Synthesis of Amine Derivatives from a “One-Pot” Synthesis of Biphenyl-4-methylazide

by

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Submitted in Partial Fulfillment of the Requirements

for the Degree of

Master of Science

in the

Chemistry

Program

YOUNGSTOWN STATE UNIVERSITY

August, 2015

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Dr. Peter Norris, Thesis Advisor Date

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Dr. John A. Jackson, Committee Member Date

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Dr. Douglas T. Genna, Committee Member Date

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Dr. Sal A. Sanders, Dean of Graduate Studies & Research Date

Thesis Abstract

This thesis explores the synthesis of amine derivatives such as amides and imines from biphenyl-4-methylazide using Staudinger chemistry. A robust one-pot synthesis of biphenyl-4-methylazide was first achieved through the use of *p*-nitrobenzenesulfonyl azide as the azide source. 1,2-Bis(diphenylphosphino)ethane (DPPE), which is cost-effective and readily available, was utilized as the mild phosphorus reducing agent. Over 40 examples of imines and amides have been synthesized producing yields as high as 70%.

Acknowledgements

I would first like to thank the Chemistry Department at Youngstown State University for not only the opportunity, but also for the funding of my research. I would like to thank the STEM college and the Cushwa family for awarding me the Cushwa Fellowship for my two years as a graduate student.

When I began the Masters of Science in Chemistry program at Youngstown State University two years, I was both inexperienced as well as lacking confidence in being able to carry out organic synthesis experiments and research. I would like to thank Dr. Peter Norris for not only being my chemistry advisor, but for also giving me the independence to carry out reactions and to learn from my mistakes, and quite frankly to allow me to think as a scientist. It's impossible to learn and to become a confident scientist without making mistakes; but it's even more important to learn from them. Dr. Norris knew that, and now after two years, I also now know this to be true. I can now confidently say that I can carry out organic synthesis research in any laboratory with the skills that I have learned. I would also like to thank you for the letters of recommendation for Ph.D. programs; and finally I would like to thank you for inviting us over your house for cookouts and for the fun night of bowling.

I would like to thank and express my gratitude to my thesis committee: Dr. John Jackson and Dr. Doug Genna. Thank you, Dr. Jackson, for teaching me about organic synthesis laboratory techniques, such as Flash Column Chromatography and thanks to Dr. Genna for teaching me that catalysts other than H^+ exist. That was clearly a topic of

which I was unaware of before I took your class this spring. You are the perfect addition the YSU Chemistry faculty.

In addition, I would like to thank Tim Styranec for teaching me about chemical safety, constantly ordering our laboratory chemicals and equipment, and more importantly being a great friend. Keep up the running!

I would like to thank Dr. Matthias Zeller for not only the crystal structures, but also for the time it took to actually solve the structures. I would also like to thank you for taking the time to teach me how to properly grow single crystals by vapor diffusion.

I would like to thank Dr. Sherri Lovelace-Cameroon for answering all of my questions even before I even started the MS program. I also want to express my gratitude for your time in writing a letters of recommendation for me when applying to Ph.D. programs.

I would like to thank Dr. Sal Sanders for the letters of recommendation and for your support and assistance throughout my graduate studies at YSU. Congratulations on becoming the Dean of Graduate Studies at YSU.

I would like to thank and wish the best to all past and present students who I was able to become great friends and had the pleasure to work with over the last two years. These include but are not limited to: Chris Copeland, Cephus Afeke, Philemon Ngoje, Kwaku Kyei-Baffour, Jen Moore, Heather Folkwein, Josh Murphy, Ish, Abby, Matt Torres, Matt LaLama, Caleb Tatebe, Amanda Sacco, Emmanuel Buabeng, Jordan Zaluski, J.J. Mihaly, and Aboagye Collins.

Finally, I would like to thank my family, especially my mom and dad for all their support in believing in me and offering endless encouragement throughout my whole life. I want to thank my grandparents for all their love and influence over me throughout my entire life and my brother, my best friend, and my runner partner Greg for all the support and constant advice he's given to me throughout the years. Congratulations on graduating from medical school and best of luck as a dermatologist. I look forward to seeing you live an exciting life with Amanda.

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Introduction

Azide synthesis in one-pot has important benefits when compared to a traditional two-pot azide synthesis. Some benefits include improved time and material efficiency. Additionally, it is well known that phosphines are effective mild reducing agents for the conversion of alkyl azides to alkyl amines. Developing methodology based off of a one-pot alkyl azide protocol in our lab, reacting alkyl azides with phosphines provides an effective method of synthesizing amides and imines. Therefore, having a background in azide chemistry would allow a better understanding of their use as ylide intermediates for aza-Wittig reactions.

Organic Azides

Azides are common intermediates for the synthesis of amine derivatives. Consequently, amine derivatives such as amides and imines are very common in natural products as well as in pharmaceutical products. Synthetically, there are many methods to prepare azides. Routes to azides include the insertion of the N_3 group (addition or substitution), the insertion of an N_2 group via diazo transfer, the insertion of the nitrogen atom itself via diazotization, cleavage of triazines, and finally rearrangement mechanisms.[1] Unfortunately, many of these methods are not only time consuming, but also can be extremely dangerous. Hydrazoic azid (HN_3), for example is a potentially explosive, deadly compound to handle. Therefore, safer, more efficient methodology is attractive for future research. Historically, a popular, direct method of azide synthesis has been the Mitsunobu displacement. **Scheme 2** shows an example of this one-pot reaction. Although effective and versatile, hydrozoic acid, the source of the azide, in the presence

of diethyl azodicarboxylate (DEAD) is a biohazard.[1] Although there has been research dedicated to finding new methods, some have disadvantages. For example, it was reported that replacing DEAD with triphenylphosphonium anhydride trifluromethanesulphonate is more less toxic.[2] Additionally, alcohols treated with triphenylphosphine and DMSO lead to azides.[2] Unfortunately, using this method, alkenes are produced when using secondary alcohols.[2] Another popular method is with a two-pot synthesis approach utilizing sulfonyl chlorides.[3] **Scheme 2** shows an example of this two-pot synthesis. Although effective, this method can be time consuming.[3]

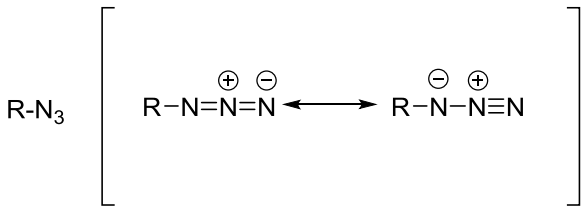
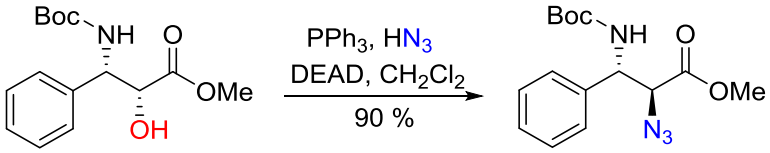
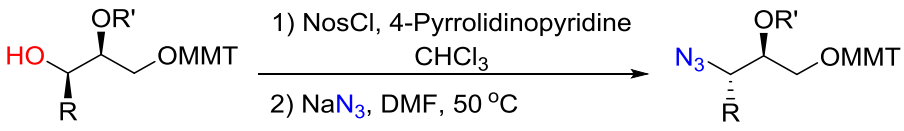


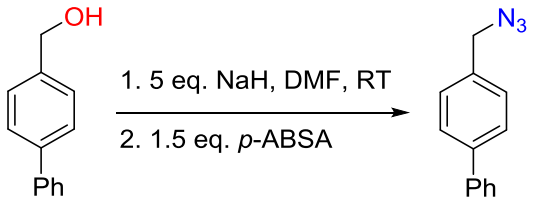
Figure 1: Resonance Structures of Alkyl Azides



Scheme 1: The classic one-pot synthesis using the Mitsunobu Reaction[1]

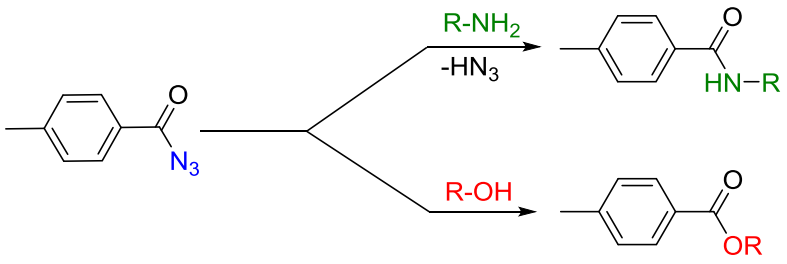


Scheme 2: Classic two-pot synthesis of alkyl azides using sulfonyl chlorides[3]



Equation 1

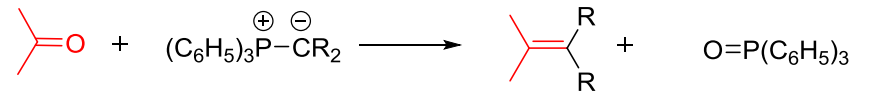
Within the specific research with azides at Youngstown State University, The Norris group have synthesized aryl sulfonyl azides, which are utilized as the azide source for efficient one-pot reactions. Previously, research was done on treating various phenyl-containing alcohols with azide compounds such as 4-nitrobenzene sulfonyl azide (*p*-NBSA), and 4-acetamidobenzene sulfonyl azide (*p*-ABSA) dissolved in a polar aprotic solvent such as DMF.[4] First, a strong base such as sodium hydride or *n*-butyllithium is added to deprotonate the alcohol making it more nucleophilic. After the addition of the electrophilic *p*-ABSA or *p*-NBSA, the nucleophilic alkoxide displaces the azide in solution forming a sulfonyl ester. The last step of the one-pot synthesis involves the now nucleophilic azide in solution attacking and displacing the sulfonyl ester in an S_N2 type reaction.[5] The mechanism for this one-pot synthesis is on page 16 (**Figure 7**). A reaction schematic published recently in this lab is shown above (**Equation 1**).[4] Additionally, research has been reported on the synthesis of esters and amides from acyl azides. In this research, 4-toluoyl azide was reacted with amines and alcohols to form the amides and esters respectively. See Equation 2 below.[6]



Equation 2

Classic Wittig Reaction

The Wittig reaction has historically been an efficient way of synthesizing alkenes. Carbonyl compounds react with phosphorus ylides to produce alkenes (either *E* or *Z*) as well as a phosphine oxide. Ylides are neutral compounds with opposite charges on adjacent atoms. A common example of the Wittig reaction is a depicted below. [7]



Equation 3: Classic Wittig Reaction[4]

In this reaction, the ylide acts as the nucleophile, attacking the carbonyl of the aldehyde or ketone. Next, an oxaphosphetane, an unstable four-membered compound collapses yielding the alkene plus the phosphine oxide.[7]

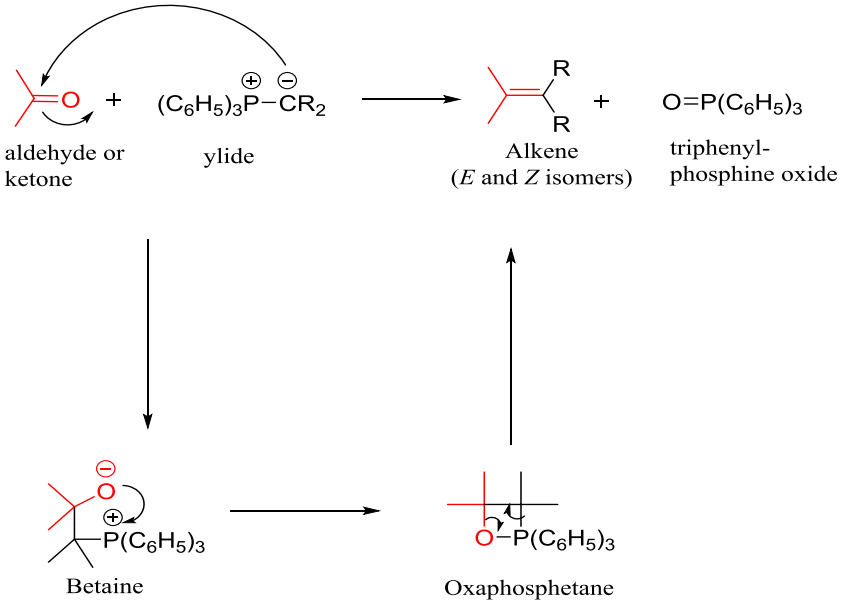


Figure 2: The Classic Wittig Mechanism Scheme[7]

Staudinger's Reduction

Although Wittig reactions are commonly seen with carbon-phosphorus ylides, an ylide can also be made using a nitrogen in place of a carbon. In 1919, Hermann Staudinger reacted azidobenzene with triphenylphosphine; this subsequently formed an ylide named an *iminophosphorane* with loss of nitrogen gas.[8] Therefore, the treatment of an azide with a phosphorus reducing agent creates the ylide needed to synthesize amine derivatives. The Staudinger reduction is a very common method in reducing azides to amines.[1] The classic Staudinger reduction is below in Figure 3. Recently, the Staudinger ligation, which converts the ylide to amides has become useful in biochemistry.[9] A mechanism for the formation of the iminophosphorane follows.

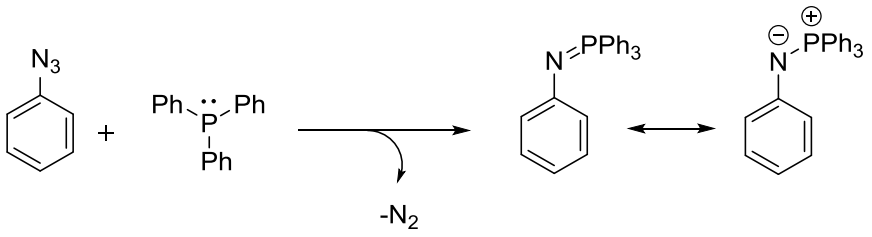


Figure 3: Staudinger's reduction in 1919

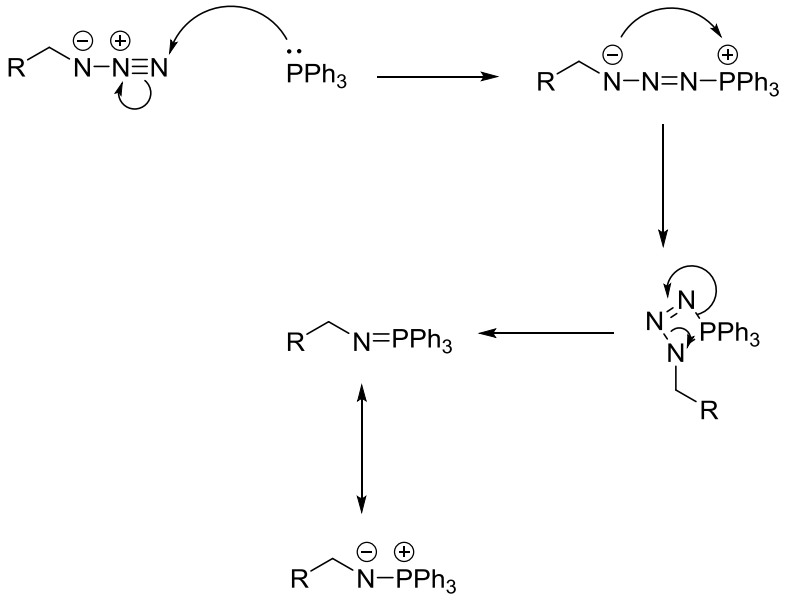


Figure 4: Mechanism for the formation of iminophosphorane[10]

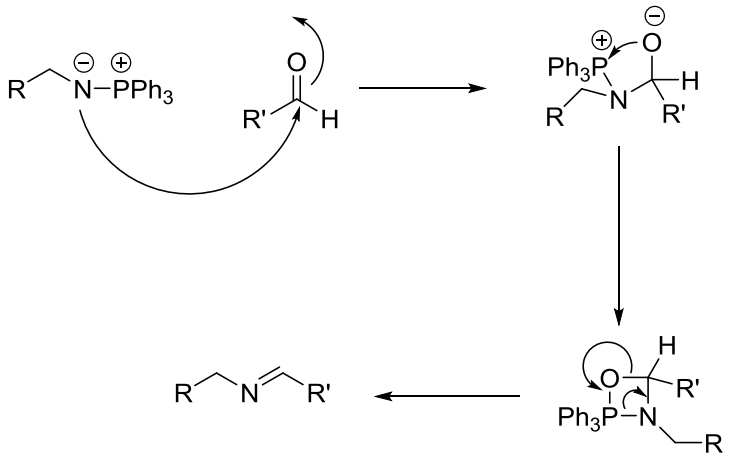
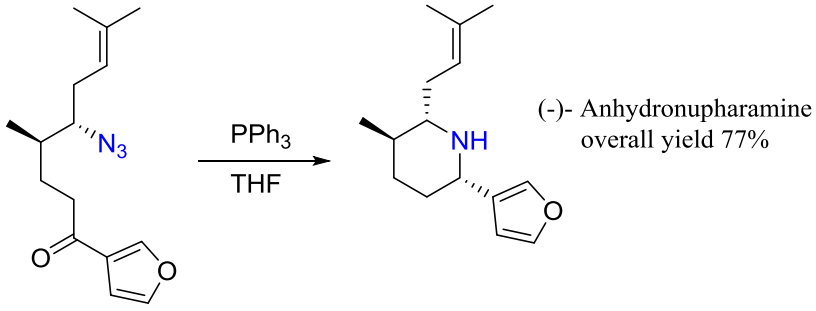
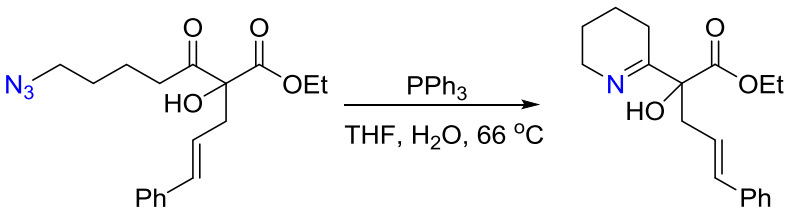


Figure 5: Mechanism for the formation of imines via aza-Wittig



Equation 4: Staudinger Reduction with PPh_3 in a natural product synthesis[11]

Equation 4, which is shown above, is an example of an intramolecular aza-Wittig reaction (followed by reduction) used in a natural product synthesis.[11] **Equation 5** depicts an intramolecular aza-Wittig reaction in Wood's 2012 synthesis of allosecurinine.[12] The Staudinger reactions have become popular within natural product chemistry due to the chemoselectivity and orthogonality of azide and phosphine compounds.[9]



Equation 5: A step in the synthesis of allosecurinine[12]

Versatility of Imines

As mentioned previously, reductions of azides are of great importance in the pharmaceutical industry because of their transformation to amines. However, another important functional group is the imine, which contains a C=N linkage. The imine functionality is of great importance in biochemistry and organic synthesis. In biochemistry, the imine group is also known by the name Schiff base.[13] The transamination of pyridoxal-5-phosphate (PLP) to pyridoxamine phosphate (PMP) forms a Schiff base via a covalent bond between PLP and an enzyme. The transimination is the first step of the conversion to PMP (aminated enzyme), which also yields a keto acid.

Like azides, imines are also versatile chemical compounds. Research has focused on using the aza-Wittig reaction to make imino acids. Reacting the P=N ylide with aldehydic and ketonic acids (as opposed to aldehydes and ketones) is an effective way to create imino acids.[14] In fact, current research attempts to react barbiturates with phosphoro aza-ylides to create imino-barbiturates. Reaction of barbiturates with azides yields amine derivatives.[15] Also, reacting fluorinated ylides (N=P) with carbonyls yields

various imines in good yields. Using fluorine affects the biological activity as a result of its high electronegativity and electron-withdrawing capability.[16]

In addition, imines are of great importance in organocatalysis. The Pictet-Spengler reaction[17], as well as the Mannich reaction[18] utilize the imine functional group when forming highly enantioselective products. Thomas Lectka has utilized imines in the synthesis of *β*-lactams[19] and Jonathan A. Ellman has utilized *N-tert*-butylsulfinyl imines as intermediates in the asymmetric synthesis of amines.[20] These examples are just a sampling of the versatility of both synthesizing imines as well as reducing imines and azides.

aza-Wittig is of great importance in fields such as natural product synthesis as well as chemical biology. For example, the aza-Wittig reaction is of great synthetic value in total syntheses, such as (-)- pancracine, (-)- coccinine, lavendamycin, as well as (+/-)-selaginoidine.[11] In addition, the total syntheses of croomine, quinine, and dendrobine utilize aza-Wittig Chemistry.[21] Total syntheses that use an aza-Wittig scheme sometimes have the advantage of using less protective group chemistry because the azide and phosphine are selectively reactive towards each other. This can sometimes allow more functional groups to be tolerated with less need to protect groups.[21] For example, the already introduced step in the synthesis of allosecurinine (**Equation 5**) didn't require the alcohol to be protected prior to the aza-Wittig reaction.

The utilization of a phosphorus compound to create ylides could potentially lead to a variety of nitrogen-containing functional groups. As shown by previous research,

alcohols can easily be converted to azides in a one pot synthesis.[4] Derivatives such as *p*-NBSA as well as *p*-ABSA act as convenient reagents for the S_N2 conversion to the azide.

Phosphine Reducing Agents

Historically, triphenyl phosphine has been utilized in the Staudinger sequence. Upon addition of water, triphenyl phosphine oxide is formed as a by-product. This can then be removed from the product mixture via flash chromatography. Recently 1,2-bis(diphenylphosphino)ethane (DPPE) (**Figure 6)** has been used in place of triphenyl phosphine in the Staudinger sequence.[22,23] Like triphenylphosphine, DPPE is commercially available. The bis(phosphine oxide) by-product is more polar than triphenylphosphine oxide with both phosphorus compounds oxidized. Therefore separation should be more convenient. Additionally, DPPE has proven successful with the Mitsunobu reaction. Although polymer-bound triphenylphosphine has been found to be very successful, it is limited in terms of cost and large scale reactions.[23]

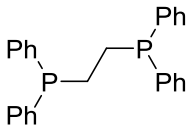
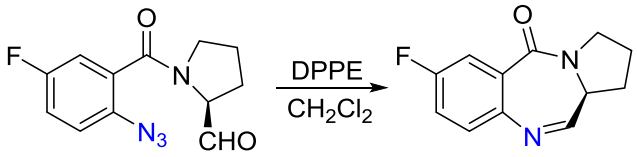


Figure 6: 1,2-bis(diphenylphosphanyl)ethane (DPPE)

DPPE has been used successfully in place of triphenylphosphine in cyclizing pyrrolo-[1,4]-benzodiazepines.[23] An intramolecular aza-Wittig reaction takes places where an imine is formed. An example of such a reaction is below (**Equation 6**).



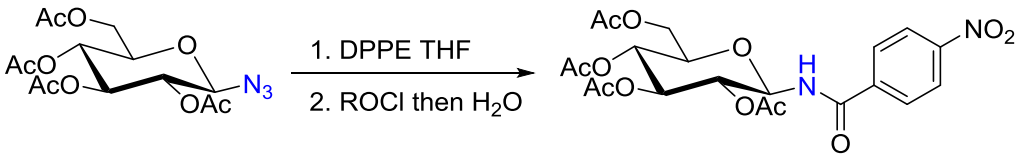
Equation 6: The aza-Wittig intramolecular formation of an imine[22]

Staudinger Ligation (Amides)

Bertozzi and Saxon introduced *Staudinger Ligation Chemistry* in 2000.[9] The importance of the Staudinger reaction to biological systems arises in that azides and phosphines do not occur in natural compounds, i.e. they are bioorthogonal. Furthermore, bioorthogonality implies that the functional groups (i.e. azide and phosphine) don't interfere with the natural chemical processes in living species.[9] In addition, although reactive, azides undergo selective ligations. Finally, the small size of azides allows them to be added to biological compounds without changing the size drastically. The aza-Wittig reaction does not need a catalyst for the azide to react with the phosphine. Although "click chemistry" only required heating when first explored, some present "click chemistry" procedures utilize a copper catalyst to perform the [3+2] cycloadditions.[21]

The use of DPPE in Staudinger ligation methods has been examined with glycosyl azides.[24] Interestingly enough, DPPE has been found to be an effective method in making β-*N*-glycosyl amide motifs [24] as is shown in **Equation 7**. The glycosyl amide functionality has become of great importance for a variety of reasons. Its usage in carbohydrate chemistry, in particular with carbohydrate scaffolds, as well as the ability to

act as donors for an array of biological reactions, are a few examples of the utility of the amide functionality. [24]



Equation 7

As a result of its bioorthogonality, the Staudinger ligation has also found uses in protein labeling of both *in vitro* and *in vivo* cells.[25] The non-toxicity of azides and phosphines at standard concentrations makes them viable candidates for protein labeling in living species.[25] As mentioned before, the small size of azides allows very insignificant perturbations in the normal functioning of a protein to be labeled.[26]

Limitations of the Staudinger Ligation

Although the chemoselectivity of the Staudinger ligation (as with the Staudinger reaction) is extremely useful in organic synthesis, there are limitations. The reaction kinetics of the Staudinger ligation are extremely slow, with rate constants at 0.0020 M/s.[10] This may relate to the possible steric hindrance of the phosphine at the ligation site. Kinetic studies show that the rate-determining step is the attack of the phosphine compound on the azide.[10] Research has attempted to speed up the reaction by increasing the nucleophilicity of the phosphine reagents by increasing the number of electron-donating groups. This has proven to be a challenge; air oxidation of the phosphine compounds tends to increase as electron- donating groups are added.[10] However, thiol-phosphine compounds such as (diphenylphosphino)methanethiol have proven to be

effective in the Staudinger ligation, with an amide yield of 95% being reported.[27] However, due to the oxidation of the phosphine compounds, higher concentrations of the phosphine reagent are needed.[10] As a whole, these reasons tend to lower the reaction yield. As a result of the slow reaction times for the Staudinger ligation, researchers are investigating complementary reactions, such as click chemistry reactions.

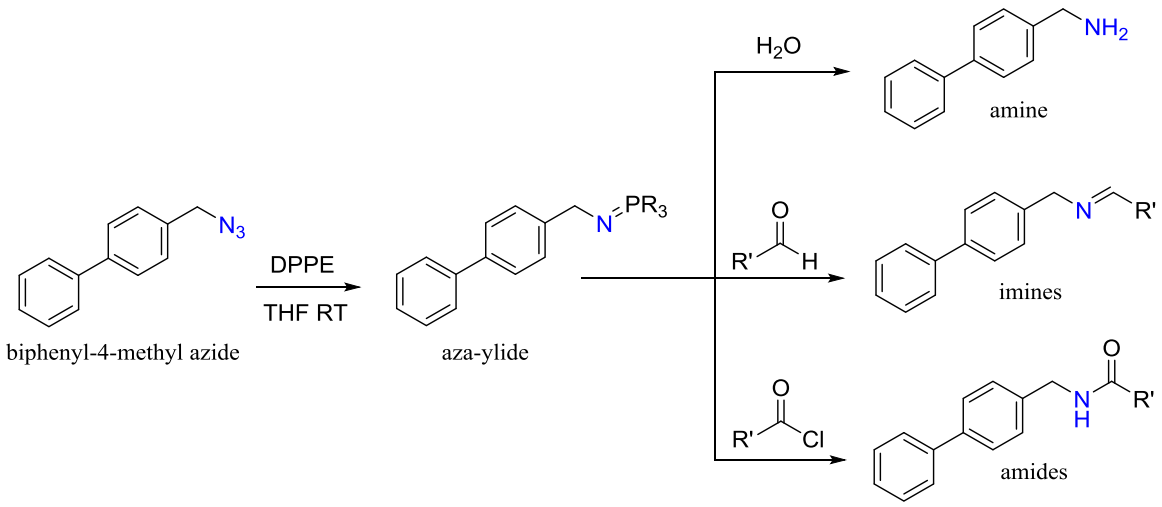
The aza-Wittig reaction is now a very powerful and efficient method of preparing amine derivatives. An appreciation was gained for the contributions of Wittig and Staudinger chemistry of the past and present. It was the goal for this research to provide future researchers in organic synthesis, medicinal chemistry, chemical biology, as well as biochemistry, a convenient synthetic route to important amine derivatives such as amides and imines. The novel "one-pot" synthesis of alkyl azide from *p*-NBSA can provide a fast, efficient route to the versatile azide functional group. Although triphenylphosphine has historically been a popular, cost-effective phosphine in Staudinger chemistry as well as aza-Wittig chemistry, the triphenylphosphine oxide by-product has proven to be a tremendous obstacle in removing during purification. There has been a very limited amount of research with the utilization of DPPE as the phosphine reducing agent in a Staudinger sequence. The more polar DPPE oxide by-product can be less burdensome to remove during purification. In conjunction with the efficient alkyl azide synthesis, this research hopes to explore DPPE as an attractive, efficient replacement to triphenylphosphine in a Staudinger and/or aza-Wittig reaction sequence.

Statement of Problem

The azide functional group is very versatile and useful in many fields of chemistry. However, traditional methods of installing the azide group can require the handling of dangerous reagents, as well as be both time-consuming and not material-efficient. The first part of this research deals with an efficient and convenient one-pot method for the installation of the azide group from a readily available alcohol. The second portion of this research explores the use of DPPE as an alternative reducing agent to the popular triphenylphosphine in a Staudinger sequence. Triphenylphosphine, although cost-effective and convenient, can propose major obstacles when attempting to remove the triphenylphospine oxide by-product during purification. On the other hand, the more polar DPPE oxide by-product is less strenuous to remove. It is the goal that this DPPE research in conjunction with the alkyl azide "one-pot" method, offers an attractive, alternative route for those researching the Staudinger reduction, the Staudinger ligation, as well as aza-Wittig chemistry.

Results and Discussion

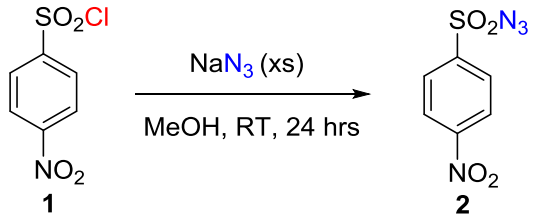
The overall goals of the research are presented below in **Scheme 3.** The mild reduction of biphenyl-4-methyl azide to the corresponding aza-ylide with DPPE allows amine derivatives such as imines and amides to be accessed.



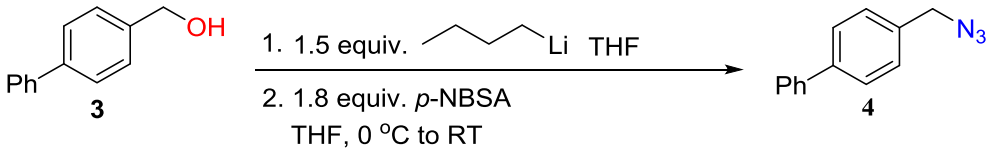
Scheme 3

One-Pot Synthesis of Biphenyl-4-methyl azide using *p*-NBSA

Research commenced with the synthesis of *para*-nitrobenzenesulfonyl azide (*p*-NBSA) (**Equation 8**). This was achieved by reacting *para*-nitrobenzenesulfonyl chloride with excess sodium azide in methanol at room temperature overnight (**Equation 8**). After *p*-NBSA was synthesized, it was utilized as the azide source in the one-pot synthesis of biphenyl-4-methylazide from biphenyl-4-methanol (**Equation 9**).



Equation 8



Equation 9: One-Pot Synthesis of Biphenyl-4-methyl azide **4** from **3**

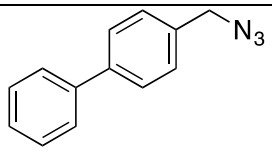
Compound Number	**Azide Structure**	**Proton CH_2 Shift (ppm)**	**IR Azide band (cm^{-1})**
4		4.38	2106

Table 1: Experimental data for Azide **4**

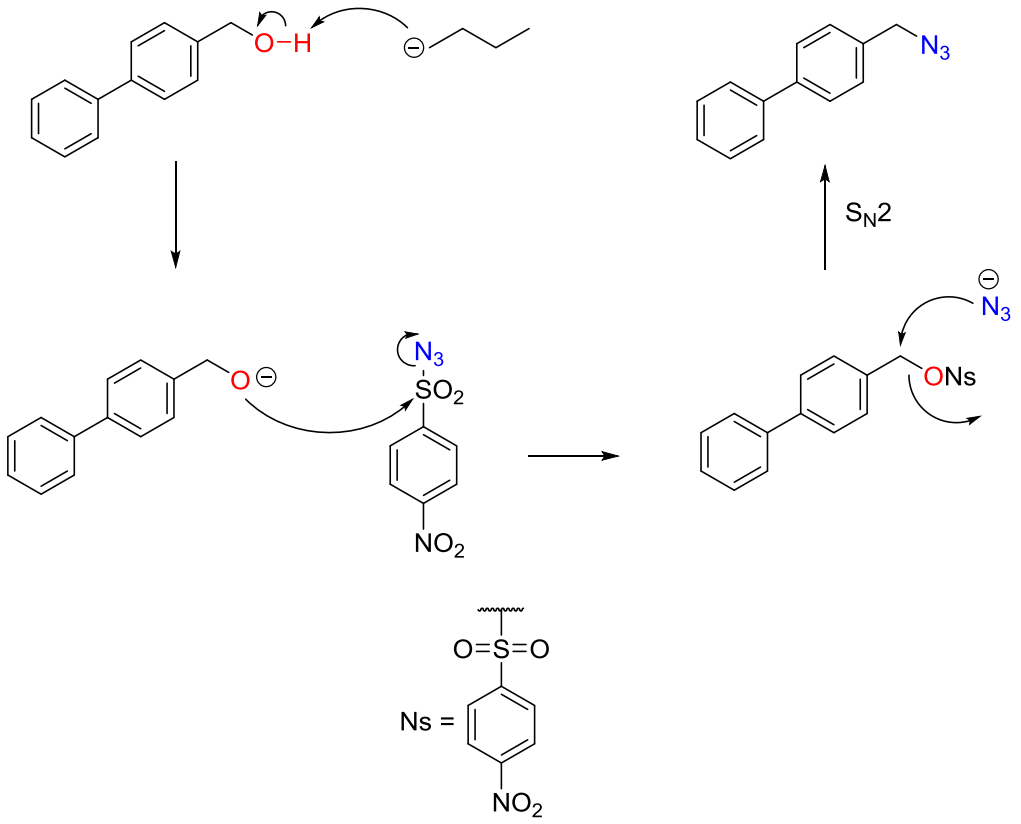


Figure 7: Mechanism for the synthesis of alkyl azide **4**

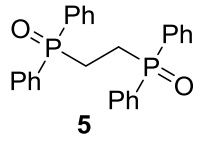
Biphenyl-4-methanol **3** was chosen for this research because it is relatively inexpensive (10 g-$43.00), as well as commercially available. In addition, the primary benzylic alcohol is easily converted to a nosyl ester in situ conveniently. Sulfonyl esters lend themselves well with S_N2 reactions with azides. Mechanistically, the first step involves butyl lithium deprotonating alcohol **3**, making it a better nucleophile. After the addition of *p*-NBSA, nucleophilic **3** performs nucleophilic sulfonylation kicking out the azide and forming a temporary nosyl ester intermediate. Finally, the now nucleophilic azide in solution attacks the nosyl ester via an S_N2 reaction yielding azide **4**. The mechanism is outlined in **Figure 7** above. Examination of the ^{1}H NMR spectrum of pure biphenyl-4-methanol revealed a methylene signal at 4.67 ppm. After the successful

synthesis of the biphenyl-4-methyl azide, the ^{1}H NMR signal for the methylene group shifted from 4.67 ppm to 4.38 ppm. This is indicative of the shielding of the $–CH_2$ group by the azide compared to the alcohol which deshielded the protons. The reaction progress was monitored with TLC and *p*-anisaldehyde stain. Successful synthesis of the alkyl azide was confirmed with a yellow spot on TLC, with a 1:1 hexane/ethyl acetate solution as eluent after 90 minutes (1 mmol scale). Employing IR spectroscopy, a sharp signal at 2010 cm^{-1} indicated the successful synthesis of the alkyl azide.

After the successful synthesis of the alkyl azide, a Staudinger reaction was employed to first create the ylide. The strategy was to (DPPE) as the mild nucleophile and reducing agent. This was achieved by adding the azide in solution dropwise to 0.5 mmol of the DPPE in DMF or THF. Progress of the reaction was monitored with TLC staining with *p*-anisaldehyde; the disappearance of the yellow azide spot indicated the reduction of the azide. A second spot appeared closer to the baseline of the TLC plate; this indicated that a new product of greater polarity was formed. Although ylides are stable and can be isolated, isolation proved to be difficult as phosphine compounds are susceptible to oxidation and hydrolysis. Examination of ^{1}H NMR spectra showed the presence of a new methylene signal at 4.9 ppm. This is indicative of the deshielding effect of the N=P bond on the $–CH_2$ portion of the alkyl group.

Although amines, amides and imines were the final goal of the research, the isolation and determination of the bis-phosphine oxide by-product was important in the identification of TLC spots. Just as the classic Wittig reaction produces triphenylphosphine oxide as the by-product, the aza-Wittig also produces a phosphine oxide by-product. Examination of the pure DPPE with ^{31}P NMR revealed a singlet at -

12.46 ppm; it was expected that the phosphine oxide signal would appear deshielded. This is exactly what was observed with the new singlet in the ^{31}P shift at 32.52 ppm. ^{1}H NMR showed the shift of the two methylene groups from 1.5 ppm to 2.5 ppm and the X-ray crystal structure (**Figure 8**) confirmed the by-product. With this result, it was decided that ^{31}P NMR spectra would be run when attempting to purify initial amine derivatives. Disappearance of the ^{31}P NMR peak at 32.52 ppm would indicate the separation of this by-product from the crude mixture. TLC with ninhydrin stain proved to be a visual aid in separating the unreacted DPPE as well as the phosphine oxide by-product. The phosphine compounds burned as purple spots; this allowed purification of the products to be easier to visualize.



5

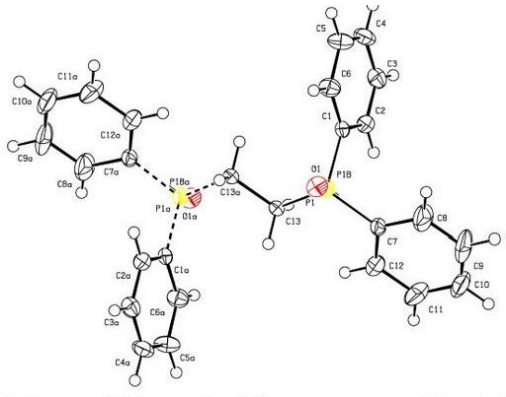


Figure 8: X-Ray crystal structure of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

Although THF worked well as the solvent for the conversion of the alkyl alcohol to the azide, the reaction progress slowed drastically when DPPE was added, both alone and in combination with the acyl chlorides in attempts at forming the amide linkage. The classic Staudinger reduction uses THF as the solvent of choice when making amines from pure azides, which likely led to faster reaction kinetics. However, with the attempted one-pot synthesis, the azide was not in its pure form. Instead it was in a solution with various

salts as well as other compounds, such as unreacted *p*-NBSA. Therefore, it was reasonable to expect reaction times to be drastically slowed as a result of the vast amount of molecules in this solution also reacting with the nucleophilic DPPE. Because the rate-determining step of the *aza*-ylide is the initial attack of the phosphine compound on the azide, the nucleophilicity of the DPPE was likely hindered by the array of other molecules in the solution such as *p*-NBSA. After one-pot attempts with THF yielded 0% amide after 10 days, a different solvent of choice was used. In fact, after 10 days of the azide/DPPE /acyl chloride stirring at room temperature, TLC showed that there was still azide which didn't react. Examination of the ^{1}H NMR further showed a lack of complete conversion of the azide to the ylide (**Table 2**). With nitrogen gas being a great leaving group, it was determined that the phosphine was not attacking the azide like it should. The nitrogen gas would only leave after the initial rate-determining step occurred.

Superior reaction kinetics with the Staudinger ligation have been reported as the polarity of the solvent increased.[27] In addition, research indicates a second order rate constant of 0.040 mol/S for the aza-Wittig reaction. Excess phosphine is sometimes used to speed up the reaction. A more polar solvent (DMF) was employed as well as excess DPPE. It was anticipated that the excess phosphine molecules would allow a greater probability of coming in contact with the azide in solution. However, with this added DPPE, additional TLC spots were seen.

Side Products from Early One-Pot Studies

Although the Staudinger ligation is chemoselective with azides and phosphines, this can prove problematic if leftover *p*-NBSA is present to react. Because *p*-NBSA also contains an azide, the DPPE can selectively attack in the same manner as with the biphenyl-4-methylazide major product. This was confirmed by mixing *p*-NBSA and DPPE in DMF. TLC in 1:1 hexanes/ethyl acetate revealed that the standard DPPE, which appears as a white spot with *p*-anisaldehyde stain, reacts with the *p*-NBSA causing the white color to disappear. TLC also revealed new products formed which appeared closer to the baseline. This likely indicates a polar ylide is formed with loss of nitrogen gas. Proposed mechanisms for the formation of the side products **6**, **7**, and **8** are shown in **Figure 9**, **Figure 11**, and **Figure 12** respectively. The mechanism for the formation of Compound **6** was proposed after an X-ray crystal structure was obtained (**Figure 10**). Side products **7** and **8** were proposed on the basis of proton NMR shifts and integrations. **Table 2** summarizes the attempted one-pot synthesis of amides and imines from **3**.

After a review of prior research, as well as analysis of this current research, it was determined that purifying the alkyl azide via flash chromatography was the most efficient route to achieve the desired results.[28] Alkyl azide **4** was synthesized in 10 mmol scales and subsequently added stoichiometrically in the next step. After the purification, reactions were attempted with both DMF and THF as solvents. Both yielded the desired amide linkage; however DMF was faster, but THF was easier to remove. **Tables 3** and **4** summarize the successfully synthesized imines and amides respectively.

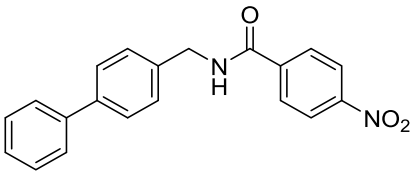
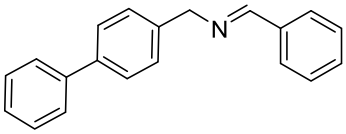
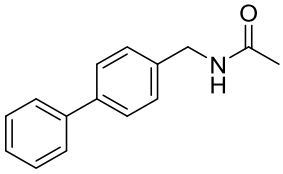
Anticipated product	Yield/comments THF, RT, 10 days	Yield/comments DMF, RT, overnight
	0% yield after 10 days. No sign of azide reacting with DPPE on TLC.	0% after 5 hours. Azide reacted with DPPE on TLC but failed to produce the amide. Compound **6** was produced instead.
	0% yield after 10 days. No sign of azide reacting with DPPE on TLC.	0% after 5 hours. Azide reacted with DPPE but failed to produce the imine. Compound **7** was produced instead
	0% yield after 10 days. No sign of azide reacting with DPPE on TLC.	0% after 5 hours. Azide reacted with DPPE on TLC but failed to produce the amide. Compound **8** produced instead.

Table 2: Preliminary Results for attempted one-pot synthesis of amides and amines.

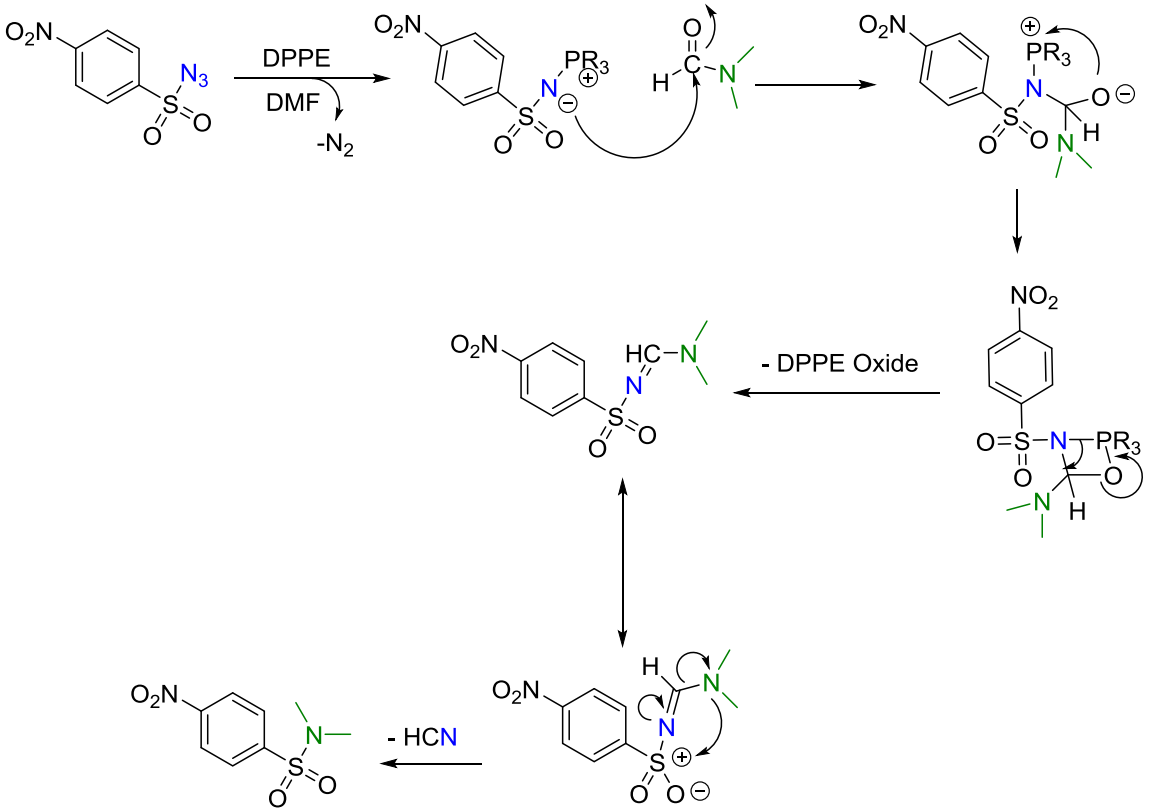


Figure 9: Proposed mechanism for the formation of **6**

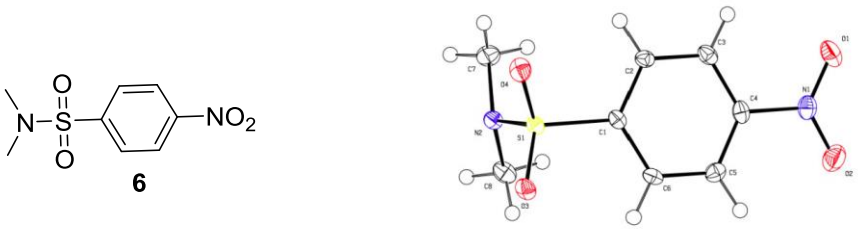


Figure 10: X-Ray crystal structure of **6**

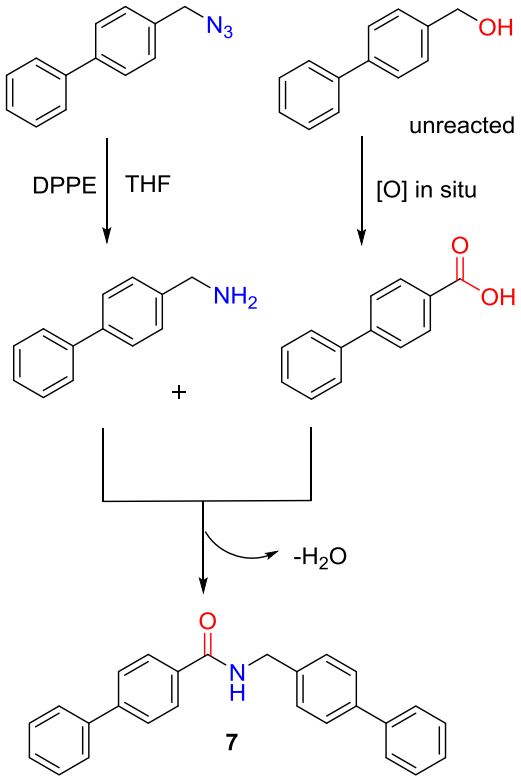
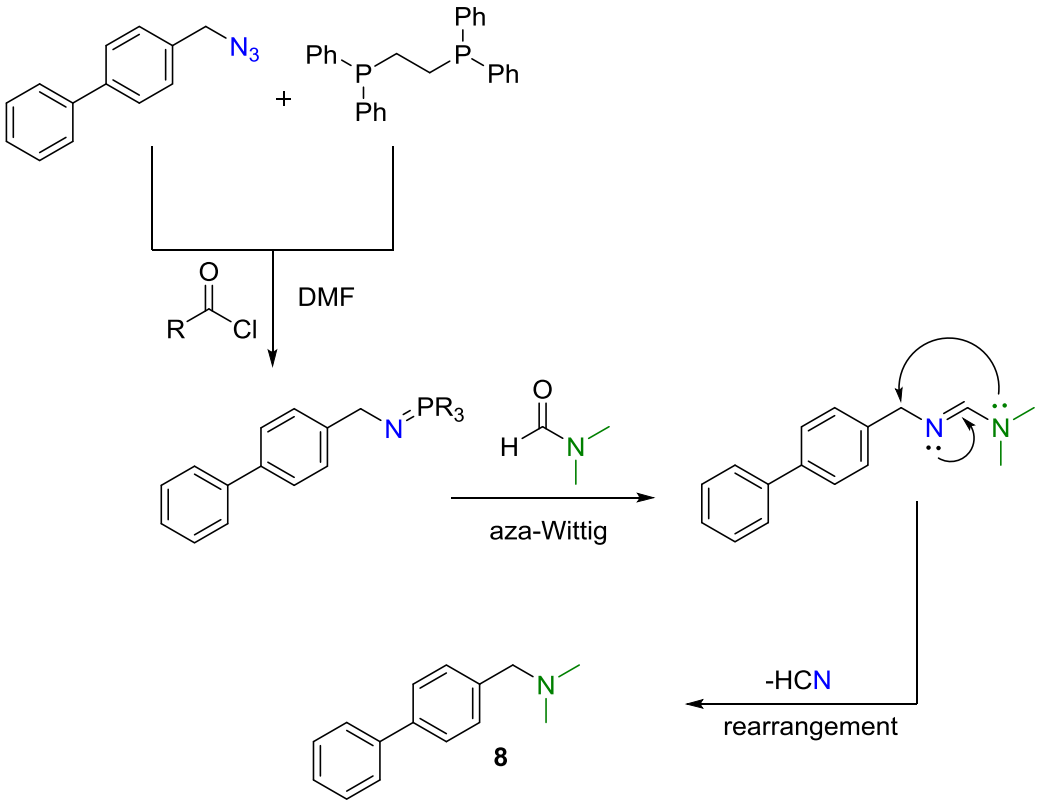


Figure 11: Proposed explanation for the formation of **7**

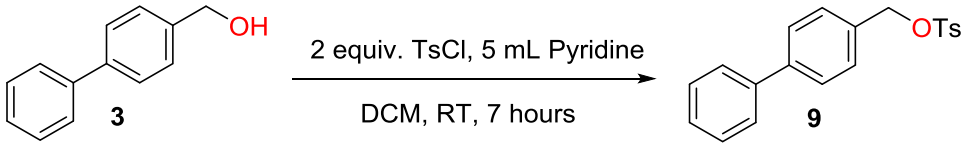


1-([1,1'-biphenyl]-4-yl)-*N*,*N*-dimethylmethanamine

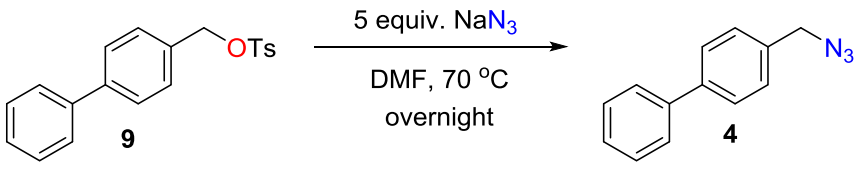
Figure 12: Proposed mechanism for the formation of **8**.

Parallel Two-Pot Synthesis of Biphenyl-4-methyl Azide

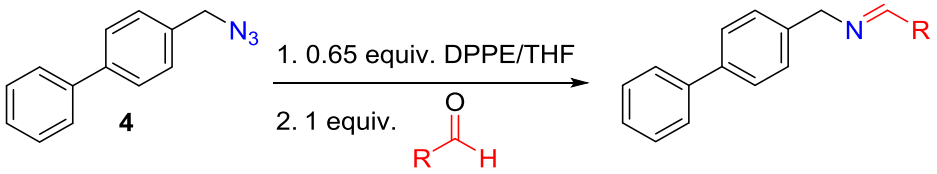
A difficulty of one-pot reactions is identifying the multiple signals that appear on ^{1}H NMR spectra. Therefore an independent synthesis of biphenyl-4-methyl azide **4** from biphenyl-4-methanol **3** was performed. Instead of the utilization of *p*-NBSA as the azide source, sodium azide was used. Biphenyl-4-methanol was first mixed with tosyl chloride in pyridine; thus forming the tosyl ester **9** (**Equation 10**). The tosyl group is a much better leaving group than the alcohol group. With a 1:1 hexane/ethyl acetate solution, a new non-polar spot was seen farther away from the baseline on TLC from the starting alkyl alcohol **3**. The formation of compound **9** was confirmed on ^{1}H NMR with the methylene singlet shift from 4.67 ppm (alcohol **3**) to 6.17 ppm (tosyl ester **9**). Thus, after the indication of the conversion to the tosyl ester **9**, sodium azide was then added producing the alkyl azide upon heating at 70 ^{o}C in DMF. The methylene ^{1}H NMR shift (4.4 ppm) was consistent with the same azide **4** formed from the one-pot method. After the alkyl azide was confirmed via ^{1}H NMR and IR, DPPE was added in the same manner as the one-pot synthesis.



Equation 10: Synthesis of ester **9** from alcohol **3**.



Equation 11: Synthesis of azide **4** from ester **9**.



Equation 12: The synthesis of aldimines from azide **4**.

Synthesis of Imines from Alkyl Azide 4

The Staudinger ligation of imines was explored through the reaction of the alkyl azide **4** with aldehydes (**Equation 12**). An advantage of using aldehydes is the ease of monitoring reactions with proton NMR (**Figure 13**). The proton shift of the aldehyde (around 10 ppm), to the imine (8.19 to 9.61 ppm range) is very noticeable. ^{13}C NMR also confirmed the formation of the imine with the N=CH carbon shift ranging from 151.59 to 165.98 ppm depending on the R group. That was not the case for the amide bond formation (N-H-C=O) which was hidden amongst the aromatic region prior to purification. Integration of the proton NMR signals confirmed the successful formation of the imine linkage with a 2:1 ratio of the methylene hydrogens to the imine proton.

Although the synthesis of the aldimines worked well, purification of imines can be very difficult. This difficulty arises from imine susceptibility to acid hydrolysis back to the aldehyde and amine respectively. This was evident when attempting to monitor the imine reactions on TLC. The slight acidity of the silica decomposed the imine back to the aldehyde. Furthermore, purification via flash chromatography was initially unsuccessful as it decomposed the imine back to the aldehyde. This was confirmed by proton NMR which showed the shift of the imine (8.4 ppm) back to the aldehyde (10 ppm) and with

the methylene portion of the imine (at 4.9 ppm) shifted to 5.2 ppm. If the crude product couldn't be recrystallized, then triethylamine had to be added to the acidic silica to prevent decomposition of the imine. Crude syrups were difficult to purify in any yield because they couldn't be immediately recrystallized. Crude syrups were almost always attributed to unreacted aldehyde by ^{1}H NMR. Alternate paths were attempted in removing unreacted aldehydes so a crude solid could be recrystallized. Employing the Cannizaro reaction,[29] saturated calcium carbonate was added to the crude imine and added to a separatory funnel. The imine was extracted with ethyl acetate into the organic layer, whereas the aldehyde should decompose into the aqueous layer. This had minimal success. Another method attempted involved the usage of the polymer-bound aldehyde scavenger Tosyl Hydrazine.[30] However, these scavengers are very expensive. If the aldehyde still remained, flash chromatography was employed as a last resort. Significant yields were only obtained by purifying through recrystallization.

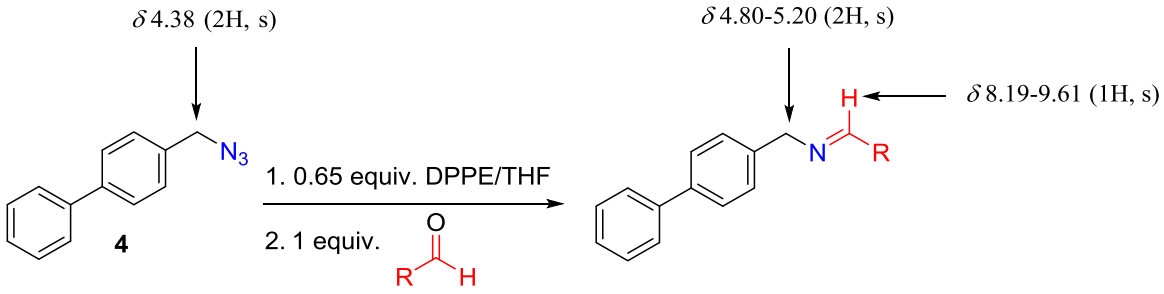
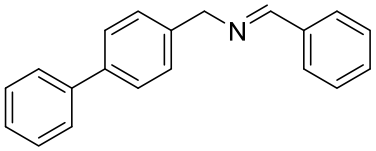
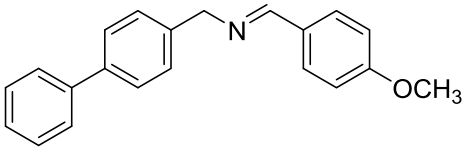
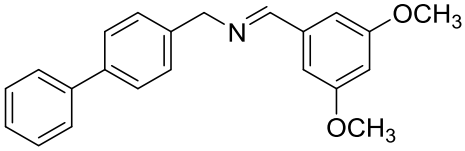
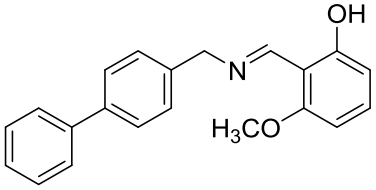
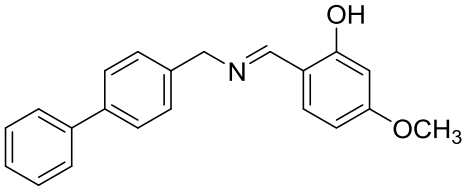
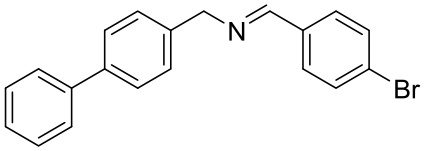
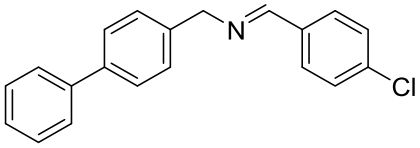
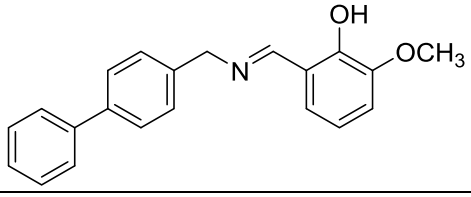
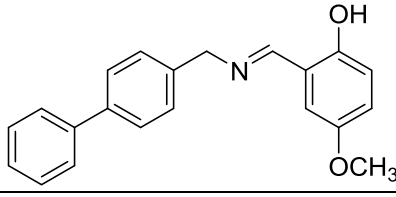
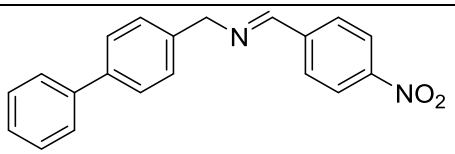
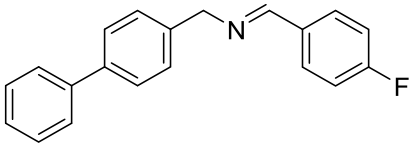
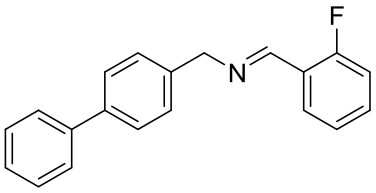
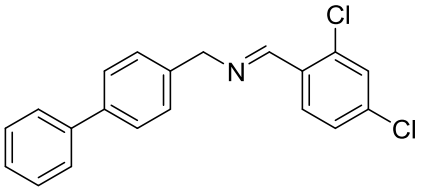
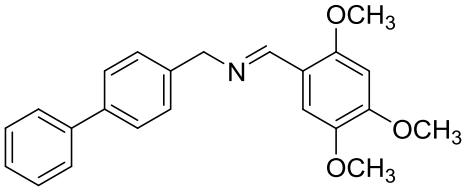
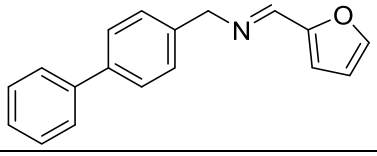
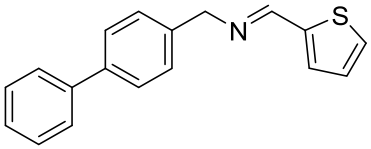
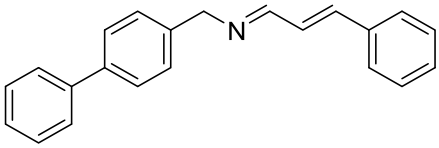
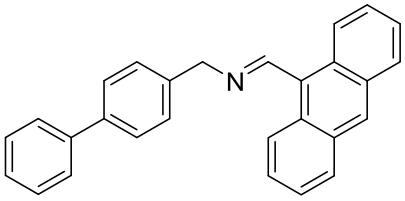
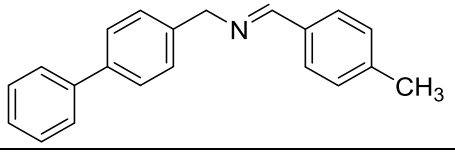


Figure 13: Monitoring the CH_2 shift and imine proton shift in proton NMR.

Imine Product Number	Imine Product	CH_2 shift (ppm)	N=CH shift (ppm)	% Yield
10		4.88	8.44	1%
11		4.83	8.36	70%
12		4.87	8.35	34%
13		4.83	8.96	43%
14		4.80	8.33	44%
15		4.85	8.37	24%
16		4.86	8.39	16%
17		4.88	8.47	38%
18		4.85	8.43	50%

19		4.93	8.50	25%
20		4.85	8.40	trace
21		4.85	8.40	trace
22		4.89	8.81	44%
23		4.84	8.80	25%
24		4.83	8.21	25%
25		4.84	8.49	10%
26		4.76	8.19	40%
27		5.20	9.61	32%
28		4.85	8.40	40%

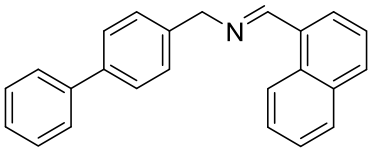
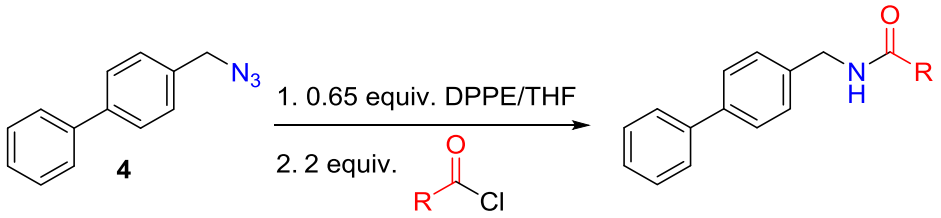
29		5.00	9.09	25%

Table 3: Synthesized aldimines with relevant CH_2 and imine proton shifts

.

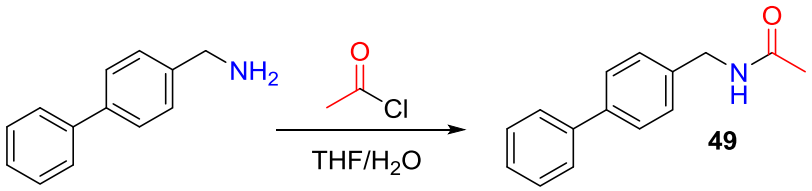


Equation 13: The synthesis of amides from azide **4**

Synthesis of Amides from alkyl azide 4.

The Staudinger ligation of amides was explored through the reaction of alkyl azide **4** with acyl chlorides. The methylene group on the azide was monitored throughout the reaction (**Figure 14**). Successful synthesis of the amide linkage was evident with the doublet signal (between 5.40 Hz and 5.92 Hz) of the methylene proton seen on proton NMR spectra. This doublet ranged from 4.48 ppm to 4.76 ppm depending on the acyl chloride used. This doublet was the result of coupling with the N-H proton from the amide directly adjacent to the methylene group. The amide N-H proton was seen on proton NMR as a broad peak ranging from 5.77 ppm to 7.0 ppm. In addition, ^{13}C NMR confirmed the presence of the carbonyl of the product for the amides ranging from 161.84 ppm to 178.38 ppm. Integration of the signals confirmed the successful formation of the amide linkage with a 2:1 ratio of the methylene hydrogens to the amide proton. The

correct proton NMR signals were confirmed through the synthesis of an amide by the classic Schotten-Baumann reaction[31] (**Equation 14**).



Equation 14: Schotten-Baumann Reaction.

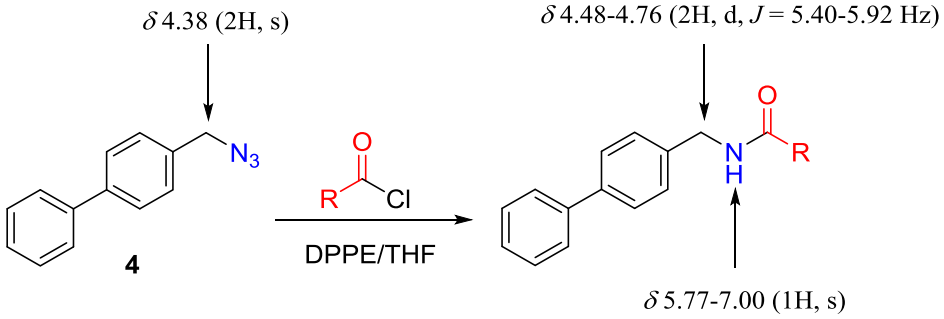
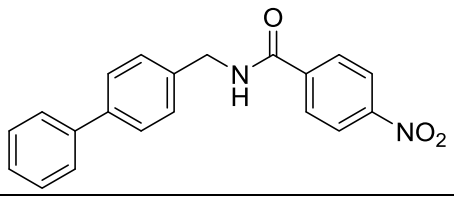
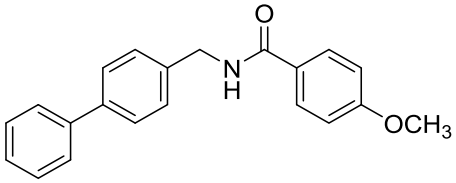
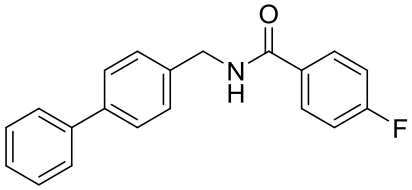
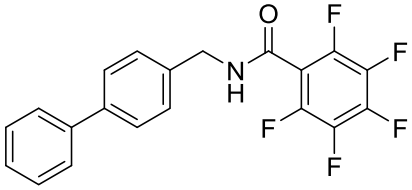
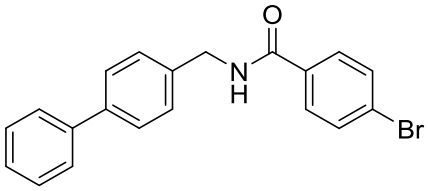
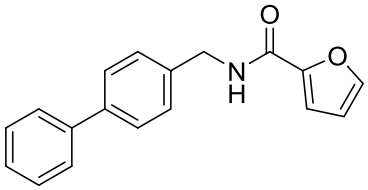
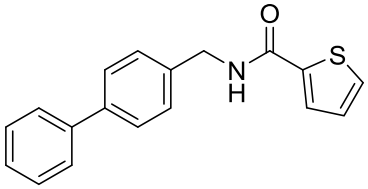
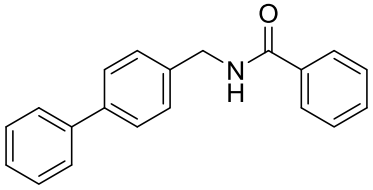
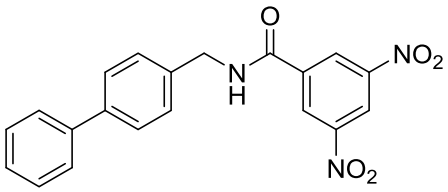
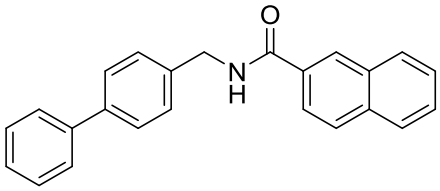
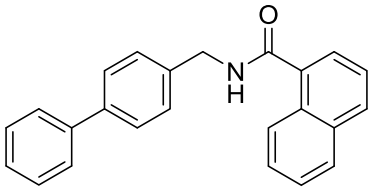
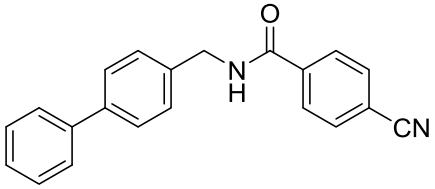
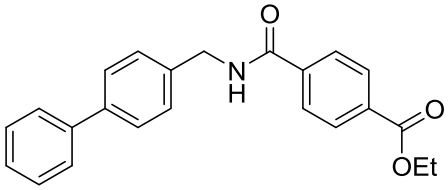
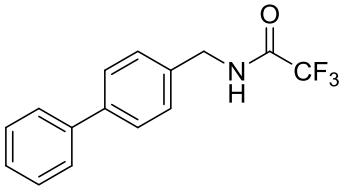
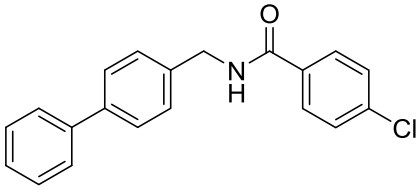
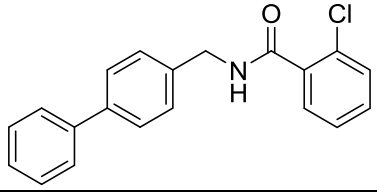


Figure 14: Monitoring the CH_2 shift and amide proton shift on proton NMR.

Amide Product Number	Amide Product	CH_2 shift (ppm)	N-H shift (ppm)	% Yield
30		4.71	6.51	33%
31		4.69	6.33	26%
32		4.69	6.39	16%
33		4.70	6.26	25%
34		4.68	6.40	27%
35		4.67	6.69	15%
36		4.67	6.37	10%
37		4.71	7.00	1%

38		4.76	6.82	16%
39		4.76	6.61	18%
40		4.79	6.32	30%
41		4.70	6.46	25%
42		4.69	6.69	5%
43		4.59	6.57	20%
44		4.69	6.40	1%
45		4.73	6.51	10%

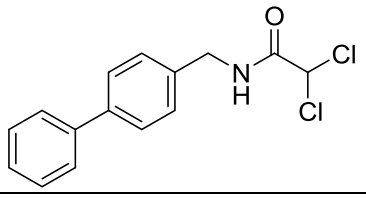
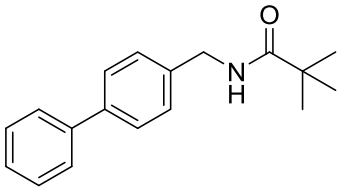
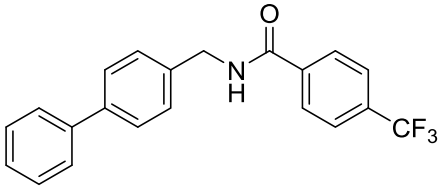
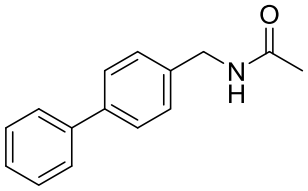
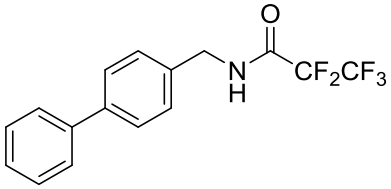
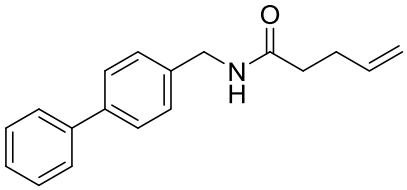
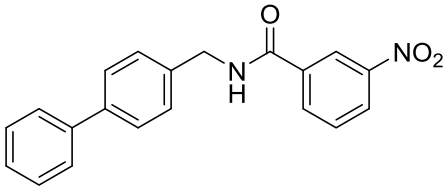
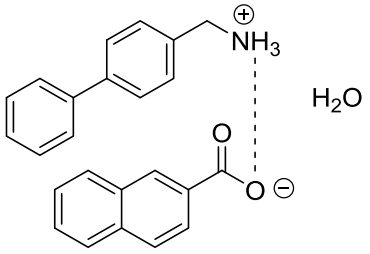
46		4.56	6.00	5%
47		4.48	5.96	1%
48		4.71	6.48	33%
49		4.48	5.77	10%
50		4.61	6.67	12%
51		4.49	5.78	10%
52		4.72	6.65	18%

Table 4: Synthesized amides with relevant CH_2 and N-H proton shifts

Amine Salt By-product and Hydrolysis Issues with Acyl Chlorides

Some of the acyl chlorides reacted did not give the amide product. Some did not give any relevant yields of any product. One particular acyl chloride, 2-naphthoyl chloride yielded a salt. This could be attributed to a few things. The first possible explanation is that the chloride hydrolyzed to a carboxylate over time when sitting on the shelf. The second possibility is that water in the solvent attributed to the formation of the salt. Although water is seen in the solid state crystal structure, that doesn't necessarily mean that water was in the flask. This could have happened after the compound was exposed to the environment. The X-ray crystal structure of the amine salt by-product is shown in **Figure 15**.



53

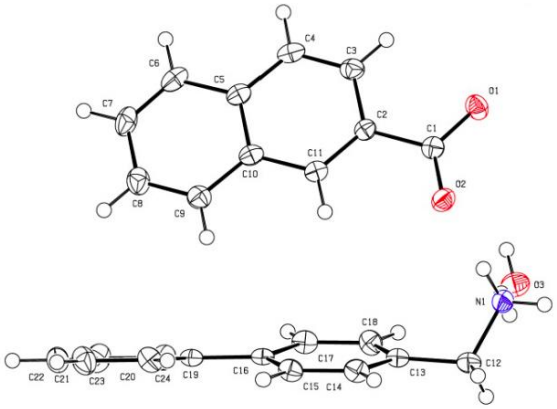


Figure 15: X-ray crystal structure of 2-naphthoyl-carboxylate by-product **53**

Hydrolysis of imine yielding 54

As previously mentioned, the acidic silica hampered initial purification of crude imines by flash column chromatography. The slightly acidic silica caused the imine to decompose back to the aldehyde and amine **54**. Therefore, the imines either had to be recrystallized or the silica needed to be ran through with triethylamine prior to purifying. The ^{1}H and ^{13}C NMR correlated with the standard **54** obtained from Sigma Aldrich.

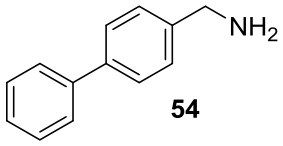


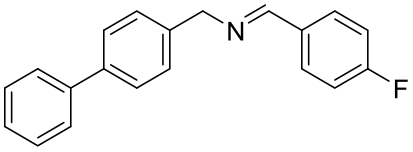
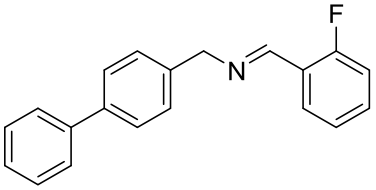
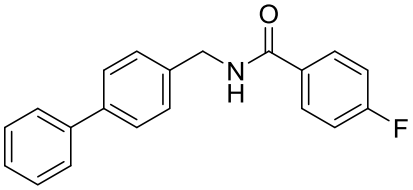
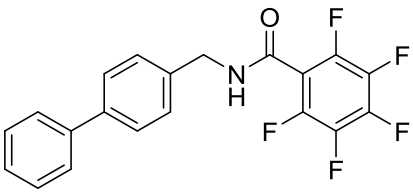
Figure 16: biphenyl-4-methyl amine

Fluorination Analysis and Work with Anhydrides

The use of anhydrides in place of some acyl chlorides has been found to be a safe alternative to creating the amide linkage in this research. For example, trifluoroacetyl chloride is much harder to handle as compared to trifluoroacetic anhydride. Trifluoroacetyl chloride is a gas at room temperature; therefore it is shipped under high pressure in a gas canister. Fluorinated compounds have been found to possess many important pharmacological properties, which are useful in the drug and agricultural industry. For example, fluorinating a compound will almost always increase the lipophilicity and bioavailability of a drug when reaching its target receptor.[32] Additionally, fluorine has been used as an isostere for hydrogen in Medicinal Chemistry as a means of altering the polarity, electronics, and stability of compounds.[33] Both

fluorine and hydrogen are similar sizes; therefore substituting a fluorine atom in place of hydrogen can vary the electron nature of the drug while avoiding any steric effects.[33]

In addition to proton and carbon NMR, additional ^{19}F NMR analysis was performed on compounds **20**, **21**, **32**, **33**, **43**, **48**, and **50**. Compounds **33**, **43**, and **50** did not contain any free protons from the acylating agent. As a result of fluorine splitting in ^{13}C NMR, ^{19}F NMR analysis was also much more convenient in identifying a variety of fluorinated functional groups. Therefore, ^{19}F NMR was used to confirm the presence of various fluorine groups (Ar-F, Ar-CF_3, $COCF_3$ and $COCF_2CF_3$). These shifts are consistent with those referenced.[34]

Fluorinated Product Number	**Structure**	**^{19}F NMR shift (ppm)**
20		-109.26
21		-121.88
32		-108.07
33		-140.19, -150.40, -159.86

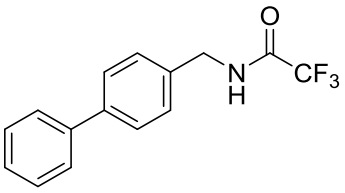
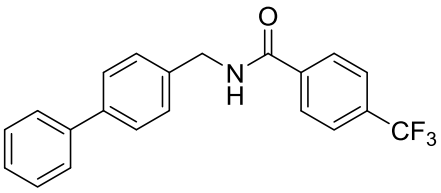
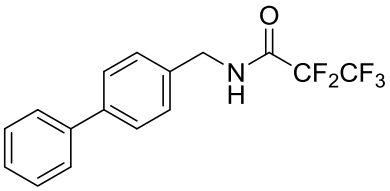
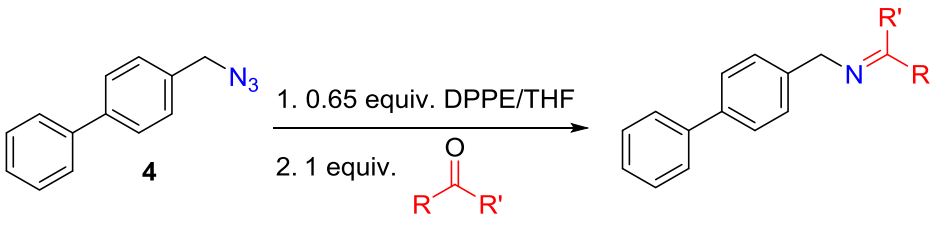
43		-75.78
48		-62.99
50		-82.76, -122.89

Table 5: Synthesized fluorinated compounds with relevant ^{19}F NMR shifts

Attempted Synthesis of Ketimines

Because the synthesis of aldimines worked from aldehydes, reactions were tried with ketones. Unfortunately, these reactions had no success in forming the desired product. This might be a consequence of the extra R group decreasing the electrophilicity of the carbonyl as well as steric factors. In the future, heating the reaction mixture may be examined. **Table 6** shows the anticipated ketimine products which weren't synthesized.



Equation 13: Attempted synthesis of ketimines from azide **4**

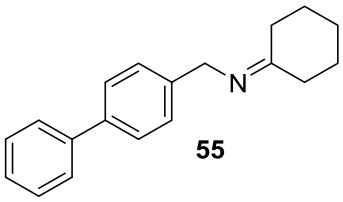
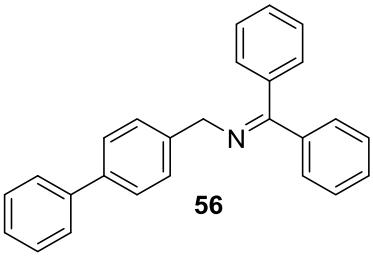
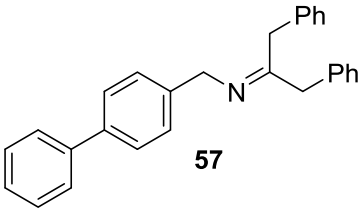
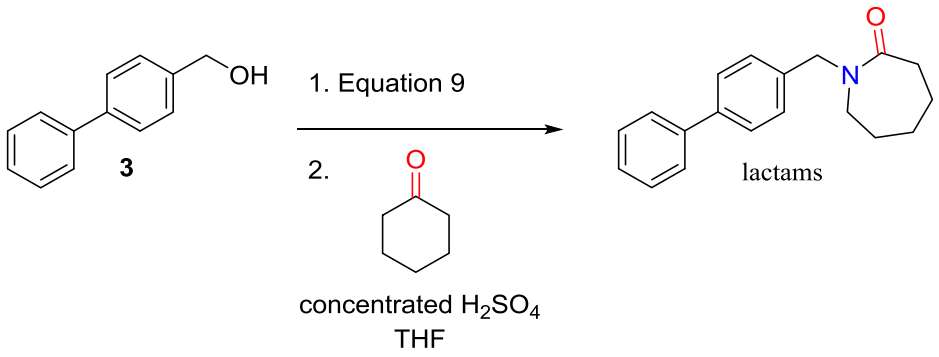
Anticipated product	% Yield
 **55**	0
 **56**	0
 **57**	0

Table 6: Attempted synthesis of ketimine compounds

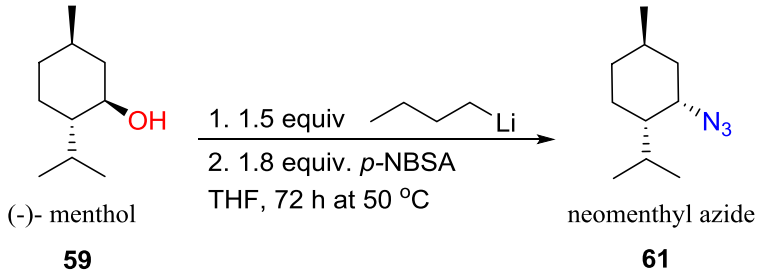
Attempted Synthesis of Lactams via Schmidt Ring Expansion[35]

After the conversion of azide **4** from alcohol **3**, azide **4** was reacted with cyclohexanone in the hope of synthesizing lactams. Unfortunately, no reactions occurred under the given conditions below in ten days. The starting materials of both azide **4** and cyclohexanone were recovered.



Equation 14: Attempted "one-pot" Schmidt ring expansion

Synthesis of Neomenthyl Azide from (-)-Menthol



Equation 15: The synthesis of neomenthyl azide from (-)-menthol.

The same one-pot scheme can be employed while converting secondary non-aromatic alcohols to azides. (-)- Menthol was successfully treated with the one-pot scheme yielding neomenthyl azide (**Equation 15**). Because the last step involves the backside attack of the azide on the Nosyl group, it should be expected that a secondary alcohol would take longer. After refluxing for up to 72 hours, neomenthyl azide was successfully synthesized from nosyl ester **60**. Azide **61** was purified via flash chromatography with a 50:1 hexanes/ethyl acetate solvent system and collected in 70% yield as yellow syrup. IR and proton NMR confirmed the synthesis of the azide. IR

displayed a sharp peak at 2105 cm^{-1} which correlated to the literature value for neomenthyl azide. Proton NMR showed a broad shift at 3.97 ppm for the proton attached to the carbon bearing the azide group. This also correlated to previous literature (3.98 ppm), which synthesized the above azide via a traditional two-pot method.[36]

X-Ray Crystal Analysis

X-ray crystal structures of amides **33**, **35**, **48**, **32** and imines **14**, **18**, and **27** were further confirmation of the amide and imine linkages formation. It is interesting to note that amide **48** produced a Z' value of **8**. Z' is defined as the number of symmetry-independent molecules in a crystal structure.[37] This implies that amide **48** exists as a polymorph. Polymorphism is defined as the ability of a compound to exist in multiple conformations in the solid state.[37] Polymorphs are of current interest in the pharmaceutical field as they can affect properties such as solubility, physical stability, bioavailability, process availability, as well as chemical and physical stability.[37] It is estimated that only around 9% of all compounds adopt multiple independent molecules in the solid state.[37] In the case of amide **48**, it can be hypothesized that the 8 independent molecules result from hydrogen bonding with the amide, as well as the biphenyl group which creates a twisted conformation. See **Appendix B** for X-ray structure data pertaining to compound **48** as well as all other compounds.

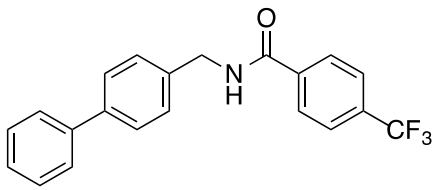
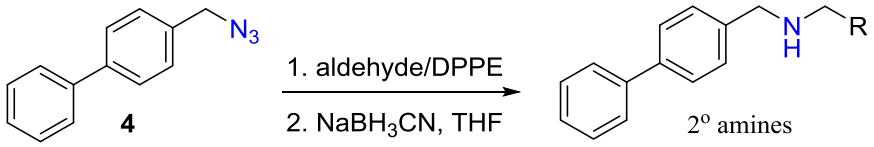
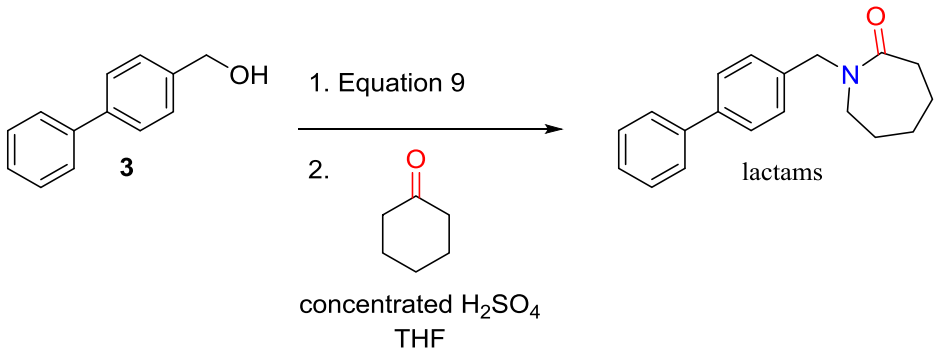


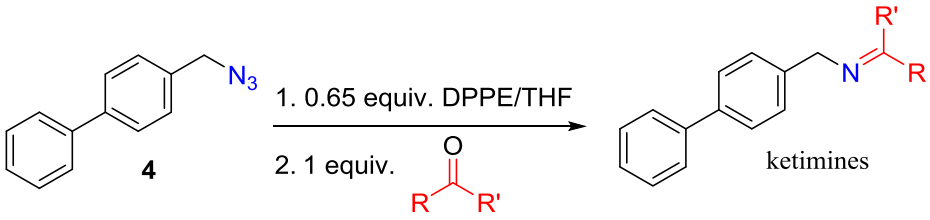
Figure 17: Compound **48** which produced Z'= 8

Future Research



Equation 16





Future research can utilize sodium cyanoborohydride to synthesize secondary amines in a "one-pot" protocol from azide **4** .[38] In addition, future research can seek to explore methods to obtain lactams (Schmidt ring expansion) via a one-pot protocol from alcohol **3**. This may involve refluxing azide **4** after the addition of the ketone. In addition, methods such as heating can be explored for synthesizing the attempted ketimines in this thesis.

Limitations

n-Butyllithium worked very effectively in this one-pot synthesis of biphenyl-4-methyl azide from biphenyl-4-methanol. However, butyllithium can be a very dangerous base to use, as it is pyrophoric.[39] Therefore extreme caution should be used when scaling up any small-scale synthesis. This is because the p*K*a of butyl lithium's conjugate acid is approximately 50, which creates an extremely exothermic reaction.[40] Additionally, like all S_N2 reactions, this one-pot procedure works best with primary or benzylic alcohols. As presented in **Equation 15**, non-aromatic secondary alcohols can be successfully converted to the azide with this same one-pot protocol. However, the reaction had to be refluxed for 72 hours at 50 ^{o}C to form the desired azide **61**. Finally, this one-pot azidation procedure is so far limited to alkyl compounds. Acyl compounds would not work with this procedure because butyl-lithium can perform Nucleophilic Acyl Substitution (NAS) and/or Nucleophilic Addition on the carbonyl.

Conclusion

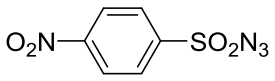
An efficient, fast, one-pot protocol was used to synthesize biphenyl-4-methyl azide **4** from biphenyl-4-methanol **3**. Afterwards**,** twenty aldimines and twenty-three amides were successfully synthesized using Staudinger chemistry; DPPE was successfully used as the mild phosphorus reducing agent. The amide and imine linkages were further confirmed by obtaining X-ray crystal structures. In addition to azide **4**, azide **61** a nonaromatic, secondary azide, was successfully synthesized in 70% yield by the same one-pot azidation protocol.

Experimental

General Procedures

All Nuclear Magnetic Resonance spectra were taken on Bruker Avance 400 Ultrashield and Bruker Avance 400 systems. 400 MHz and 100 MHz were the frequencies for ^{1}H and ^{13}C spectra, respectively. 376 MHz and 161 MHz were the frequencies for ^{19}F and ^{31}P spectra, respectively. Tetramethylsilane was used as the internal standard for ^{1}H and ^{13}C NMR. Phosphonic acid was used as the internal standard for ^{31}P NMR. Trichlorofluoromethane was used as the internal standard for ^{19}F NMR. Coupling constants (*J*) were measured in Hertz. Peak signals were labeled by the following abbreviations: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublets), dt (doublet of triplets), b s (broad singlet). Infrared specta were recorded and analyzed on a Thermo Electro Corporation IR 200 spectrophotometer. Aluminum-backed plates were used to monitor the reaction progress via TLC.

Synthesis of *para*-nitrobenzenesulfonyl azide 2 (*p*-NBSA) from *para*-nitrobenzene sulfonyl-chloride 1.



2

To a flame-dried round-bottom flask containing a magnetic stirrer, 10.0 g of *p*-nitrobenzenesulfonyl chloride was dissolved in 100 mL of methanol. Next, 10.0 g of sodium azide was added and the reaction mixture was left to stir overnight at room temperature. After excess sodium azide was filtered off, an aqueous workup was performed with ethyl acetate as the extracting solvent. After drying with anhydrous

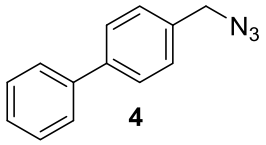
magnesium sulfate, the ethyl acetate was evaporated under reduced pressure; the solid was recrystallized from hot methanol yielding *p*-NBSA as yellow crystals which correlated to previous data of the known azide **2**.

^{1}H NMR ($CDCl_3$) δ 8.18 (2H, d, J = 9.08 Hz), 8.48 (2H, d, J = 9.08 Hz).

^{13}C NMR ($CDCl_3$) δ 151.21, 143.72, 128.94 (double intensity), 124.98 (double intensity).

IR absorption: 2135 cm^{-1} for azide functional group.

Synthesis of biphenyl-4-methylazide 4 from biphenyl-4-methanol 3.



4

To a flame-dried round-bottom flask containing a magnetic stirrer, 0.184 g (1.00 mmol) of biphenyl-4-methanol was added. A rubber septum was then attached and a balloon filled with nitrogen gas was attached through a needle connector apparatus. The round-bottom flask was subsequently flushed with nitrogen gas. 3.00 mL of dry THF was added via syringe, and immediately following, the flask was submerged in an ice bath. 0.750 mL (1.50 mmol) of 2.00 M *n*-butyllithium in cyclohexane was added drop-wise. After waiting 15 minutes, 0.420 g (1.80 mmol) of *p*-nitrobenzenesulfonyl azide (*p*-NBSA) in 3.00 mL of dry THF was added. The reaction was monitored by thin-layer chromatography with silica and *p*-anisaldehyde stain. After 90 minutes, TLC showed complete consumption of the biphenyl -4-methanol. A bright yellow spot with an R_f of 0.69 in 1:1 hexanes/ethyl acetate solvent system indicated formation of the desired azide. The reaction mixture was quenched with ammonium chloride, then an aqueous workup

was performed; ethyl acetate was used as the extracting solvent. After drying with anhydrous magnesium sulfate, the solvent was removed under reduced pressure, the azide was purified via flash chromatography with a 6:1 hexanes/ethyl acetate solvent system.[20] The pure yellow oil was collected and its structure confirmed with ^{1}H NMR and IR Spectroscopy of the known azide **4**.

^{1}H NMR ($CDCl_3$) δ 4.38 (2H, s), 7.35-7.62 (9H, m).

^{13}C NMR ($CDCl_3$) δ 141.33, 140.57, 134.39, 128.84 (double intensity), 128.67, 127.59 (double intensity), 127.53, 127.13 (double intensity), 54.60.

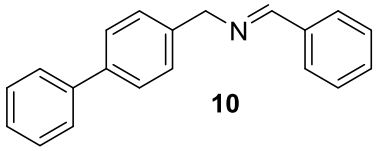
IR absorption: 2106 cm^{-1} for azide functional group.

General Procedures for the Synthesis of the Imines

To a flame-dried round-bottom flask containing a magnetic stirrer, 0.209 g (1.00 mmol) of biphenyl-4-methylazide **4** was added in 3.00 mL of dry THF. A rubber septum was then attached, and a balloon filled with nitrogen gas was attached to a needle connector apparatus. Immediately following, 0.260 g (0.650 mmol) of DPPE was dissolved in 3.00 mL of THF and syringed into the flask. 1.00 mmol of the aldehyde was either directly added via syringe (if it was a liquid), or dissolved in 3.00 mL of THF and then syringed into the flask. TLC plates needed to first be dipped in triethylamine and dried before being used to monitor the reaction. If not, the imine would decompose on the plate. When complete, the reaction mixture was first put on a rotary evaporator to remove the THF. The reaction mixture (if it was a solid) was immediately recrystallized from hot ethanol. If the reaction mixture was A crude syrup, the imine was purified via flash

chromatography. Note: the silica had to be run through with triethylamine to prevent decomposition of the product.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-phenylmethanimine 10 from azide 4.



10

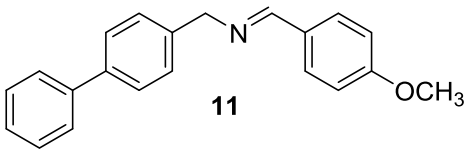
The synthesis of compound **10**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.102 mL (1.00 mmol) of benzaldehyde. After 5 hours, **10** was purified by Flash Chromatography with 1:1 Hexanes/Ethyl Acetate solvent system and collected as a colorless solid in 1% yield.

^{1}H NMR ($CDCl_3$) δ 4.88 (2H, s), 7.32-7.82 (14H, m), 8.44 (1H, s).

^{13}C NMR ($CDCl_3$) δ 162.16, 138.36, 130.85, 128.77 (double intensity), 128.65 (double intensity), 128.44 (double intensity), 128.32 (double intensity), 127.31 (double intensity), 127.20, 127.12 (double intensity), 64.80.

IR absorption: 1685 cm^{-1} for imine functional group.

Synthesis of (*E*)-*N*-(1,1'biphenyl]-4-ylmethyl)-1-(4-methoxyphenyl)methanimine 11 from azide 4.



11

The synthesis of compound **11**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.122 mL (1.00 mmol) of *p*-anisaldehyde overnight. The crude mixture was recrystallized from hot ethanol and collected as colorless crystals in 70 % yield.

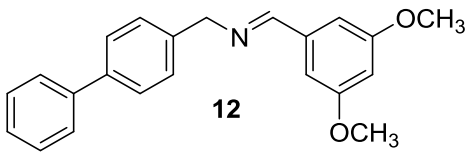
^{1}H NMR ($CDCl_3$) δ 3.85 (3H, s), 4.83 (2H, s), 6.94 (2H, d, J = 8.70 Hz), 7.32-7.60 (9H, m), 7.75 (2H, d, J = 8.80 Hz), 8.36 (1H, s).

^{13}C NMR ($CDCl_3$) δ 161.74, 161.46, 141.05, 139.90, 138.67, 129.88 (double intensity), 128.76 (double intensity), 128.40 (double intensity), 127.64, 127.27 (double intensity), 127.17, 127.11 (double intensity), 114.00 (double intensity), 64.71, 55.39.

IR absorption: 1684 cm^{-1} for imine functional group .

Melting Point: 83-86 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(3,5 dimethoxyphenyl)methanimine 12 from azide 4.



12

The synthesis of compound **12**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.166 mL (1.00 mmol) of 2,4-dimethoxybenzaldehyde overnight.

The reaction mixture was recrystallized from hot ethanol yielding imine **12** in 34% yield as colorless crystals.

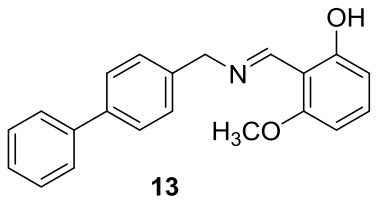
^{1}H NMR ($CDCl_3$) δ 3.83 (6H, s), 4.87 (2H, s), 6.55 (1H, s), 6.96 (2H, s), 7.34-7.61 (9H, m), 8.80 (1H, s).

^{13}C NMR ($CDCl_3$) δ 162.13, 160.94 (double intensity), 140.98, 140.03, 138.21, 138.19, 128.78 (double intensity), 128.47 (double intensity), 127.31 (double intensity), 127.22, 127.11 (double intensity), 105.94 (double intensity), 103.57, 64.70, 55.56 (double intensity).

IR absorption: 1646 cm^{-1} for imine functional group.

Melting Point: 105-106 ^{o}C.

Synthesis of (*E*)-2-((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-3-methoxphenol 13 from azide 4.



13

The synthesis of compound **13**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.136 g (1.00 mmol) of 2-hydroxy-6-methoxybenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **13** in 43% as a yellow crystals.

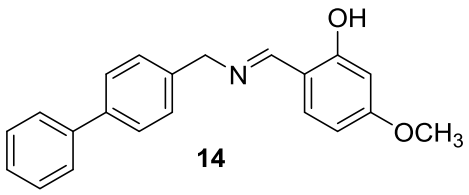
^{1}H NMR ($CDCl_3$) δ 3.86 (3H, s), 4.83 (2H, s), 6.32 (1H, d, J= 8.20 Hz), 6.56 (1H, d, J= 8.20 Hz), 7.22-7.59 (10H, m), 8.96 (1H, s), 14.46 (1H, s).

^{13}C NMR ($CDCl_3$) δ 163.93, 161.92, 159.58, 140.84, 140.26, 137.54, 133.59, 128.80 (double intensity), 128.09 (double intensity), 127.40, 127.30, 127.12 (double intensity), 110.40, 108.12, 62.65, 55.62.

IR absorption: 1634 cm^{-1} for imine functional group.

Melting Point: 78-80 ^{o}C.

Synthesis of (*E*)-1-((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-5-methoxyphenol 14 from azide 4.



14

The synthesis of compound **14**: 0.209 g (1 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.136 g (1.00 mmol) of 2-hydroxy-4-methoxybenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **14** in 44% as yellow crystals.

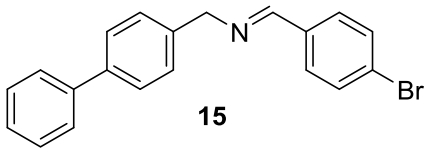
^{1}H NMR ($CDCl_3$) δ 3.82 (3H, s), 4.80 (2H, s), 6.41-6.46 (2H, m), 7.15 (1H, d, J = 8.52 Hz), 7.33-7.60 (9H, m), 8.33 (1H, s), 13.95 (1H, s).

^{13}C NMR ($CDCl_3$) δ 165.03, 164.67, 163.59, 140.78, 140.35, 137.31, 132.72, 128.82 (double intensity), 128.15 (double intensity), 127.44 (double intensity), 127.34, 127.12 (double intensity), 112.44, 106.53, 101.18, 61.72, 55.41.

IR absorption: 1626 cm^{-1} for imine functional group.

Melting Point: 100-102 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-bromophenyl)methanimine 15 from azide 4.



15

The synthesis of compound **15**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.185 g (1.00 mmol) of 4-bromobenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **15** in 24% as colorless crystals.

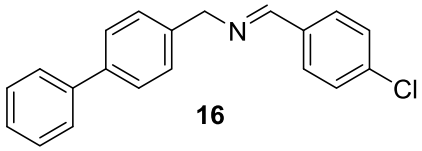
^{1}H NMR ($CDCl_3$) δ 4.85 (2H, s), 7.32-7.45 (9H, m), 7.56 (2H, d, J = 8.48 Hz), 7.67 (2H, d, J = 8.52 Hz), 8.37 (1H, s).

^{13}C NMR ($CDCl_3$) δ 160.80, 140.95, 140.10, 138.06, 135.00, 131.88 (double intensity), 129.73 (double intensity), 128.79 (double intensity), 128.46 (double intensity), 127.36 (double intensity), 127.25, 127.12 (double intensity), 125.25 64.78.

IR absorption: 1644 cm^{-1} for imine functional group.

Melting Point: 116-118 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-chlorophenyl)methanimine 16 from azide 4.



16

The synthesis of compound **16**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.141 g (1.00 mmol) of 4-chlorobenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **16** in 16% as a colorless crystals.

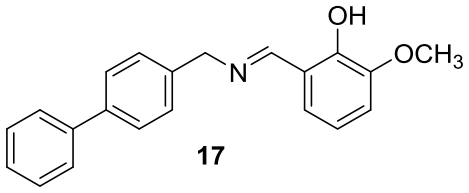
^{1}H NMR ($CDCl_3$) δ 4.86 (2H, s), 7.33-7.45 (9H, m), 7.59 (2H, d, J = 8.24 Hz), 7.74 (2H, d, J = 8.48 Hz), 8.39 (1H, s).

^{13}C NMR ($CDCl_3$) δ 160.70, 140.95, 140.09, 138.10, 136.77, 134.58, 129.50 (double intensity), 128.93 (double intensity), 128.78 (double intensity), 128.45 (double intensity), 127.36, 127.24, 127.11 (double intensity), 64.77.

IR absorption: 1644 cm^{-1} for imine functional group.

Melting Point: 116-118 ^{o}C.

Synthesis of (*E*)-1-((([1,1' biphenyl]-4-ylmethyl)imino)methyl)-6-methoxyphenol 17 from azide 4.



17

The synthesis of compound **17**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.136 g (1.00 mmol) of 2-hydroxy-3-methoxybenzaldehyde overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **17** in 38% as yellow crystals.

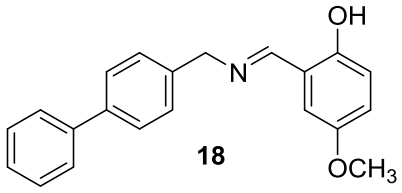
^{1}H NMR ($CDCl_3$) δ 3.92 (3H, s), 4.88 (2H, s), 6.84 (1H, t, J= 7.82 Hz), 6.91-6.96 (2H m), 7.35-7.60 (9H, m), 8.47 (1H, s), 13.94 (1H, s).

^{13}C NMR ($CDCl_3$) δ 165.98, 151.82, 148.56, 140.80, 140.36, 137.13, 128.83 (double intensity), 128.10 (double intensity), 127.41 (double intensity), 127.13 (double intensity), 123.05, 118.65, 118.07, 114.01, 62.49, 56.12.

IR absorption: 1634 cm^{-1} for imine functional group.

Melting Point: 98-100 ^{o}C.

Synthesis of (*E*)-2-((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-4-methoxyphenol 18 from azide 4.



18

The synthesis of compound **18**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.136 g (1.00 mmol) of 2-hydroxy-5-methoxybenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **18** in 50% as yellow crystals.

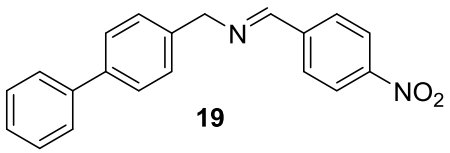
^{1}H NMR ($CDCl_3$) δ 3.78 (3H, s), 4.85 (2H, s), 6.81 (1H, d, J= 1.96 Hz), 6.91-6.96 (2H m), 7.33-7.58 (9H, m), 8.43 (1H, s), 12.91 (1H, s).

^{13}C NMR ($CDCl_3$) δ 165.45, 155.21, 152.01, 140.78, 140.36, 137.21, 128.82 (double intensity), 128.24 (double intensity), 127.45 (double intensity), 127.35 127.12 (double intensity), 119.42, 118.46, 117.81, 114.92, 63.03, 55.96.

IR absorption: 1634 cm^{-1} for imine functional group.

Melting Point: 108-110 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-nitrophenyl)methanimine 19 from azide 4.



19

The synthesis of compound **19**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.151 g (1.00 mmol) of 4-nitrobenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **19** in 25% as an orange solid.

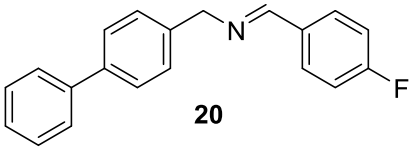
^{1}H NMR ($CDCl_3$) δ 4.93 (2H, s), 7.42-7.61 (9H, m), 7.97 (2H, d, J = 8.84 Hz), 8.28 (2H, d, J = 8.80), 8.50 (1H, s).

^{13}C NMR ($CDCl_3$) δ 159.61, 149.09, 141.57, 140.82, 140.34, 137.47, 129.00 (double intensity), 128.82 (double intensity), 128.55, 127.45 (double intensity), 127.35, 127.11 (double intensity), 123.93 (double intensity), 64.97.

IR absorption: 1644 cm^{-1} for imine functional group.

Melting Point: 104-107 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl}-4-ylmethyl)-1-(4-fluorophenyl)methanimine 20 from azide 4.



20

The synthesis of compound **20**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.107 mL (1.00 mmol) of 4-fluorobenzaldehyde overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **20** in trace amounts as a yellow solid with unreacted DPPE.

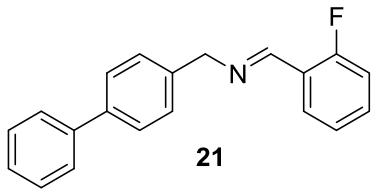
^{1}H NMR ($CDCl_3$) δ 4.85 (2H, s), 7.11 (2H, d, J = 8.66 Hz), 7.31-7.60 (9H, m), 7.80 (2H, d, J = 8.78 Hz), 8.40 (1H, s).

^{13}C NMR ($CDCl_3$) δ 160.60, 140.98, 140.06, 138.25, 132.88, 132.66, 130.20 (d, $^3J_{CF}$ = 8.71 Hz), 128.77 (double intensity), 128.43, (double intensity), 127.34 (double intensity), 127.22, 127.10 (double intensity), 115.75 (d, $^2J_{FC}$ = 21.9 Hz), 64.69.

^{19}F NMR ($CDCl_3$) -109.26.

IR absorption: 1645 cm^{-1} for imine functional group.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2-fluorophenyl)methanimine 21 from azide 4.



21

The synthesis of compound **21**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.105 mL (1.00 mmol) of 2-fluorobenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **21** in trace amounts as a yellow solid with DPPE.

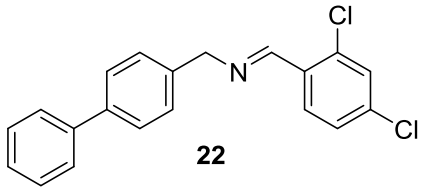
^{1}H NMR ($CDCl_3$) δ 4.85 (2H, s), 7.11 (2H, d, J = 8.66 Hz), 7.31-7.60 (9H, m), 7.80 (2H, d, J = 8.80 Hz), 8.40 (1H, s).

^{13}C NMR ($CDCl_3$) δ 155.32, 141.00, 140.06, 138.21, 132.70 (d, $^3J_{CF}$ = 11.24 Hz), 131.86 (double intensity), 129.33, 128.93 (double intensity), 128.74, 128.42, 127.30 (double intensity), 127.18, 127.08 (double intensity), 124.36, 115.73 (d, $^2J_{FC}$ = 20.8 Hz), 65.18.

^{19}F NMR ($CDCl_3$) -121.88.

IR absorption: 1640 cm^{-1} for imine functional group.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4-dichlorophenyl)methanimine 22 from azide 4.



22

The synthesis of compound **22**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.175 g (1.00 mmol) of 2-4-dichlorobenzaldehyde overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **22** in 44% as a yellow solid.

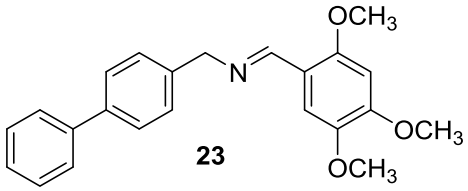
^{1}H NMR ($CDCl_3$) δ 4.89 (2H, s), 6.51 (1H, s), 7.25-7.59 (10H, m), 8.07 (2H, d, J = 8.48 Hz), 8.81 (1H, s).

^{13}C NMR ($CDCl_3$) δ 157.66, 140.93, 140.17, 137.91, 137.09, 135.71, 131.71, 129.58, 129.46, 128.80 (double intensity), 128.46 (double intensity), 127.57, 127.39 (double intensity), 127.27, 127.12 (double intensity), 65.07.

IR absorption: 1638 cm^{-1} for imine functional group.

Melting Point: 110 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4,5-trimethoxyphenyl) methanimine 23 from azide 4.



23

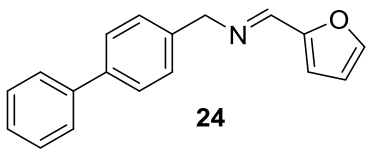
The synthesis of compound **23**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.196 g (1.00 mmol) of 2-4-5-trimethoxybenzaldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **23** in 25 % as a colorless solid.

^{1}H NMR ($CDCl_3$) δ 3.88 (3H, s), 3.89 (3H, s), 3.94 (3H, s), 4.84 (2H, s), 6.51 (1H, s), 7.33-7.60 (10H, m), 8.80 (1H, s).

^{13}C NMR ($CDCl_3$) δ 157.56, 154.30, 152.31, 143.58, 141.08, 139.81, 139.10, 128.75 (double intensity), 128.42 (double intensity), 127.22 (double intensity), 127.14, 127.10 (double intensity), 116.55, 109.02, 96.62, 65.14, 56.60, 56.33, 56.04.

IR absorption: 1633 cm^{-1} for imine functional group.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(furan-2-yl)methanimine 24 from azide 4.



24

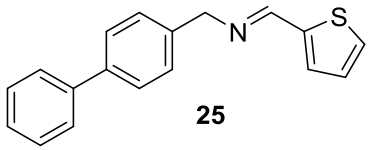
The synthesis of compound **24**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.830 mL (1.00 mmol) of furfural overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **24** in 10% as an orange solid.

^{1}H NMR ($CDCl_3$) δ 4.83 (2H, s), 6.49 (1H, m), 6.79 (1H, d, J = 3.24 Hz), 7.32-7.60 (10H, m), 8.20 (1H, s).

^{13}C NMR ($CDCl_3$) δ 151.59, 150.54, 144.92, 140.98, 140.09, 137.96, 128.83 (double intensity), 128.75 (double intensity), 127.36 (double intensity), 127.27 127.12 (double intensity), 114.41, 111.76, 64.89.

IR absorption: 1646 cm^{-1} for imine functional group.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(thiophen-2-yl)methanimine 25 from azide 4.



25

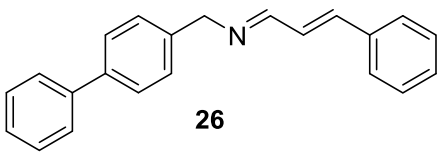
The synthesis of compound **25**: 0.209 g (1.00 mmol) of **4** was stirred with 0.269 g (0.650 mmol) of DPPE and 0.930 mL (1.00 mmol) of thiophene2-carbaldehyde overnight. The crude syrup was purified via flash chromatography with 1:1 Hexanes/Ethyl Acetate yielding imine **25** in trace amounts (1% yield) as an orange solid. Note: triethylamine was run though the silica gel prior to purification to prevent decomposition of the imine.

^{1}H NMR ($CDCl_3$) δ 4.84 (2H, s), 7.29 (1H, dd, J = 5.00 Hz, 3.64 Hz), 7.34-7.60 (11H, m), 8.49 (1H, s).

^{13}C NMR ($CDCl_3$) δ 155.32, 142.44, 141.00, 140.00, 138.12, 130.76, 129.14, 128.77 (double intensity), 128.48 (double intensity), 127.41, 127.29 (double intensity), 127.20, 127.10 (double intensity), 64.20.

IR absorption: 1634 cm^{-1} for imine functional group.

Synthesis of (1*E*,2*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-3-phenylprop-2-en-1-imine 26 from azide 4.



26

The synthesis of compound **26**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.126 mL (1.00 mmol) of *trans*-cinnamaldehyde overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **26** in 40% as a yellow crystals.

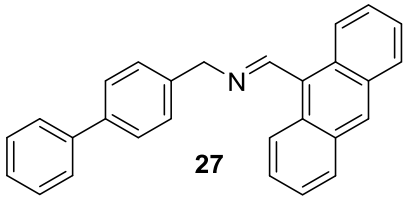
^{1}H NMR ($CDCl_3$) δ 4.76 (2H, s), 6.51 (2H, d, J = 5.36 Hz), 7.32-7.60 (14H, m), 8.19 (1H, s).

^{13}C NMR ($CDCl_3$) δ 163.62, 140.05, 138.28, 137.26, 135.69, 132.11, 129.27, 128.88 (double intensity), 128.78 (double intensity), 128.67, 128.56 (double intensity), 127.39 (double intensity), 127.31 (double intensity), 127.22, 127.11 (double intensity), 65.00.

IR absorption: 1636 cm^{-1} for imine functional group.

Melting Point: 100-105 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(anthracen-9-yl)methanimine 27 from azide 4.



27

The synthesis of compound **27:** 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.206 g (1.00 mmol) of 9-anthraldehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **27** in 32% as a bronze colored solid.

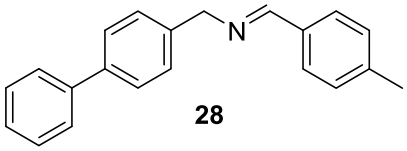
^{1}H NMR ($CDCl_3$) δ 5.20 (2H, s), 7.35-7.67 (13H, m), 8.03 (2H, d, J = 8.04 Hz), 8.52 (1H, s), 8.56 (2H, d, J = 8.84 Hz), 9.61 (1H, s).

^{13}C NMR ($CDCl_3$) δ 161.32, 140.97, 140.11, 138.28, 131.30, 130.13, 129.60, 128.92 (double intensity), 128.80 (double intensity), 128.67 (double intensity), 128.01, 127.45 (double intensity), 127.26, 127.13 (double intensity), 126.83 (double intensity), 125.30 (double intensity), 124.82, 66.50.

IR absorption: 1655 cm^{-1} for imine functional group.

Melting Point: 130-135 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(p-tolyl)methanimine 28 from azide 4.



28

The synthesis of compound **28:** 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.120 mL (1.00 mmol) of tolualdehyde overnight. The reaction mixture was recrystallized from hot ethanol yielding imine **28** in 40% as a colorless crystals.

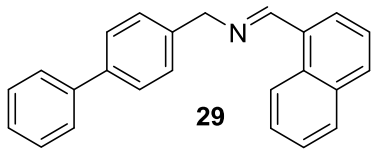
^{1}H NMR ($CDCl_3$) δ 2.39 (3H, s), 4.85 (2H, s), 7.23 (2H, d, J = 7.92 Hz), 7.32-7.60 (9H, m), 7.70 (2H, d, J = 8.08 Hz), 8.40 (1H, s).

^{13}C NMR ($CDCl_3$) δ 162.07, 141.14, 138.54, 133.53, 130.86, 130.80, 129.37 (double intensity), 128.75 (double intensity), 128.41 (double intensity), 128.29 (double intensity), 127.28 (double intensity), 127.17, 127.11 (double intensity), 64.78, 21.56.

IR absorption: 1646 cm^{-1} for imine functional group.

Melting Point: 96-98 ^{o}C.

Synthesis of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(naphthalen-1-yl)methanimine 29 from azide 4.



29

The synthesis of compound **29**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.140 mL (1.00 mmol) of 9-anthraldehyde overnight. The reaction mixture was immediately recrystallized from hot ethanol yielding imine **29** in 25% as a colorless crystals.

^{1}H NMR ($CDCl_3$) δ 5.00 (2H, s), 7.23 (2H, d, J = 7.92 Hz), 7.32-7.62 (10H, m), 7.89-7.98 (3H, m), 8.98 (1H, d, J = 8.60 Hz); 9.09 (1H, s).

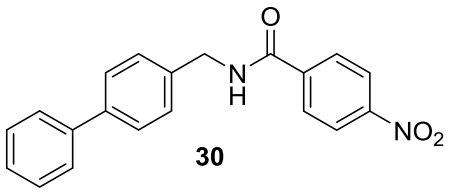
^{13}C NMR ($CDCl_3$) δ 161.83, 141.03, 139.97, 138.61, 133.86, 131.52, 131.38, 131.23, 129.15, 128.77 (double intensity), 128.67, 128.40 (double intensity), 127.33 (double intensity), 127.26, 127.20, 127.12 (double intensity), 126.09, 125.28, 124.37, 65.80.

IR absorption: 1642 cm^{-1} for imine functional group.

General Procedures for the Synthesis of the Amides

To a flame-dried round-bottom flask containing a magnetic stirrer, 0.209 g (1.00 mmol) of biphenyl-4-methylazide was added in 3.00 mL of dry THF. A rubber septum was then attached and a balloon filled with nitrogen gas was attached to a needle connector apparatus. Immediately following, 0.260 g (0.650 mmol) of DPPE was dissolved in 3.00 mL of THF and syringed into the flask. 2.00 mmol of the acyl chloride (or anhydride) was either directly added via syringe (if it was a liquid), or dissolved in 3.00 mL of THF and then syringed into the flask. TLC was used to monitor the reaction. When complete, the reaction mixture was first put on a rotary evaporator to remove the THF under reduced pressure. The reaction mixture (if it was a solid) was immediately recrystallized from hot ethanol. If the reaction mixture was a crude syrup, the amide was purified via flash chromatography.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-nitrobenzamide 30 from azide 4.



30

The synthesis of compound **30**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.371 g (2.00 mmol) of 4-nitrobenzoyl chloride overnight. The reaction was recrystallized from hot ethanol yielding amide **30** in 33% as a colorless crystals.

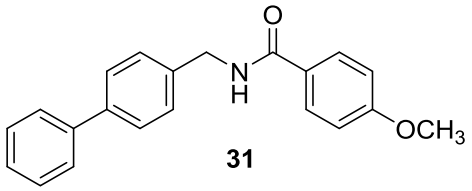
^{1}H NMR ($CDCl_3$) δ 4.71 (2H, d, J = 5.64 Hz), 6.51 (1H, s), 7.34-7.61 (9H, m), 7.97 (2H, d, J = 8.92 Hz), 8.30 (2H, d, J = 8.88 Hz).

^{13}C NMR ($CDCl_3$) δ 165.30, 149.75, 141.09, 140.53, 139.94, 136.43, 128.87 (double intensity), 128.52 (double intensity), 128.20 (double intensity), 127.71 (double intensity), 127.54, 127.09 (double intensity), 123.90 (double intensity), 44.25.

IR absorption: 1641 cm^{-1} for carbonyl functional group on amide.

Melting Point: 170-172 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-methoxybenzamide 31 from azide 4.



31

The synthesis of compound **31**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.271 mL (2.00 mmol) of 4-methoxybenzoyl chloride overnight. The reaction was immediately recrystallized from hot ethanol yielding amide **31** in 26% yield as a colorless crystals.

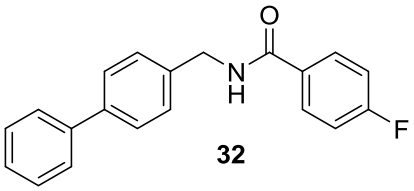
^{1}H NMR ($CDCl_3$) δ 3.85 (3H, s), 4.69 (2H, d, J = 5.68 Hz), 6.33 (1H, s), 6.93 (2H, d, J = 8.88 Hz), 7.34-7.60 (9H, m), 7.78 (2H, d, J = 8.92 Hz).

^{13}C NMR ($CDCl_3$) δ 166.87, 162.31, 140.75, 140.65, 137.47, 128.81 (double intensity), 128.78 (double intensity), 128.39 (double intensity), 127.54 (double intensity), 127.38, 127.10 (double intensity), 126.70, 113.84 (double intensity), 55.43, 43.83.

IR absorption: 1655 cm^{-1} for carbonyl functional group on amide.

Melting Point: 159-161 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-fluorobenzamide 32 from azide 4.



32

The synthesis of compound **32**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.240 mL (2.00 mmol) of 4-fluorobenzoyl chloride overnight. The reaction was recrystallized from hot ethanol yielding 16% of amide **32** as a colorless solid.

^{1}H NMR ($CDCl_3$) δ 4.69 (2H, d, J = 5.64 Hz), 6.38 (1H, s), 7.13 (2H, d, J = 8.60 Hz), 7.34-7.60 (9H, m), 7.83 (2H, d, J = 8.80 Hz).

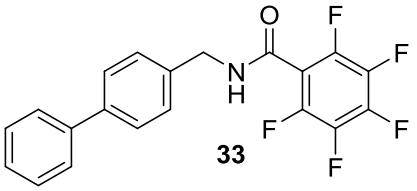
^{13}C NMR ($CDCl_3$) δ 166.20 (d, $^{1}J_{FC}$ = 28.59), 163.55, 140.76, 140.64, 137.05, 130.53, 129.34 (d, $^{3}J_{FC}$ = 8.75 Hz), 128.85 (double intensity), 128.45 (double intensity), 127.60 (double intensity), 127.45, 127.10 (double intensity), 115.69 (d, $^{2}J_{FC}$ = 21.74 Hz), 43.95.

^{19}F NMR ($CDCl_3$) -108.07.

IR absorption: 1700 cm^{-1} for carbonyl functional group on amide.

Melting Point: 163-165 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide 33 from azide 4.



33

The synthesis of compound **33**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.288 mL (2.00 mmol) of 2,3,4,5,6-Pentafluorobenzoyl chloride overnight. The reaction was recrystallized from hot ethanol yielding amide **33** in 25% as a colorless crystals.

^{1}H NMR ($CDCl_3$) δ 4.70 (2H, d, J = 5.72 Hz), 6.26 (1H, s), 7.35-7.61 (9H, m).

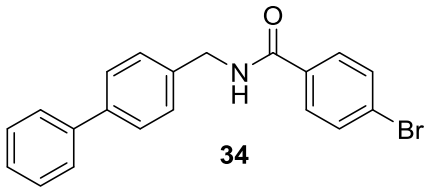
^{13}C NMR ($CDCl_3$) δ 160.64, 144.05-146.60 (m), 138.92-143.64 (m), 140.96, 140.45, 129.00-136.56 (m), 135.72, 132.57, 128.86 (double intensity), 128.22 (double intensity), 127.62 (double intensity), 127.06 (double intensity), 111.20-114.64 (m), 44.08.

^{19}F NMR ($CDCl_3$) δ -140.20, -150.40, -159.87.

IR absorption: 1684 cm^{-1} for carbonyl functional group on amide.

Melting Point: 150-152 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-bromobenzamide 34 from azide 4.



34

The synthesis of compound **34**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.440 g (2.00 mmol) of 4-bromobenzoyl chloride overnight. The reaction was immediately recrystallized from hot ethanol yielding amide **34** in 27% as a colorless crystals.

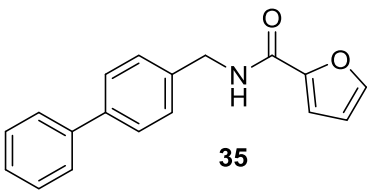
^{1}H NMR ($CDCl_3$) δ 4.68 (2H, d, J = 5.64 Hz), 6.40 (1H, s), 7.34-7.60 (9H, m), 7.68 (2H, d, J = 8.92 Hz), 7.68 (2H, d, J = 8.64 Hz).

^{13}C NMR ($CDCl_3$) δ 166.38, 140.85, 140.65, 136.94, 133.23, 131.89 (double intensity), 128.83 (double intensity), 128.61 (double intensity), 128.44 (double intensity), 127.61 (double intensity), 127.45, 127.09 (double intensity), 126.31, 44.00.

IR absorption: 1642 cm^{-1} for carbonyl functional group on amide.

Melting Point: 173-176 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)furan-2-carboxamide 35 from azide 4.



35

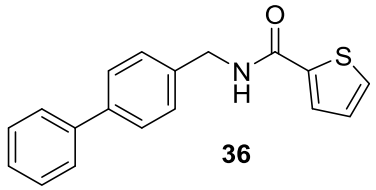
The synthesis of compound **35**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.197 mL (2.00 mmol) of 2-furoyl chloride overnight. The reaction was immediately recrystallized from hot ethanol yielding amide **35** in 15% as a colorless crystals.

^{1}H NMR ($CDCl_3$) δ 4.67 (2H, d, J = 5.92 Hz), 6.52 (1H, dd, J = 3.48 Hz, 1.76 Hz), 6.69 (1H, s), 7.18 (1H, d, J = 3.50 Hz), 7.32 (1H, d, J = 3.54 Hz), 7.31-7.65 (9H, m).

^{13}C NMR ($CDCl_3$) δ 128.82 (double intensity), 127.55 (double intensity), 114.59, 112.27.

IR absorption: 1708 cm^{-1} for carbonyl functional group on amide.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)thiophene-2-carboxamide 36 from azide 4.



36

The synthesis of compound **36**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.214 mL (2.00 mmol) of 2-thiophenecarbonyl chloride overnight. The reaction was recrystallized from hot ethanol yielding amide **36** in 17% yield as a colorless solid.

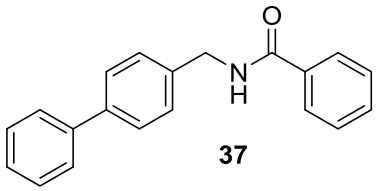
^{1}H NMR ($CDCl_3$) δ 4.67 (2H, d, J = 5.72 Hz), 6.37 (1H, s), 7.08 (1H, dd, J = 3.72 Hz, 5.00 Hz), 7.33-7.59 (11H, m).

^{13}C NMR ($CDCl_3$) δ 161.84, 140.71, 140.67, 138.68, 137.05, 130.11, 128.83 (double intensity), 128.45 (double intensity), 128.24, 127.69, 127.55 (double intensity), 127.42, 127.10 (double intensity), 43.73.

IR absorption: 1741 cm^{-1} for carbonyl functional group on amide.

Melting Point: 144-150 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)benzamide 37 from azide 4.



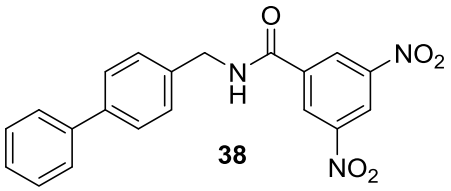
37

The synthesis of **37**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.232 mL (2.00 mmol) of benzoyl chloride overnight. The crude reaction syrup was purified via flash chromatography with a 1:1 hexanes/ethyl acetate solvent system and amide **37** was collected as a colorless solid in 1% yield.

^{1}H NMR ($CDCl_3$) δ 4.71 (2H, d, J = 5.68 Hz), 6.41 (1H, s), 7.34-7.83 (14H, m).

^{13}C NMR ($CDCl_3$) δ 141.42, 140.51, 136.45, 129.10 (double intensity), 128.85 (double intensity), 128.70, 127.56 (double intensity), 127.53 (double intensity), 127.34 (double intensity), 127.15 (double intensity), 46.01.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-3,5-dinitrobenzamide 38 from azide 4.



38

The synthesis of **38**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.461 g (2.00 mmol) of 3,5-dinitrobenzoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **38** was collected as yellow crystals in 16% yield.

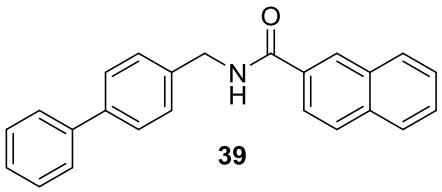
^{1}H NMR ($CDCl_3$) δ 4.74 (2H, d, J = 5.60 Hz), 6.82 (1H, s), 7.35-7.62 (9H, m), 8.99 (1H, s), 9.17 (2H, s).

^{13}C NMR ($CDCl_3$) δ 162.60, 148.67 (double intensity), 141.24, 140.35, 137.70, 135.81, 128.90 (double intensity), 128.69 (double intensity), 128.44 (double intensity), 127.76 (double intensity), 127.63, 127.25 (double intensity), 127.07 (double intensity), 121.22, 44.55.

IR absorption: 1639 cm^{-1} for carbonyl functional group on amide.

Melting Point: 169-171 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-naphthamide 39 from azide 4.



39

The synthesis of **39**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.382 g (2.00 mmol) of 2-naphthoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **39** was collected as colorless crystals in 18% yield.

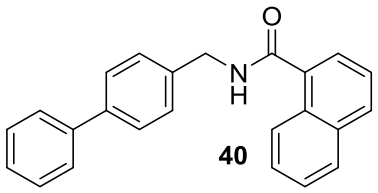
^{1}H NMR ($CDCl_3$) δ 4.76 (2H, d, J = 5.64 Hz), 6.61 (1H, s), 7.33-7.61 (11H, m), 7.87-7.93 (4H, m), 8.33 (1H, s).

^{13}C NMR ($CDCl_3$) δ 167.44, 140.72, 140.68, 137.22, 134.80, 132.63, 131.53, 128.95, 128.84 (double intensity), 128.57, 128.50 (double intensity), 127.79 127.74, 127.60 (double intensity), 127.49 127.42, 127.11 (double intensity), 126.83, 123.59, 44.01.

IR absorption: 1652 cm^{-1} for carbonyl functional group on amide.

Melting Point: 172-176 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-1-naphthamide 40 from azide 4.



40

The synthesis of **40**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.382 g (2.00 mmol) of 1-naphthoyl chloride overnight. The crude reaction mixture was recrystallized in hot ethanol and amide **40** was collected as colorless crystals in 30% yield.

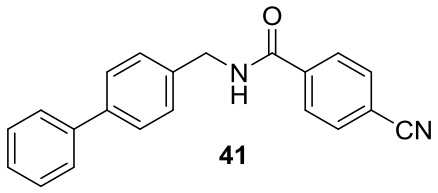
^{1}H NMR ($CDCl_3$) δ 4.79 (2H, d, J = 5.76 Hz), 6.32 (1H, s), 7.33-7.67 (11H, m), 7.88 (1H, d, J = 7.76 Hz), 7.93 (2H, d, J = 8.28 Hz), 8.38 (2H, d, J = 8.24 Hz).

^{13}C NMR ($CDCl_3$) δ 169.45, 140.71, 140.66, 137.10, 134.28, 133.73, 130.81, 130.19, 128.84 (double intensity), 128.39 (double intensity), 128.36, 127.61 (double intensity), 127.43, 127.25, 127.10 (double intensity), 126.51, 125.42, 124.96, 124.72, 43.87.

IR absorption: 1657 cm^{-1} for carbonyl functional group on amide.

Melting Point: 168-170 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-cyanobenzamide 41 from azide 4.



41

The synthesis of **41**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.331 g (2.00 mmol) of 4-cyanobenzoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **41** was collected as a colorless solid in 25% yield.

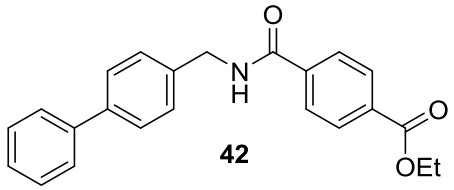
^{1}H NMR ($CDCl_3$) δ 4.70 (2H, d, J = 5.48 Hz), 6.46 (1H, s), 7.34-7.61 (9H, m), 7.75 (2H, d, J = 8.24 Hz), 7.91 (2H, d, J = 8.24 Hz).

^{13}C NMR ($CDCl_3$) δ 165.55, 141.05, 140.55, 138.30, 136.52, 132.52 (double intensity), 128.87 (double intensity), 128.51 (double intensity), 127.73 (double

intensity), 127.69 (double intensity), 127.54, 127.09 (double intensity), 117.93, 115.32, 44.16.

IR absorption: 1660 cm^{-1} for carbonyl functional group on amide.

Synthesis of ethyl 4-(([1,1'-biphenyl]-4-ylmethyl) carbamoyl)benzoate 42 from azide 4.



42

The synthesis of **42**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.410 g (2.00 mmol) of terephthaloyl chloride overnight. When complete, the reaction mixture was first put on a rotary evaporator to remove the THF under reduced pressure. The reaction mixture was recrystallized from hot ethanol yielding amide **42** as a colorless solid in 5 % yield. Note: the ethanol converted the remaining chloride group to the ethyl ester.

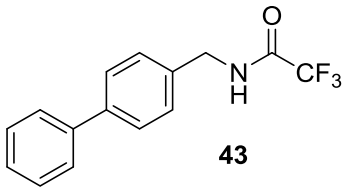
^{1}H NMR ($CDCl_3$) δ 1.40 (3H, t, J = 7.14 Hz), 4.38 (2H, q, J = 7.10 Hz) 4.69 (2H, d, J = 5.52 Hz), 6.69 (1H, s), 7.33-7.59 (9H, m), 7.87 (2H, d, J = 8.32 Hz), 8.09 (2H, d, J = 8.36 Hz).

^{13}C NMR ($CDCl_3$) δ 166.61, 165.84, 140.74, 140.60, 138.11, 136.89, 133.13, 129.84 (double intensity), 128.86 (double intensity), 128.47 (double intensity), 127.58 (double intensity), 127.46, 127.09 (double intensity), 127.07 (double intensity), 61.43, 43.98, 14.31.

IR absorption: 1714 cm^{-1} for carbonyl functional group on ester.

1658 cm^{-1} for carbonyl functional group on amide.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2,2-trifluoroacetamide 43 from azide 4.



43

The synthesis of **43**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.282 mL (2.00 mmol) of trifluoroacetic anhydride overnight. The reaction mixture was recrystallized from hot ethanol yielding amide **43** as a pink solid in 20% yield.

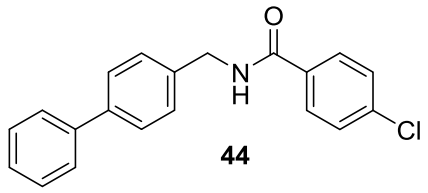
^{1}H NMR ($CDCl_3$) δ 4.59 (2H, d, J = 5.76 Hz), 6.57 (1H, s), 7.35-7.72 (9H, m).

^{13}C NMR ($CDCl_3$) δ 157.49, 141.23, 140.45, 135.12, 128.86 (double intensity), 128.45 (double intensity), 127.82 (double intensity), 127.67, 127.08 (double intensity), 116.01 (q, $^{1}J_{FC}$ = 287.78 Hz), 43.70.

^{19}F NMR ($CDCl_3$) δ -75.78.

IR absorption: 1726 cm^{-1} for carbonyl functional group on amide.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-chlorobenzamide 44 from azide 4.



44

The synthesis of **44**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.256 mL (2.00 mmol) of 4-chlorobenzoyl chloride overnight. The reaction mixture was recrystallized from hot ethanol yielding amide **44** as a colorless solid in 1% yield.

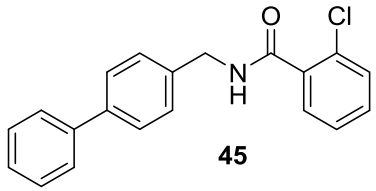
^{1}H NMR ($CDCl_3$) δ 4.69 (2H, d, J = 5.52 Hz), 6.40 (1H, s), 7.36-7.60 (11H, m), 7.76 (2H, d, J = 8.52 Hz).

^{13}C NMR ($CDCl_3$) δ 166.29, 140.84, 140.65, 137.90, 136.97, 132.77, 130.84 (double intensity), 128.91 (double intensity), 128.84 (double intensity), 128.44 (double intensity), 127.61 (double intensity), 127.45, 127.09 (double intensity), 44.00.

IR absorption: 1659 cm^{-1} for carbonyl functional group on amide.

Melting Point: 152-156 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-chlorobenzamide 45 from azide 4.



45

The synthesis of **45**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.253 mL (2.00 mmol) of 2-chlorobenzoyl chloride overnight. The reaction mixture was recrystallized from hot ethanol yielding amide **45** as a colorless solid in 10% yield.

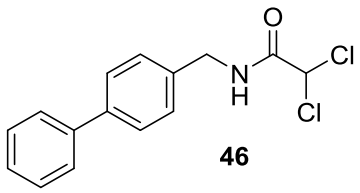
^{1}H NMR ($CDCl_3$) δ 4.73 (2H, d, J = 5.68 Hz), 6.51 (1H, s), 7.32-7.47 (8H, m), 7.58-7.61 (4H, m) 7.72-7.74 (1H, m).

^{13}C NMR ($CDCl_3$) δ 166.43, 140.67, 136.70, 134.87, 131.45, 130.69, 130.33, 130.30, 128.82 (double intensity), 128.36 (double intensity), 127.54 (double intensity), 127.41, 127.17, 127.10 (double intensity), 43.98.

IR absorption: 1655 cm^{-1} for carbonyl functional group on amide.

Melting Point: 148-150 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2-dichloroacetamide 46 from azide 1.



46

The synthesis of **46**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.192 mL (2.00 mmol) of dichloroacetyl chloride overnight. The reaction mixture was recrystallized from hot ethanol yielding 24% of amide **46** as a colorless solid.

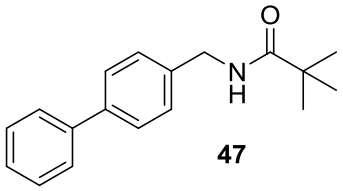
^{1}H NMR ($CDCl_3$) δ 4.56 (2H, d, J = 5.40 Hz), 5.99 (1H, s), 6.84 (1H, s) 7.36-7.60 (9H, m).

^{13}C NMR ($CDCl_3$) δ 164.11, 141.08, 140.52. 135.66, 128.83 (double intensity), 128.17 (double intensity), 127.67 (double intensity), 127.50, 127.08 (double intensity), 66.42, 44.01.

IR absorption: 1694 cm^{-1} for carbonyl functional group on amide.

Melting Point: 146-148 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)pivalamide 47 from azide 4.



47

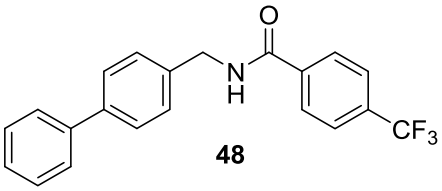
The synthesis of **47**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.250 mL (2.00 mmol) of trimethylacetyl chloride overnight. The crude reaction syrup was purified via flash chromatography with a 6:1 Hexanes/Ethyl Acetate solvent system and amide **47** collected as a colorless solid in 1% yield.

^{1}H NMR ($CDCl_3$) δ 1.25 (9H, s), 4.47 (2H, d, J = 5.64 Hz), 5.96 (1H, s), 7.32-7.59 (9H, m).

^{13}C NMR ($CDCl_3$) δ 178.38, 140.72, 140.45, 137.68, 128.82 (double intensity), 128.13 (double intensity), 127.49 (double intensity), 127.36, 127.09 (double intensity), 43.31, 38.77, 27.66 (triple intensity).

IR absorption: 1650 cm^{-1} for carbonyl functional group on amide.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-(trifluoromethyl)benzamide 48 from azide 4.



48

The synthesis of **48**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.300 mL (2.00 mmol) of 4-(trifluoromethyl)benzoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **48** was collected as colorless crystals in 33 % yield.

^{1}H NMR ($CDCl_3$) δ 4.71 (2H, d, J = 5.64 Hz), 6.48 (1H, s), 7.36-7.61 (9H, m), 7.71 (2H, d, J = 8.20 Hz), 7.92 (2H, d, J = 8.12 Hz).

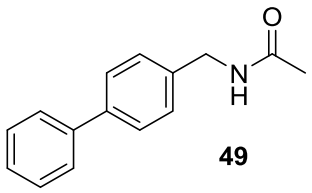
^{13}C NMR ($CDCl_3$) δ 166.09, 140.91, 140.60, 137.69, 136.76, 133.38 (q, $^{2}J_{FC}$ = 32.74 Hz), 128.85 (double intensity), 128.43 (double intensity), 127.63 (double intensity), 127.49 (double intensity), 127.08 (double intensity), 125.70 (q, $^{3}J_{FC}$ = 3.63 Hz), 123.66 (q, $^{1}J_{FC}$ = 272.41 Hz), 44.07.

^{19}F NMR ($CDCl_3$) δ -63.00.

IR absorption: 1664 cm^{-1} for carbonyl functional group on amide.

Melting Point: 163-165 ^{o}C.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)acetamide 49 from azide 4.



49

The synthesis of **49**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.143 mL (2.00 mmol) of acetyl chloride overnight. The crude reaction syrup was purified via flash chromatography with 6:1 Hexanes/Ethyl Acetate solvent system and amide **49** collected as a colorless solid in 10%.

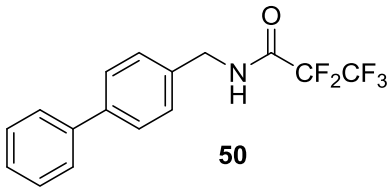
^{1}H NMR ($CDCl_3$) δ 2.05 (3H, s) 4.48 (2H, d, J = 5.64 Hz), 5.77 (1H, s), 7.35-7.59 (9H, m).

^{13}C NMR ($CDCl_3$) δ 169.87, 140.71, 140.65, 137.29, 128.82 (double intensity), 128.34 (double intensity), 127.50 (double intensity), 127.40, 127.09 (double intensity), 43.52, 23.38.

IR absorption: 1644 cm^{-1} for carbonyl functional group on amide.

Melting Point: 178-180 ^{o}C

Synthesis of *N*-(1,1'-biphenyl]-4-ylmethyl)-2,2,3,3,3-pentafluoropropanamide 50 from azide 4.



50

The synthesis of **50**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.395 mL (2.00 mmol) of Pentafluoropropionic anhydride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **50** was collected as colorless crystals in 12 % yield.

^{1}H NMR ($CDCl_3$) δ 4.61 (2H, d, J = 5.80 Hz), 6.67 (1H, s), 7.35-7.61 (9H, m).

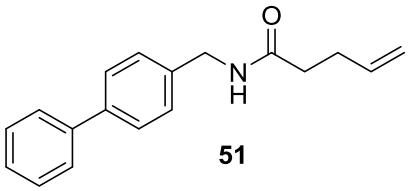
^{13}C NMR ($CDCl_3$) δ 157.67, 141.47, 140.39, 134.75, 128.89 (double intensity), 128.37 (double intensity), 127.83 (double intensity), 127.63, 127.11 (double intensity), 43.80.

^{19}F NMR ($CDCl_3$) δ -82.76, -122.89.

IR absorption: 1721 cm^{-1} for carbonyl functional group on amide.

Melting Point: 202-205 ^{o}C.

Synthesis of *N*-(1,1'-biphenyl]-4-ylmethyl)pent-4-enamide 51 from azide 4.



51

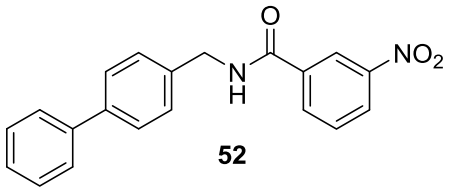
The synthesis of **51**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.221 mL (2.00 mmol) of 4-pentenoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **51** was collected as a colorless solid in 10 % yield.

^{1}H NMR ($CDCl_3$) δ 2.60 (4H, m), 4.49 (2H, d, J = 5.68 Hz), 5.06 (2H, m), 5.78 (1H, s), 5.84 (1H, m), 7.34-7.59 (9H, m).

^{13}C NMR ($CDCl_3$) δ 172.12, 140.68, 140.55, 137.32, 137.02, 128.83 (double intensity), 128.32 (double intensity), 127.47 (double intensity), 127.40, 127.09 (double intensity), 115.76, 43.33, 35.92, 29.63.

IR absorption: 1665 cm^{-1} for carbonyl functional group on amide.

Synthesis of *N*-([1,1'-biphenyl]-4-ylmethyl)-3-nitrobenzamide 52 from azide 4.



52

The synthesis of **52**: 0.209 g (1.00 mmol) of **4** was stirred with 0.260 g (0.650 mmol) of DPPE and 0.186 g (1.00 mmol) of 3-nitrobenzoyl chloride overnight. The crude reaction mixture was recrystallized from hot ethanol and amide **52** was collected as a colorless solid in 18 % yield. Note: only 1 mmol of 3-nitrobenzoyl chloride was used as compared to the 2 mmol scale used on the other compounds.

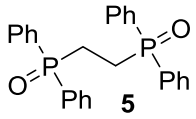
^{1}H NMR ($CDCl_3$) δ 4.72 (2H, d, J = 5.64 Hz), 6.65 (1H, s), 7.36-7.68 (8H, m), 8.21 (2H, d, J = 10.48 Hz), 8.37 (2H, d, J = 11.52 Hz), 8.63 (1H, s).

^{13}C NMR ($CDCl_3$) δ 164.92, 148.19, 140.97, 140.53, 136.45, 135.10, 133.32, 129.95, 128.85 (double intensity), 128.55 (double intensity), 127.67 (double intensity), 127.49, 127.09 (double intensity), 126.21, 121.79, 44.18.

IR absorption: 1667 cm^{-1} for carbonyl functional group on amide.

Melting Point: 145-148 ^{o}C.

Ethane-1,2-diylbis(diphenylphosphine oxide) 5



5

Side product **5** recrystallized out of a crude mixture of while attempting to purify amide **49** in early one-pot studies. Hot ethanol was used as the recrystallizing solvent which yielded **5** as colorless crystals.

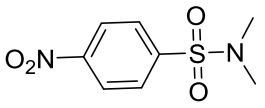
^{1}H NMR ($CDCl_3$) δ 2.65 (4H, d, J_{PH} = 2.76 Hz), 7.42-7.73 (20H, m).

^{13}C NMR ($CDCl_3$) δ 160.77, 144.08, 132.60, 130.87, 130.82, 130.77 (double intensity), 130.16, 129.65 (double intensity), 129.16, 129.10 (double intensity), 129.04, 21.52, 20.86.

^{31}P NMR ($CDCl_3$) δ 32.49.

IR absorption: 1718 cm^{-1} for carbonyl functional groups.

***N,N*-dimethyl-4-nitrobenzenesulfonamide 6**



6

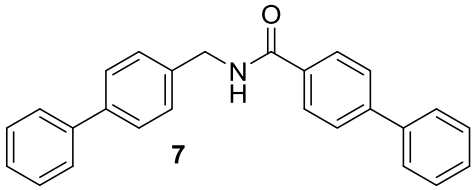
Side product **6** recrystallized out of a crude mixture of while attempting to purify amide **30** in early one pot studies. Hot ethanol was used as the recrystallizing solvent which yielded **6** as yellow crystals.

Note the solvent used in the reaction was DMF.

^{1}H NMR ($CDCl_3$) δ 2.78 (6H, s), 7.97 (2H, d, J = 8.92 Hz), 8.41 (2H, d, J = 8.88 Hz).

^{13}C NMR ($CDCl_3$) δ 128.82 (double intensity), 124.36 (double intensity), 37.81.

***N*-([1,1'-biphenyl]-4-ylmethyl)-[1,1'-biphenyl]-4-carboxamide 7**



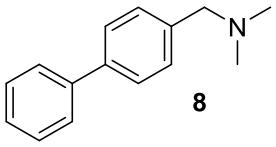
7

Side product **7** recrystallized out of a crude mixture of while attempting to purify amide **49** in early one pot studies. Hot ethanol was used as the recrystallizing solvent which yielded **7** as a colorless powder.

^{1}H NMR ($CDCl_3$) δ 4.89 (2H, s), 7.31-7.65 (16H, m), 7.88 (2H, d, J = 8.32), 8.47 (1H, s).

IR absorption: 1643 cm^{-1} for amide functional group.

1-([1,1'-biphenyl]-4-yl)-*N,N*-dimethylmethanamine 8



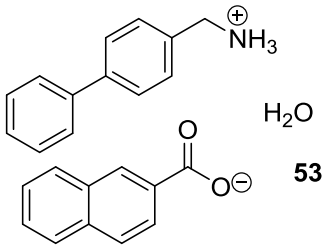
8

Side product **8** recrystallized out of a crude mixture of while attempting to purify amide **33** in early one pot studies. Hot ethanol was used as the recrystallizing solvent which yielded **8** as colorless crystals.

Note: the solvent used in the reaction was DMF.

^{1}H NMR ($CDCl_3$) δ 1.55 (6H, s), 5.45 (2H, s), 7.37-7.64 (9H, s).

[1,1'-biphenyl]-4-ylmethanaminium 2-naphthoate hydrate 53



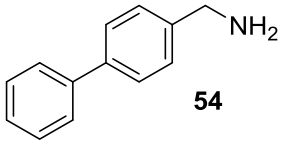
53

Side product **53** recrystallized out of a crude mixture of while attempting to purify amide **39** in the optimized experiments. Hot ethanol was used as the recrystallizing solvent which yielded **53** as yellow crystals.

^{1}H NMR ($CDCl_3$) δ 2.56 (2H, b s), 4.00, (2H, s), 7.31-7.62 (11H, m), 7.89 (2H, d, J = 8.24 Hz), 7.95 (1H, d, J = 8.00 Hz), 8.09 (1H, d, J = 8.76 Hz), 8.65 (1H, s).

IR absorption: 1643 cm^{-1} for amine functional group.

[1,1'-biphenyl]-4-ylmethanamine 54



54

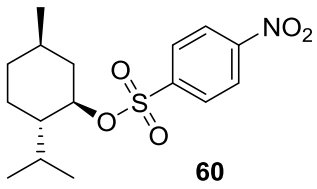
Amine **54** recrystallized out of a crude mixture of while attempting to purify imine **10** in the optimized experiments. Amine **54** formed upon hydrolysis of imine **10** during purification on a flash column. It was collected as a colorless solid in trace amounts.

^{1}H NMR ($CDCl_3$) δ 3.92 (2H, s), 7.32-7.60 (9H, m).

^{13}C NMR ($CDCl_3$) δ 142.35, 140.95, 139.81, 128.78 (double intensity), 128.67 (double intensity), 127.56 (double intensity), 127.33 (double intensity), 127.21 (double intensity), 127.08 (double intensity), 46.21.

IR absorption: 1643 cm^{-1} for primary amine group.

Synthesis of menthyl nosyl ester 60 from (-)-menthol.



60

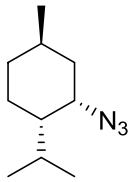
To a flame dried round bottom flask containing a magnetic stirrer, 0.780 g (5.00 mmol) of (-)-menthol was added. A rubber septum was then attached. A balloon filled with nitrogen gas was attached through a needle connector apparatus. The round bottom flask was subsequently flushed with the nitrogen gas. 10.0 mL of dry THF was added via syringe, and immediately following, the flask was submerged in an ice bath. 3.75 mL

(7.50 mmol) of 2.00 M *n*-butyl lithium in cyclohexane was added dropwise. After waiting 15 minutes, 2.10 g (9.00 mmol) of *para*-nitrobenzenesulfonyl azide in 15.0 mL of dry THF was added dropwise. The reaction was monitored by thin-layer chromatography and *p*-anisaldehyde stain. After 60 minutes, proton NMR confirmed the formation of the nosyl ester. Afterwards, an aqueous workup was performed with ethyl acetate as the extracting solvent. **60** was collected under reduced pressure as a yellow syrup.

^{1}H NMR ($CDCl_3$) δ 0.83-1.40 (13H, m), 1.41-1.88 (4H, m), 2.05-2.14 (1H, m), 4.57 (1H, dt, J = 15.47 Hz, 10.90 Hz, 8.13 Hz), (2H, d, J = 9.00 Hz), 8.40 (2H, d, J = 8.96 Hz).

IR absorption: 1351 cm^{-1} for sulfonate ester functional group.

Synthesis of (+)-neomenthyl azide 61 from (-)-menthol.



61

To a flame-dried round-bottom flask containing a magnetic stirrer, 0.780 g (5.00 mmol) of (-)-menthol was added. A rubber septum was then attached, and a balloon filled with nitrogen gas was attached through a needle connector apparatus. The round bottom flask was subsequently flushed with the nitrogen gas. 10.0 mL of dry THF was added via syringe. Immediately following, the flask was submerged in an ice bath. 3.75 mL (7.50 mmol) of 2.00 M *n*-butyl lithium in cyclohexane was added dropwise. After 15 minutes, 2.10 g (9.00 mmol) of *p*-nitrobenzenesulfonyl azide in 15.0 mL of dry THF was

added dropwise. TLC monitored the reaction with *p*-anisaldehyde stain. After 60 minutes, proton NMR confirmed the formation of the nosyl ester. The reaction mixture was then refluxed at 50 ^{o}C for 72 hours. Afterwards, an aqueous workup was performed; ethyl acetate was used as the extracting solvent. After the solvent was removed under reduced pressure, the azide was purified via flash chromatography with 50:1 Hexanes: Ethyl Acetate solvent system.[20] The pure yellow oil was collected in 70% yield.

^{1}H NMR ($CDCl_3$) δ 0.77-1.32 (13H, m), 1.43-1.75 (4H, m), 1.99-2.03 (1H, m), 3.97, (1H, b s).

^{13}C NMR ($CDCl_3$) δ 60.53, 47.33, 38.91, 35.00, 29.48, 26.52, 24.89, 22.67, 20.92, 20.68.

IR absorption: 2106 cm^{-1} for azide functional group.

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APPENDIX A

IR and NMR SPECTRA

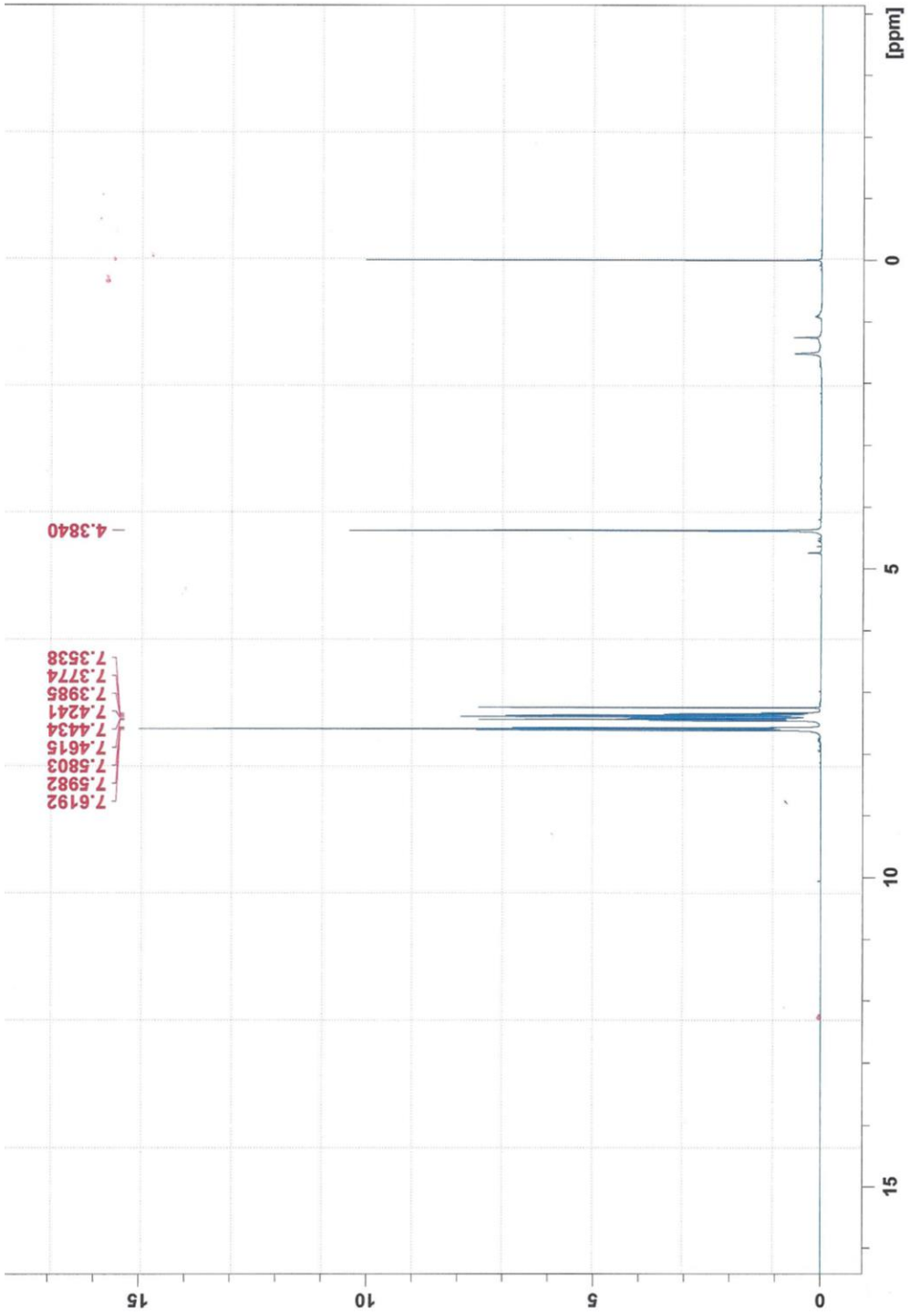

Figure 18: ^{1}H NMR spectrum of 4 -(azidomethyl)-1,1'-biphenyl **4**

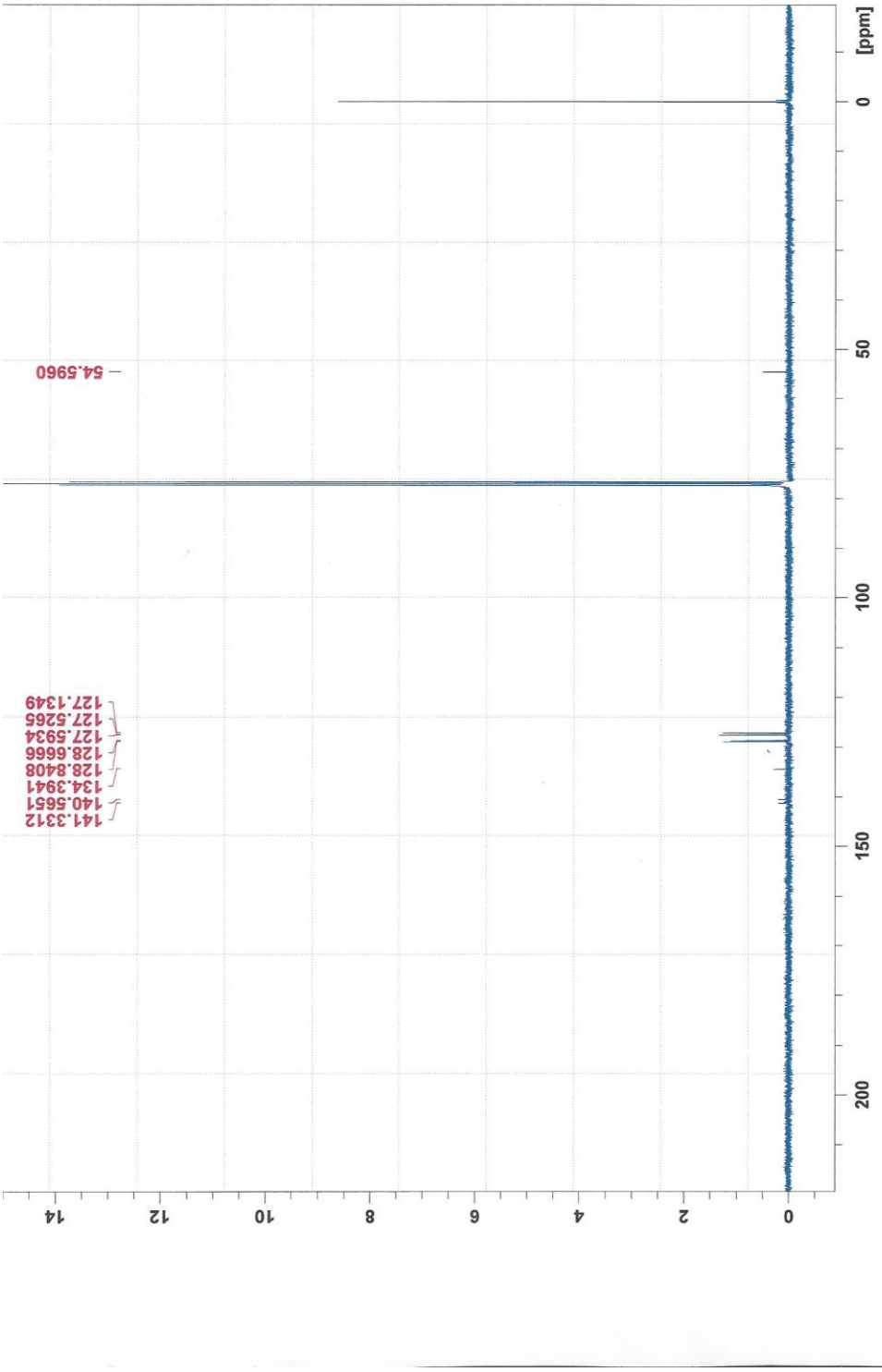

Figure 19: ^{13}C NMR spectrum of ^{4}-(azidomethyl)-1,1'-biphenyl **4**

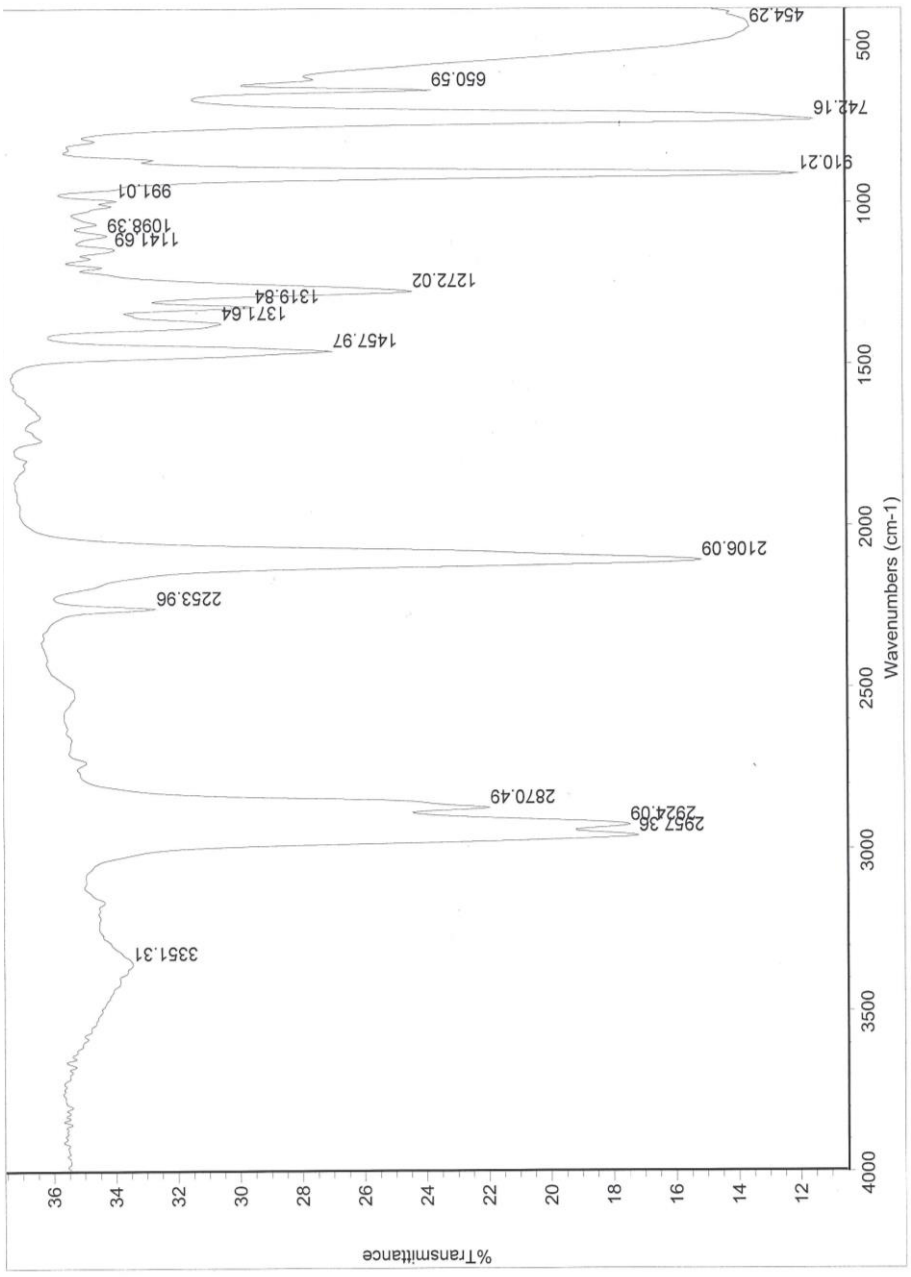

Figure 20: IR spectrum of [4] -(azidomethyl)-1,1'-biphenyl **4**

IR and NMR Spectra of Imines

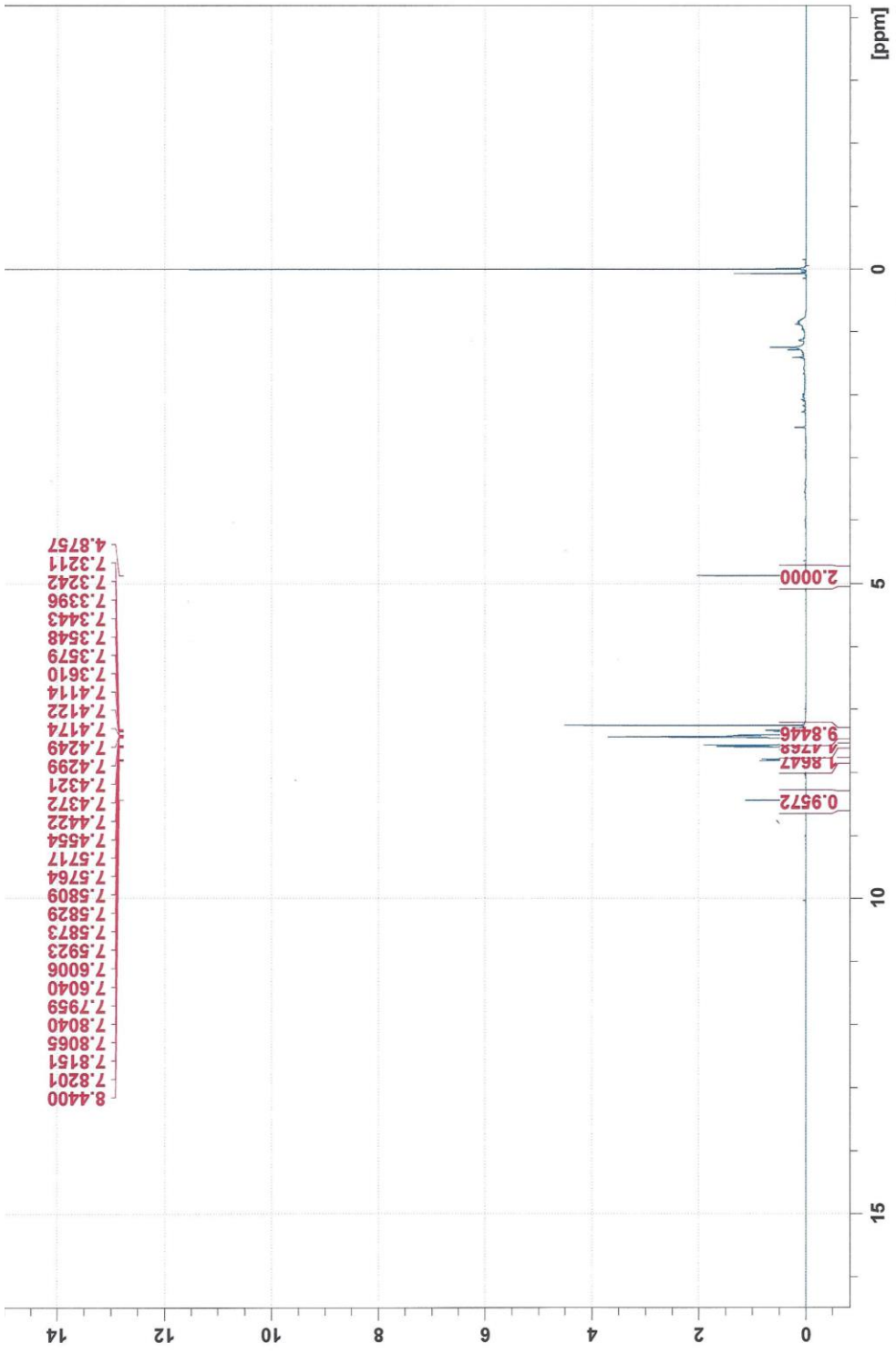

Figure 21: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-phenylmethanimine **10**

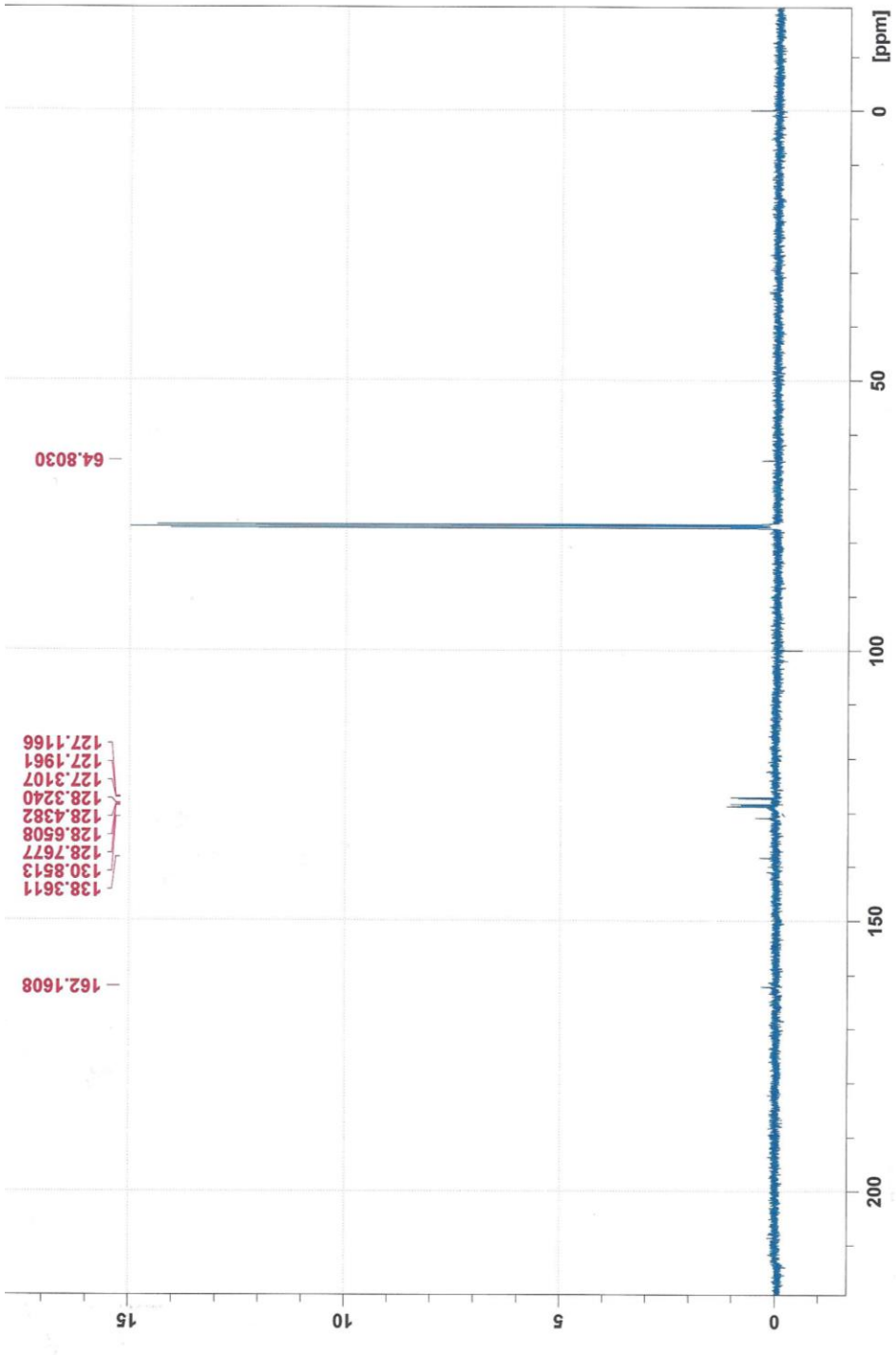

Figure 22: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-phenylmethanimine **10**

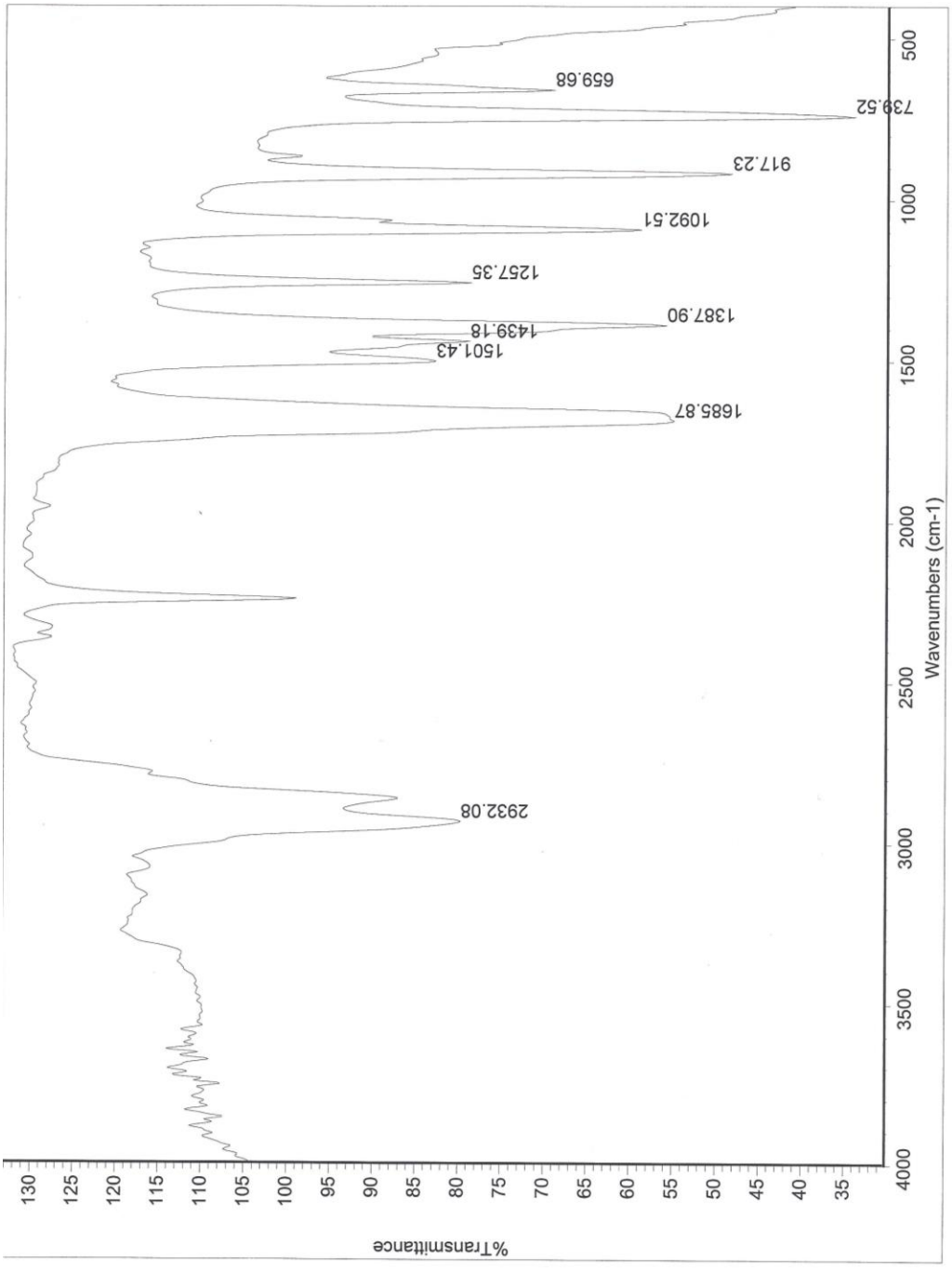

Figure 23: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-phenylmethanimine **10**

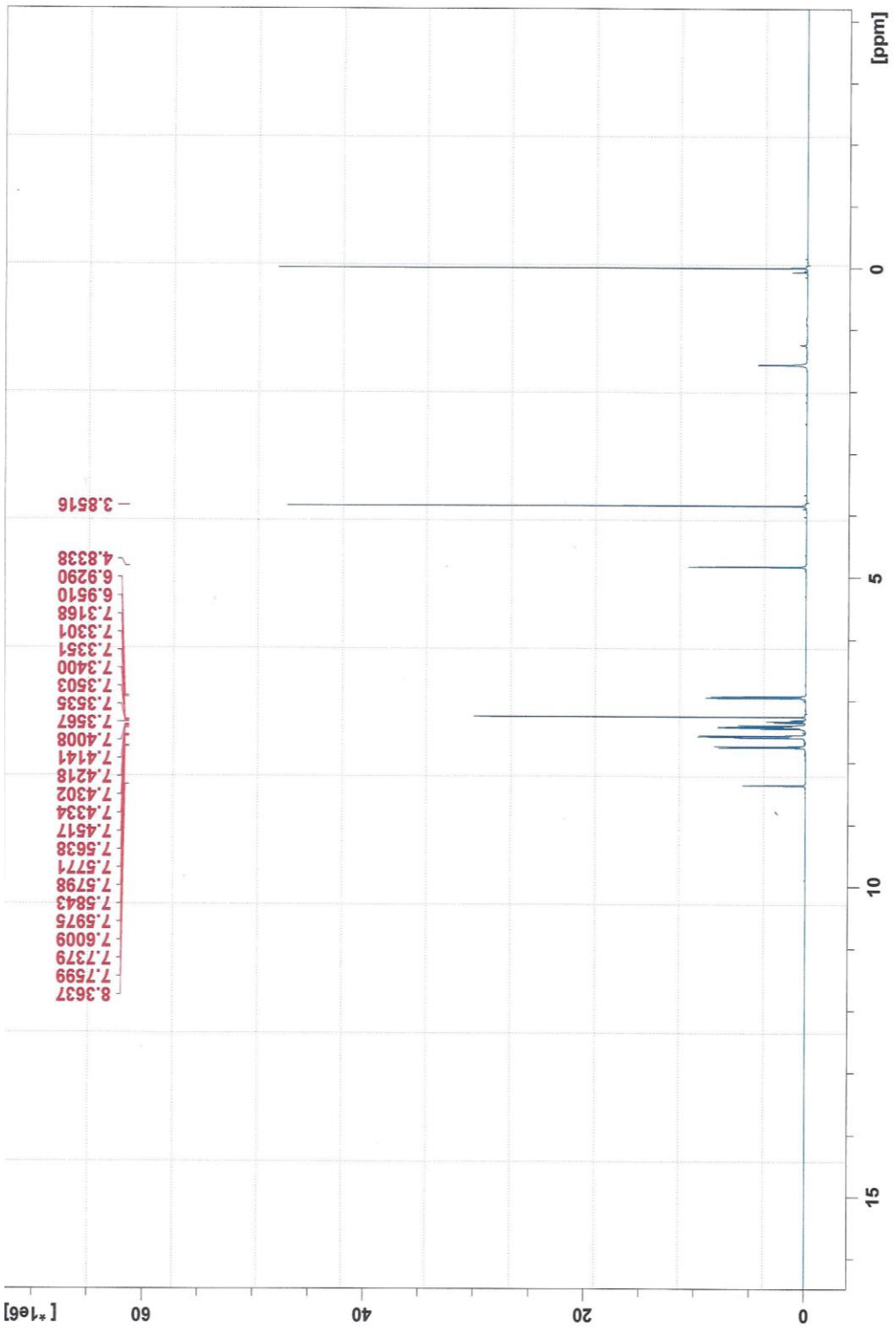

Figure 24: ^{1}H NMR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-methoxyphenyl)methanimine **11**

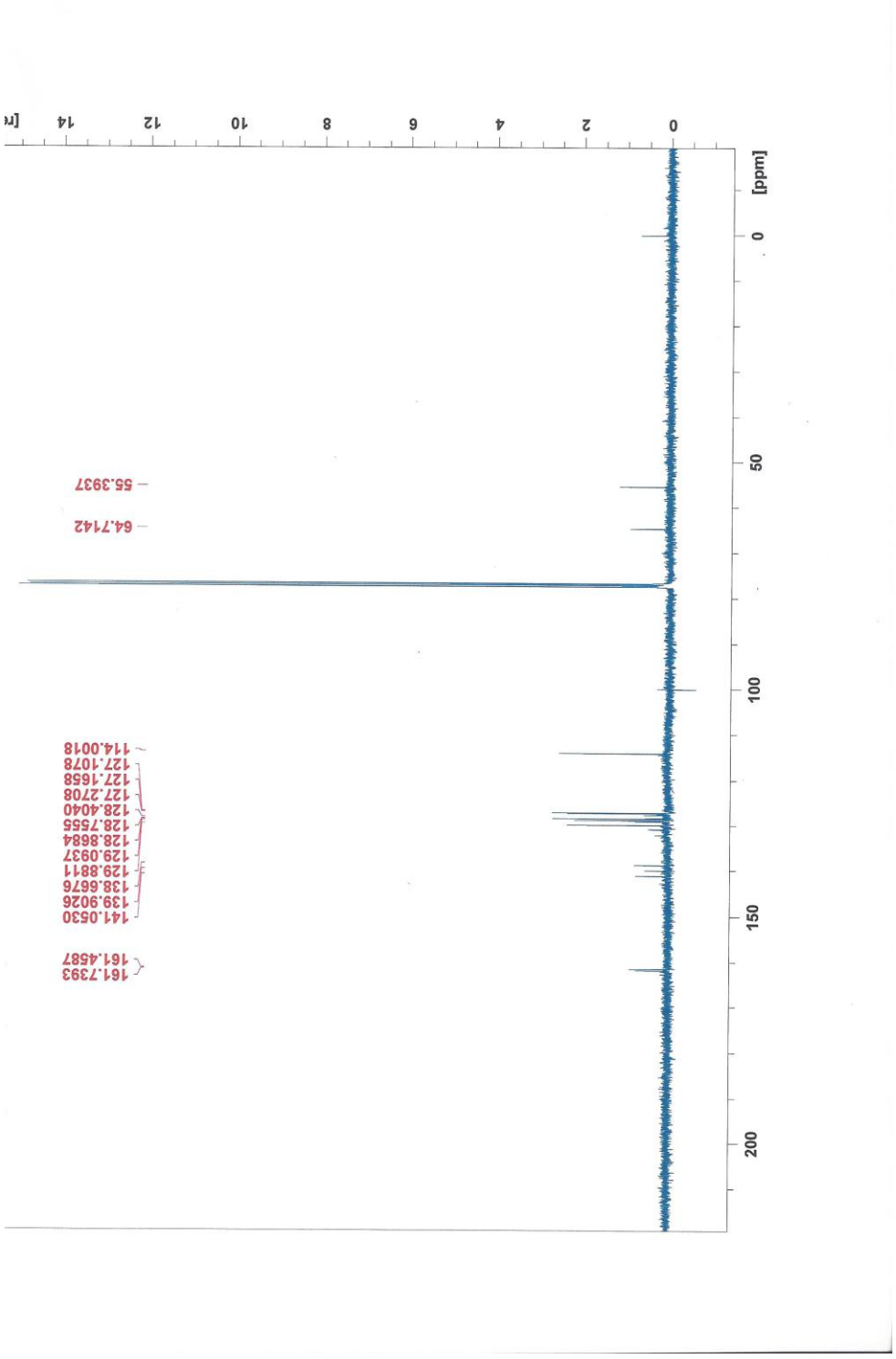

Figure 25: ^{13}C NMR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-methoxyphenyl)methanimine **11**

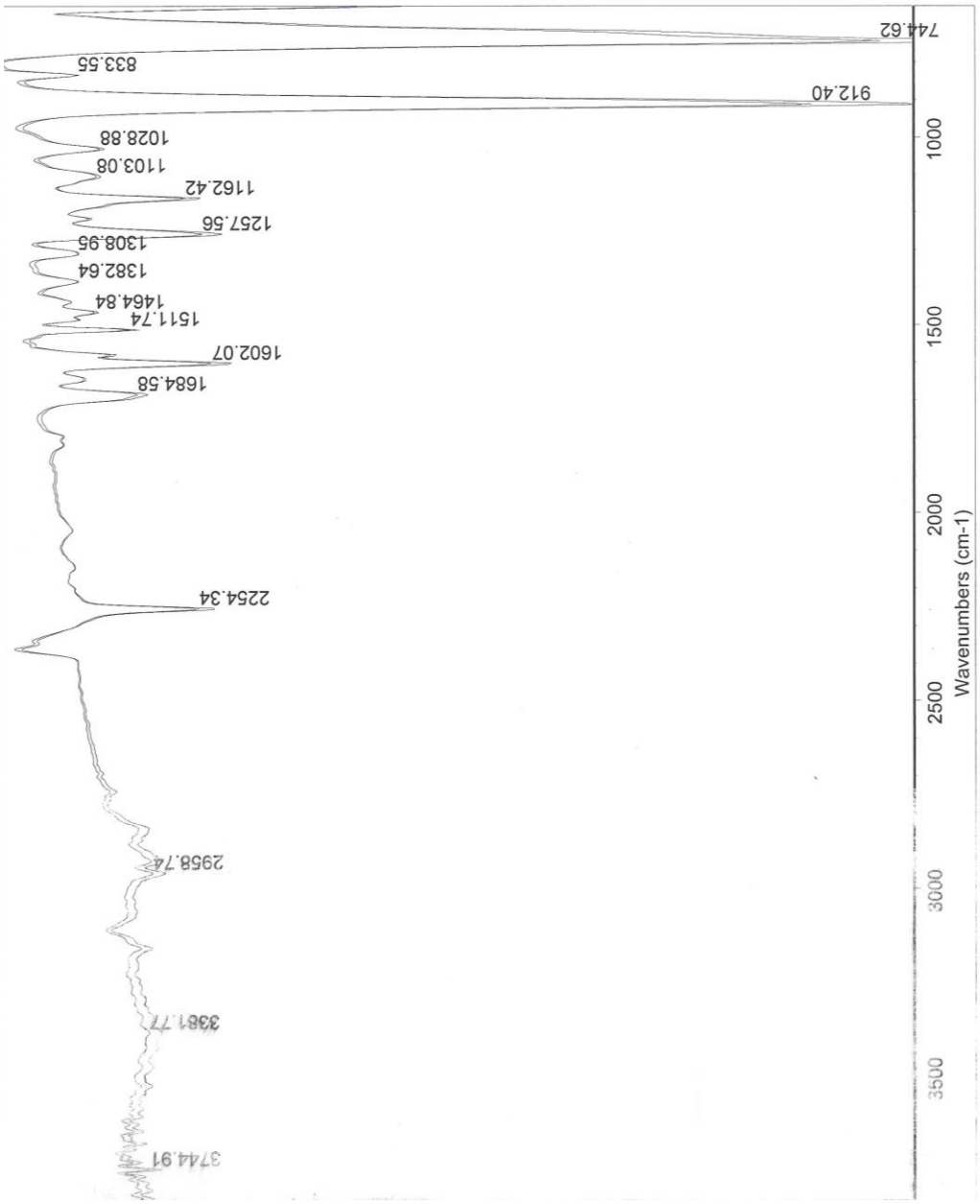

Figure 26: IR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-methoxyphenyl)methanimine **11**

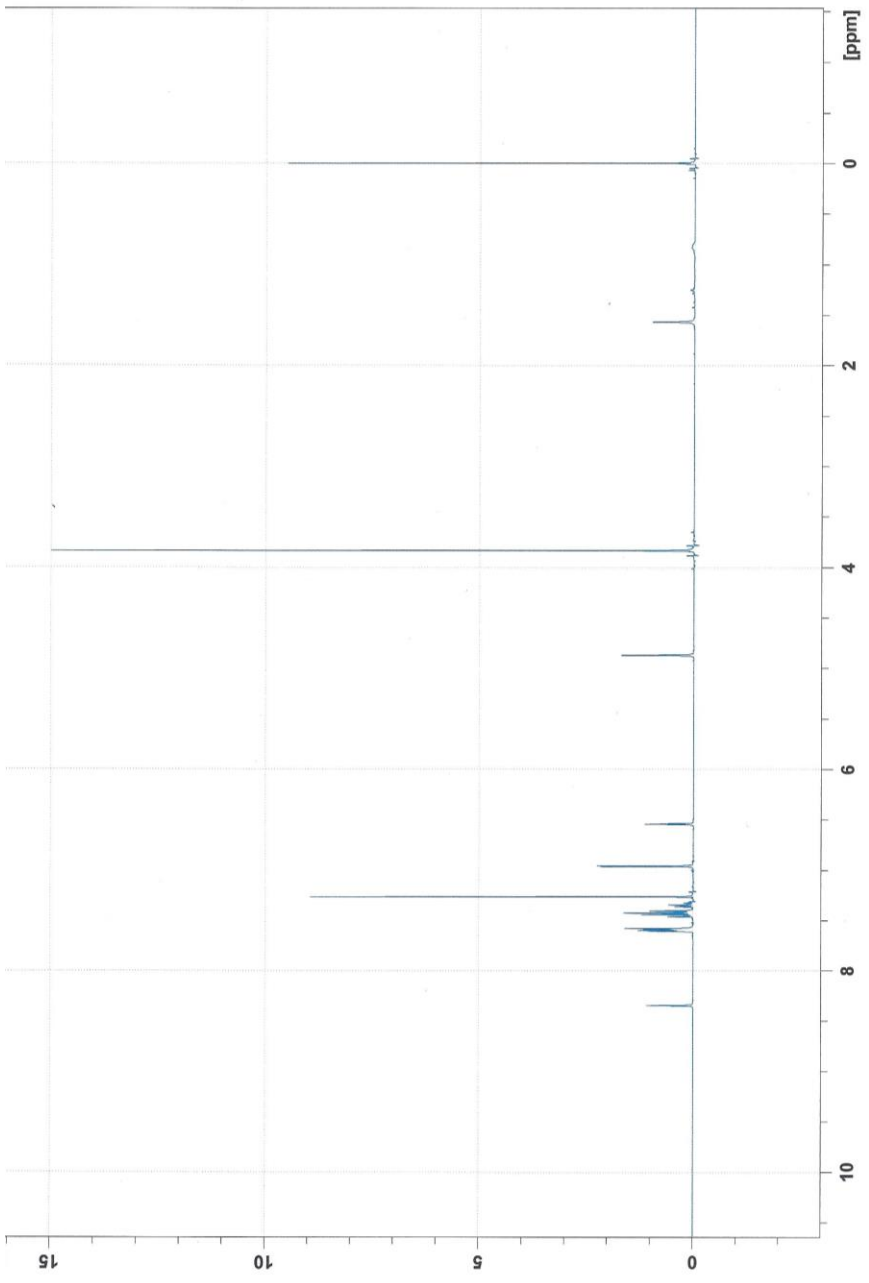

Figure 27:^{1}H NMR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(3,5-dimethoxyphenyl)methanimine **12**

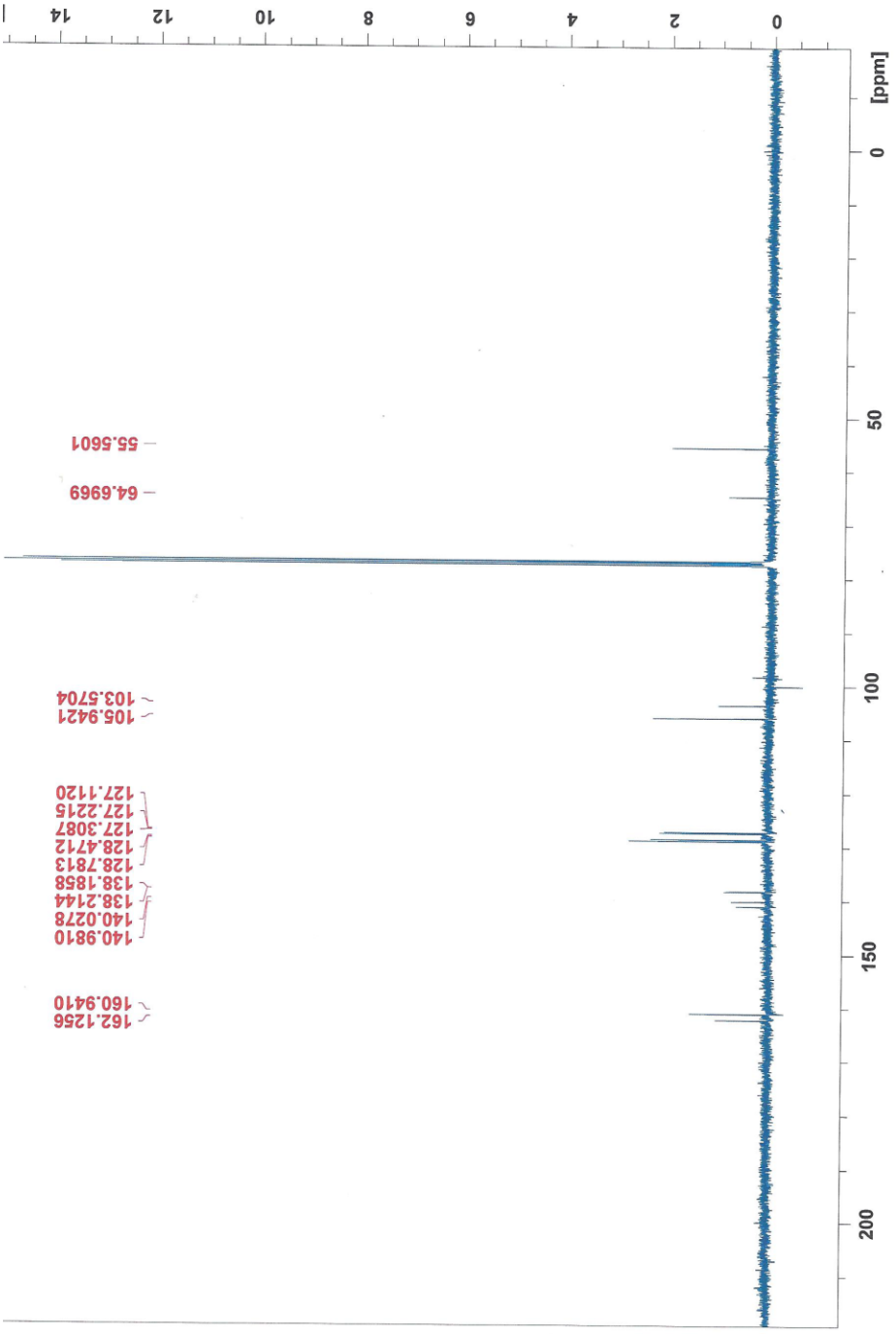

Figure 28: ^{13}C NMR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(3,5-dimethoxyphenyl)methanimine **12**

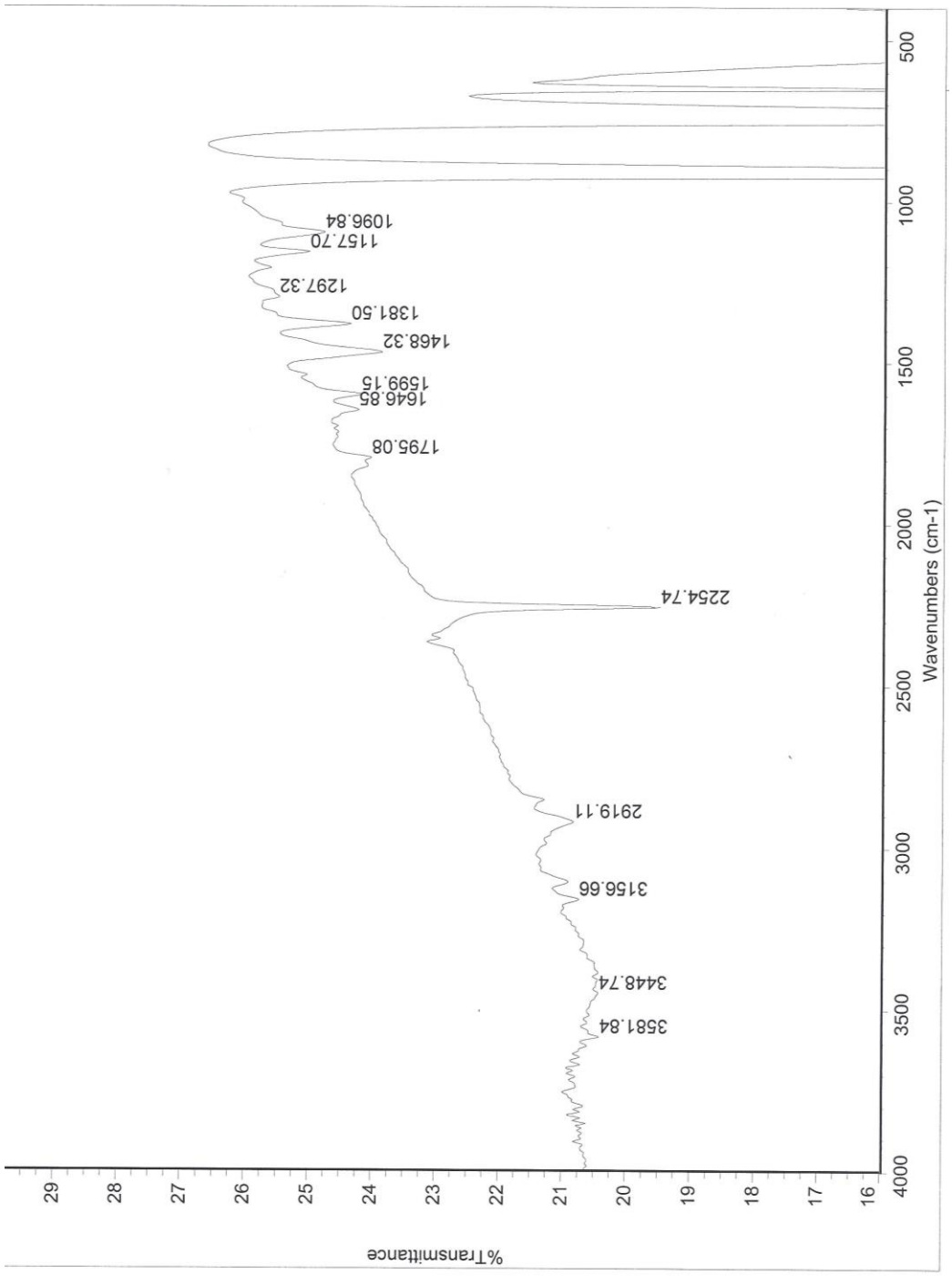

Figure 29: IR spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(3,5-dimethoxyphenyl)methanimine **12**

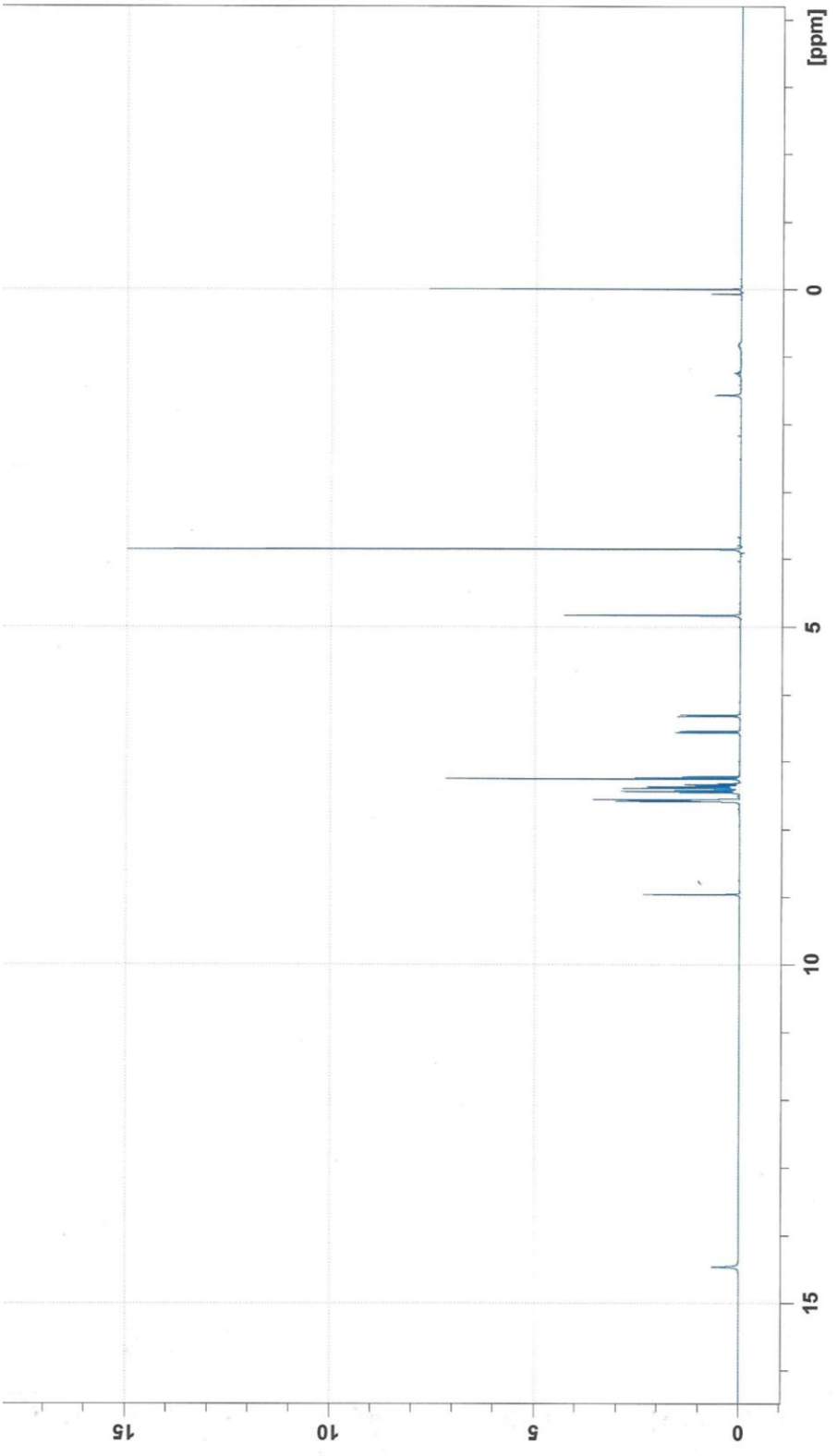

Figure 30: ^{1}H NMR Spectrum of (*E*)-2-(([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-3-methoxyphenol **13**

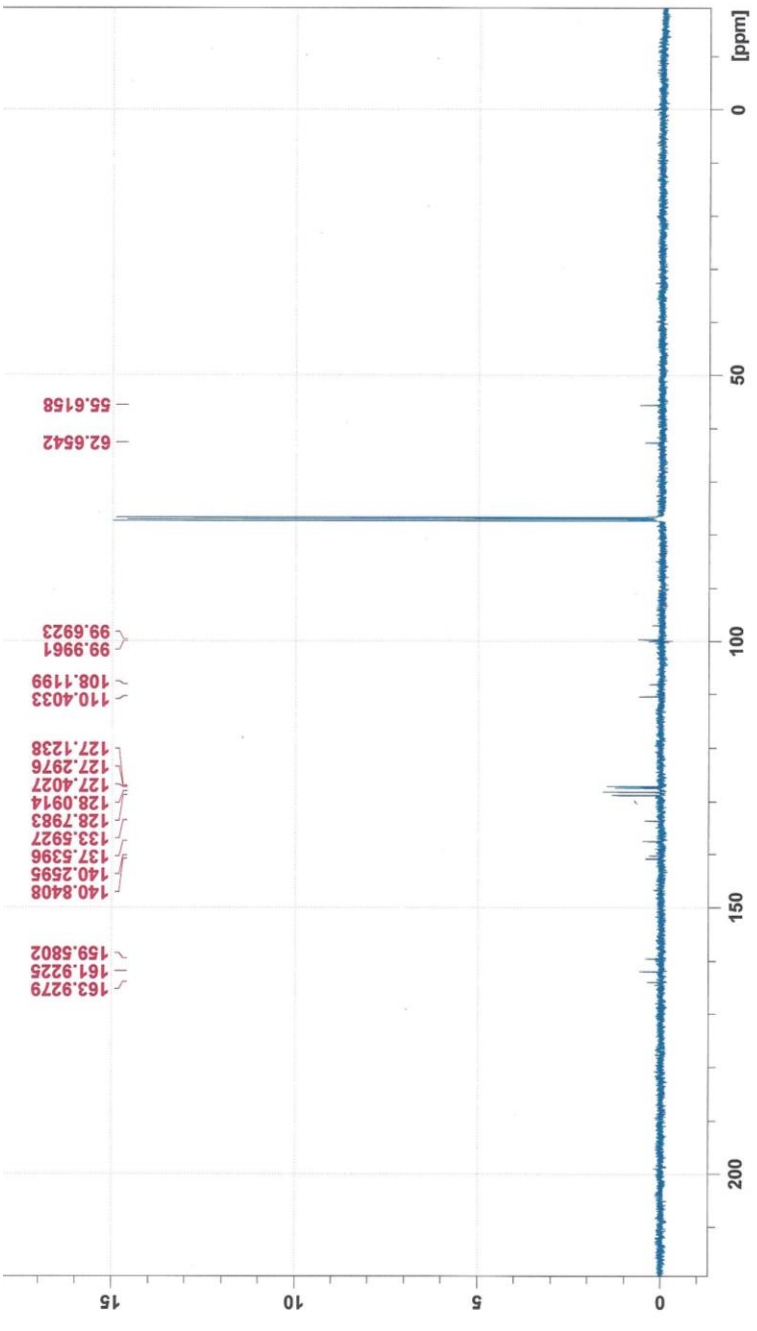

Figure 31: ^{13}C NMR Spectrum of (*E*)-2-(([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-3-methoxyphenol **13**

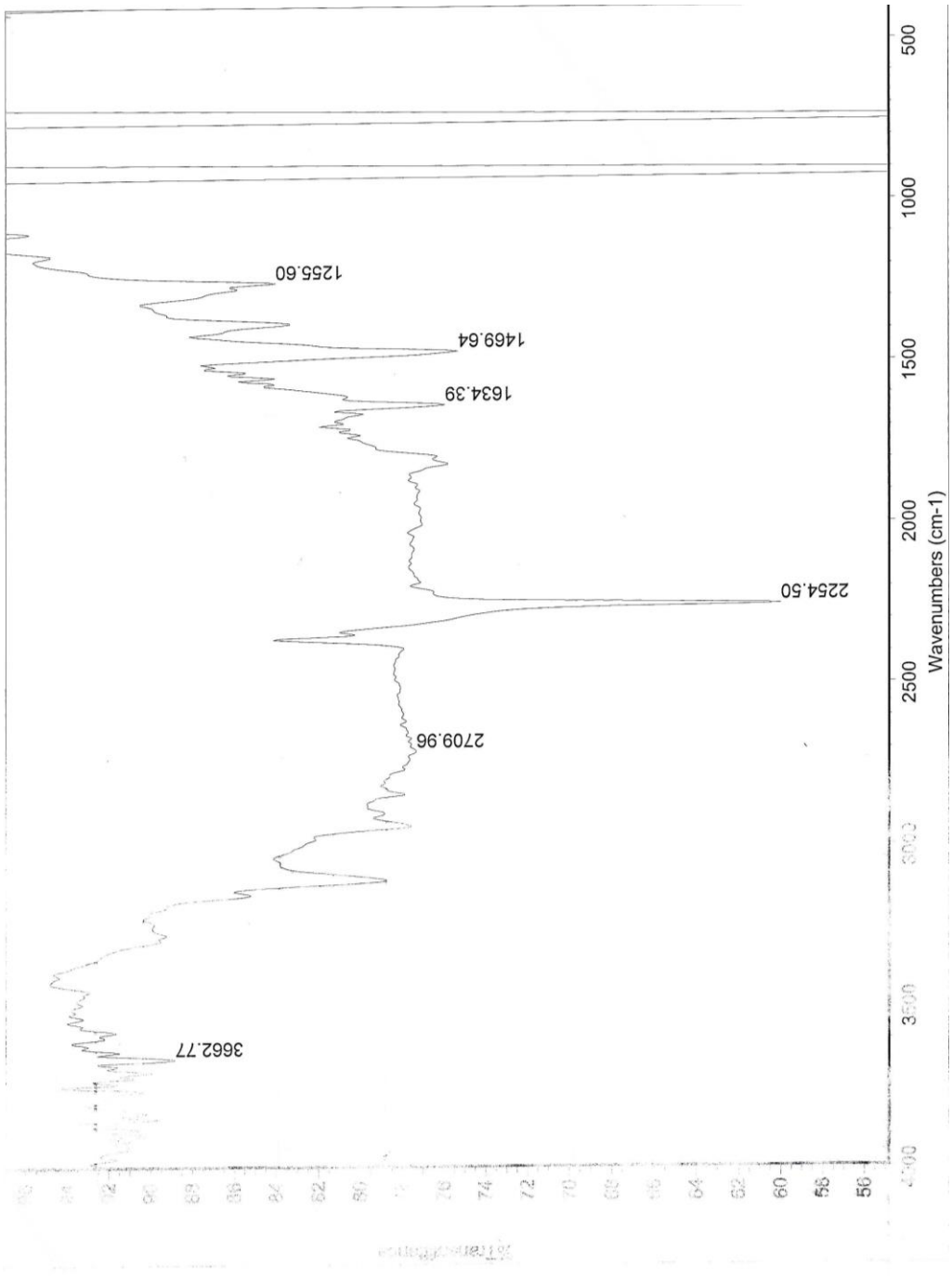

Figure 32: IR Spectrum of (*E*)-2-(((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-3-methoxyphenol **13**

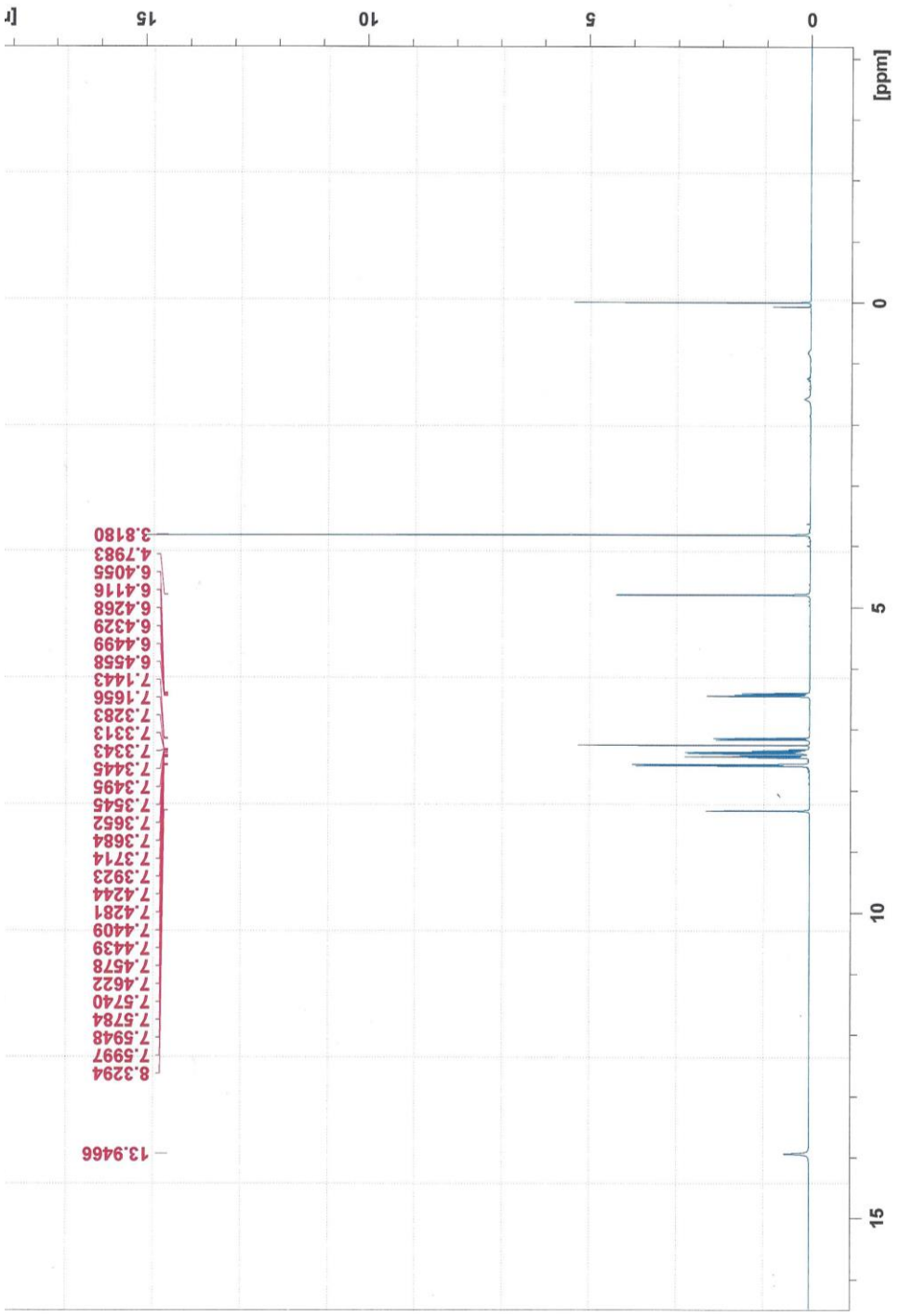

Figure 33: ^{1}H NMR Spectrum (*E*)-2-(((1,1'-biphenyl]-4-ylmethyl)imino)methyl)-5-methoxyphenol **14**

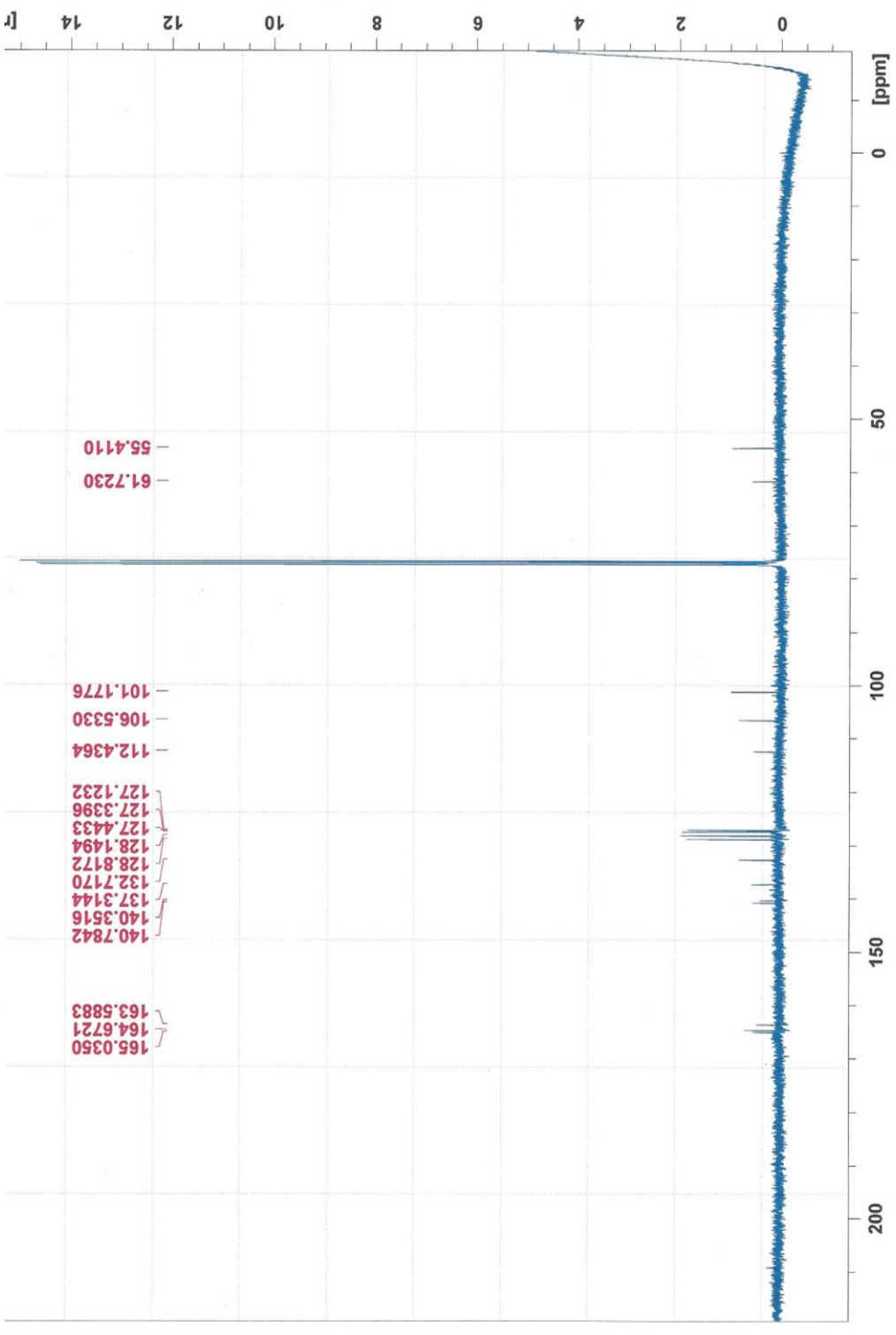

Figure 34: ^{13}C NMR Spectrum (*E*)-2-(([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-5-methoxyphenol

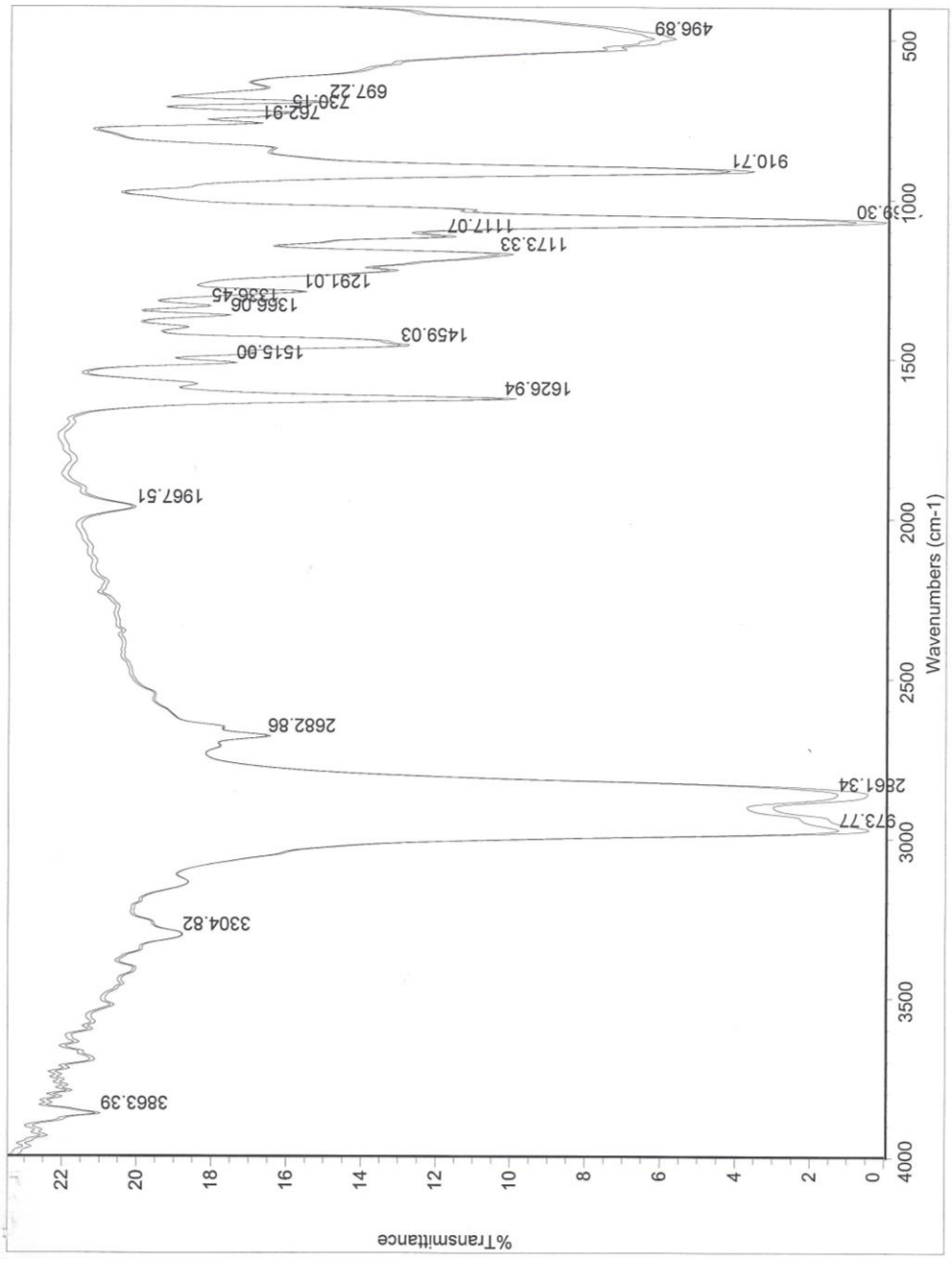

Figure 35: IR Spectrum (*E*)-2-(((1,1'-biphenyl]-4-ylmethyl)imino)methyl)-5-methoxyphenol **14**

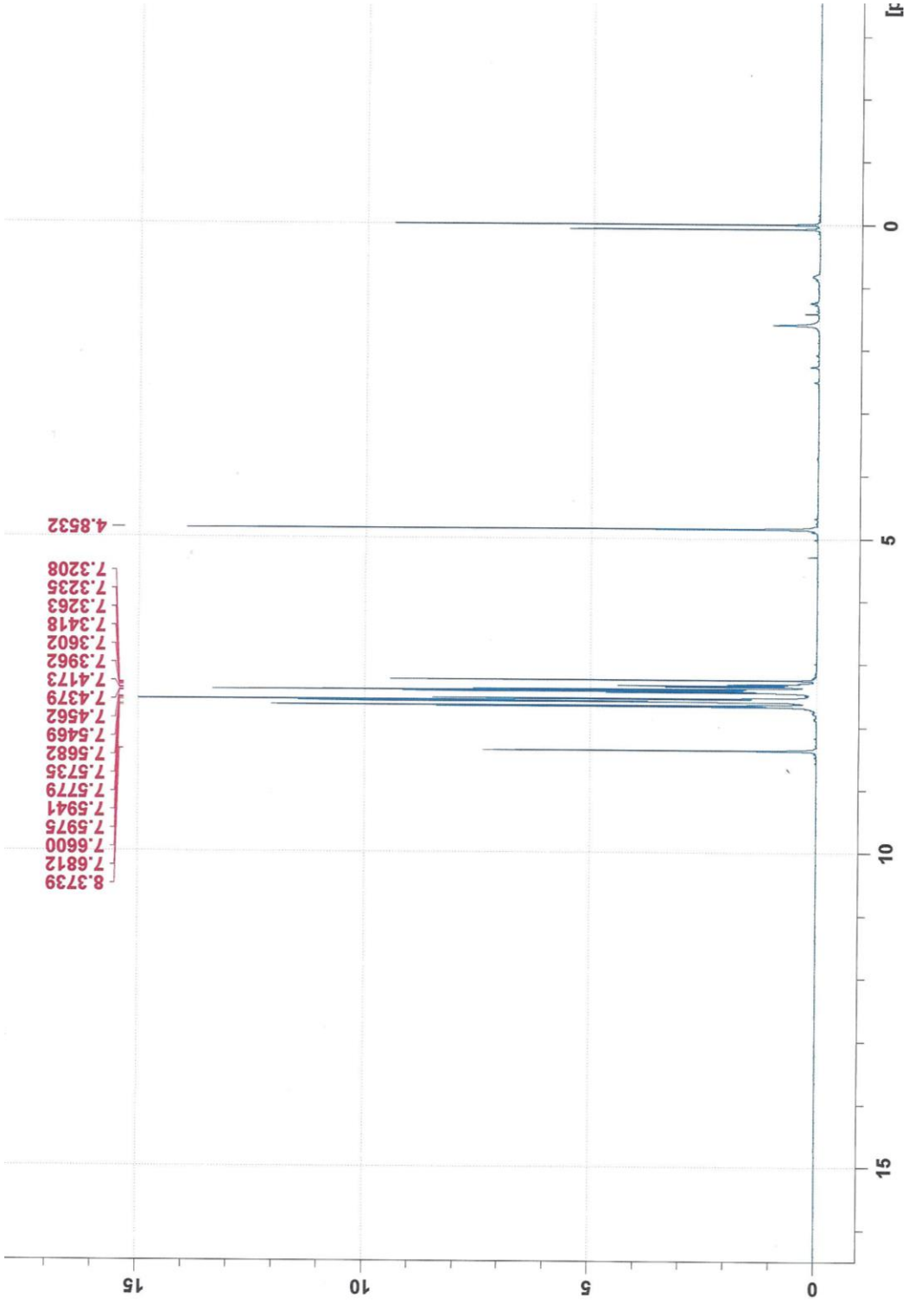

Figure 36: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-bromophenyl)methanimine **15**

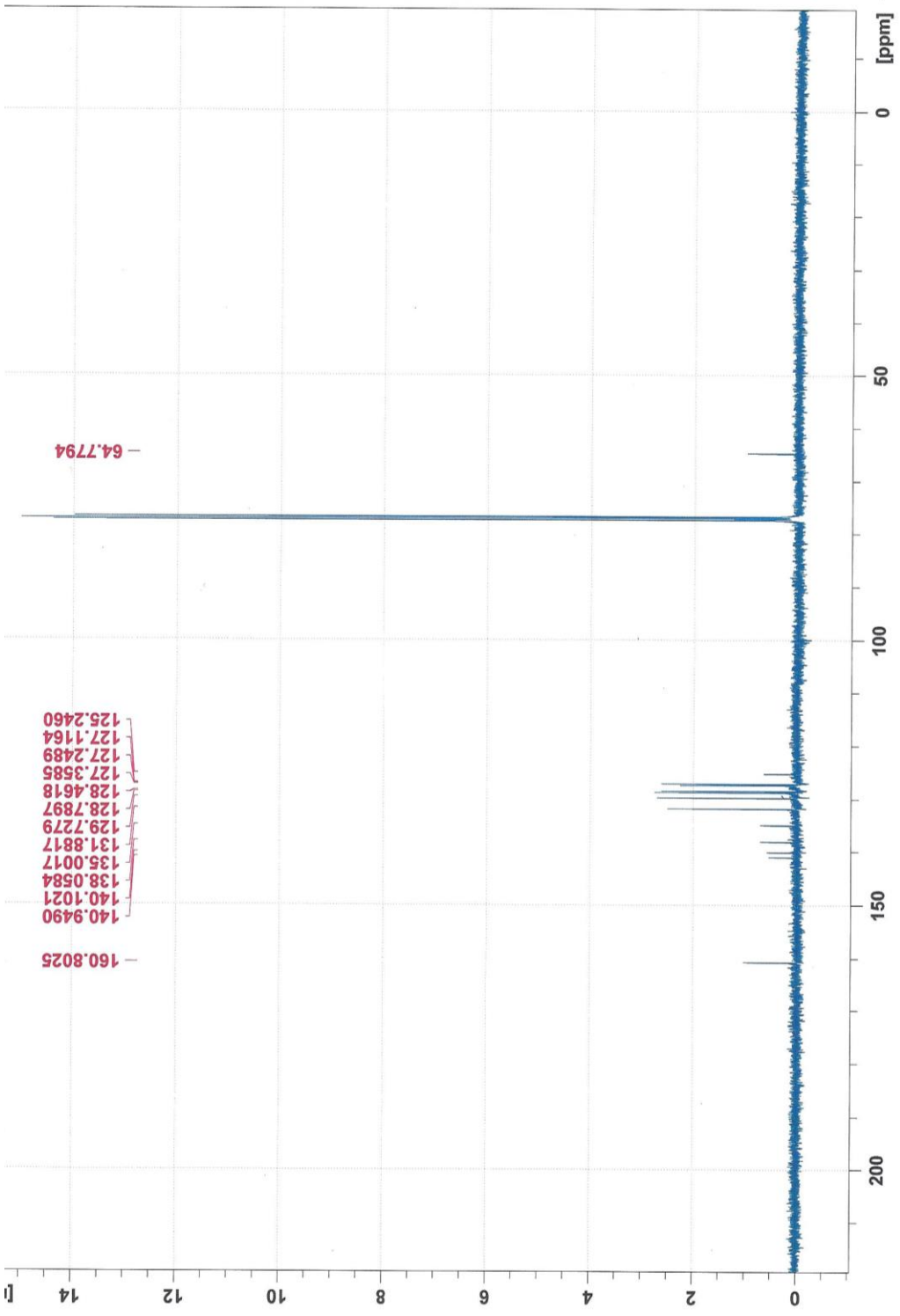

Figure 37.[13] C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-bromophenyl)methanimine **15**

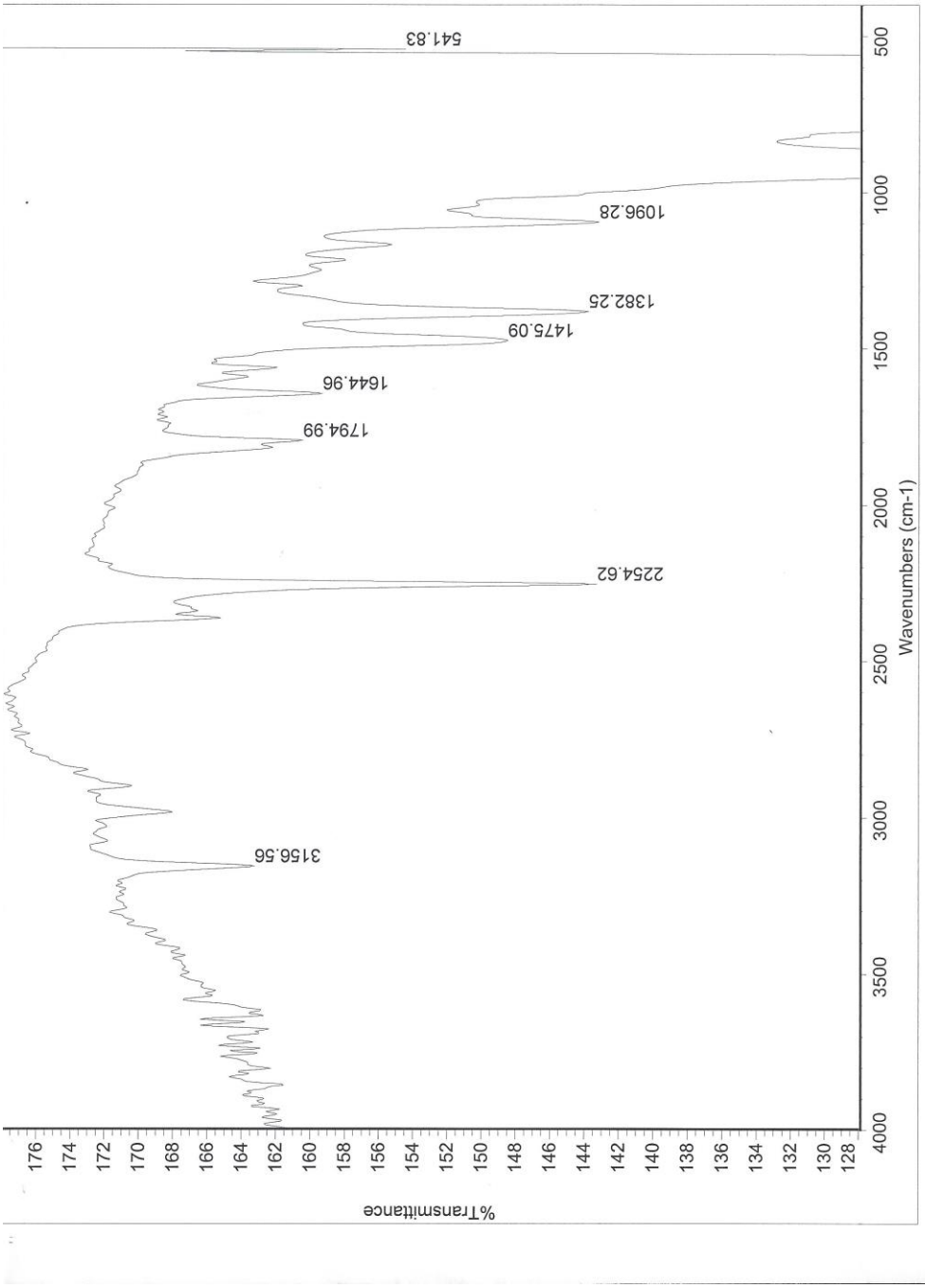

Figure 38: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-bromophenyl)methanimine **15**

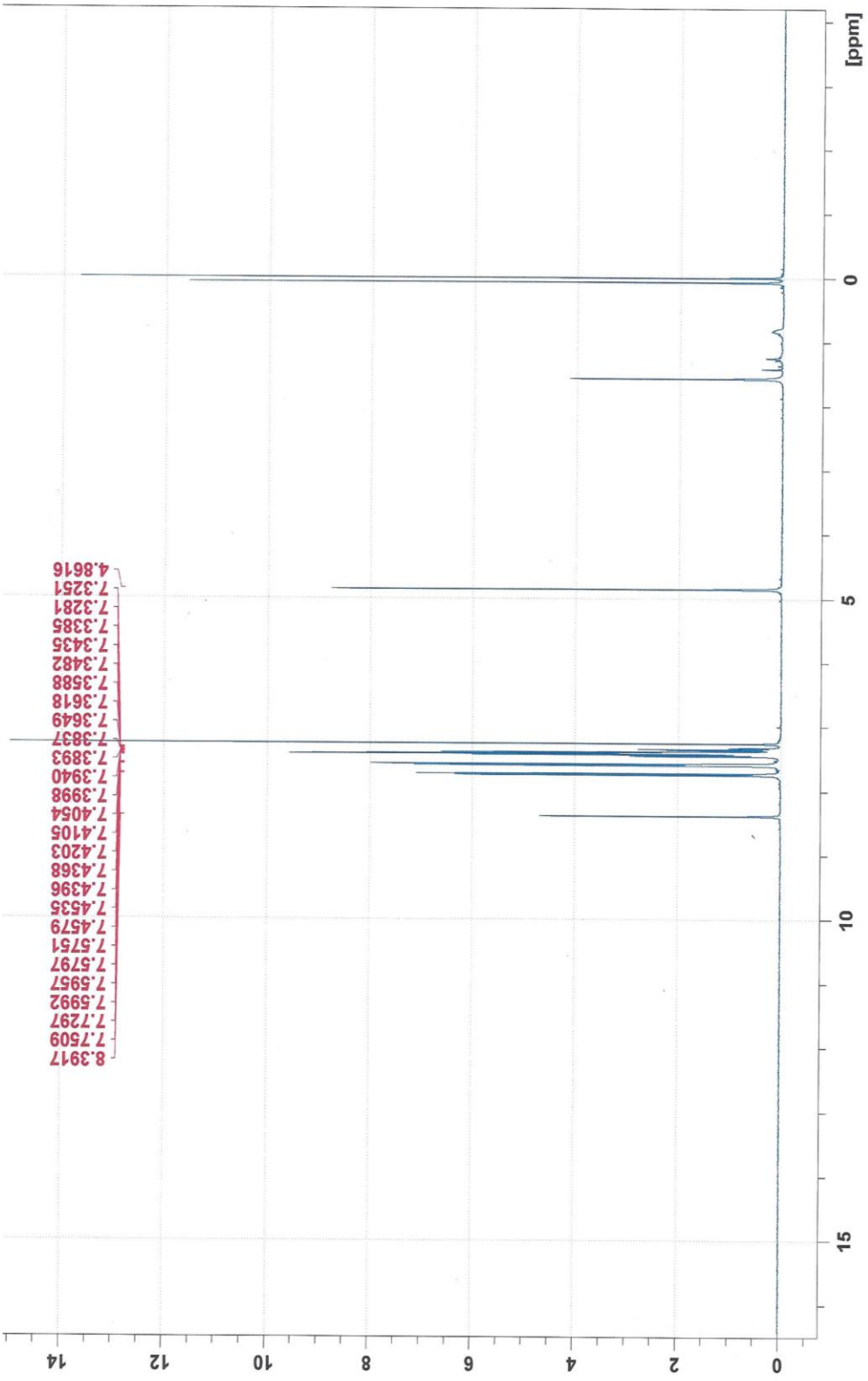

Figure 39: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-chlorophenyl)methanimine **16**

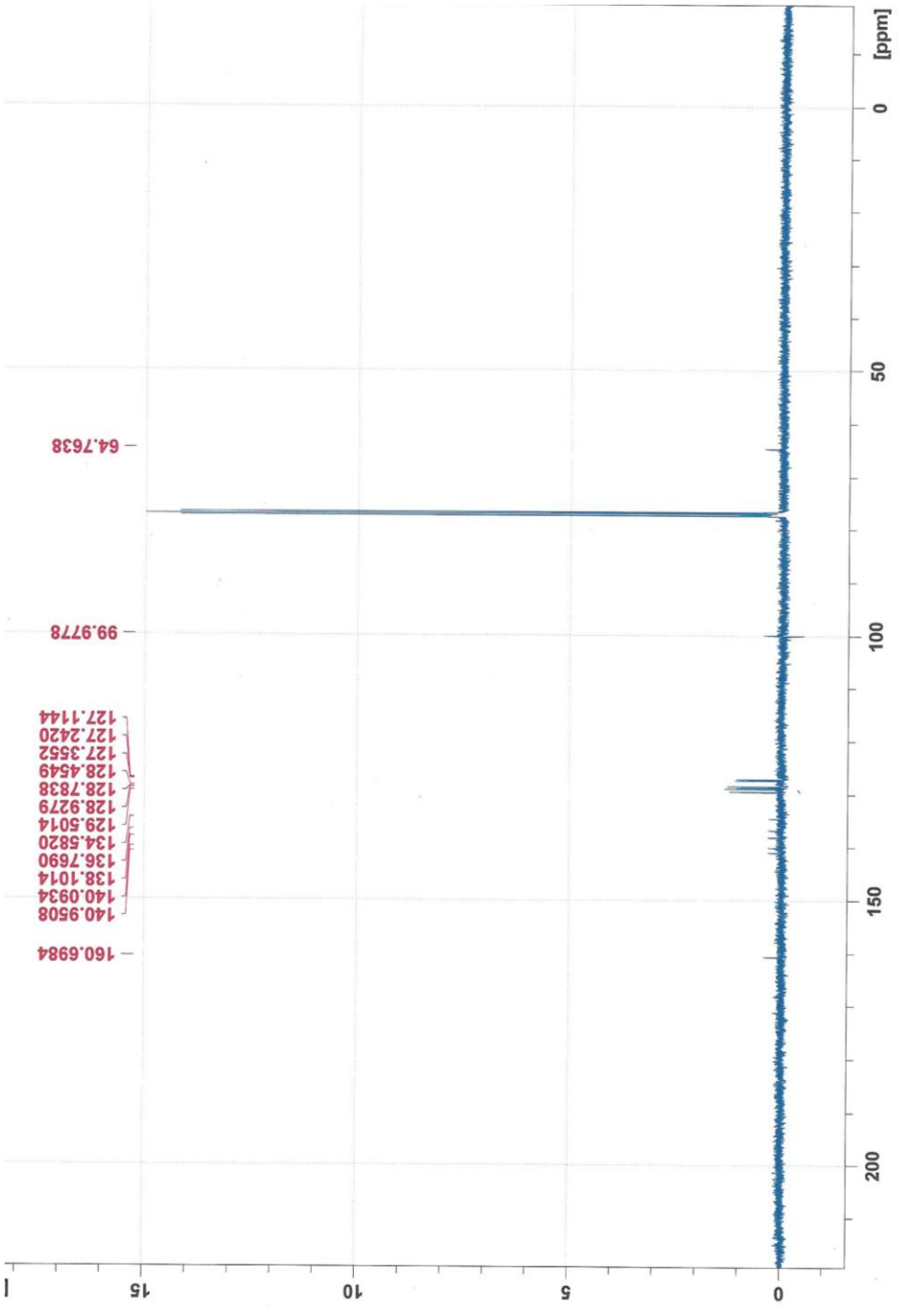

Figure 40: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-chlorophenyl)methanimine **16**

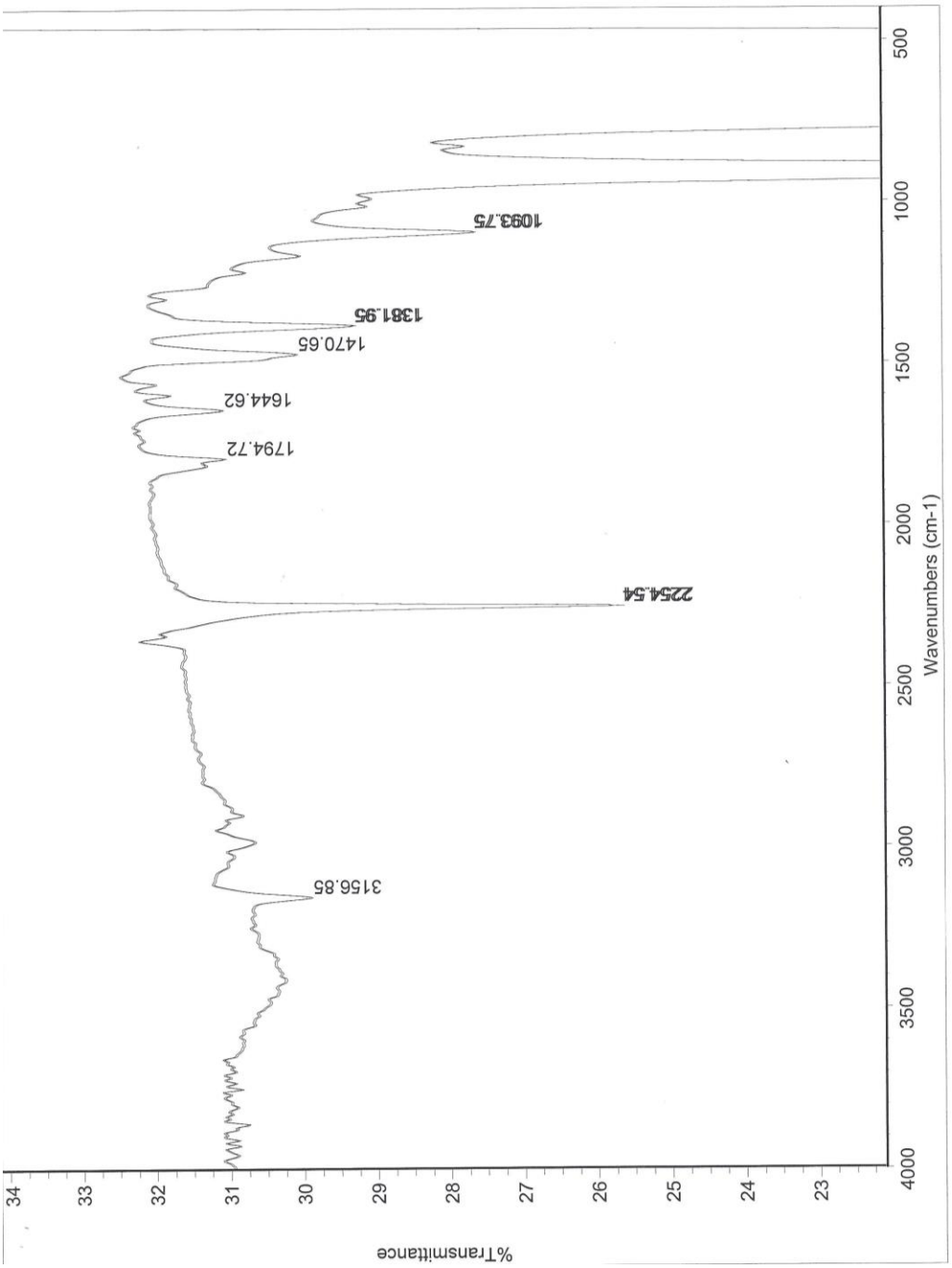

Figure 41: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-chlorophenyl)methanimine **16**

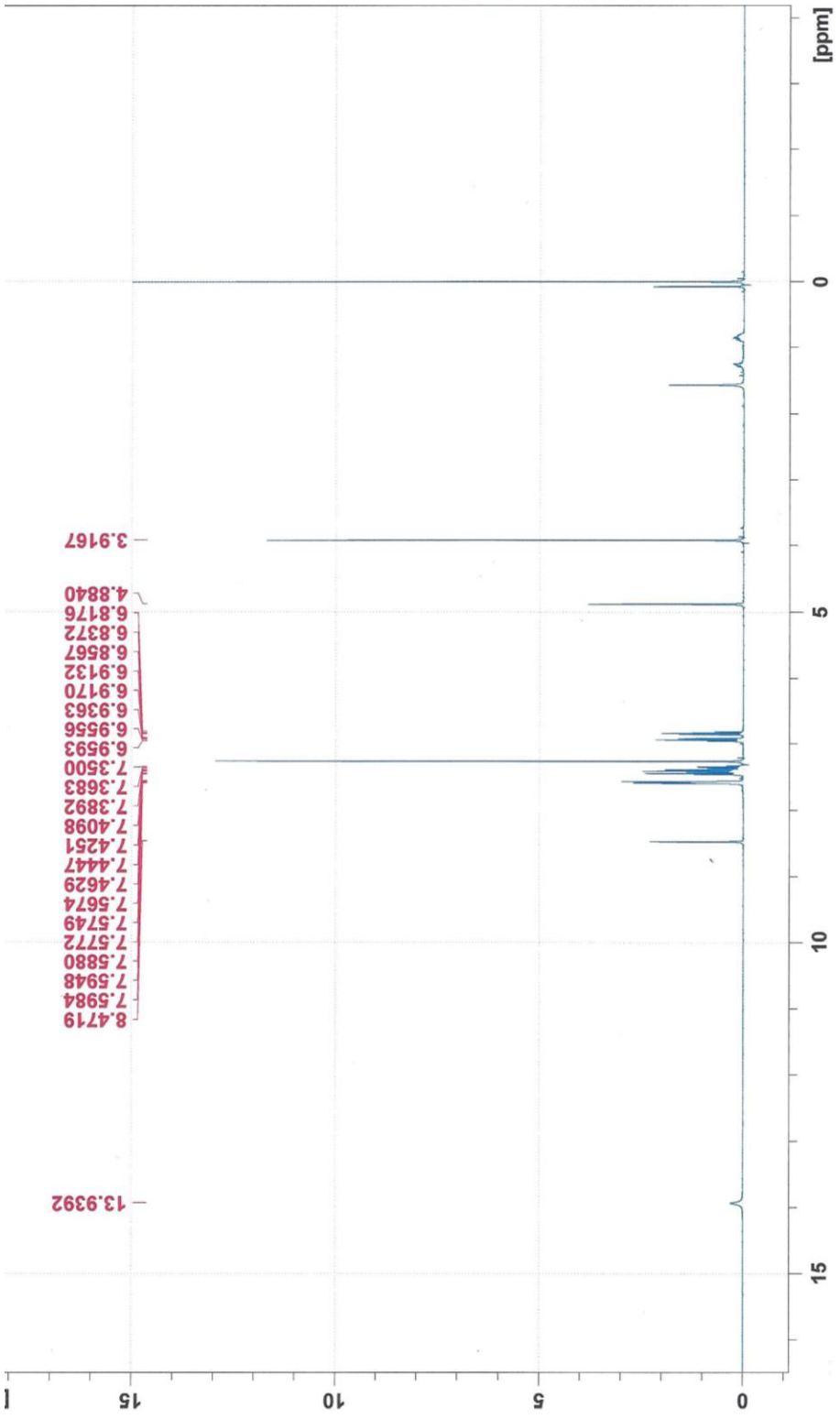

Figure 42: ^{1}H NMR Spectrum of (*E*)-2-(((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-6-methoxyphenol **17**

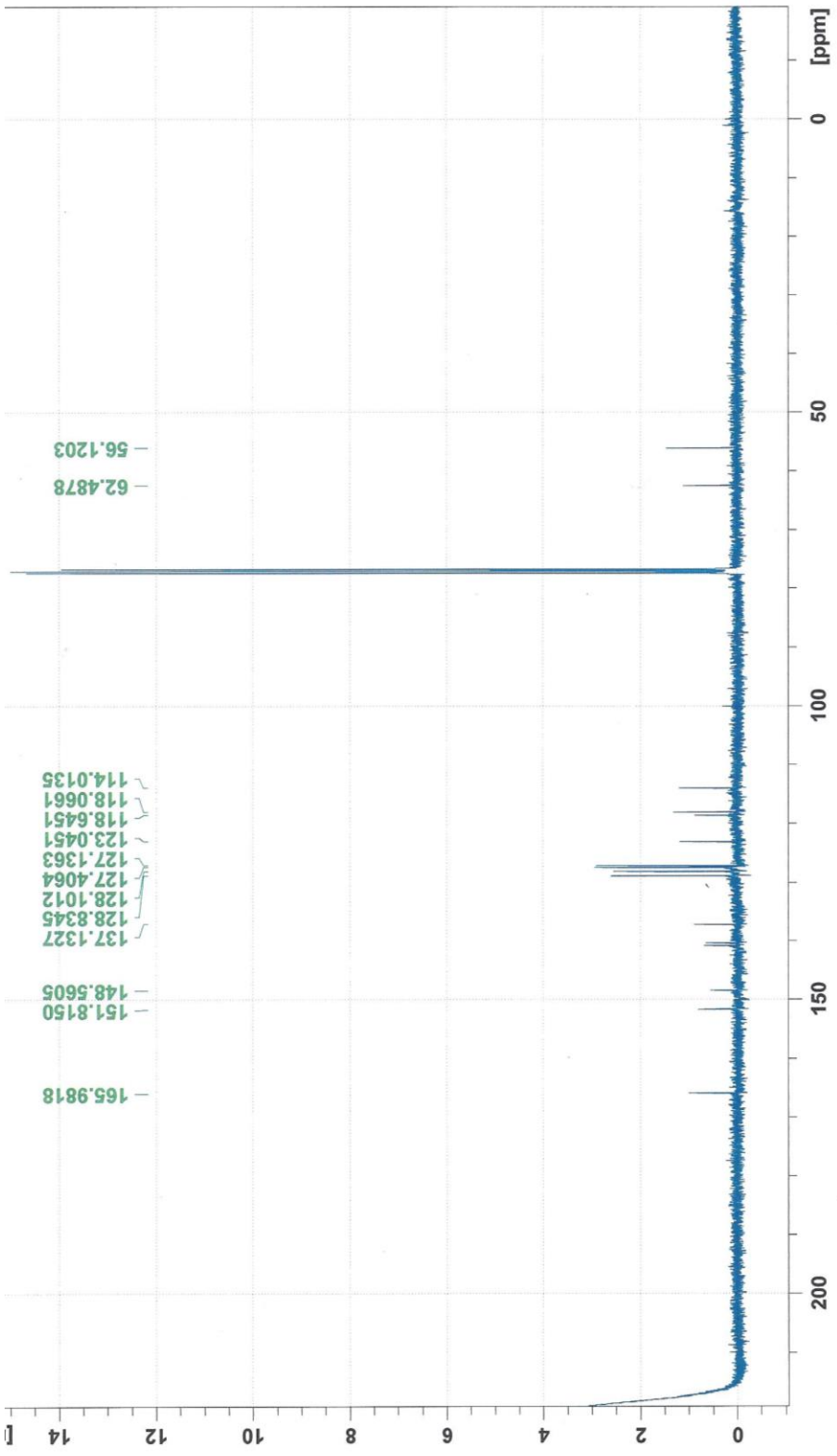

Figure 43: ^{13}C NMR Spectrum of (*E*)-2-(((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-6-methoxyphenol **17**

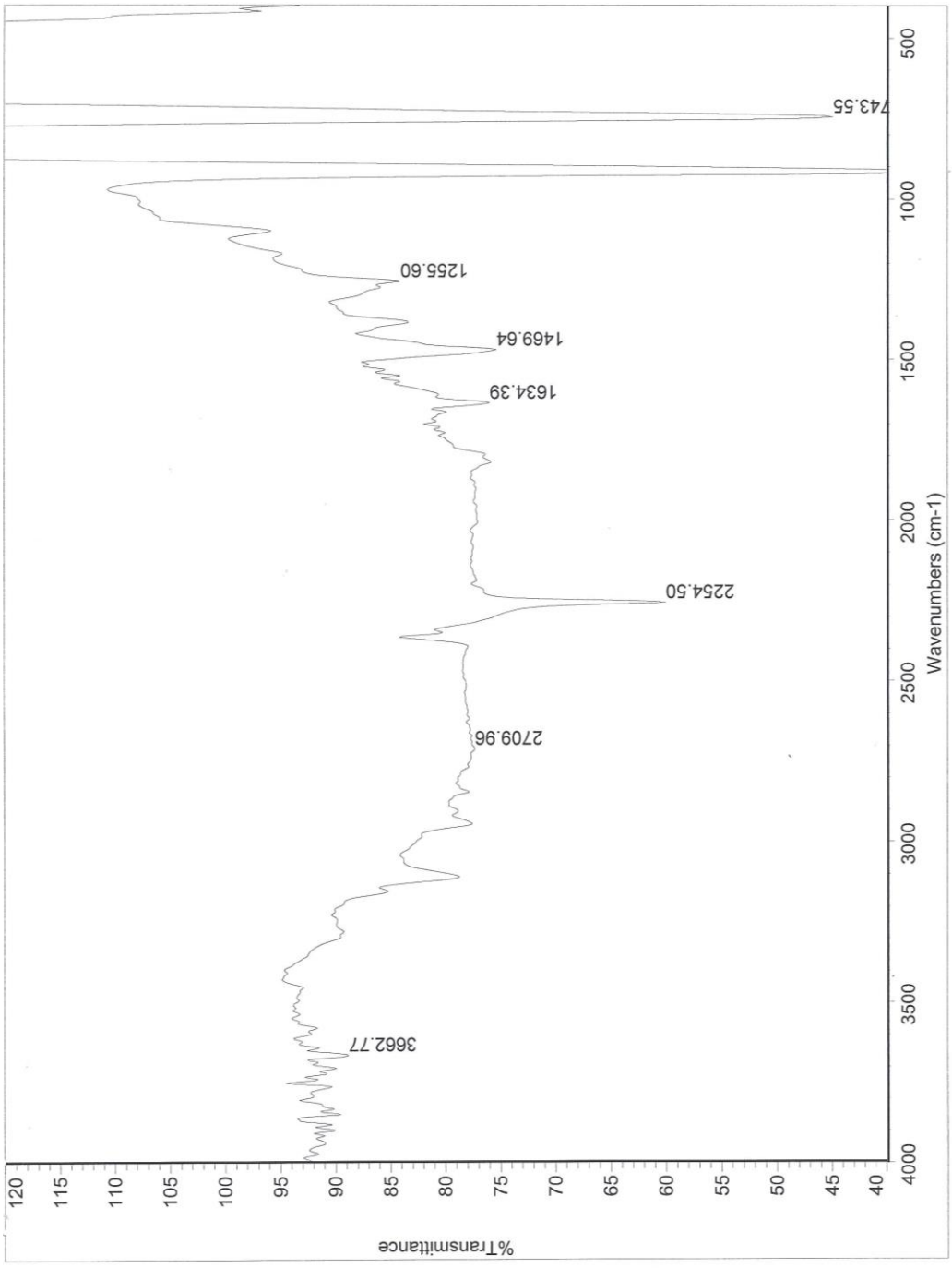

Figure 44: IR Spectrum of (*E*)-2-(([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-6-methoxyphenol **17**

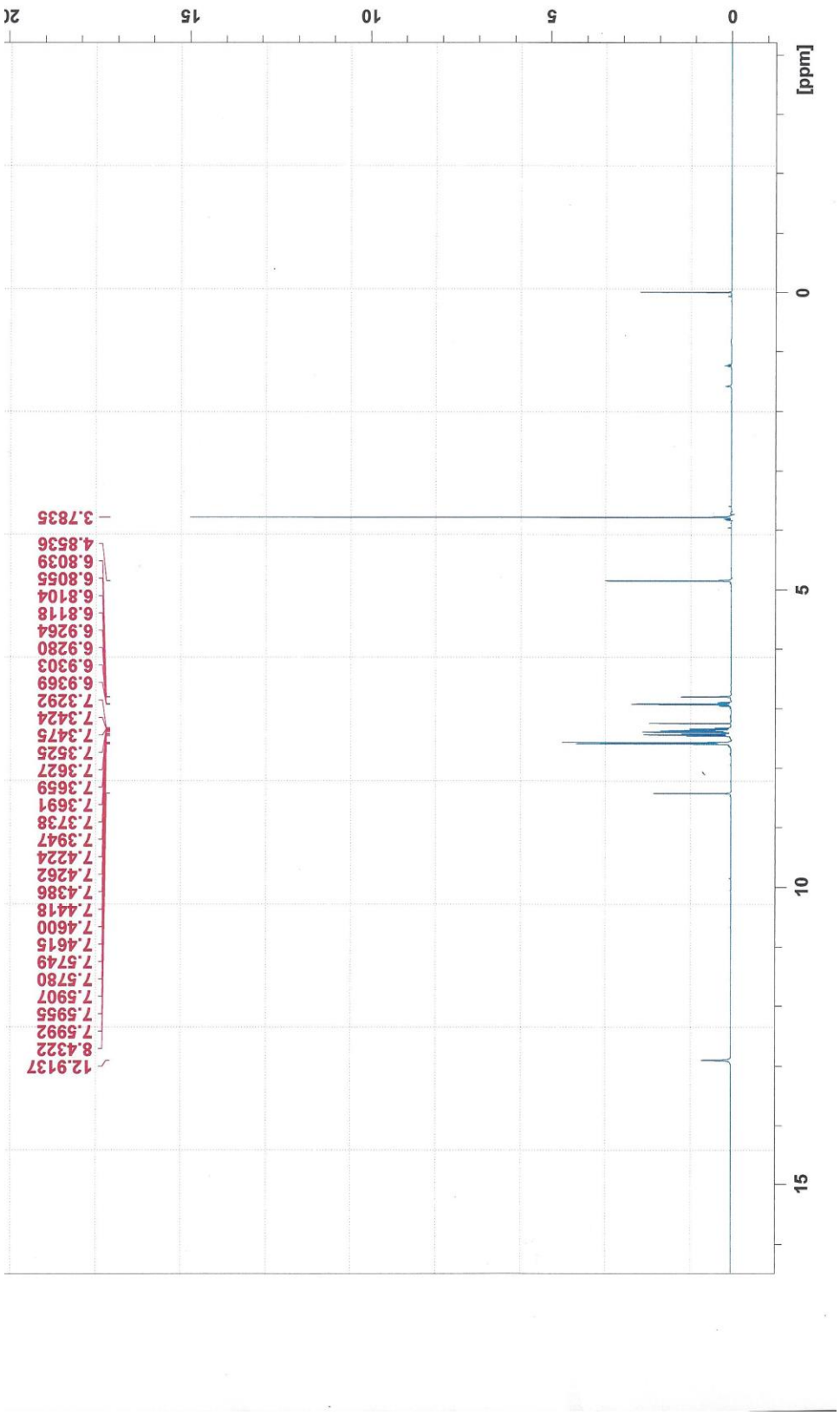

Figure 45: ^{1}H NMR Spectrum of (*E*)-2-(((1,1'-biphenyl]-4-ylmethyl)imino)methyl)-4-methoxyphenol **18**

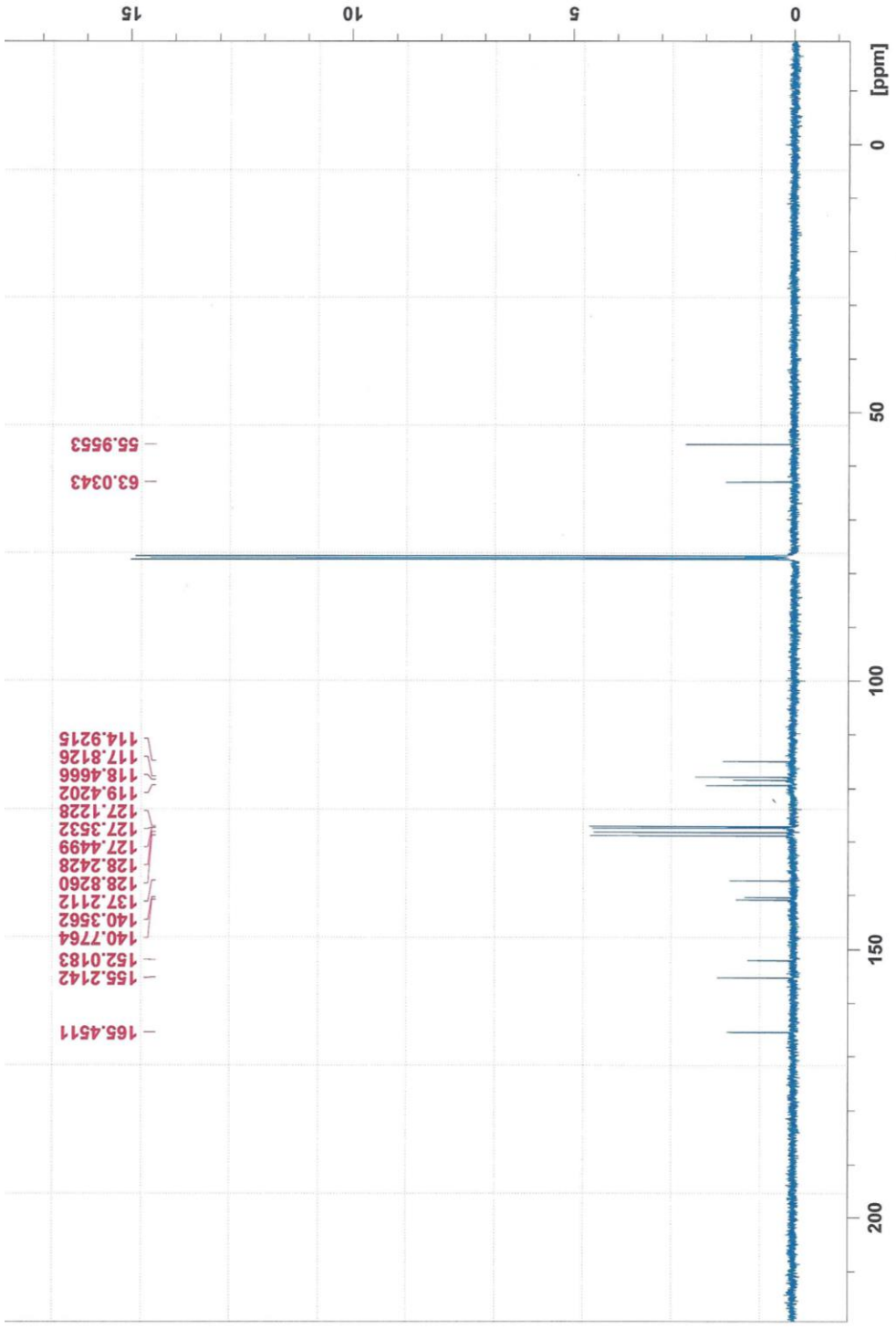

Figure 46: ^{13}C NMR Spectrum of (*E*)-2-(((1,1'-biphenyl]-4-ylmethyl)imino)methyl)-4-methoxyphenol **18**

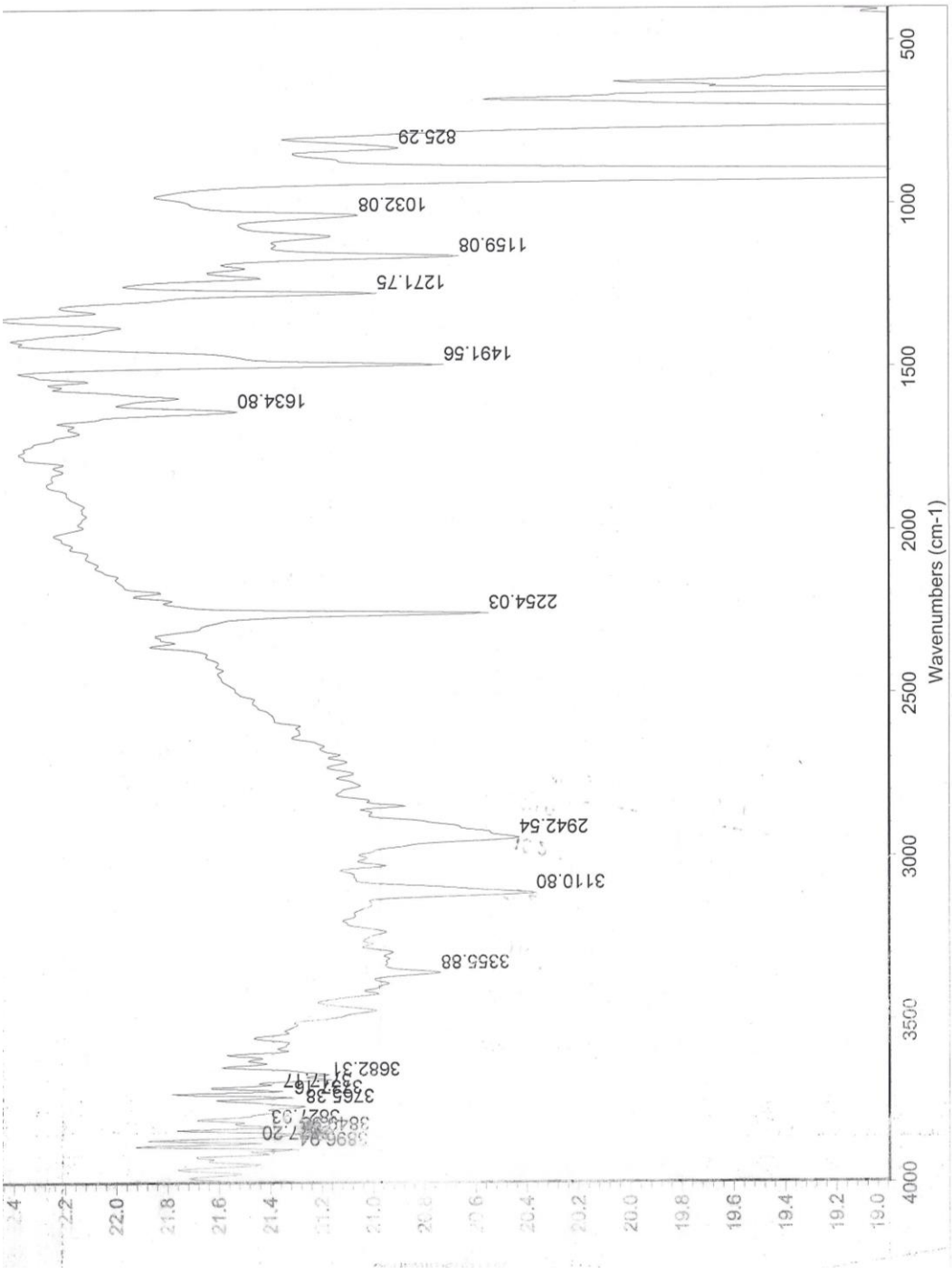

Figure 47: IR Spectrum of (*E*)-2-(((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-4-methoxyphenol **18**

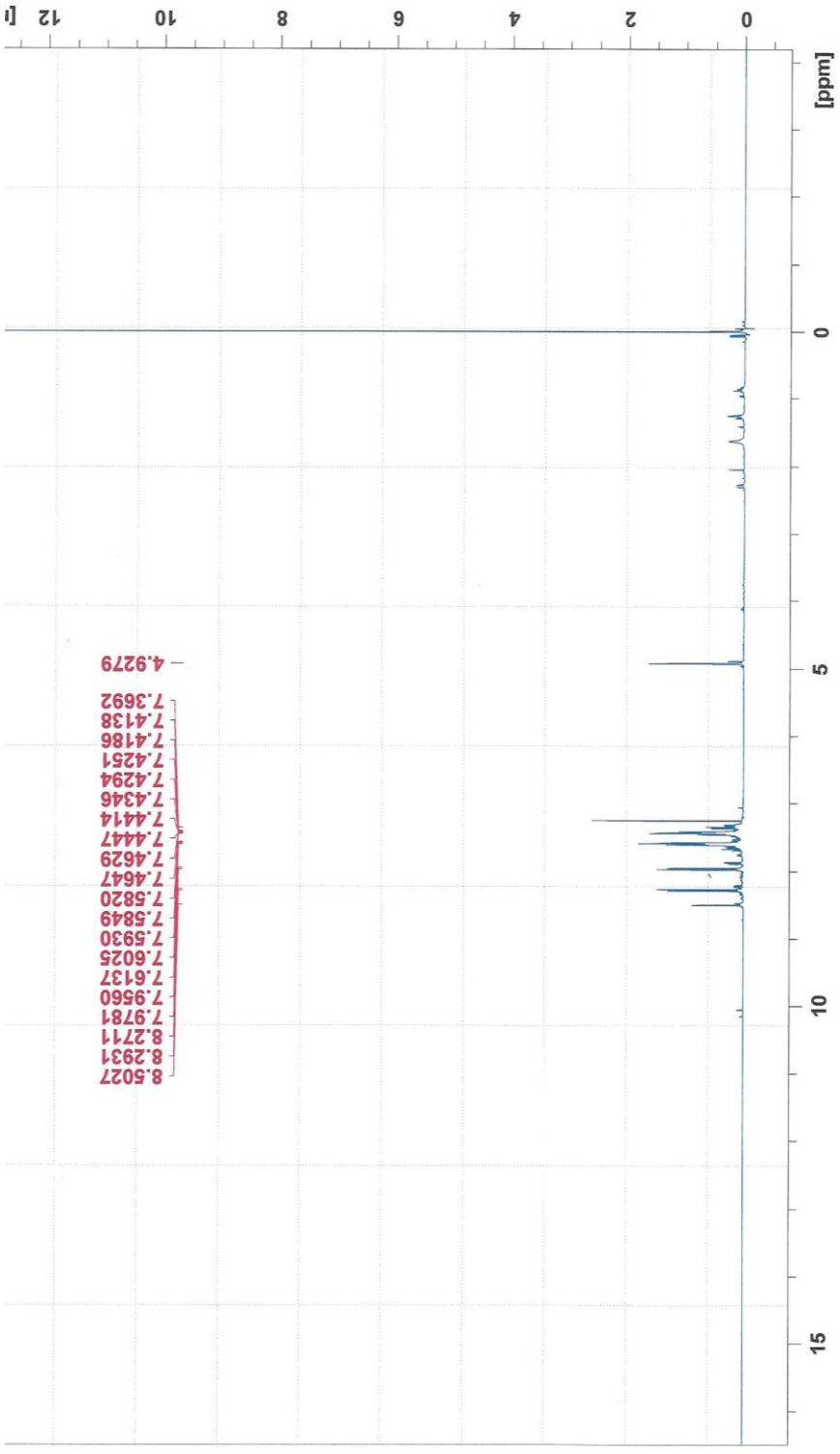

Figure 48: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-nitrophenyl)methanimine **19**

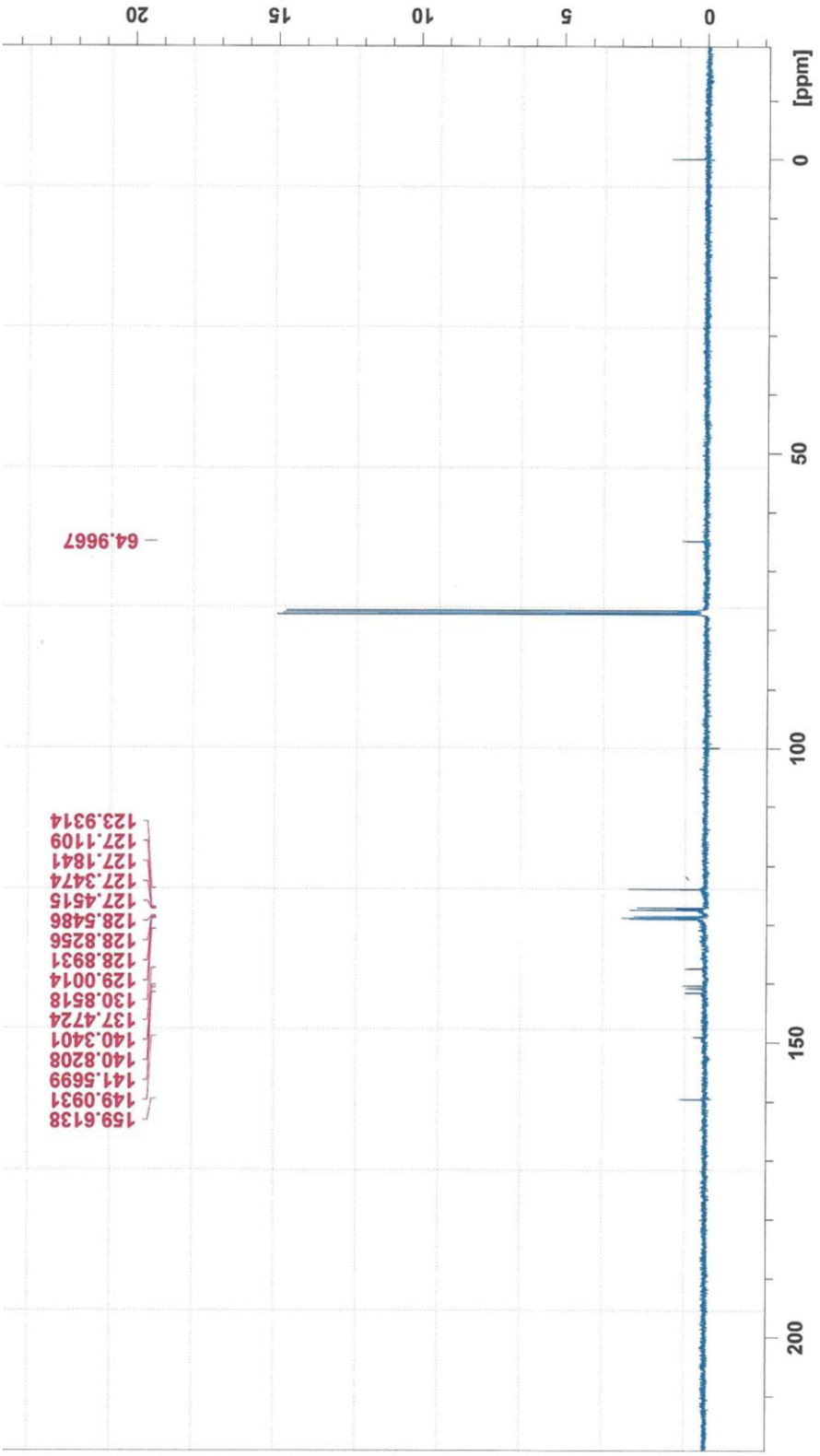

Figure 49: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-nitrophenyl)methanimine **19**

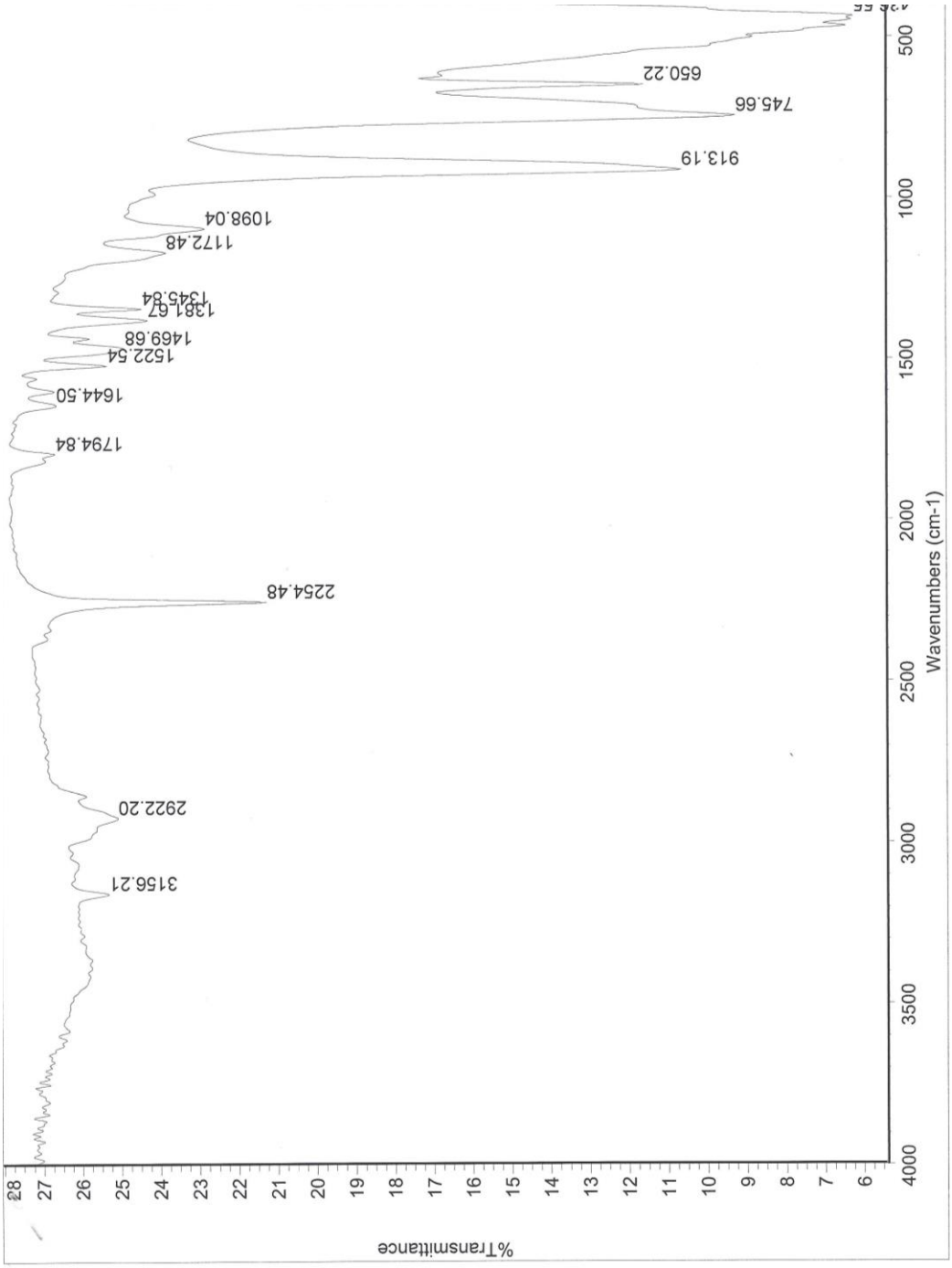

Figure 50: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-nitrophenyl)methanimine **19**

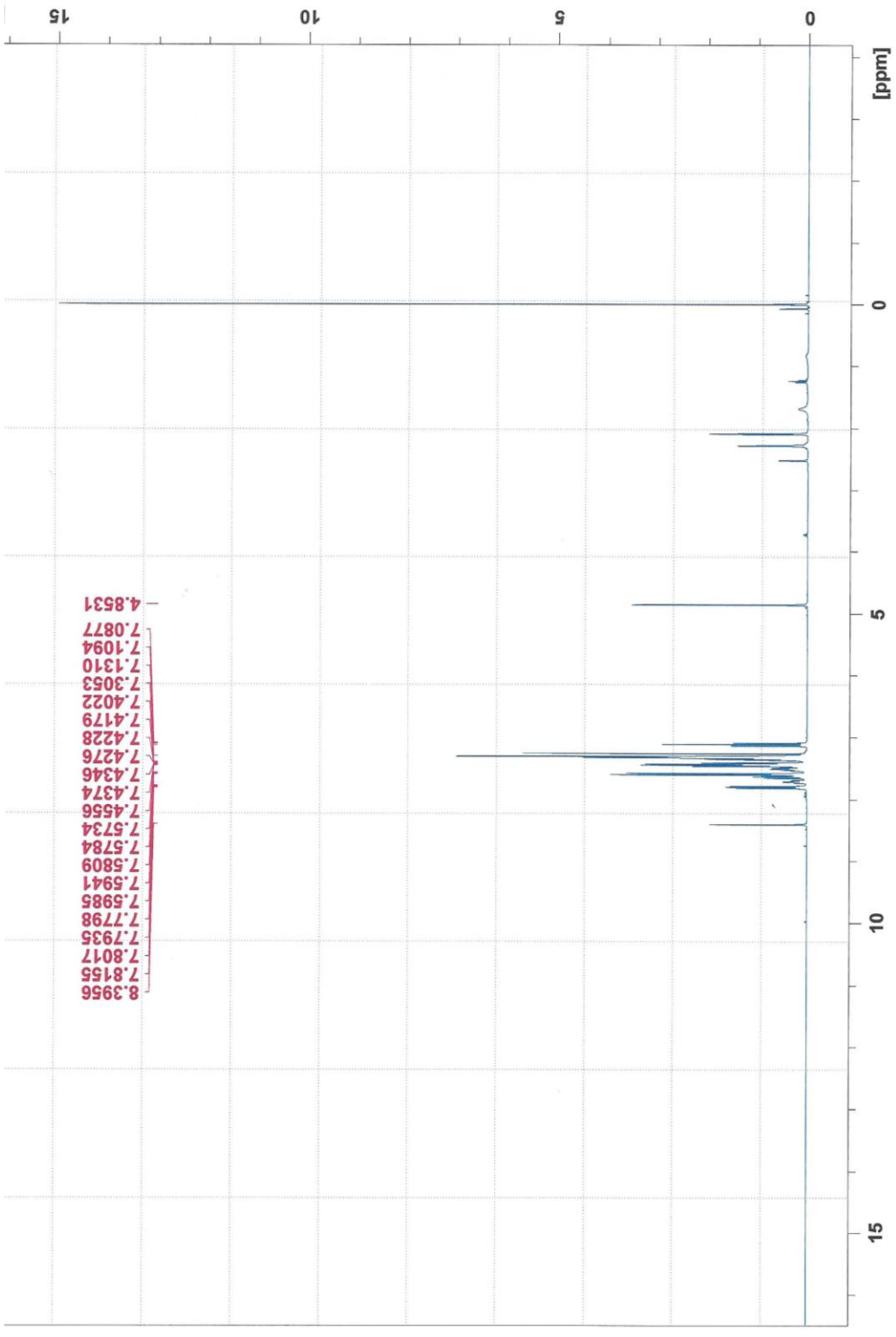

Figure 51: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-fluorophenyl)methanimine **20**

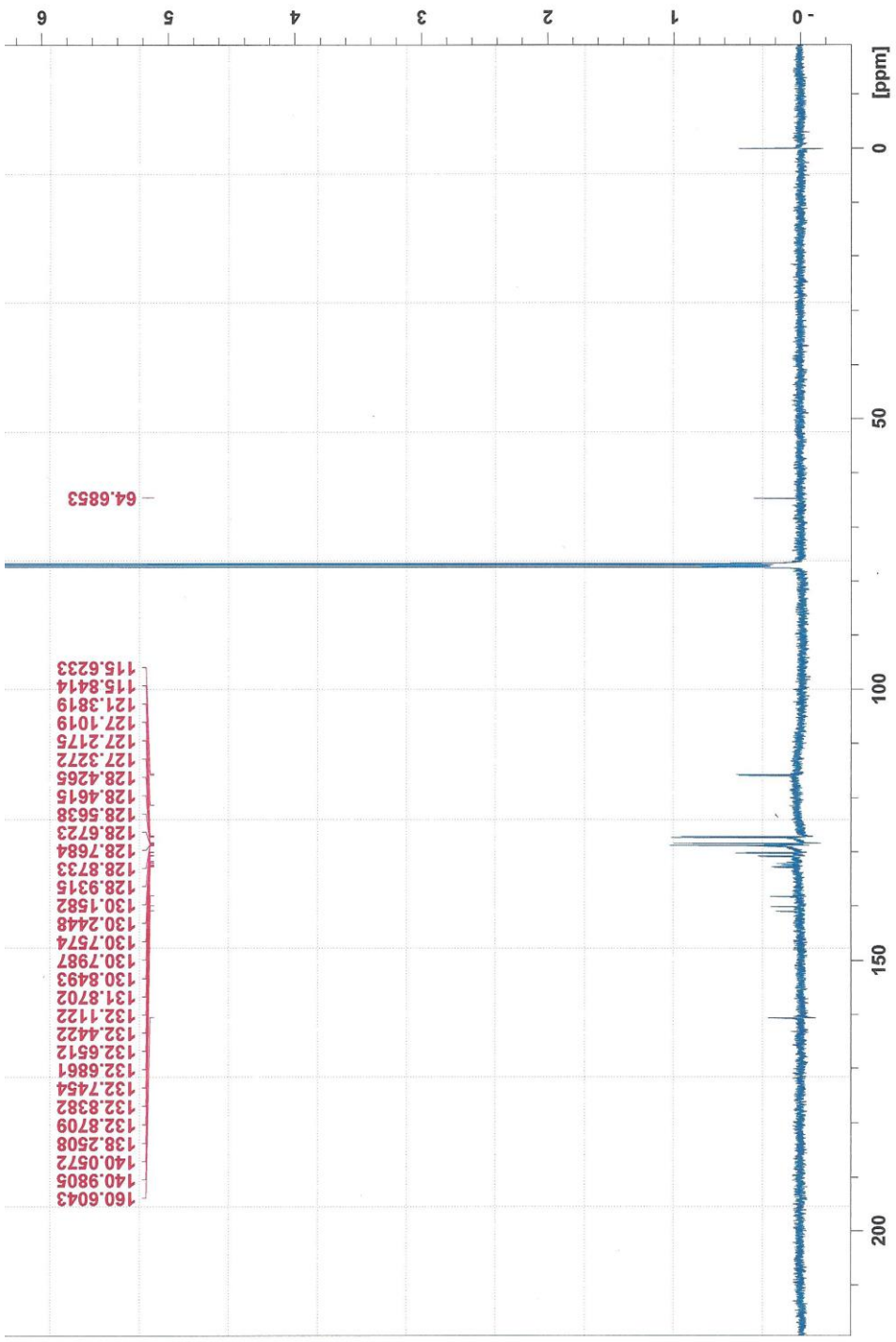

Figure 52: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-fluorophenyl)methanimine **20**

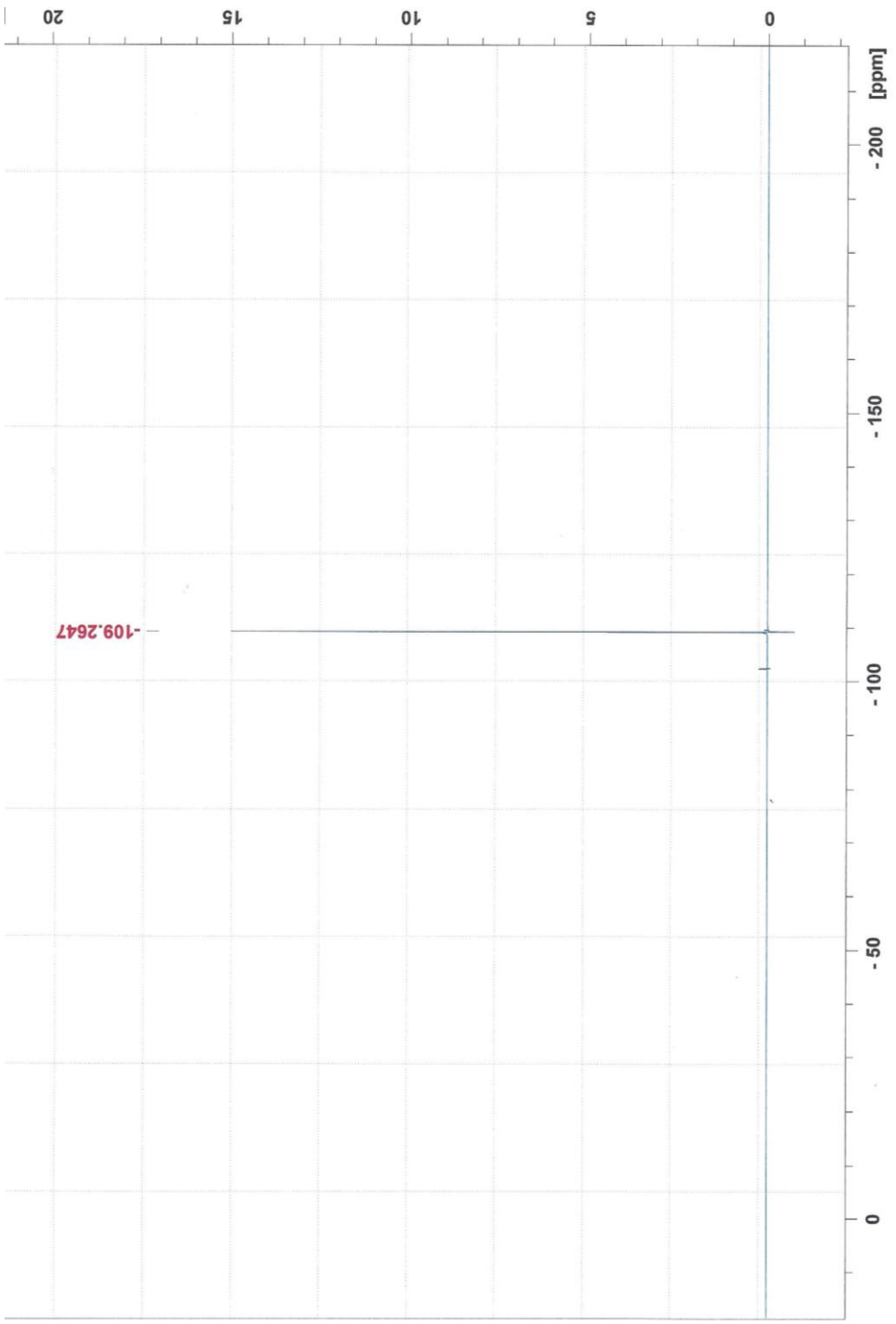

Figure 53: ^{19}F NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-fluorophenyl)methanimine **20**

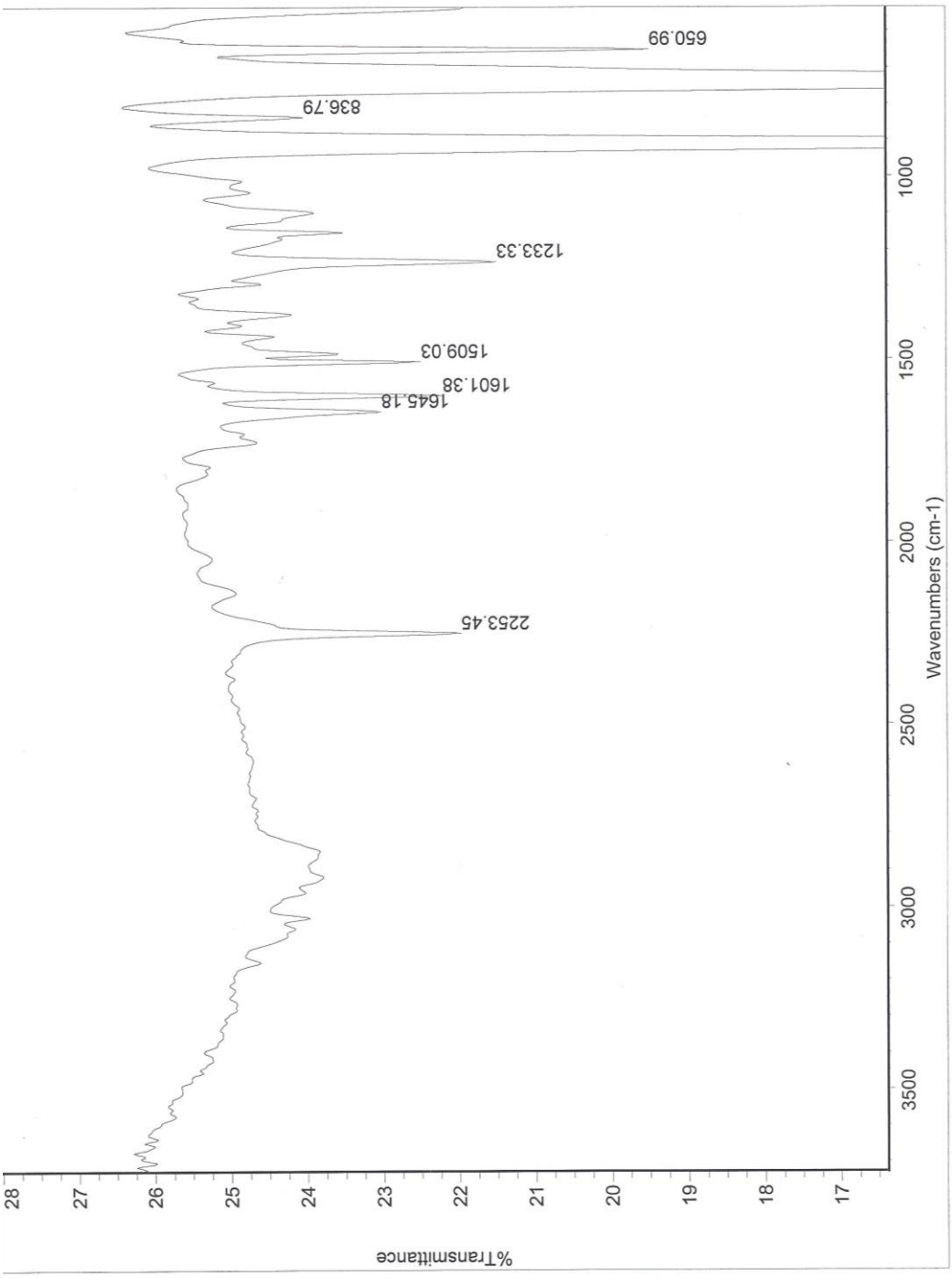

Figure 54: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(4-fluorophenyl)methanimine **20**

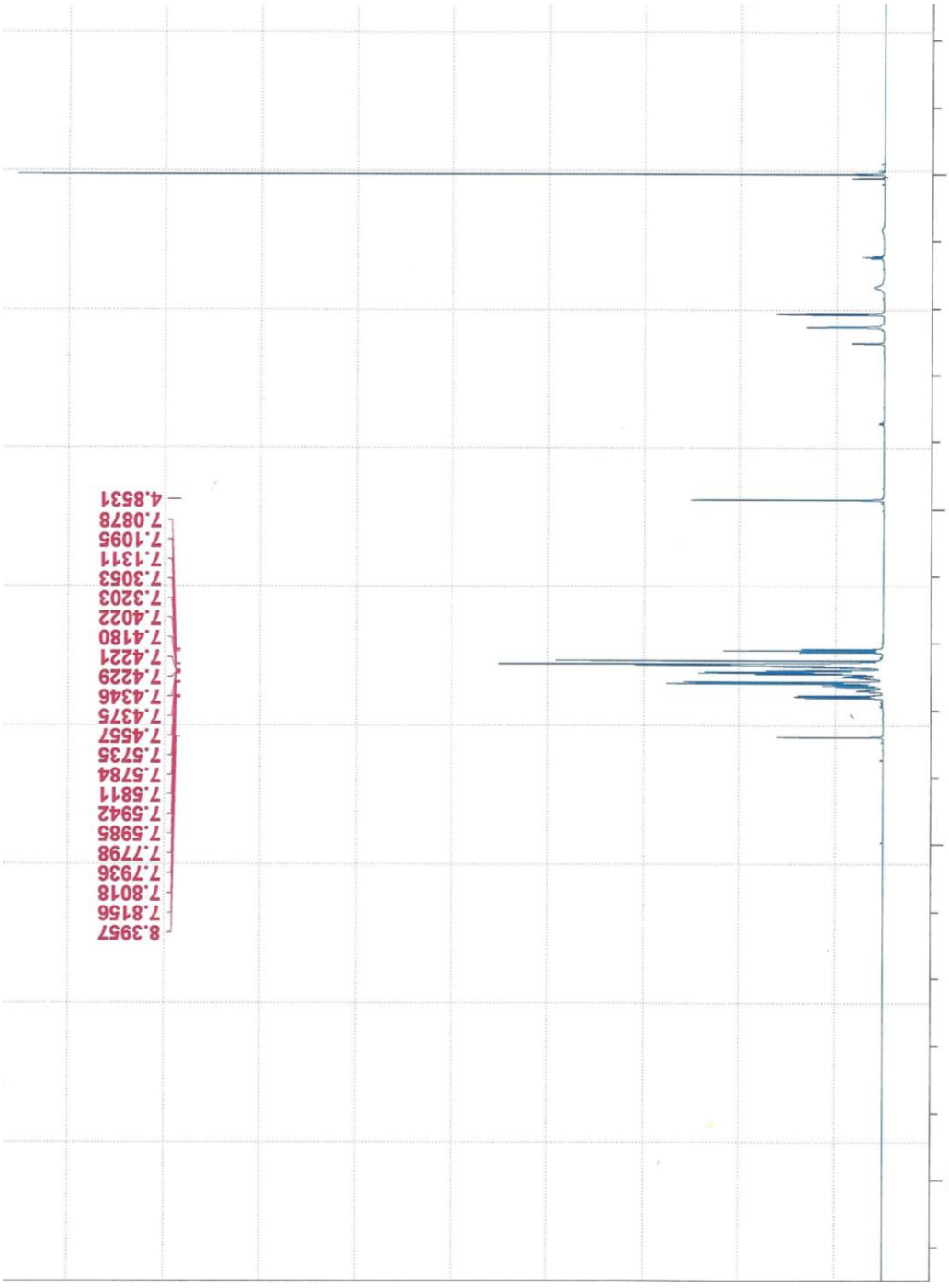

Figure 55: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2-fluorophenyl)methanimine **21**

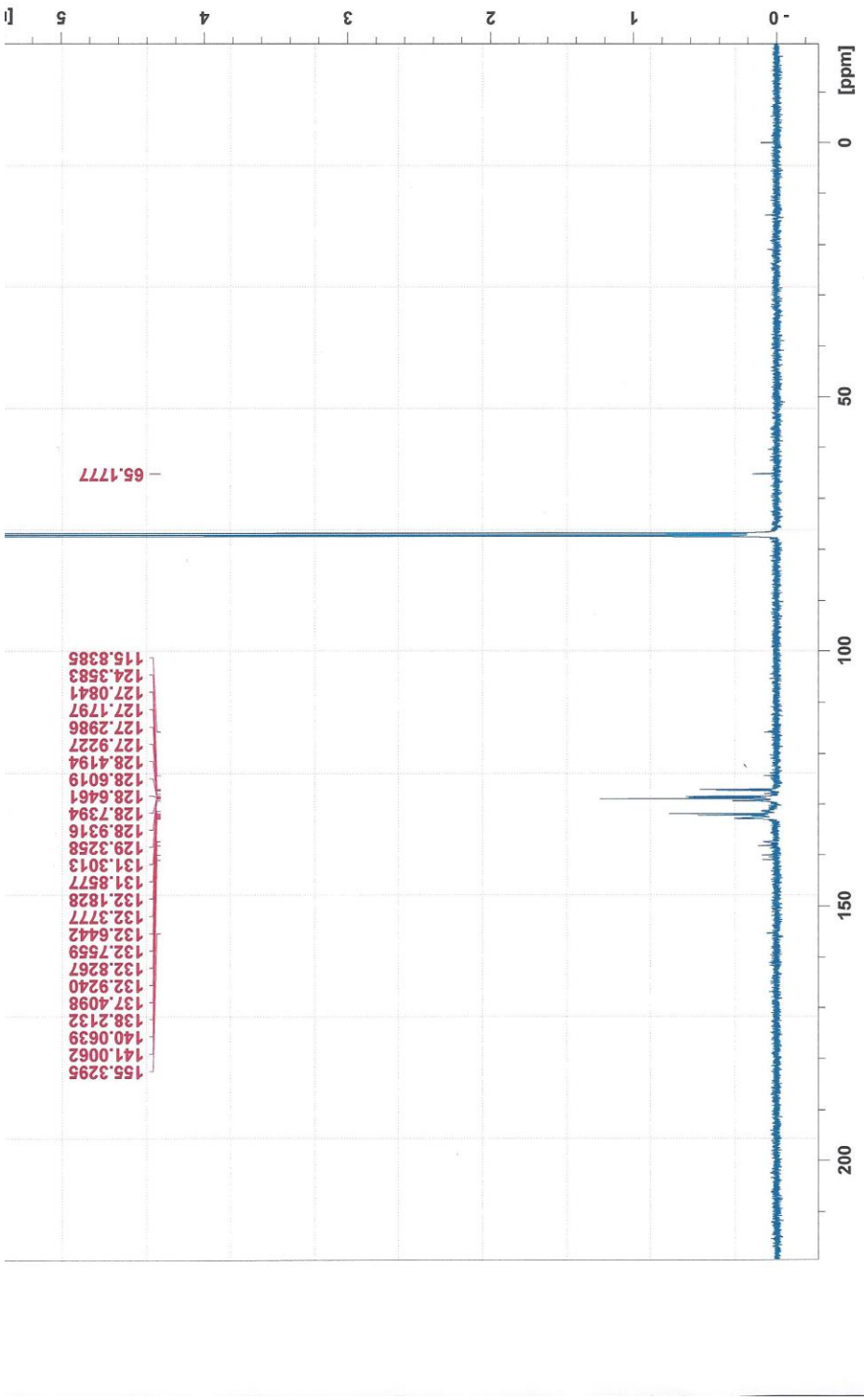

Figure 56: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2-fluorophenyl)methanimine **21**

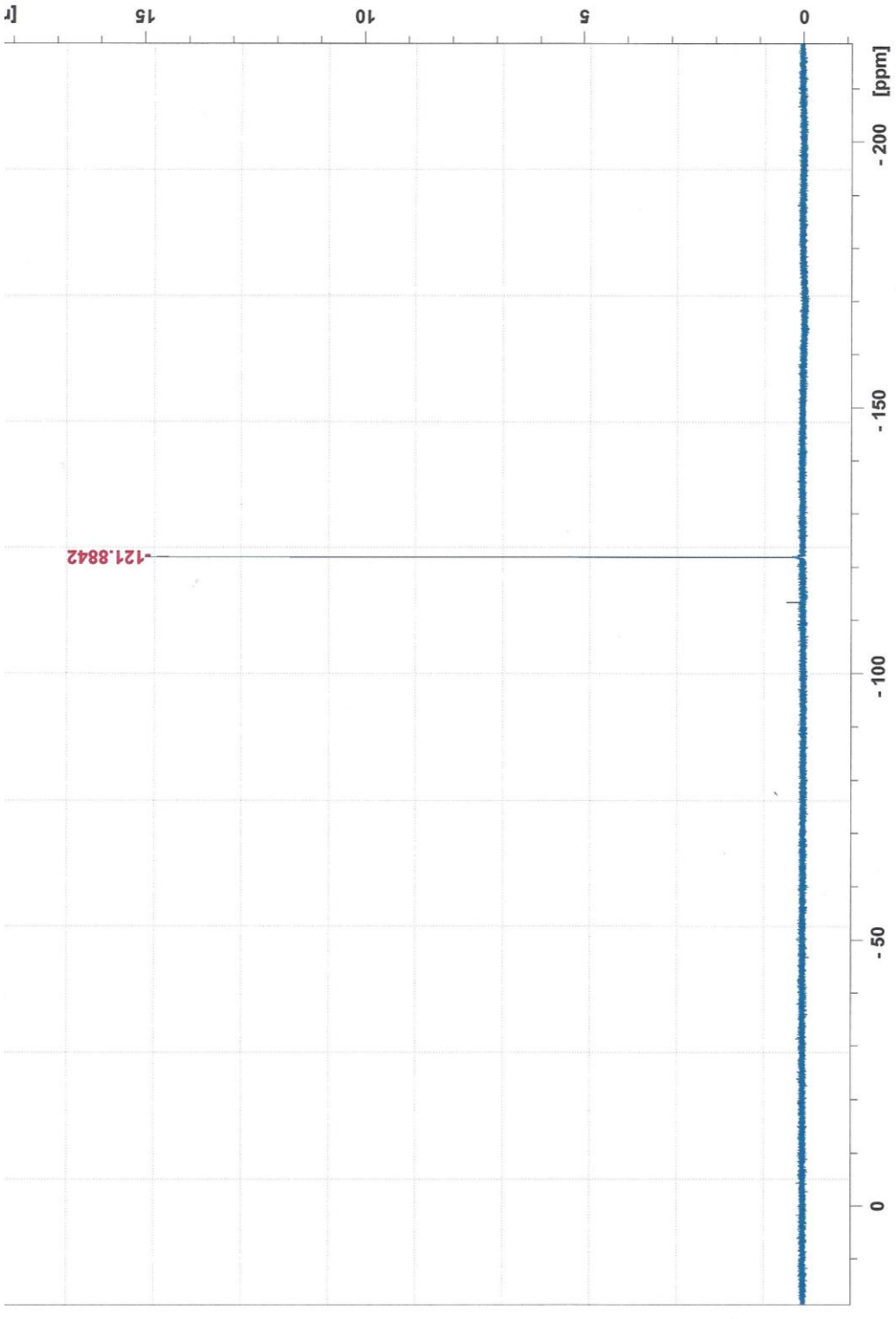

Figure 57: ^{19}F NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2-fluorophenyl)methanimine **21**

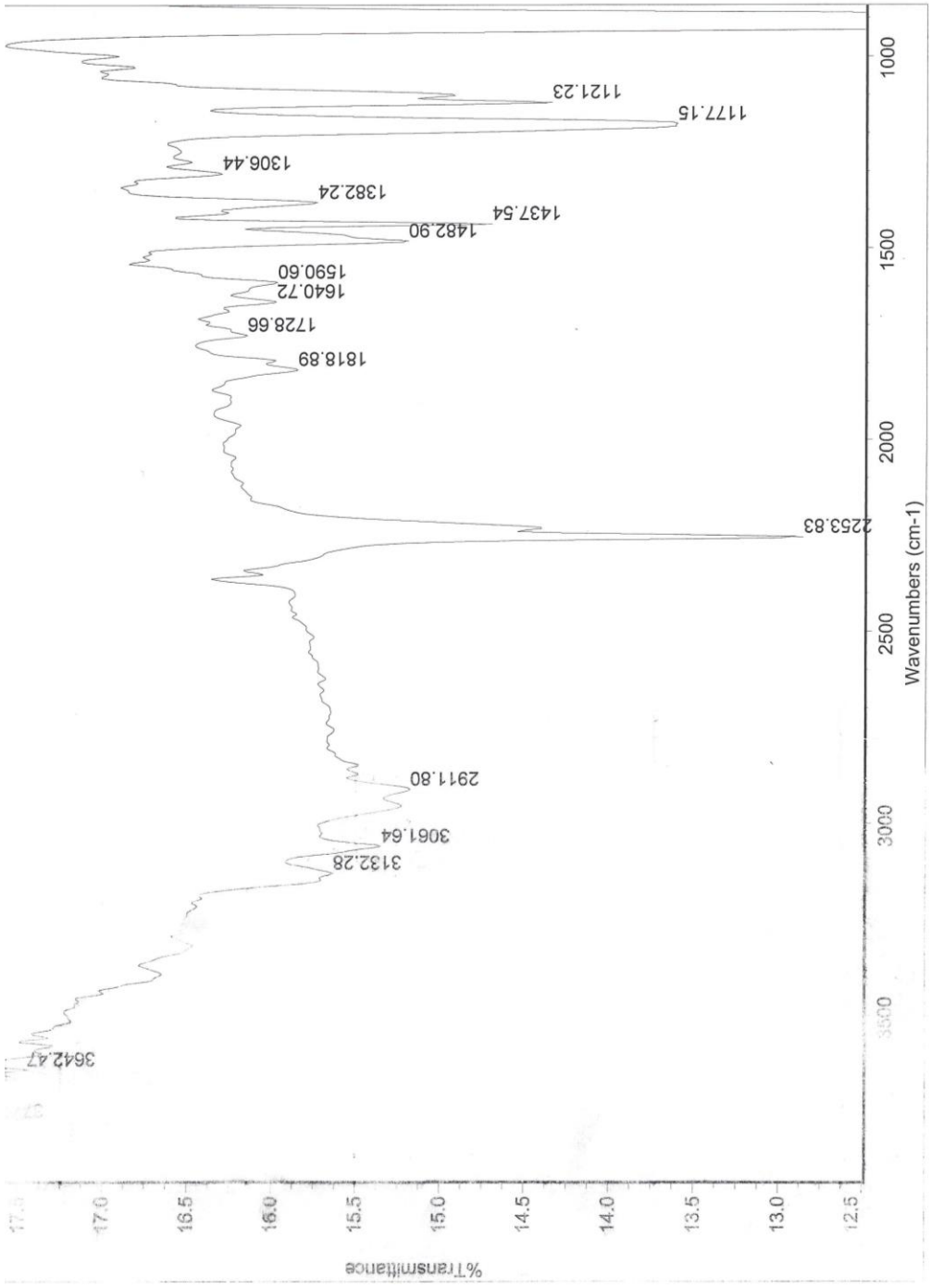

Figure 58: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2-fluorophenyl)methanimine **21**

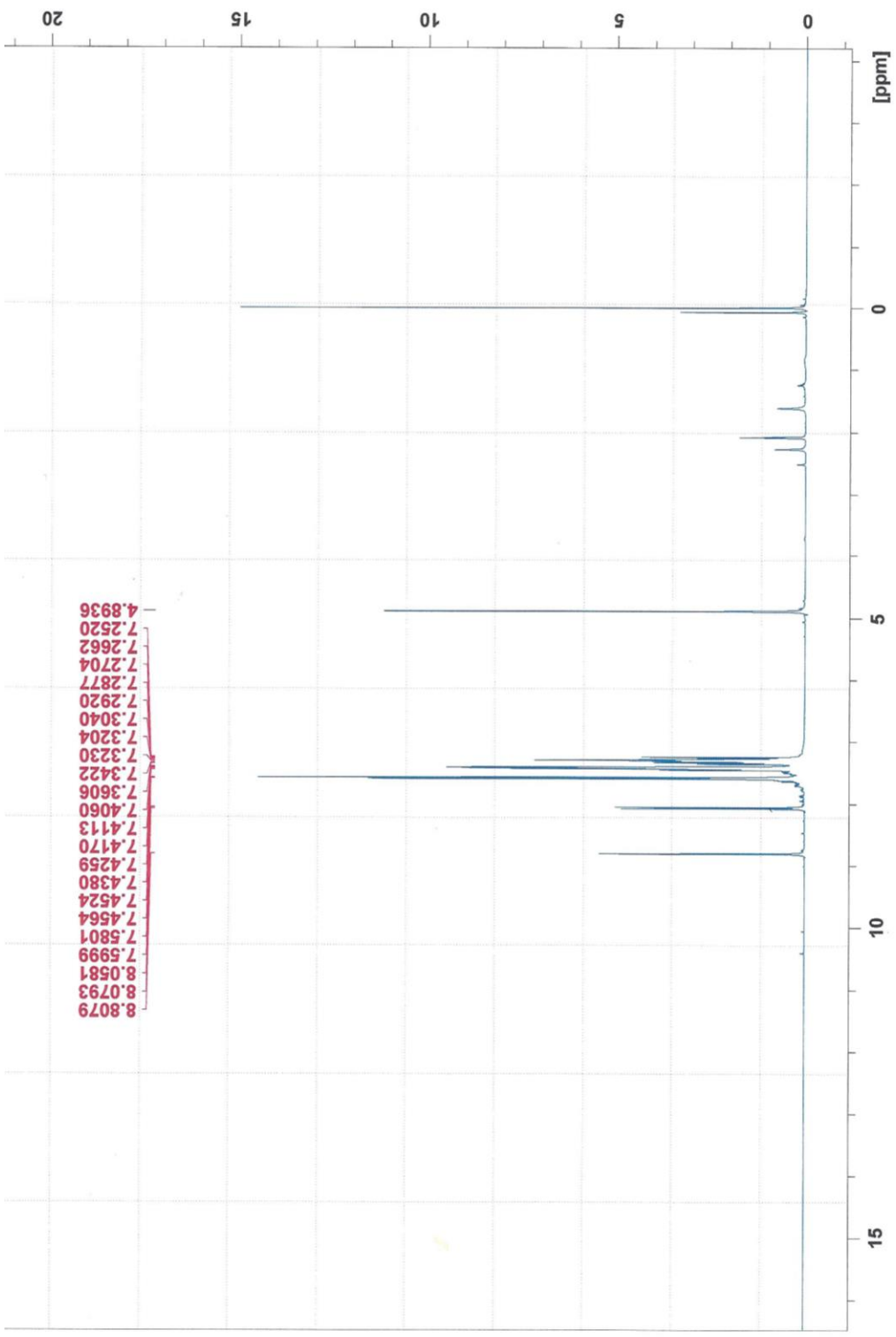

Figure 59: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4-dichlorophenyl)methanimine **22**

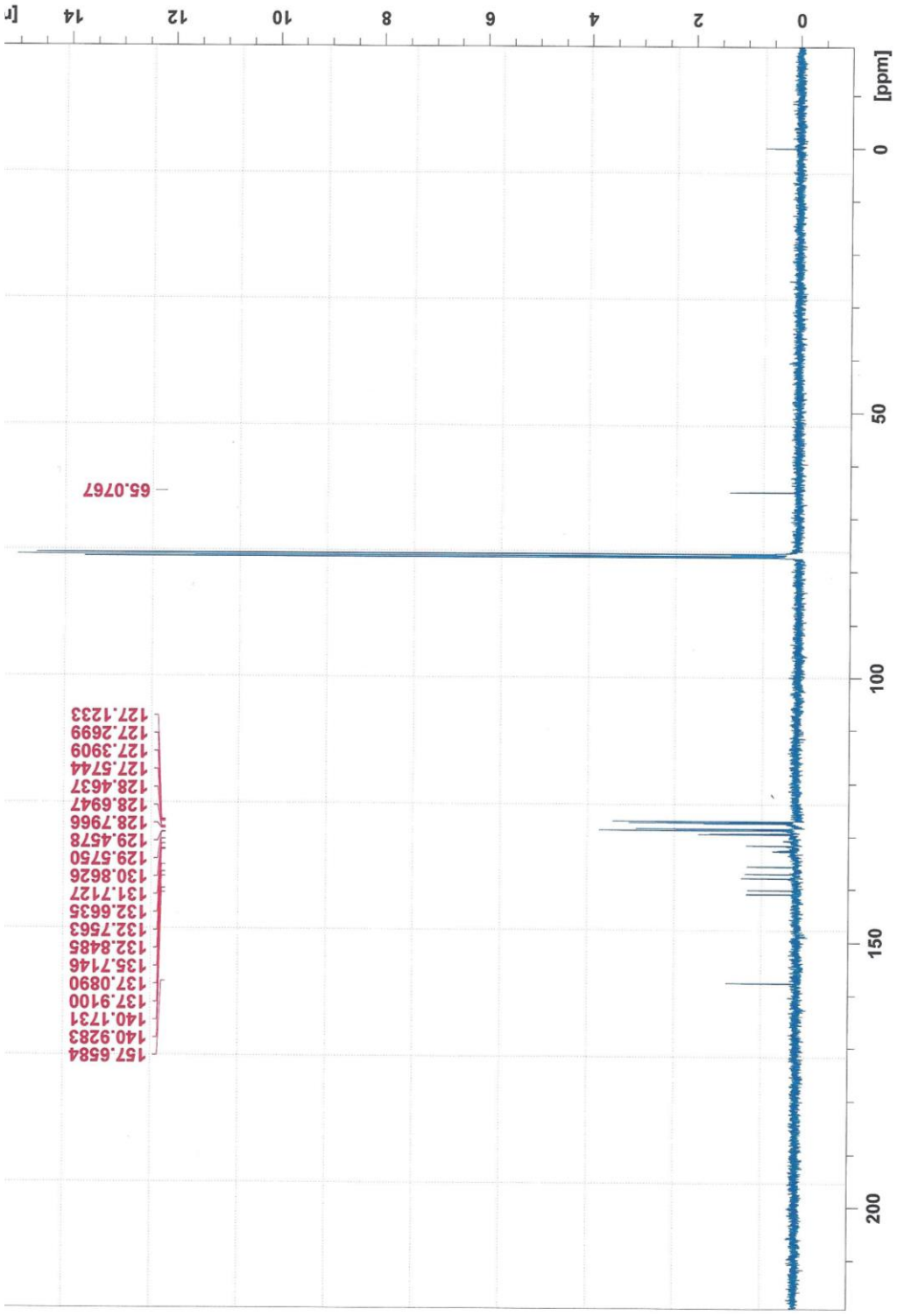

Figure 60: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4-dichlorophenyl)methanimine **22**

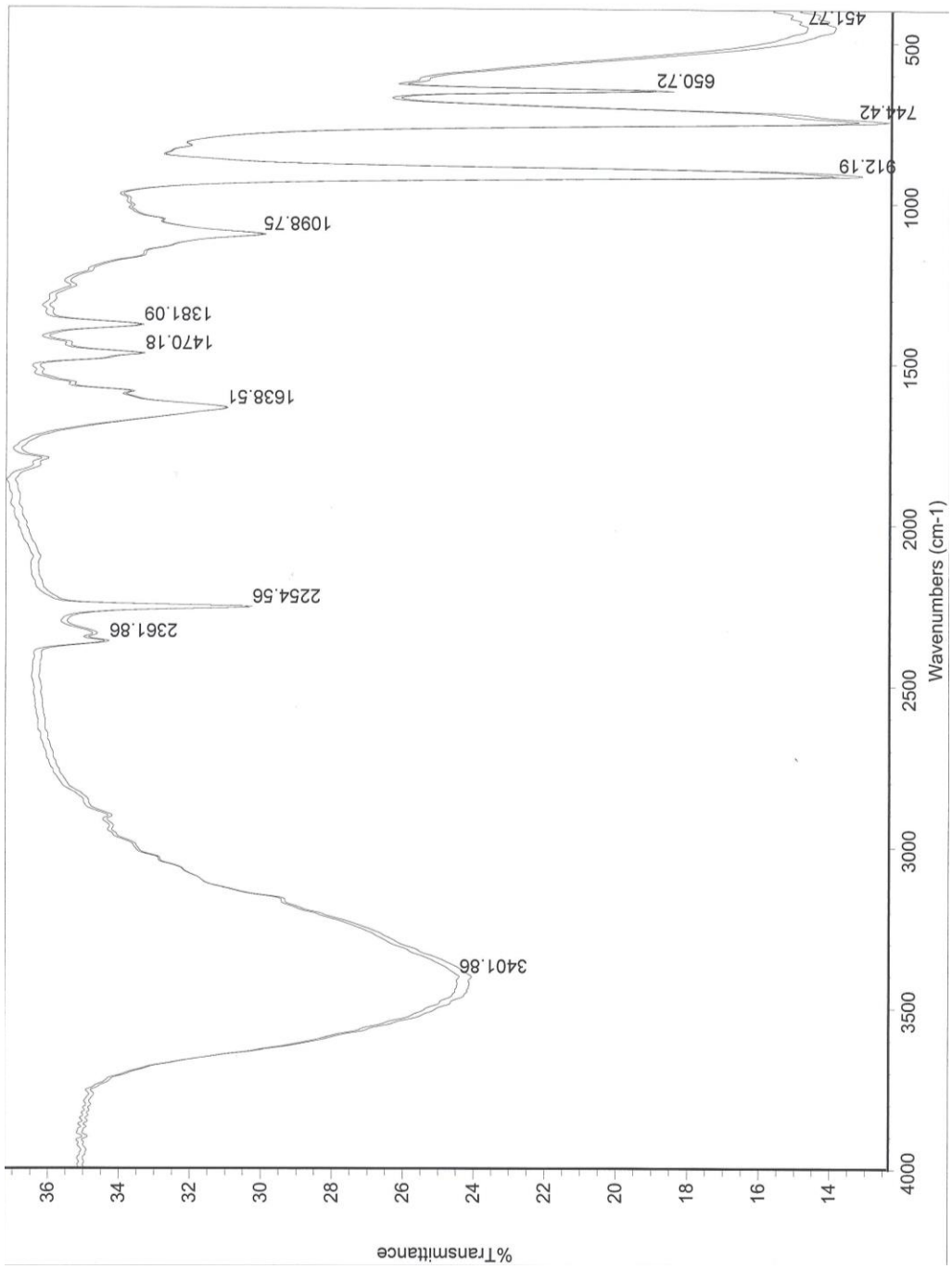

Figure 61: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4-dichlorophenyl)methanimine **22**

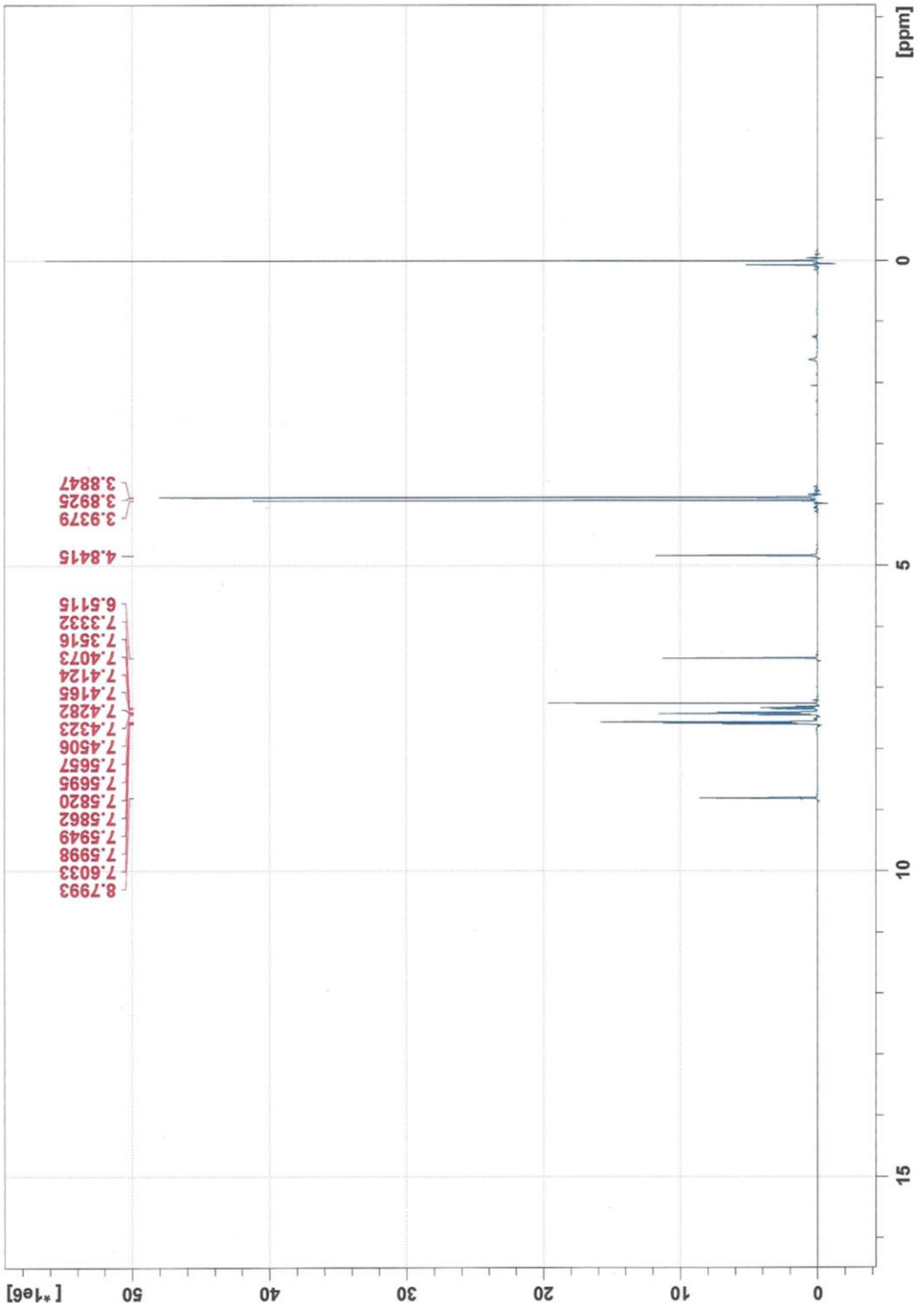

Figure 62: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4,5-trimethoxyphenyl)methanimine **23**

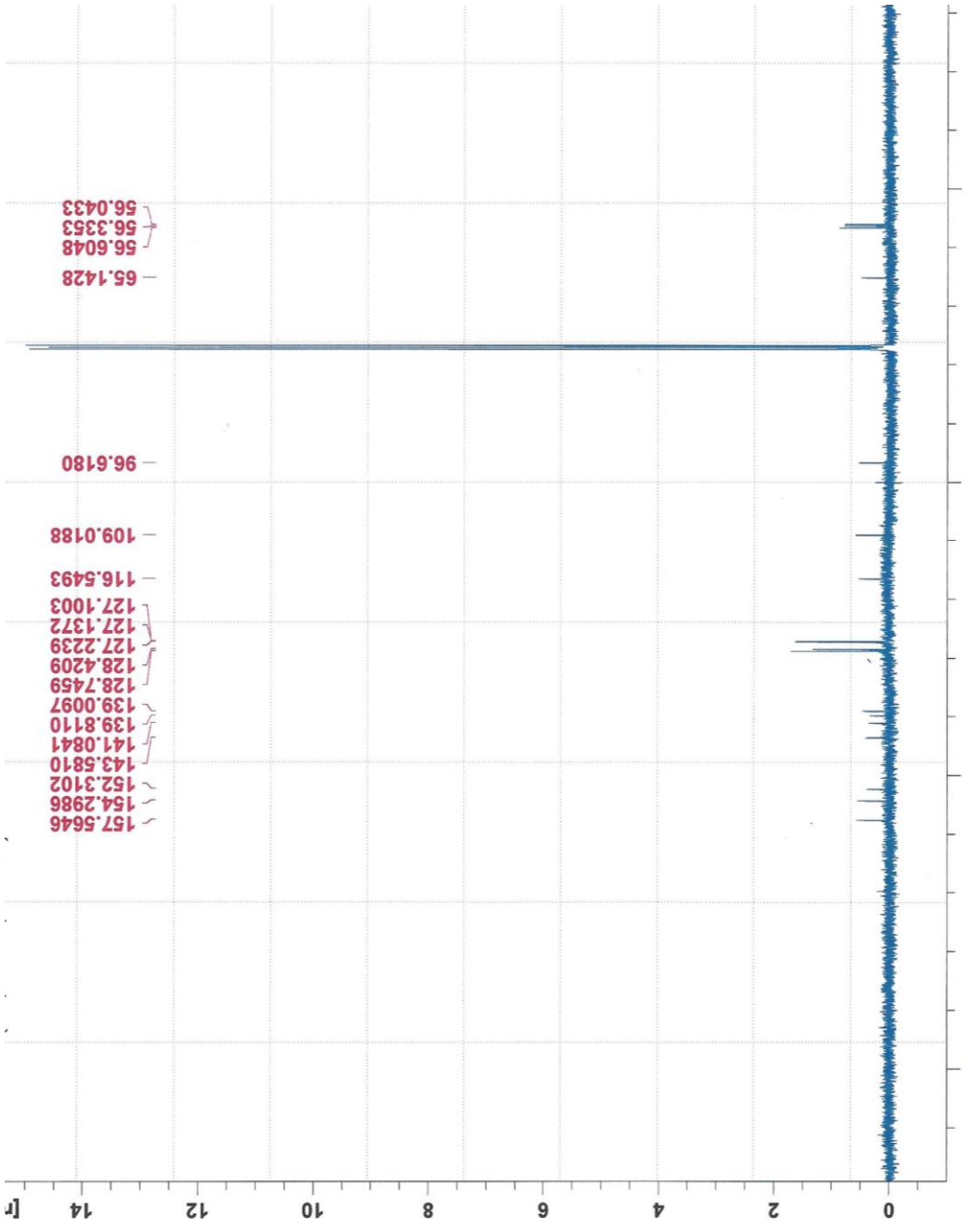

Figure 63: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4,5-trimethoxyphenyl)methanimine **23**

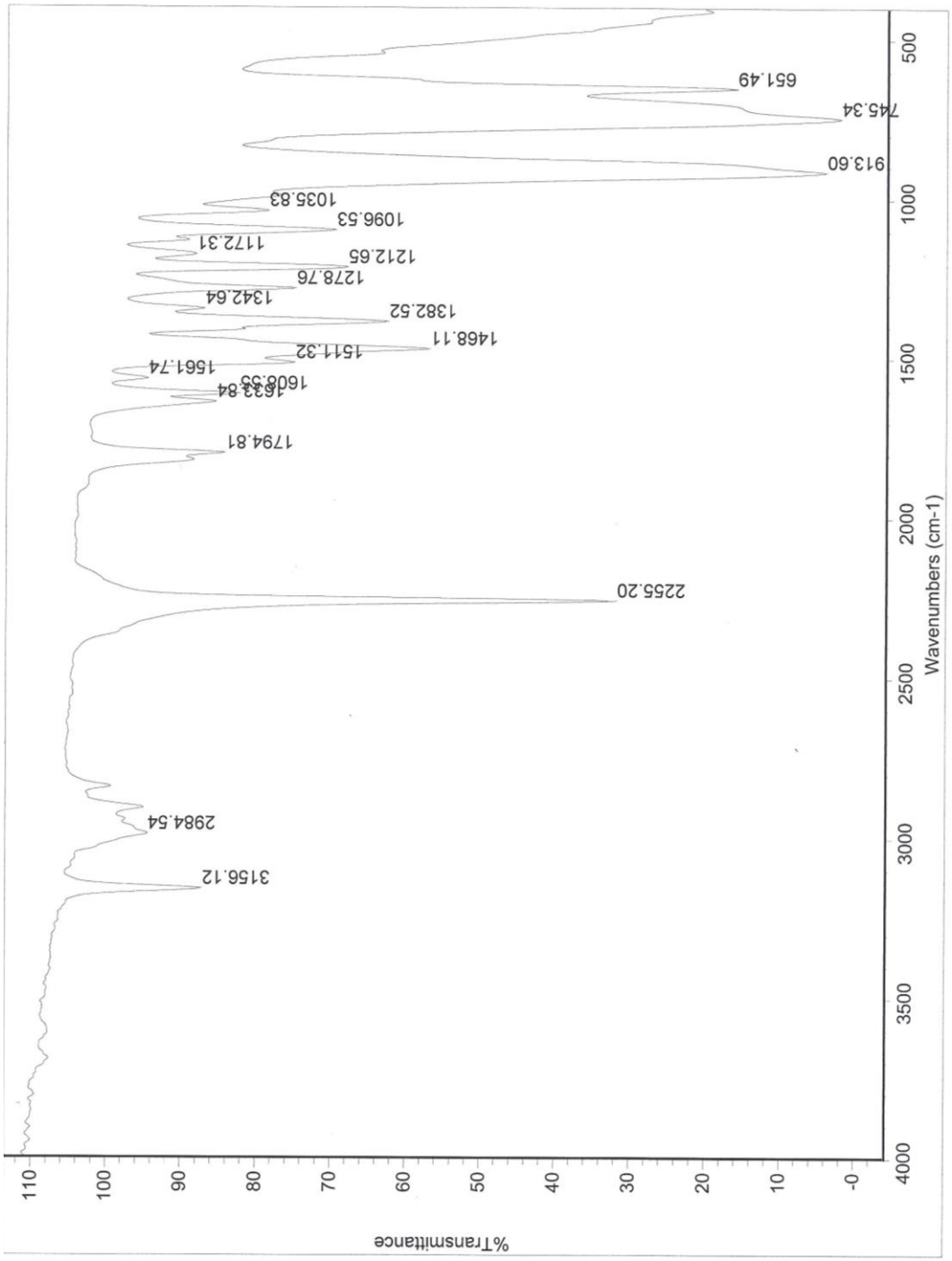

Figure 64: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(2,4,5-trimethoxyphenyl)methanimine **23**

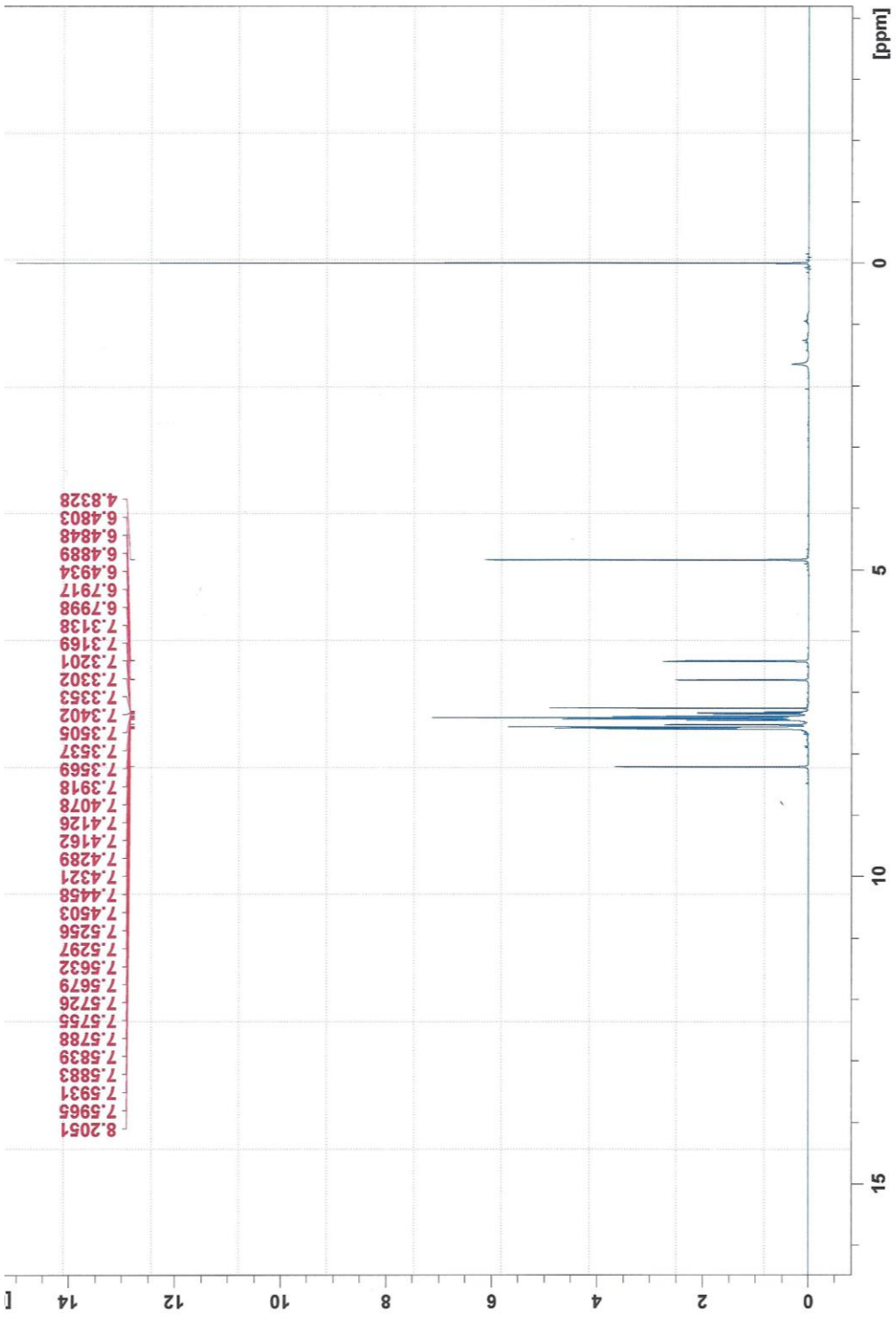

Figure 65: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(furan-2-yl)methanimine **24**

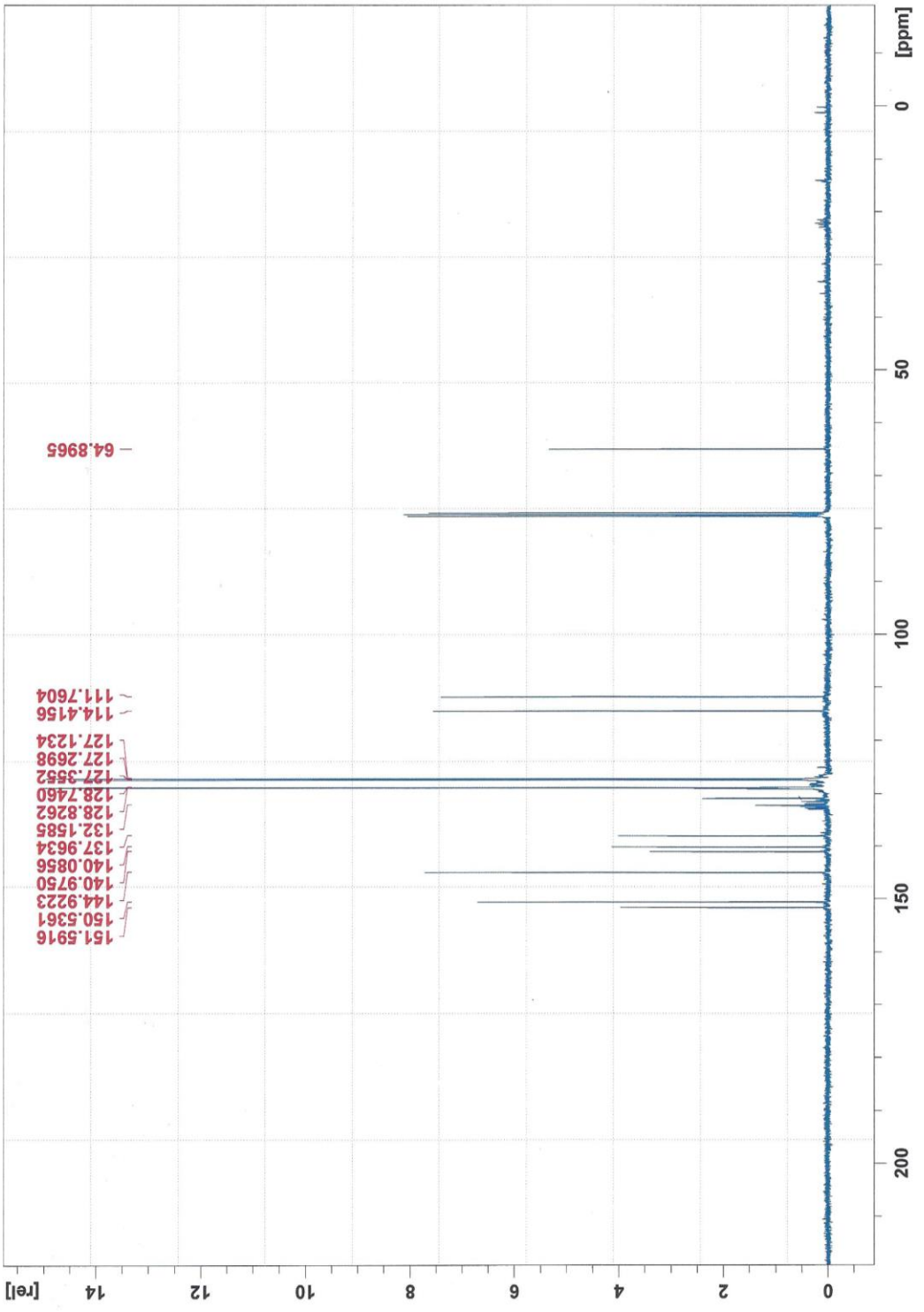

Figure 66: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(furan-2-yl)methanimine **24**

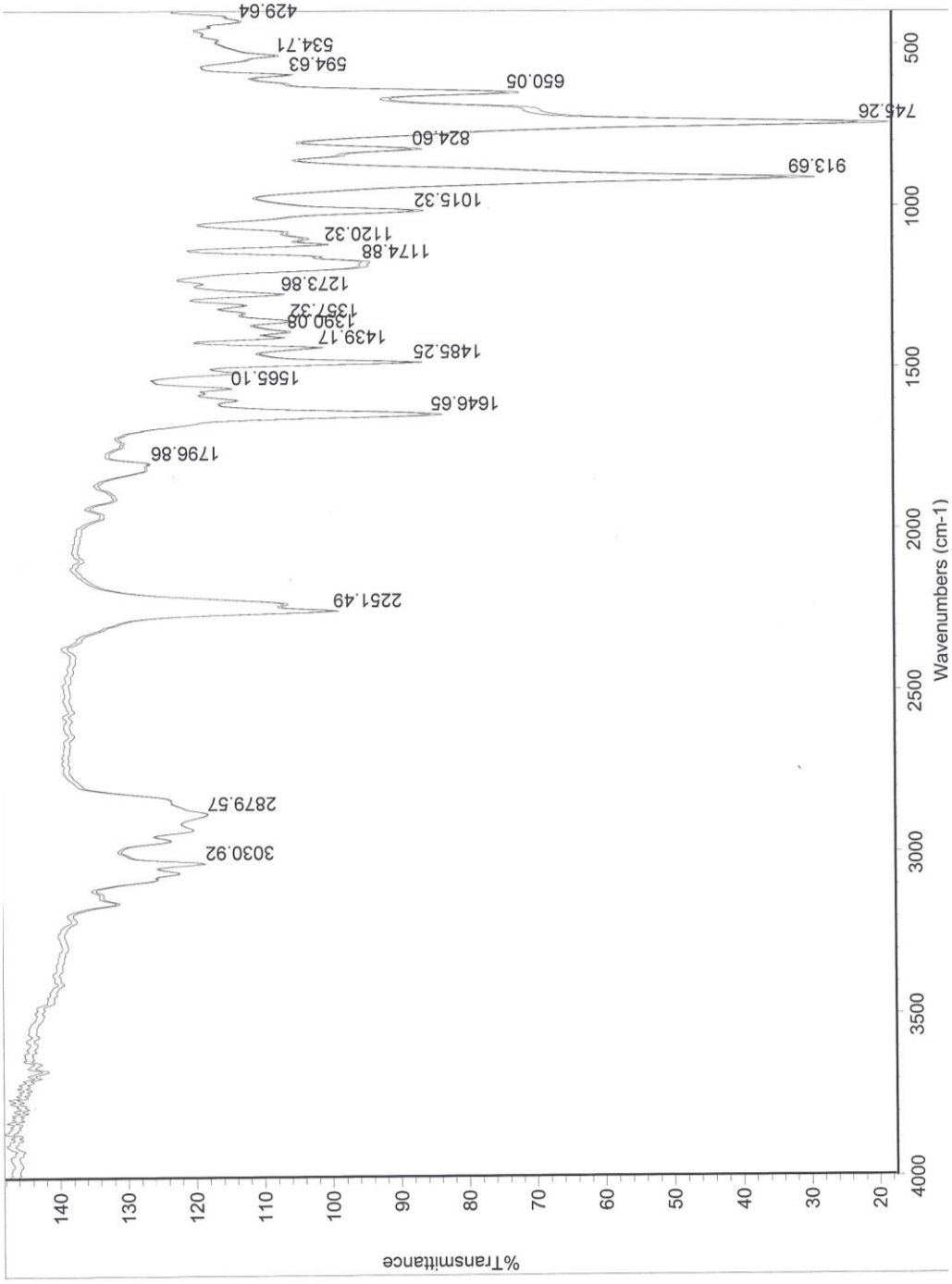

Figure 67: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(furan-2-yl)methanimine **24**

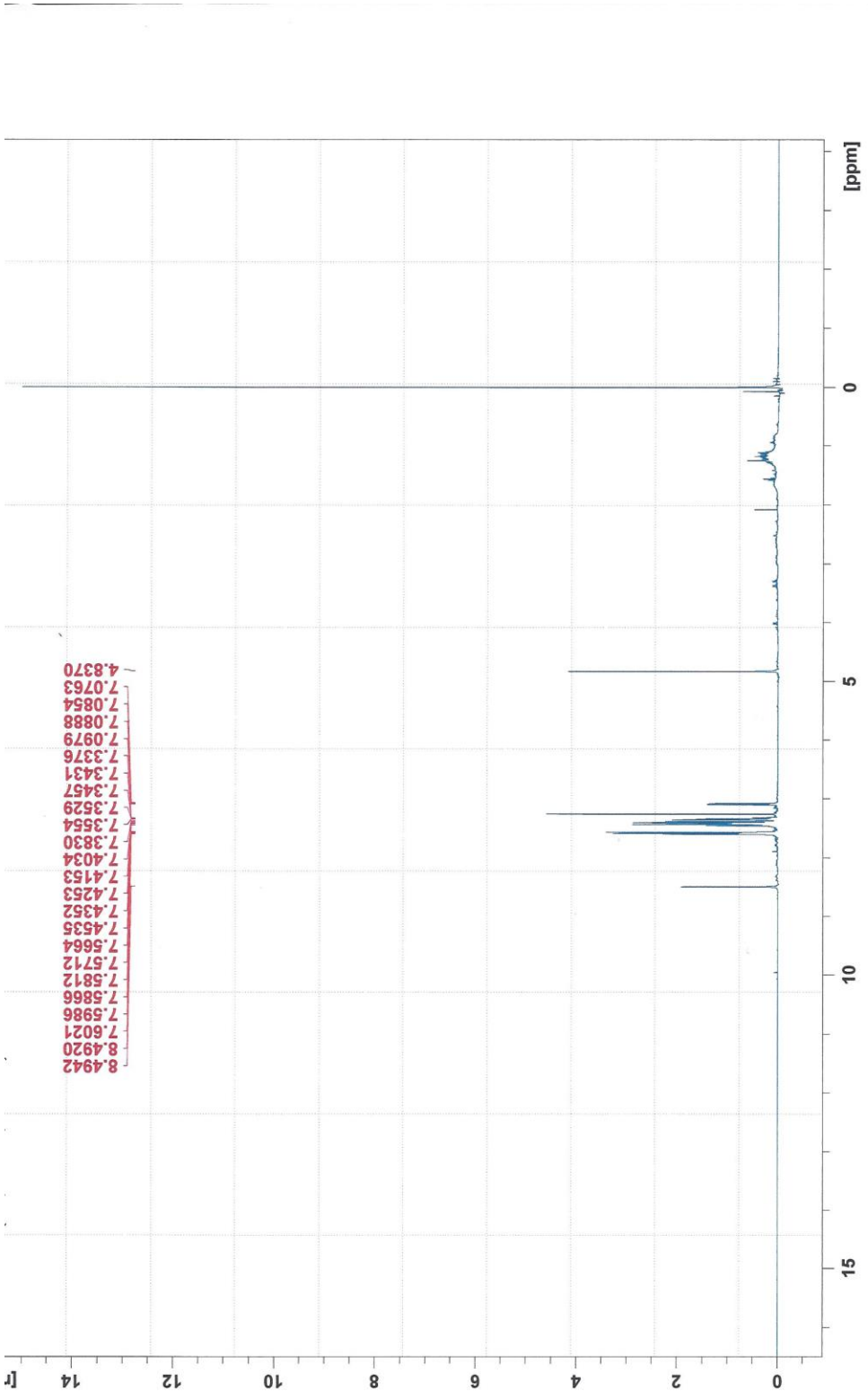

Figure 68: ^{1}H NMR Spectrum of(*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(thiophen-2-yl)methanimine **25**

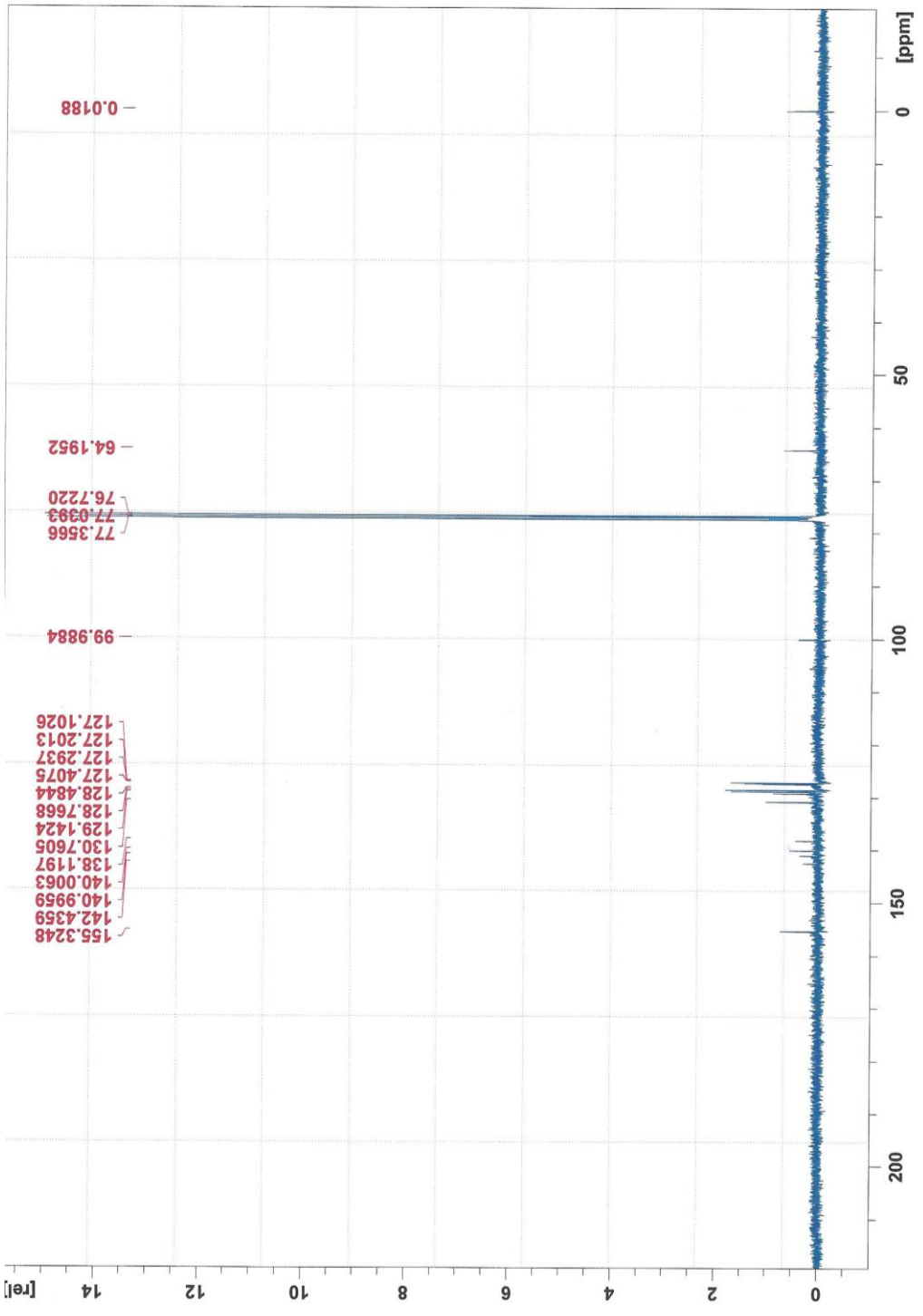

Figure 69: ^{13}C NMR Spectrum of(*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(thiophen-2-yl)methanimine **25**

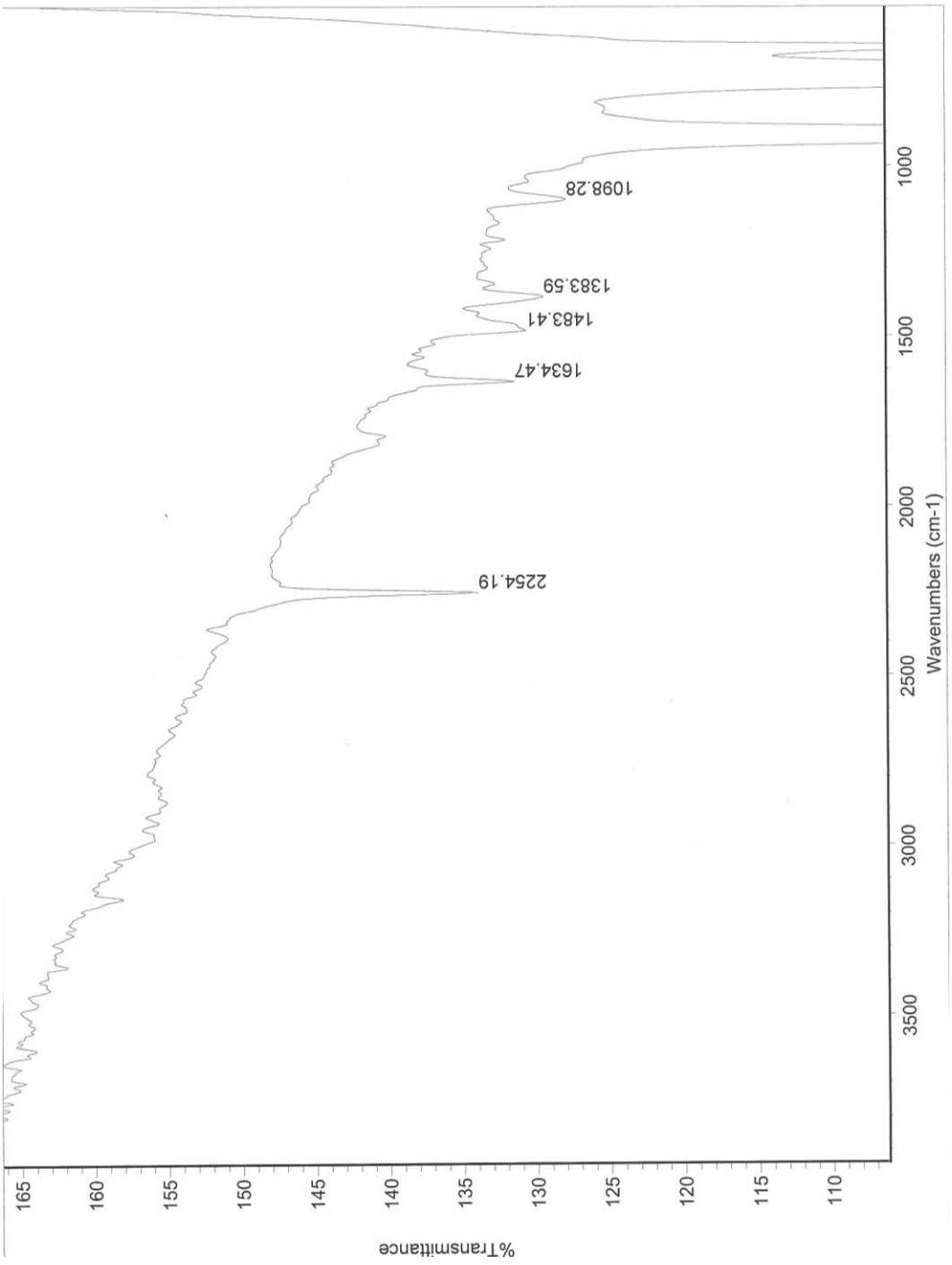

Figure 70: IR Spectrum of(*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(thiophen-2-yl)methanimine **25**

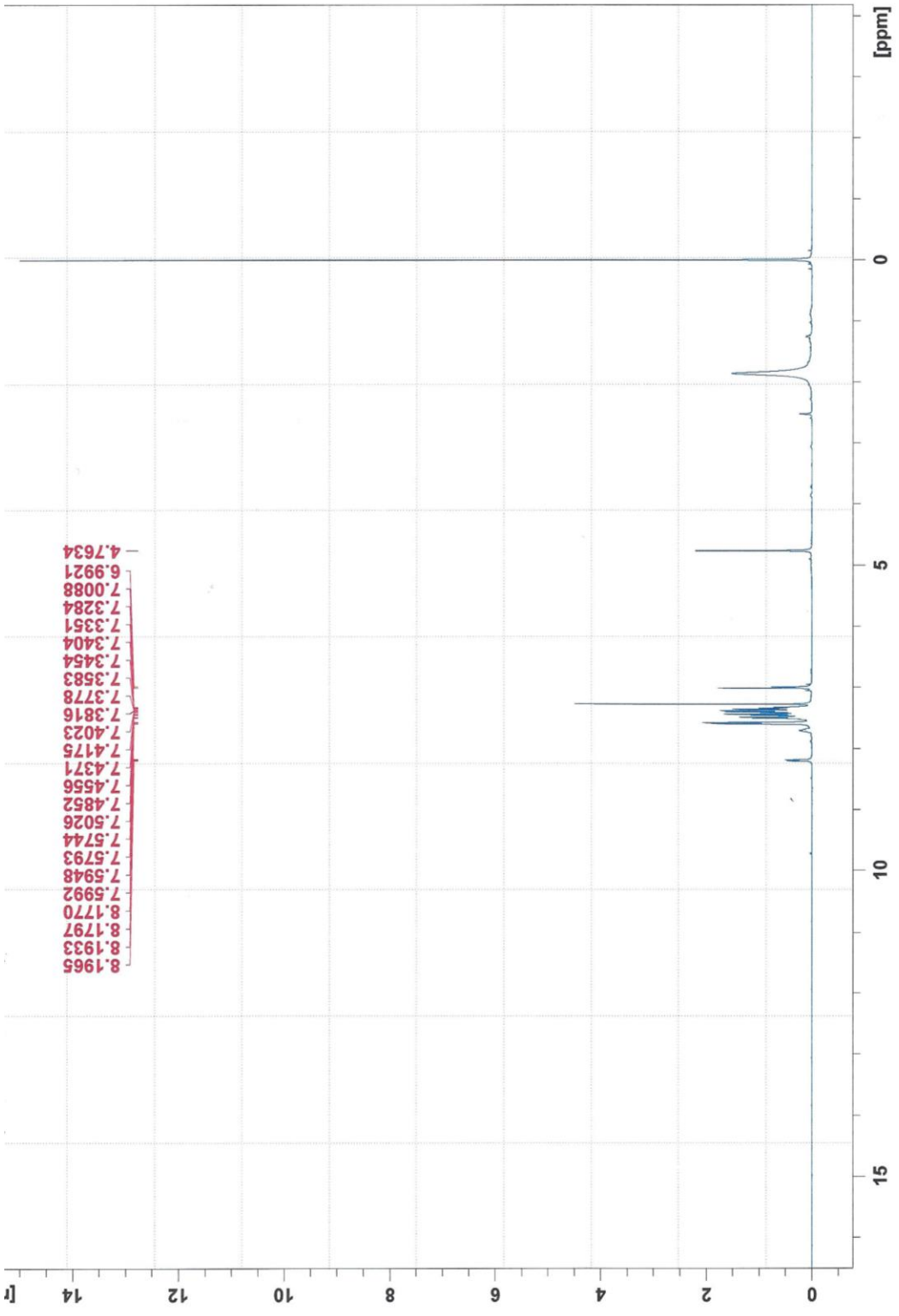

Figure 71: ^{1}H NMR Spectrum of(1*E*,2*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-3-phenylprop-2-en-1-imine **26**

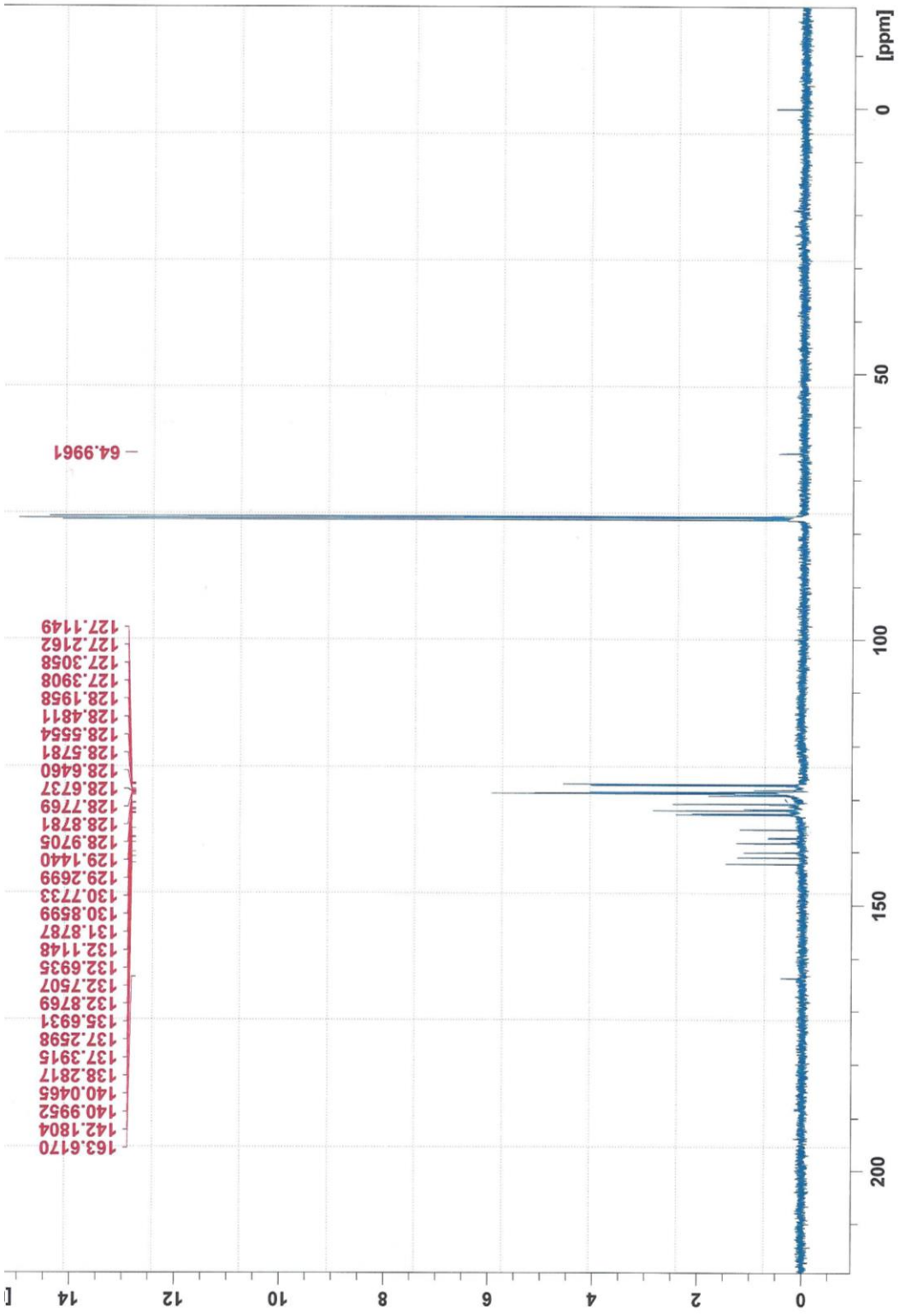

Figure 72: ^{13}C NMR Spectrum of(1*E*,2*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-3-phenylprop-2-en-1-imine **26**

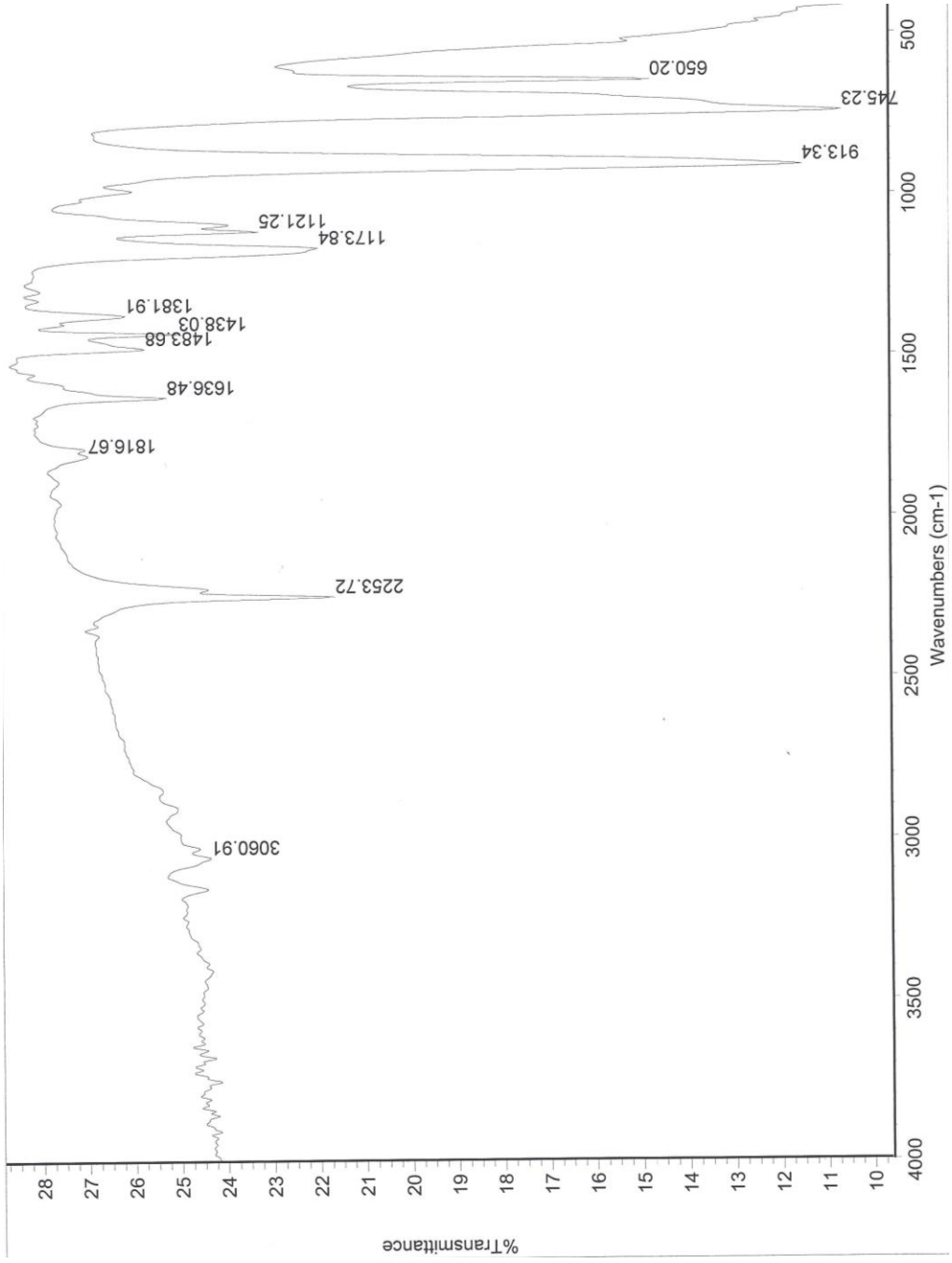

Figure 73: IR Spectrum of(1*E*,2*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-3-phenylprop-2-en-1-imine **26**

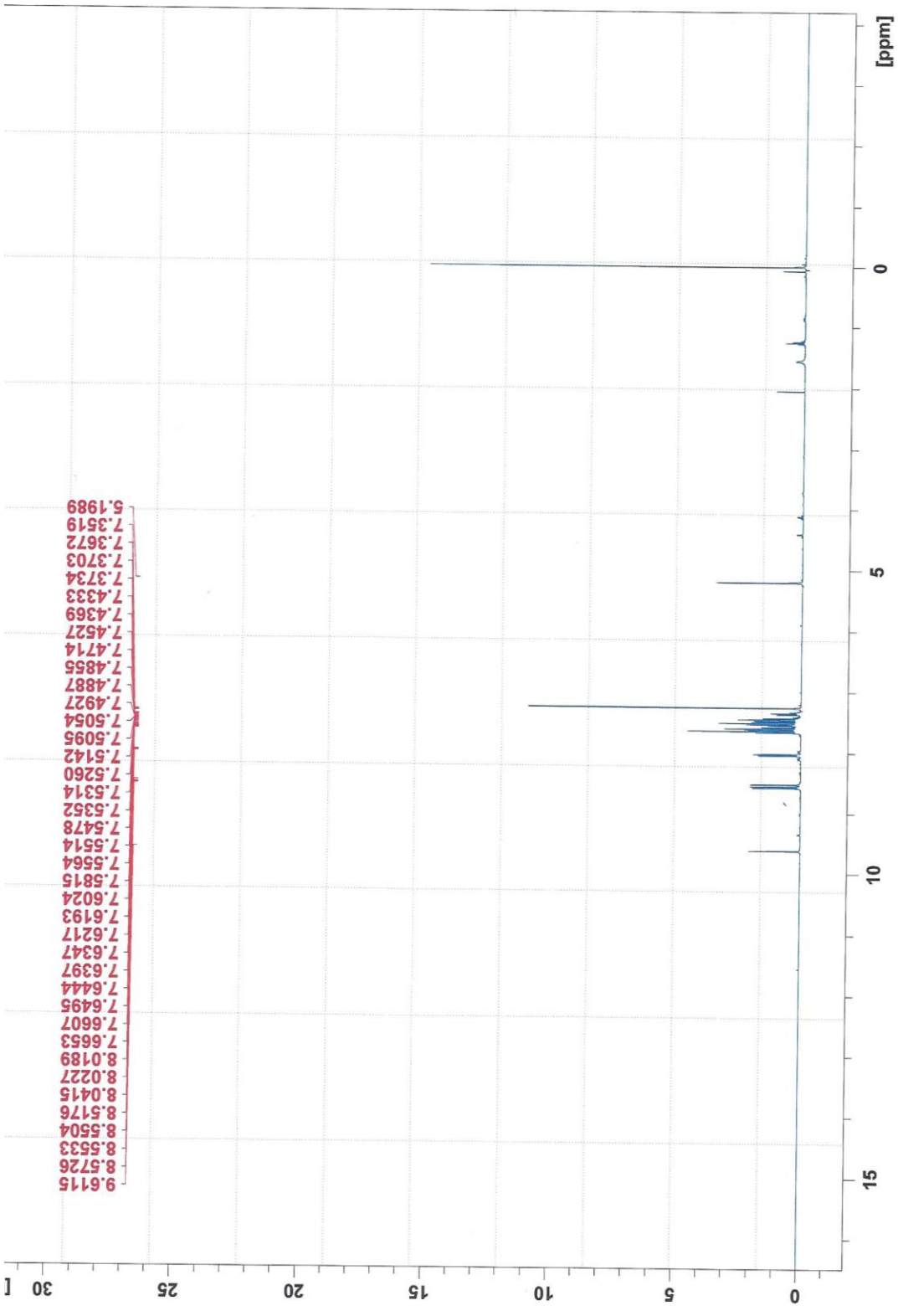

Figure 74: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(anthracen-9-yl)methanimine **27**

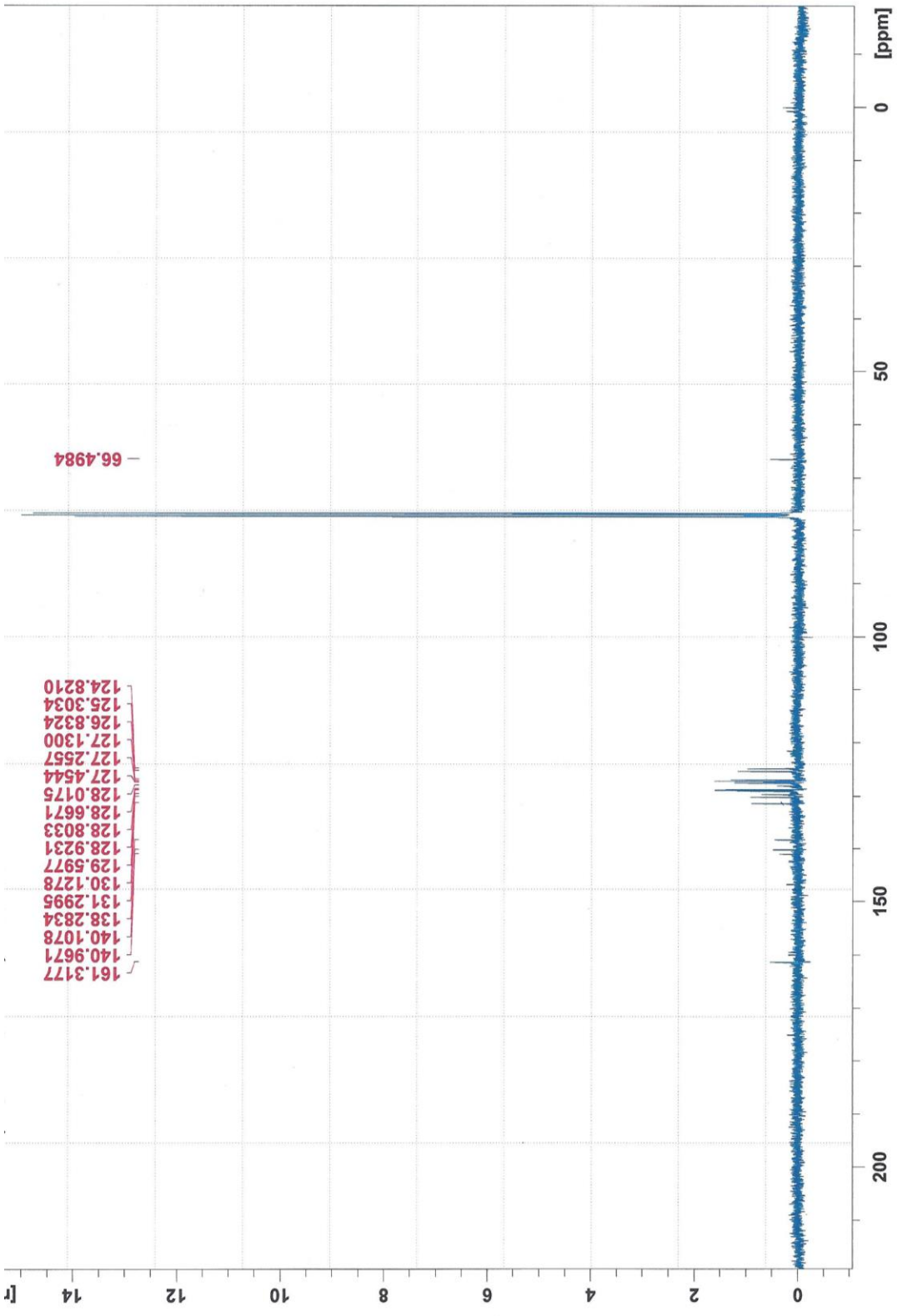

Figure 75: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(anthracen-9-yl)methanimine **27**

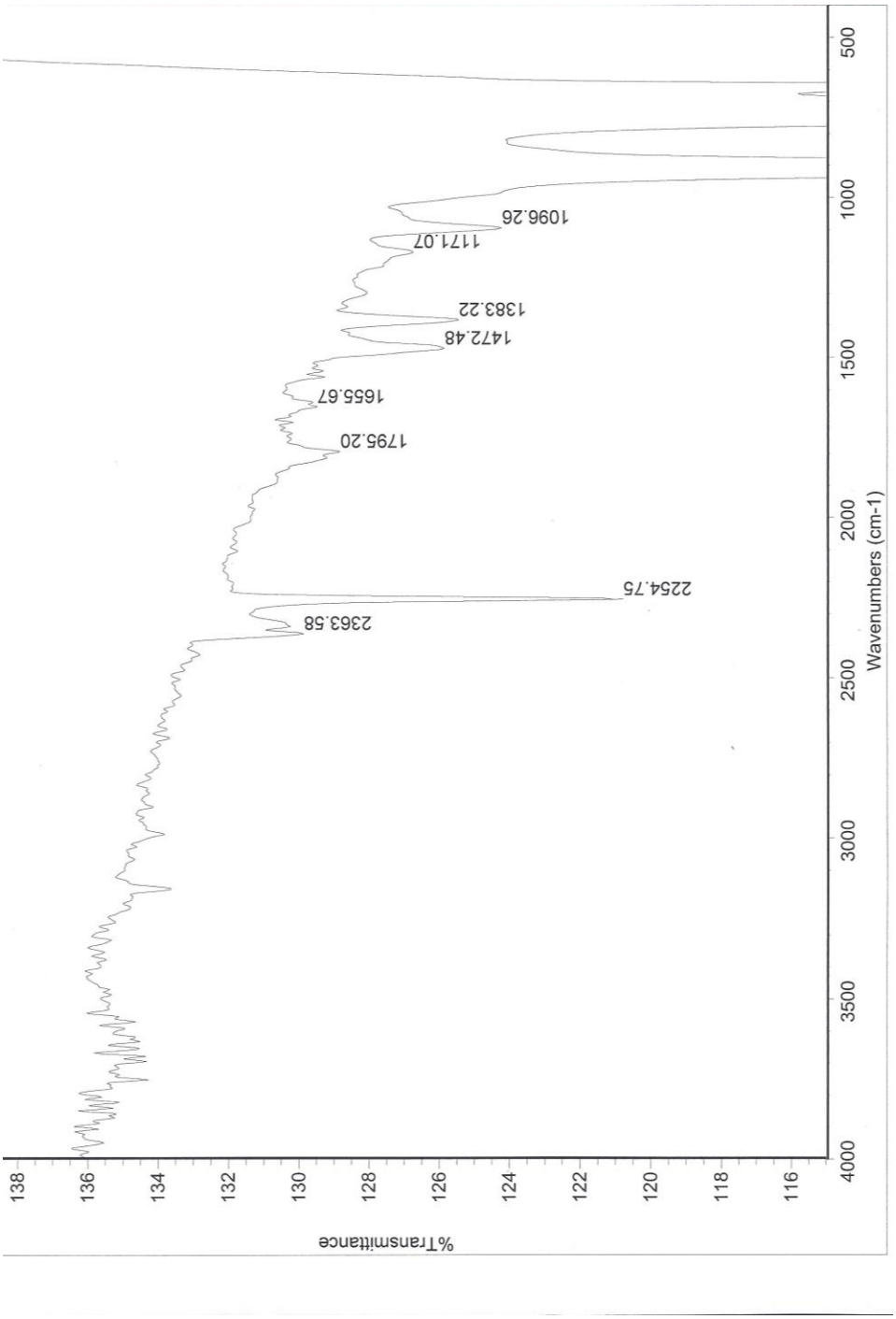

Figure 76: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(anthracen-9-yl)methanimine **27**

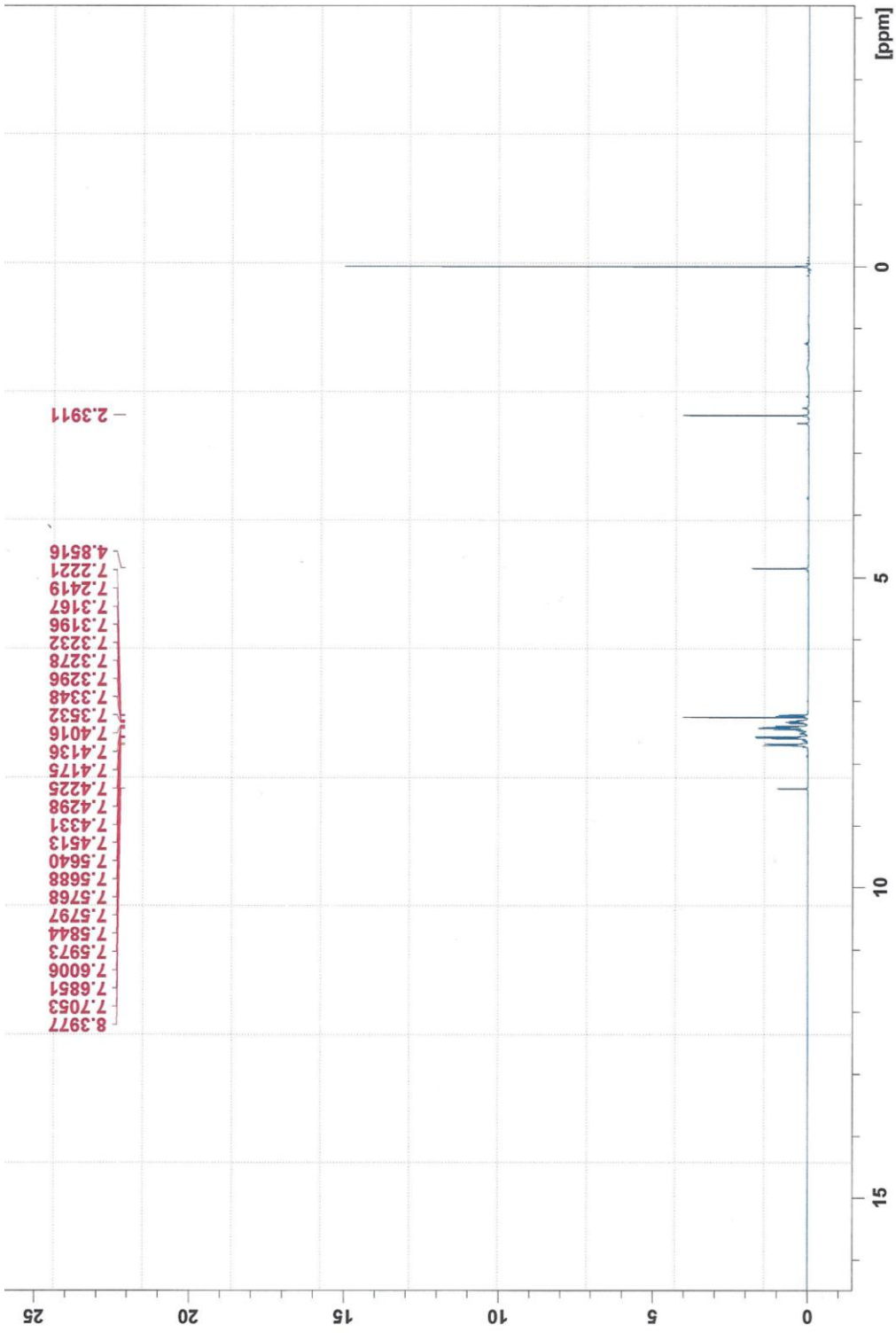

Figure 77: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(*p*-tolyl)methanimine **28**

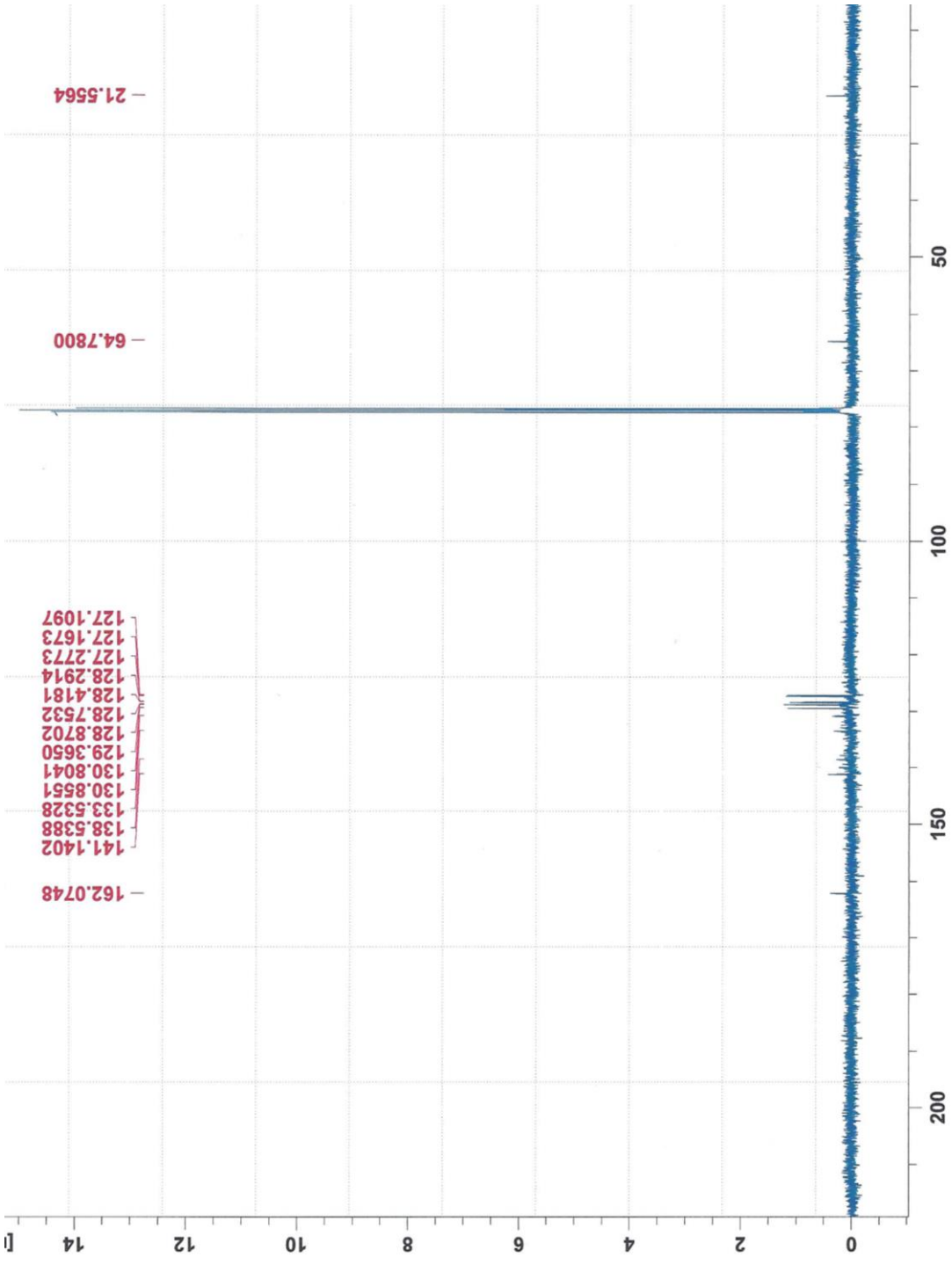

Figure 78: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(*p*-tolyl)methanimine **28**

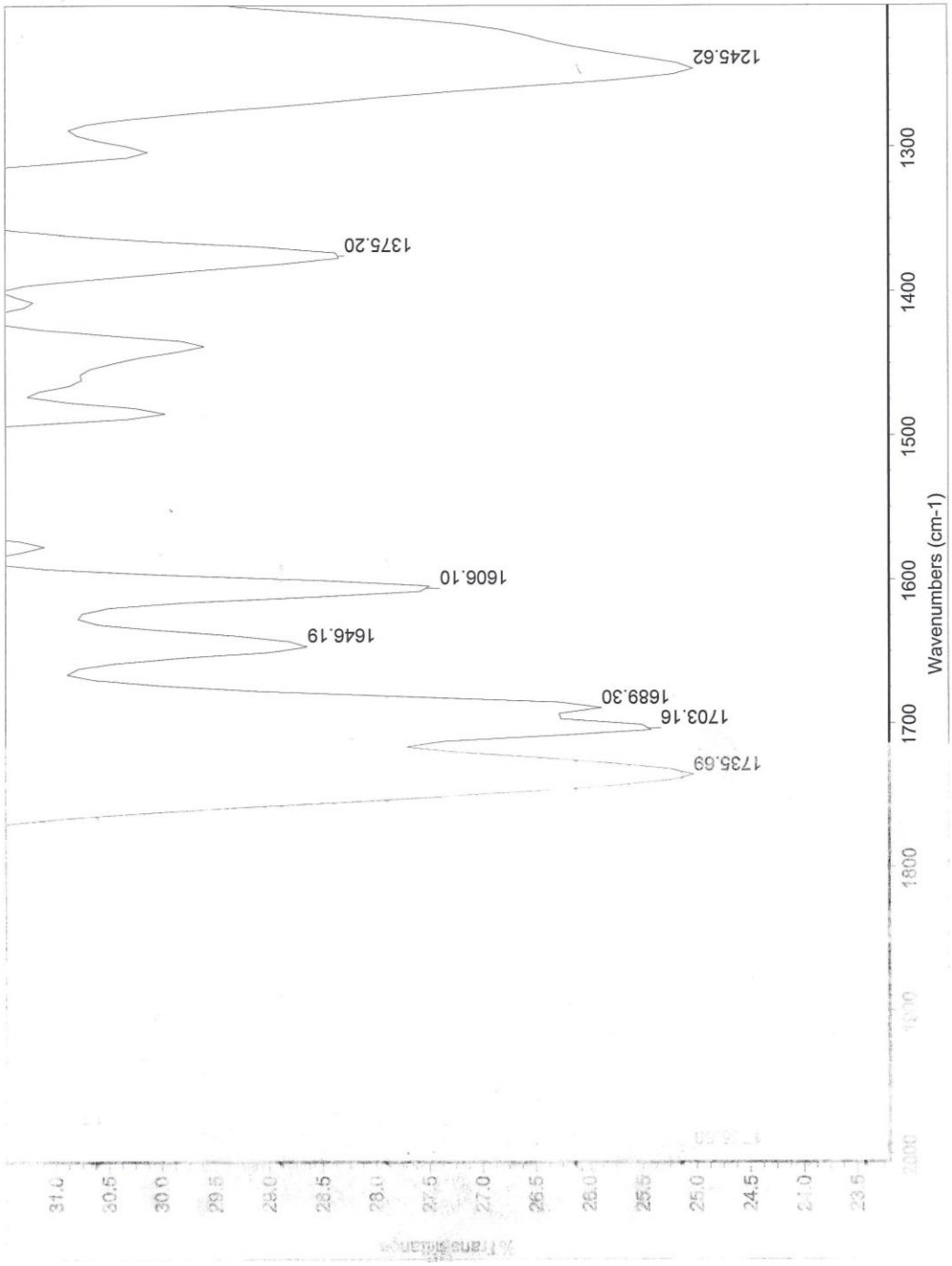

Figure 79: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(*p*-tolyl)methanimine **28**

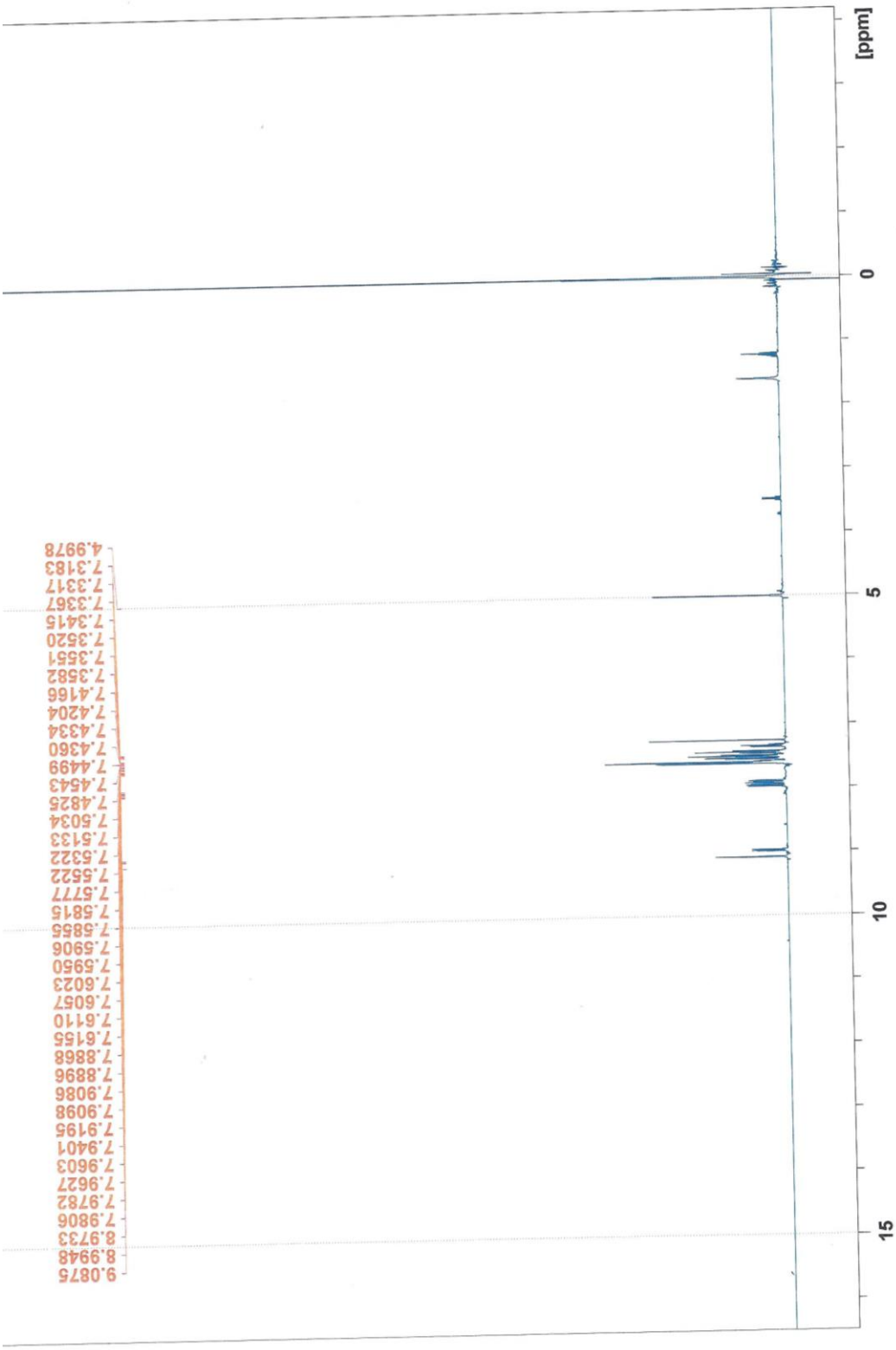

Figure 80: ^{1}H NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(naphthalen-1-yl)methanimine **29**

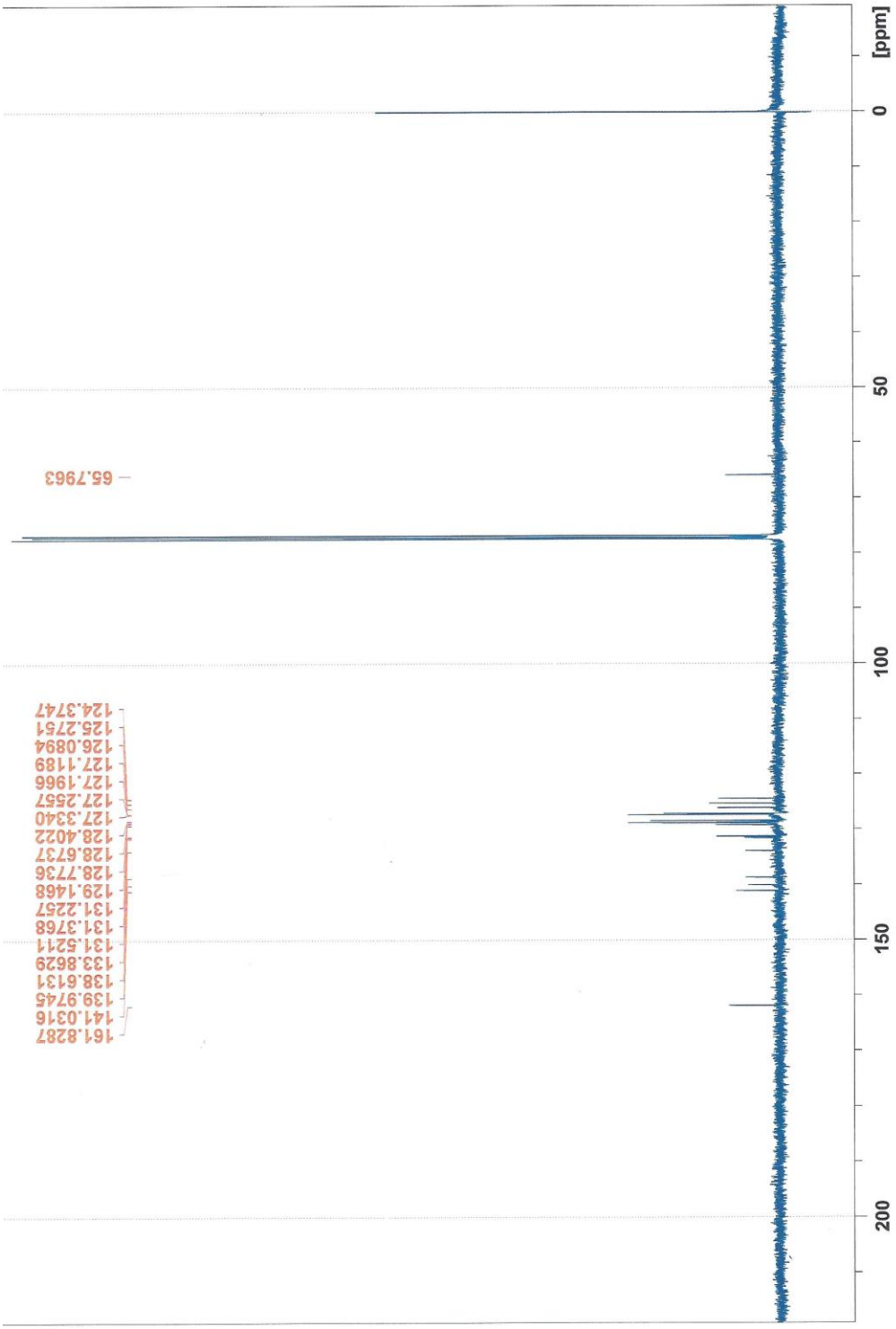

Figure 81: ^{13}C NMR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(naphthalen-1-yl)methanimine **29**

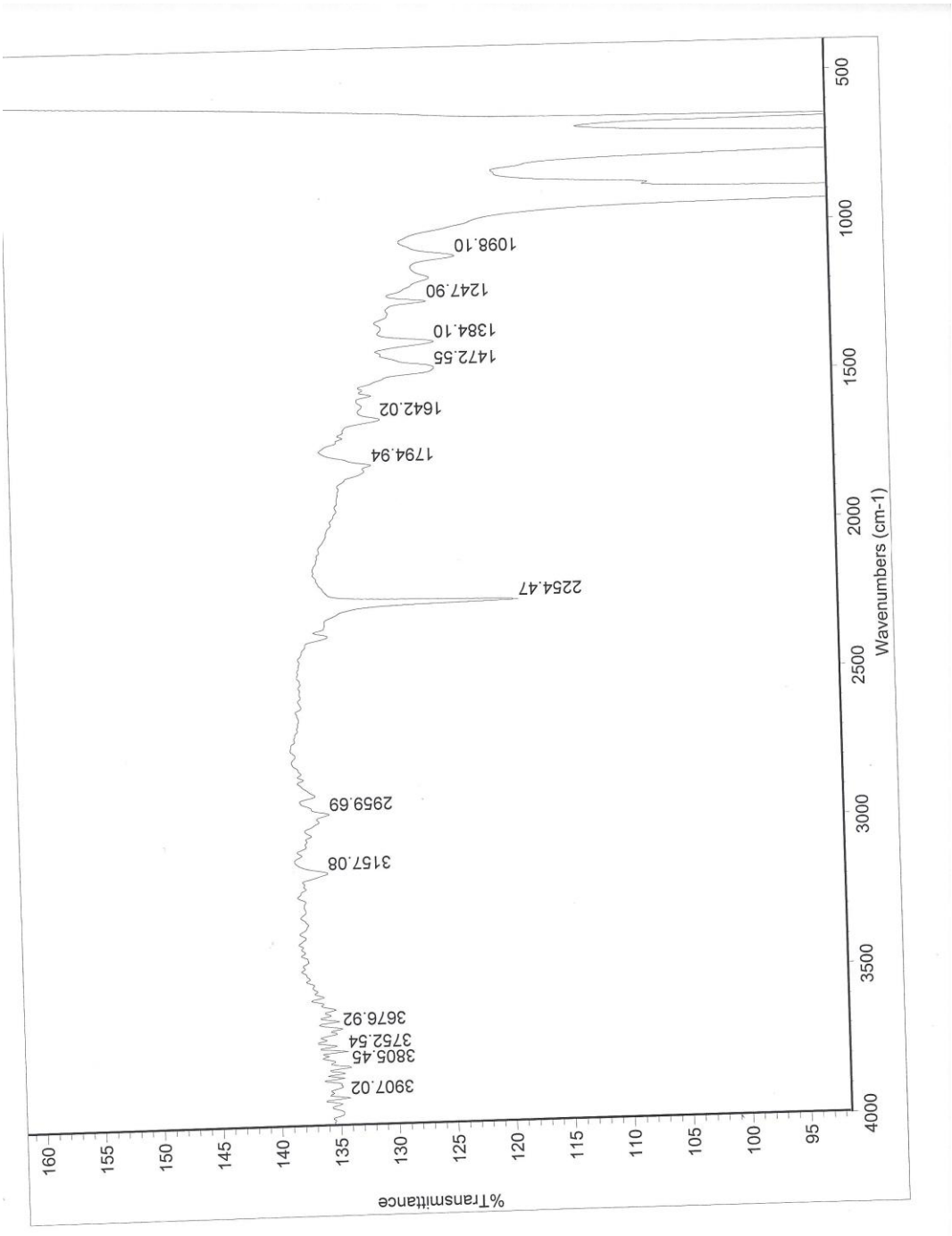

Figure 82: IR Spectrum of (*E*)-*N*-([1,1'-biphenyl]-4-ylmethyl)-1-(naphthalen-1-yl)methanimine **29**

NMR and IR Spectra of Amides

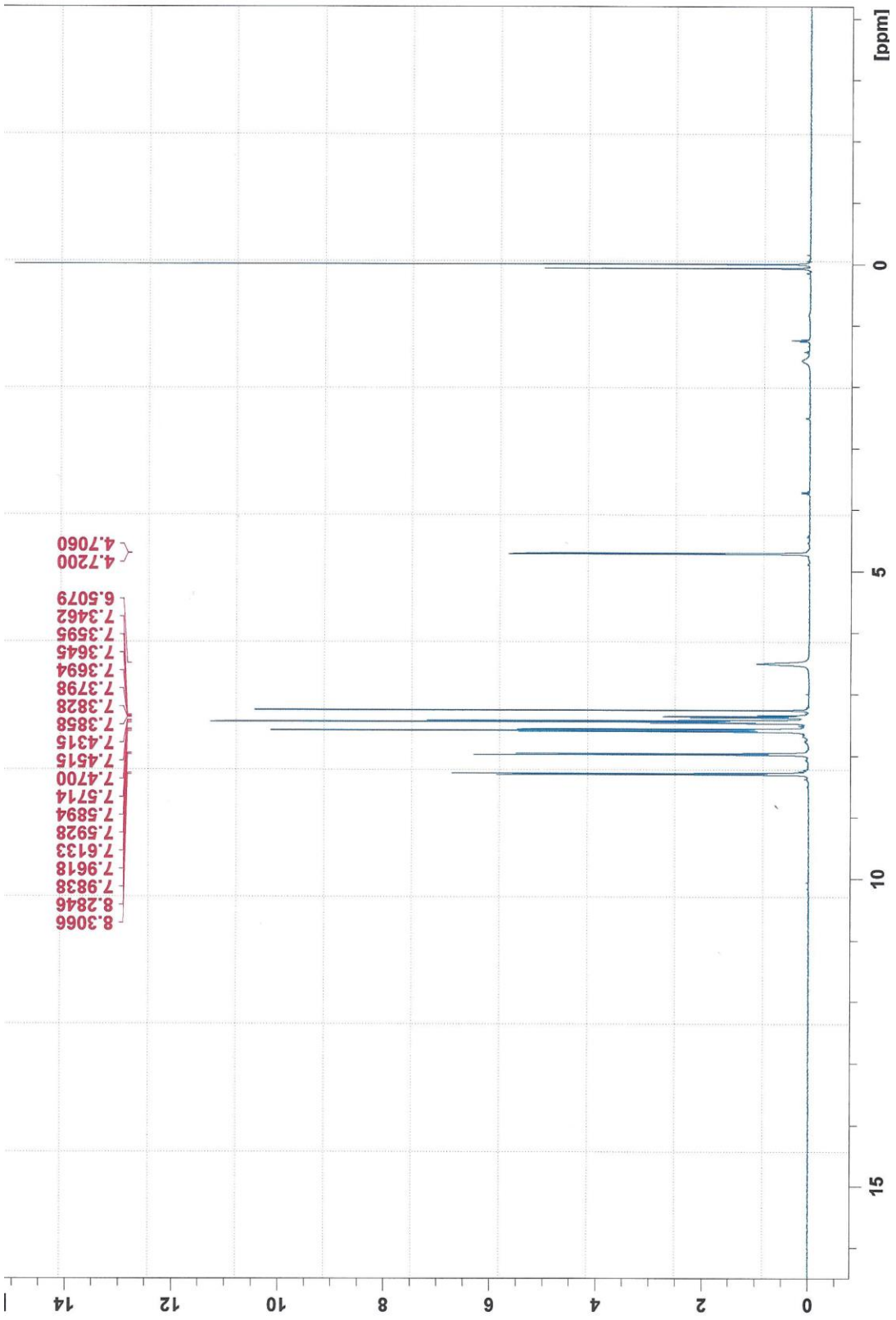

Figure 83: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-nitrobenzamide **30**

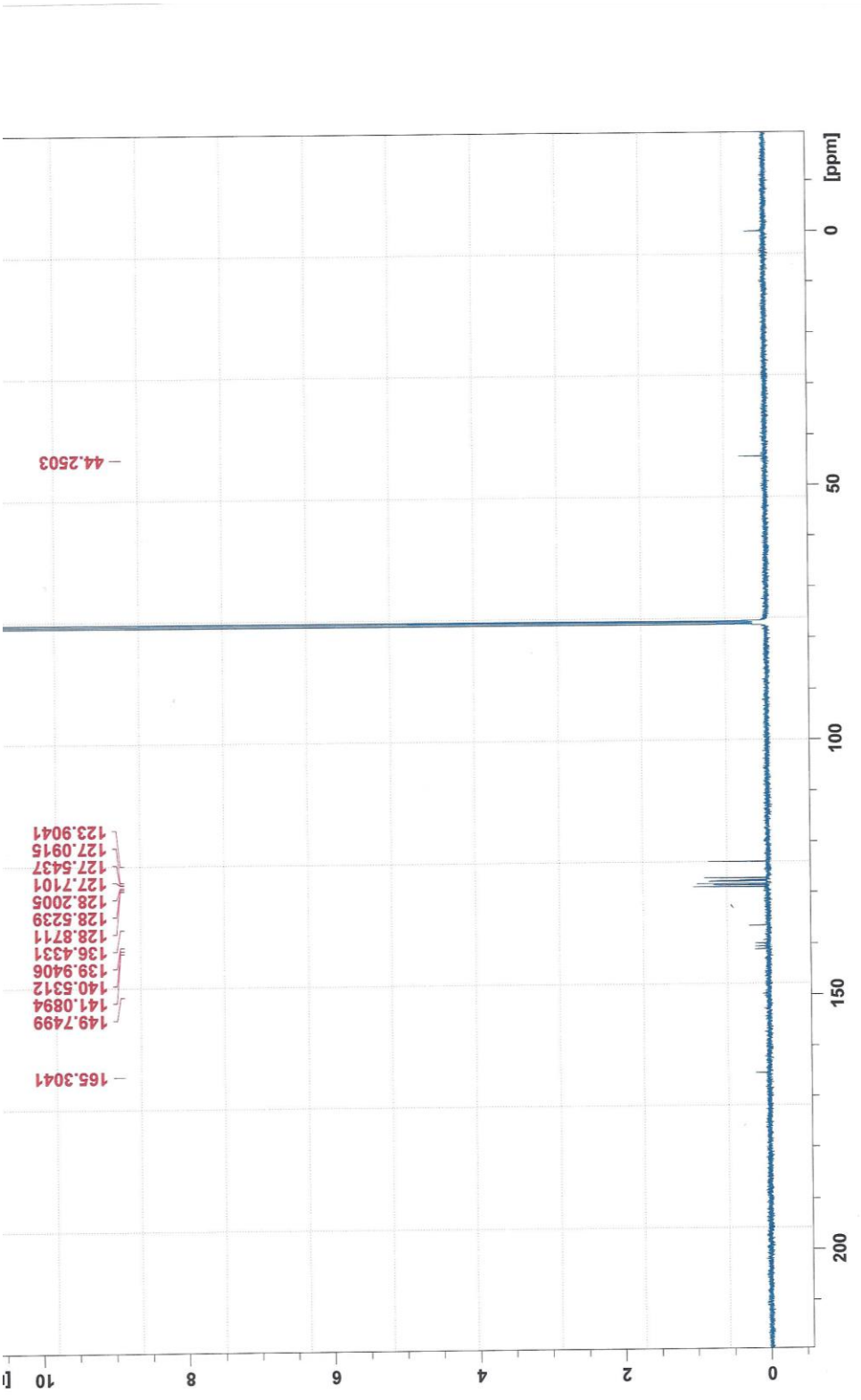

Figure 84: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-nitrobenzamide **30**

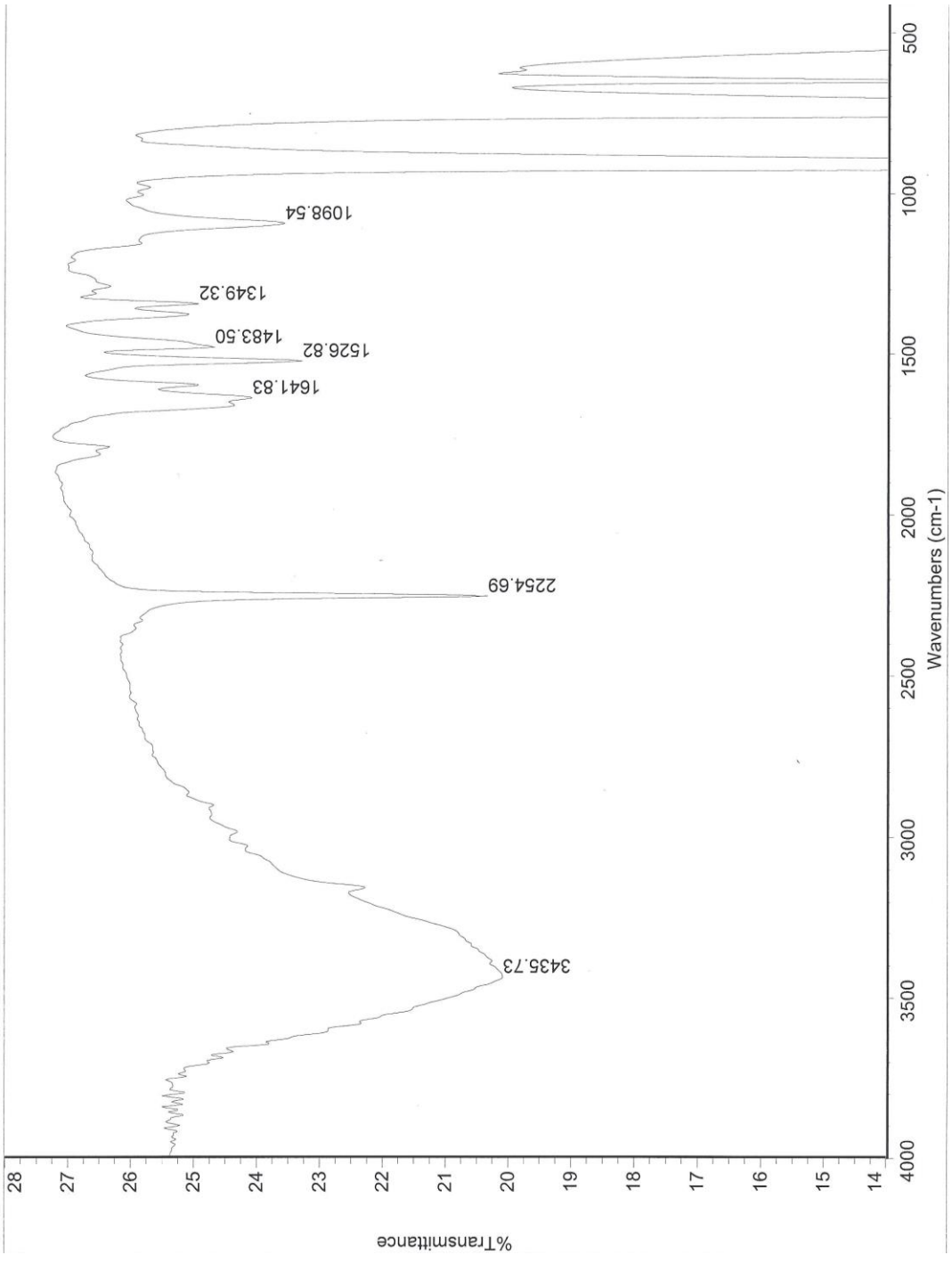

Figure 85: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-nitrobenzamide **30**

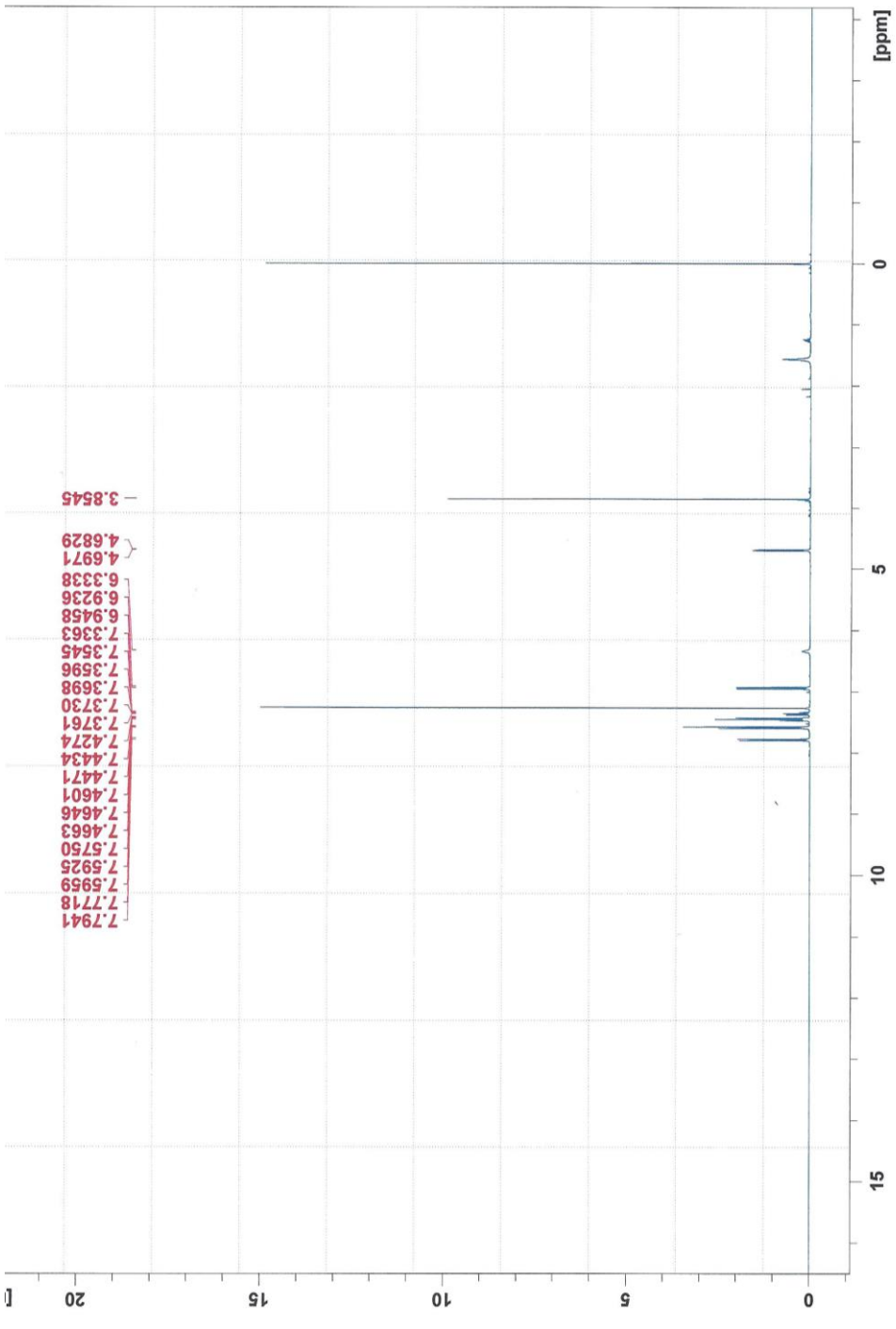

Figure 86: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-methoxybenzamide **31**

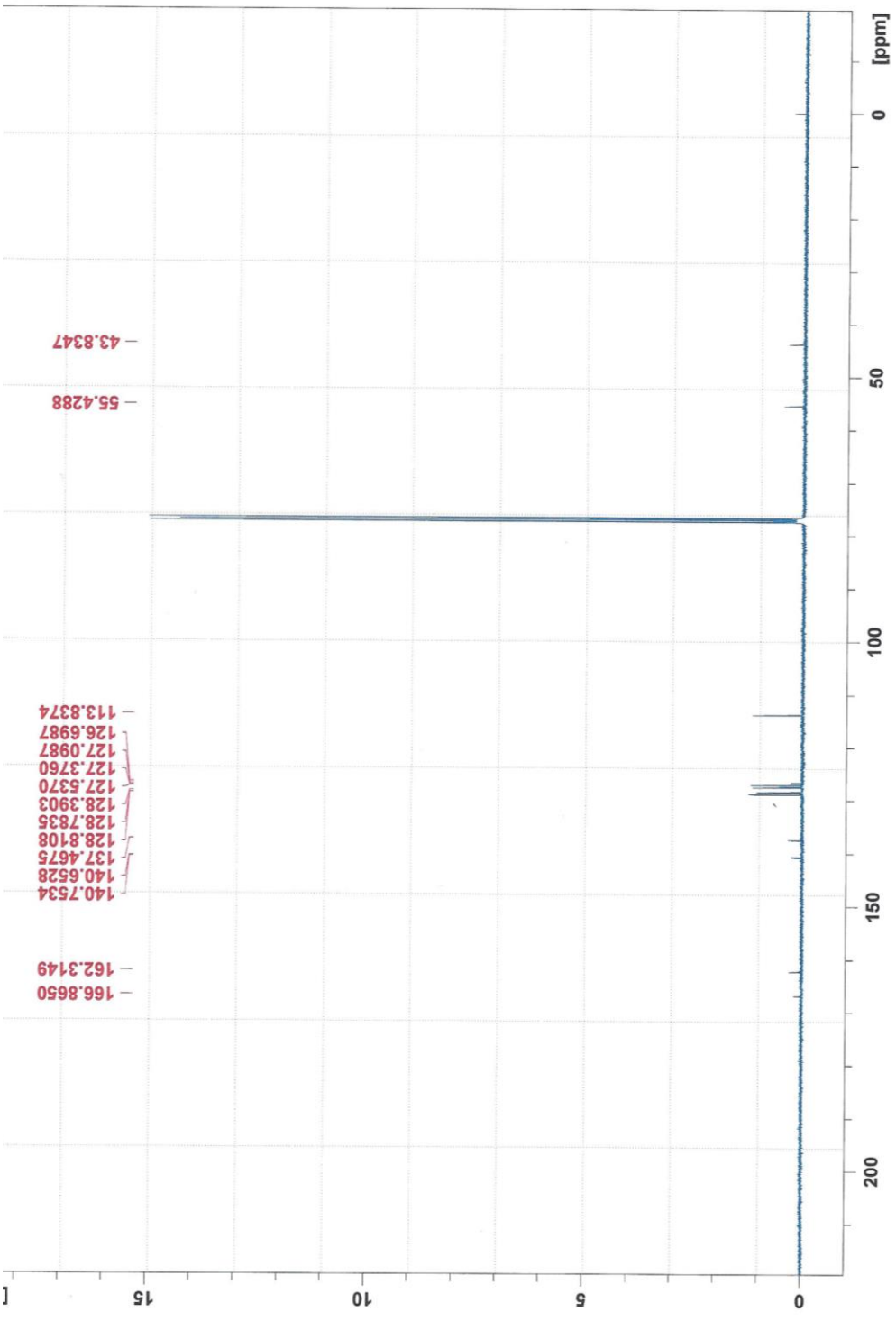

Figure 87: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-methoxybenzamide **31**

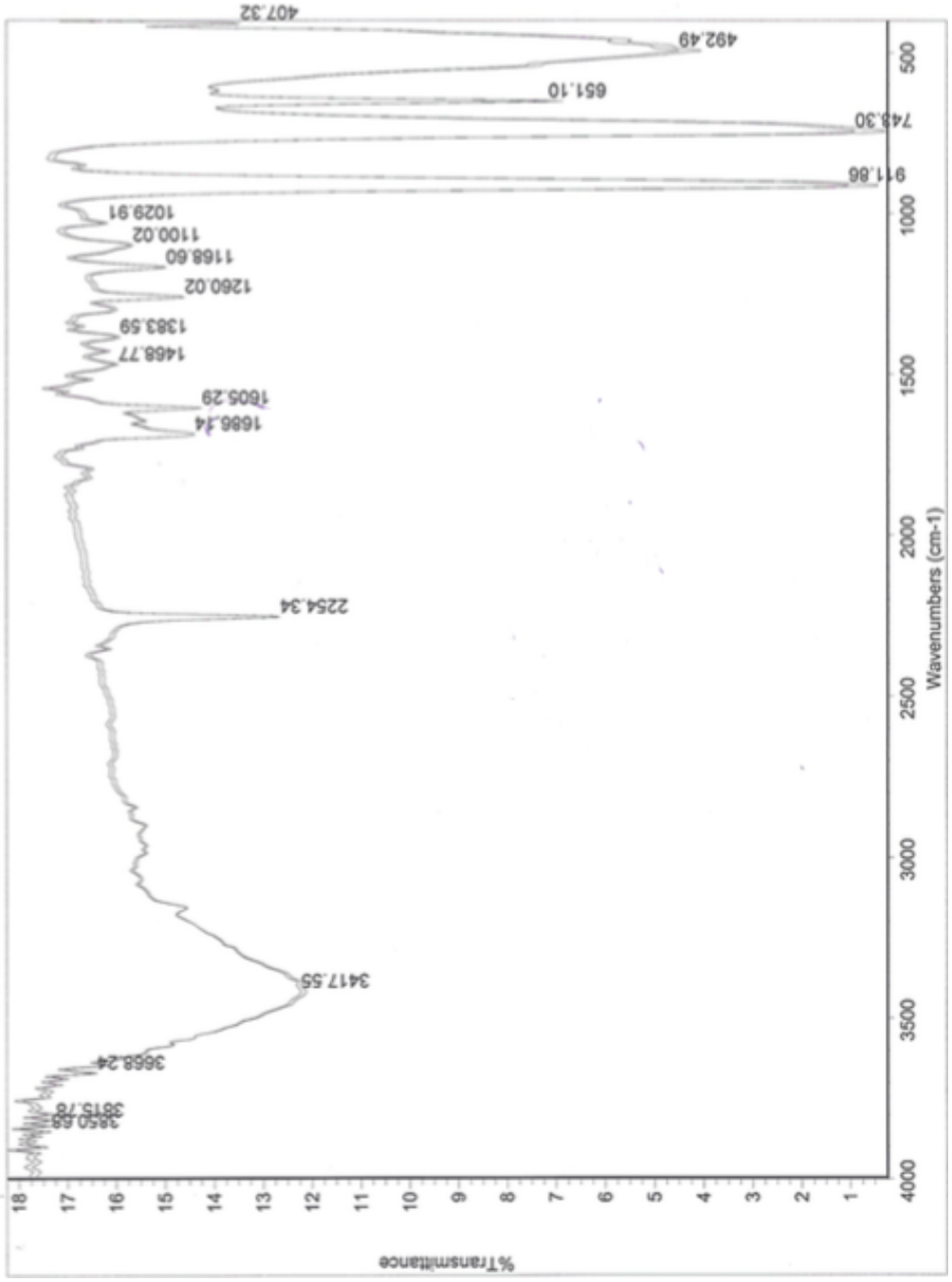

Figure 88: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-methoxybenzamide **31**

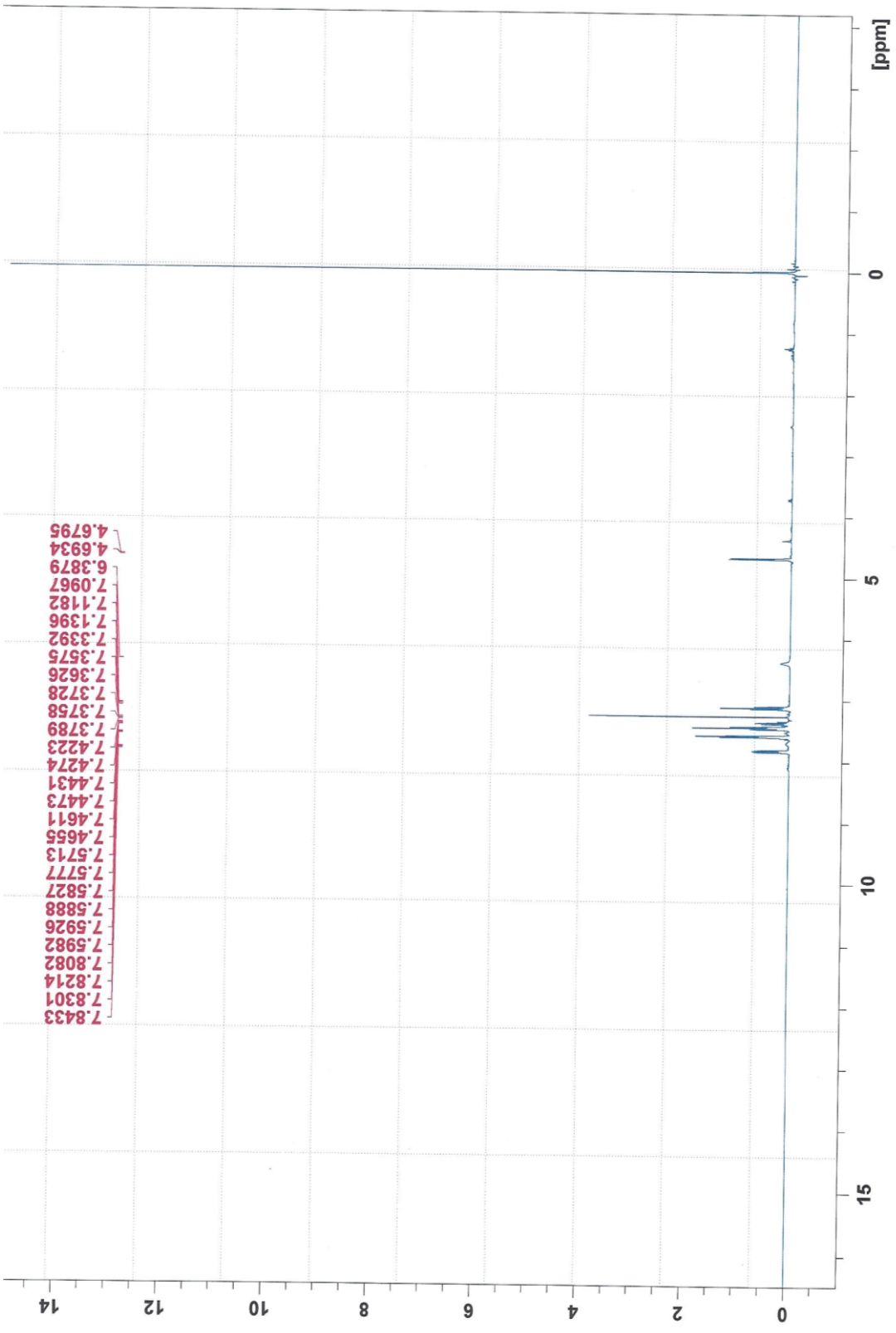

Figure 89: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-fluorobenzamide **32**

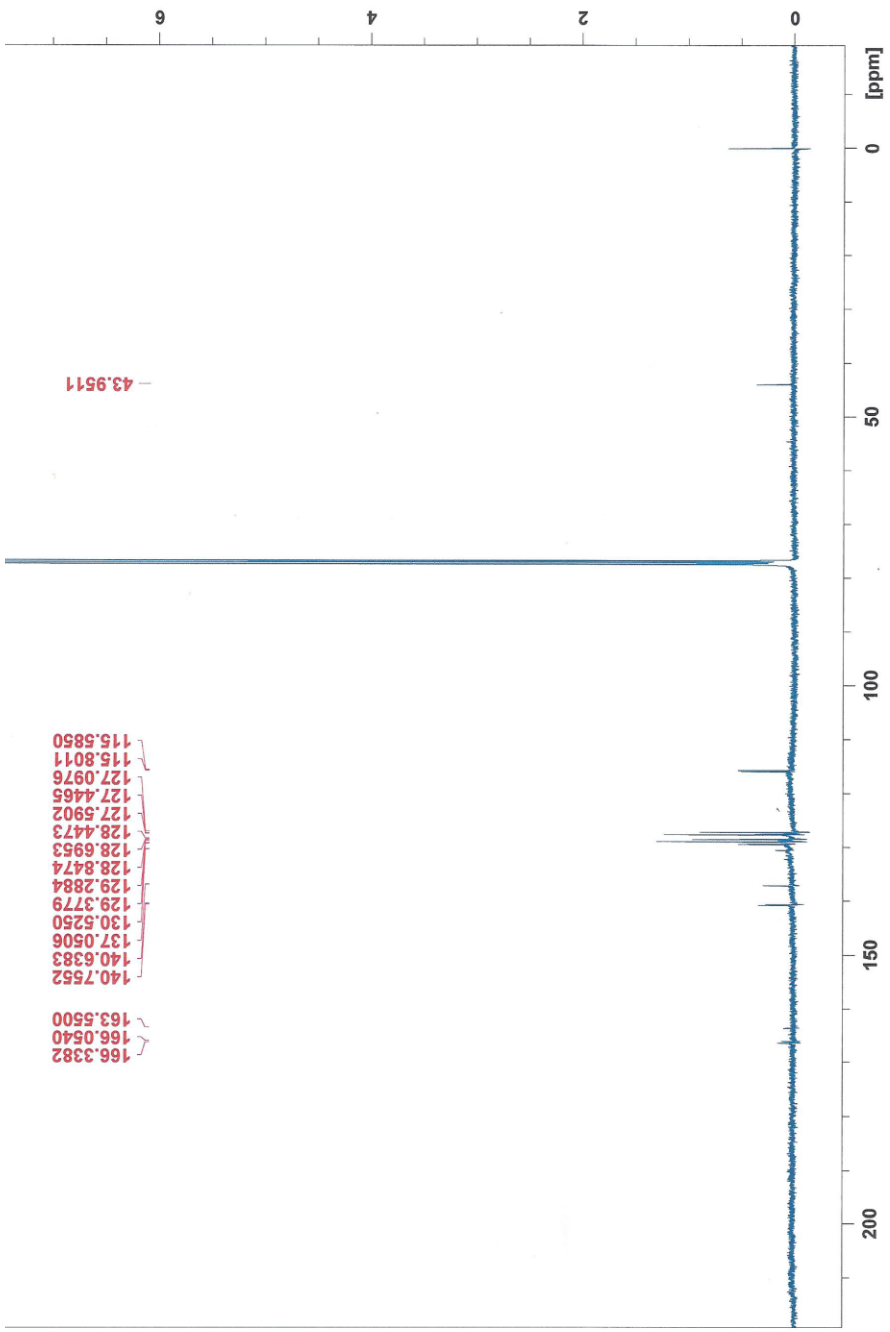

Figure 90: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-fluorobenzamide **32**

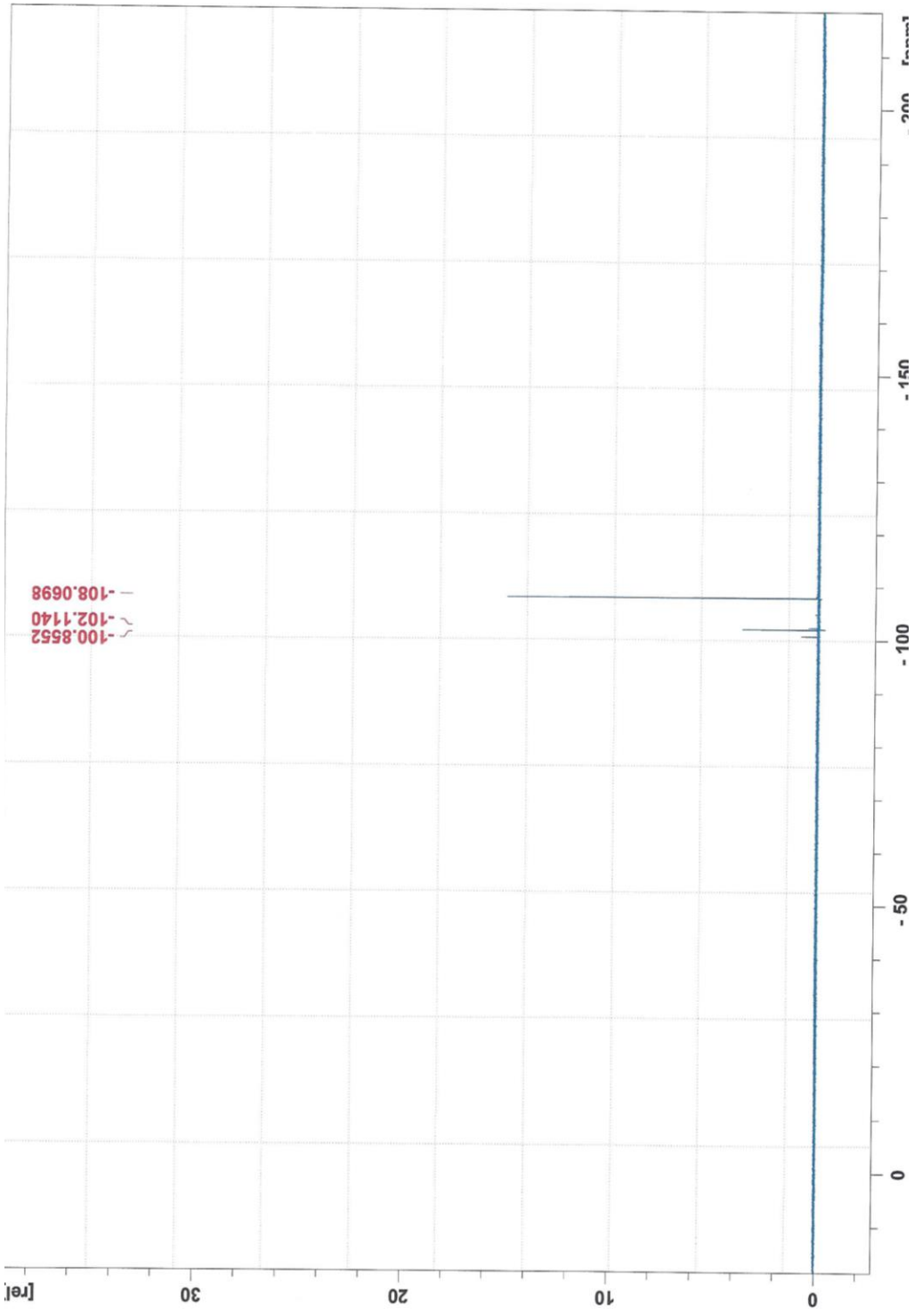

Figure 91: ^{19}F NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-fluorobenzamide **32**

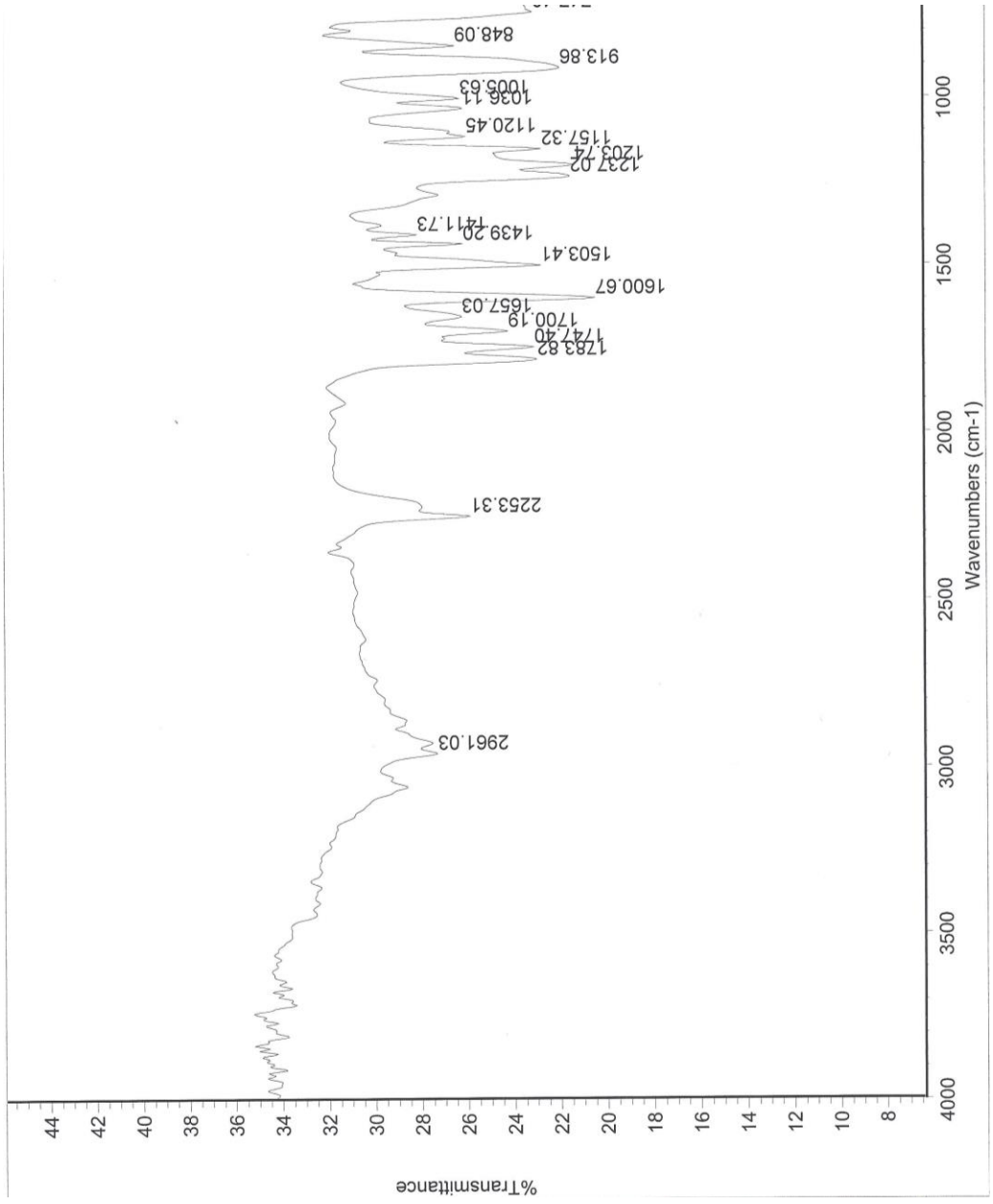

Figure 92: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-fluorobenzamide **32**

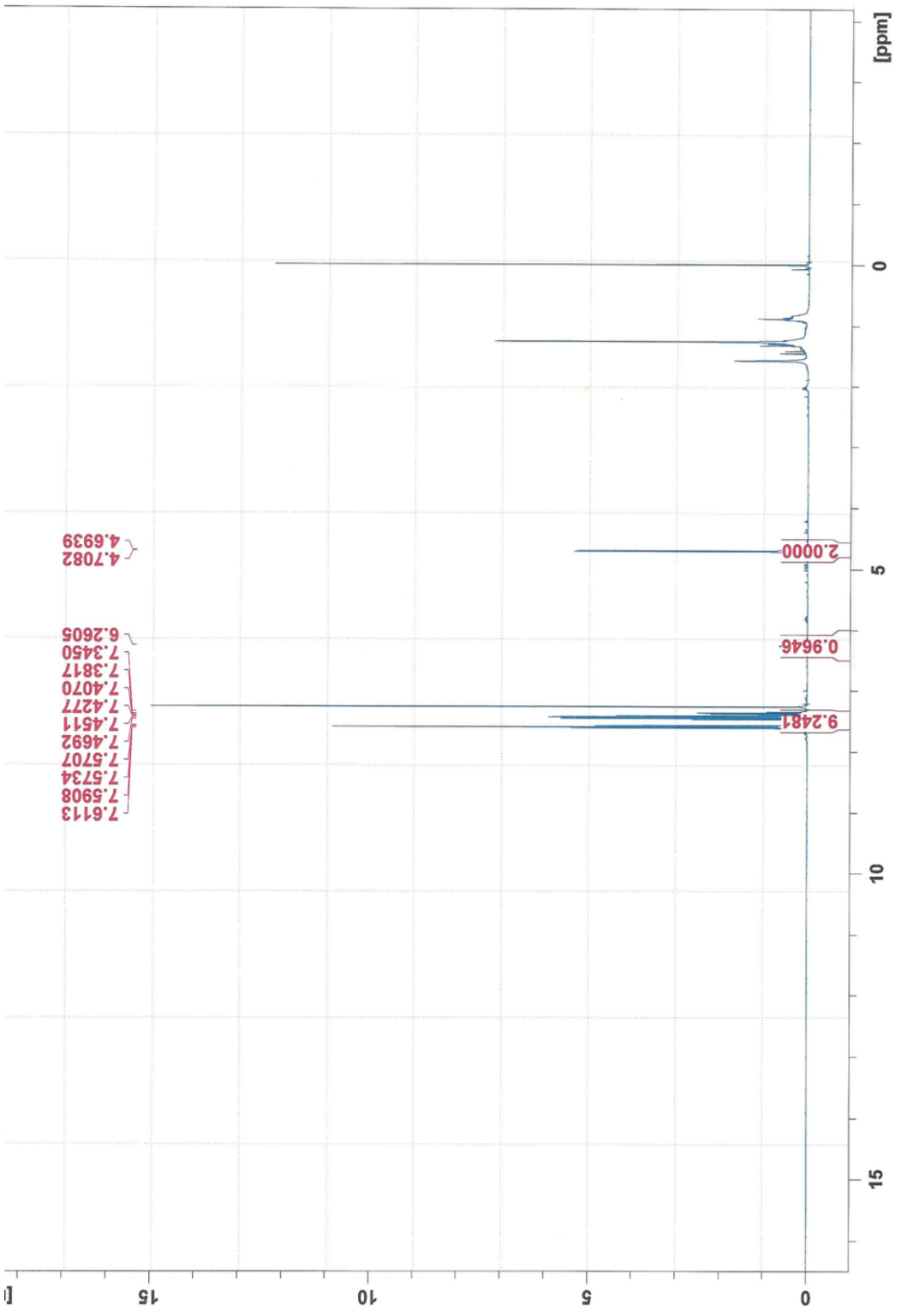

Figure 93: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide **33**

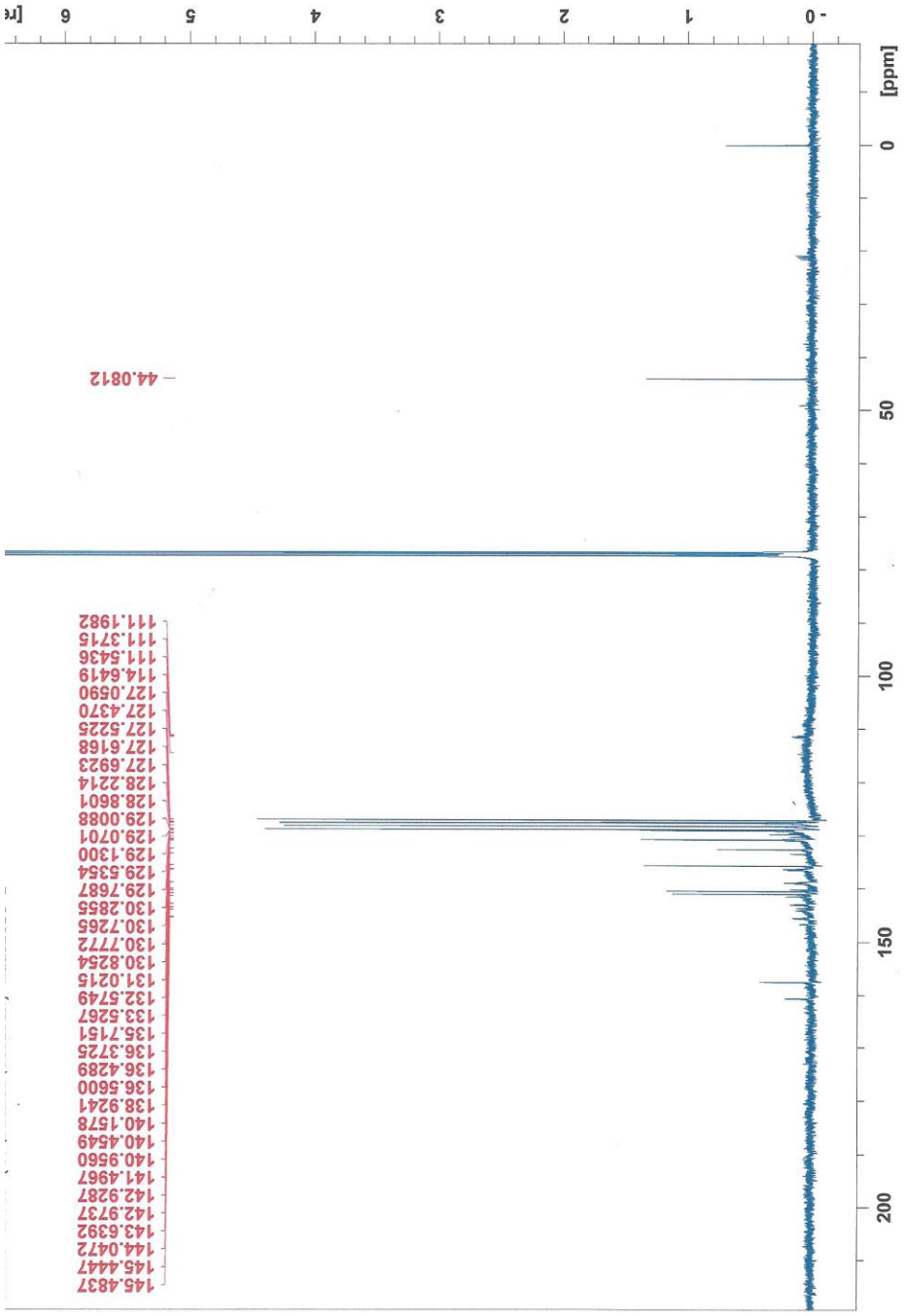

Figure 94: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide **33**

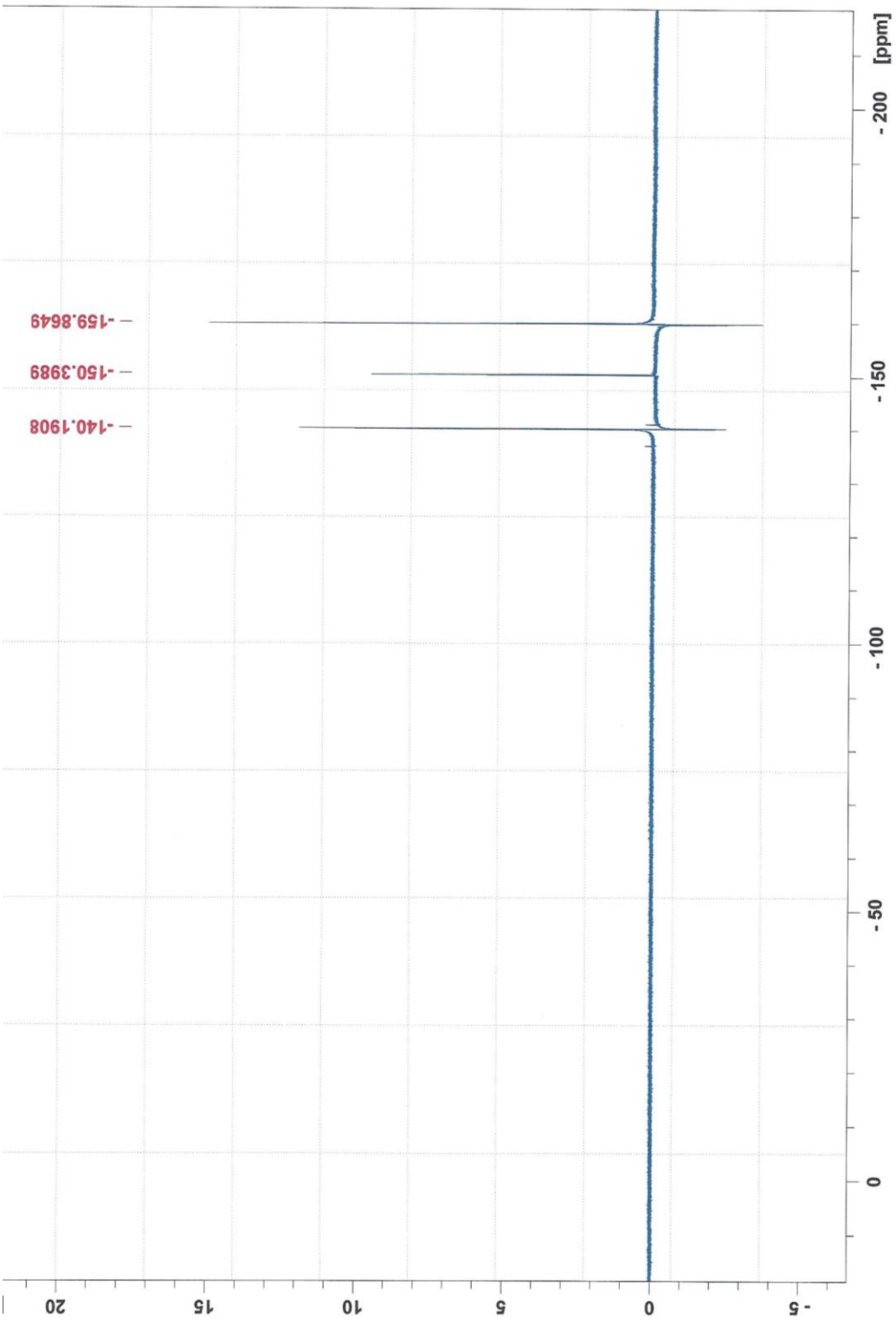

Figure 95: ^{19}F NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide **33**

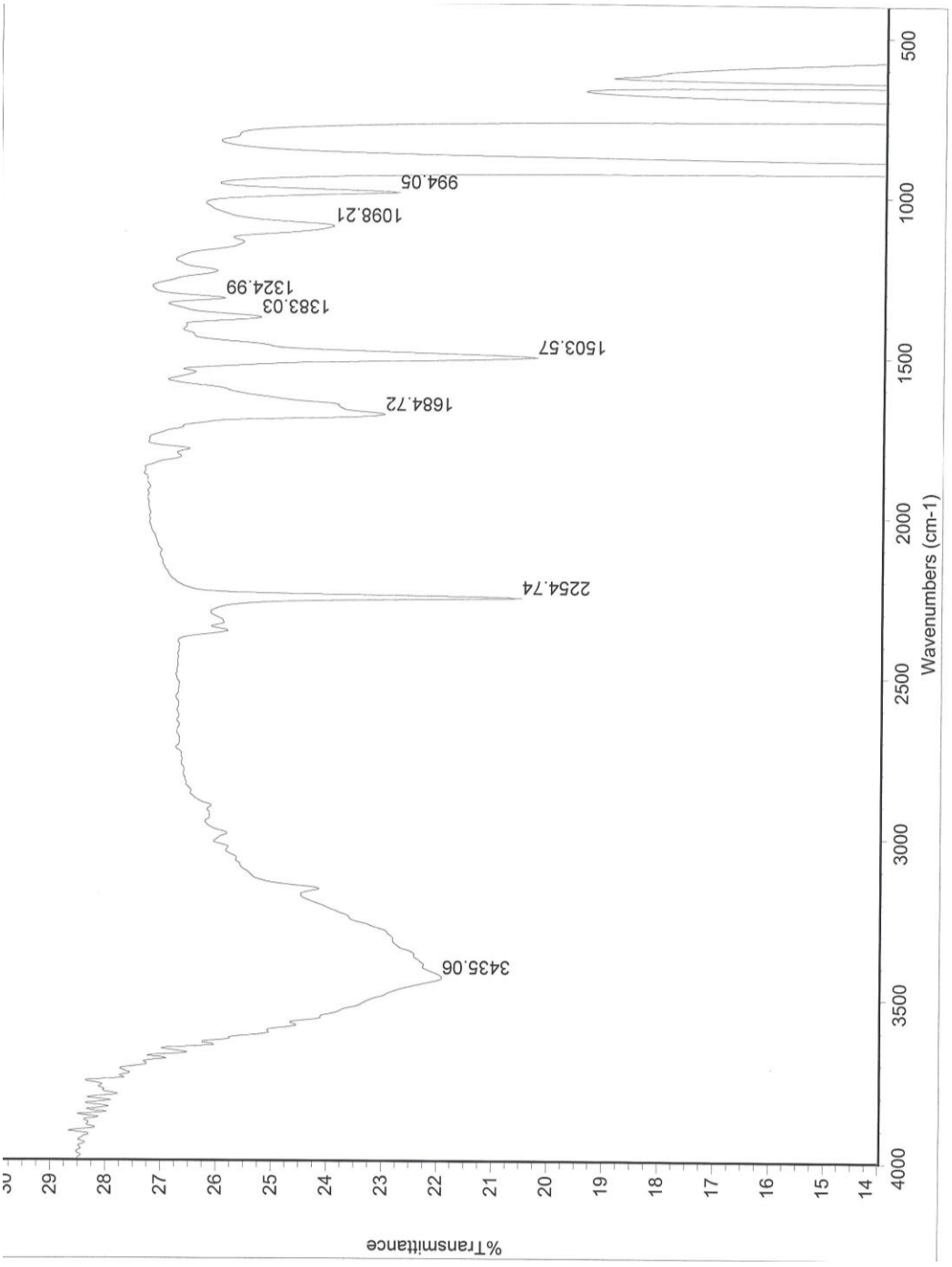

Figure 96: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide **33**

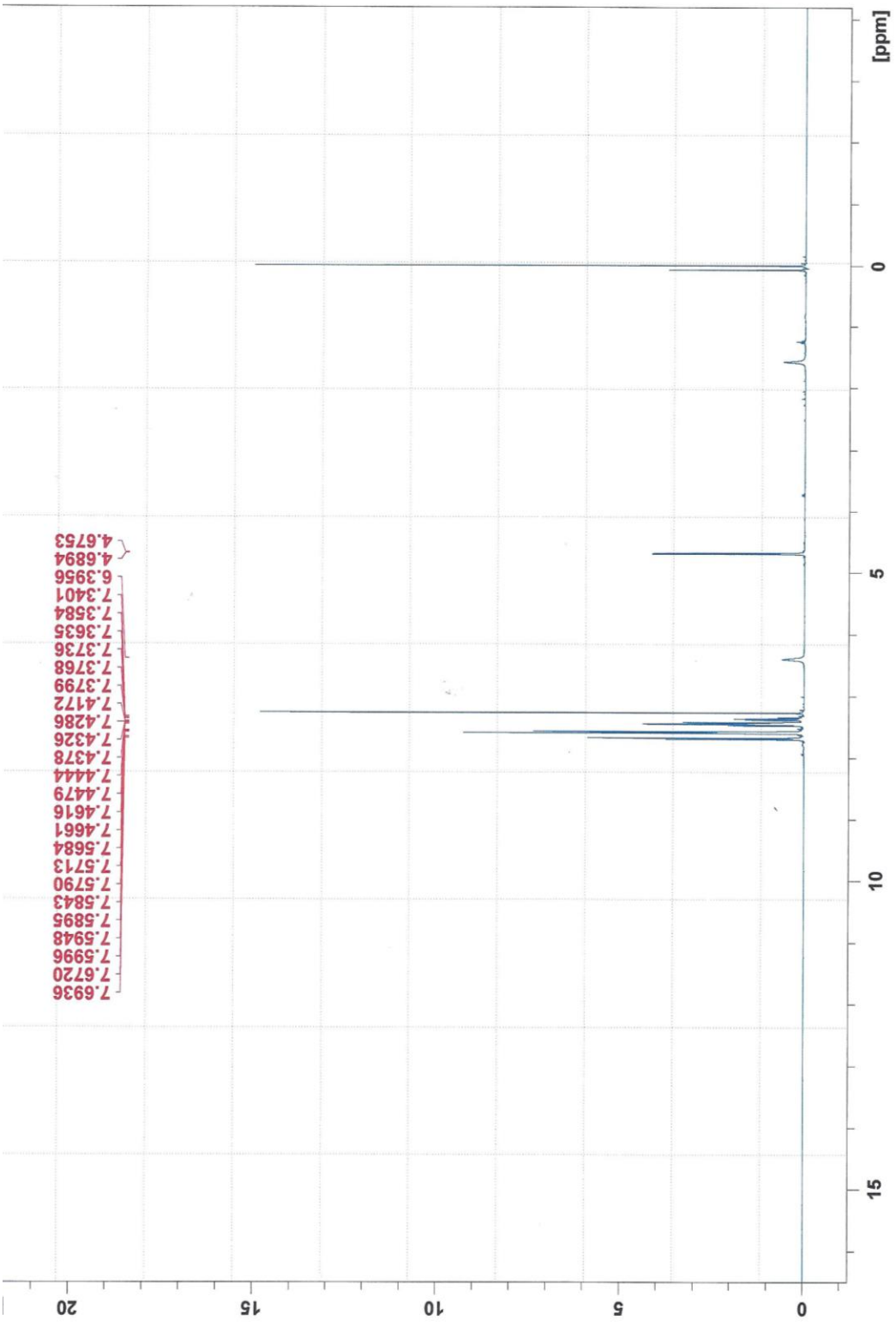

Figure 97: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-bromobenzamide **34**

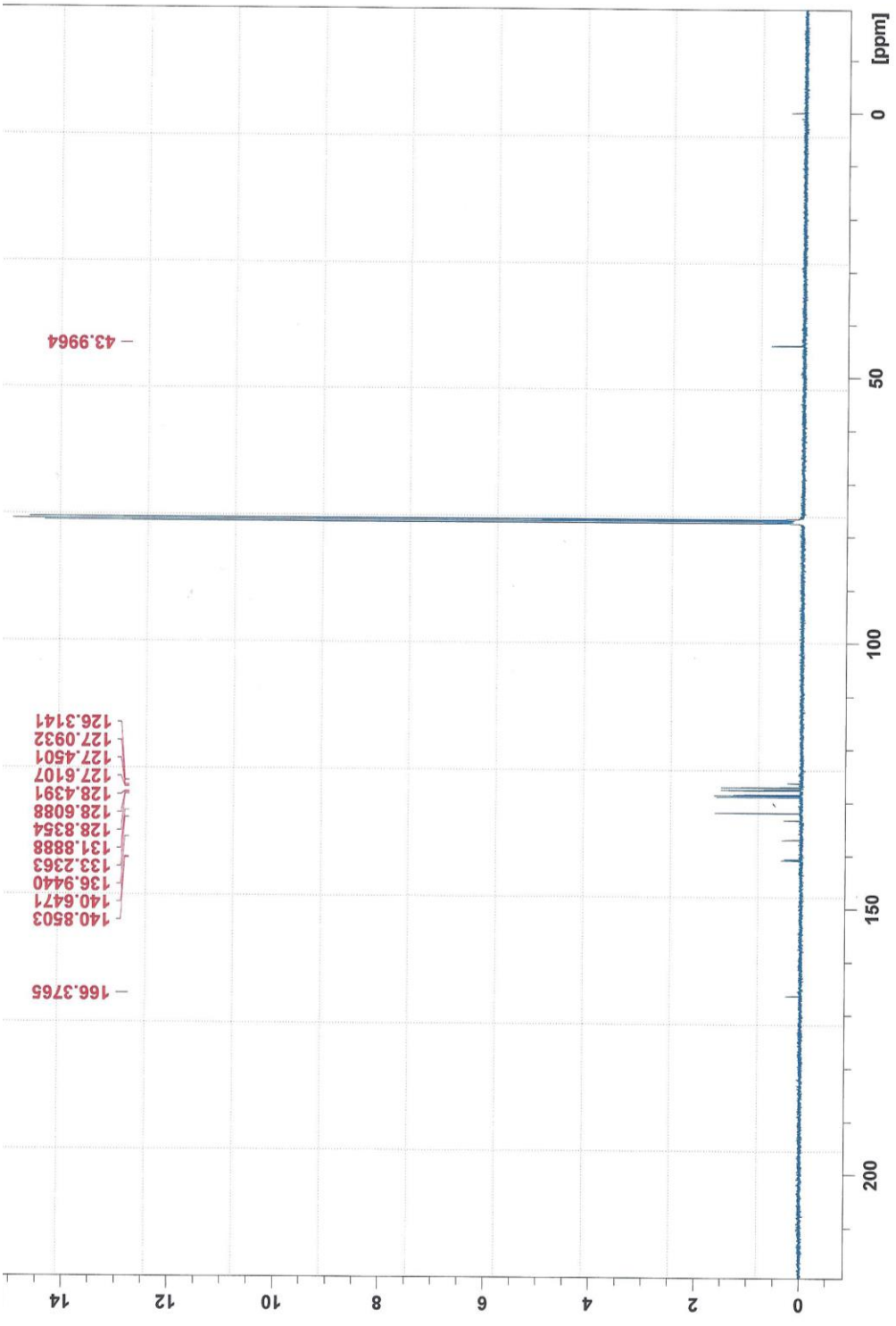

Figure 98: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-bromobenzamide **34**

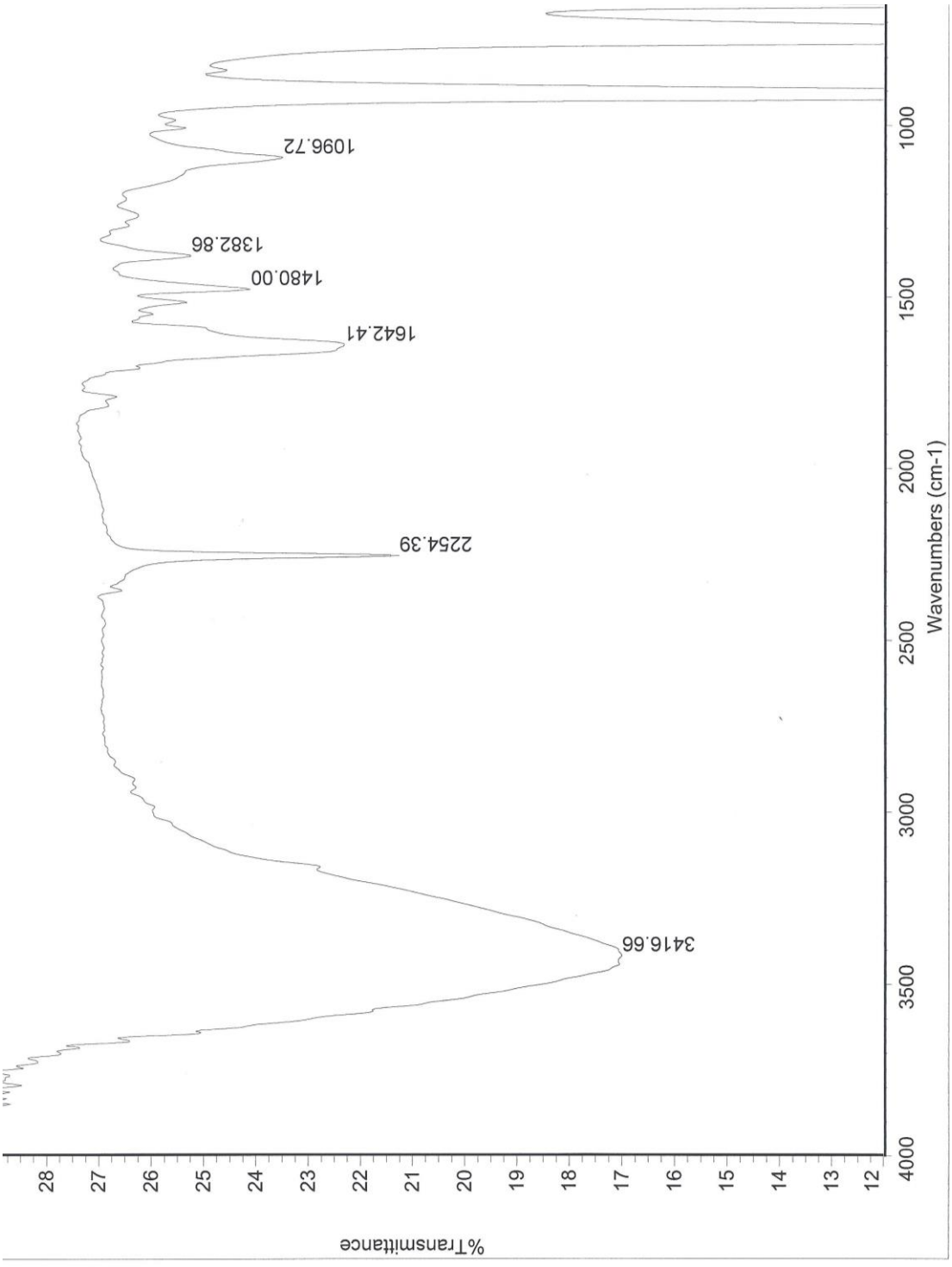

Figure 99: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-bromobenzamide **34**

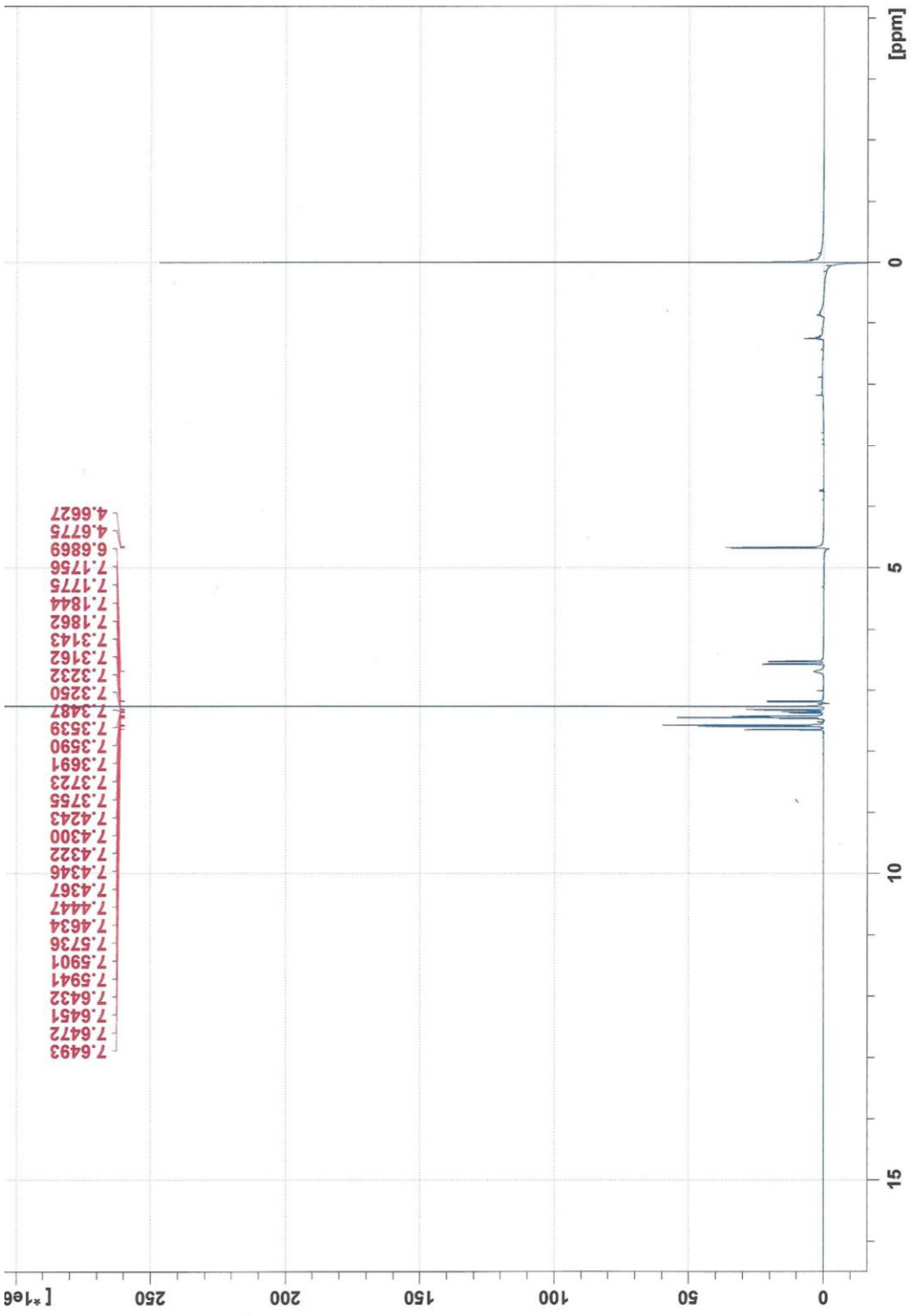

Figure 100: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)furan-2-carboxamide **35**

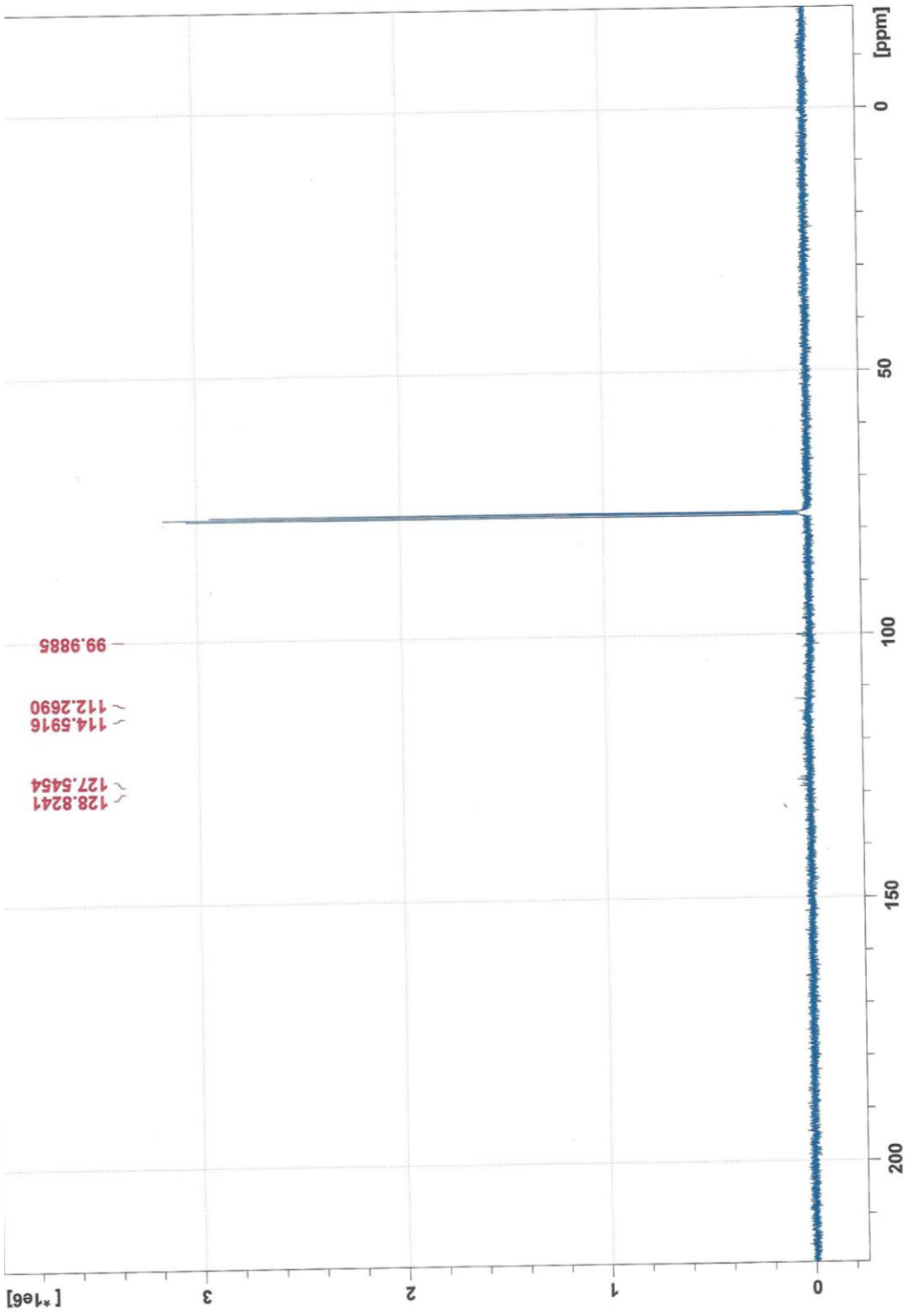

Figure 101: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)furan-2-carboxamide **35**

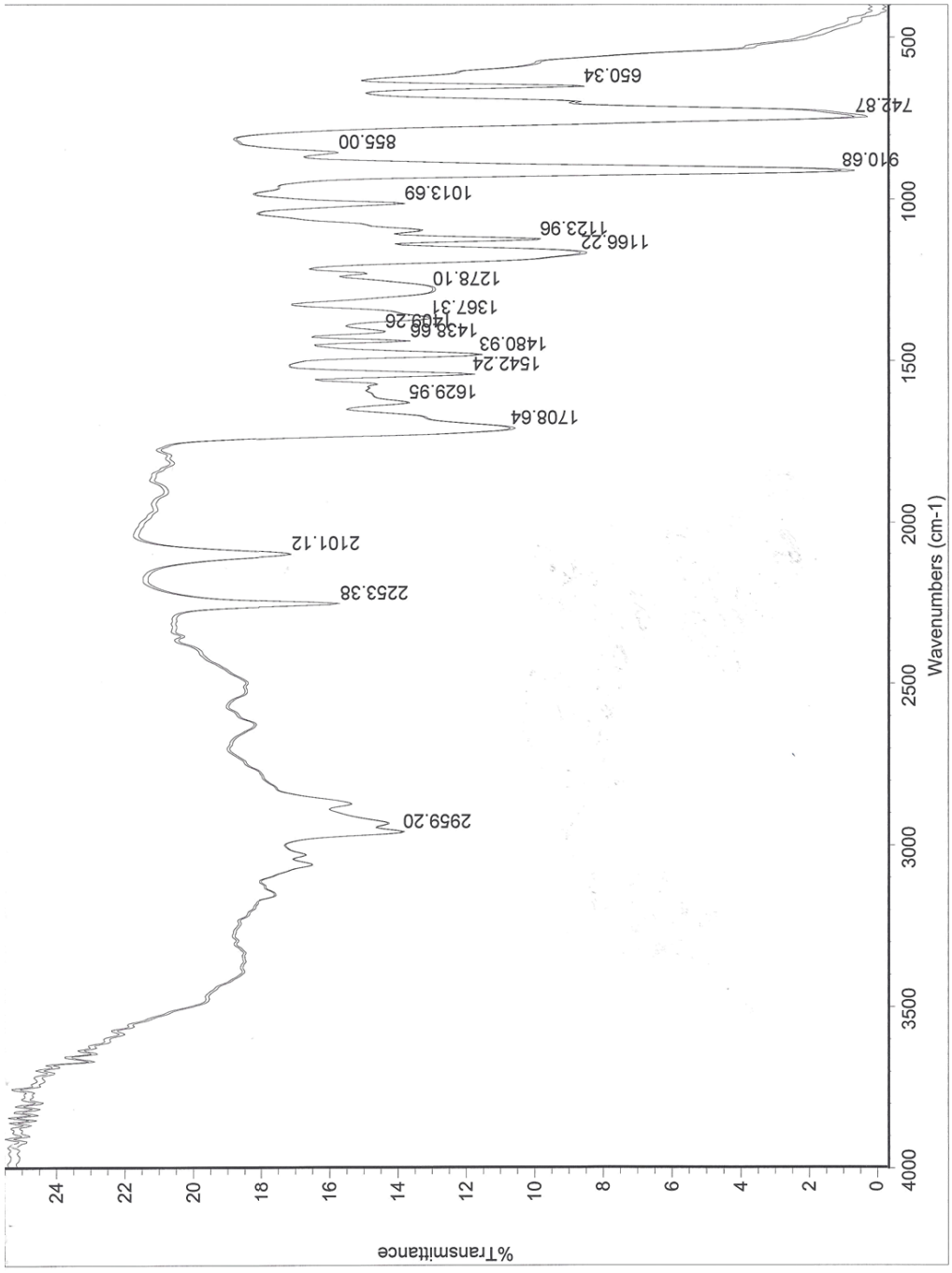

Figure 102: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)furan-2-carboxamide **35**

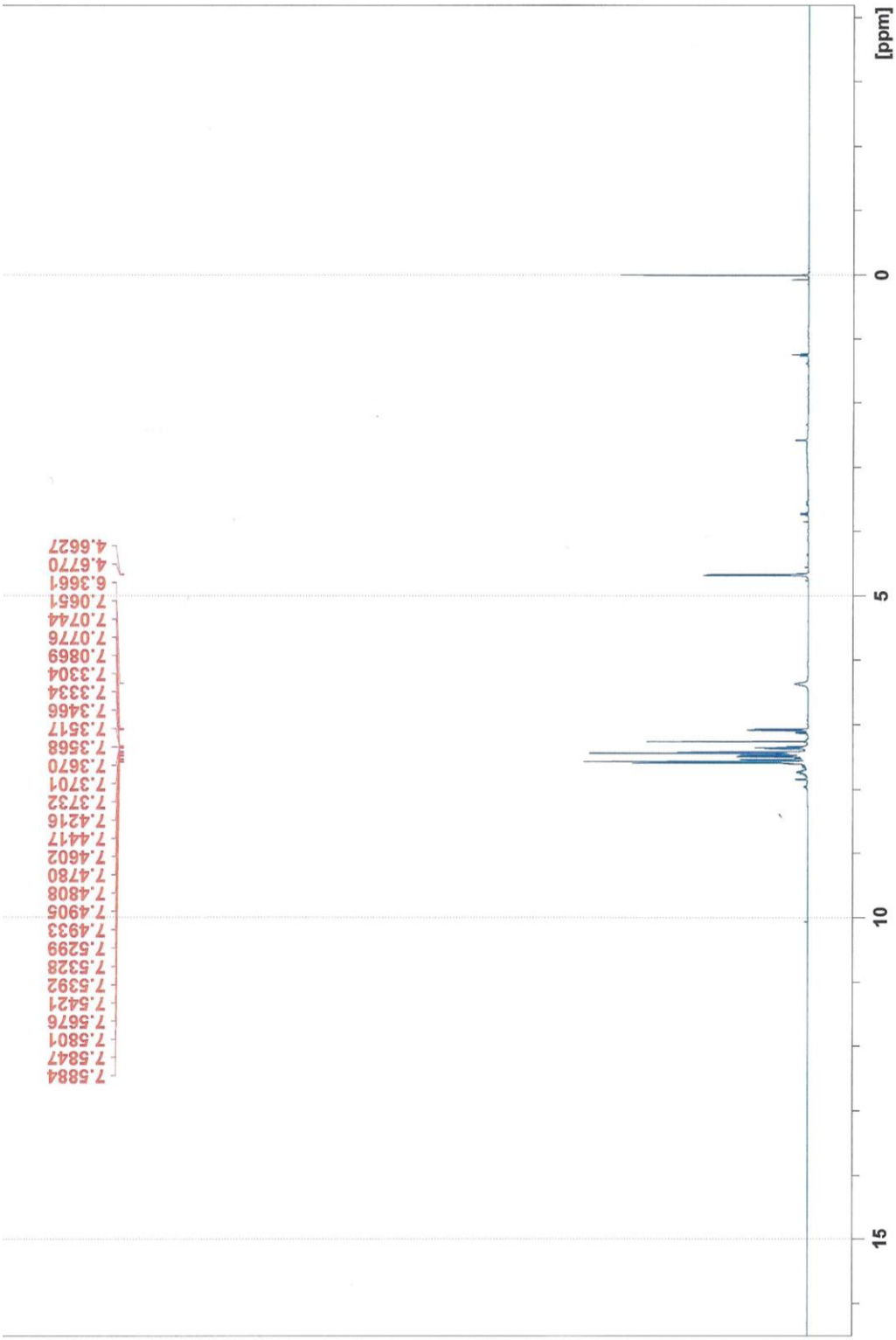

Figure 103: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)thiophene-2-carboxamide **36**

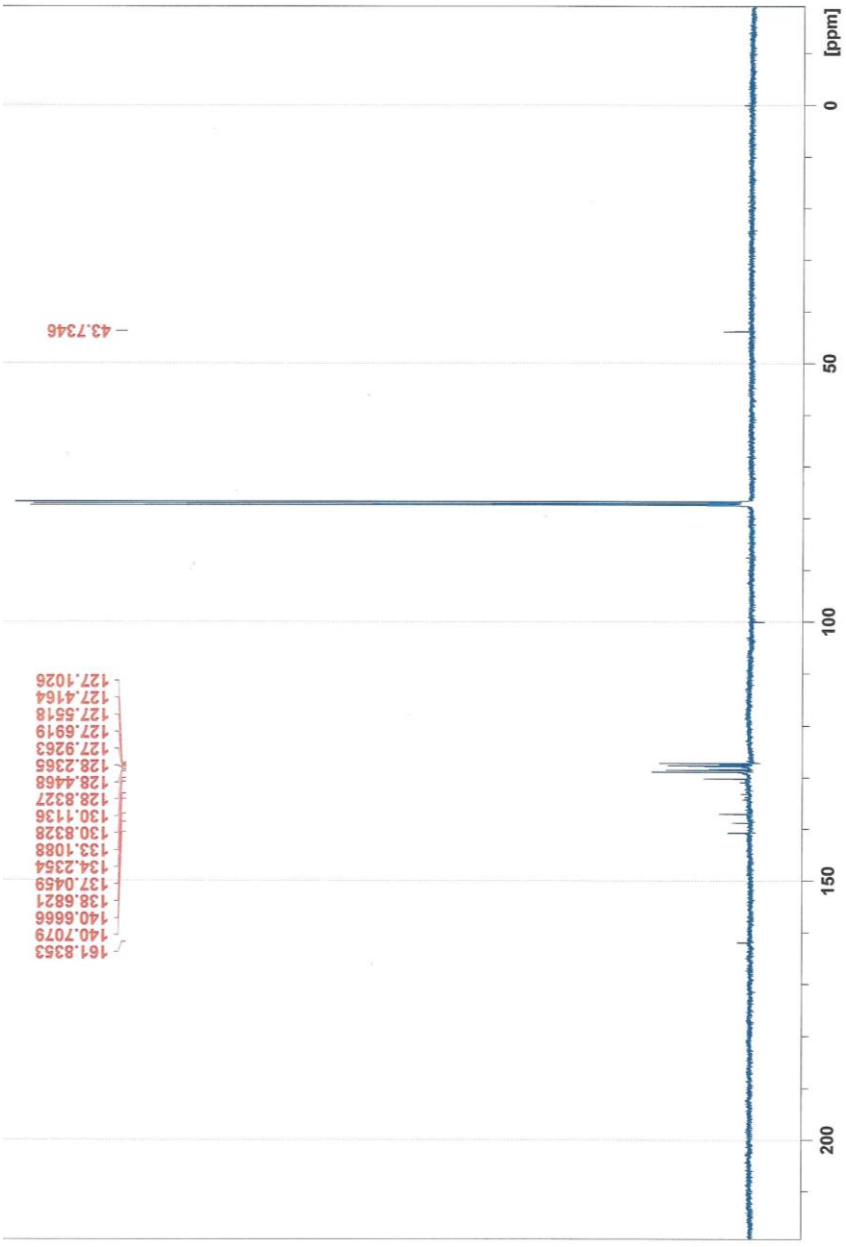

Figure 104: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)thiophene-2-carboxamide **36**

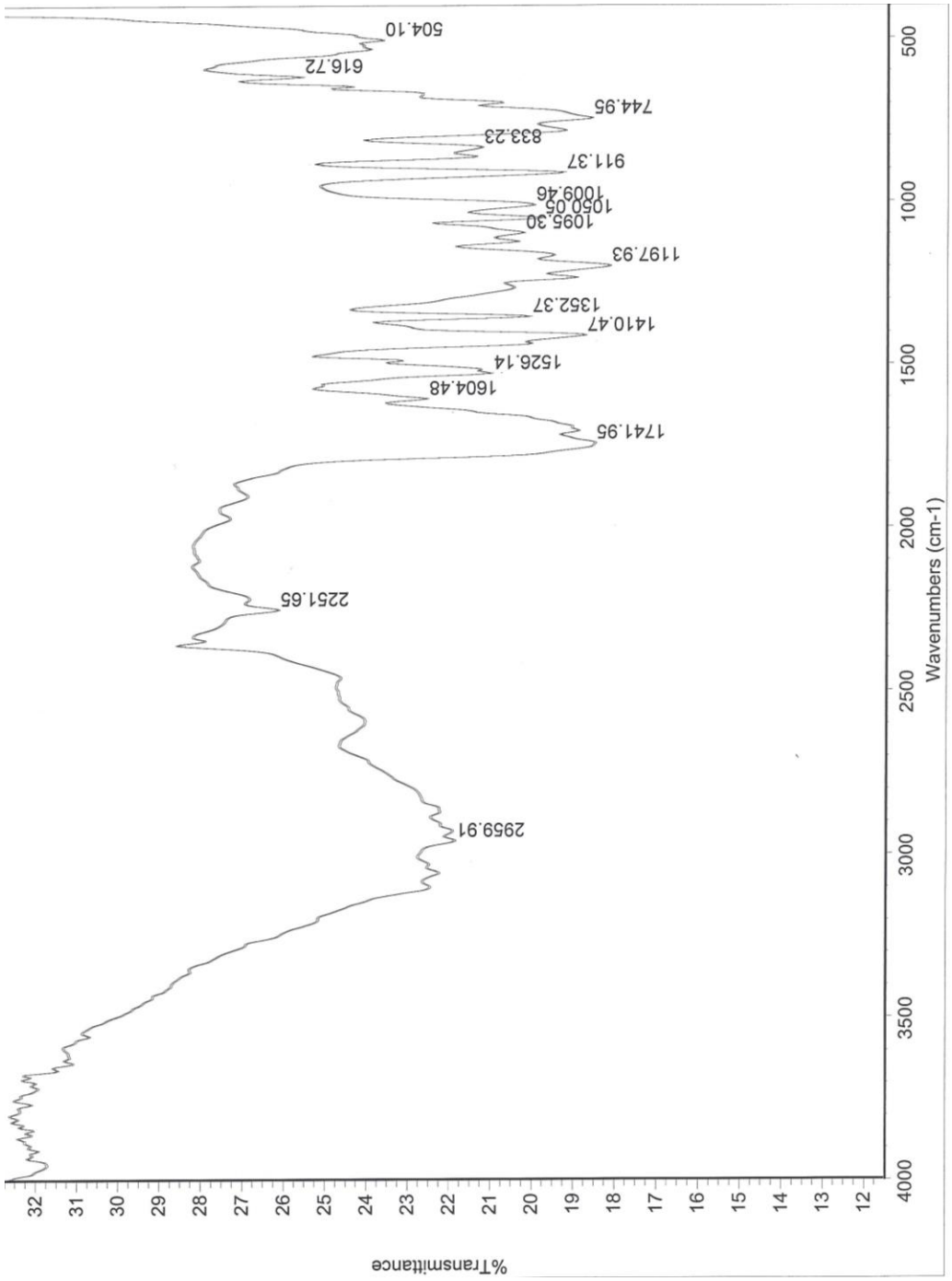

Figure 105: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)thiophene-2-carboxamide **36**

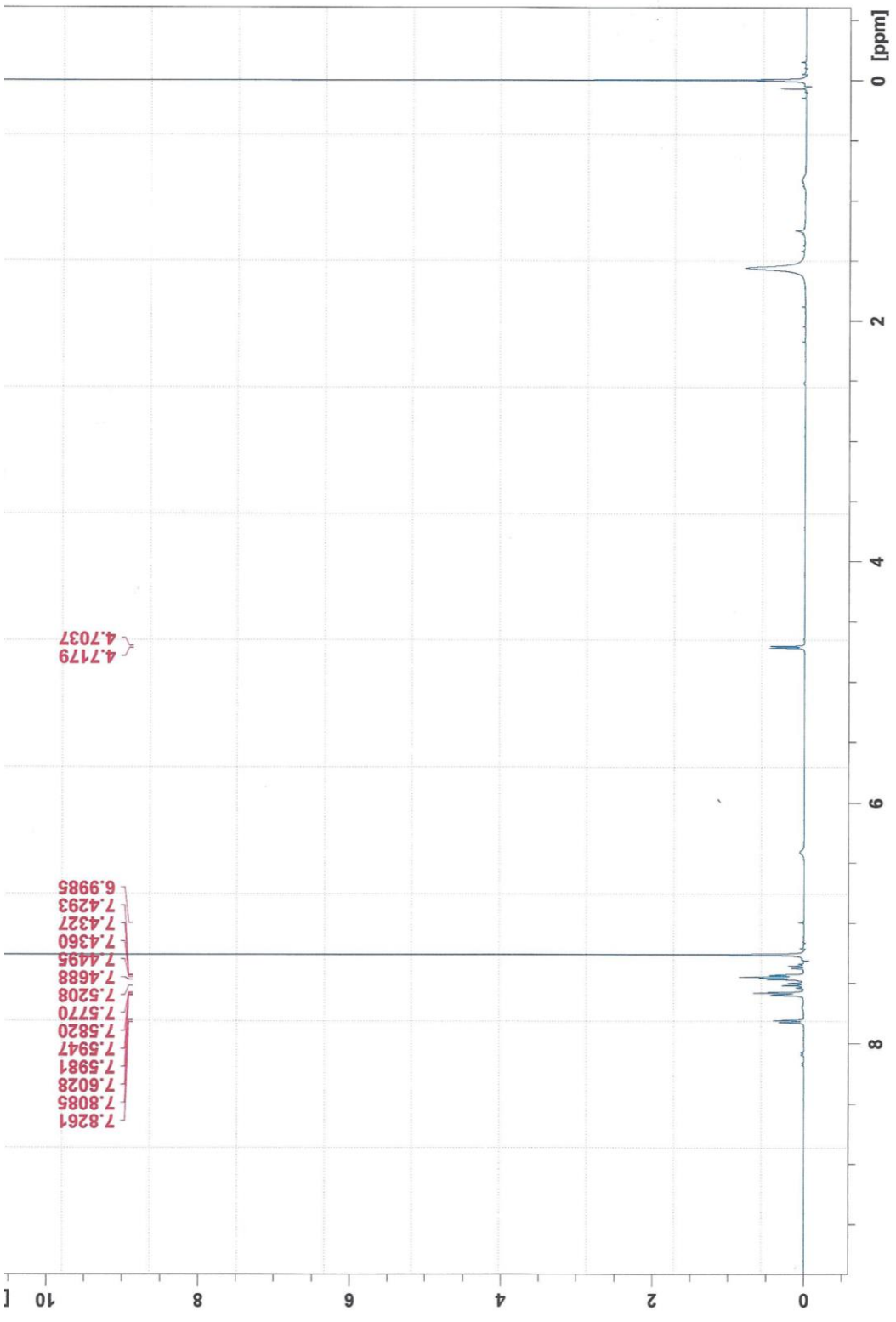

Figure 106: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)benzamide **37**

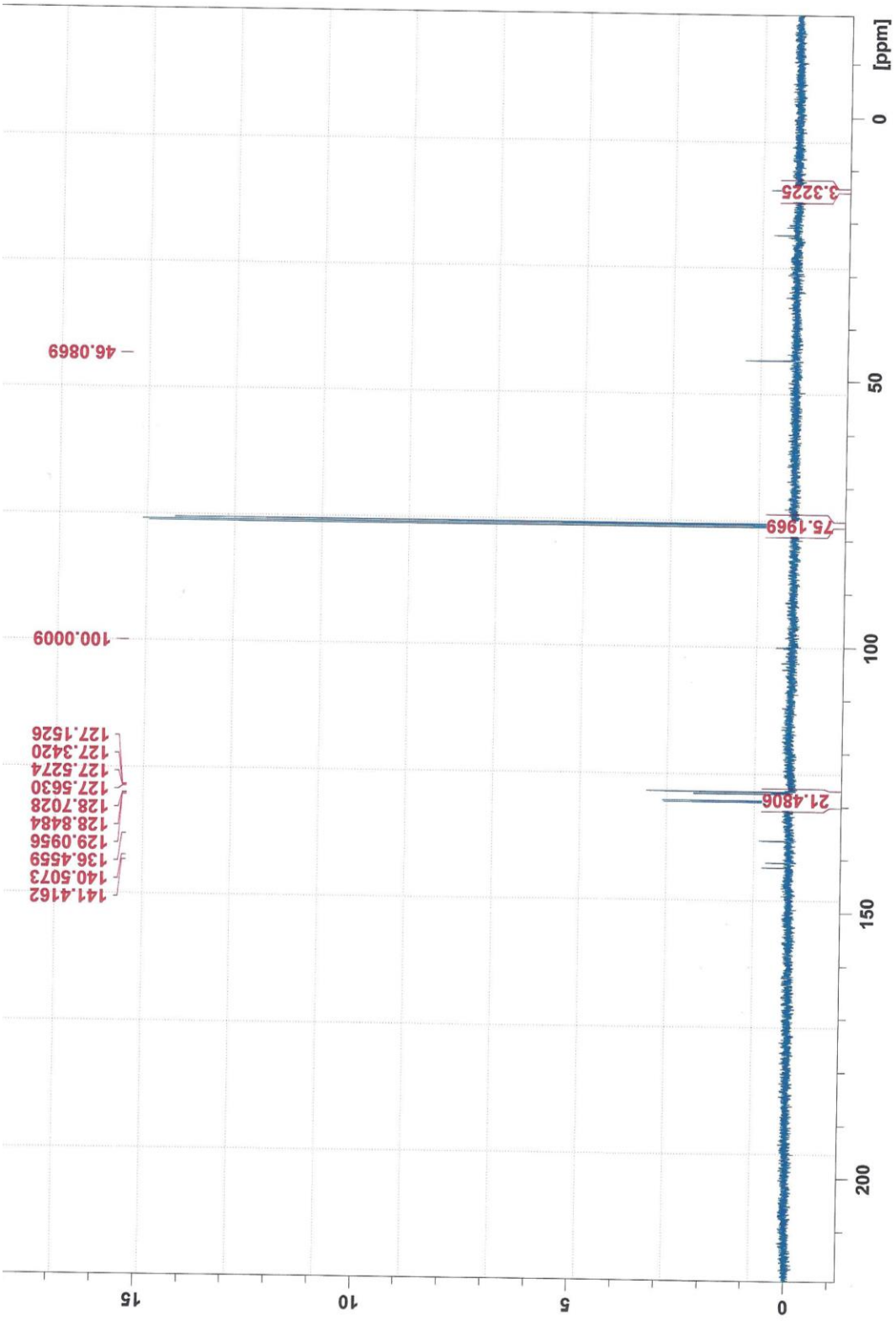

Figure 107: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)benzamide **37**

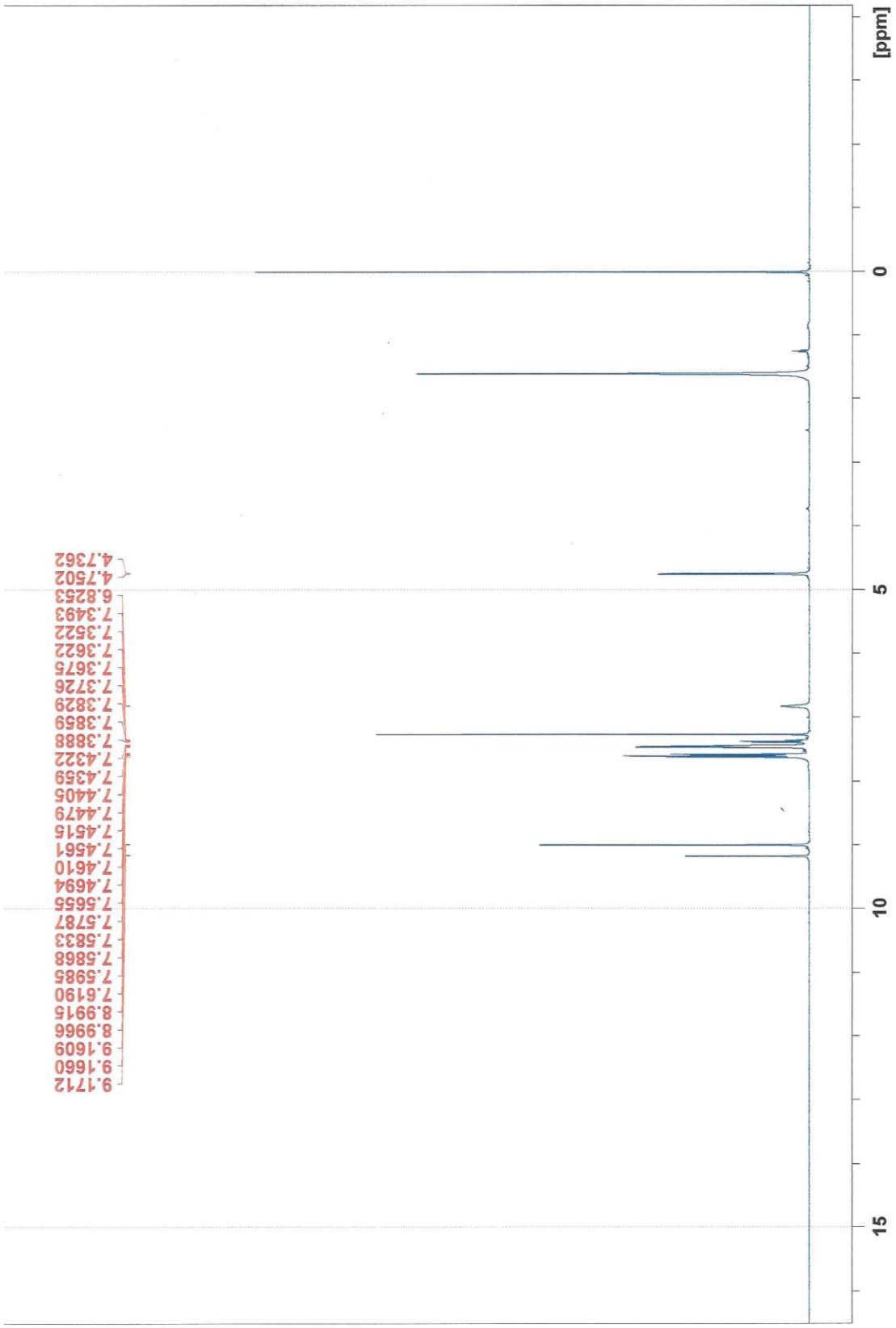

Figure 108: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3,5-dinitrobenzamide **38**

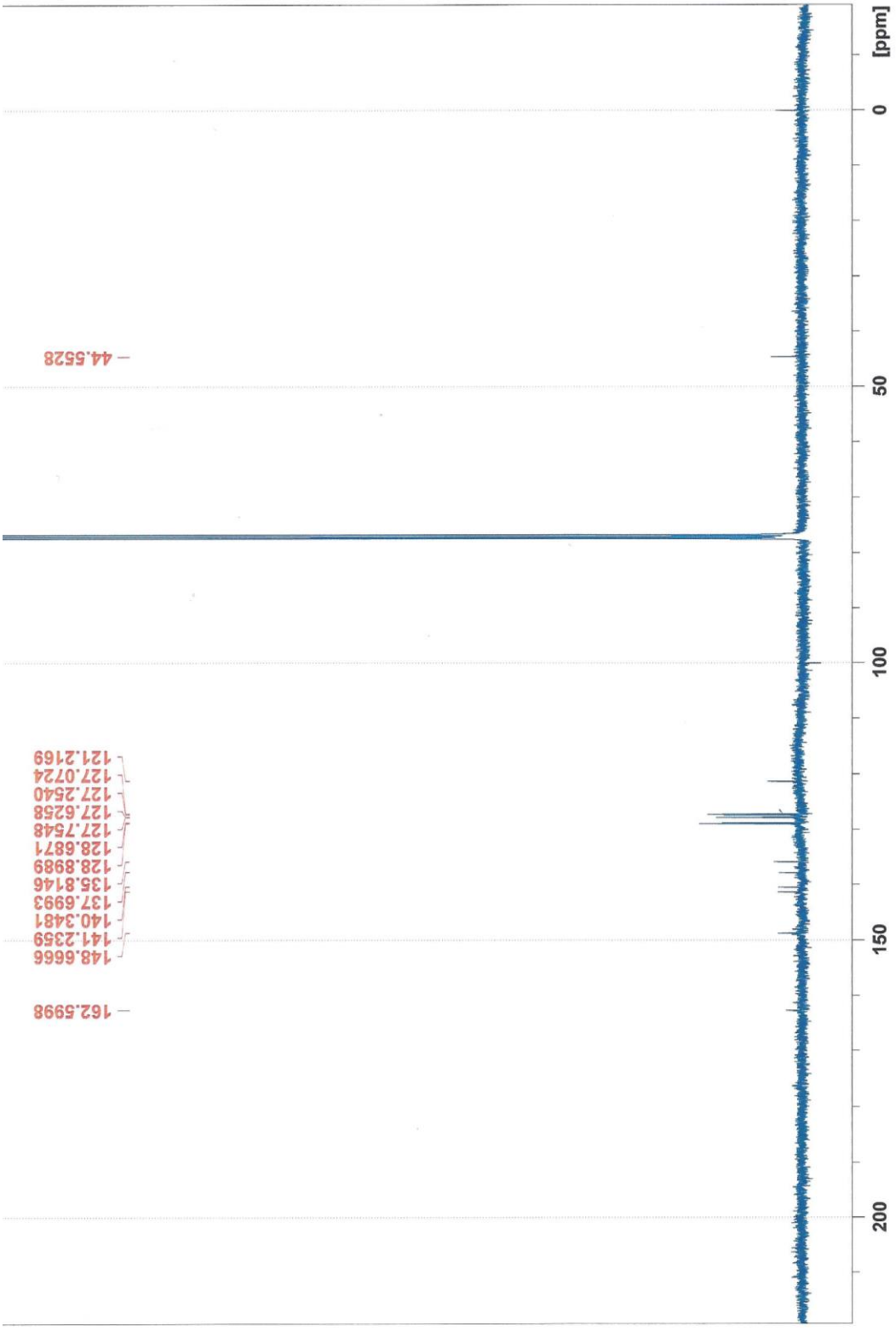

Figure 109: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3,5-dinitrobenzamide **38**

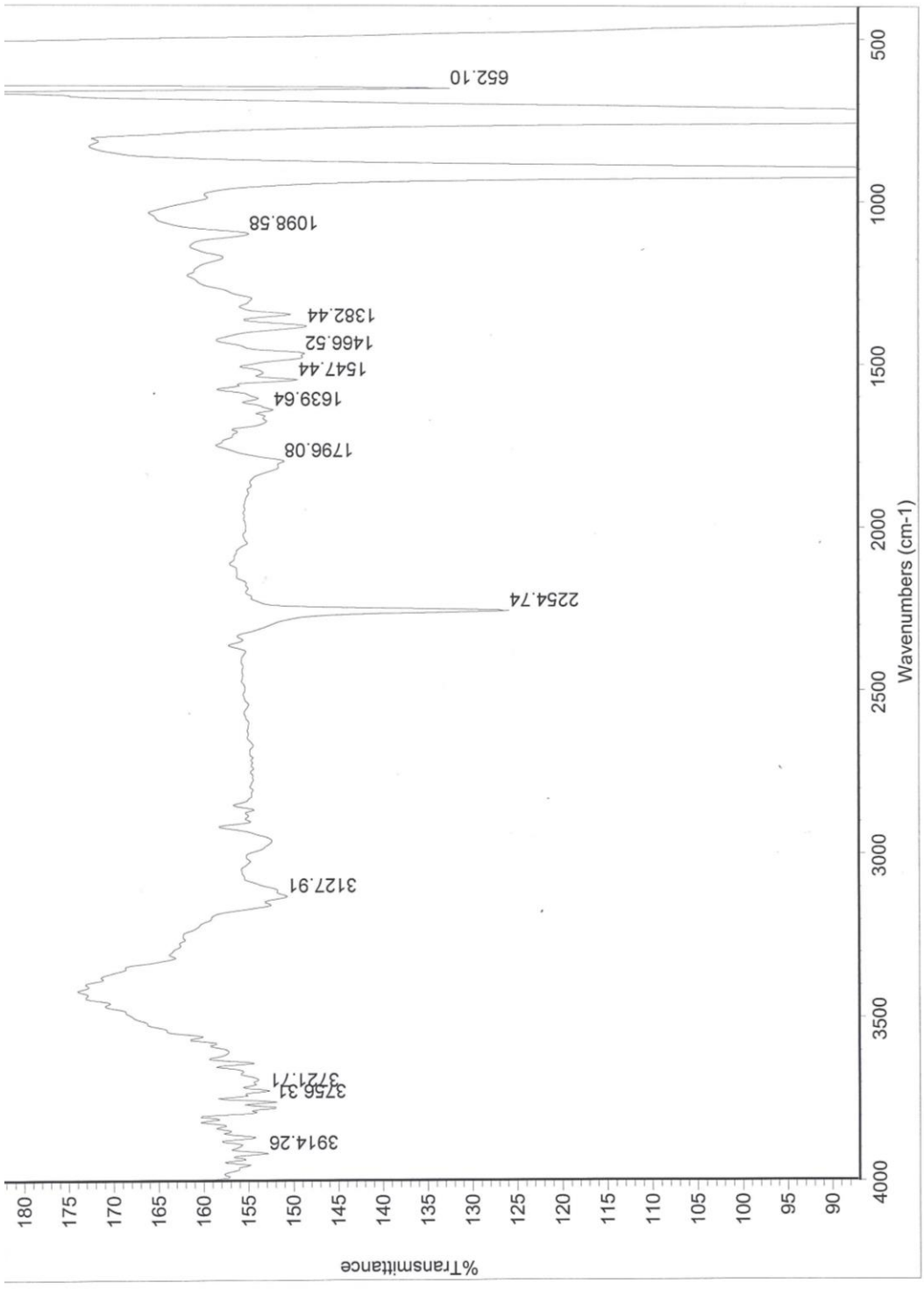

Figure 110: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3,5-dinitrobenzamide **38**

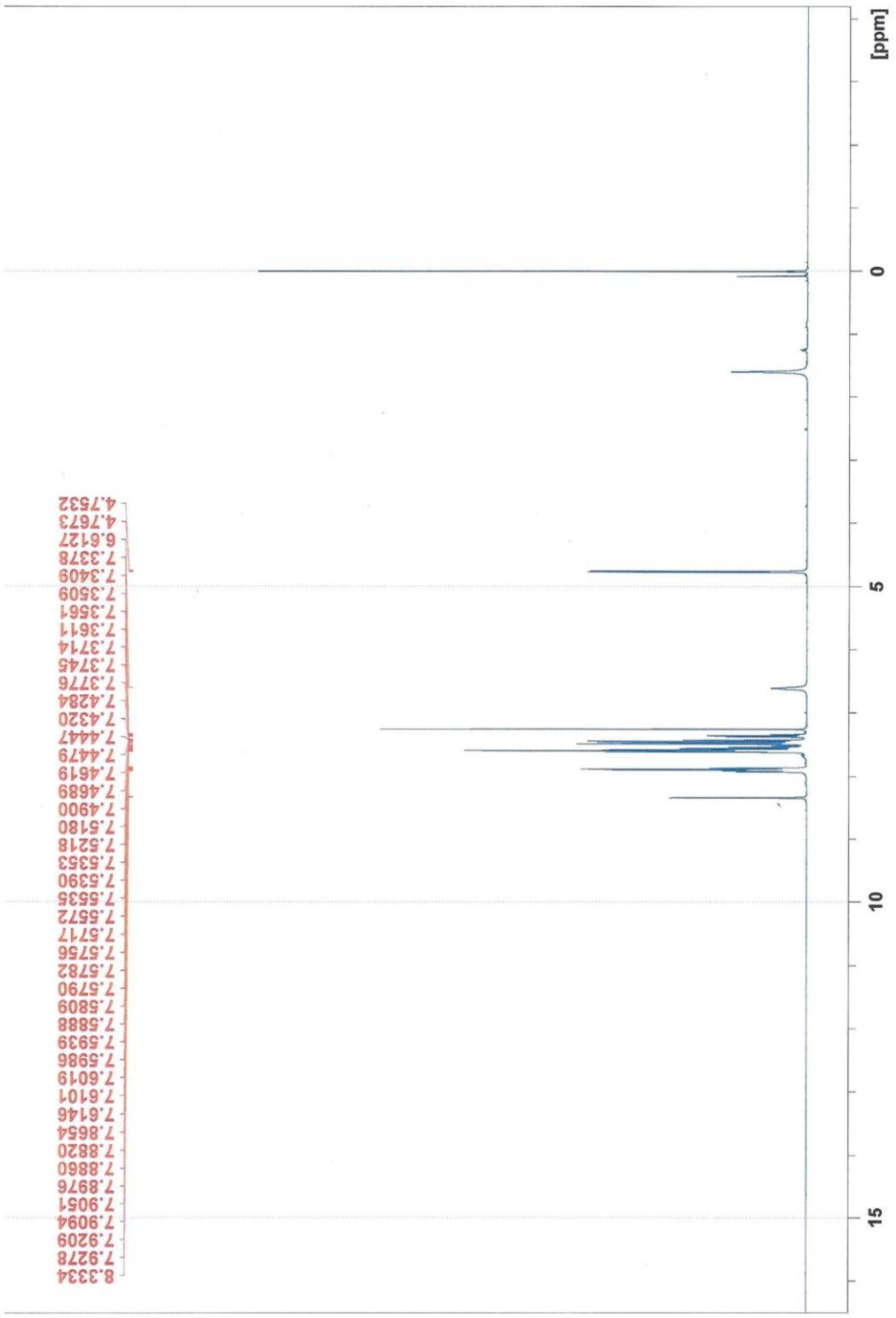

Figure 111: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-naphthamide **39**

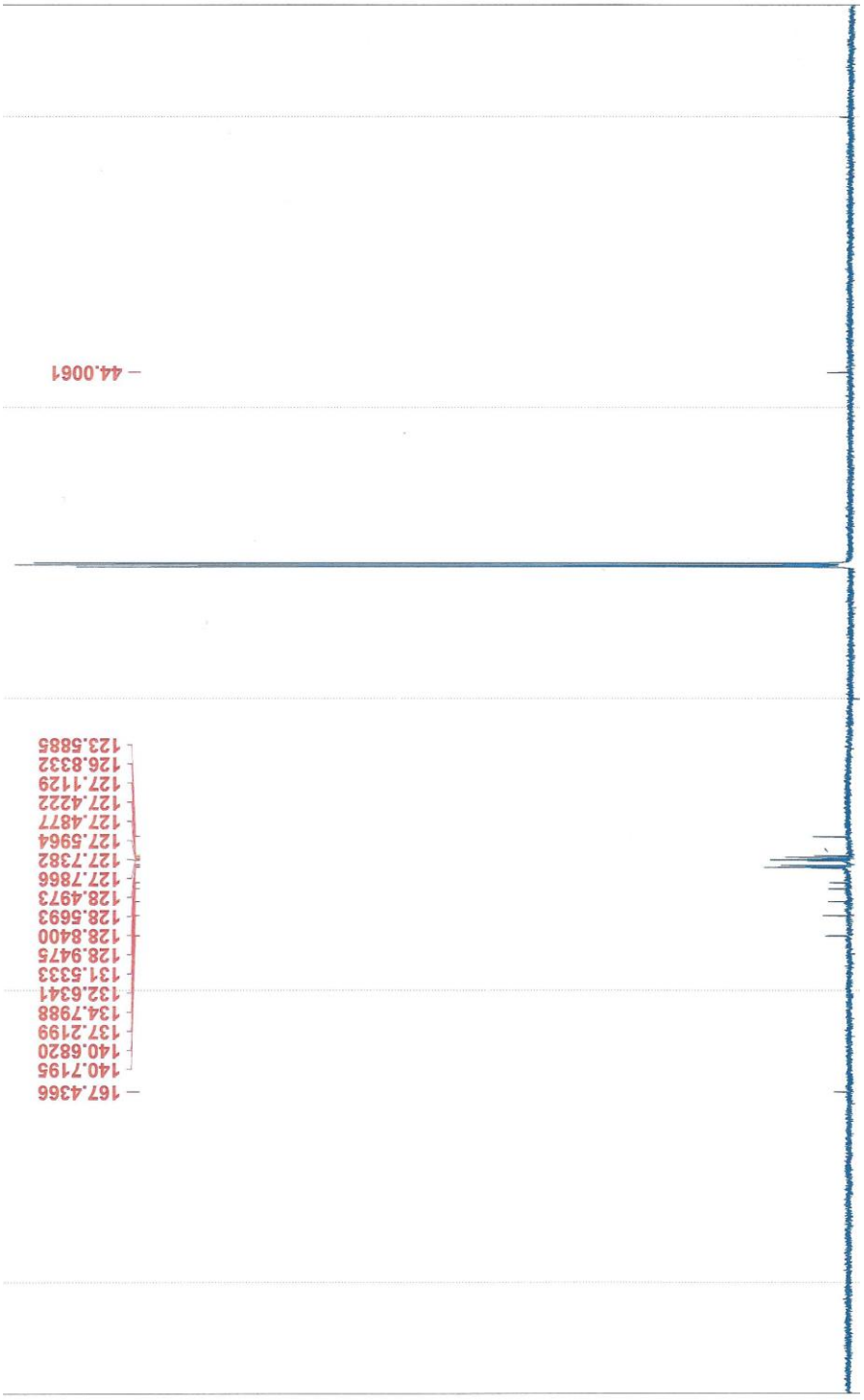

Figure 112: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-naphthamide **39**

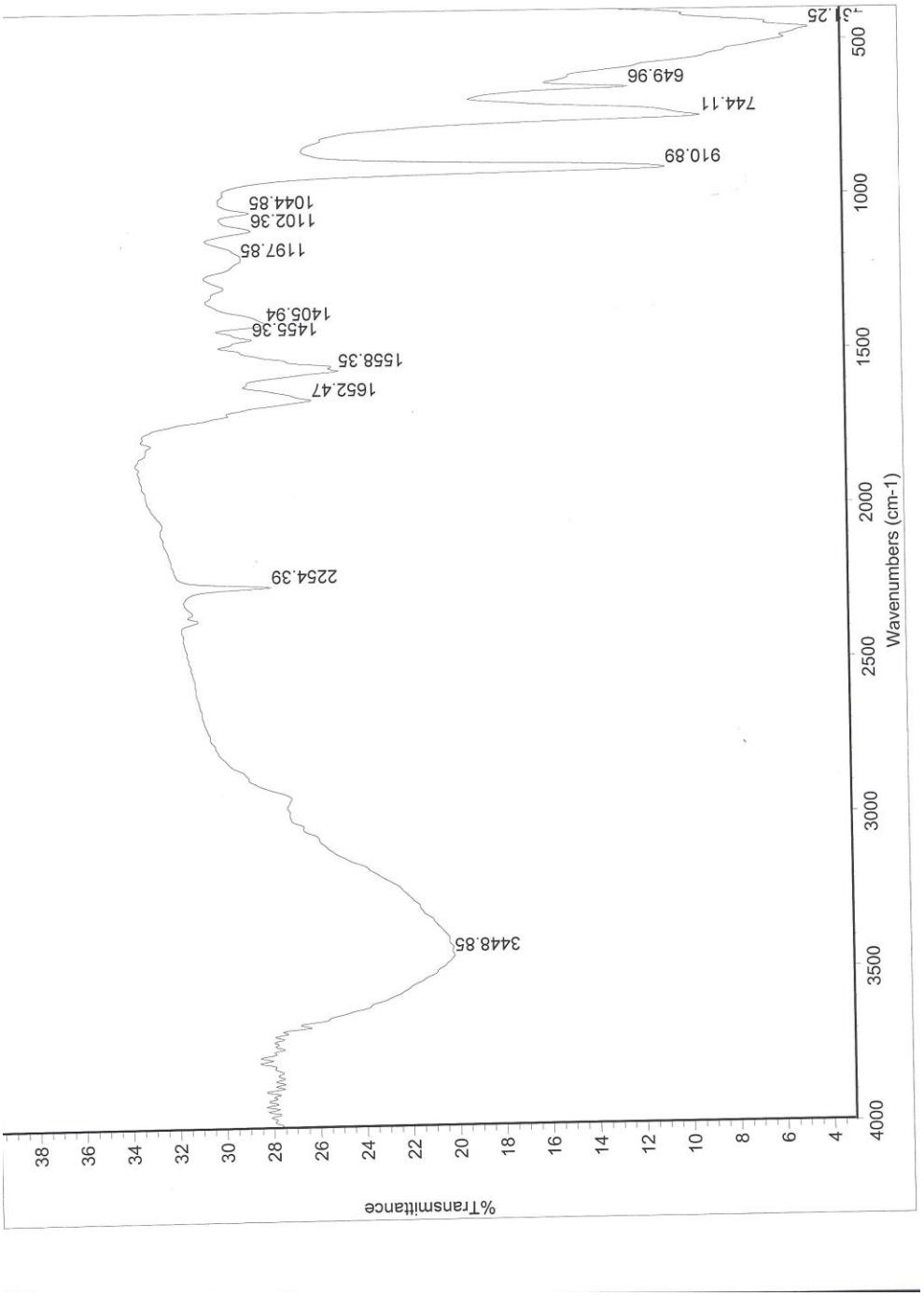

Figure 113: IR NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-naphthamide **39**

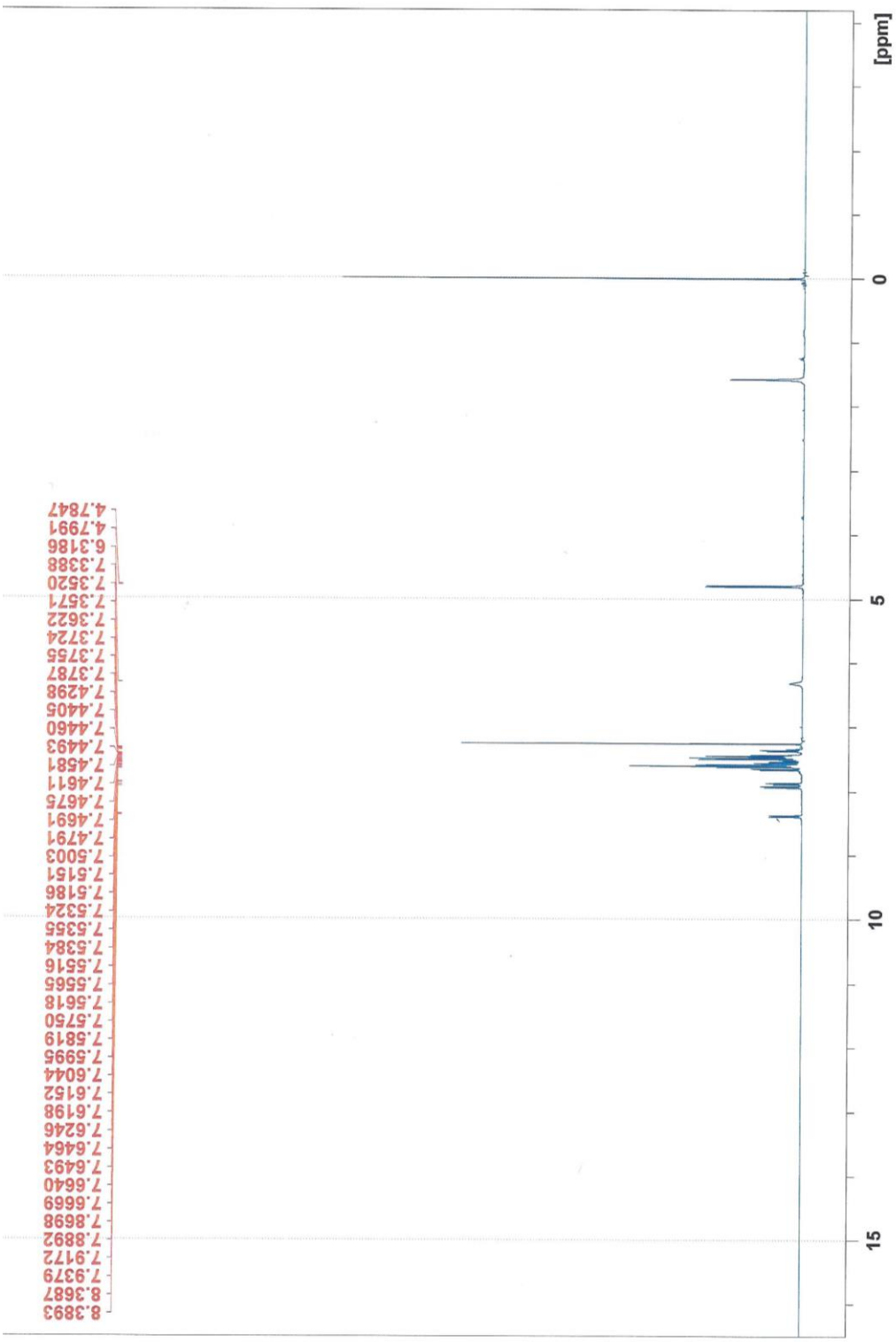

Figure 114: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1-naphthamide **40**

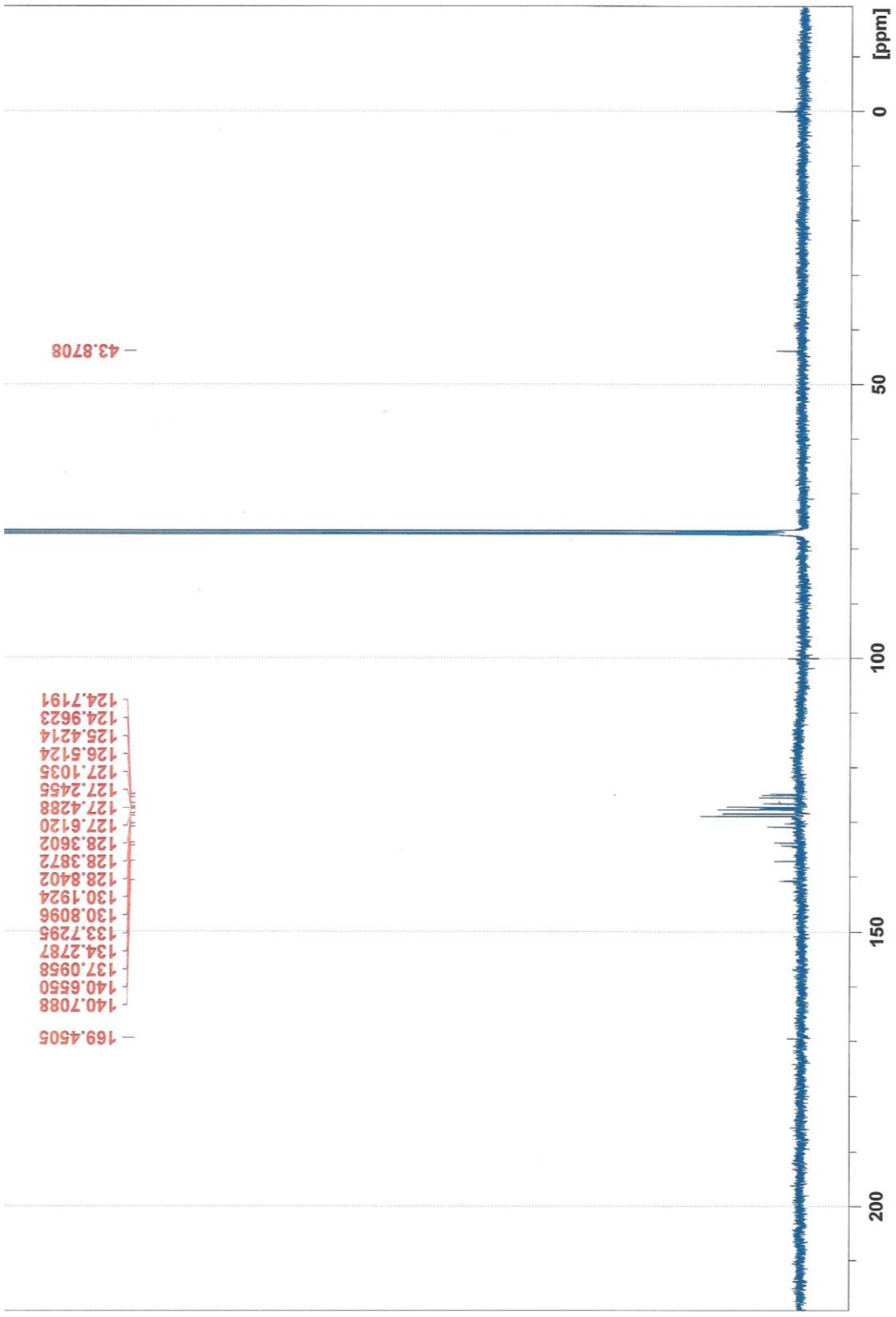

Figure 115: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1-naphthamide **40**

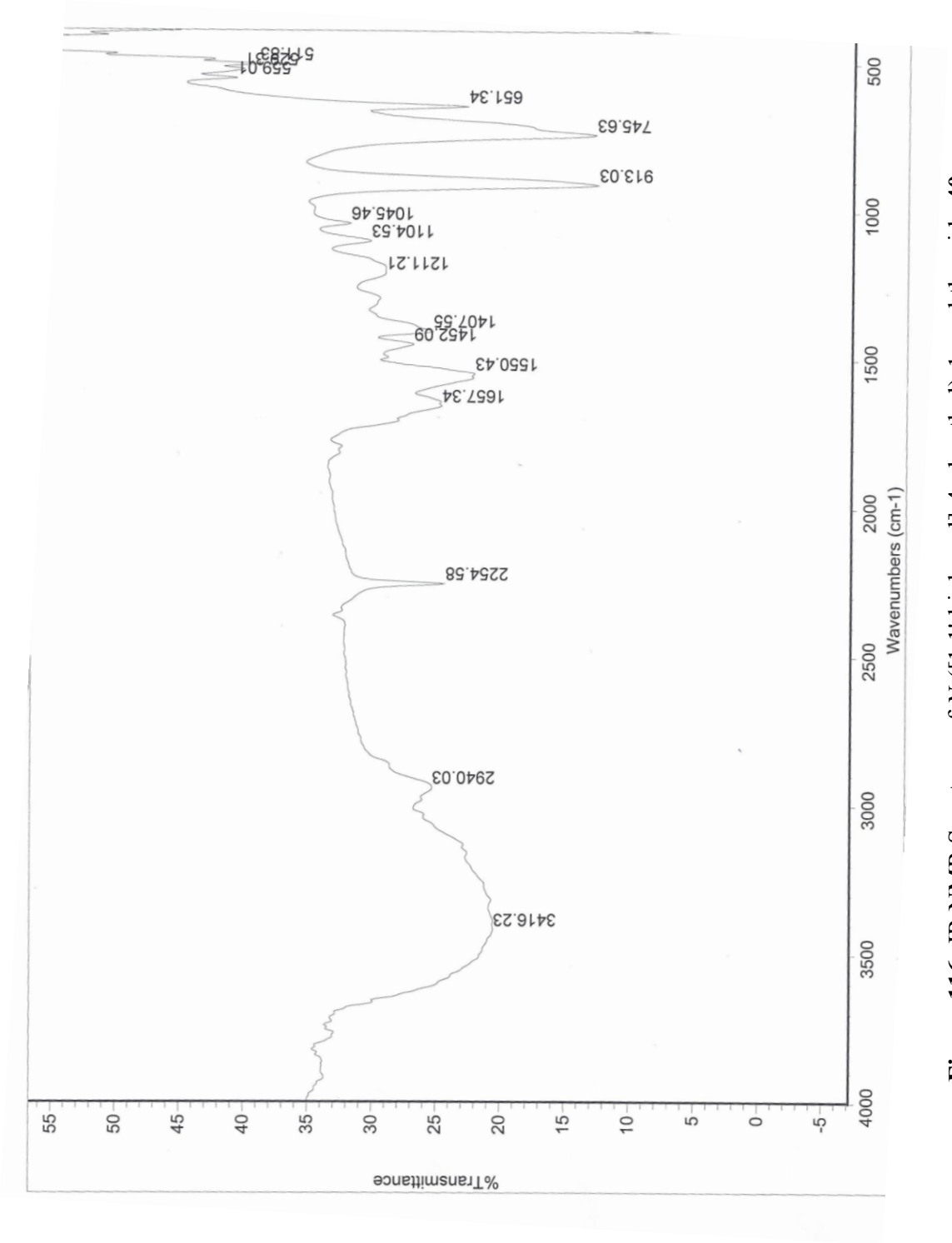

Figure 116: IR NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1-naphthamide **40**

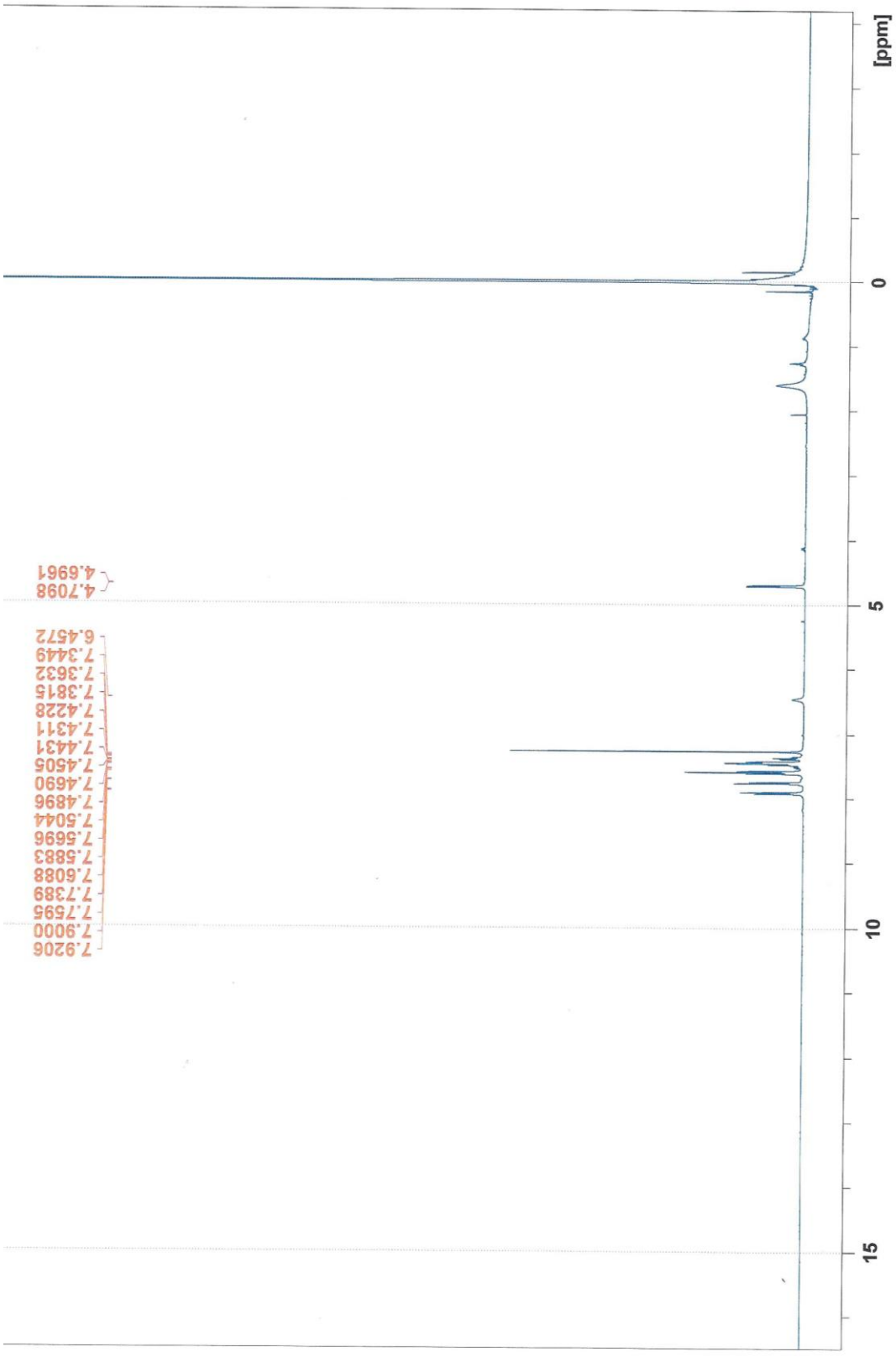

Figure 117: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-cyanobenzamide **41**

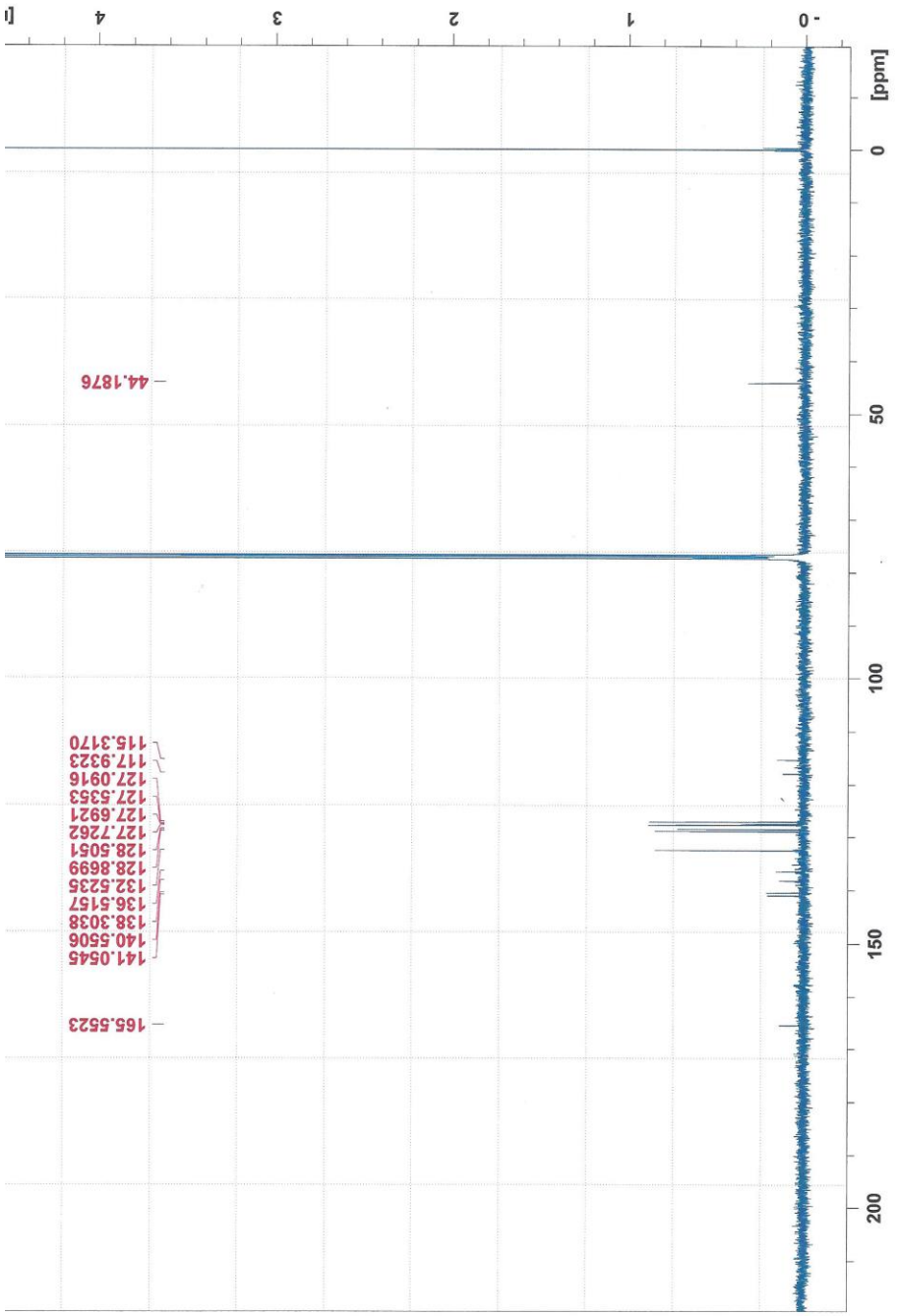

Figure 118: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-cyanobenzamide **41**

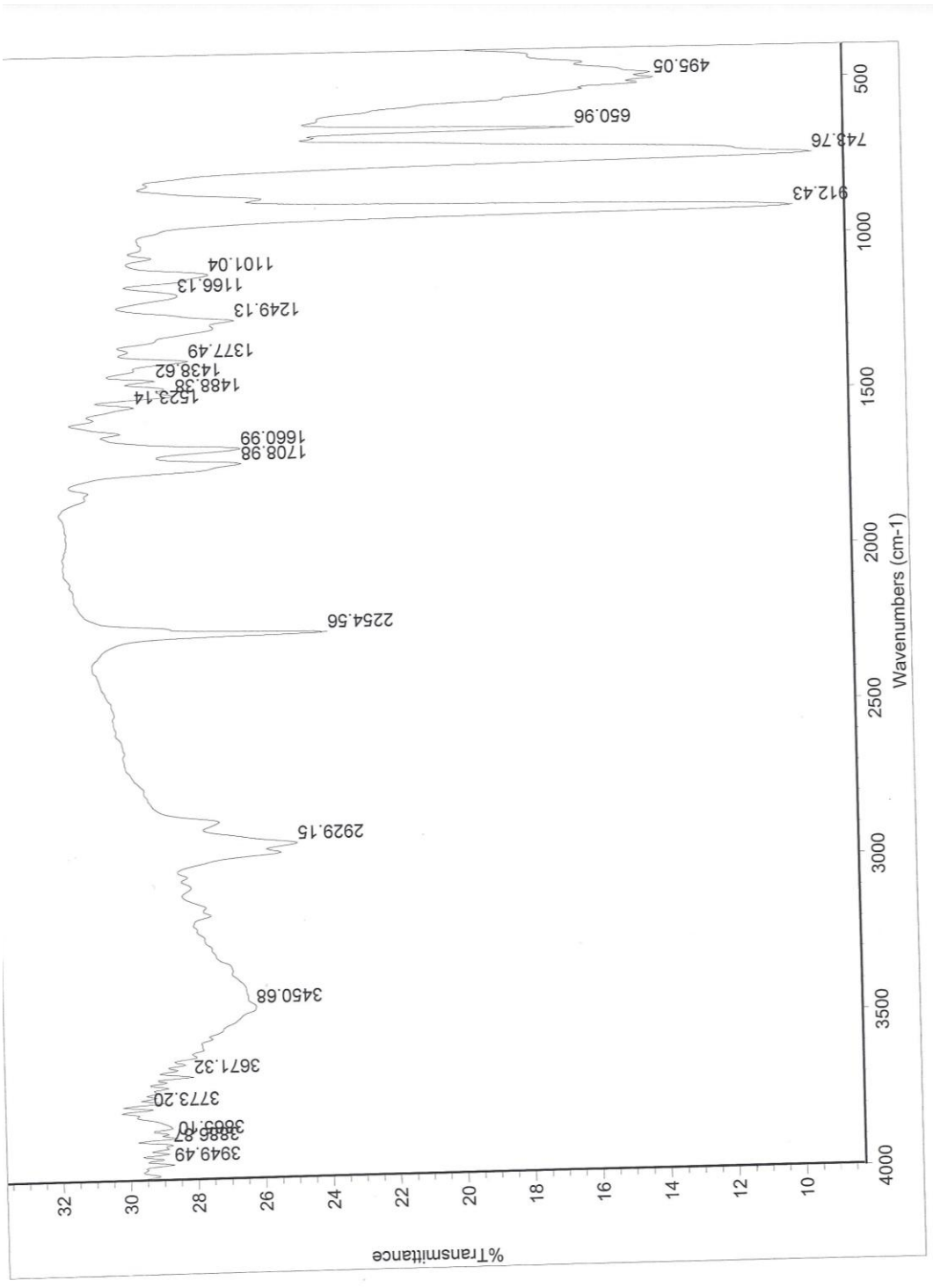

Figure 119: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-cyanobenzamide **41**

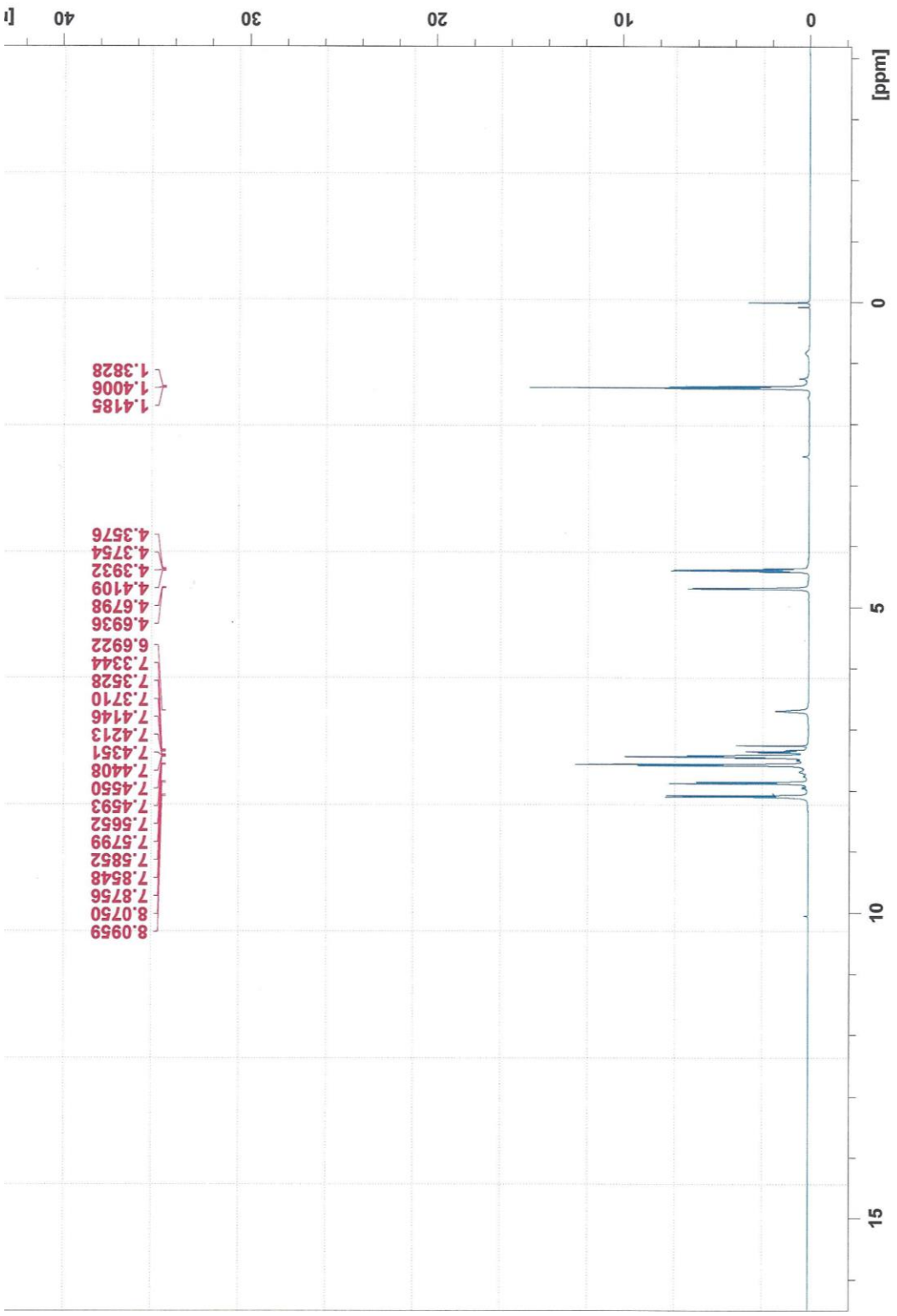

Figure 120: ^{1}H NMR Spectrum of ethyl 4-(([1,1'-biphenyl]-4-ylmethyl)carbamoyl)benzoate **42**

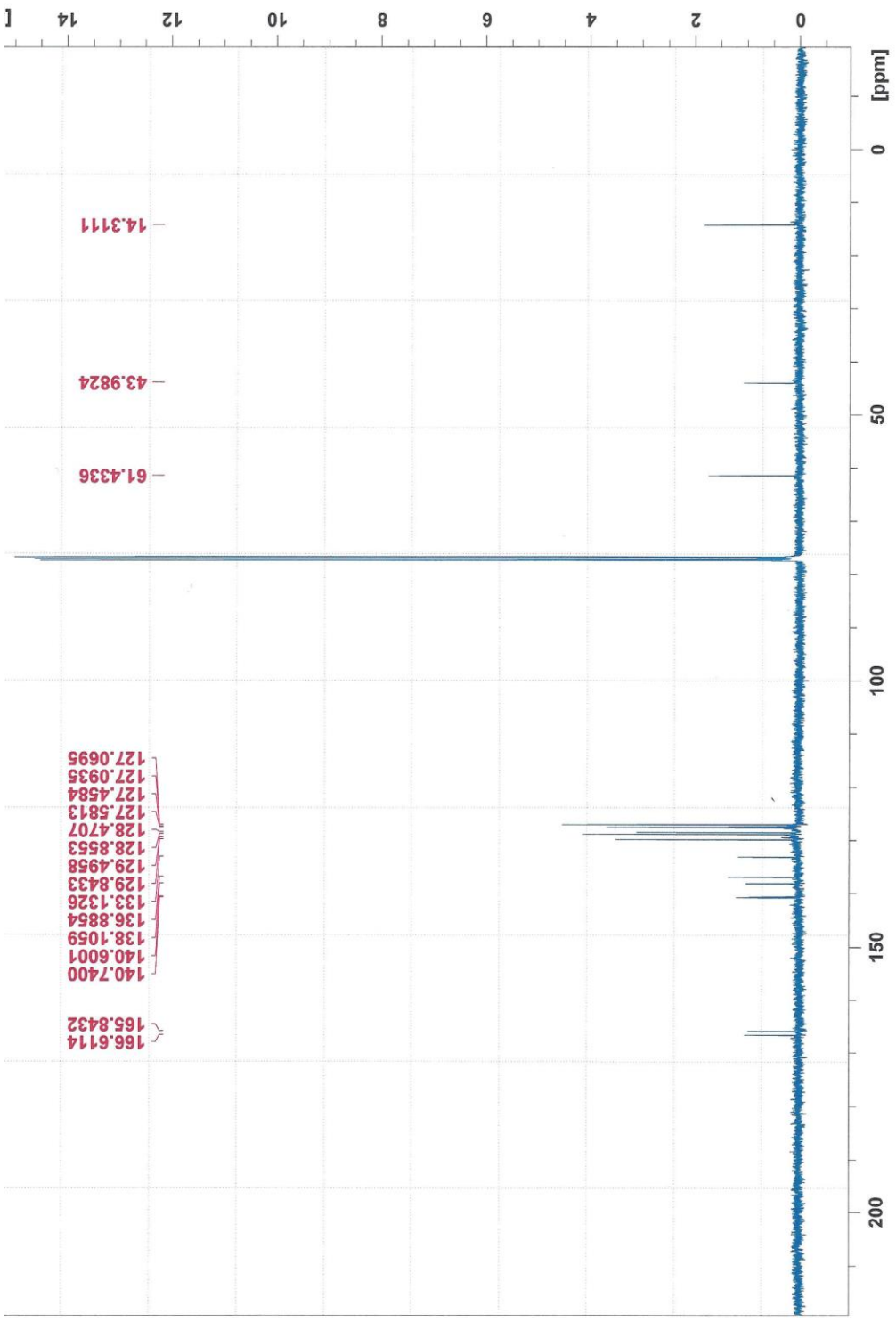

Figure 121: ^{13}C NMR Spectrum of ethyl 4-(([1,1'-biphenyl]-4-ylmethyl)carbamoyl)benzoate **42**

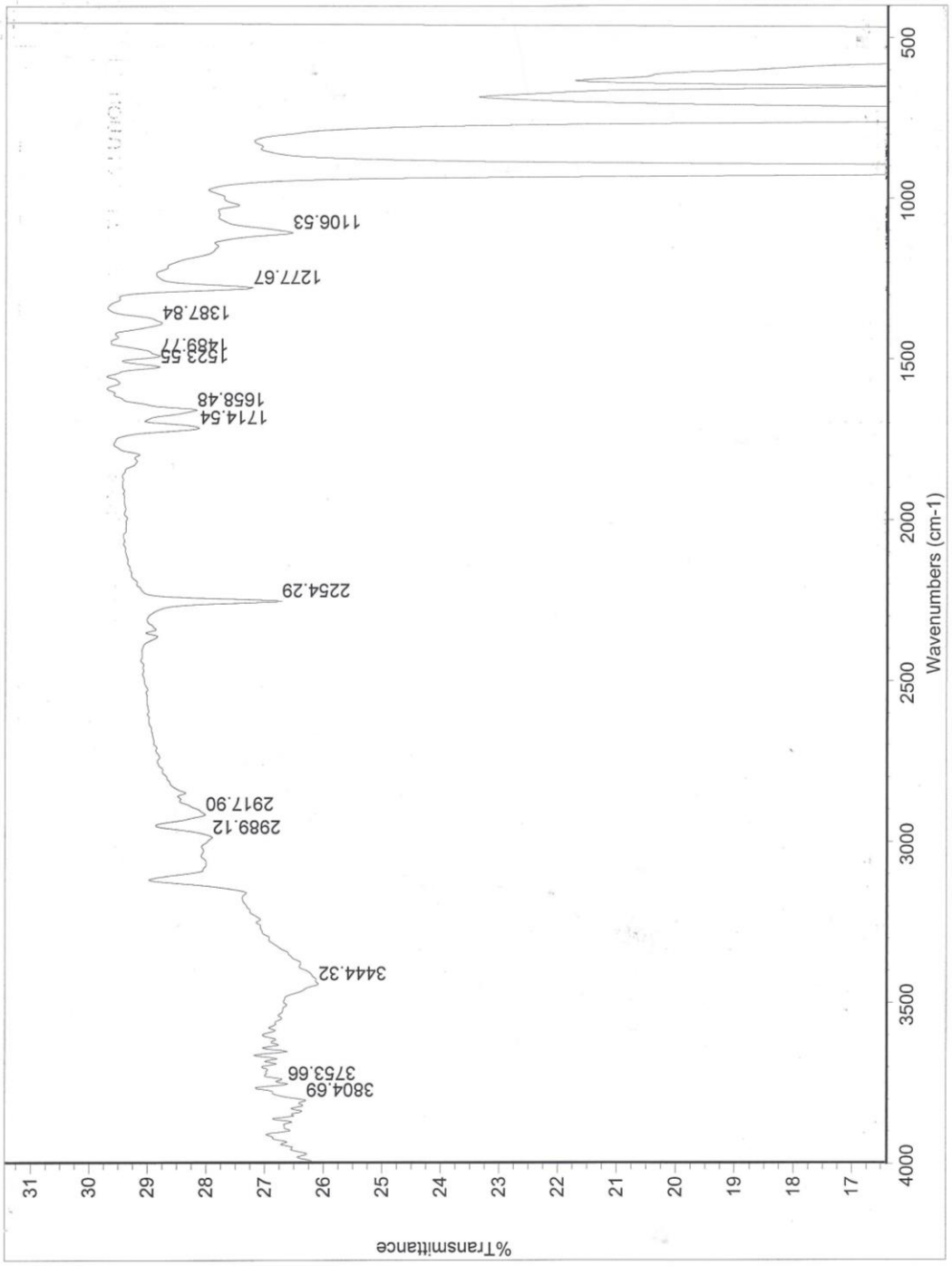

Figure 122: IR Spectrum of ethyl 4-(([1,1'-biphenyl]-4-ylmethyl)carbamoyl)benzoate **42**

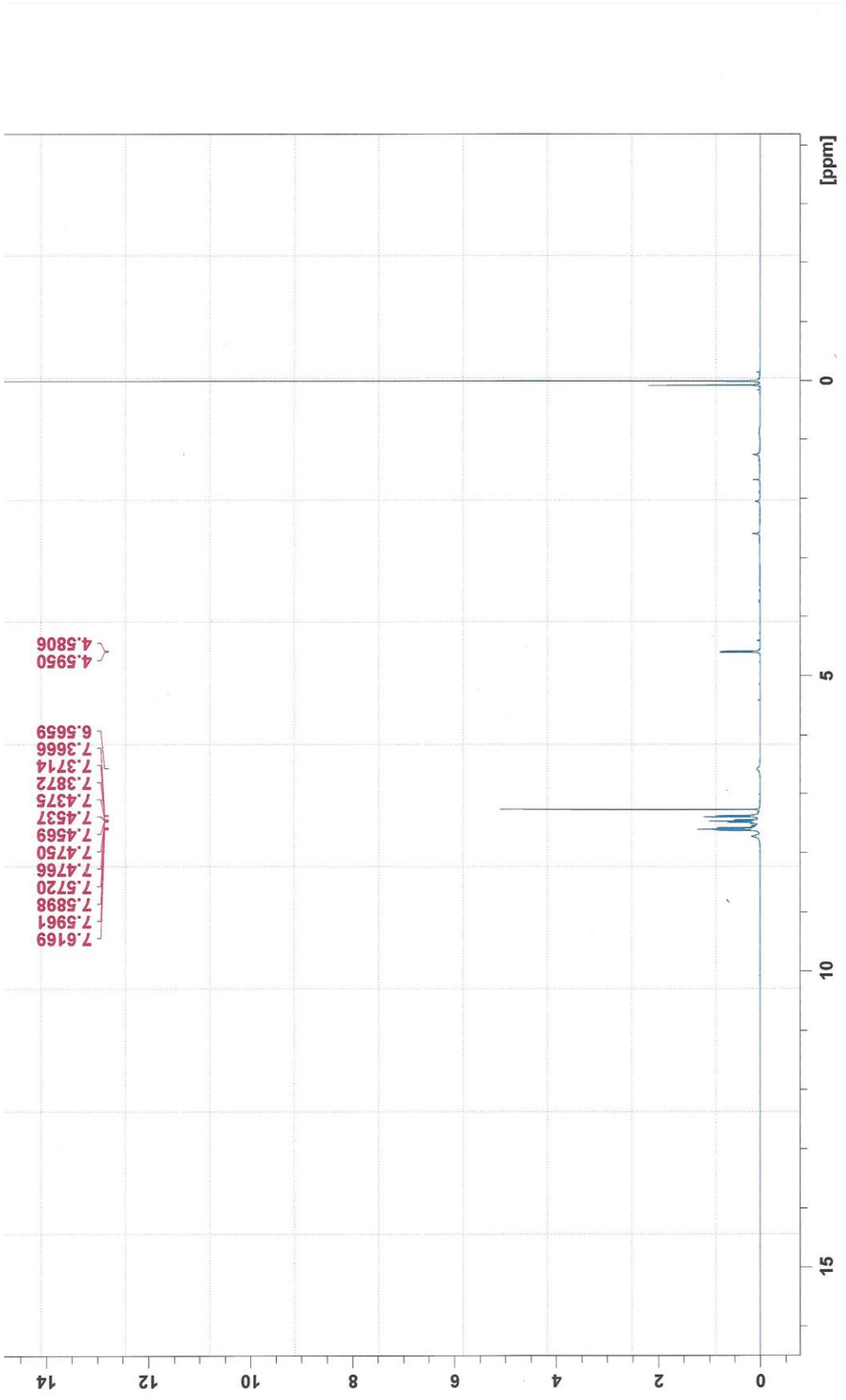

Figure 123: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2,2-trifluoroacetamide **43**

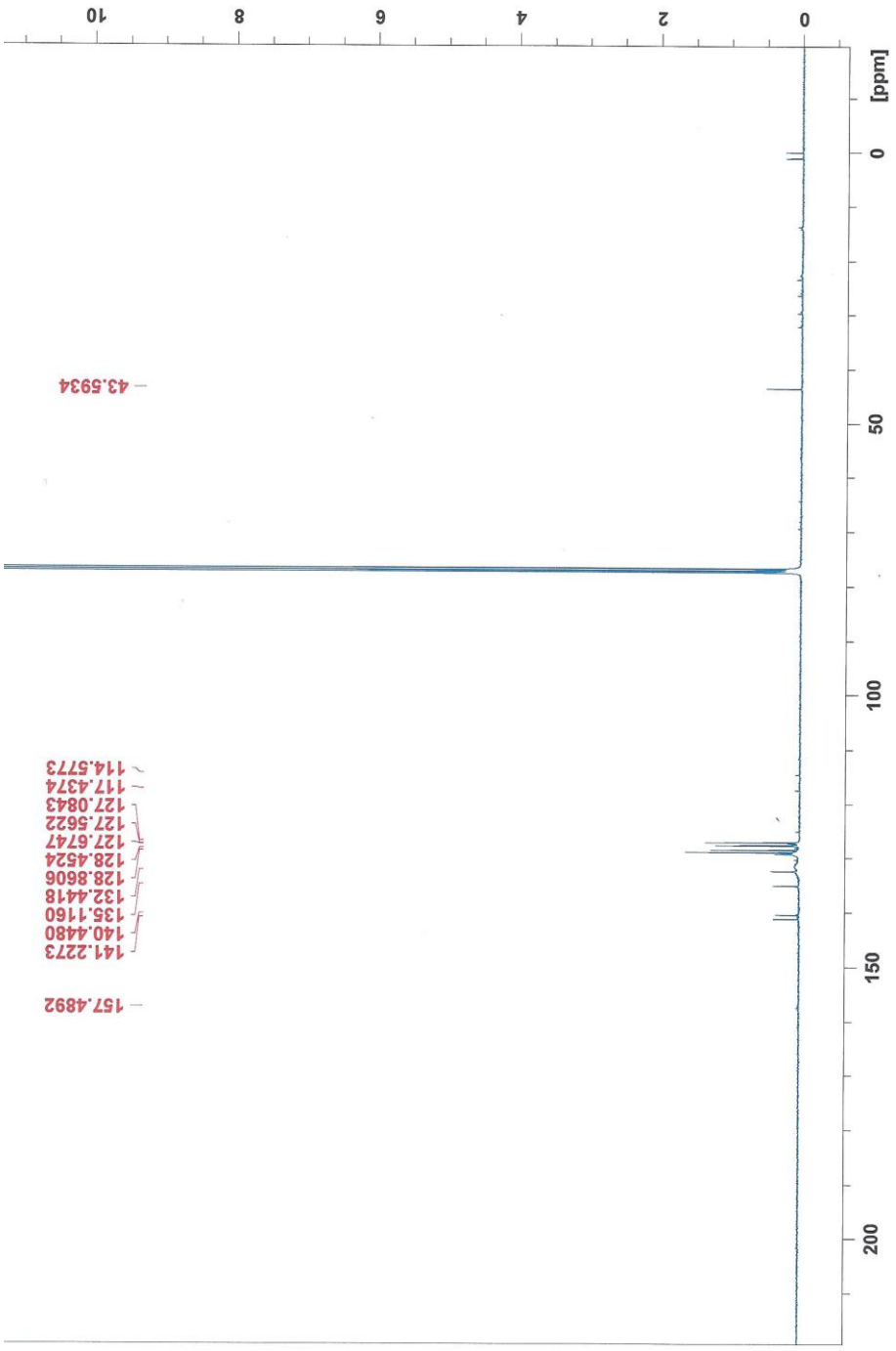

Figure 124: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2,2-trifluoroacetamide **43**

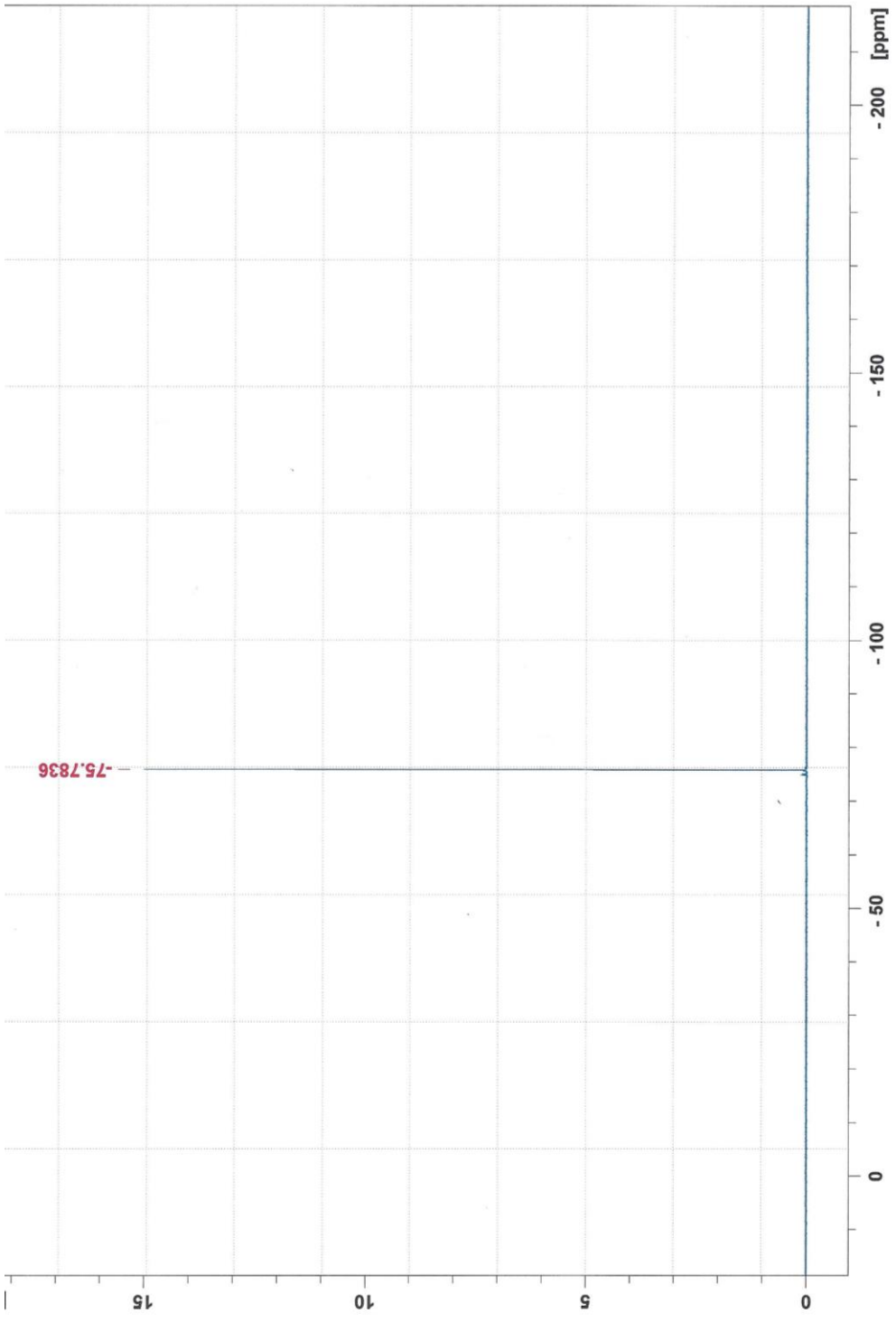

Figure 125: ^{19}F NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2,2-trifluoroacetamide **43**

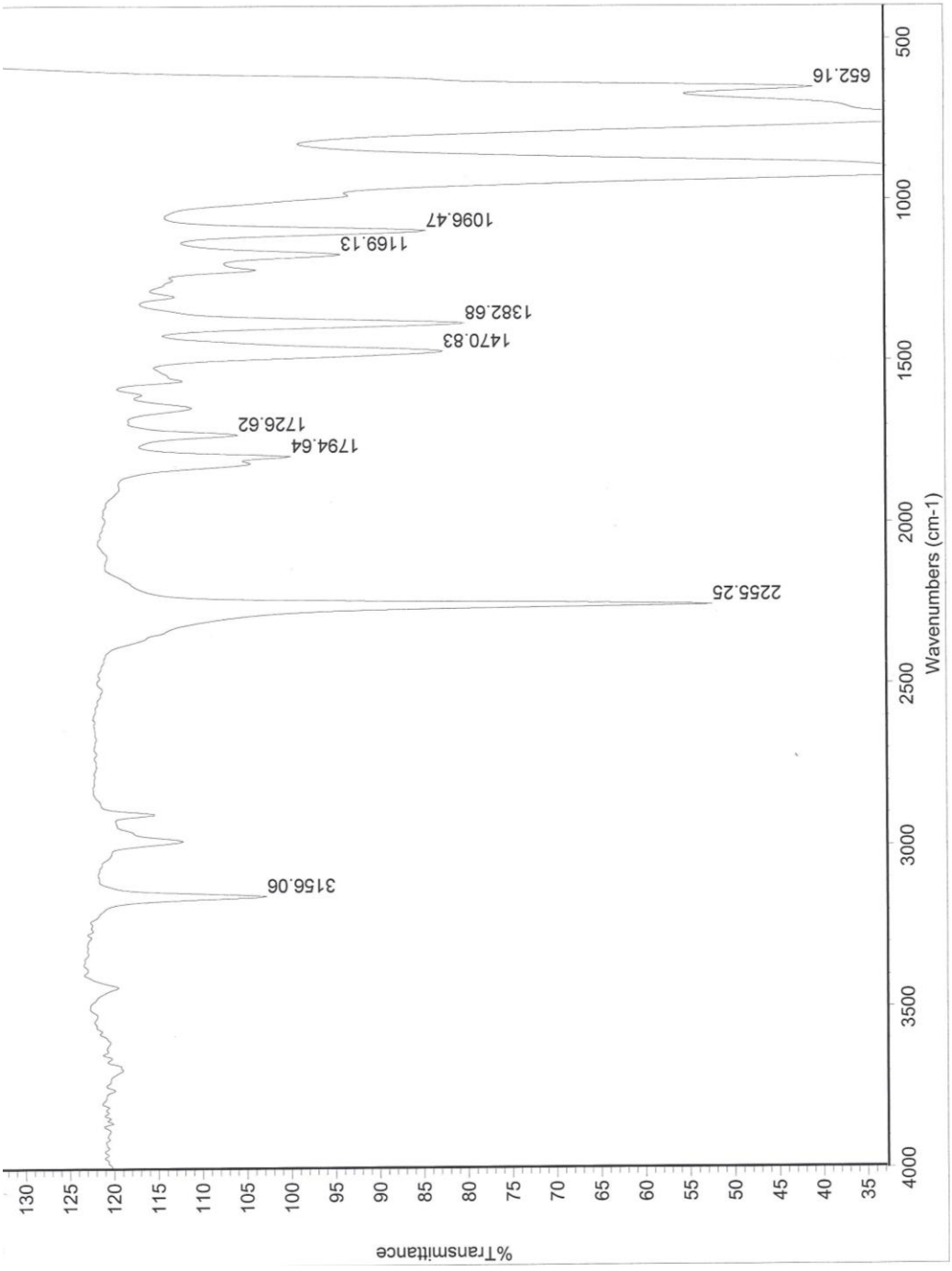

Figure 126: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2,2-trifluoroacetamide **43**

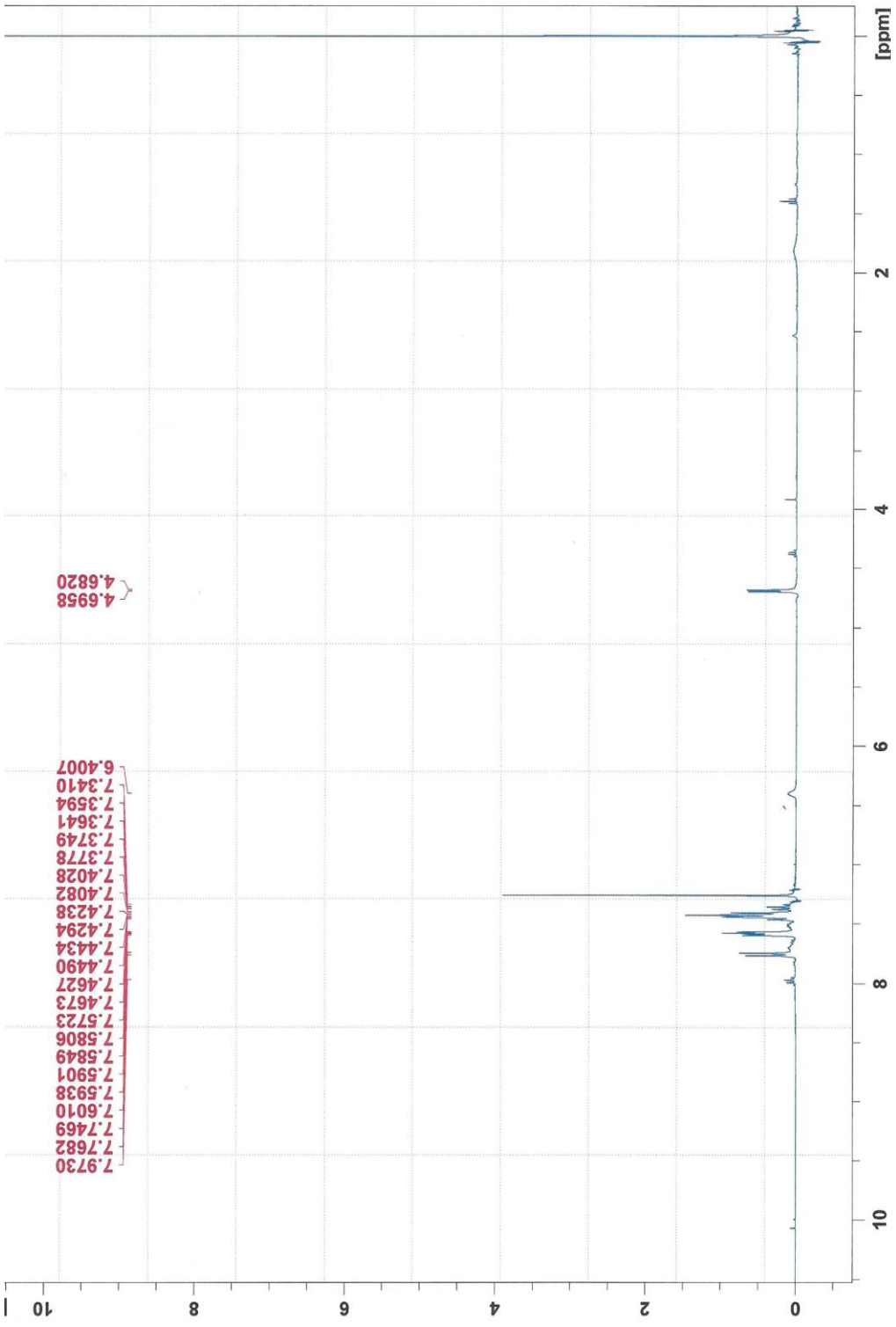

Figure 127: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-chlorobenzamide **44**

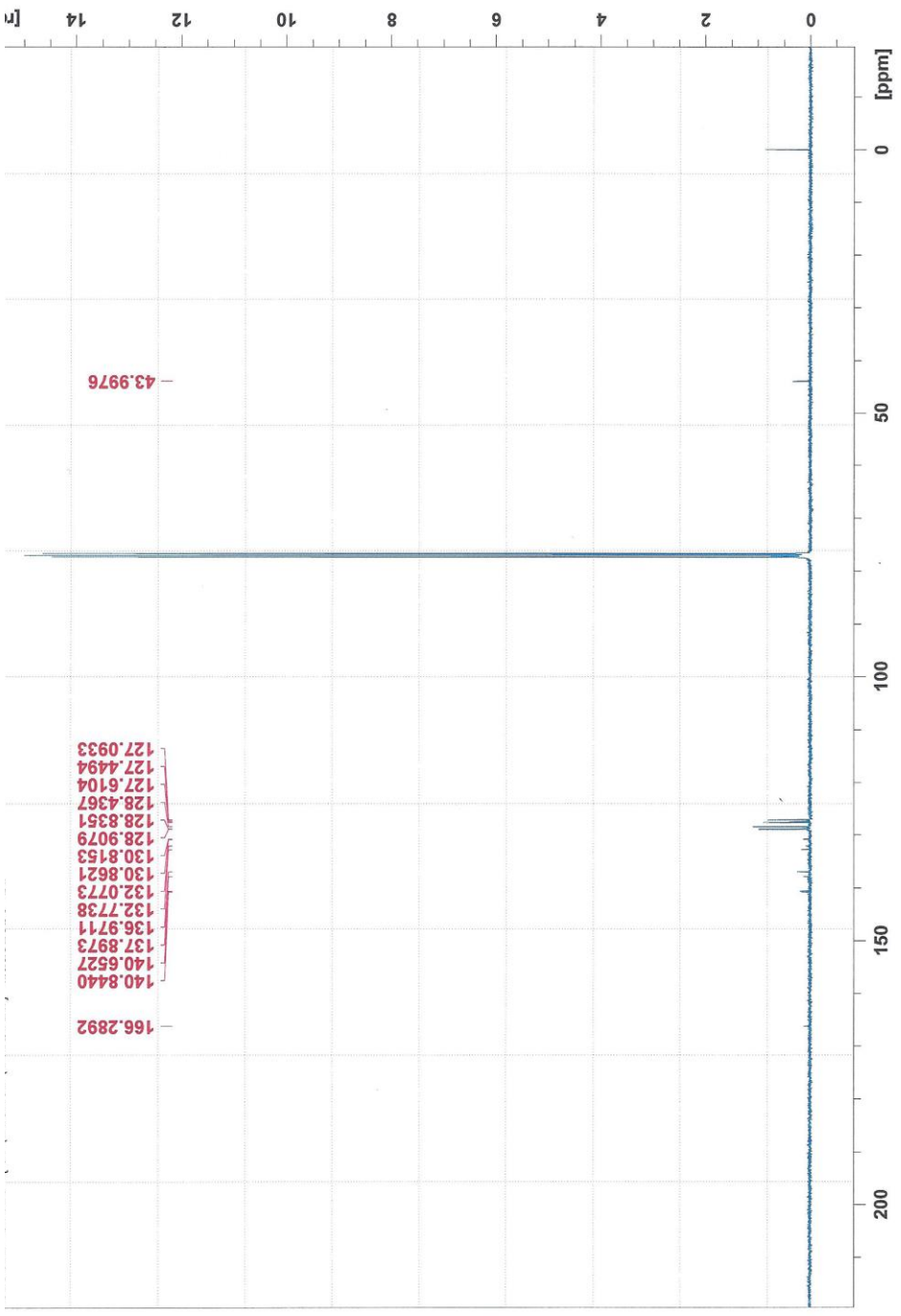

Figure 128: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-chlorobenzamide **44**

Figure 129: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-chlorobenzamide **44**

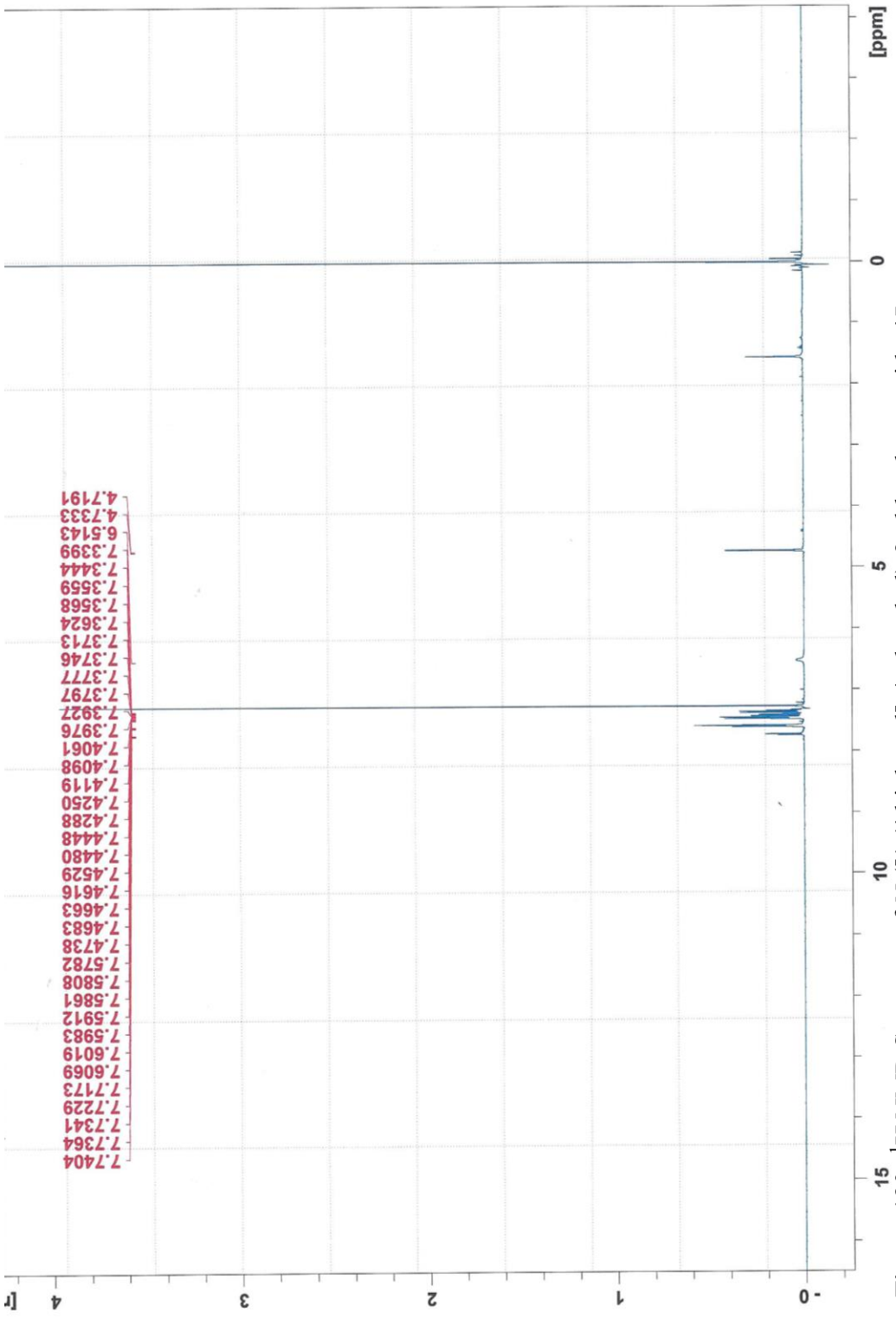

Figure 130: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-chlorobenzamide **45**

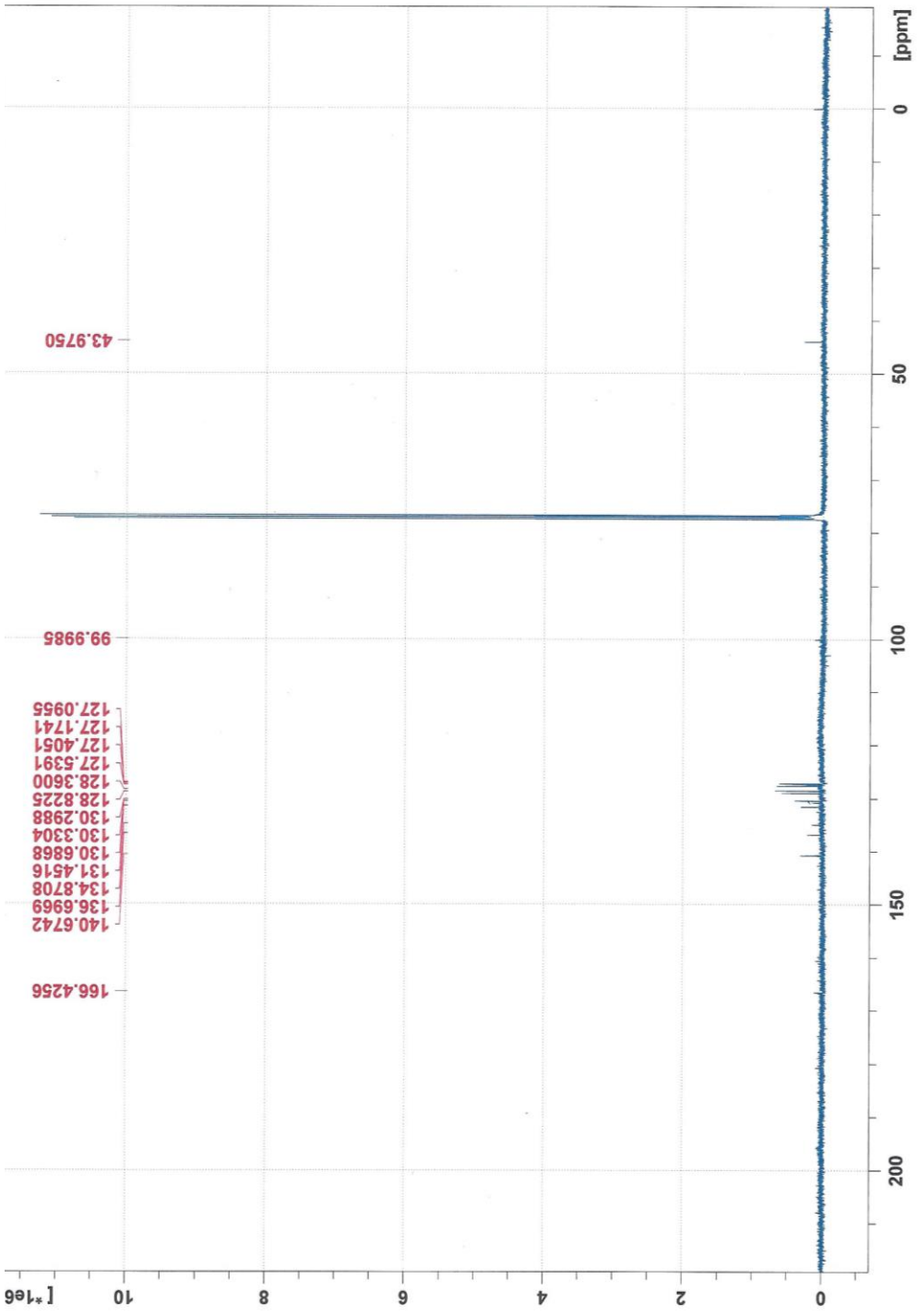

Figure 131: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-chlorobenzamide **45**

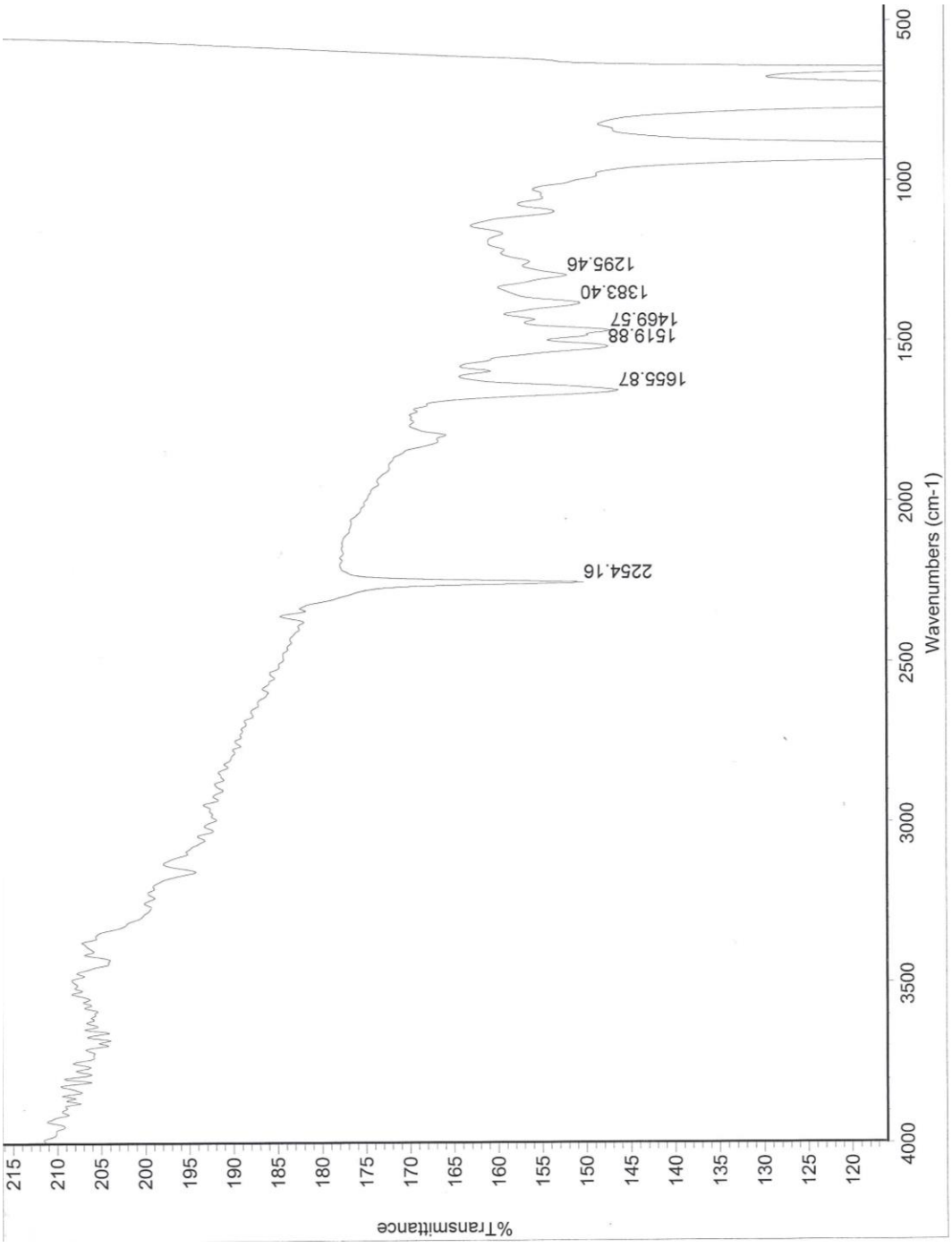

Figure 132: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2-chlorobenzamide **45**

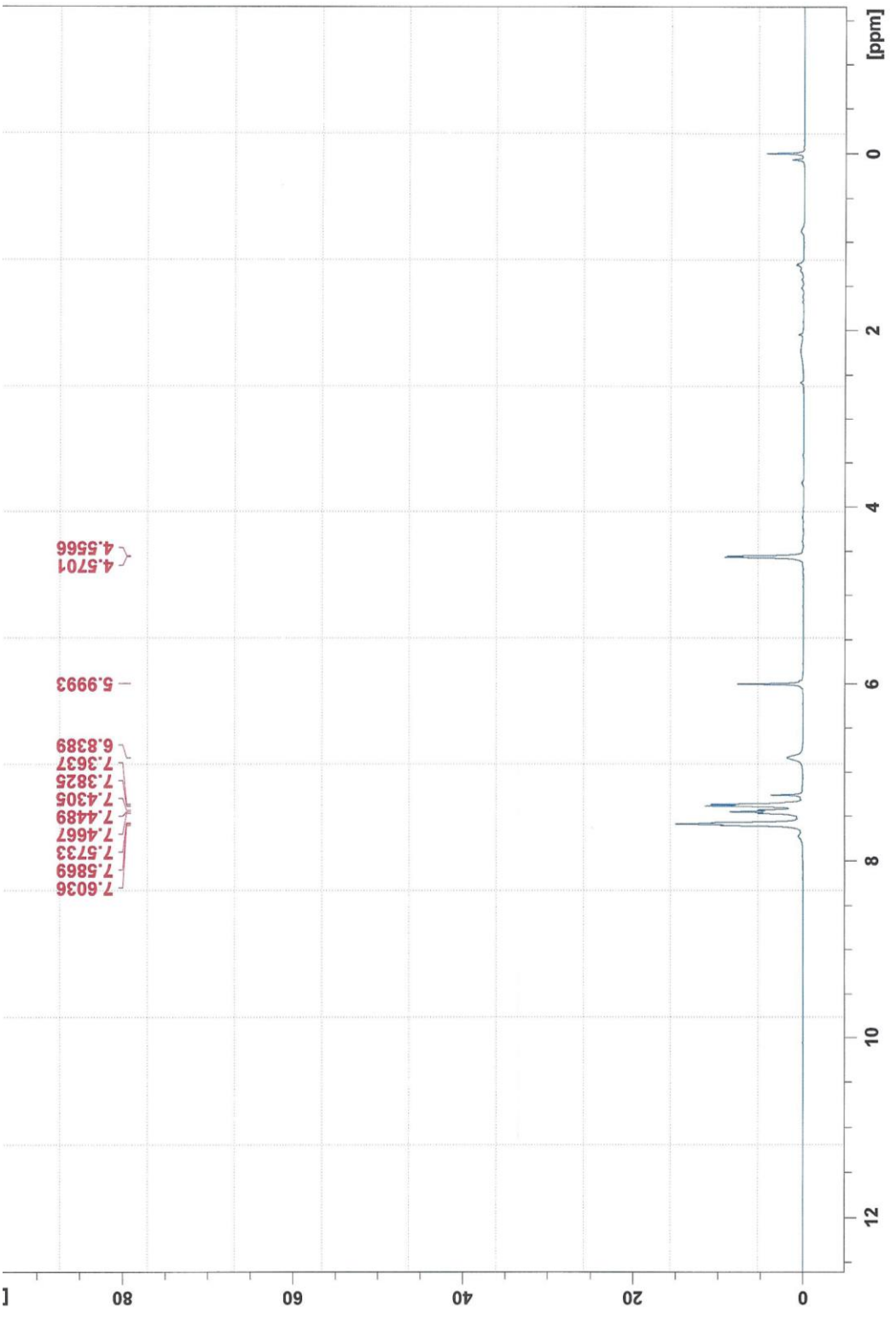

Figure 133: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2-dichloroacetamide **46**

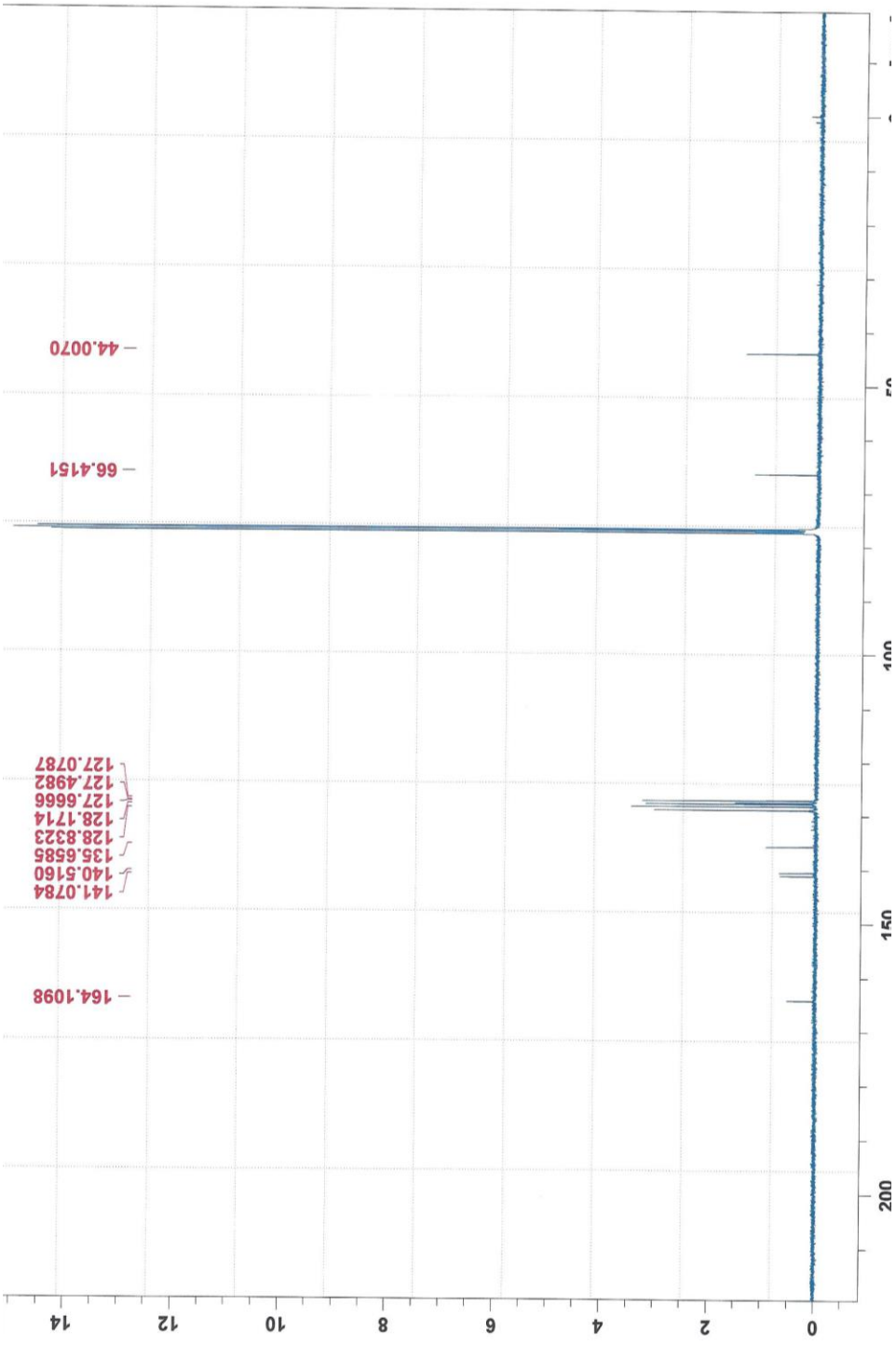

Figure 134: ^{13}C NMR Spectrum of *N*-([1,1'-biphemyl]-4-ylmethyl)-2,2-dichloroacetamide **46**

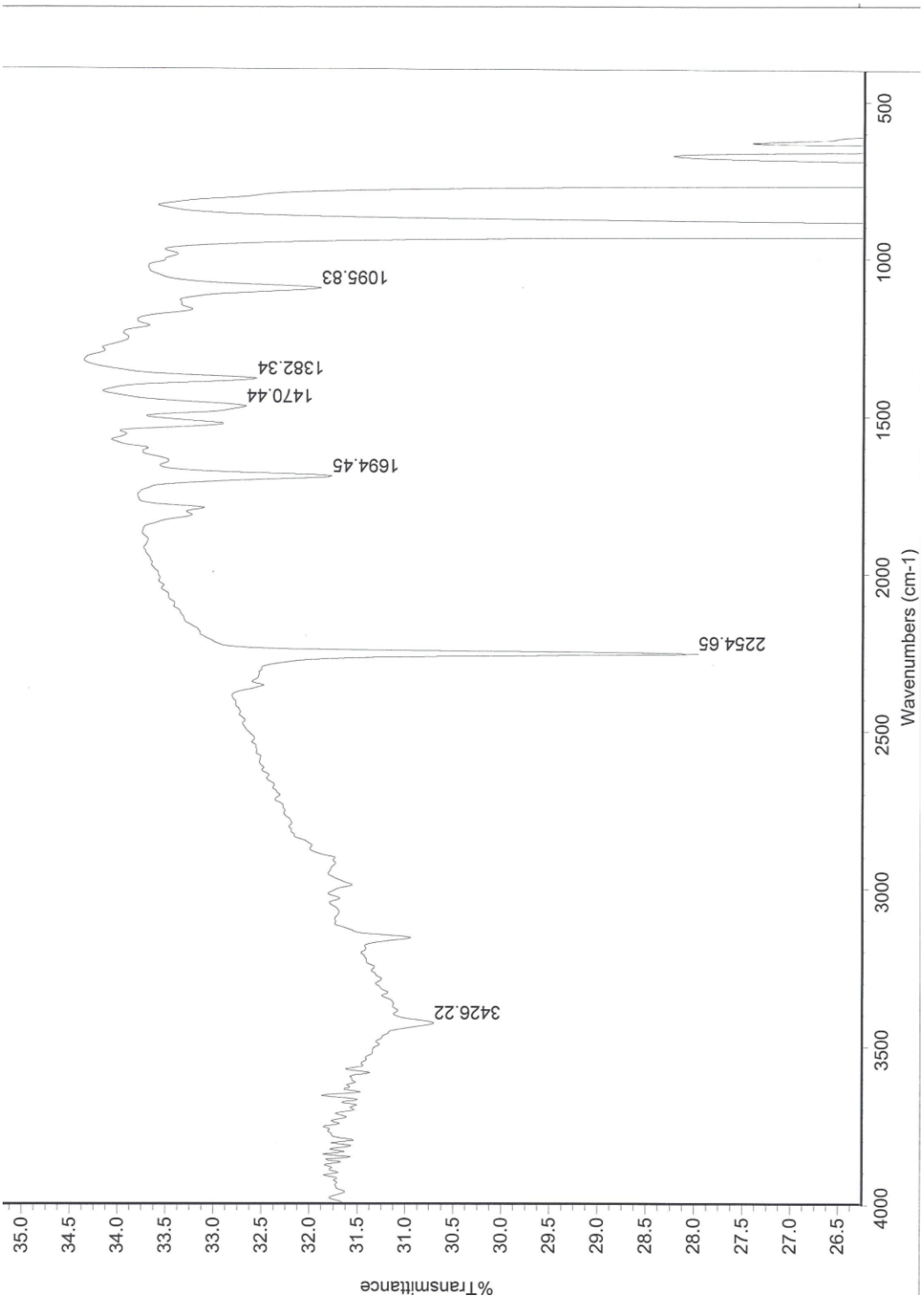

Figure 135: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,2-dichloroacetamide **46**

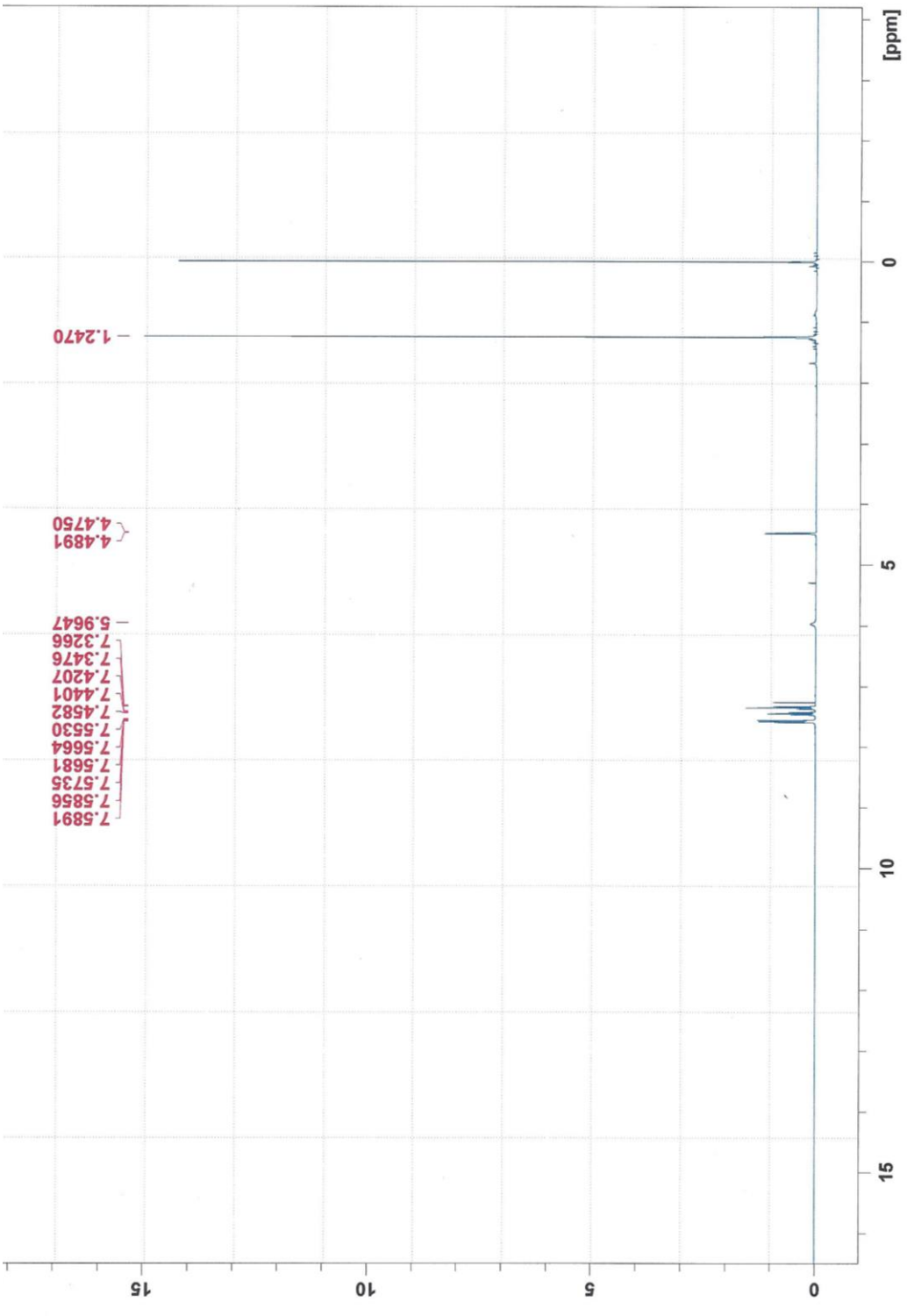

Figure 136: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pivalamide **47**

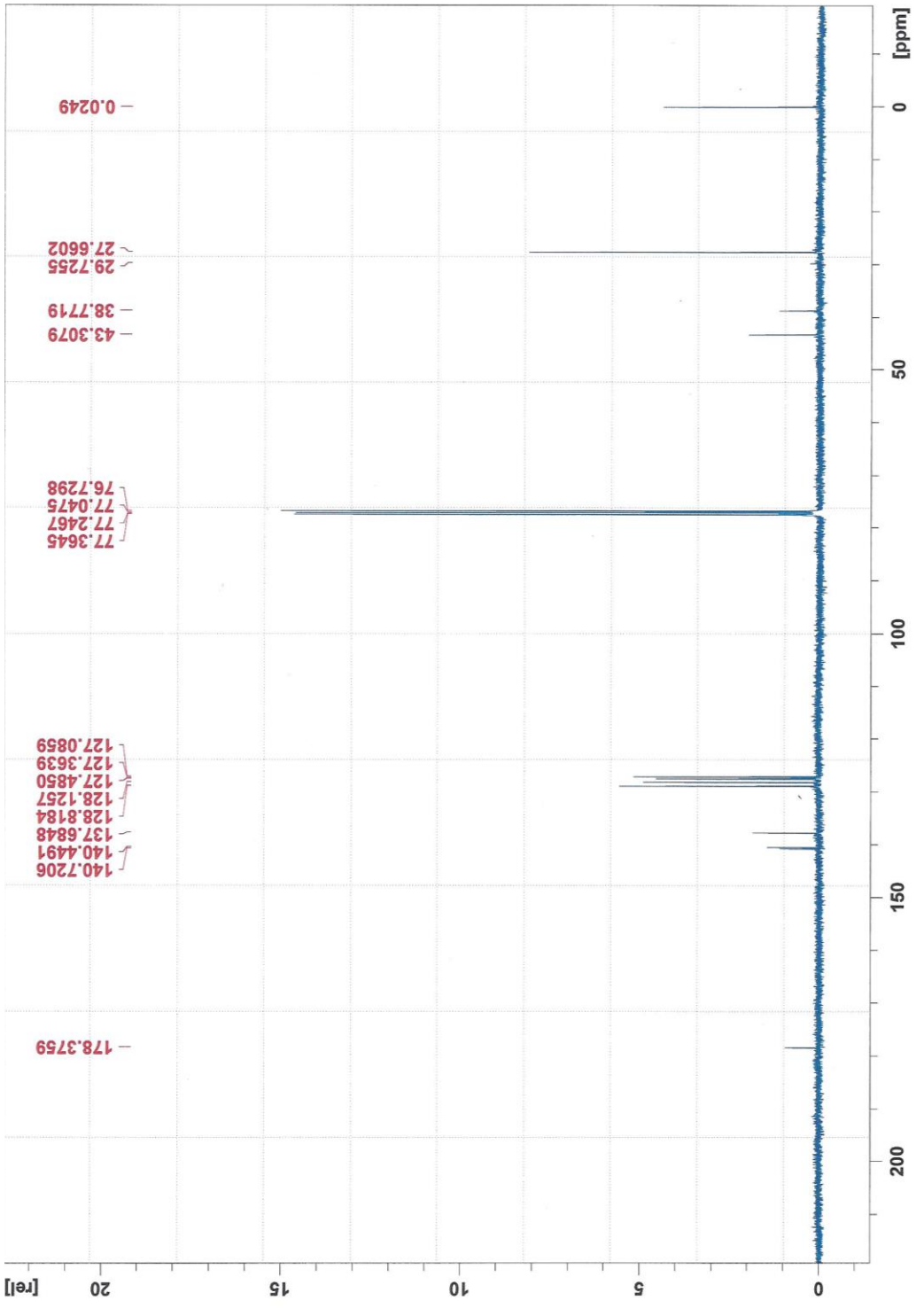

Figure 137: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pivalamide **47**

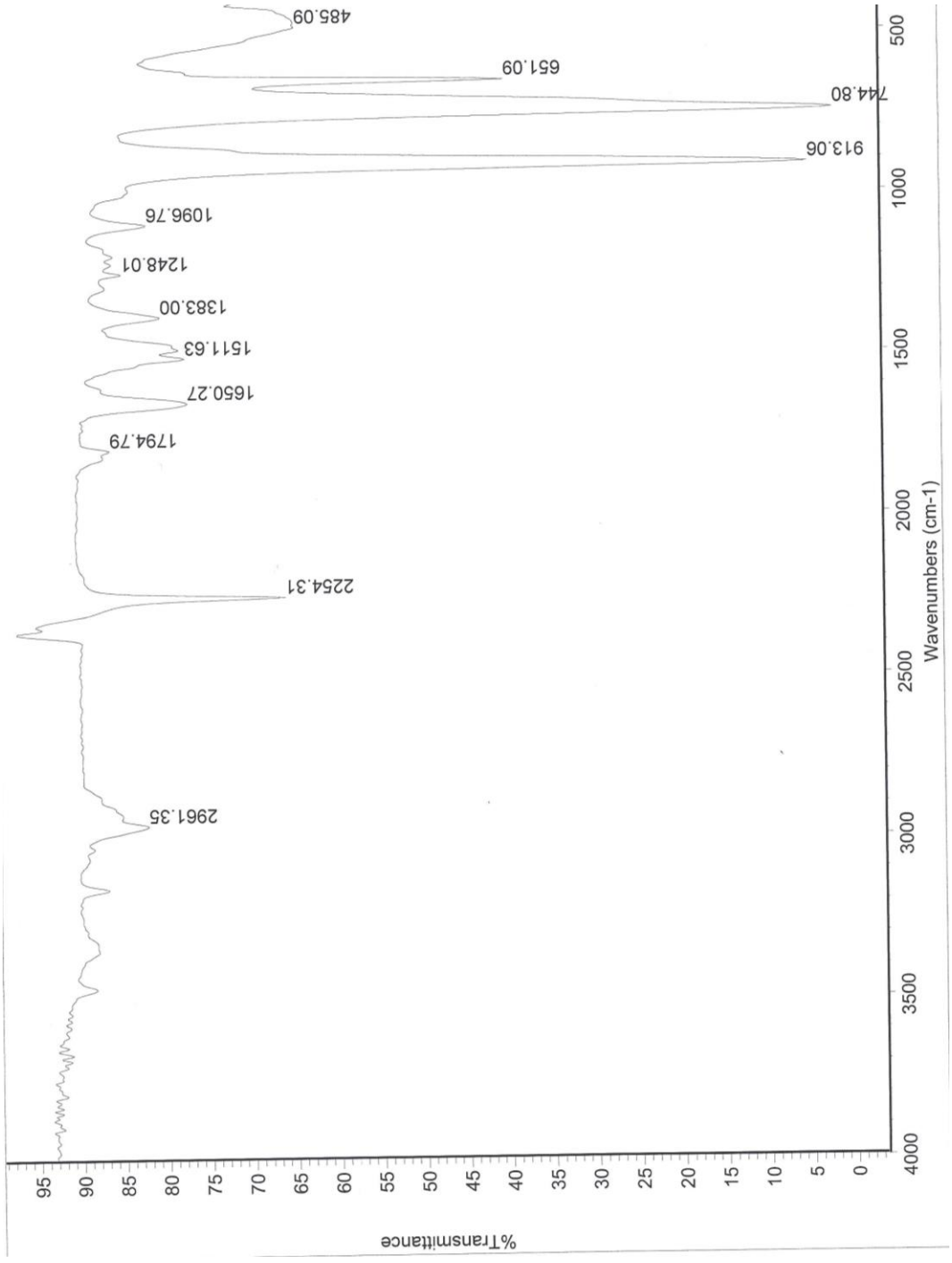

Figure 138: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pivalamide **47**

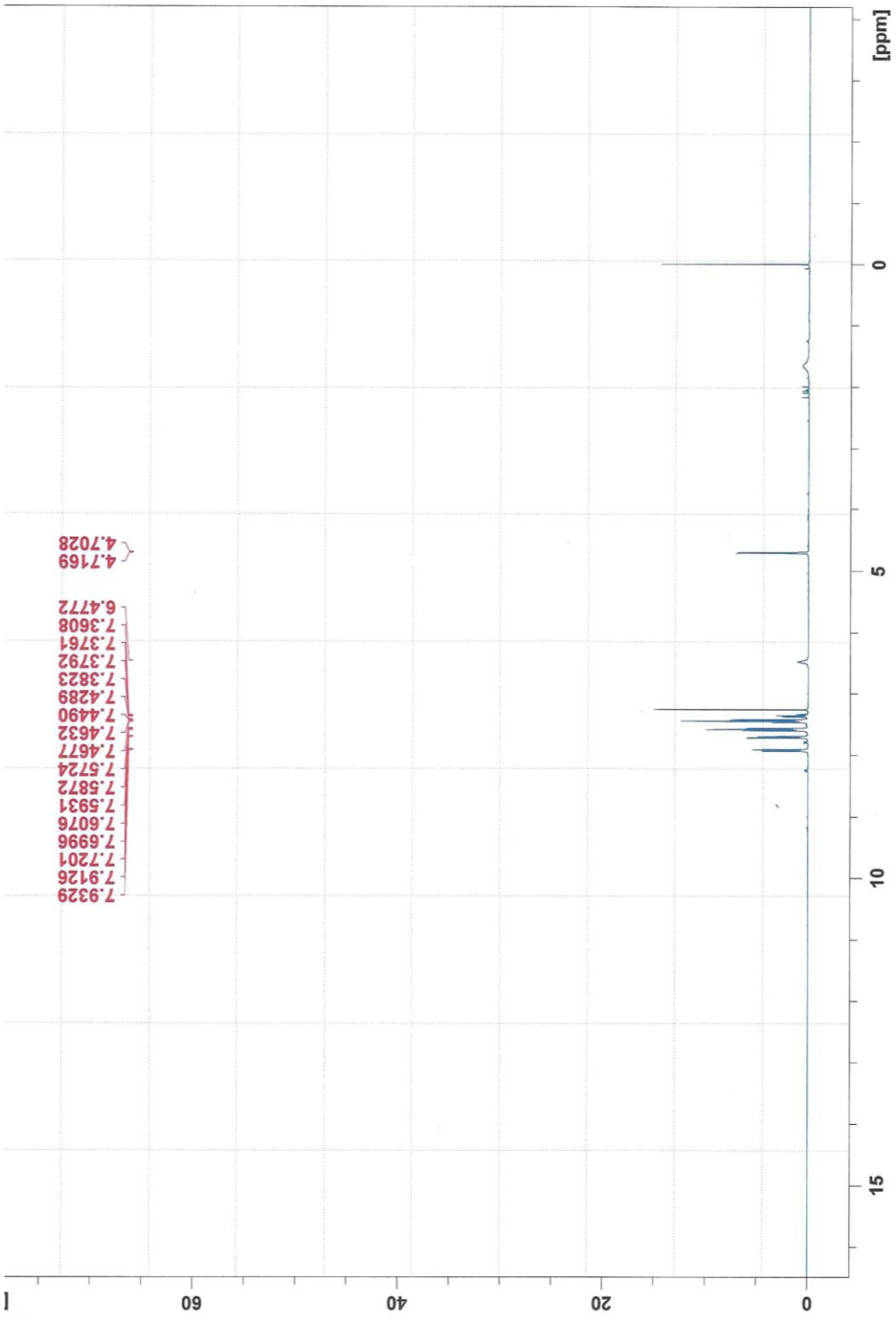

Figure 139: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-(trifluoromethyl)benzamide **48**

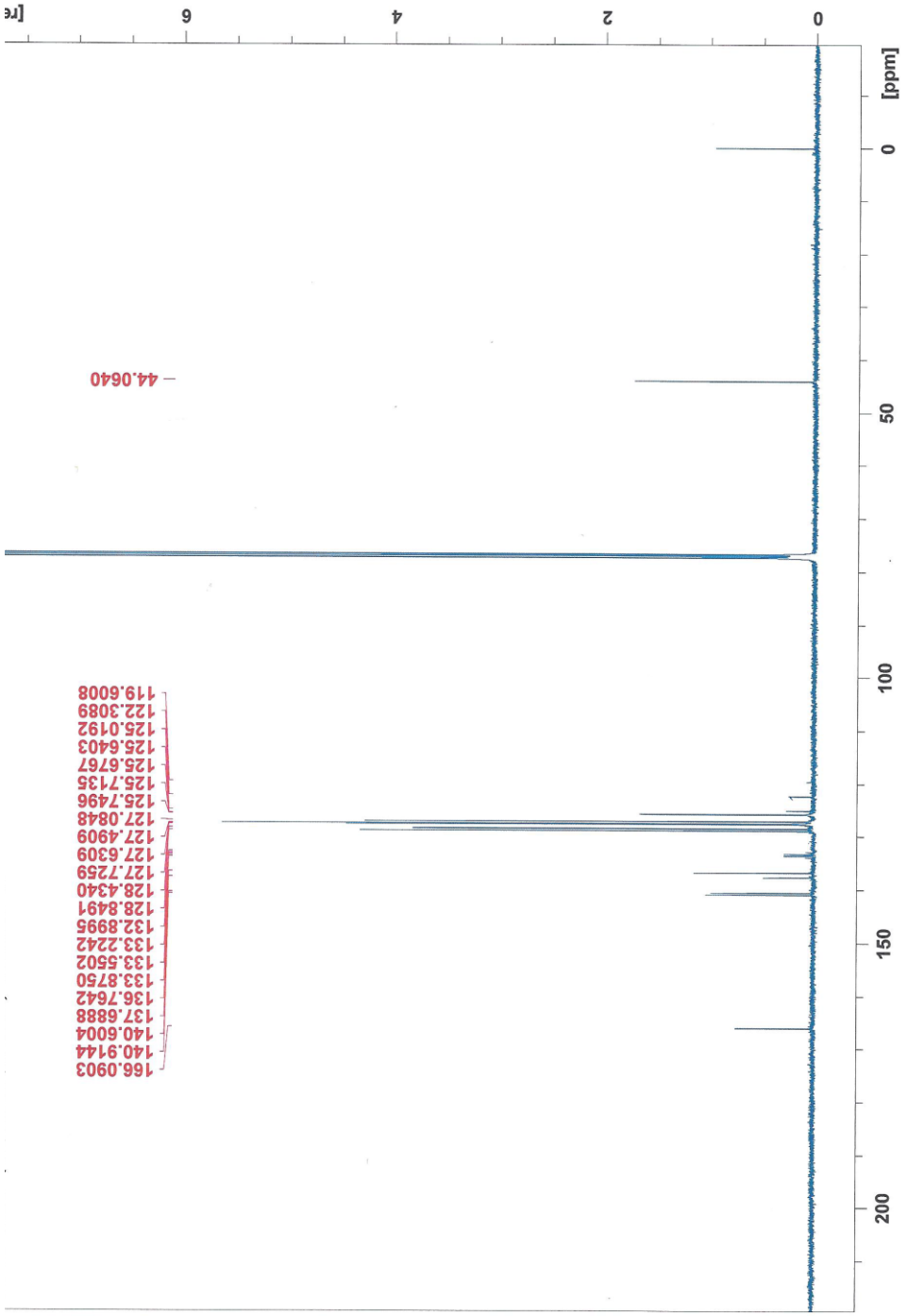

Figure 140: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-(trifluoromethyl)benzamide **48**

Figure 141: ^{19}F NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-(trifluoromethyl)benzamide **48**

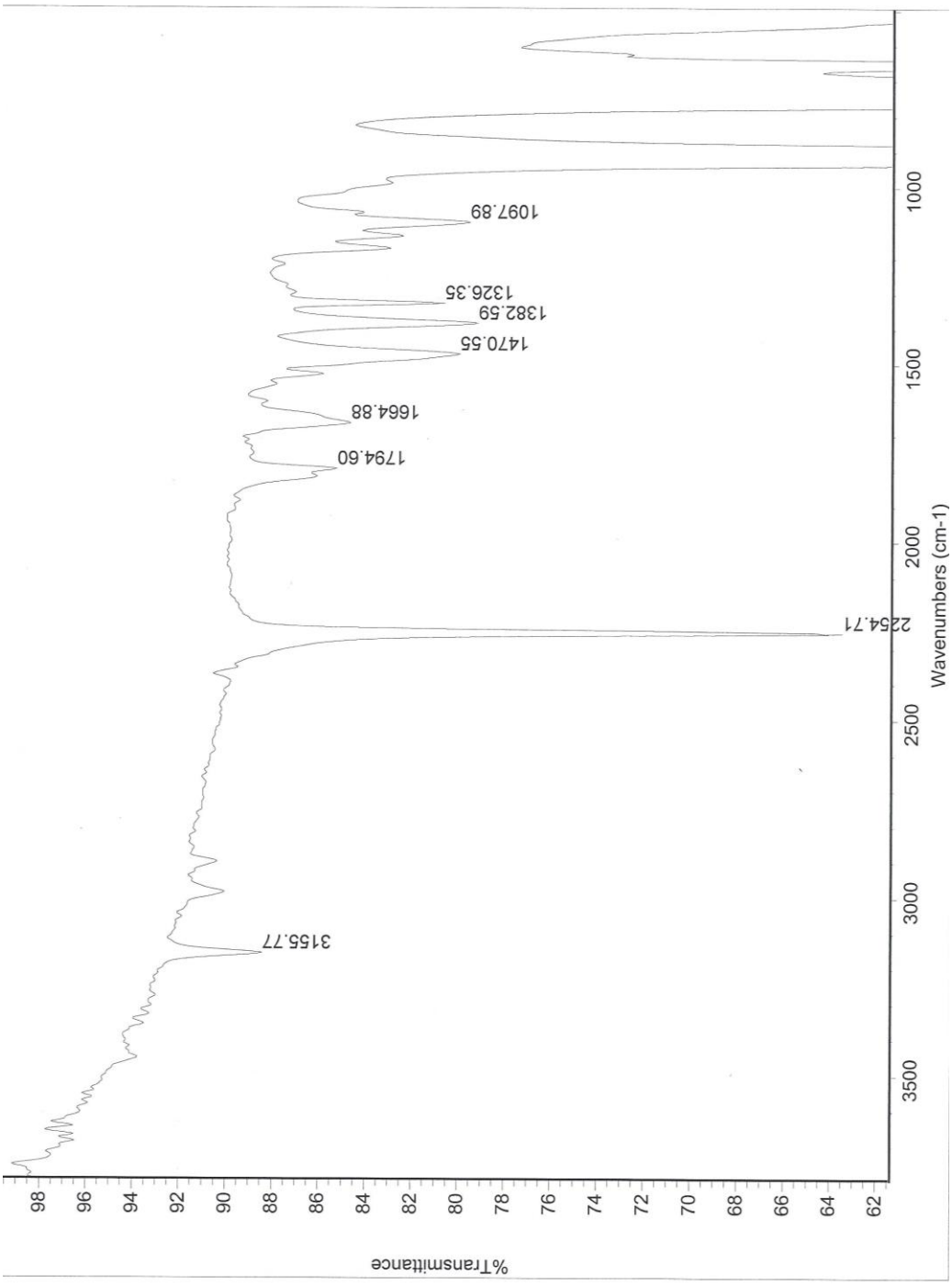

Figure 142: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-4-(trifluoromethyl)benzamide **48**

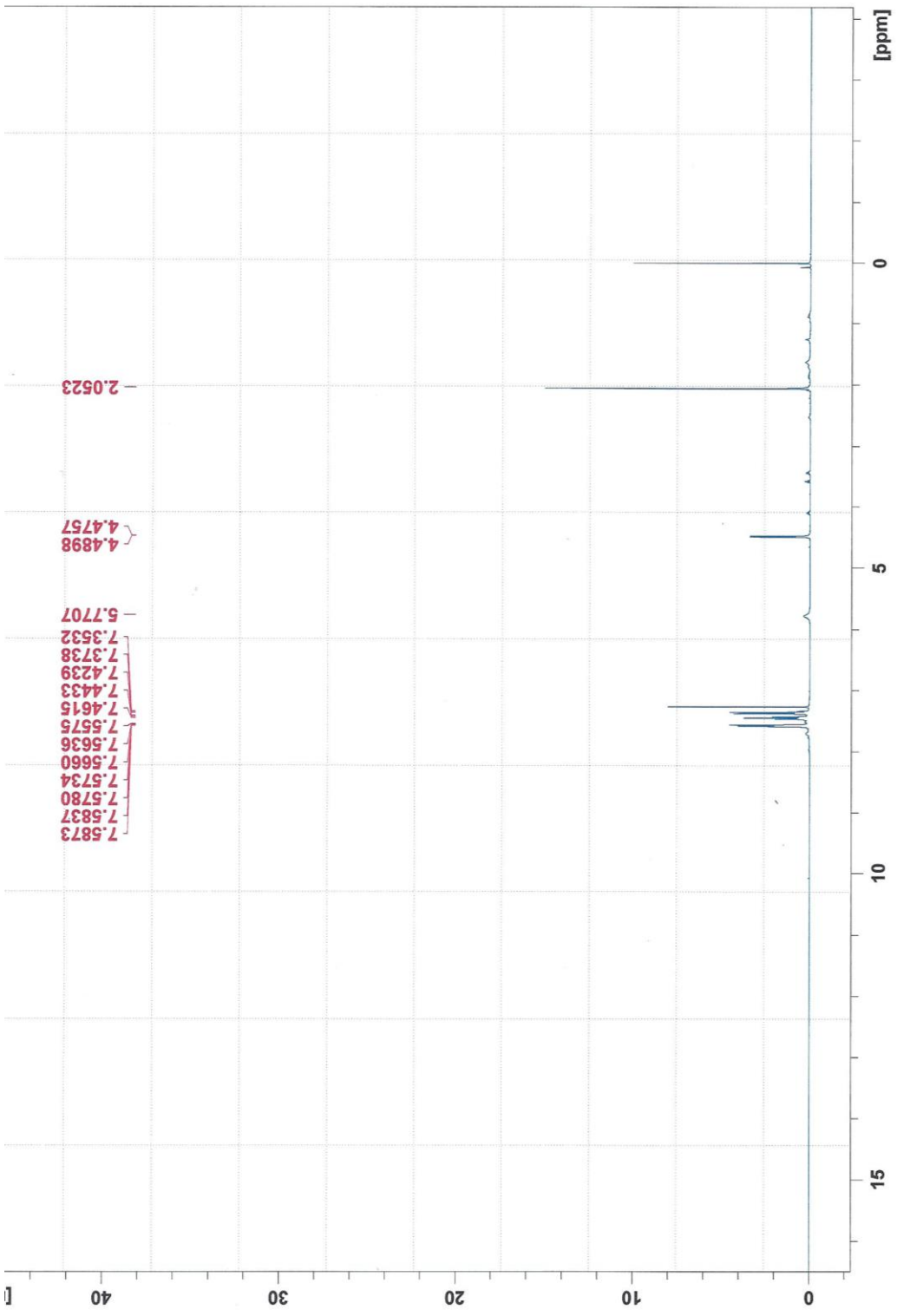

Figure 143: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)acetamide **49**

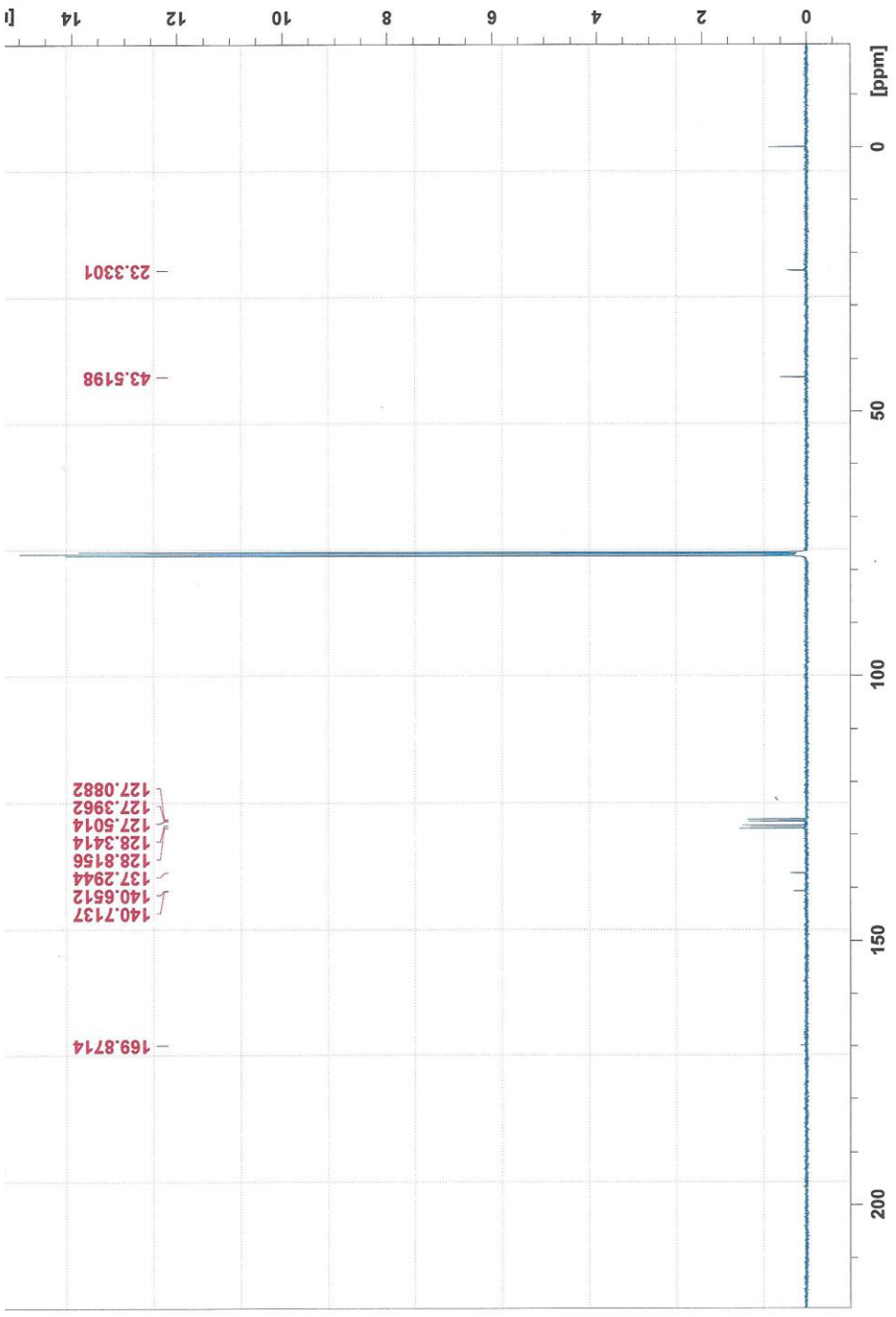

Figure 144: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)acetamide **49**

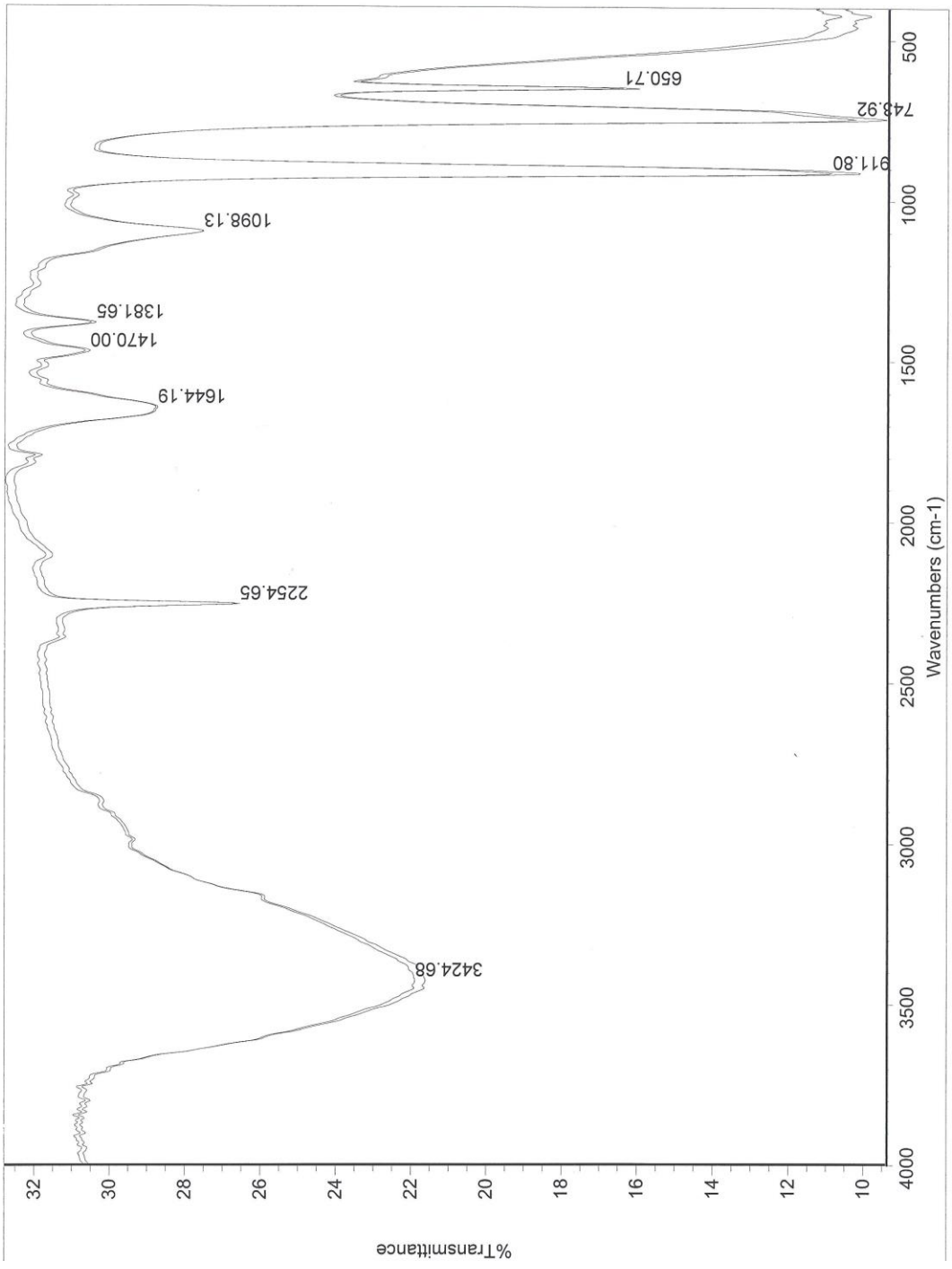

Figure 145: IR NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)acetamide **49**

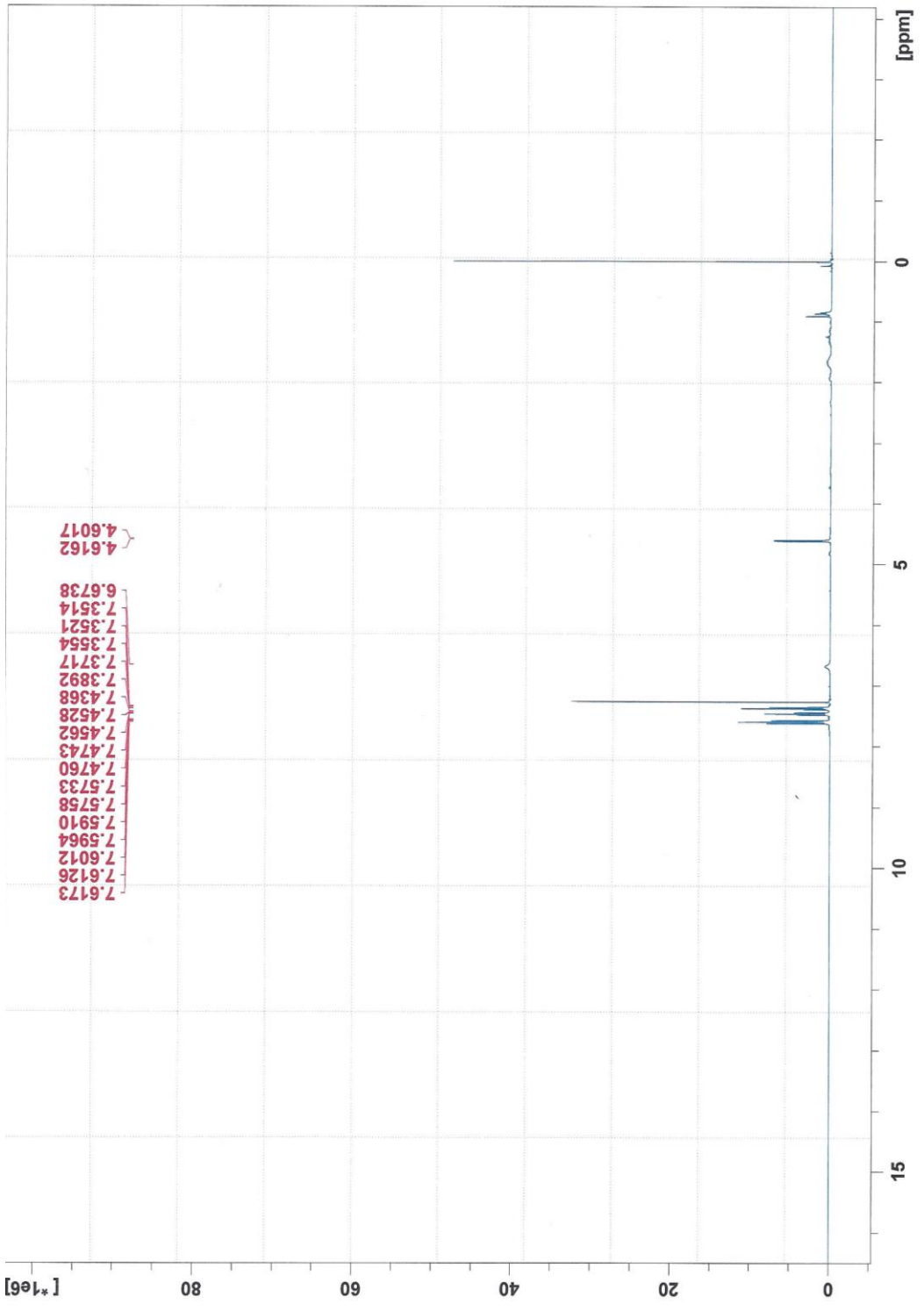

Figure 146: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1,1,2,2,2-pentafluoroethan-1-amine **50**

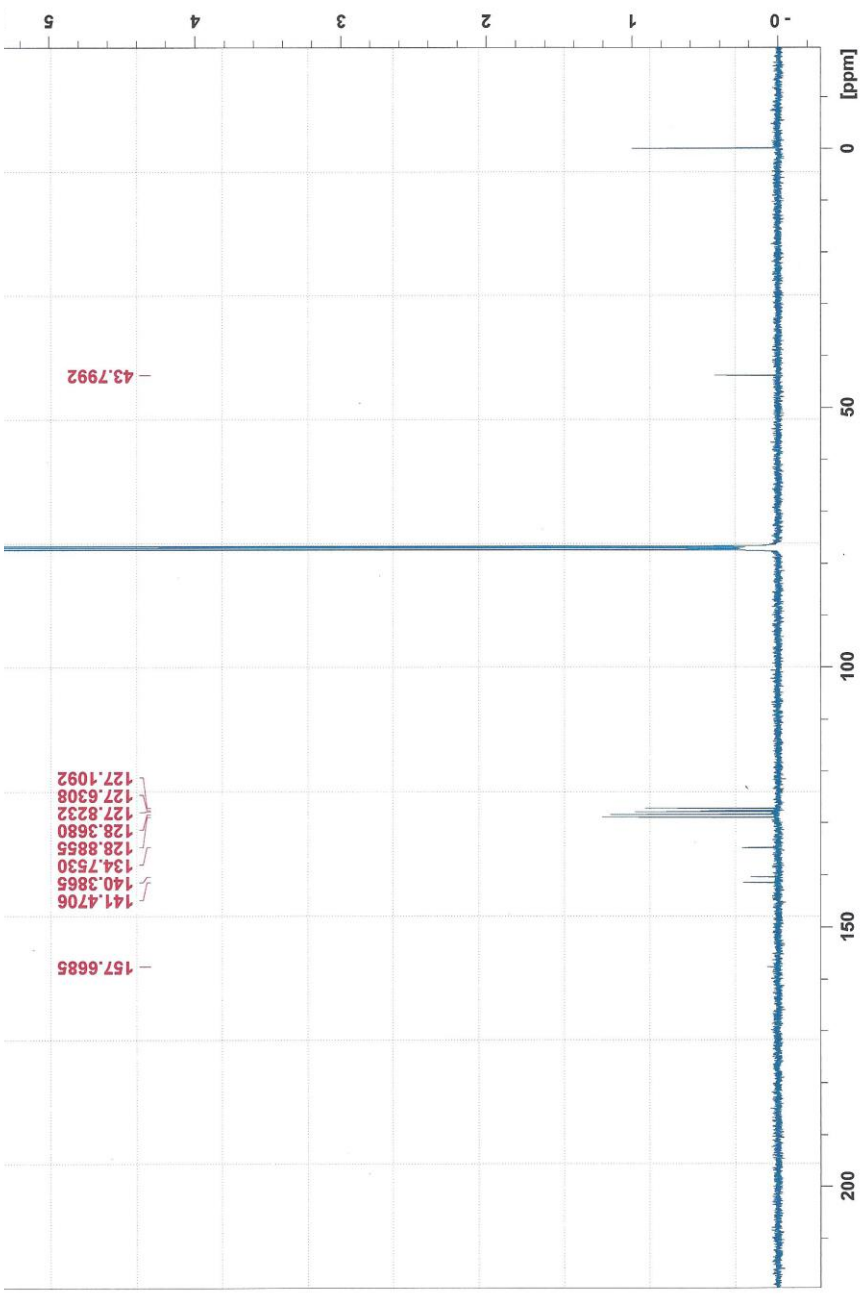

Figure 147: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1,1,2,2,2-pentafluoroethan-1-amine **50**

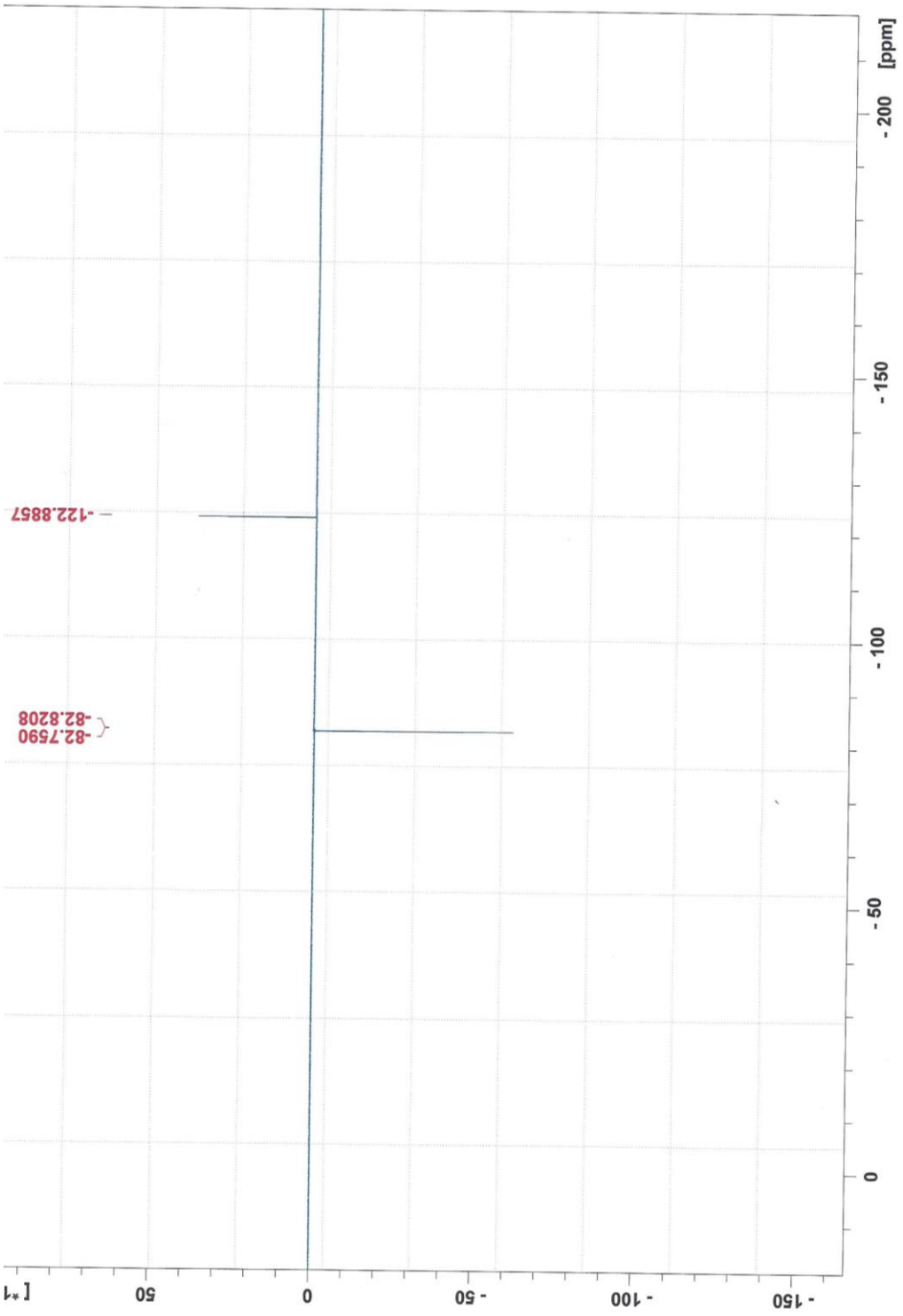

Figure 148: ^{19}F NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1,1,2,2,2-pentafluoroethan-1-amine **50**

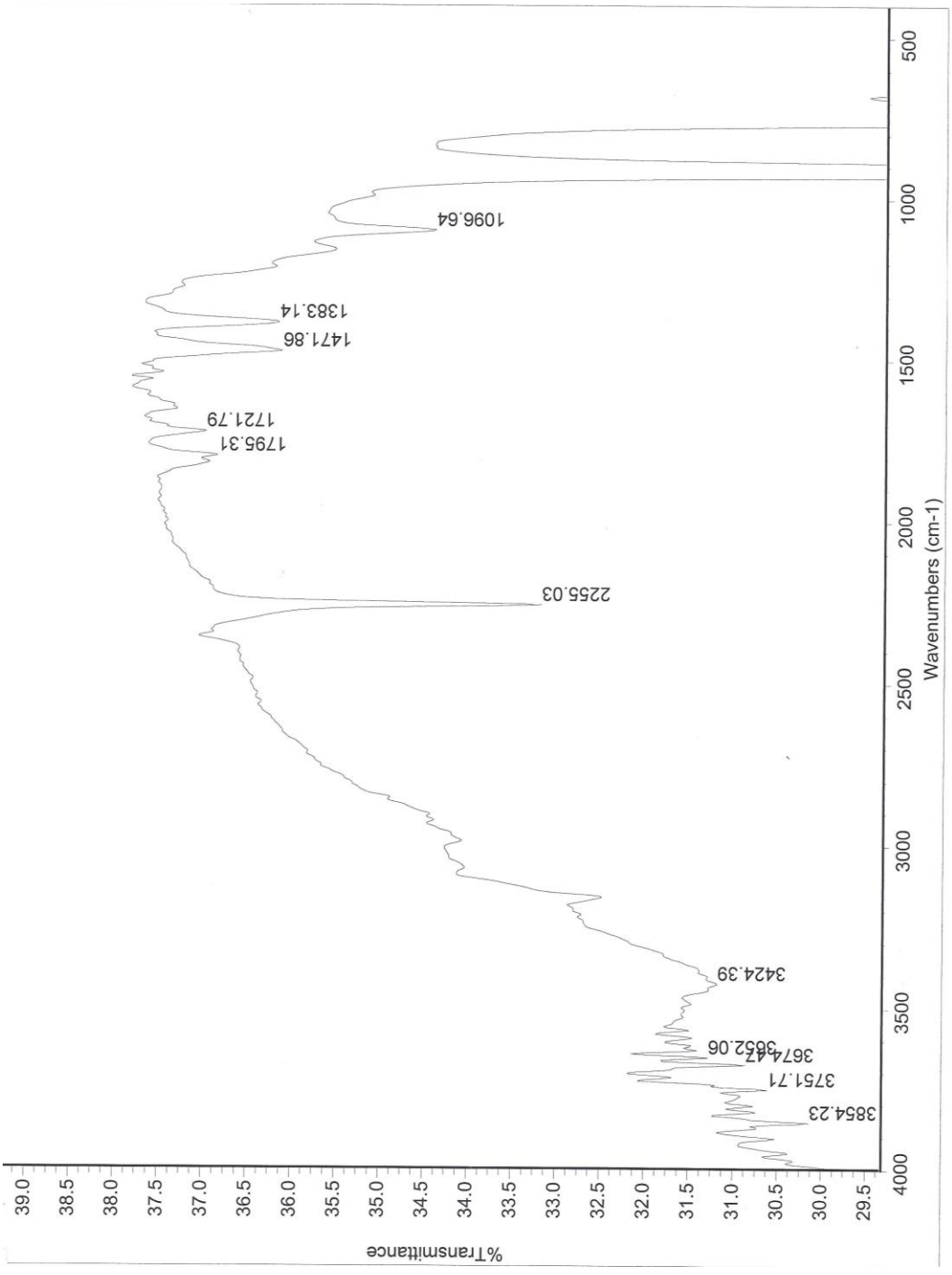

Figure 149: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-1,1,2,2,2-pentafluoroethan-1-amine **51**

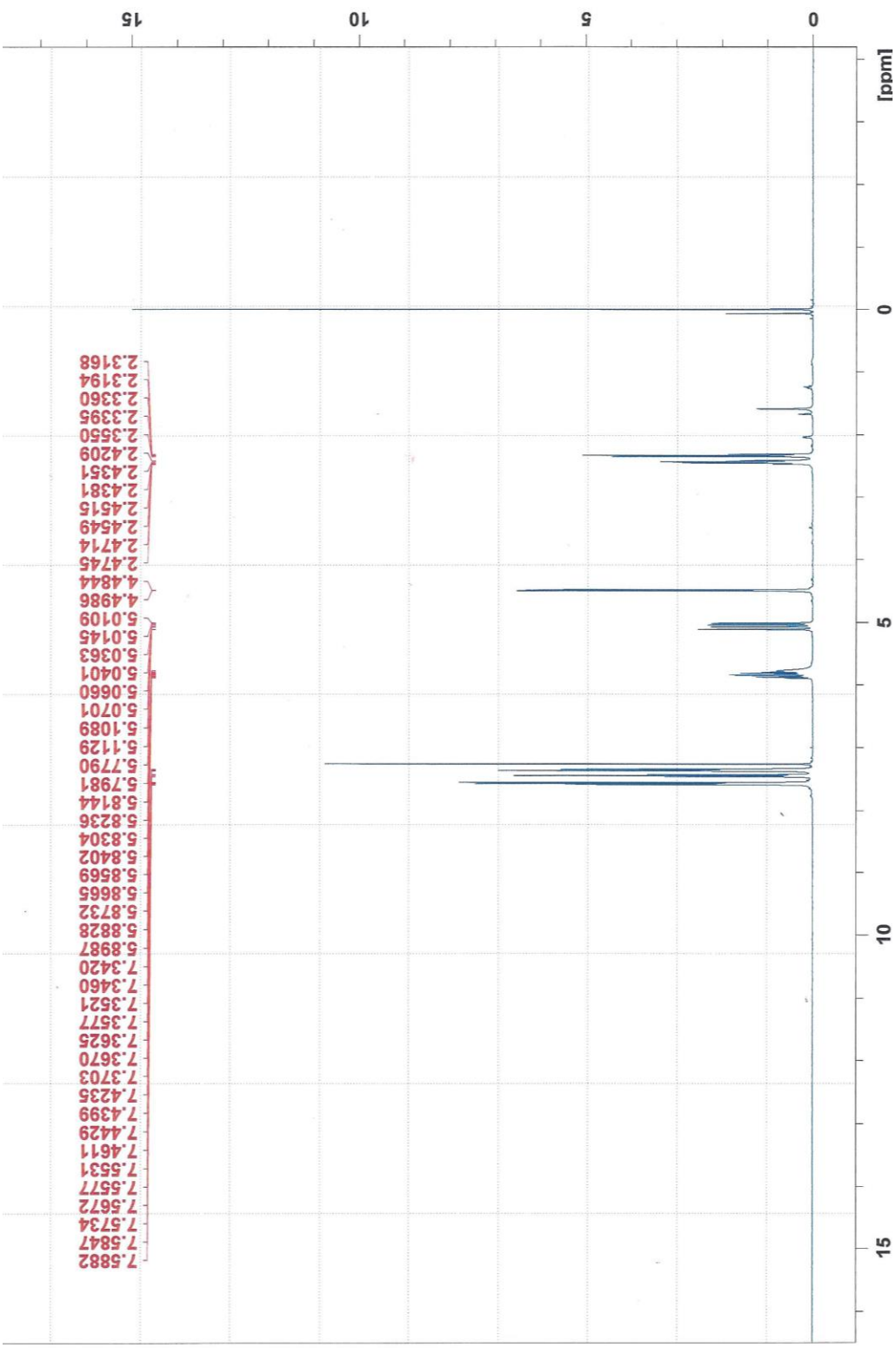

Figure 150: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pent-4-enamide **51**

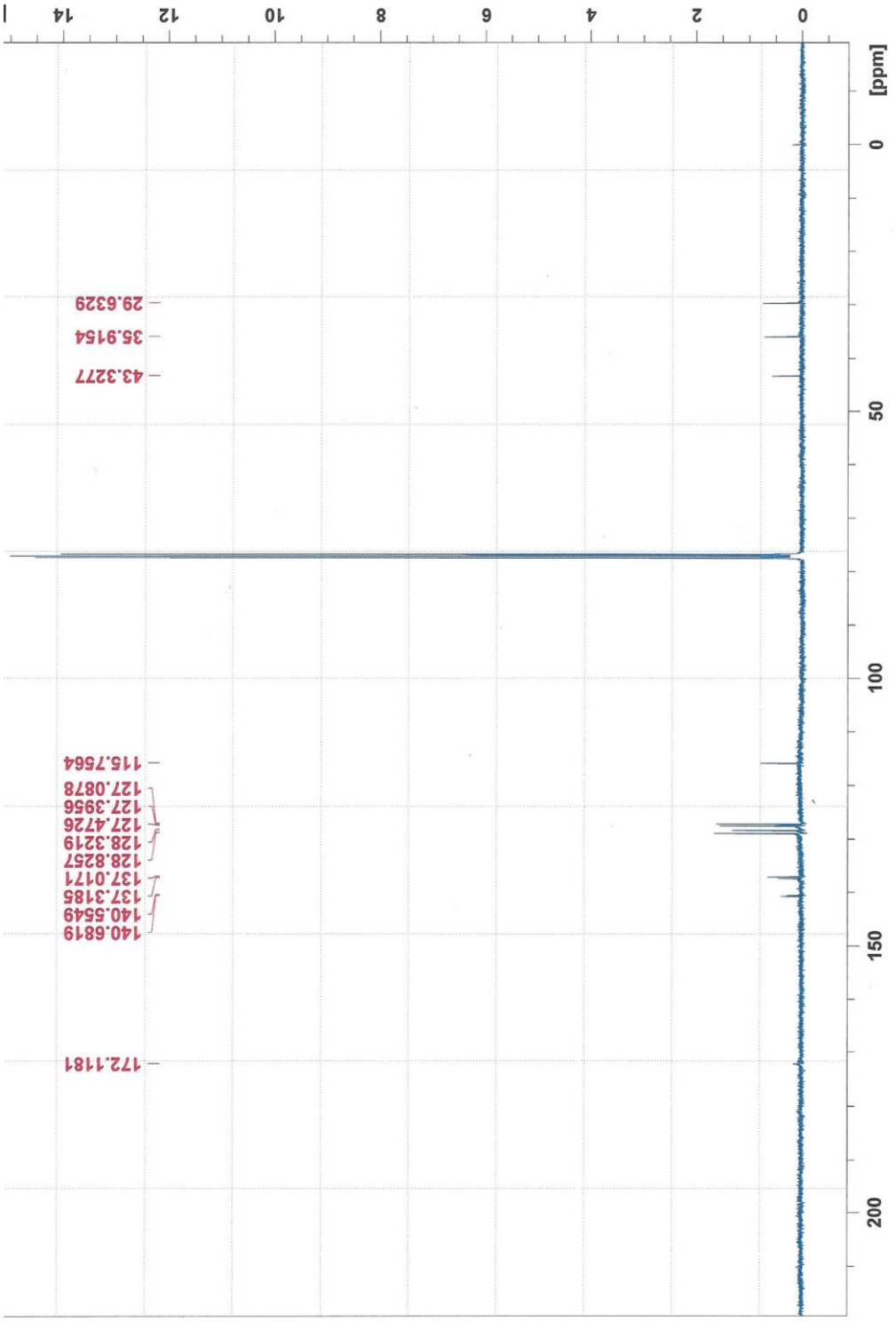

Figure 151: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pent-4-enamide **51**

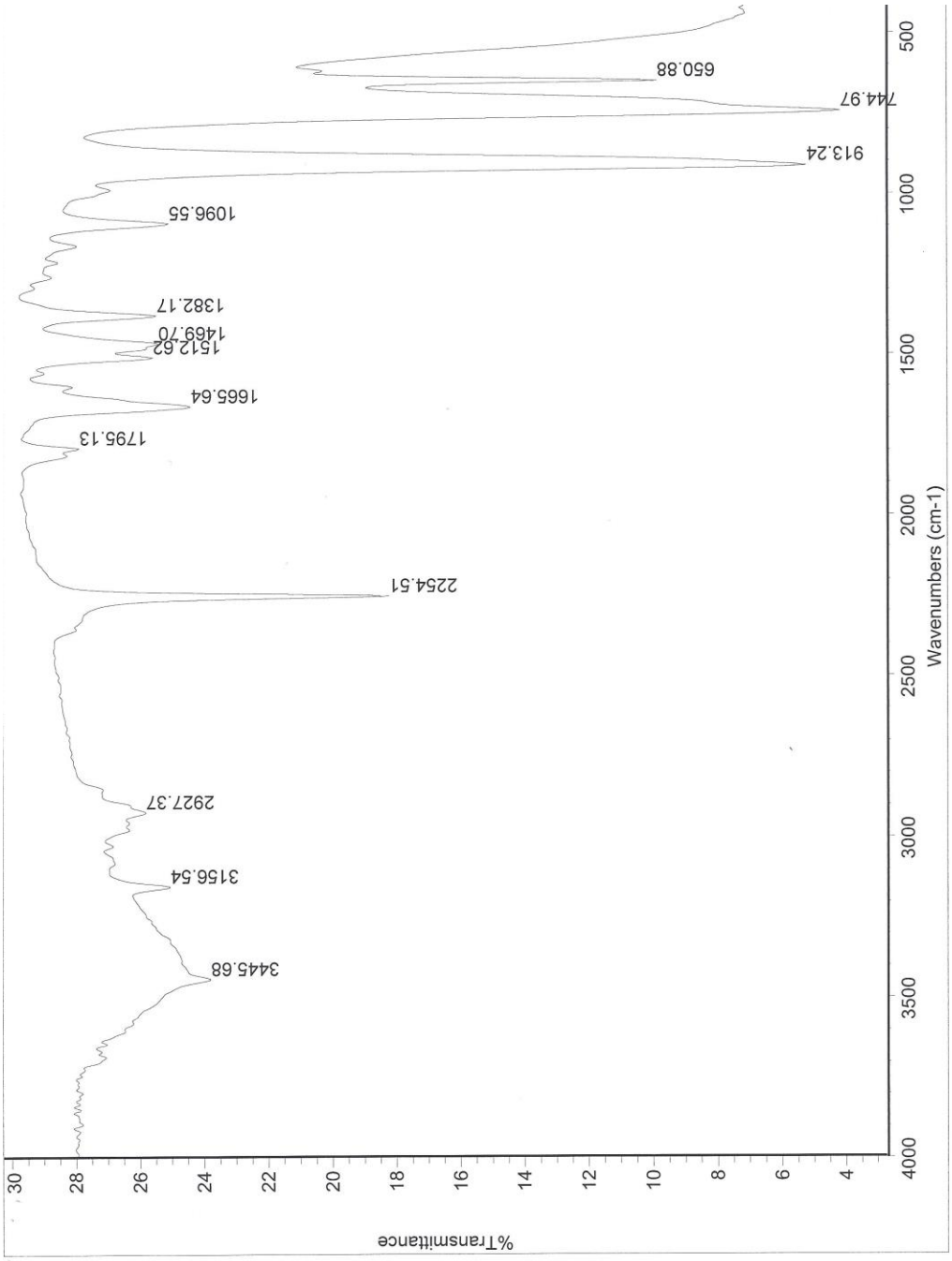

Figure 152: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)pent-4-enamide **51**

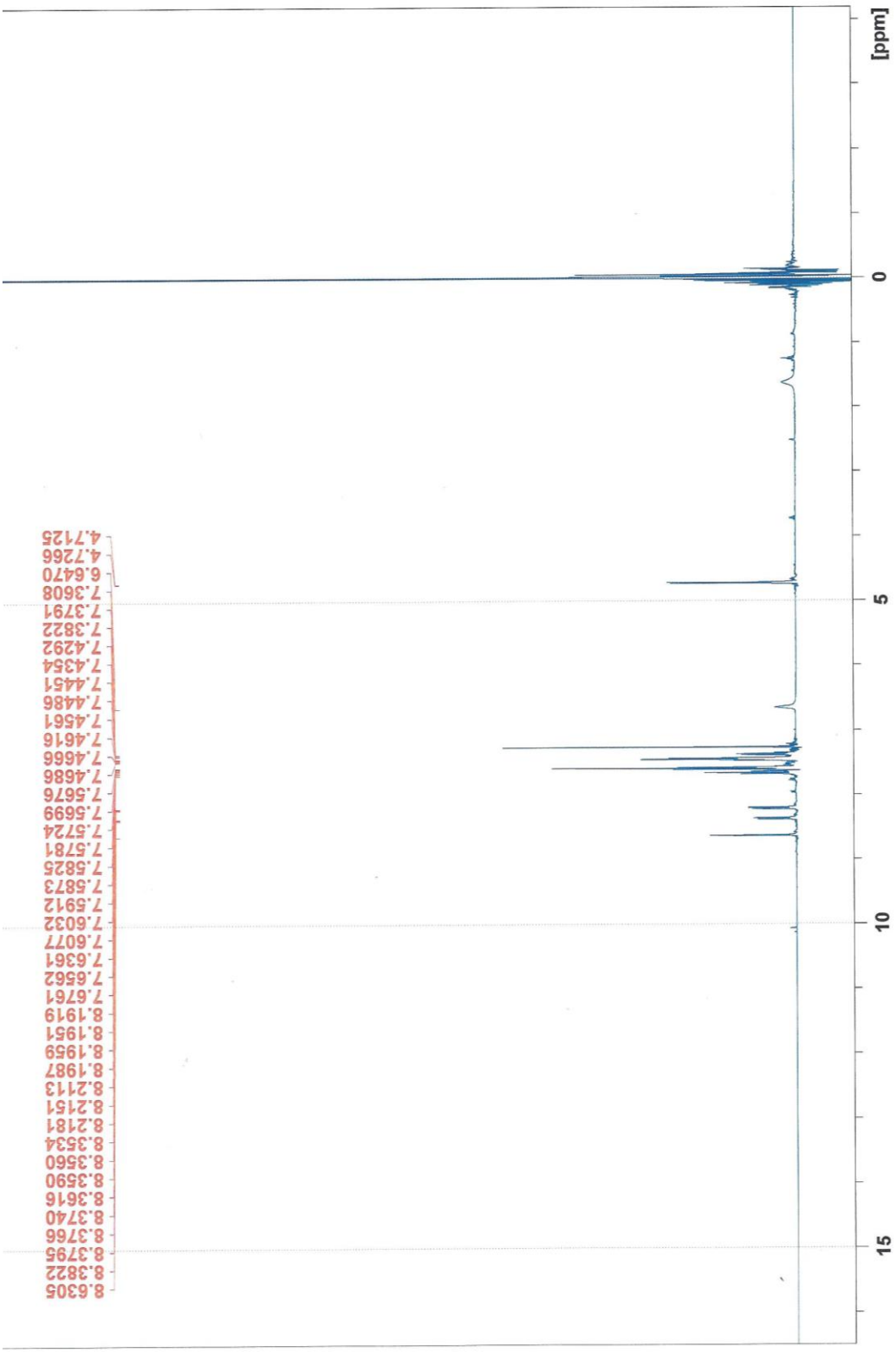

Figure 153: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3-nitrobenzamide **52**

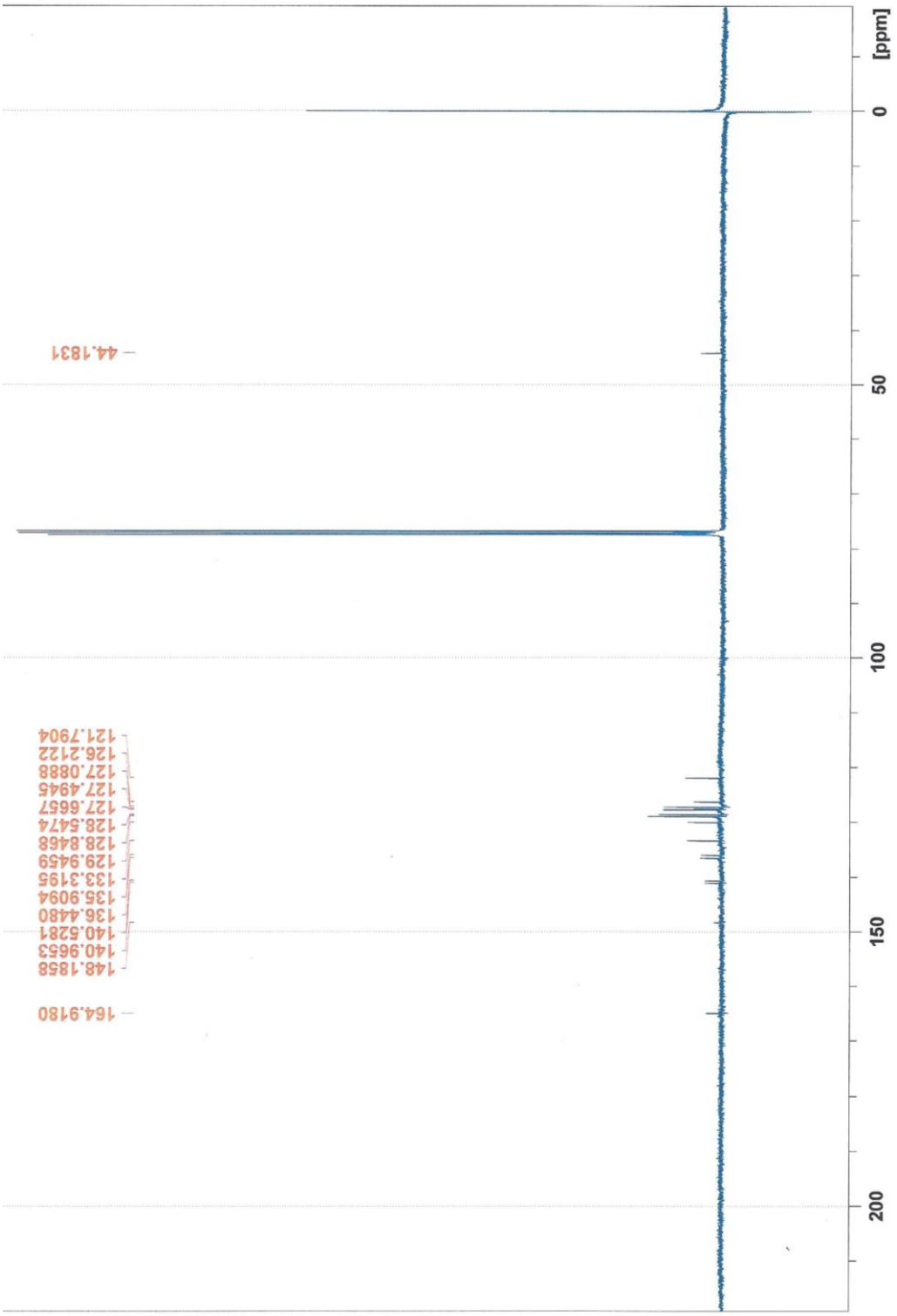

Figure 154: ^{13}C NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3-nitrobenzamide **52**

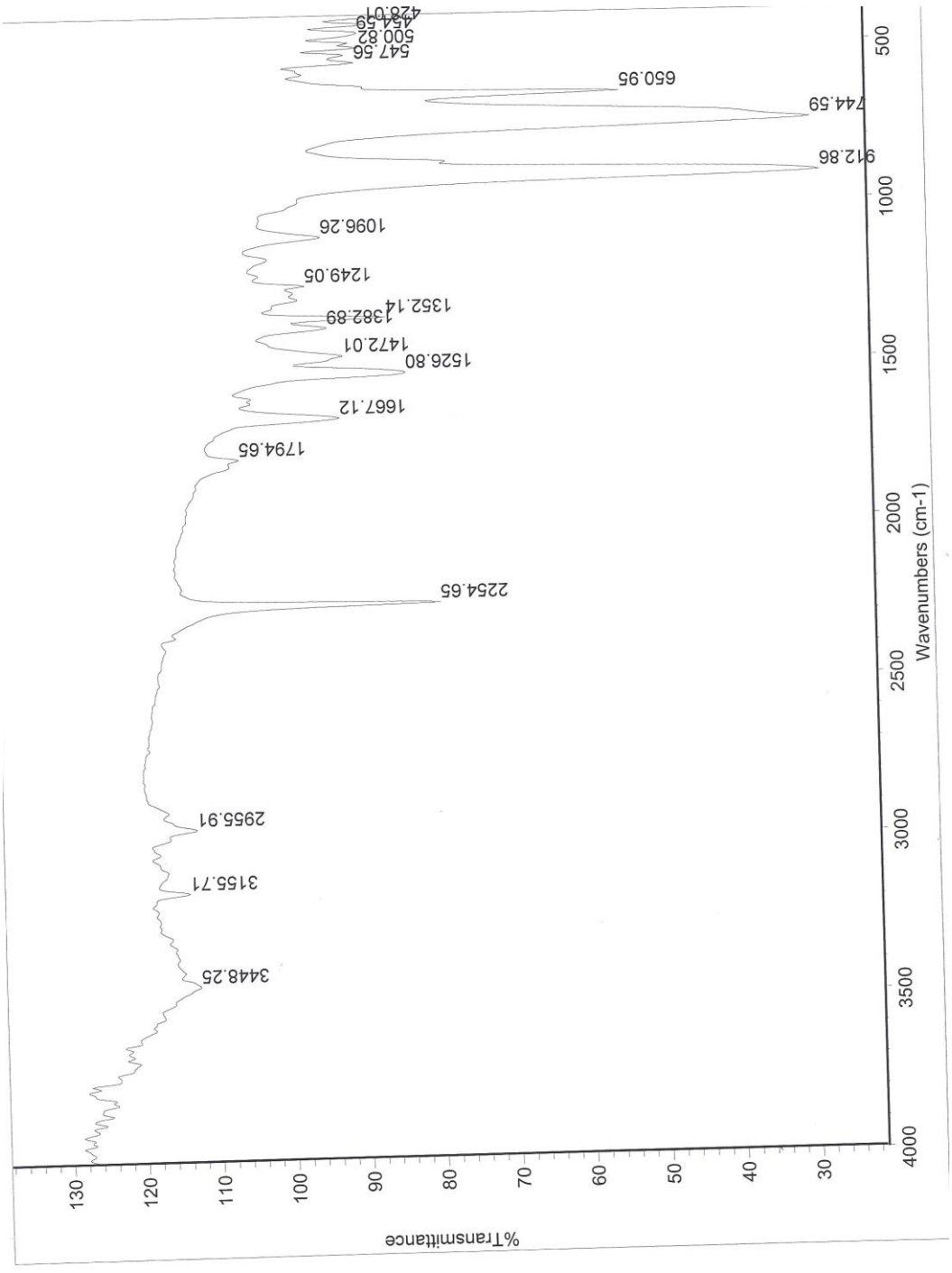

Figure 155: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-3-nitrobenzamide **52**

NMR & IR Spectra of Side Products

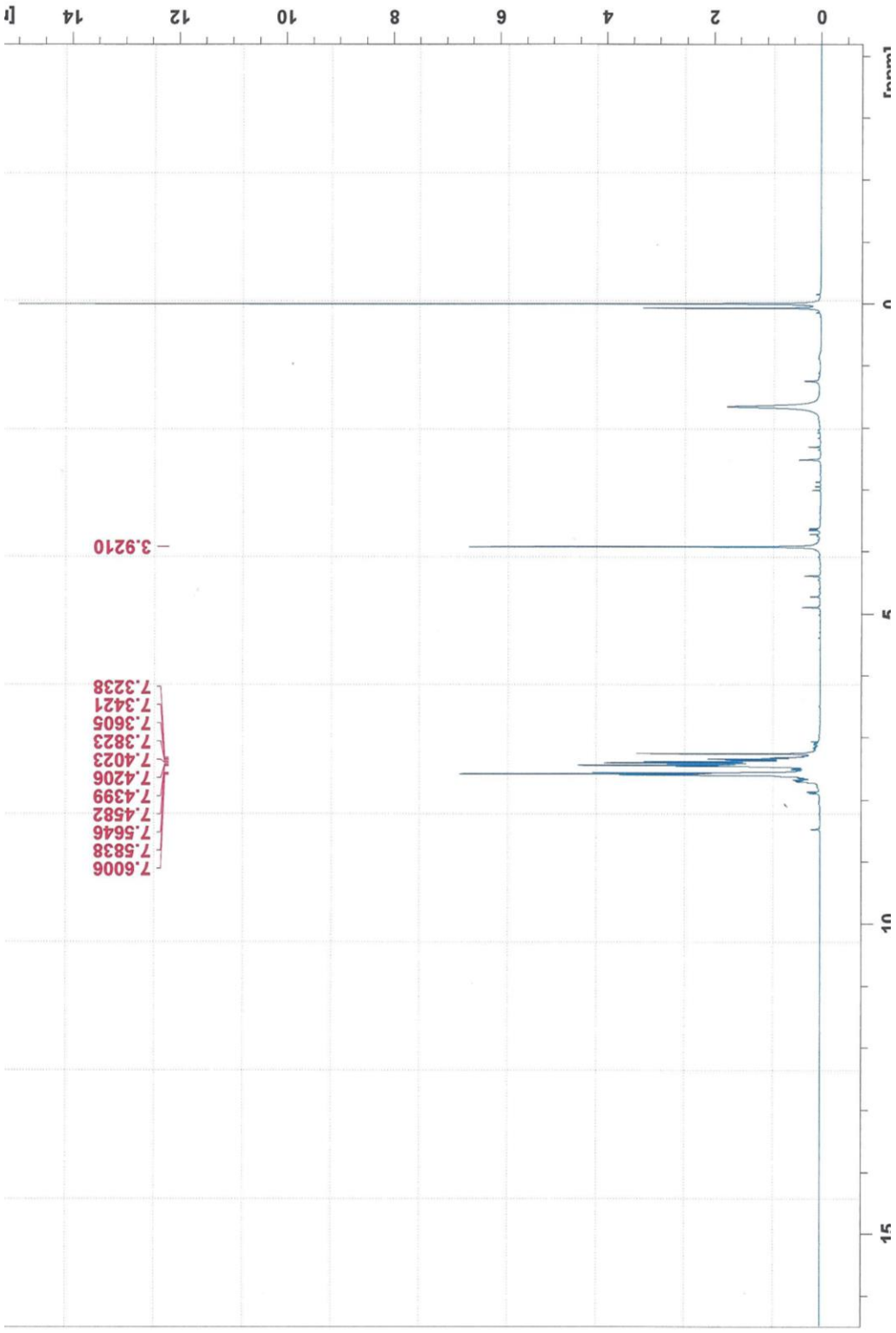

Figure 156: ^{1}H NMR Spectrum of [1,1'-biphenyl]-4-ylmethanamine **54**

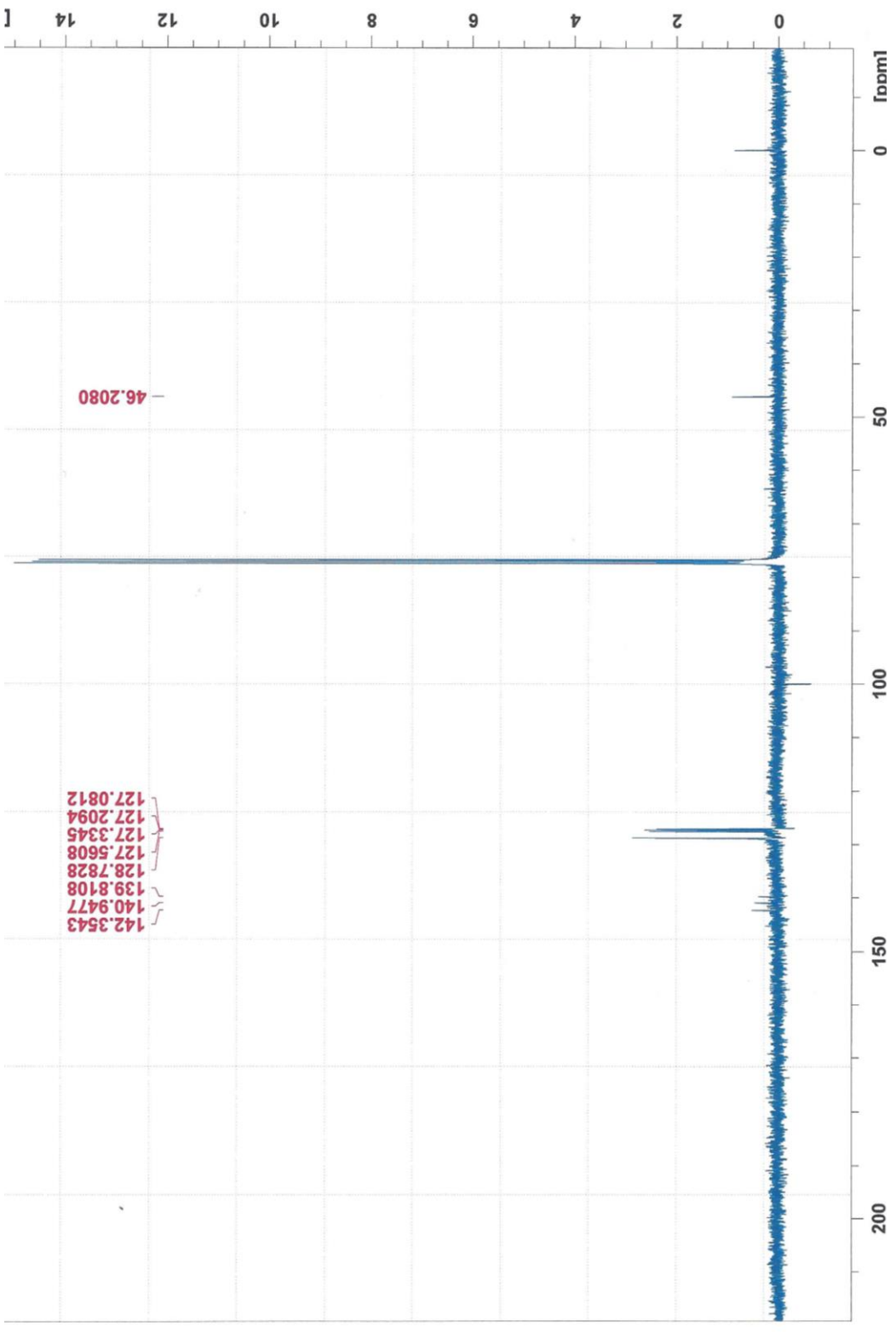

Figure 157: ^{13}C NMR Spectrum of [1,1'-biphenyl]-4-ylmethanamine **54**

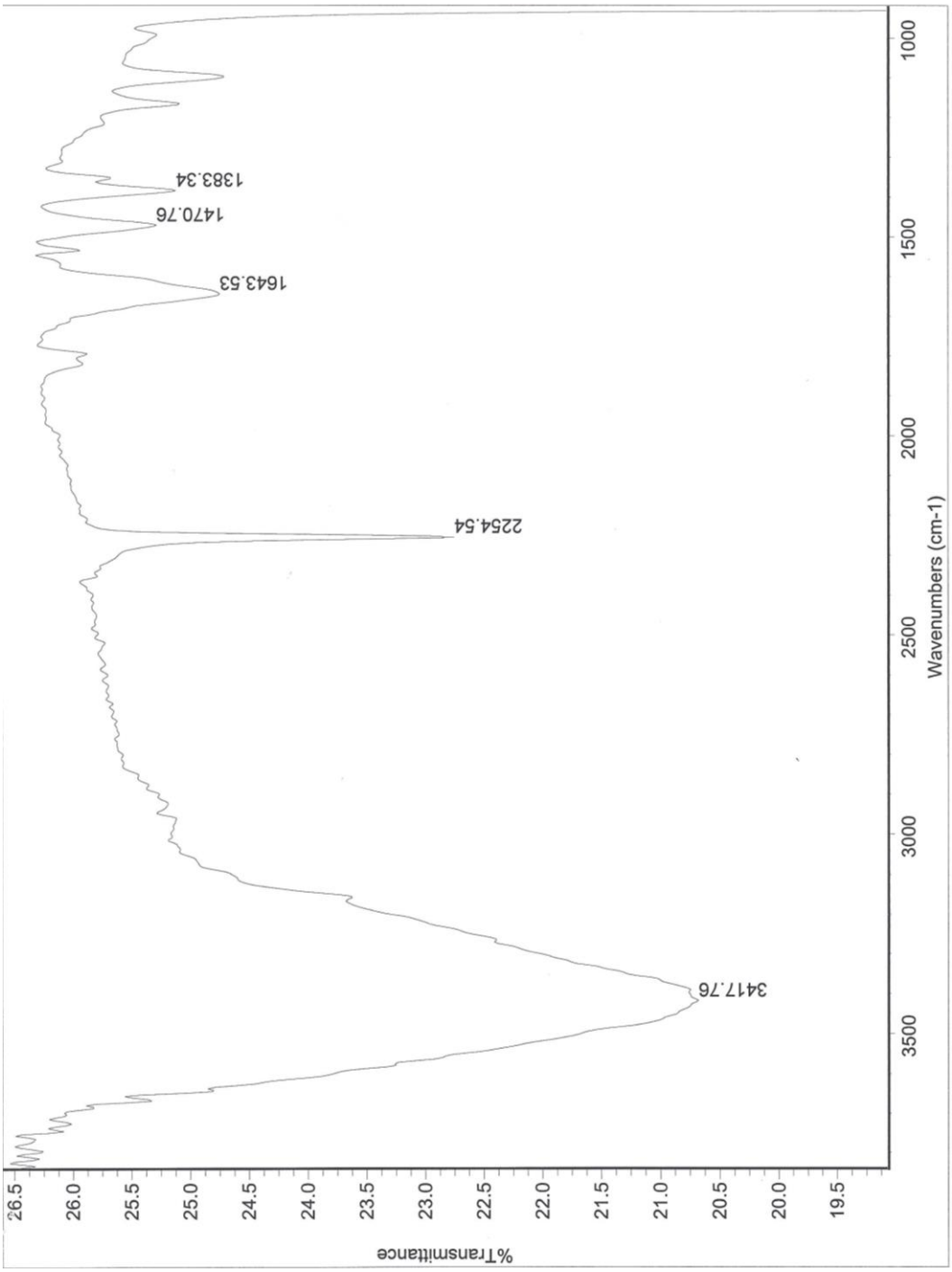

Figure 158: IR Spectrum of [1,1'-biphenyl]-4-ylmethanamine **54**

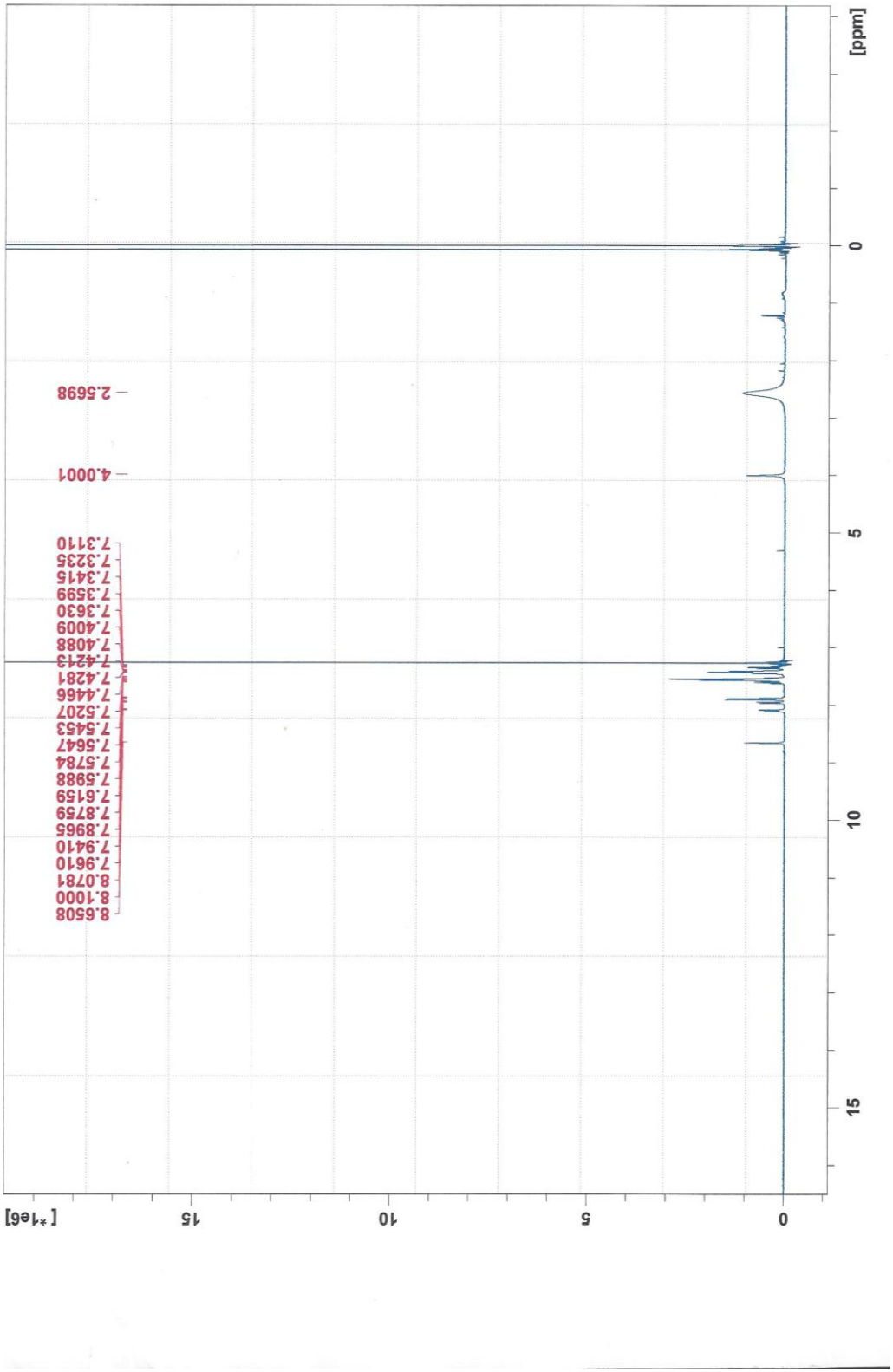

Figure 159: ^{1}H NMR Spectrum of [1,1'-biphenyl]-4-ylmethanaminium 2-naphthoate hydrate **53**

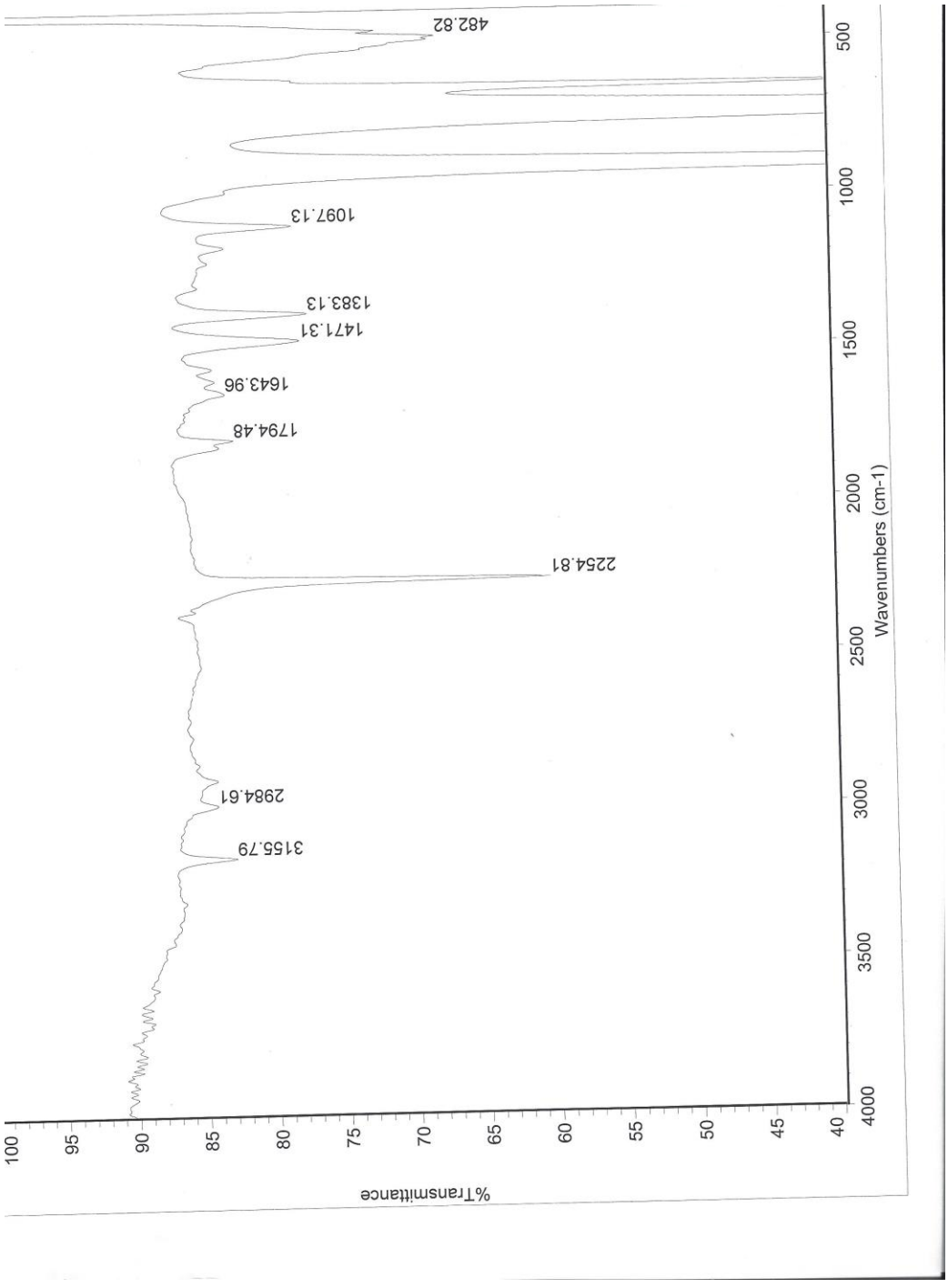

Figure 160: IR Spectrum of [1,1'-biphenyl]-4-ylmethanaminium 2-naphthoate hydrate **53**

Figure 161: ^{1}H NMR Spectrum of *N,N*-dimethyl-4-nitrobenzenesulfonamide **6**

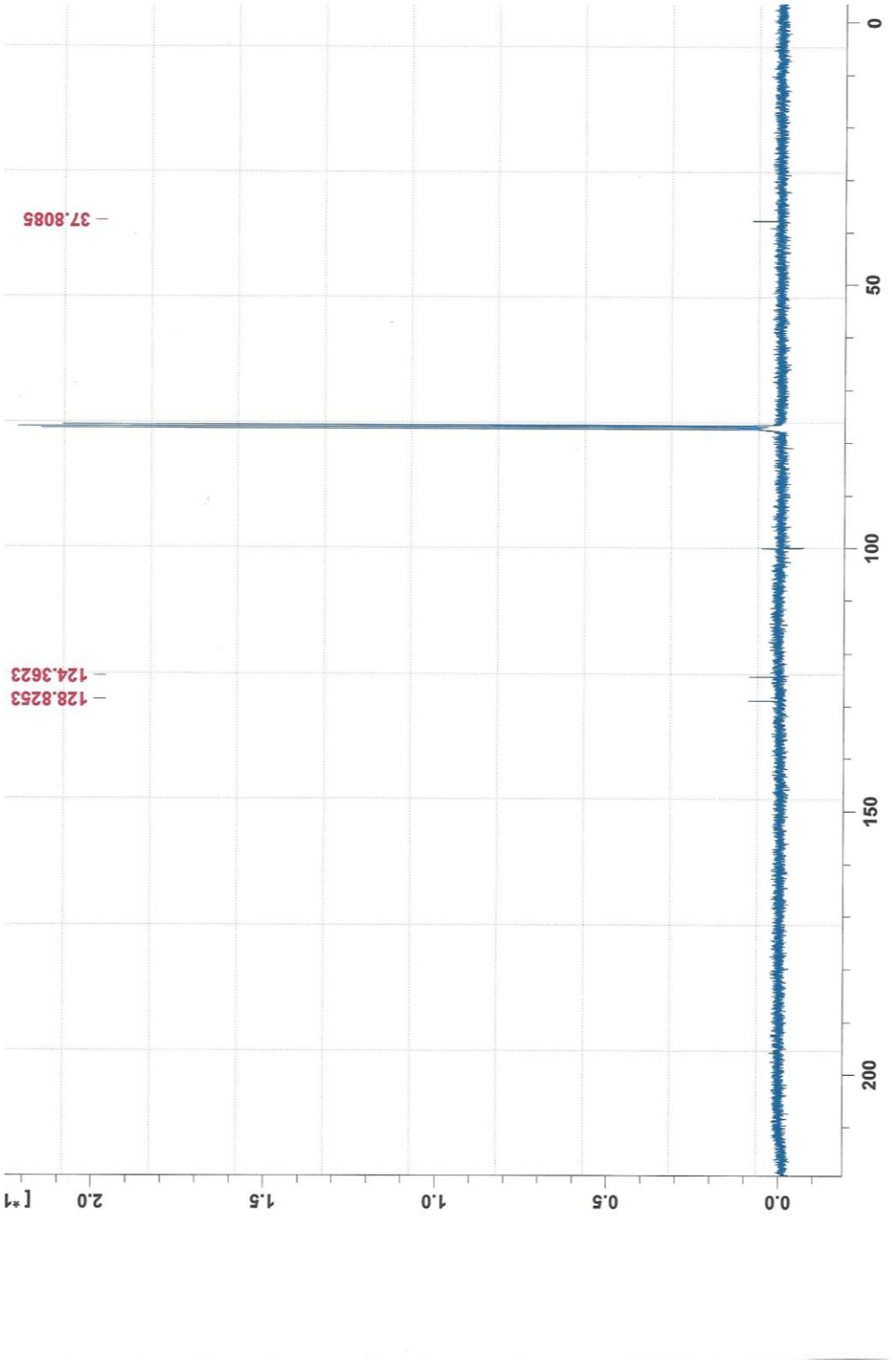

Figure 162: ^{13}C NMR Spectrum of *N,N*-dimethyl-4-nitrobenzenesulfonamide **6**

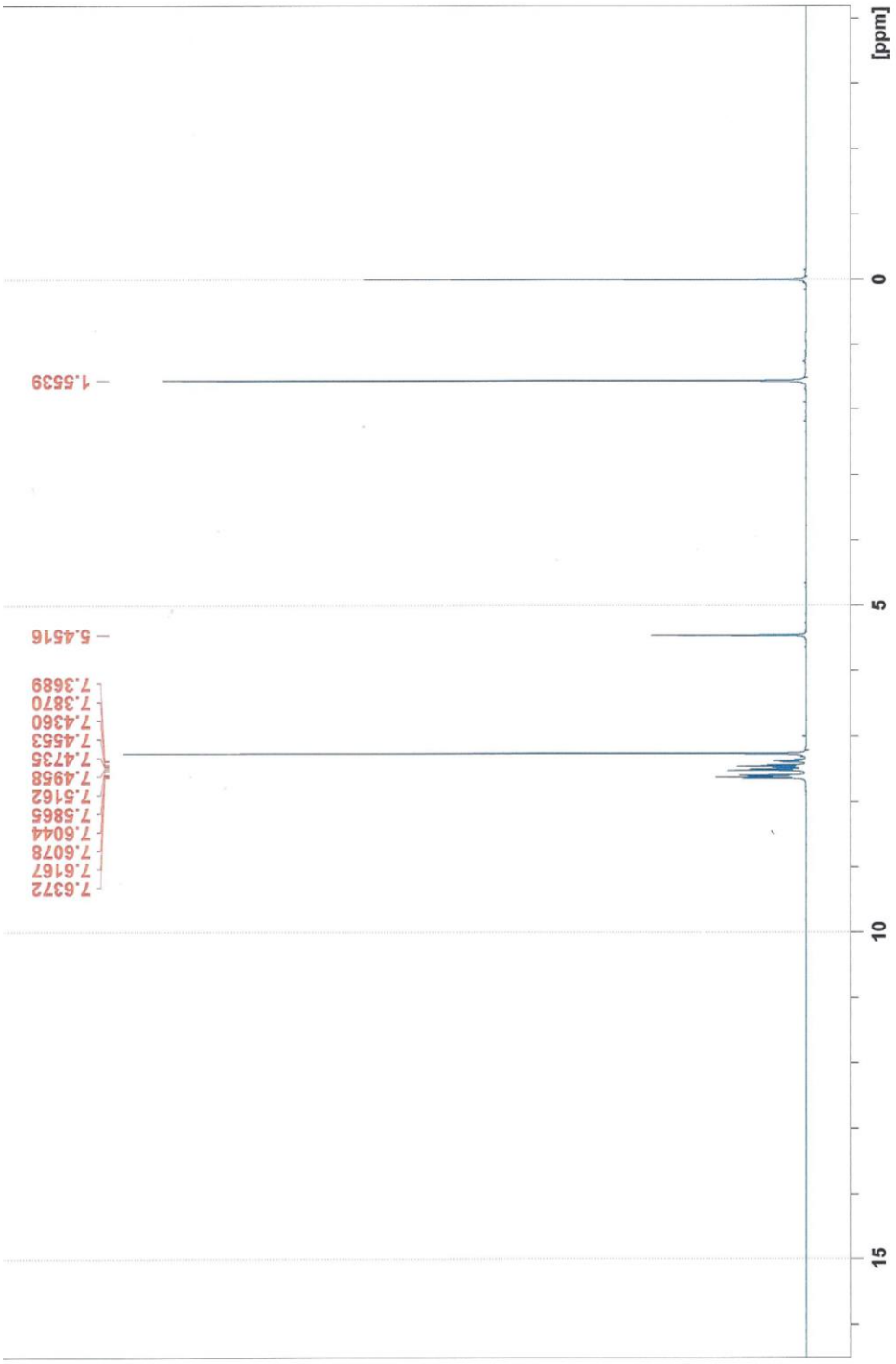

Figure 163: ^{1}H NMR Spectrum of 1-([1,1'-biphenyl]-4-yl)-N,N-dimethylmethanamine **8**

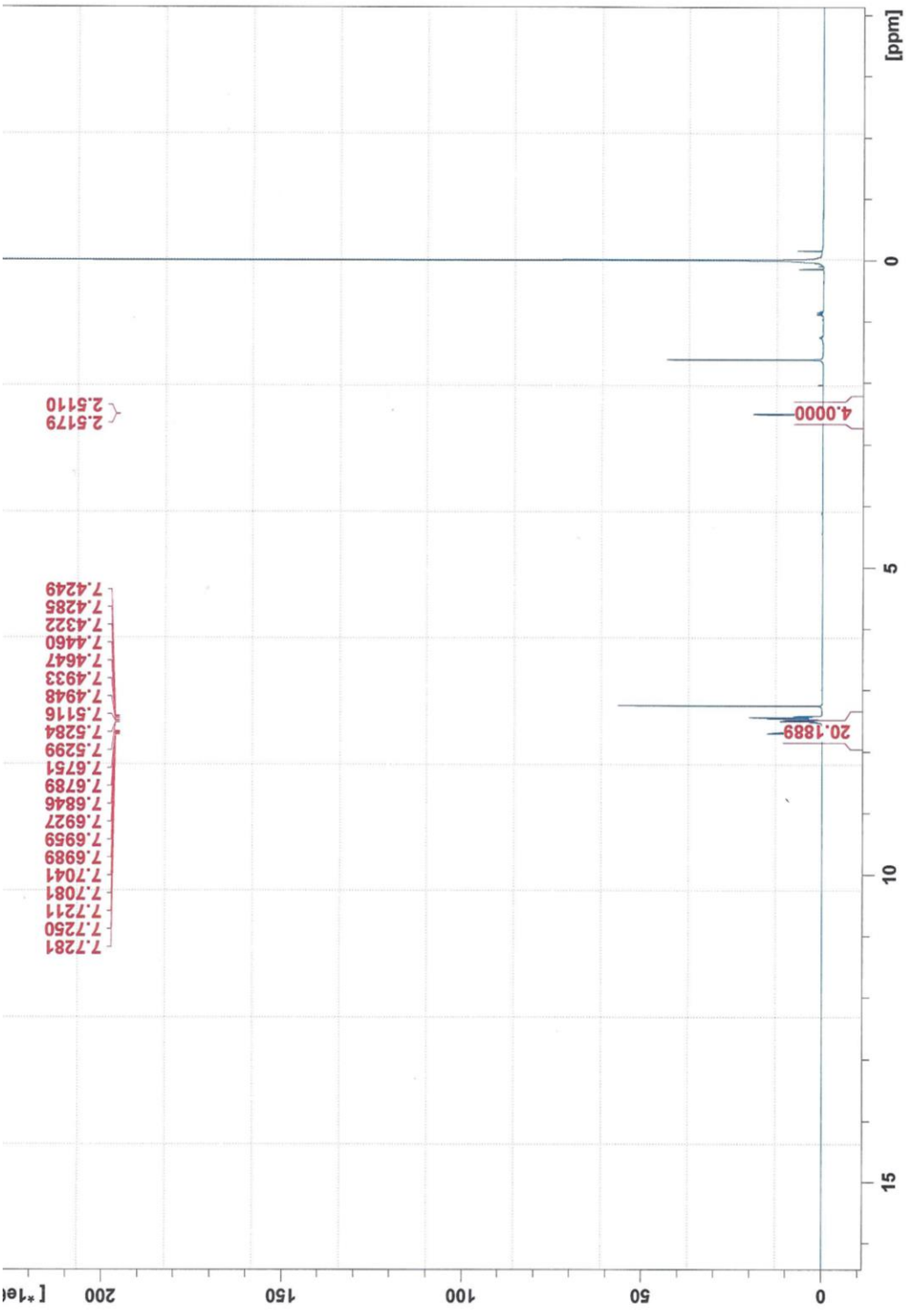

Figure 164: ^{1}H NMR Spectrum of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

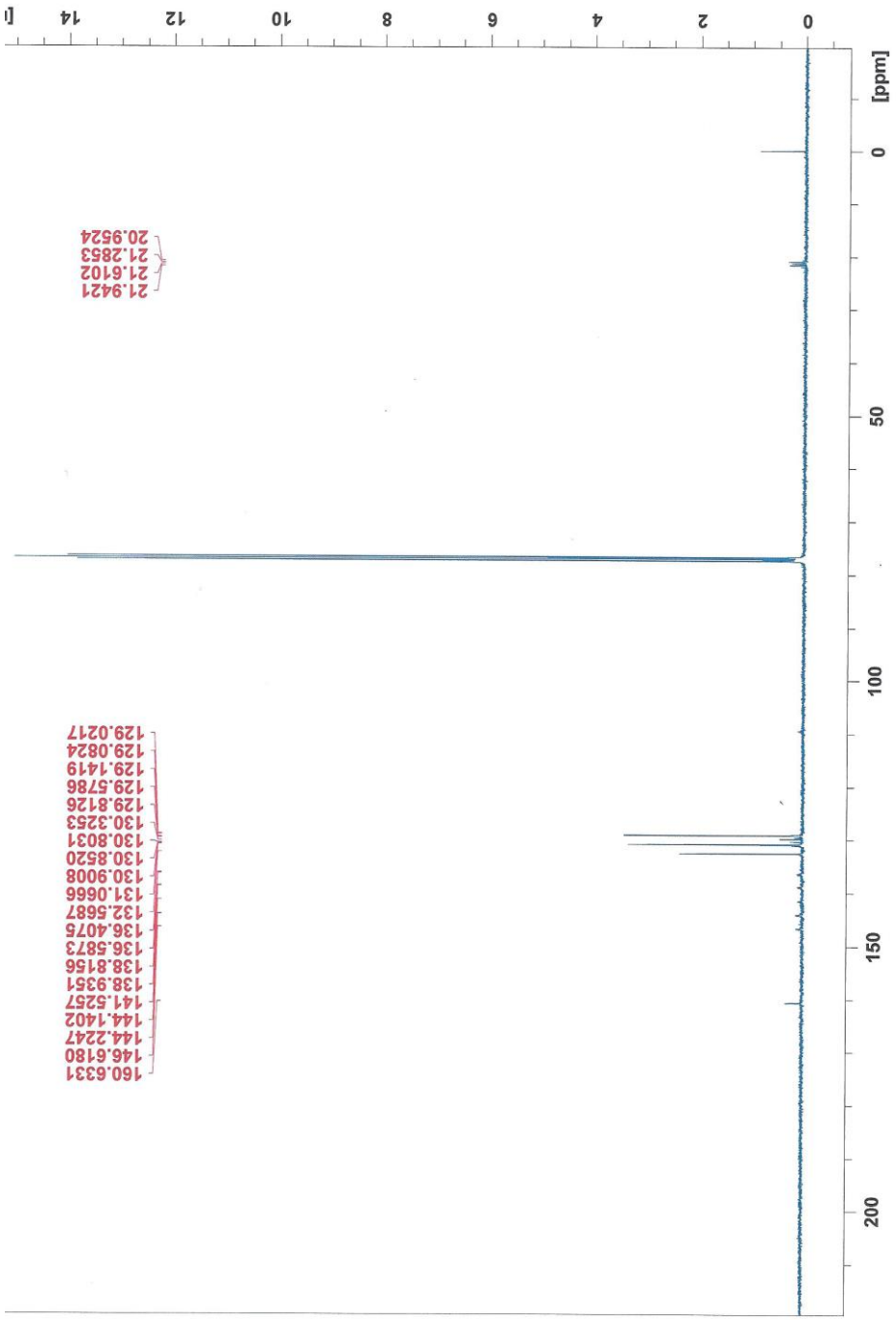

Figure 165: ^{13}C NMR Spectrum of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

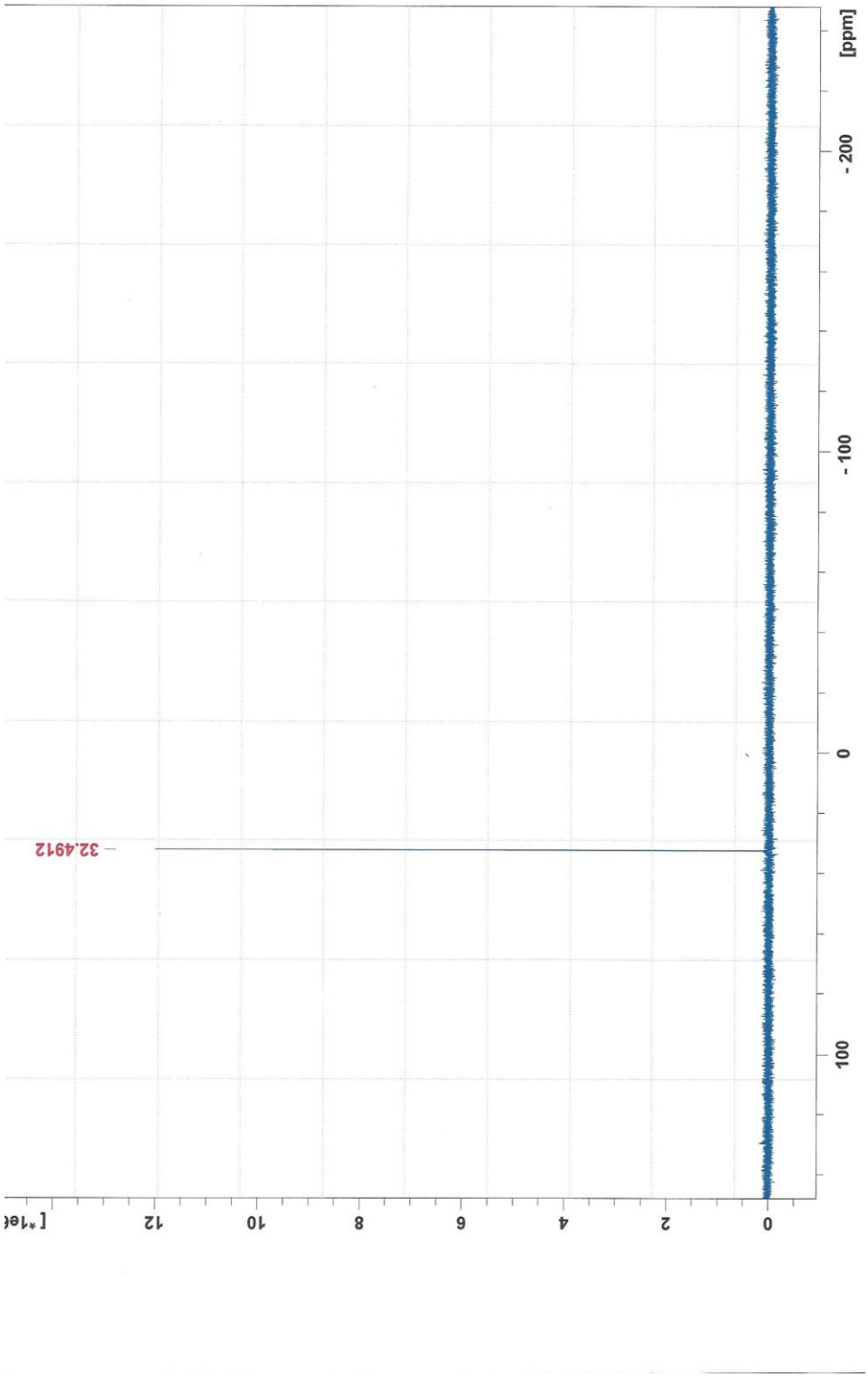

Figure 166: ^{31}P NMR Spectrum of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

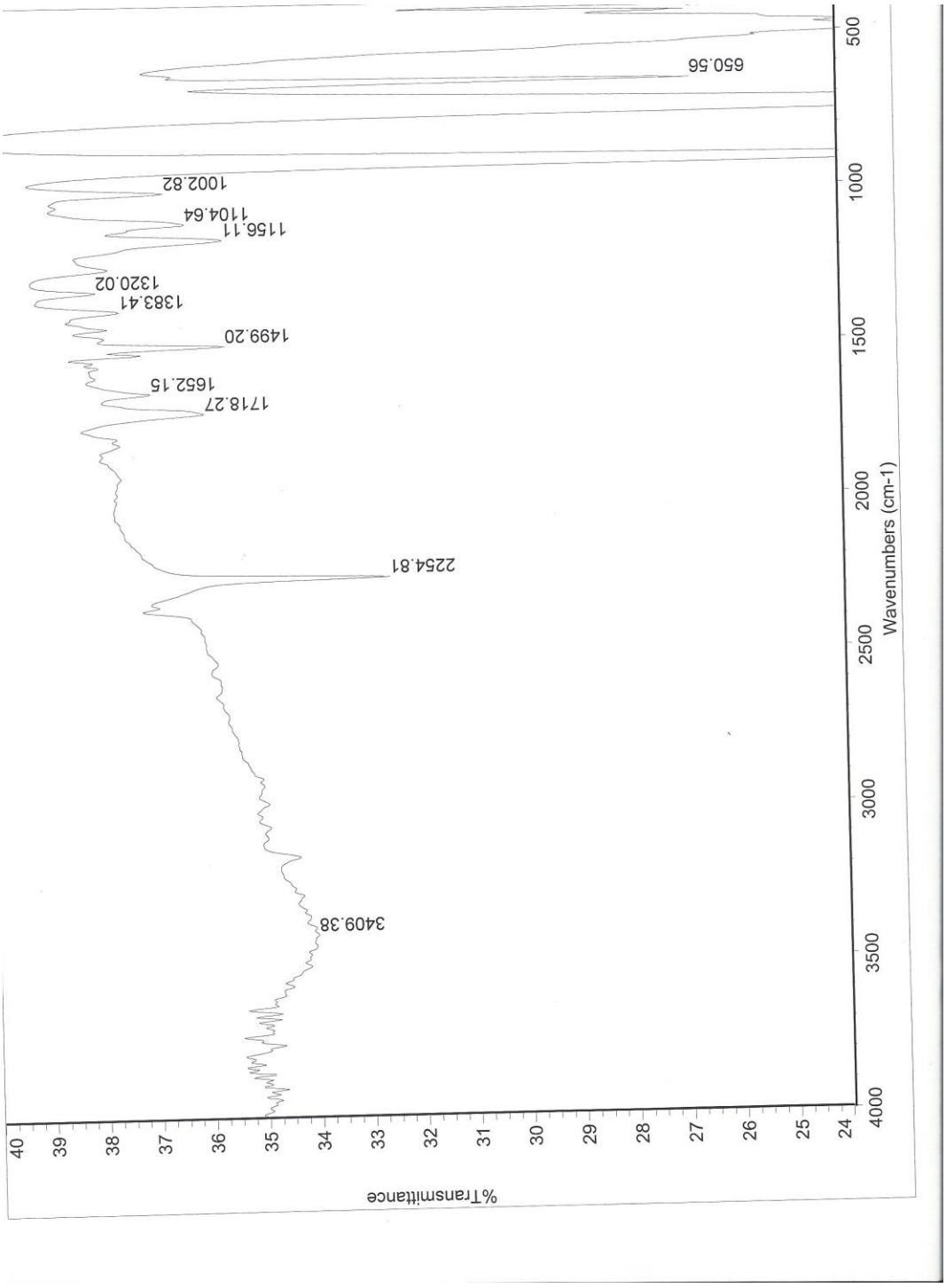

Figure 167: IR Spectrum of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

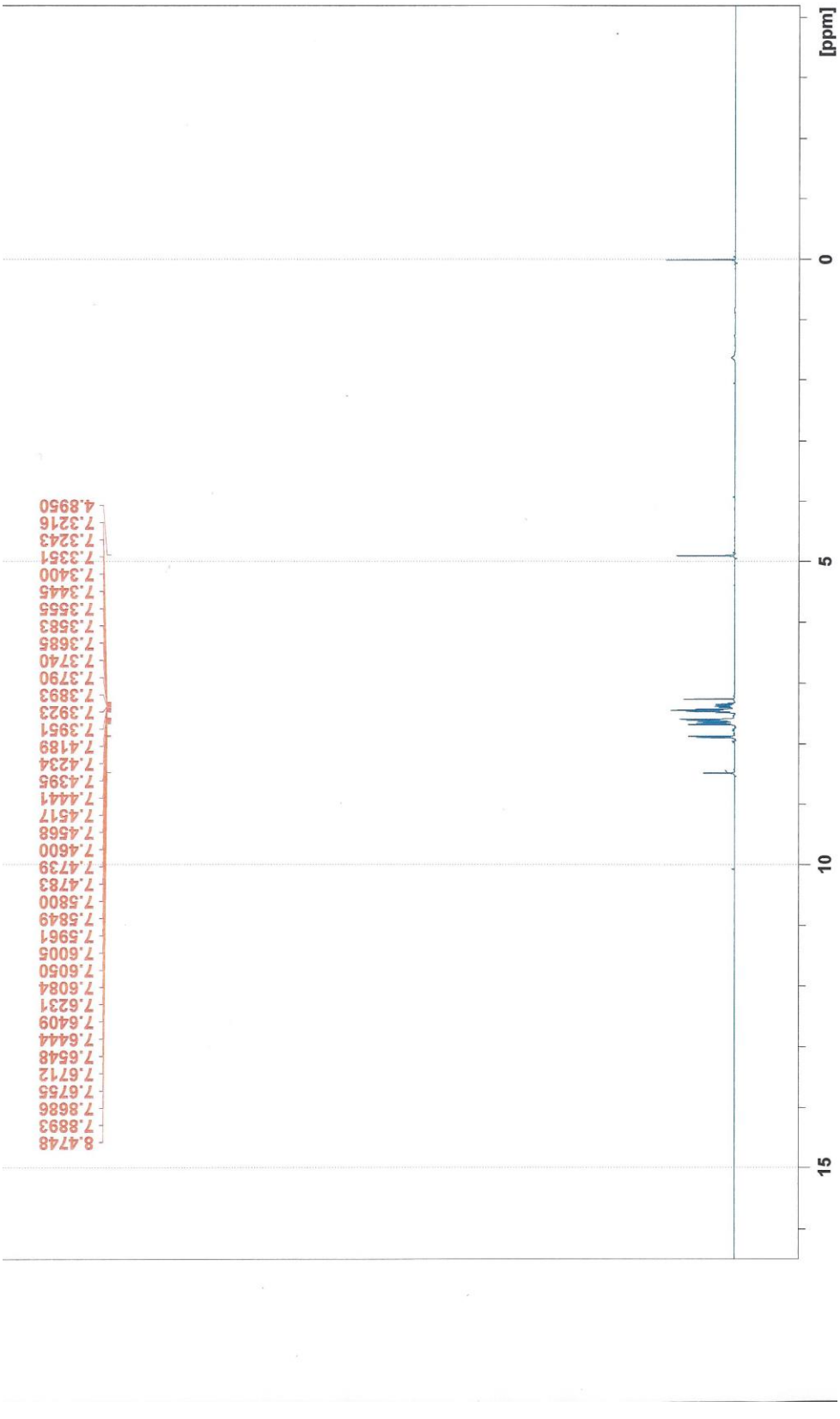

Figure 168: ^{1}H NMR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-[1,1'-biphenyl]-4-carboxamide **7**

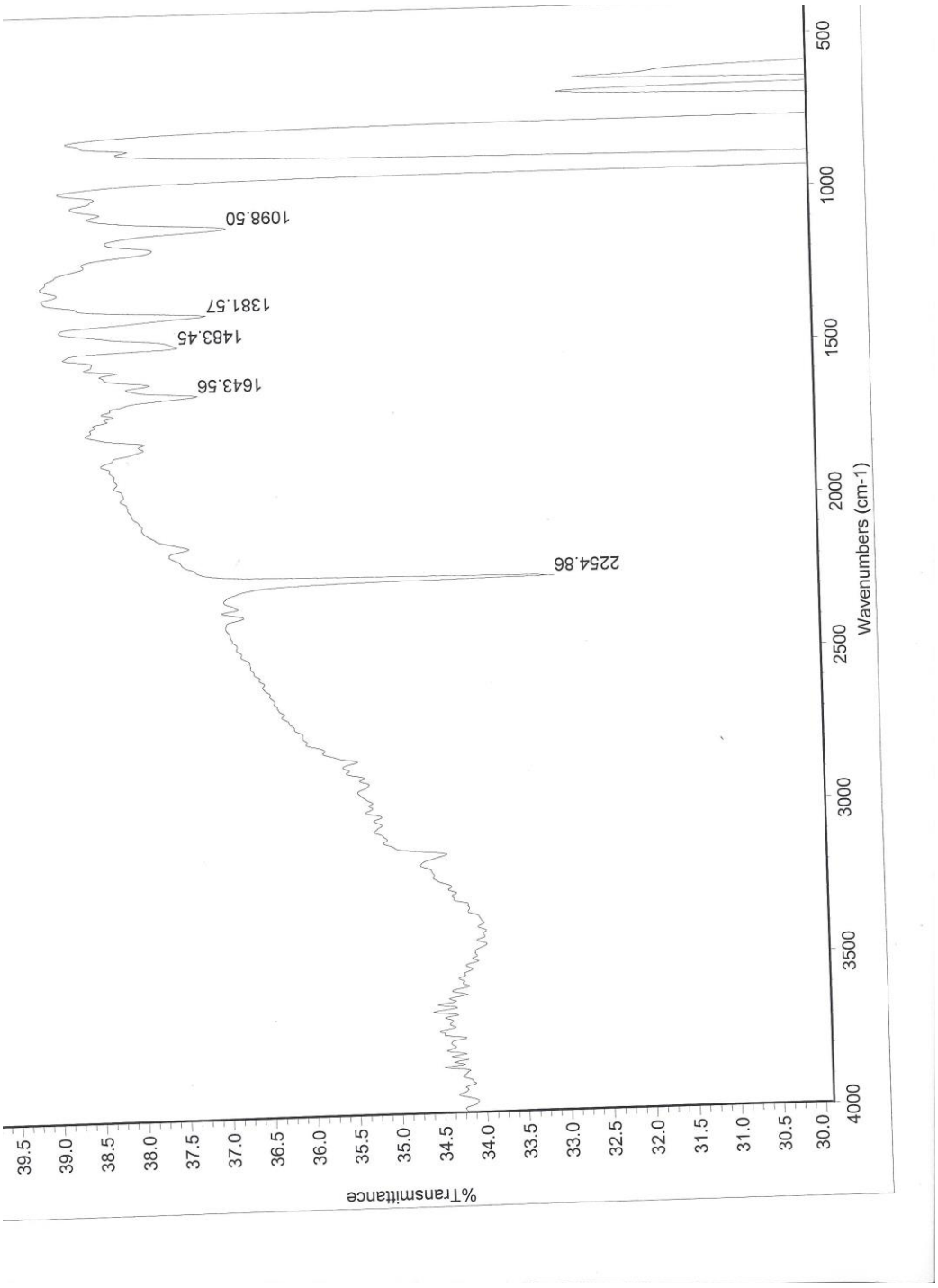

Figure 169: IR Spectrum of *N*-([1,1'-biphenyl]-4-ylmethyl)-[1,1'-biphenyl]-4-carboxamide **7**

IR and NMR SPECTRA of NEOMENTHYL AZIDE PROJECT

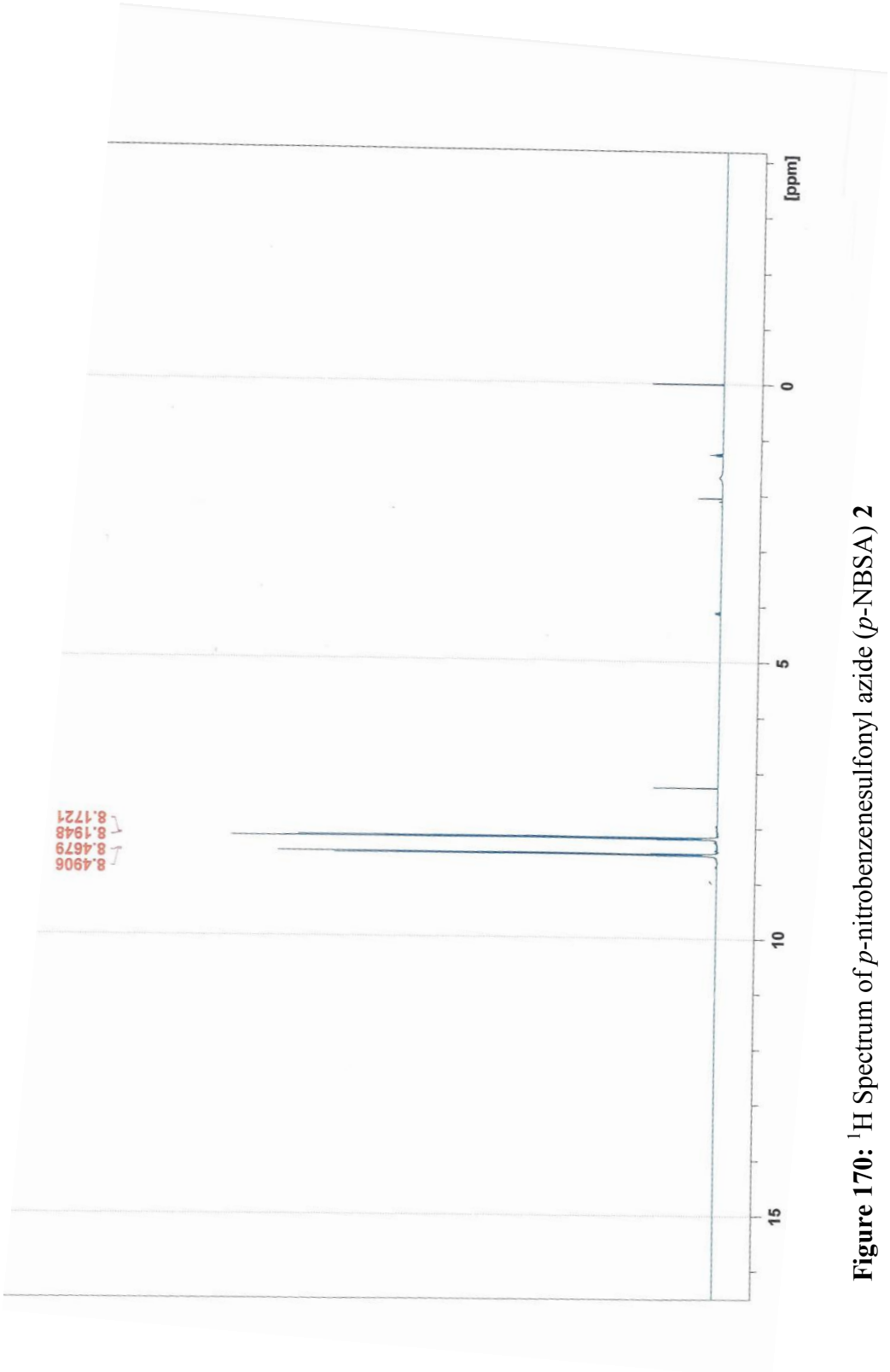

Figure 170: ^{1}H Spectrum of *p*-nitrobenzenesulfonyl azide (*p*-NBSA) **2**

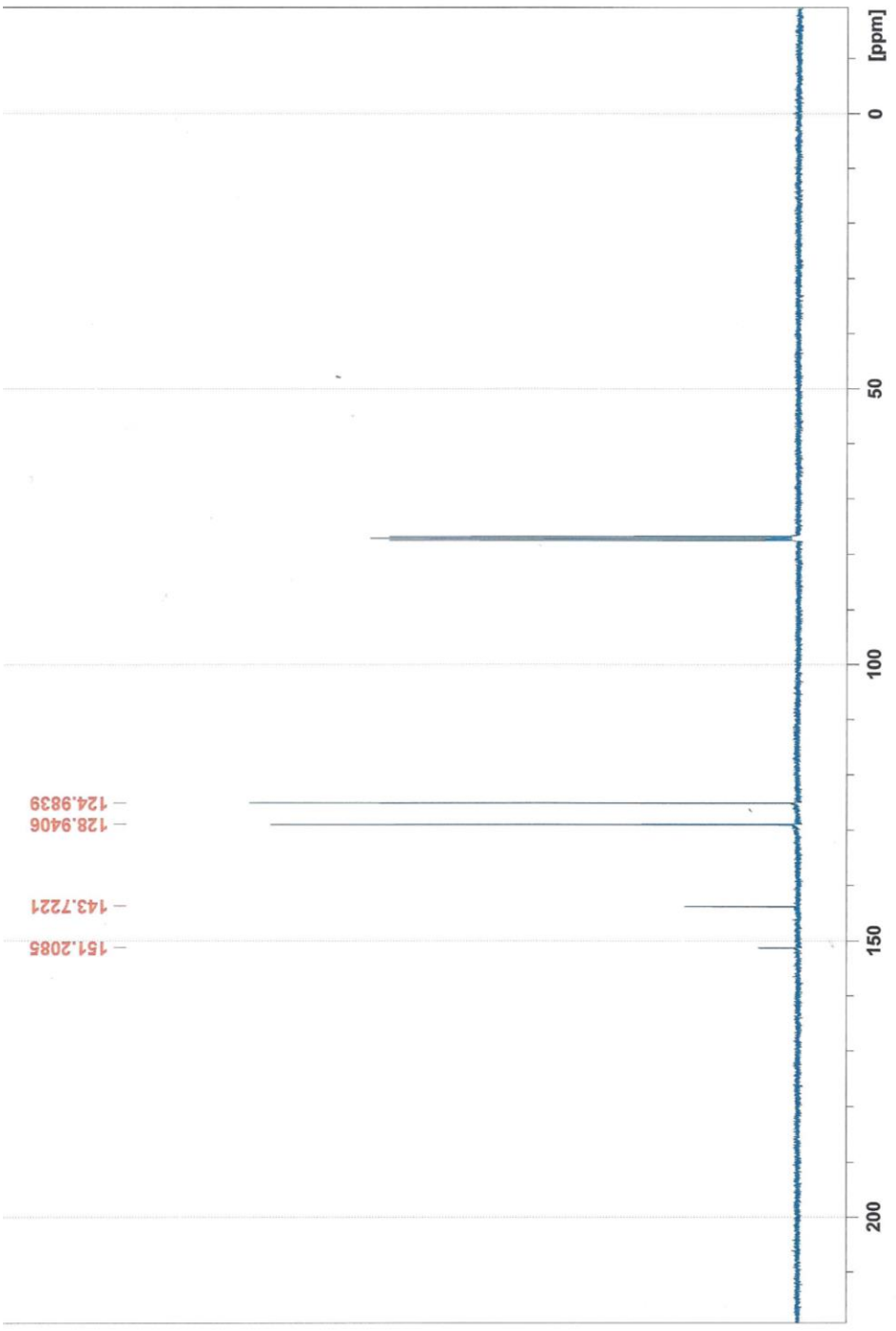

Figure 171: ^{13}C Spectrum of *p*-nitrobenzenesulfonyl azide (*p*-NBSA) **2**

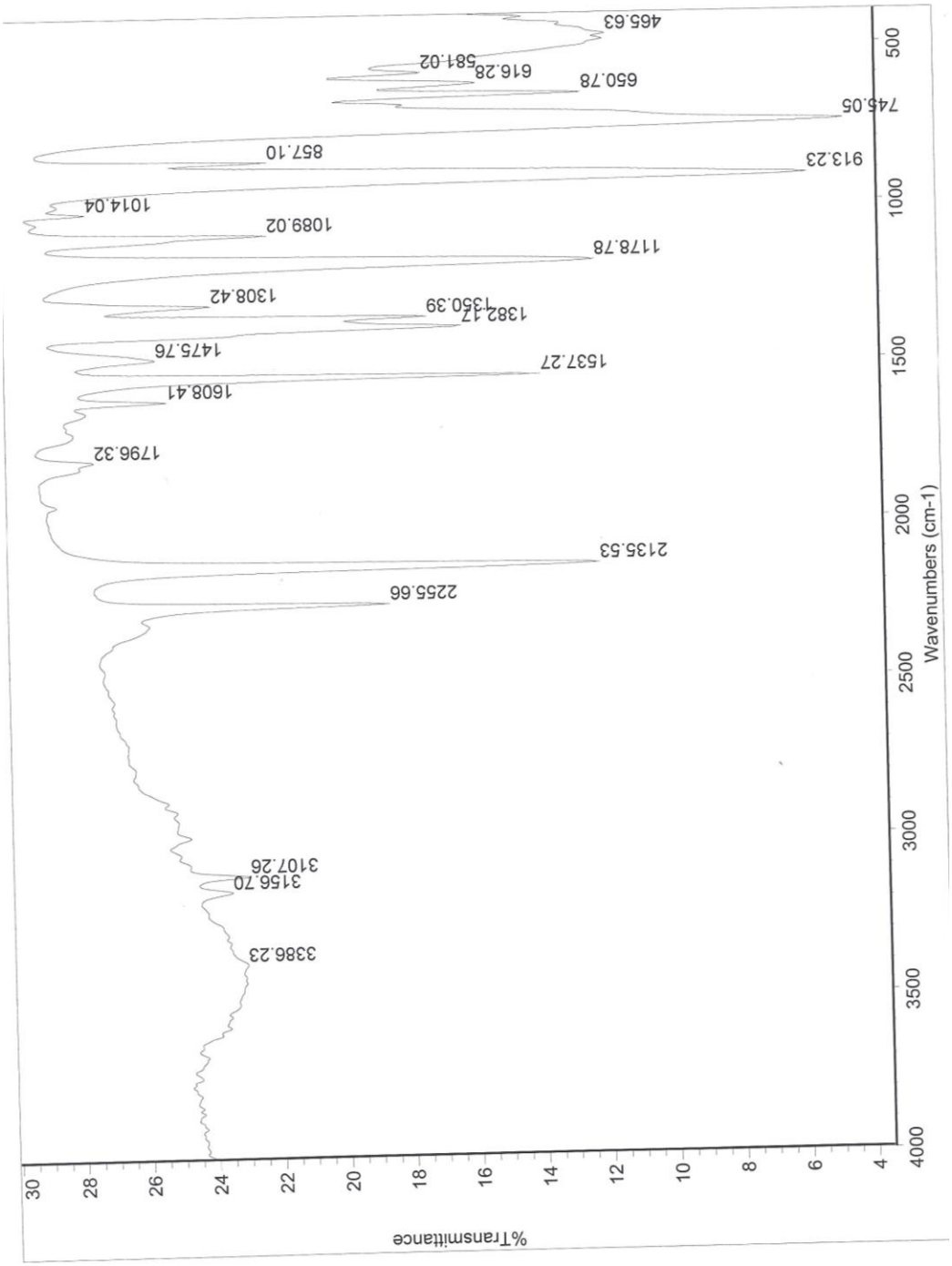

Figure 172: IR Spectrum of *p*-nitrobenzenesulfonyl azide (*p*-NBSA) **2**

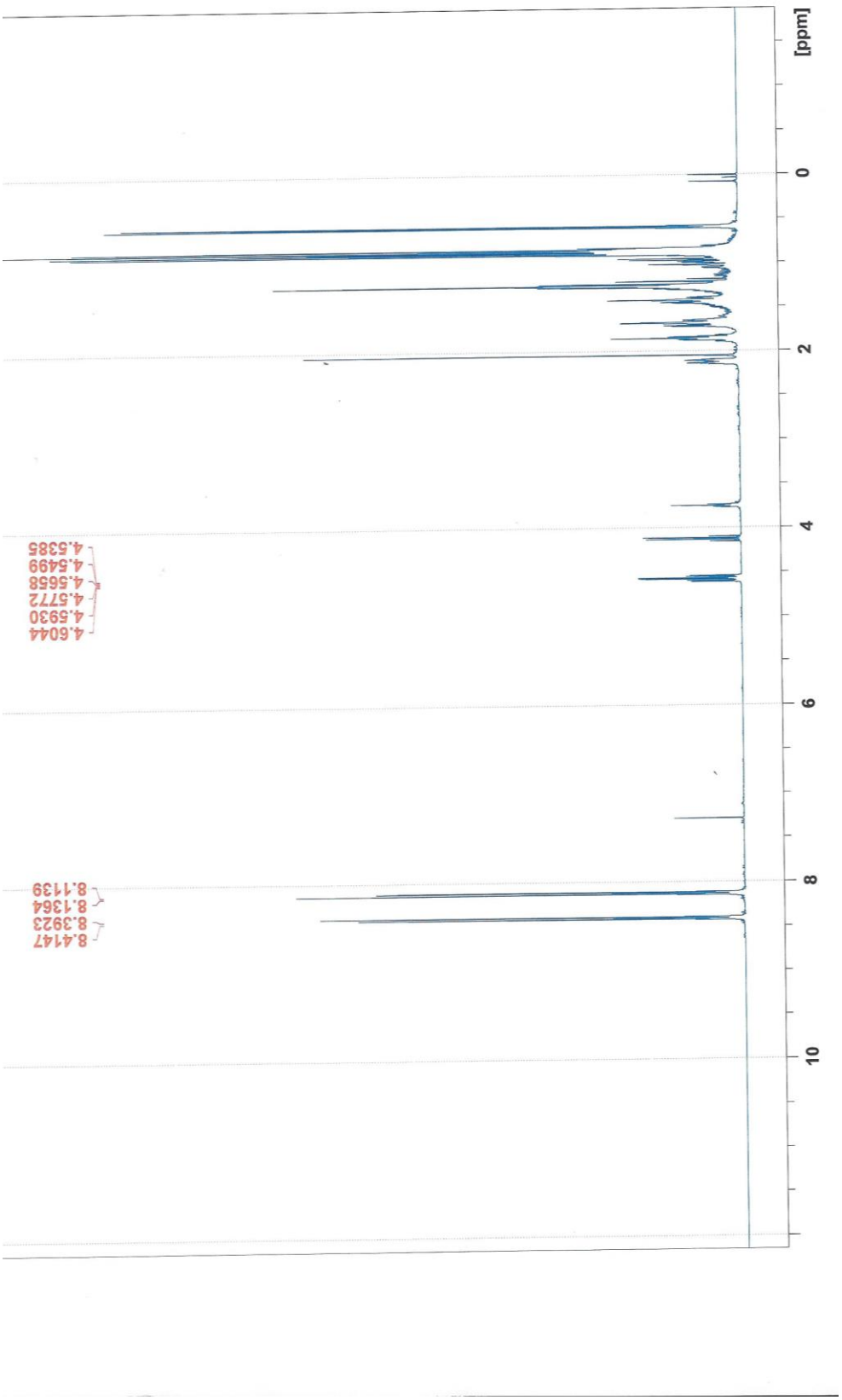

Figure 173: ^{1}H NMR Spectrum of menthyl nosyl ester **60**

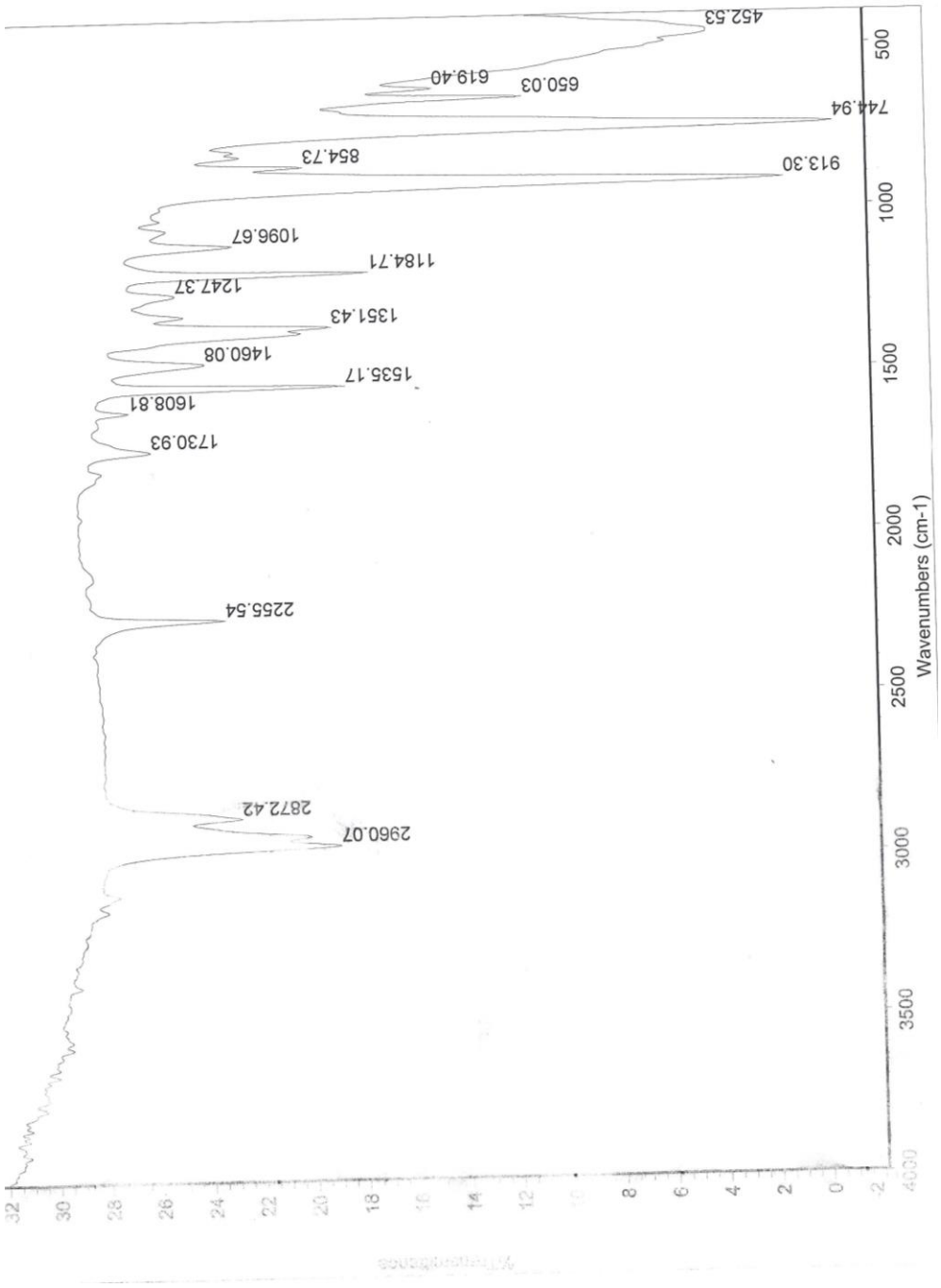

Figure 174: IR Spectrum of menthyl nosyl ester **60**

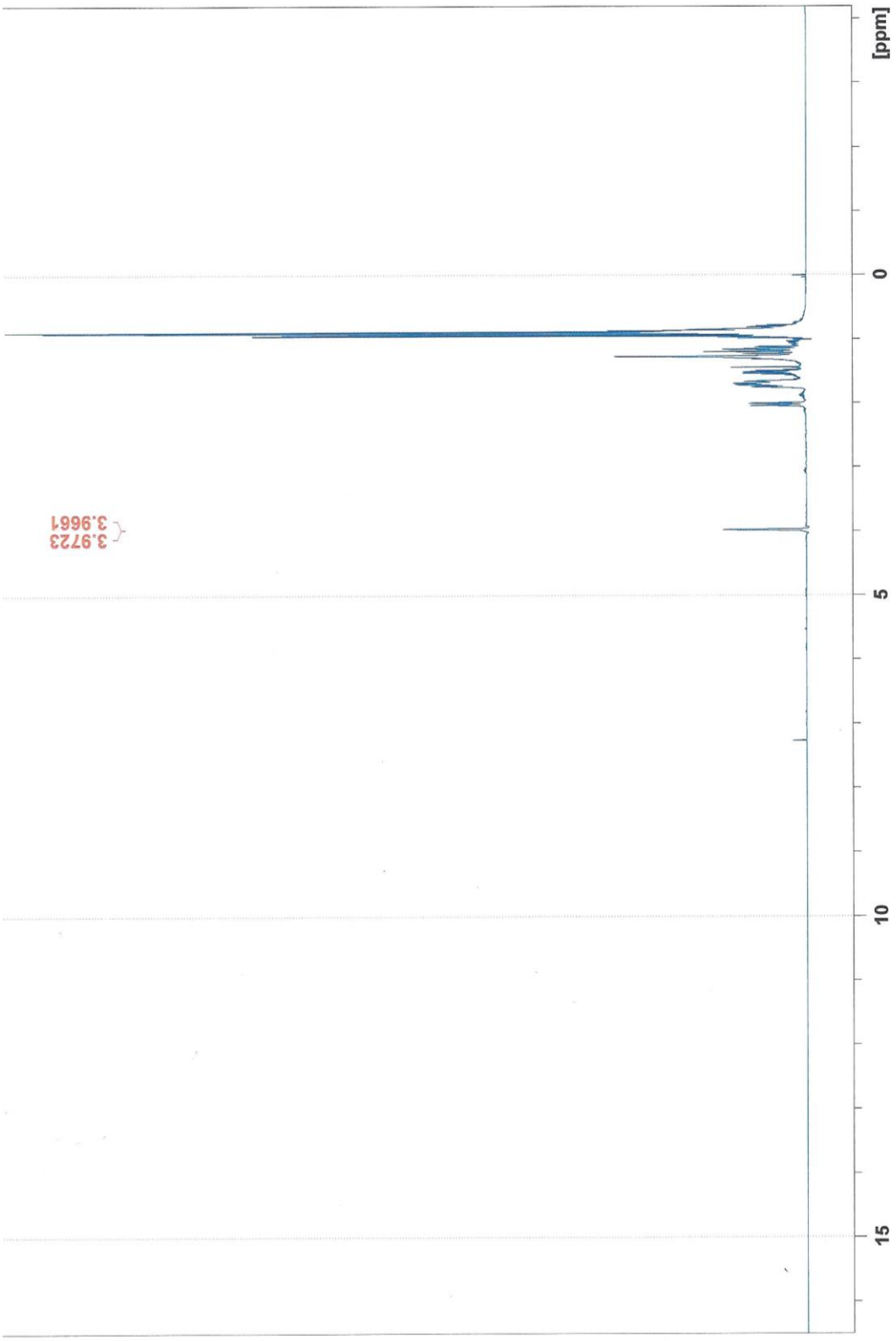

Figure 175: ^{1}H NMR Spectrum of neomenthyl azide **61**

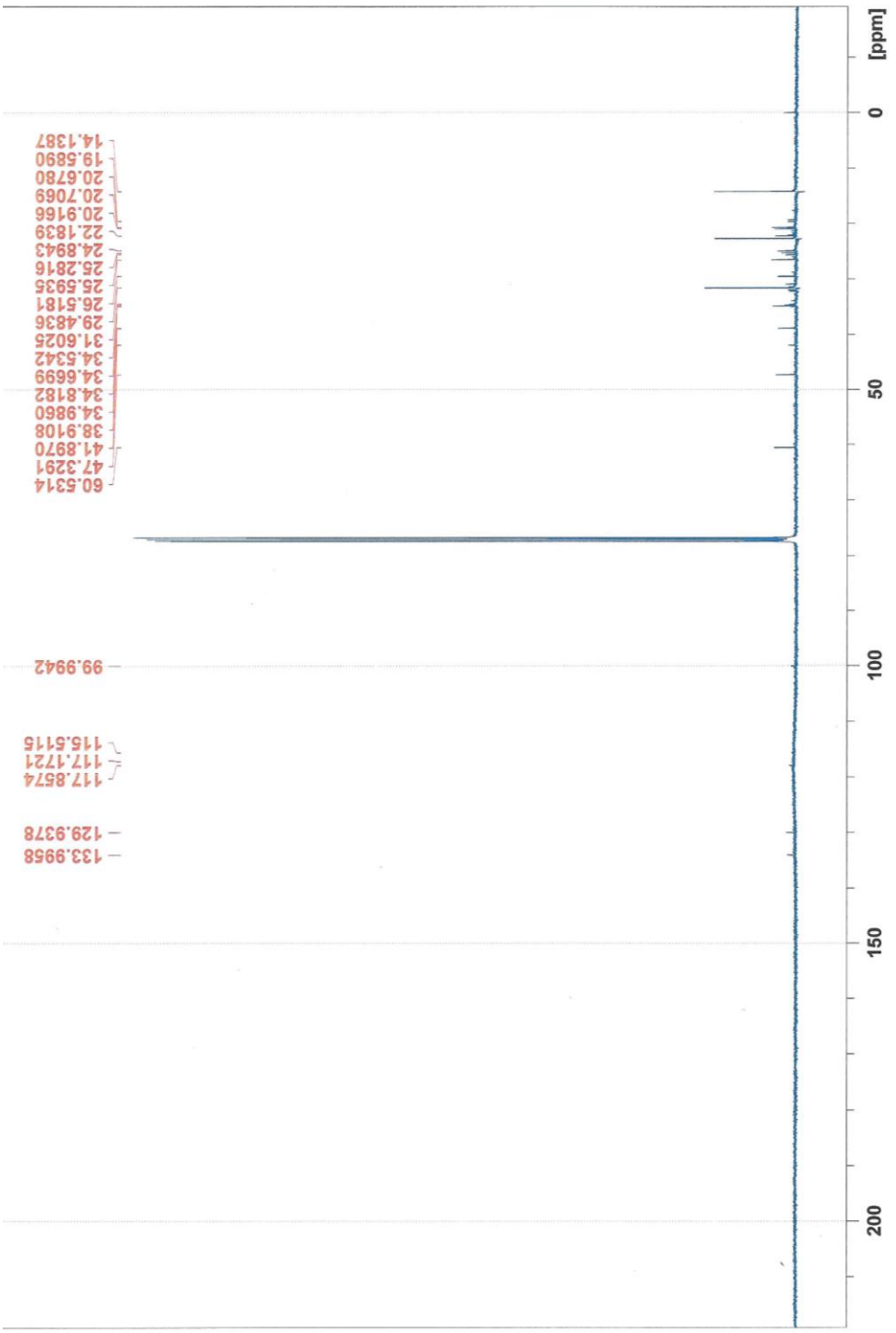

Figure 176: ^{13}C NMR Spectrum of neomenthyl azide **61**

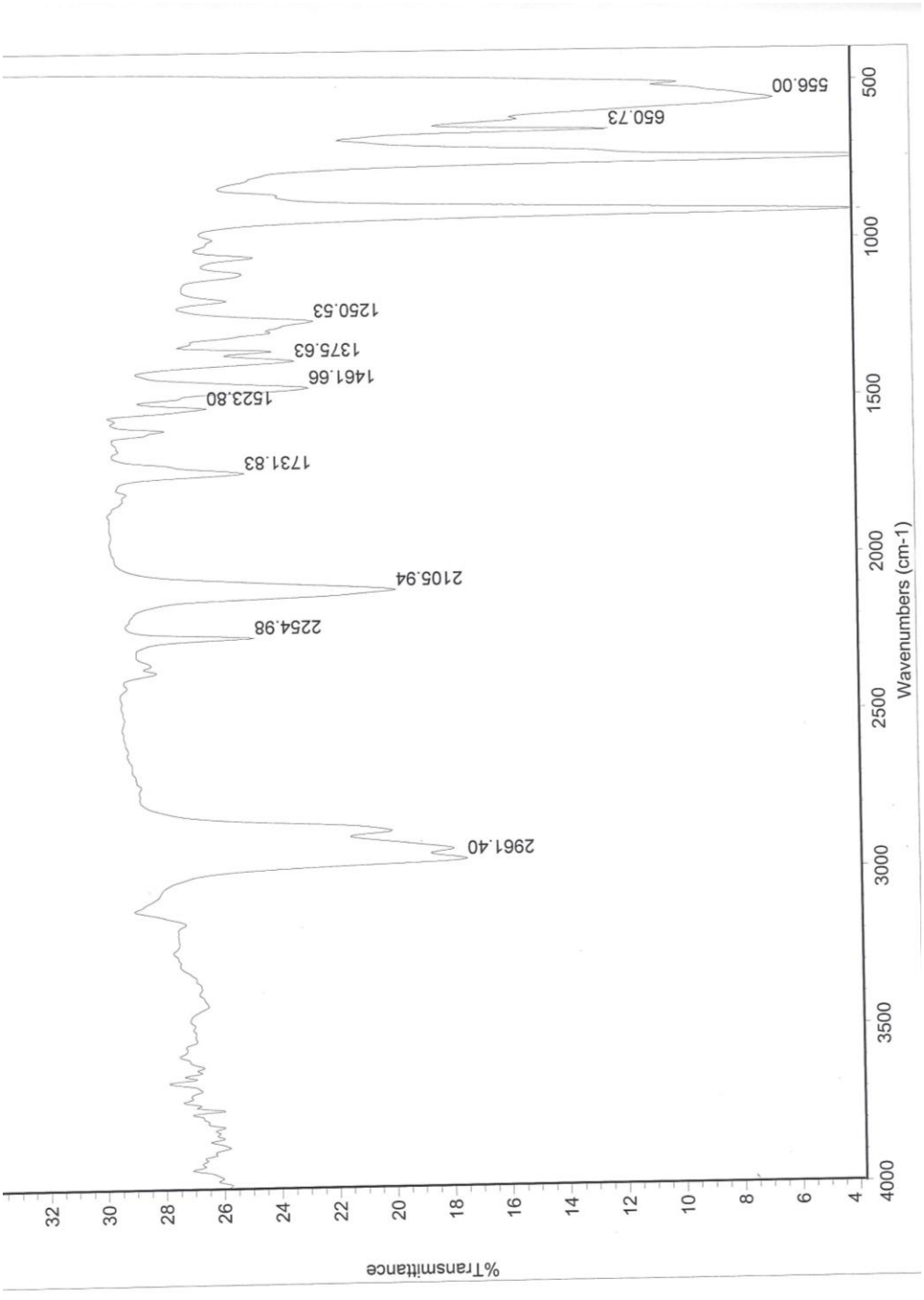

Figure 177: IR Spectrum of neomenthyl azide **61**

Appendix B

X-Ray Crystallography

Figure 178: X-Ray Crystal Structure of *N*-([1,1'-biphenyl]-4-ylmethyl)furan-2-carboxamide **35**

Table 1. Experimental details. **35**

Crystal data-	
Chemical formula	$C_{18}H_{15}NO_2$
M_r	277.31
Crystal system, space group	Triclinic, *P*1
Temperature (K)	100
a, *b*, *c* (Å)	cellsetting:triclinic 9.8399 (8), 10.0468 (8), 15.4115 (13)
α, β, γ (°)	cellsetting:triclinic 98.900 (3), 97.295 (4), 112.119 (3)
V (Å^3)	1365.6 (2)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.09
Crystal size (mm)	0.41 × 0.19 × 0.05
Data collection	
Diffractometer	Bruker AXS D8 Quest CMOS diffractometer
Absorption correction	Multi-scan TWINABS (Sheldrick, 2012)
T_{min}, T_{max}	0.679, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	49282, 8212, 6673
R_{int}	0.041
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.715
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.057, 0.132, 1.03
No. of reflections	8212
No. of parameters	380
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.59, −0.31

Computer programs: Apex2 v2014.1-1 (Bruker, 2014), *SAINT* V8.34A (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2013), SHELXLE Rev656 (Hübschle *et al.*, 2011

Table 2. Bond Lengths (Å). **35**

O1A—C1A	1.237(2)
O2A—C5A	1.369(2)
O2A—C2A	1.374(2)
N1A—C1A	1.343(2)
N1A—C6A	1.4619(19)
N1A—H1A	0.8800
C1A—C2A	1.476(2)
C2A—C3A	1.356(2)
C3A—C4A	1.436(3)
C3A—H3A	0.9500
C4A—C5A	1.337(3)
C4A—H4A	0.9500
C5A—H5A	0.9500
C6A—C7A	1.511(2)
C6A—H6AA	0.9900
C6A—H6AB	0.990
C7A—C12A	1.389(2)
C7A—C8A	1.394(2)
C8A—C9A	1.389(2)
C8A—H8A	0.9500
C9A—C10A	1.405(2)
C9A—H9A	0.9500
C10A—C11A	1.398(2)
C10A—C13A	1.490(2)
C11A—C12A	1.393(2)
C11A—H11A	0.9500
C12A—H12A	0.9500
C13A—C14A	1.402(2)
C13A—C18A	1.407(2)
C14A—C15A	1.390(2)
C14A—H14A	0.9500
C15A—C16A	1.392(2)
C15A—H15A	0.9500
C16A—C17A	1.389(2)
C16A—H16A	0.9500
C17A—C18A	1.392(2)
C17A—H17A	0.9500
C18A—H18A	0.9500
O1B—C2B	1.3684(19)
O1B—C5B	1.3699(19)
O2B—C1B	1.2398(19)
N1B—C1B	1.3408(19)
N1B—C6B	1.4567(19)
N1B—H1B	0.8800
C1B—C2B	1.475(2)
C2B—C3B	1.355(2)
C3B—C4B	1.428(2
C3B—H3B	0.9500
C4B—C5B	1.346(3)
C4B—H4B	0.9500
C5B—H5B	0.9500
C6B—C7B	1.513(2)
C6B—H6BA	0.9900
C6B—H6BB	0.9900
C7B—C12B	1.388(2)
C7B—C8B	1.395(2)
C8B—C9B	1.388(2)
C8B—H8B	0.9500
C9B—C10B	1.401(2)
C9B—H9B	0.9500
C10B—C11B	1.399(2)
C10B—C13B	1.489(2)
C11B—C12B	1.393(2)
C11B—H11B	0.9500
C12B—H12B	0.9500
C13B—C14B	1.401(2)
C13B—C18B	1.404(2)
C14B—C15B	1.393(2)
C14B—H14B	0.9500
C15B—C16B	1.390(2)
C15B—H15B	0.9500
C16B—C17B	1.389(2)
C16B—H16B	0.9500
C17B—C18B	1.393(2)
C17B—H17B	0.9500
C18B—H18B	0.9500

Table 3. Bond Angles (deg). 35

C5A—O2A—C2A	106.28 (14)	C2B—O1B—C5B	106.17 (13)
C1A—N1A—C6A	122.53 (14)	C1B—N1B—C6B	120.97 (13)
C1A—N1A—H1A	118.7	C1B—N1B—H1B	119.5
C6A—N1A—H1A	118.7	C6B—N1B—H1B	119.5
O1A—C1A—N1A	124.25 (15)	O2B—C1B—N1B	123.52 (14)
O1A—C1A—C2A	120.21 (15)	O2B—C1B—C2B	119.59 (14)
N1A—C1A—C2A	115.52 (14)	N1B—C1B—C2B	116.88 (14)
C3A—C2A—O2A	110.06 (15)	C3B—C2B—O1B	110.30 (14)
C3A—C2A—C1A	130.93 (16)	C3B—C2B—C1B	131.13 (15)
O2A—C2A—C1A	118.96 (13)	O1B—C2B—C1B	118.57 (13)
C2A—C3A—C4A	106.14 (16)	C2B—C3B—C4B	106.43 (15)
C2A—C3A—H3A	126.9	C2B—C3B—H3B	126.8
C4A—C3A—H3A	126.9	C4B—C3B—H3B	126.8
C5A—C4A—C3A	106.59 (16)	C5B—C4B—C3B	106.31 (14)
C5A—C4A—H4A	126.7	C5B—C4B—H4B	126.8
C3A—C4A—H4A	126.7	C3B—C4B—H4B	126.8
C4A—C5A—O2A	110.93 (17)	C4B—C5B—O1B	110.78 (15)
C4A—C5A—H5A	124.5	C4B—C5B—H5B	124.6
O2A—C5A—H5A	124.5	O1B—C5B—H5B	124.6
N1A—C6A—C7A	114.33 (13)	N1B—C6B—C7B	114.97 (12)
N1A—C6A—H6AA	108.7	N1B—C6B—H6BA	108.5
C7A—C6A—H6AA	108.7	C7B—C6B—H6BA	108.5
N1A—C6A—H6AB	108.7	N1B—C6B—H6BB	108.5
C7A—C6A—H6AB	108.7	C7B—C6B—H6BB	108.5

H6AA—C6A—H6A	107.6	H6BA—C6B—H6BB	107.5
C12A—C7A—C8A	118.25 (14)	C12B—C7B—C8B	118.25 (14)
C12A—C7A—C6A	119.82 (14)	C12B—C7B—C6B	123.31 (13)
C8A—C7A—C6A	121.87 (14)	C8B—C7B—C6B	118.44 (13)
C9A—C8A—C7A	120.81 (14)	C9B—C8B—C7B	121.20 (14)
C9A—C8A—H8A	119.6	C9B—C8B—H8B	119.4
C7A—C8A—H8A	119.6	C7B—C8B—H8B	119.4
C8A—C9A—C10A	121.36 (15)	C8B—C9B—C10B	120.96 (14)
C8A—C9A—H9A	119.3	C8B—C9B—H9B	119.5
C10A—C9A—H9A	119.3	C10B—C9B—H9B	119.5
C11A—C10A—C9A	117.29 (14)	C11B—C10B—C9B	117.42 (14)
C11A—C10A—C13A	121.37 (14)	C11B—C10B—C13B	121.56 (13)
C9A—C10A—C13A	121.33 (14)	C9B—C10B—C13B	121.01 (13)
C12A—C11A—C10A	121.13 (15)	C12B—C11B—C10B	121.47 (14)
C12A—C11A—H11A	119.4	C12B—C11B—H11B	119.3
C10A—C11A—H11A	119.4	C10B—C11B—H11B	119.3
C7A—C12A—C11A	121.11 (15)	C7B—C12B—C11B	120.69 (14)
C7A—C12A—H12A	119.4	C7B—C12B—H12B	119.7
C11A—C12A—H12A	119.4	C11B—C12B—H12B	119.7
C14A—C13A—C18A	117.55 (14)	C14B—C13B—C18B	117.85 (14)
C14A—C13A—C10A	121.24 (14)	C14B—C13B—C10B	121.47 (13)
C18A—C13A—C10A	121.21 (14)	C18B—C13B—C10B	120.68 (14)
C15A—C14A—C13A	121.41 (15)	C15B—C14B—C13B	121.01 (15)
C15A—C14A—H14A	119.3	C15B—C14B—H14B	119.5
C13A—C14A—H14A	119.3	C13B—C14B—H14B	119.5
C14A—C15A—C16A	120.16 (16)	C16B—C15B—C14B	120.36 (16)

C14A—C15A—H15A 119.9	C16B—C15B—H15B 119.8
C16A—C15A—H15A 119.9	C14B—C15B—H15B 119.8
C17A—C16A—C15A 119.45 (15)	C17B—C16B—C15B 119.45 (15)
C17A—C16A—H16A 120.3	C17B—C16B—H16B 120.3
C15A—C16A—H16A 120.3	C15B—C16B—H16B 120.3
C16A—C17A—C18A 120.37 (15)	C16B—C17B—C18B 120.27 (15)
C16A—C17A—H17A 119.8	C16B—C17B—H17B 119.9
C18A—C17A—H17A 119.8	C18B—C17B—H17B 119.9
C17A—C18A—C13A 121.05 (15)	C17B—C18B—C13B 121.05 (15)
C17A—C18A—H18A 119.5	C17B—C18B—H18B 119.5
C13A—C18A—H18A 119.5	C13B—C18B—H18B 119.5
C6A—N1A—C1A—O1A5.7 (2)	C6B—N1B—C1B—O2B 3.9 (2)
C6A—N1A—C1A—C2A−173.12 (13)	C6B—N1B—C1B—C2B −175.19 (13)
C5A—O2A—C2A—C3A0.38 (18)	C5B—O1B—C2B—C3B 0.64 (18)
C5A—O2A—C2A—C1A178.07 (14)	C5B—O1B—C2B—C11 79.94 (13)
O1A—C1A—C2A—C3A−20.8 (3)	O2B—C1B—C2B—C3 −8.8 (3)
N1A—C1A—C2A—C3A158.09 (17)	N1B—C1B—C2B—C3 170.37 (16)
O1A—C1A—C2A—O2A162.11 (14)	O2B—C1B—C2B—O1 172.09 (14)
N1A—C1A—C2A—O2A−19.0 (2)	N1B—C1B—C2B—O1B−8.8 (2)
O2A—C2A—C3A—C4A−0.03 (19)	O1B—C2B—C3B—C4 −0.44 (18)
C1A—C2A—C3A—C4A−177.36 (16)	C1B—C2B—C3B—C4 −179.63 (15)
C2A—C3A—C4A—C5A−0.3 (2)	C2B—C3B—C4B—C5 0.07 (19)
C3A—C4A—C5A—O2A0.6 (2)	C3B—C4B—C5B—O1B0.3 (2)
C2A—O2A—C5A—C4A−0.6 (2)	C2B—O1B—C5B—C4B−0.59 (19)
C1A—N1A—C6A—C7A−100.95 (18)	C1B—N1B—C6B—C7B−82.71 (18)
N1A—C6A—C7A—C12A−131.14 (16)	N1B—C6B—C7B—C12B −14.8 (2)

N1A—C6A—C7A—C8A	51.5 (2)	N1B—C6B—C7B—C8B	165.42 (14)
C12A—C7A—C8A—C9A	−1.6 (2)	C12B—C7B—C8B—C9B	1.2 (2)
C6A—C7A—C8A—C9A	175.80 (15)	C6B—C7B—C8B—C9B	−179.09 (15)
C7A—C8A—C9A—C10A	−0.5 (2)	C7B—C8B—C9B—C10B	−.2 (3)
C8A—C9A—C10A—C11A	1.9 (2)	C8B—C9B—C10B—C11B	−0.9 (2)
C8A—C9A—C10A—C13A	−177.30 (14)	C8B—C9B—C10B—C13B1	77.75 (15)
C9A—C10A—C11A—C12A	−1.2 (2)	C9B—C10B—C11B—C12B	0.9 (2)
C13A—C10A—C11A—C12A	177.92 (15)	C13B—C10B—C11B—C12B	−177.72 (15)
C8A—C7A—C12A—C11A	2.2 (2)	C8B—C7B—C12B—C11B	−1.1 (2)
C6A—C7A—C12A—C11A	−175.24 (15)	C6B—C7B—C12B—C11B	179.14 (15)
C10A—C11A—C12A—C7A	−0.8 (3)	C10B—C11B—C12B—C7B	0.1 (3)
C11A—C10A—C13A—C14A	−160.87 (15)	C11B—C10B—C13B—C14B	21.4 (2)
C9A—C10A—C13A—C14A	18.2 (2)	C9B—C10B—C13B—C14B	−157.14 (15)
C11A—C10A—C13A—C18A	18.6 (2)	C11B—C10B—C13B—C18B	−159.27 (15)
C9A—C10A—C13A—C18A	−162.24 (14)	C9B—C10B—C13B—C18B	22.2 (2)
C18A—C13A—C14A—C15A	1.1 (2)	C18B—C13B—C14B—C15B	0.0 (2)
C10A—C13A—C14A—C15A	−179.32 (14)	C10B—C13B—C14B—C15B	179.36 (15)
C13A—C14A—C15A—C16A	−0.6 (2)	C13B—C14B—C15B—C16B	−0.1 (2)
C14A—C15A—C16A—C17A	−0.2 (2)	C14B—C15B—C16B—C17B	−0.2 (3)
C15A—C16A—C17A—C18A	0.5 (2)	C15B—C16B—C17B—C18B	0.5 (3)
C16A—C17A—C18A—C13A	0.1 (2)	C16B—C17B—C18B—C13B	−0.5 (3)
C14A—C13A—C18A—C17A	−0.9 (2)	C14B—C13B—C18B—C17B	0.2 (2)
C10A—C13A—C18A—C17A	179.58 (14)	C10B—C13B—C18B—C17B	−179.09 (15)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2). **35**

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.52658 (14)	0.22244 (14)	0.15524 (9)	0.0248 (3)
O2A	0.78350 (13)	0.09747 (14)	0.06441 (8)	0.0226 (3)
N1A	0.61580 (15)	0.05683 (15)	0.19538 (9)	0.0173 (3)
H1A	0.6760	0.0127	0.1848	0.021*
C1A	0.59907 (17)	0.14642 (17)	0.14259 (10)	0.0175 (3)
C2A	0.67168 (17)	0.14801 (17)	0.06443 (10)	0.0178 (3)
C3A	0.6442 (2)	0.18737 (19)	−0.01410 (12)	0.0248 (4)
H3A	0.5724	0.2256	−0.0311	0.030*
C4A	0.7454 (2)	0.1597 (2)	−0.06639 (12)	0.0287 (4)
H4A	0.7535	0.1755	−0.1251	0.034*
C5A	0.8261 (2)	0.1072 (2)	−0.01637 (12)	0.0287 (4)
H5A	0.9027	0.0803	−0.0345	0.034*
C6A	0.53780 (18)	0.02979 (18)	0.26969 (10)	0.0186 (3)
H6AA	0.5004	−0.0766	0.2694	0.022*
H6AB	0.4498	0.0549	0.2597	0.022*
C7A	0.63300 (17)	0.11582 (17)	0.36101 (10)	0.0160 (3)
C8A	0.71410 (18)	0.26788 (18)	0.37888 (10)	0.0187 (3)
H8A	0.7154	0.3188	0.3317	0.022*
C9A	0.79300 (17)	0.34584 (18)	0.46475 (11)	0.0181 (3)
H9A	0.8481	0.4493	0.4752	0.022*

C10A	0.79303 (16)	0.27482 (17)	0.53643 (10)	0.0149 (3)
C11A	0.71407 (18)	0.12188 (18)	0.51727 (11)	0.0196 (3)
H11A	0.7135	0.0702	0.5640	0.024*
C12A	0.63624 (19)	0.04394 (18)	0.43084 (11)	0.0207 (3)
H12A	0.5845	−0.0601	0.4195	0.025*
C13A	0.87222 (16)	0.35936 (17)	0.62932 (10)	0.0150 (3)
C14A	0.91268 (17)	0.51153 (18)	0.65423 (11)	0.0186 (3)
H14A	0.8881	0.5614	0.6112	0.022*
C15A	0.98795 (18)	0.59107 (19)	0.74057 (11)	0.0210 (3)
H15A	1.0151	0.6943	0.7558	0.025*
C16A	1.02368 (18)	0.51977 (19)	0.80485 (11)	0.0203 (3)
H16A	1.0755	0.5740	0.8638	0.024*
C17A	0.98298 (18)	0.36863 (19)	0.78202 (11)	0.0201 (3)
H17A	1.0064	0.3193	0.8257	0.024*
C18A	0.90813 (17)	0.28913 (18)	0.69547 (10)	0.0182 (3)
H18A	0.8808	0.1858	0.6808	0.022*
O1B	0.73081 (14)	0.52182 (13)	0.03785 (8)	0.0217 (2)
O2B	0.59298 (13)	0.75742 (13)	0.16030 (8)	0.0205 (2)
N1B	0.57275 (15)	0.52798 (15)	0.17157 (8)	0.0159 (3)
H1B	0.5968	0.4541	0.1528	0.019*
C1B	0.61537 (16)	0.64570 (17)	0.13449 (10)	0.0151 (3)
C2B	0.69076 (17)	0.63767 (17)	0.05823 (10)	0.0153 (3)
C3B	0.72916 (19)	0.72726 (19)	0.00007 (11)	0.0216 (3)

H3B	0.7130	0.8146	−0.0004	0.026*
C4B	0.79878 (19)	0.66445 (19)	−0.06032 (11)	0.0217 (3)
H4B	0.8382	0.7017	−0.1089	0.026*
C5B	0.7974 (2)	0.5419 (2)	−0.03468 (11)	0.0235 (3)
H5B	0.8373	0.4781	−0.0630	0.028*
C6B	0.48726 (17)	0.52064 (18)	0.24256 (10)	0.0175 (3)
H6BA	0.4302	0.4158	0.2426	0.021*
H6BB	0.4138	0.5636	0.2285	0.021*
C7B	0.57990 (17)	0.59904 (16)	0.33573 (10)	0.0146 (3)
C8B	0.50670 (17)	0.62681 (18)	0.40369 (10)	0.0180 (3)
H8B	0.4009	0.5950	0.3905	0.022*
C9B	0.58562 (17)	0.70009 (18)	0.49023 (10)	0.0175 (3)
H9B	0.5331	0.7178	0.5353	0.021*
C10B	0.74159 (16)	0.74827 (16)	0.51216 (10)	0.0143 (3)
C11B	0.81369 (17)	0.71780 (18)	0.44394 (10)	0.0181 (3)
H11B	0.9193	0.7479	0.4571	0.022*
C12B	0.73416 (17)	0.64429 (18)	0.35715 (10)	0.0185 (3)
H12B	0.7861	0.6249	0.3122	0.022*
C13B	0.82699 (17)	0.83169 (17)	0.60416 (10)	0.0147 (3)
C14B	0.97969 (17)	0.92369 (18)	0.62017 (10)	0.0184 (3)
H14B	1.0305	0.9326	0.5715	0.022*
C15B	1.05823 (19)	1.00239 (19)	0.70638 (11)	0.0215 (3)
H15B	1.1618	1.0642	0.7160	0.026*

C16B	0.9859 (2)	0.99101 (19)	0.77846 (11)	0.0222 (3)
H16B	1.0396	1.0445	0.8372	0.027*
C17B	0.8343 (2)	0.90073 (19)	0.76383 (10)	0.0211 (3)
H17B	0.7840	0.8930	0.8127	0.025*
C18B	0.75574 (18)	0.82148 (18)	0.67775 (10)	0.0182 (3)
H18B	0.6523	0.7595	0.6687	0.022*

Table 5: Atomic displacement parameters (Å2). **35**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1A	0.0271 (6)	0.0181 (6)	0.0344 (7)	0.0136 (5)	0.0079 (5)	0.0078 (5)
O2A	0.0240 (6)	0.0239 (6)	0.0209 (6)	0.0107 (5)	0.0054 (5)	0.0045 (5)
N1A	0.0220 (6)	0.0165 (6)	0.0164 (6)	0.0112 (5)	0.0039 (5)	0.0031 (5)
C1A	0.0176 (7)	0.0123 (7)	0.0200 (7)	0.0050 (6)	0.0005 (5)	0.0017 (6)
C2A	0.0191 (7)	0.0123 (7)	0.0190 (7)	0.0049 (6)	−0.0001 (6)	0.0020 (6)
C3A	0.0267 (8)	0.0149 (8)	0.0239 (8)	0.0025 (7)	−0.0061 (7)	0.0036 (6)
C4A	0.0342 (9)	0.0207 (8)	0.0194 (8)	−0.0007 (7)	0.0038 (7)	0.0029 (6)
C5A	0.0285 (9)	0.0267 (9)	0.0244 (8)	0.0046 (7)	0.0105 (7)	0.0008 (7)
C6A	0.0199 (7)	0.0164 (7)	0.0182 (7)	0.0067 (6)	0.0043 (6)	0.0016 (6)
C7A	0.0162 (7)	0.0157 (7)	0.0173 (7)	0.0080 (6)	0.0050 (5)	0.0022 (6)
C8A	0.0221 (7)	0.0170 (7)	0.0175 (7)	0.0069 (6)	0.0062 (6)	0.0061 (6)
C9A	0.0188 (7)	0.0138 (7)	0.0206 (7)	0.0048 (6)	0.0052 (6)	0.0049 (6)
C10A	0.0128 (6)	0.0158 (7)	0.0180 (7)	0.0070 (6)	0.0050 (5)	0.0048 (5)
C11A	0.0233 (7)	0.0157 (7)	0.0203 (7)	0.0070 (6)	0.0048 (6)	0.0074 (6)
C12A	0.0228 (8)	0.0141 (7)	0.0231 (8)	0.0056 (6)	0.0042 (6)	0.0036 (6)
C13A	0.0116 (6)	0.0162 (7)	0.0182 (7)	0.0056 (6)	0.0058 (5)	0.0046 (6)
C14A	0.0186 (7)	0.0162 (7)	0.0215 (7)	0.0072 (6)	0.0046 (6)	0.0050 (6)
C15A	0.0220 (7)	0.0161 (7)	0.0226 (8)	0.0061 (6)	0.0050 (6)	0.0014 (6)
C16A	0.0185 (7)	0.0219 (8)	0.0178 (7)	0.0059 (6)	0.0042 (6)	0.0020 (6)
C17A	0.0211 (7)	0.0226 (8)	0.0177 (7)	0.0088 (6)	0.0048 (6)	0.0072 (6)
C18A	0.0174 (7)	0.0177 (7)	0.0204 (7)	0.0075 (6)	0.0052 (6)	0.0048 (6)

O1B	0.0310 (6)	0.0192 (6)	0.0215 (5)	0.0143 (5)	0.0117 (5)	0.0074 (5)
O2B	0.0270 (6)	0.0154 (5)	0.0208 (5)	0.0110 (5)	0.0045 (5)	0.0027 (4)
N1B	0.0214 (6)	0.0127 (6)	0.0146 (6)	0.0077 (5)	0.0051 (5)	0.0029 (5)
C1B	0.0154 (6)	0.0142 (7)	0.0137 (6)	0.0058 (6)	−0.0009 (5)	0.0012 (5)
C2B	0.0170 (7)	0.0128 (7)	0.0136 (6)	0.0051 (6)	−0.0004 (5)	0.0005 (5)
C3B	0.0262 (8)	0.0184 (8)	0.0205 (7)	0.0086 (7)	0.0046 (6)	0.0063 (6)
C4B	0.0230 (8)	0.0220 (8)	0.0177 (7)	0.0057 (7)	0.0051 (6)	0.0055 (6)
C5B	0.0274 (8)	0.0240 (8)	0.0198 (7)	0.0100 (7)	0.0099 (6)	0.0040 (6)
C6B	0.0170 (7)	0.0166 (7)	0.0159 (7)	0.0036 (6)	0.0046 (5)	0.0020 (6)
C7B	0.0172 (7)	0.0123 (7)	0.0144 (6)	0.0055 (6)	0.0033 (5)	0.0044 (5)
C8B	0.0137 (6)	0.0193 (8)	0.0191 (7)	0.0055 (6)	0.0034 (5)	0.0022 (6)
C9B	0.0162 (7)	0.0209 (8)	0.0157 (7)	0.0073 (6)	0.0063 (5)	0.0028 (6)
C10B	0.0162 (6)	0.0128 (7)	0.0144 (6)	0.0058 (5)	0.0037 (5)	0.0042 (5)
C11B	0.0139 (6)	0.0216 (8)	0.0191 (7)	0.0071 (6)	0.0045 (5)	0.0043 (6)
C12B	0.0176 (7)	0.0216 (8)	0.0169 (7)	0.0081 (6)	0.0070 (6)	0.0030 (6)
C13B	0.0182 (7)	0.0129 (7)	0.0149 (6)	0.0085 (6)	0.0025 (5)	0.0037 (5)
C14B	0.0185 (7)	0.0181 (7)	0.0194 (7)	0.0088 (6)	0.0026 (6)	0.0042 (6)
C15B	0.0205 (7)	0.0178 (8)	0.0247 (8)	0.0089 (6)	−0.0019 (6)	0.0030 (6)
C16B	0.0291 (8)	0.0190 (8)	0.0182 (7)	0.0134 (7)	−0.0039 (6)	0.0009 (6)
C17B	0.0305 (8)	0.0216 (8)	0.0149 (7)	0.0145 (7)	0.0044 (6)	0.0048 (6)
C18B	0.0218 (7)	0.0184 (7)	0.0169 (7)	0.0099 (6)	0.0047 (6)	0.0053 (6)

Table 6: Hydrogen-bond geometry (Å, º). **35**

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O2Bi	0.88	2.33	2.8894 (18)	122
C3A—H3A···O2Bii	0.95	2.48	3.295 (2)	144
N1B—H1B···O1A	0.88	2.17	2.8938 (18)	139
C3B—H3B···O2Aiii	0.95	2.65	3.511 (2)	151

Symmetry codes: (i) x, y−1, z; (ii) −x+1, −y+1, −z; (iii) x, y+1, z.

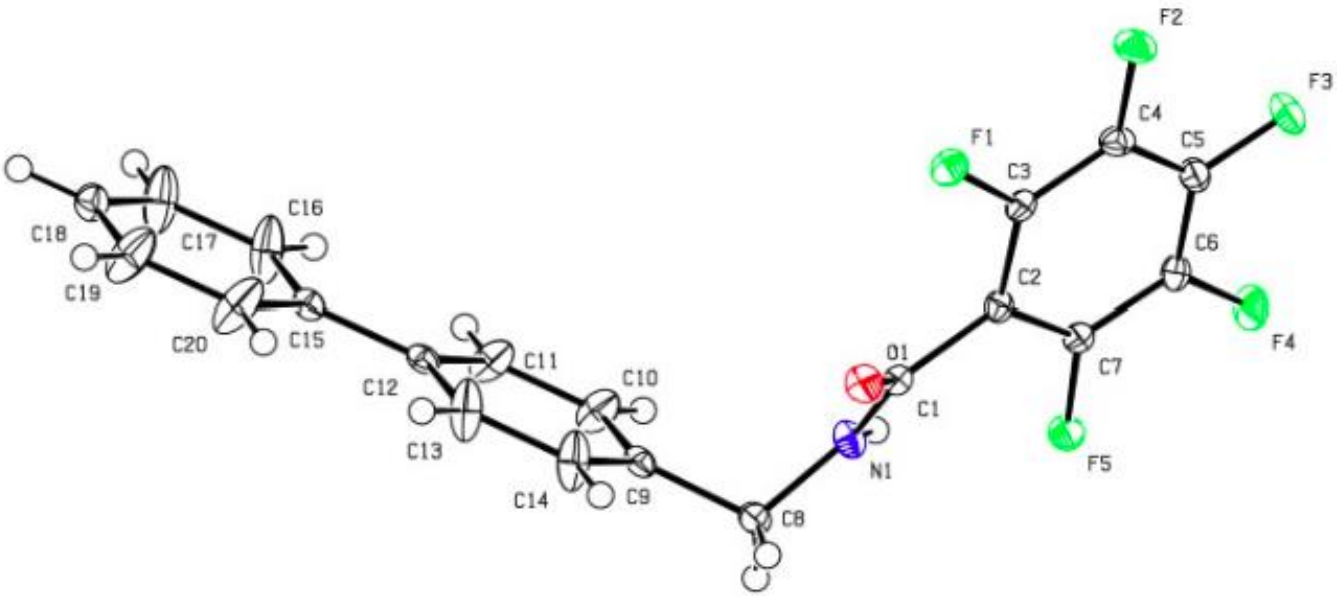

Figure 179: X-Ray Crystal Structure of *N*-([1,1'-biphenyl]-4-ylmethyl)-2,3,4,5,6-pentafluorobenzamide **33.**

Table 1. Experimental details. **33**

	APEX14AN012_0m
Crystal data	
Chemical formula	C20H12F5NO
*M*r	377.31
Crystal system, space group	Orthorhombic, *P*212121
Temperature (K)	100
a, *b*, *c* (Å)	cellsetting:orthorhombic 5.2988 (4), 9.2762 (6), 31.828 (2)
V (Å3)	1564.42 (19)
Z	4
Radiation type	Mo *K*α
μ (mm−1)	0.14
Crystal size (mm)	0.55 × 0.34 × 0.10
Data collection	
Diffractometer	Bruker AXS APEXII CCD diffractometer
Absorption correction	Multi-scan Apex2 v2013.4-1 (Bruker, 2013)
*T*min, *T*max	0.619, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18196, 4808, 4491
*R*int	0.028
(sin θ/λ)max (Å−1)	0.733
Refinement	
$R[F2 > 2\sigma(F2)]$, *wR*(*F*2), *S*	0.040, 0.109, 1.04
No. of reflections	4808
No. of parameters	244
H-atom treatment	H-atom parameters constrained
Δρmax, Δρmin (e Å−3)	0.47, −0.22
Absolute structure	Flack x determined using 1747 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter	−0.29 (16)

Table 2: Bond Lengths (Å). **33**

F1—C3	1.3373(19)
F2—C4	1.339(2)
F3—C5	1.3319(19)
F4—C6	1.3411(19)
F5—C7	1.339(2)
O1—C1	1.231(2)
N1—C1	1.342(2)
N1—C8	1.467(2)
N1—H1	0.8800
C1—C2	1.512(2)
C2—C7	1.394(2)
C2—C3	1.398(2)
C3—C4	1.385(2)
C4—C5	1.384(3
C5—C6	1.376(3)
C6—C7	1.384(2)
C8—C9	1.510(2)
C8—H8A	0.9900
C8—H8B	0.9900
C9—C14	1.378(3)
C9—C10	1.391(3)
C10—C11	1.389(3)
C10—H10	0.9500
C11—C12	1.397(2)
C11—H11	0.9500
C12—C13	1.392(3)
C12—C15	1.491(2)
C13—C14	1.396(3)
C13—H13	0.9500
C14—H14	0.9500
C15—C16	1.389(3)
C15—C20	1.392(3)
C16—C17	1.394(3)
C16—H16	0.9500
C17—C18	1.380(3)
C17—H17	0.9500
C18—C19	1.374(3)
C18—H18	0.9500
C19—C20	1.394(3)
C19—H19	0.9500
C20—H20	0.9500

Table 3: Bond Angles (deg). **33**

C1—N1—C8	120.96 (15)	C11—C10—C9	120.81 (17)
C1—N1—H1	119.5	C11—C10—H10	119.6
C8—N1—H1	119.5	C9—C10—H10	119.6
O1—C1—N1	123.87(15)	C10—C11—C12	121.59 (18)
O1—C1—C2	119.76 (15)	C10—C11—H11	119.2
N1—C1—C2	116.37 (15)	C12—C11—H11	119.2
C7—C2—C3	115.98 (14)	C13—C12—C11	116.88 (16)
C7—C2—C1	123.54 (15)	C13—C12—C15	121.80 (16)
C3—C2—C1	120.31 (15)	C11—C12—C15	121.31 (16)
F1—C3—C4	116.90 (15)	C12—C13—C14	121.52 (17)
F1—C3—C2	121.08 (15)	C12—C13—H13	119.2
C4—C3—C2	122.02 (15)	C14—C13—H13	119.2
F2—C4—C5	119.81 (16)	C9—C14—C13	120.98 (18)
F2—C4—C3	120.13 (16)	C9—C14—H14	119.5
C5—C4—C3	120.06 (16)	C13—C14—H14	119.5
F3—C5—C6	120.41 (16)	C16—C15—C20	116.78 (17)
F3—C5—C4	120.07 (16)	C16—C15—C12	121.44 (16)
C6—C5—C4	119.52 (16)	C20—C15—C12	121.79 (16)
F4—C6—C5	119.89 (15)	C15—C16—C17	121.83 (18)
F4—C6—C7	120.38 (16)	C15—C16—H16	119.1
C5—C6—C7	119.73 (16)	C17—C16—H16	119.1
F5—C7—C6	116.60 (15)	C18—C17—C16	120.30 (19)
F5—C7—C2	120.70 (14)	C18—C17—H17	119.8
C6—C7—C2	122.68 (15)	C16—C17—H17	119.8

N1—C8—C9	113.16 (15)	C19—C18—C17	118.90 (18)
N1—C8—H8A	108.9	C19—C18—H18	120.5
C9—C8—H8A	108.9	C17—C18—H18	120.5
N1—C8—H8B	108.9	C18—C19—C20	120.65 (19)
C9—C8—H8B	108.9	C18—C19—H19	119.7
H8A—C8—H8B	107.8	C20—C19—H19	119.7
C14—C9—C10	118.21(17)	C15—C20—C19	121.54 (19)
C14—C9—C8	121.21 (17)	C15—C20—H20	119.2
C10—C9—C8	120.57 (16)	C19—C20—H20	119.2
C8—N1—C1—O1	−3.2 (3)	C3—C2—C7—C6	0.9 (2)
C8—N1—C1—C2	175.88 (15)	C1—C2—C7—C6	−174.41 (16)
O1—C1—C2—C7	143.94 (17)	C1—N1—C8—C9	85.7 (2)
N1—C1—C2—C7	−35.1 (2)	N1—C8—C9—C14	−117.5 (2)
O1—C1—C2—C3	−31.1 (2)	N1—C8—C9—C10	63.6 (2)
N1—C1—C2—C3	149.79 (17)	C14—C9—C10—C11	−0.2 (3)
C7—C2—C3—F1	179.13 (15	C8—C9—C10—C11	178.70 (19)
C1—C2—C3—F1	−5.4 (2)	C9—C10—C11—C12	0.4 (3)
C7—C2—C3—C4	−0.4 (2)	C10—C11—C12—C13	−0.5 (3)
C1—C2—C3—C4	175.01 (16)	C10—C11—C12—C15	−179.71 19)
F1—C3—C4—F2	0.4 (2)	C11—C12—C13—C14	0.4 (3)
C2—C3—C4—F2	179.99 (15)	C15—C12—C13—C14	179.6 (2)
F1—C3—C4—C5	−179.57 (15)	C10—C9—C14—C13	0.1 (3)
C2—C3—C4—C5	0.0 (3)	C8—C9—C14—C13	−178.8 (2)
F2—C4—C5—F3	0.4 (3)	C12—C13—C14—C9	−0.2 (4)
C3—C4—C5—F3	−179.59 (16)	C13—C12—C15—C16	178.2 (2)

F2—C4—C5—C6	−179.97 (16)	C11—C12—C15—C16	−2.6 (3)
C3—C4—C5—C6	0.0 (3)	C13—C12—C15—C20	−1.5 (3)
F3—C5—C6—F4	0.3 (3)	C11—C12—C15—C20	177.6 (2)
C4—C5—C6—F4	−179.26 (16)	C20—C15—C16—C17	−0.3 (4)
F3—C5—C6—C7	−179.99 (15)	C12—C15—C16—C17	179.9 (2)
C4—C5—C6—C7	0.4 (3)	C15—C16—C17—C18	0.0 (4)
F4—C6—C7—F5	0.6 (2)	C16—C17—C18—C19	0.1 (4)
C5—C6—C7—F5	−179.11 (15)	C17—C18—C19—C20	0.0 (4)
F4—C6—C7—C2	178.79 (15)	C16—C15—C20—C19	0.4 (4)
C5—C6—C7—C2	−0.9 (3)	C12—C15—C20—C19	−179.8 (2)
C3—C2—C7—F5	179.03 (15)	C18—C19—C20—C15	−0.3 (4)
C1—C2—C7—F5	3.8 (2)		

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2).**33**

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.9493 (2)	0.85305 (12)	0.15830 (3)	0.0192 (2)
F2	0.9605 (2)	1.00184 (12)	0.08682 (4)	0.0219 (2)
F3	0.6025 (2)	0.95988 (13)	0.02696 (4)	0.0240 (3)
F4	0.2288 (2)	0.76391 (13)	0.03969 (3)	0.0223 (3)
F5	0.2113 (2)	0.61266 (12)	0.11070 (3)	0.0187 (2)
O1	0.7943 (2)	0.59915 (15)	0.19217 (4)	0.0174 (3)
N1	0.3660 (3)	0.59147 (17)	0.19301 (5)	0.0157 (3)
H1	0.2253	0.6231	0.1816	0.019*
C1	0.5888 (3)	0.63317 (17)	0.17709 (5)	0.0136 (3)
C2	0.5790 (3)	0.72532 (18)	0.13796 (5)	0.0124 (3)
C3	0.7680 (3)	0.82690 (19)	0.13007 (5)	0.0137 (3)
C4	0.7762 (3)	0.90555 (19)	0.09313 (6)	0.0157 (3)
C5	0.5938 (4)	0.88485 (18)	0.06261 (5)	0.0161 (3)
C6	0.4048 (3)	0.78577 (19)	0.06930 (5)	0.0154 (3)
C7	0.3982 (3)	0.70867 (17)	0.10651 (5)	0.0134 (3)
C8	0.3534 (3)	0.4939 (2)	0.22924 (5)	0.0165 (3)
H8A	0.4935	0.4236	0.2273	0.020*
H8B	0.1930	0.4394	0.2281	0.020*
C9	0.3697 (3)	0.57147 (19)	0.27088 (5)	0.0145 (3)
C10	0.1832 (4)	0.6687 (2)	0.28285 (6)	0.0235 (4)

H10	0.0464	0.6878	0.2644	0.028*
C11	0.1946 (4)	0.7381 (2)	0.32146 (7)	0.0236 (4)
H11	0.0643	0.8035	0.3290	0.028*
C12	0.3930 (3)	0.71409 (18)	0.34949 (5)	0.0137 (3)
C13	0.5779 (4)	0.6164 (3)	0.33706 (6)	0.0276 (5)
H13	0.7152	0.5969	0.3554	0.033*
C14	0.5662 (4)	0.5464 (3)	0.29828 (6)	0.0271 (5)
H14	0.6956	0.4805	0.2907	0.033*
C15	0.4034 (3)	0.78846 (17)	0.39097 (5)	0.0135 (3)
C16	0.2217 (4)	0.8887 (3)	0.40267 (6)	0.0326 (5)
H16	0.0884	0.9103	0.3837	0.039*
C17	0.2298 (4)	0.9586 (3)	0.44141 (7)	0.0339 (6)
H17	0.1027	1.0267	0.4485	0.041*
C18	0.4214 (4)	0.9295 (2)	0.46951 (6)	0.0197 (4)
H18	0.4273	0.9767	0.4960	0.024*
C19	0.6036 (5)	0.8309 (3)	0.45858 (7)	0.0350 (6)
H19	0.7365	0.8100	0.4777	0.042*
C20	0.5953 (5)	0.7614 (3)	0.41976 (8)	0.0335 (5)
H20	0.7237	0.6940	0.4128	0.040*

Table 5: Atomic displacement parameters (Å2). **33**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.0160 (5)	0.0221 (5)	0.0195 (5)	−0.0052 (4)	−0.0051 (4)	−0.0019 (4)
F2	0.0195 (5)	0.0198 (5)	0.0264 (6)	−0.0075 (5)	0.0032 (4)	0.0025 (4)
F3	0.0296 (6)	0.0253 (6)	0.0170 (5)	−0.0012 (5)	0.0018 (5)	0.0082 (4)
F4	0.0226 (6)	0.0286 (6)	0.0156 (5)	−0.0028 (5)	−0.0079 (4)	0.0014 (4)
F5	0.0164 (5)	0.0219 (5)	0.0178 (5)	−0.0075 (4)	−0.0029 (4)	0.0010 (4)
O1	0.0133 (6)	0.0210 (6)	0.0179 (6)	0.0004 (5)	−0.0025 (5)	0.0013 (5)
N1	0.0132 (6)	0.0211 (7)	0.0127 (6)	−0.0001 (6)	−0.0004 (5)	0.0032 (5)
C1	0.0153 (7)	0.0129 (7)	0.0126 (7)	−0.0005 (6)	−0.0002 (6)	−0.0013 (5)
C2	0.0117 (7)	0.0133 (7)	0.0122 (6)	0.0002 (6)	0.0004 (6)	−0.0009 (5)
C3	0.0124 (7)	0.0147 (7)	0.0140 (7)	−0.0006 (6)	−0.0010 (6)	−0.0027 (6)
C4	0.0146 (7)	0.0134 (7)	0.0192 (8)	−0.0018 (6)	0.0031 (6)	−0.0009 (6)
C5	0.0192 (8)	0.0156 (7)	0.0136 (7)	0.0024 (7)	0.0017 (6)	0.0015 (6)
C6	0.0153 (8)	0.0185 (7)	0.0124 (7)	0.0005 (6)	−0.0023 (6)	−0.0007 (5)
C7	0.0131 (7)	0.0132 (7)	0.0139 (7)	−0.0005 (6)	−0.0005 (6)	−0.0010 (5)
C8	0.0183 (8)	0.0164 (7)	0.0147 (7)	−0.0029 (6)	−0.0005 (6)	0.0024 (6)

C9	0.0138 (7)	0.0157 (7)	0.0139 (7)	−0.0028 (6)	0.0005 (6)	0.0038 (6)
C10	0.0221 (9)	0.0224 (9)	0.0261 (9)	0.0072 (7)	−0.0128 (7)	−0.0052 (7)
C11	0.0228 (9)	0.0201 (8)	0.0278 (9)	0.0090 (7)	−0.0116 (7)	−0.0071 (7)
C12	0.0112 (7)	0.0144 (7)	0.0154 (7)	−0.0028 (6)	−0.0007 (6)	0.0039 (5)
C13	0.0170 (8)	0.0516 (13)	0.0142 (8)	0.0161 (9)	−0.0026 (7)	−0.0037 (8)
C14	0.0196 (9)	0.0466 (12)	0.0151 (8)	0.0165 (9)	−0.0011 (7)	−0.0041 (8)
C15	0.0124 (7)	0.0138 (7)	0.0144 (7)	−0.0012 (6)	−0.0003 (6)	0.0029 (5)
C16	0.0232 (9)	0.0592 (15)	0.0155 (8)	0.0225 (11)	−0.0056 (7)	−0.0066 (9)
C17	0.0262 (10)	0.0576 (15)	0.0179 (9)	0.0232 (11)	−0.0015 (8)	−0.0083 (9)
C18	0.0211 (9)	0.0213 (8)	0.0166 (8)	−0.0019 (7)	−0.0013 (7)	−0.0002 (6)
C19	0.0355 (12)	0.0352 (11)	0.0344 (11)	0.0160 (10)	−0.0237 (10)	−0.0149 (9)
C20	0.0329 (12)	0.0316 (11)	0.0360 (11)	0.0189 (10)	−0.0215 (10)	−0.0170 (9)

Table 6: Hydrogen-bond geometry (Å, º). **33**

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F5	0.88	2.26	2.7521 (19)	115
N1—H1···O1i	0.88	2.32	3.030 (2)	138

Figure 180: X-Ray Crystal Structure of (*E*)-2-((([1,1'-biphenyl]-4-ylmethyl)imino)methyl)-4-methoxyphenol **18.**

Table1.Experimental details.**18**

	Apex15mz027_0m
Crystal data	
Chemical formula	$C_{21}H_{19}NO_2$
M_r	317.37
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	cellsetting:monoclinic 7.2819 (17), 18.775 (4), 11.704 (3)
β (°)	cellsetting:monoclinic 94.462 (4)
V (Å^3)	1595.3 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.55 × 0.13 × 0.12
Data collection	
Diffractometer	Bruker AXS APEXII CCD diffractometer
Absorption correction	Multi-scan Apex2 v2013.4-1 (Bruker, 2013)
T_{min}, T_{max}	0.663, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25970, 4867, 3787
R_{int}	0.032
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.715
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.146, 1.04
No. of reflections	4867
No. of parameters	220
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.64, −0.25

Table 2. Bond Lengths (Å).**18**

C1—N1 1.2814(14)	C10—C15 1.3944(15)
C1—C2 1.4615(15)	C11—C12 1.3873(15)
C1—H1 0.9500	C11—H11 0.9500
C2—C3 1.4003(15)	C12—C13 1.4004(15)
C2—C7 1.4110(15)	C12—H12 0.9500
C3—O1 1.3583(13)	C13—C14 1.3961(15)
C3—C4 1.3954(16)	C13—C16 1.4843(15)
C4—C5 1.3813(16)	C14—C15 1.3934(15)
C4—H4 0.9500	C14—H14 0.9500
C5—C6 1.4006(16)	C15—H15 0.9500
C5—H5 0.9500	C16—C21 1.3988(15)
C6—O2 1.3736(13)	C16—C17 1.3999(15)
C6—C7 1.3874(15)	C17—C18 1.3900(15)
C7—H7 0.9500	C17—H17 0.9500
C8—O2 1.4269(15)	C18—C19 1.3914(17)
C8—H8A 0.9800	C18—H18 0.9500
C8—H8B 0.9800	C19—C20 1.3913(17)
C8—H8C 0.9800	C19—H19 0.9500
C9—N1 1.4562(15)	C20—C21 1.3944(15)
C9—C10 1.5077(15)	C20—H20 0.9500
C9—H9A 0.9900	C21—H21 0.9500
C10—C11 1.3911 (15)	O1—H1A 0.981(18)

Table 3: Bond Angles (Deg). **18**

N1—C1—C2	121.05 (10)	C12—C11—C10	121.16 (10)
N1—C1—H1	119.5	C12—C11—H11	119.4
C2—C1—H1	119.5	C10—C11—H11	119.4
C3—C2—C7	119.84 (10)	C11—C12—C13	120.94 (10)
C3—C2—C1	120.75 (10)	C11—C12—H12	119.5
C7—C2—C1	119.26 (10)	C13—C12—H12	119.5
O1—C3—C4	118.45 (10)	C14—C13—C12	117.75 (10)
O1—C3—C2	121.76 (10)	C14—C13—C16	121.78 (9)
C4—C3—C2	119.78 (10)	C12—C13—C16	120.47 (9)
C5—C4—C3	120.13 (10)	C15—C14—C13	121.19 (10)
C5—C4—H4	119.9	C15—C14—H14	119.4
C3—C4—H4	119.9	C13—C14—H14	119.4
C4—C5—C6	120.64 (10)	C14—C15—C10	120.64 (10)
C4—C5—H5	119.7	C14—C15—H15	119.7
C6—C5—H5	119.7	C10—C15—H15	119.7
O2—C6—C7	124.82 (10)	C21—C16—C17	118.46 (10)
O2—C6—C5	115.28 (9)	C21—C16—C13	121.53 (10)
C7—C6—C5	119.87 (10)	C17—C16—C13	120.00 (10)
C6—C7—C2	119.71 (10)	C18—C17—C16	120.95 (10)
C6—C7—H7	120.1	C18—C17—H17	119.5
C2—C7—H7	120.1	C16—C17—H17	119.5
O2—C8—H8A	109.5	C17—C18—C19	120.20 (11)
O2—C8—H8B	109.5	C17—C18—H18	119.9
H8A—C8—H8B	109.5	C19—C18—H18	119.9

O2—C8—H8C	109.5	C20—C19—C18	119.42 (11)
H8A—C8—H8C	109.5	C20—C19—H19	120.3
H8B—C8—H8C	109.5	C18—C19—H19	120.3
N1—C9—C10	113.66 (9)	C19—C20—C21	120.44 (11)
N1—C9—H9A	108.8	C19—C20—H20	119.8
C10—C9—H9A	108.8	C21—C20—H20	119.8
N1—C9—H9B	108.8	C20—C21—C16	120.52 (10)
C10—C9—H9B	108.8	C20—C21—H21	119.7
H9A—C9—H9B	107.7	C16—C21—H21	119.7
C11—C10—C15	118.30 (10)	C1—N1—C9	117.86 (10)
C11—C10—C9	117.91 (10)	C3—O1—H1A	109.5
C15—C10—C9	123.79 (10)	C6—O2—C8	116.97 (9)
N1—C1—C2—C3	3.11 (17)	C12—C13—C14—C	151.36 (17)
N1—C1—C2—C7	−172.39 (11)	C16—C13—C14—C15	−177.64 (10)
C7—C2—C3—O1	−179.62 (10)	C13—C14—C15—C10	−0.81 (18)
C1—C2—C3—O1	4.91 (17)	C11—C10—C15—C14	−0.46 (17)
C7—C2—C3—C4	1.56 (17)	C9—C10—C15—C14	−179.87 (11)
C1—C2—C3—C4	−173.92 (10)	C14—C13—C16—C21	−34.62 (16)
O1—C3—C4—C5	−179.06 (11)	C12—C13—C16—C21	146.40 (11)
C2—C3—C4—C5	−0.19 (18)	C14—C13—C16—C17	144.28 (11)
C3—C4—C5—C6	−1.13 (18)	C12—C13—C16—C17	−34.70 (16)
C4—C5—C6—O2	179.25 (10)	C21—C16—C17—C18	0.61 (17)
C4—C5—C6—C7	1.06 (17)	C13—C16—C17—C18	−178.32 (10)
O2—C6—C7—C2	−177.69 (10)	C16—C17—C18—C19	−0.49 (18)
C5—C6—C7—C2	0.32 (17)	C17—C18—C19—C20	−0.22 (18)

C3—C2—C7—C6	−1.62 (16)	C18—C19—C20—C21	0.80 (19)
C1—C2—C7—C6	173.92 (10)	C19—C20—C21—C16	−0.68 (18)
N1—C9—C10—C11	169.63 (10)	C17—C16—C21—C20	−0.03 (17)
N1—C9—C10—C15	−10.95 (17)	C13—C16—C21—C20	178.89 (10)
C15—C10—C11—C12	1.15 (17)	C2—C1—N1—C9	172.05 (10)
C9—C10—C11—C12	−179.40 (11)	C10—C9—N1—C1	157.49 (11)
C10—C11—C12—C13	−0.58 (18)	C7—C6—O2—C8	−0.36 (16)
C11—C12—C13—C14	−0.67 (17)	C5—C6—O2—C8	−178.44 (11)
C11—C12—C13—C16	178.34 (11)		

Table 4. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2).**18.**

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.21916 (16)	0.61732 (6)	0.92113 (9)	0.0169 (2)
H1	0.1570	0.6386	0.9809	0.020*
C2	0.25746 (16)	0.65976 (6)	0.82104 (9)	0.0161 (2)
C3	0.34083 (16)	0.62893 (6)	0.72918 (10)	0.0177 (2)
C4	0.39210 (17)	0.67174 (6)	0.63952 (10)	0.0201 (2)
H4	0.4490	0.6510	0.5772	0.024*
C5	0.36024 (16)	0.74424 (6)	0.64118 (9)	0.0191 (2)
H5	0.3975	0.7732	0.5805	0.023*
C6	0.27355 (16)	0.77551 (6)	0.73140 (9)	0.0170 (2)
C7	0.22165 (15)	0.73362 (6)	0.82108 (9)	0.0158 (2)
H7	0.1622	0.7546	0.8822	0.019*
C8	0.16795 (19)	0.88216 (6)	0.81604 (11)	0.0236 (3)
H8A	0.1599	0.9335	0.8015	0.035*
H8B	0.2426	0.8736	0.8881	0.035*
H8C	0.0440	0.8628	0.8217	0.035*
C9	0.24728 (17)	0.51570 (6)	1.03793 (10)	0.0199 (2)
H9A	0.1341	0.5333	1.0703	0.024*
H9B	0.3531	0.5282	1.0925	0.024*

C10	0.23608 (15)	0.43576 (6)	1.02739 (9)	0.0165 (2)
C11	0.24665 (16)	0.39585 (6)	1.12783 (9)	0.0181 (2)
H11	0.2593	0.4196	1.1997	0.022*
C12	0.23905 (16)	0.32203 (6)	1.12486 (9)	0.0173 (2)
H12	0.2477	0.2960	1.1947	0.021*
C13	0.21878 (15)	0.28538 (6)	1.02043 (9)	0.0148 (2)
C14	0.20455 (16)	0.32564 (6)	0.91984 (9)	0.0173 (2)
H14	0.1880	0.3021	0.8479	0.021*
C15	0.21418 (16)	0.39975 (6)	0.92302 (9)	0.0180 (2)
H15	0.2057	0.4260	0.8534	0.022*
C16	0.21587 (15)	0.20634 (6)	1.01821 (9)	0.0154 (2)
C17	0.32453 (16)	0.16766 (6)	1.10037 (9)	0.0172 (2)
H17	0.3973	0.1923	1.1585	0.021*
C18	0.32751 (17)	0.09365 (6)	1.09809 (10)	0.0207 (2)
H18	0.4027	0.0682	1.1541	0.025*
C19	0.22067 (18)	0.05672 (6)	1.01399 (10)	0.0224 (2)
H19	0.2229	0.0061	1.0122	0.027*
C20	0.11052 (18)	0.09450 (6)	0.93257 (10)	0.0215 (2)

H20	0.0361	0.0695	0.8755	0.026*
C21	0.10863 (16)	0.16876 (6)	0.93411 (9)	0.0179 (2)
H21	0.0339	0.1940	0.8776	0.021*
N1	0.26807 (14)	0.55185 (5)	0.92961 (8)	0.0191 (2)
O1	0.37678 (13)	0.55802 (4)	0.72539 (8)	0.0235 (2)
H1A	0.350 (2)	0.5360 (4)	0.7983 (14)	0.035*
O2	0.25149 (13)	0.84807 (4)	0.72423 (7)	0.0216 (2)

Table 5: Atomic displacement parameters (Å2).**18**

	*U*11	*U*22	*U*33	*U*12	*U*13
C1	0.0188 (5)	0.0160 (5)	0.0159 (5)	−0.0009 (4)	0.0021 (4)
C22	0.0166 (5)	0.0157 (5)	0.0159 (5)	−0.0015 (4)	0.0012 (4)
C3	0.0179 (5)	0.0169 (5)	0.0180 (5)	−0.0014 (4)	0.0007 (4)
C4	0.0206 (6)	0.0242 (6)	0.0157 (5)	−0.0012 (4)	0.0027 (4)
C5	0.0190 (5)	0.0235 (5)	0.0148 (5)	−0.0042 (4)	0.0016 (4)
C6	0.0181 (5)	0.0166 (5)	0.0161 (5)	−0.0017 (4)	−0.0002 (4)
C7	0.0174 (5)	0.0162 (5)	0.0137 (5)	−0.0016 (4)	0.0010 (4)
C8	0.0298 (7)	0.0183 (5)	0.0231 (6)	0.0022 (5)	0.0039 (5)
C9	0.0278 (6)	0.0152 (5)	0.0166 (5)	0.0012 (4)	0.0018 (4)
C10	0.0165 (5)	0.0146 (5)	0.0186 (5)	0.0009 (4)	0.0024 (4)
C11	0.0212 (6)	0.0175 (5)	0.0157 (5)	−0.0006 (4)	0.0013 (4)
C12	0.0209 (5)	0.0168 (5)	0.0140 (5)	−0.0004 (4)	0.0004 (4)
C13	0.0137 (5)	0.0142 (5)	0.0165 (5)	0.0001 (4)	0.0022 (4)
C14	0.0212 (5)	0.0165 (5)	0.0143 (5)	0.0008 (4)	0.0031 (4)
C15	0.0226 (6)	0.0168 (5)	0.0148 (5)	0.0015 (4)	0.0030 (4)
C16	0.0165 (5)	0.0147 (5)	0.0153 (5)	−0.0001 (4)	0.0039 (4)
C17	0.0172 (5)	0.0173 (5)	0.0172 (5)	−0.0005 (4)	0.0015 (4)
C18	0.0218 (6)	0.0181 (5)	0.0225 (6)	0.0015 (4)	0.0034 (4)
C19	0.0279 (6)	0.0146 (5)	0.0252 (6)	−0.0016 (4)	0.0057 (5)
C20	0.0254 (6)	0.0187 (5)	0.0203 (5)	−0.0032 (4)	0.0022 (4)
C21	0.0200 (5)	0.0175 (5)	0.0162 (5)	−0.0004 (4)	0.0008 (4)
N1	0.0227 (5)	0.0155 (4)	0.0192 (5)	−0.0004 (4)	0.0029 (4)
O1	0.0317 (5)	0.0165 (4)	0.0229 (4)	0.0003 (3)	0.0066 (4)
O2	0.0290 (5)	0.0161 (4)	0.0200 (4)	0.0000 (3)	0.0041 (3)

Table 6: Hydrogen-bond geometry (Å, º).**18**

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.98	1.72	2.5782 (14)	144

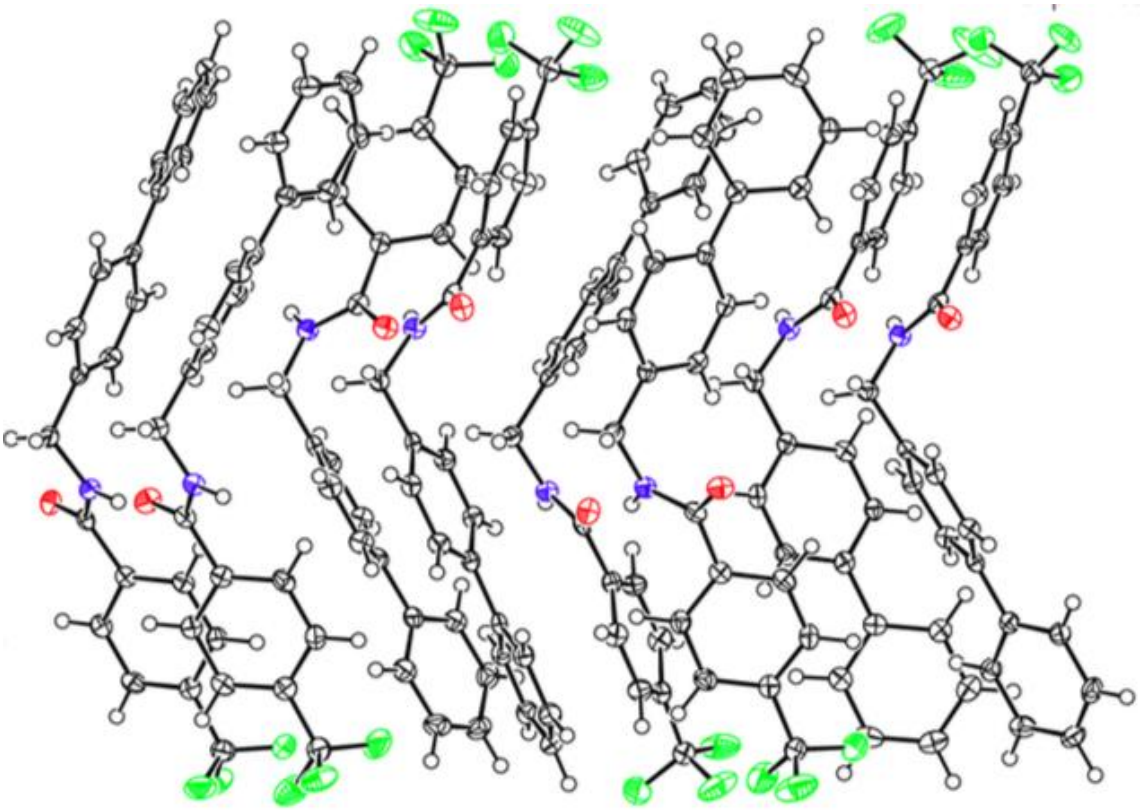

Figure 181: X-Ray Crystal Structure of **48.**

Table 1. Experimental details**. 48**

	Prosp15mz070_0m
Crystal data	
Chemical formula	C21H16F3NO
*M*r	355.35
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	100
a, *b*, *c* (Å)	cellsetting:monoclinic 72.578 (3), 10.0576 (4), 37.9188 (14)
β (°)	cellsetting:monoclinic 105.496 (2)
V (Å3)	26673.2 (18)
Z	64
Radiation type	Cu *K*α
μ (mm−1)	0.93
Crystal size (mm)	0.20 × 0.10 × 0.07
Data collection	
Diffractometer	Bruker AXS X8 Prospector CCD diffractometer
Absorption correction	Multi-scan TWINABS (Sheldrick, 2012)
*T*min, *T*max	0.616, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	133943, 23185, 22554
*R*int	0.116
(sin θ/λ)max (Å−1)	0.598
Refinement	
$R[F2 > 2\sigma(F2)]$, $wR(F2)$, S	0.045, 0.125, 1.05
No. of reflections	23185
No. of parameters	1874
H-atom treatment	H-atom parameters constrained
	$w = 1/[\sigma2(Fo2) + (0.073P)2 + 28.5534P]$ where $P = (Fo2 + 2Fc2)/3$
Δρmax, Δρmin (e Å−3)	0.49, −0.34

Computer programs: Apex2 v2014.1-0 (Bruker, 2014), *SAINT* V8.34A (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2014), SHELXLE Rev714 (Hübschle *et al.*, 2011).

Special details

Refinement:

The crystal under investigation was found to be non-merohedrically twinned. The orientation matrices for the two components were identified using the program Cell_Now, with the two components being related by a 180 degree rotation around the

reciprocal a axis. The two components were integrated using Saint and corrected for absorption using twinabs, resulting in the following statistics:

11095 data (4569 unique) involve domain 1 only, mean I/σ 20.8 11140 data (4500 unique) involve domain 2 only, mean I/σ 18.3 117504 data (23206 unique) involve 2 domains, mean I/σ 25.4 167 data (167 unique) involve 3 domains, mean I/σ 28.1

The exact twin matrix identified by the integration program was found to be:

0.99994 0.00085 1.02408 0.00004 − 1.00000 − 0.00014 0.00012 0.00231 − 0.99994

The structure was solved using direct methods with only the non-overlapping reflections of component 1. The structure was refined using the hklf 5 routine with all reflections of component 1 (including the overlapping ones), resulting in a BASF value of 0.4091 (6).

The Rint value given is for all reflections and is based on agreement between observed single and composite intensities and those calculated from refined unique intensities and twin fractions (TWINABS (Sheldrick, 2012).

Table 2: Bond Lengths (Å)**.48**

N1A—C14A 1.338(3)	N1E—C14E 1.337(3)
N1A—C1A 1.455(3)	N1E—C1E 1.459(3)
N1A—H1A 0.8800	N1E—H1E 0.8800
C1A—C2A 1.515(3)	C1E—C2E 1.520(3)
C1A—H1AA 0.9900	C1E—H1EA 0.9900
C1A—H1AB 0.9900	C1E—H1EB 0.9900
F1A—C21A 1.315(3)	F1E—C21E 1.325(3)
O1A—C14A 1.234(3)	O1E—C14E 1.240(3)
F2A—C21A 1.291(3)	F2E—C21E 1.331(3)
C2A—C3A 1.383(4)	C2E—C7E 1.380(3)
C2A—C7A 1.395(3)	C2E—C3E 1.388(4)
F3A—C21A 1.306(3)	F3E—C21E 1.337(3)
C3A—C4A 1.389(3)	C3E—C4E 1.394(3)
C3A—H3A 0.9500	C3E—H3E 0.9500
C4A—C5A 1.394(3)	C4E—C5E 1.399(3)
C4A—H4A 0.9500	C4E—H4E 0.9500
C5A—C6A 1.400(4)	C5E—C6E 1.391(4)
C5A—C8A 1.488(3)	C5E—C8E 1.493(3)
C6A—C7A 1.388(3)	C6E—C7E 1.387(3)
C6A—H6A 0.9500	C6E—H6E 0.9500
C7A—H7A 0.9500	C7E—H7E 0.9500
C8A—C13A 1.393(3)	C8E—C13E 1.387(3)
C8A—C9A 1.399(4)	C8E—C9E 1.402(4)
C9A—C10A 1.384(3)	C9E—C10E 1.394(3)
C9A—H9A 0.9500	C9E—H9E 0.9500
C10A—C11A 1.385(4)	C10E—C11E 1.385(4)
C10A—H10A 0.9500	C10E—H10E 0.9500
C11A—C12A 1.385(4)	C11E—C12E 1.377(4)
C11A—H11A 0.9500	C11E—H11E 0.9500
C12A—C13A 1.383(4)	C12E—C13E 1.393(4)
C12A—H12A 0.9500	C12E—H12E 0.9500
C13A—H13A 0.9500	C13E—H13E 0.9500
C14A—C15A 1.499(3)	C14E—C15E 1.506(3)
C15A—C16A 1.384(3)	C15E—C16E 1.382(4)
C15A—C20A 1.399(3)	C15E—C20E 1.400(3)
C16A—C17A 1.395(3)	C16E—C17E 1.390(3)
C16A—H16A 0.9500	C16E—H16E 0.9500
C17A—C18A 1.390(3)	C17E—C18E 1.394(3)
C17A—H17A 0.9500	C17E—H17E 0.9500
C18A—C19A 1.391(4)	C18E—C19E 1.384(4)
C18A—C21A 1.503(3)	C18E—C21E 1.497(3)
C19A—C20A 1.377(4)	C19E—C20E 1.383(3)
C19A—H19A 0.9500	C19E—H19E 0.9500
C20A—H20A 0.9500	C20E—H20E 0.9500
N1B—C14B 1.337(3)	N1F—C14F 1.334(3)

N1B—C1B 1.459(3)	N1F—C1F 1.456(3)
N1B—H1B 0.8800	N1F—H1F 0.8800
C1B—C2B 1.513(3)	C1F—C2F 1.515(3)
C1B—H1BA 0.9900	C1F—H1FA 0.9900
C1B—H1BB 0.9900	C1F—H1FB 0.9900
F1B—C21B 1.335(3)	O1F—C14F 1.237(3)
O1B—C14B 1.234(3)	F1F—C21F 1.322(4)
F2B—C21B 1.324(3)	C2F—C7F 1.388(3)
C2B—C3B 1.384(3)	C2F—C3F 1.391(4)
C2B—C7B 1.390(3)	F2F—C21F 1.325(3)
F3B—C21B 1.347(3)	C3F—C4F 1.388(3)
C3B—C4B 1.387(3)	C3F—H3F 0.9500
C3B—H3B 0.9500	F3F—C21F 1.323(3)
C4B—C5B 1.401(3)	C4F—C5F 1.393(3)
C4B—H4B 0.9500	C4F—H4F 0.9500
C5B—C6B 1.393(3)	C5F—C6F 1.394(3)
C5B—C8B 1.492(3)	C5F—C8F 1.490(3)
C6B—C7B 1.394(3)	C6F—C7F 1.394(3)
C6B—H6B 0.9500	C6F—H6F 0.9500
C7B—H7B 0.9500	C7F—H7F 0.9500
C8B—C9B 1.390(3)	C8F—C13F 1.394(3)
C8B—C13B 1.394(3)	C8F—C9F 1.398(3)
C9B—C10B 1.391(3)	C9F—C10F 1.392(3)
C9B—H9B 0.9500	C9F—H9F 0.9500
C10B—C11B 1.384(4)	C10F—C11F 1.385(4)
C10B—H10B 0.9500	C10F—H10F 0.9500
C11B—C12B 1.385(4)	C11F—C12F 1.382(4)
C11B—H11B 0.9500	C11F—H11F 0.9500
C12B—C13B 1.396(3)	C12F—C13F 1.394(3)
C12B—H12B 0.9500	C12F—H12F 0.9500
C13B—H13B 0.9500	C13F—H13F 0.9500
C14B—C15B 1.499(3)	C14F—C15F 1.503(3)
C15B—C16B 1.393(3)	C15F—C16F 1.392(3)
C15B—C20B 1.398(3)	C15F—C20F 1.393(3)
C16B—C17B 1.387(3)	C16F—C17F 1.379(4)
C16B—H16B 0.9500	C16F—H16F 0.9500
C17B—C18B 1.388(4)	C17F—C18F 1.388(3)
C17B—H17B 0.9500	C17F—H17F 0.9500
C18B—C19B 1.398(4)	C18F—C19F 1.376(4)
C18B—C21B 1.499(3)	C18F—C21F 1.501(3)
C19B—C20B 1.381(3)	C19F—C20F 1.388(4)
C19B—H19B 0.9500	C19F—H19F 0.9500
C20B—H20B 0.9500	C20F—H20F 0.9500
N1C—C14C 1.339(3)	N1G—C14G 1.336(3)
N1C—C1C 1.463(3)	N1G—C1G 1.463(3)
N1C—H1C 0.8800	N1G—H1G 0.8800

F1C—C21C 1.322(3)
O1C—C14C 1.233(3)
C1C—C2C 1.510(3)
C1C—H1CA 0.9900
C1C—H1CB 0.9900
F2C—C21C 1.335(3)
C2C—C7C 1.393(3)
C2C—C3C 1.395(3)
F3C—C21C 1.327(3)
C3C—C4C 1.388(3)
C3C—H3C 0.9500
C4C—C5C 1.409(3)
C4C—H4C 0.9500
C5C—C6C 1.398(3)
C5C—C8C 1.487(3)
C6C—C7C 1.384(3)
C6C—H6C 0.9500
C7C—H7C 0.9500
C8C—C9C 1.393(3)
C8C—C13C 1.396(3)
C9C—C10C 1.381(4)
C9C—H9C 0.9500
C10C—C11C 1.388(4)
C10C—H10C 0.9500
C11C—C12C 1.381(4)
C11C—H11C 0.9500
C12C—C13C 1.383(4)
C12C—H12C 0.9500
C13C—H13C 0.9500
C14C—C15C 1.505(3)
C15C—C16C 1.383(3)
C15C—C20C 1.392(3)
C16C—C17C 1.386(3)
C16C—H16C 0.9500
C17C—C18C 1.393(3)
C17C—H17C 0.9500
C18C—C19C 1.380(4)
C18C—C21C 1.498(3)
C19C—C20C 1.389(3)
C19C—H19C 0.9500
C20C—H20C 0.9500
N1D—C14D 1.336(3)
N1D—C1D 1.460(3)
N1D—H1D 0.8800
C1D—C2D 1.512(3)
C1D—H1DA 0.9900

C1G—C2G 1.504(3)
C1G—H1GA 0.9900
C1G—H1GB 0.9900
F1G—C21G 1.329(3)
O1G—C14G 1.239(3)
F2G—C21G 1.335(3)
C2G—C7G 1.391(3)
C2G—C3G 1.393(4)
F3G—C21G 1.344(3)
C3G—C4G 1.378(3)
C3G—H3G 0.9500
C4G—C5G 1.405(3)
C4G—H4G 0.9500
C5G—C6G 1.395(4)
C5G—C8G 1.483(3)
C6G—C7G 1.387(3)
C6G—H6G 0.9500
C7G—H7G 0.9500
C8G—C9G 1.396(4)
C8G—C13G 1.396(3)
C9G—C10G 1.385(4)
C9G—H9G 0.9500
C10G—C11G 1.395(4)
C10G—H10G 0.9500
C11G—C12G 1.377(4)
C11G—H11G 0.9500
C12G—C13G 1.388(3)
C12G—H12G 0.9500
C13G—H13G 0.9500
C14G—C15G 1.506(3)
C15G—C16G 1.380(3)
C15G—C20G 1.392(3)
C16G—C17G 1.391(3)
C16G—H16G 0.9500
C17G—C18G 1.388(3)
C17G—H17G 0.9500
C18G—C19G 1.384(4)
C18G—C21G 1.504(3)
C19G—C20G 1.394(3)
C19G—H19G 0.9500
C20G—H20G 0.9500
N1H—C14H 1.339(3)
N1H—C1H 1.465(3)
N1H—H1H 0.8800
C1H—C2H 1.509(3)
C1H—H1HA 0.9900

C1D—H1DB 0.9900	C1H—H1HB 0.9900
F1D—C21D 1.338(3)	F1H—C21H 1.323(3)
O1D—C14D 1.233(3)	O1H—C14H 1.231(3)
F2D—C21D 1.328(3)	F2H—C21H 1.344(3)
C2D—C3D 1.383(4)	C2H—C7H 1.387(3)
C2D—C7D 1.403(3)	C2H—C3H 1.390(3)
F3D—C21D 1.327(3)	F3H—C21H 1.326(3)
C3D—C4D 1.383(3)	C3H—C4H 1.383(3)
C3D—H3D 0.9500	C3H—H3H 0.9500
C4D—C5D 1.397(3)	C4H—C5H 1.406(3)
C4D—H4D 0.9500	C4H—H4H 0.9500
C5D—C6D 1.400(4)	C5H—C6H 1.387(4)
C5D—C8D 1.481(3)	C5H—C8H 1.480(3)
C6D—C7D 1.385(3)	C6H—C7H 1.388(3)
C6D—H6D 0.9500	C6H—H6H 0.9500
C7D—H7D 0.9500	C7H—H7H 0.9500
C8D—C9D 1.394(4)	C8H—C9H 1.389(4)
C8D—C13D 1.400(3)	C8H—C13H 1.403(4)
C9D—C10D 1.383(4)	C9H—C10H 1.392(4)
C9D—H9D 0.9500	C9H—H9H 0.9500
C10D—C11D 1.391(4)	C10H—C11H 1.388(4)
C10D—H10D 0.9500	C10H—H10H 0.9500
C11D—C12D 1.378(4)	C11H—C12H 1.380(5)
C11D—H11D 0.9500	C11H—H11H 0.9500
C12D—C13D 1.385(4)	C12H—C13H 1.399(4)
C12D—H12D 0.9500	C12H—H12H 0.9500
C13D—H13D 0.9500	C13H—H13H 0.9500
C14D—C15D 1.505(3)	C14H—C15H 1.504(3)
C15D—C16D 1.385(4)	C15H—C16H 1.387(4)
C15D—C20D 1.392(3)	C15H—C20H 1.389(3)
C16D—C17D 1.383(3)	C16H—C17H 1.392(3)
C16D—H16D 0.9500	C16H—H16H 0.9500
C17D—C18D 1.389(3)	C17H—C18H 1.384(4)
C17D—H17D 0.9500	C17H—H17H 0.9500
C18D—C19D 1.383(4)	C18H—C19H 1.393(4)
C18D—C21D 1.497(3)	C18H—C21H 1.498(3)
C19D—C20D 1.388(3)	C19H—C20H 1.396(3)
C19D—H19D 0.9500	C19H—H19H 0.9500
C20D—H20D 0.9500	C20H—H20H 0.9500

Table 3: Bond Angles (deg).**48**

C14A—N1A—C1A	121.30 (19)	C14E—N1E—C1E	120.87 (19)
C14A—N1A—H1A	119.3	C14E—N1E—H1E	119.6
C1A—N1A—H1A	119.3	C1E—N1E—H1E	119.6
N1A—C1A—C2A	115.2 (2)	N1E—C1E—C2E	114.5 (2)
N1A—C1A—H1AA	108.5	N1E—C1E—H1EA	108.6
C2A—C1A—H1AA	108.5	C2E—C1E—H1EA	108.6
N1A—C1A—H1AB	108.5	N1E—C1E—H1EB	108.6
C2A—C1A—H1AB	108.5	C2E—C1E—H1EB	108.6
H1AA—C1A—H1AB	107.5	H1EA—C1E—H1EB	107.6
C3A—C2A—C7A	117.7 (2)	C7E—C2E—C3E	118.6 (2)
C3A—C2A—C1A	122.6 (2)	C7E—C2E—C1E	120.8 (2)
C7A—C2A—C1A	119.7 (2)	C3E—C2E—C1E	120.5 (2)
C2A—C3A—C4A	121.6 (2)	C2E—C3E—C4E	120.3 (2)
C2A—C3A—H3A	119.2	C2E—C3E—H3E	119.8
C4A—C3A—H3A	119.2	C4E—C3E—H3E	119.8
C3A—C4A—C5A	121.1 (2)	C3E—C4E—C5E	121.3 (2)
C3A—C4A—H4A	119.4	C3E—C4E—H4E	119.4
C5A—C4A—H4A	119.4	C5E—C4E—H4E	119.4
C4A—C5A—C6A	117.3 (2)	C6E—C5E—C4E	117.5 (2)
C4A—C5A—C8A	120.5 (2)	C6E—C5E—C8E	121.2 (2)
C6A—C5A—C8A	122.2 (2)	C4E—C5E—C8E	121.3 (2)
C7A—C6A—C5A	121.3 (2)	C7E—C6E—C5E	121.0 (2)
C7A—C6A—H6A	119.4	C7E—C6E—H6E	119.5
C5A—C6A—H6A	119.4	C5E—C6E—H6E	119.5

C6A—C7A—C2A	121.0 (2)	C2E—C7E—C6E	121.3 (2)
C6A—C7A—H7A	119.5	C2E—C7E—H7E	119.4
C2A—C7A—H7A	119.5	C6E—C7E—H7E	119.4
C13A—C8A—C9A	117.4 (2)	C13E—C8E—C9E	118.3 (2)
C13A—C8A—C5A	121.3 (2)	C13E—C8E—C5E	121.5 (2)
C9A—C8A—C5A	121.2 (2)	C9E—C8E—C5E	120.2 (2)
C10A—C9A—C8A	121.3 (2)	C10E—C9E—C8E	120.8 (2)
C10A—C9A—H9A	119.3	C10E—C9E—H9E	119.6
C8A—C9A—H9A	119.3	C8E—C9E—H9E	119.6
C9A—C10A—C11A	120.3 (3)	C11E—C10E—C9E	119.9 (3)
C9A—C10A—H10A	119.9	C11E—C10E—H10E	120.1
C11A—C10A—H10	19.9	C9E—C10E—H10E	120.1
C12A—C11A—C10A	119.2 (2)	C12E—C11E—C10E	119.8 (2)
C12A—C11A—H11A	120.4	C12E—C11E—H11E	120.1
C10A—C11A—H11A	120.4	C10E—C11E—H11E	120.1
C13A—C12A—C11A	120.3 (2)	C11E—C12E—C13E	120.6 (2)
C13A—C12A—H12A	119.8	C11E—C12E—H12E	119.7
C11A—C12A—H12	119.8	C13E—C12E—H12E	119.7
C12A—C13A—C8A	121.5 (3)	C8E—C13E—C12E	120.7 (3)
C12A—C13A—H13A	119.3	C8E—C13E—H13E	119.7
C8A—C13A—H13A	119.3	C12E—C13E—H13E	119.7
O1A—C14A—N1A	122.3 (2)	O1E—C14E—N1E	122.9 (2)
O1A—C14A—C15A	121.3 (2)	O1E—C14E—C15E	120.6 (2)
N1A—C14A—C15A	116.35 (19)	N1E—C14E—C15E	116.5 (2)
C16A—C15A—C20A	119.5 (2)	C16E—C15E—C20E	119.9 (2)

C16A—C15A—C14A	123.2 (2)	C16E—C15E—C14E	119.7 (2)
C20A—C15A—C14A	117.2 (2)	C20E—C15E—C14E	120.3 (2)
C15A—C16A—C17A	120.3 (2)	C15E—C16E—C17E	120.6 (2)
C15A—C16A—H16A	119.8	C15E—C16E—H16E	119.7
C17A—C16A—H16A	119.8	C17E—C16E—H16E	119.7
C18A—C17A—C16A	119.3 (2)	C16E—C17E—C18E	119.4 (2)
C18A—C17A—H17A	120.4	C16E—C17E—H17E	120.3
C16A—C17A—H17A	120.4	C18E—C17E—H17E	120.3
C17A—C18A—C19A	120.7 (2)	C19E—C18E—C17E	120.0 (2)
C17A—C18A—C21A	120.3 (2)	C19E—C18E—C21E	119.3 (2)
C19A—C18A—C21A	119.0 (2)	C17E—C18E—C21E	120.7 (2)
C20A—C19A—C18A	119.5 (2)	C20E—C19E—C18E	120.6 (2)
C20A—C19A—H19A	120.2	C20E—C19E—H19E	119.7
C18A—C19A—H19A	120.2	C18E—C19E—H19E	119.7
C19A—C20A—C15A	120.6 (2)	C19E—C20E—C15E	119.5 (2)
C19A—C20A—H20A	119.7	C19E—C20E—H20E	120.3
C15A—C20A—H20A	119.7	C15E—C20E—H20E	120.3
F2A—C21A—F3A	107.7 (3)	F1E—C21E—F2E	106.6 (2)
F2A—C21A—F1A	106.4 (3)	F1E—C21E—F3E	105.5 (2)
F3A—C21A—F1A	104.1 (3)	F2E—C21E—F3E	106.3 (2)
F2A—C21A—C18A	112.8 (2)	F1E—C21E—C18E	113.7 (2)
F3A—C21A—C18A	112.2 (2)	F2E—C21E—C18E	112.3 (2)
F1A—C21A—C18A	113.1 (2)	F3E—C21E—C18E	111.9 (2)
C14B—N1B—C1B	121.20 (19)	C14F—N1F—C1F	121.72 (19)
C14B—N1B—H1B	119.4	C14F—N1F—H1F	119.1

C1B—N1B—H1B	119.4	C1F—N1F—H1F	119.1
N1B—C1B—C2B	113.1 (2)	N1F—C1F—C2F	114.1 (2)
N1B—C1B—H1BA	108.9	N1F—C1F—H1FA	108.7
C2B—C1B—H1BA	108.9	C2F—C1F—H1FA	108.7
N1B—C1B—H1BB	108.9	N1F—C1F—H1FB	108.7
C2B—C1B—H1BB	108.9	C2F—C1F—H1FB	108.7
H1BA—C1B—H1BB	107.8	H1FA—C1F—H1FB	107.6
C3B—C2B—C7B	118.6 (2)	C7F—C2F—C3F	118.5 (2)
C3B—C2B—C1B	120.9 (2)	C7F—C2F—C1F	120.7 (2)
C7B—C2B—C1B	120.5 (2)	C3F—C2F—C1F	120.8 (2)
C2B—C3B—C4B	121.0 (2)	C4F—C3F—C2F	120.7 (2)
C2B—C3B—H3B	119.5	C4F—C3F—H3F	119.7
C4B—C3B—H3B	119.5	C2F—C3F—H3F	119.7
C3B—C4B—C5B	120.9 (2)	C3F—C4F—C5F	121.1 (2)
C3B—C4B—H4B	119.6	C3F—C4F—H4F	119.4
C5B—C4B—H4B	119.6	C5F—C4F—H4F	119.4
C6B—C5B—C4B	117.9 (2)	C4F—C5F—C6F	118.1 (2)
C6B—C5B—C8B	121.0 (2)	C4F—C5F—C8F	121.3 (2)
C4B—C5B—C8B	121.1 (2)	C6F—C5F—C8F	120.5 (2)
C5B—C6B—C7B	120.8 (2)	C7F—C6F—C5F	120.6 (2)
C5B—C6B—H6B	119.6	C7F—C6F—H6F	119.7
C7B—C6B—H6B	119.6	C5F—C6F—H6F	119.7
C2B—C7B—C6B	120.8 (2)	C2F—C7F—C6F	120.9 (2)
C2B—C7B—H7B	119.6	C2F—C7F—H7F	119.5
C6B—C7B—H7B	119.6	C6F—C7F—H7F	119.5

C9B—C8B—C13B	118.6 (2)	C13F—C8F—C9F	118.2 (2)
C9B—C8B—C5B	120.8 (2)	C13F—C8F—C5F	121.6 (2)
C13B—C8B—C5B	120.6 (2)	C9F—C8F—C5F	120.3 (2)
C8B—C9B—C10B	120.8 (2)	C10F—C9F—C8F	121.0 (2)
C8B—C9B—H9B	119.6	C10F—C9F—H9F	119.5
C10B—C9B—H9B	119.6	C8F—C9F—H9F	119.5
C11B—C10B—C9B	120.0 (2)	C11F—C10F—C9F	119.9 (2)
C11B—C10B—H10B	120.0	C11F—C10F—H10F	120.0
C9B—C10B—H10B	120.0	C9F—C10F—H10F	120.0
C10B—C11B—C12B	120.1 (2)	C12F—C11F—C10F	120.0 (2)
C10B—C11B—H11B	119.9	C12F—C11F—H11F	120.0
C12B—C11B—H11B	119.9	C10F—C11F—H11F	120.0
C11B—C12B—C13B	119.6 (2)	C11F—C12F—C13F	120.1 (2)
C11B—C12B—H12B	120.2	C11F—C12F—H12F	119.9
C13B—C12B—H12B	120.2	C13F—C12F—H12F	119.9
C8B—C13B—C12B	120.8 (2)	C8F—C13F—C12F	120.8 (2)
C8B—C13B—H13B	119.6	C8F—C13F—H13F	119.6
C12B—C13B—H13B	119.6	C12F—C13F—H13F	119.6
O1B—C14B—N1B	122.8 (2)	O1F—C14F—N1F	122.8 (2)
O1B—C14B—C15B	121.3 (2)	O1F—C14F—C15F	121.4 (2)
N1B—C14B—C15B	115.88 (19)	N1F—C14F—C15F	115.8 (2)
C16B—C15B—C20B	119.7 (2)	C16F—C15F—C20F	119.5 (2)
C16B—C15B—C14B	122.5 (2)	C16F—C15F—C14F	122.6 (2)
C20B—C15B—C14B	117.7 (2)	C20F—C15F—C14F	117.9 (2)
C17B—C16B—C15B	120.0 (2)	C17F—C16F—C15F	119.9 (2)

C17B—C16B—H16B	120.0	C17F—C16F—H16F	120.0
C15B—C16B—H16B	120.0	C15F—C16F—H16F	120.0
C16B—C17B—C18B	119.8 (2)	C16F—C17F—C18F	120.0 (2)
C16B—C17B—H17B	120.1	C16F—C17F—H17F	120.0
C18B—C17B—H17B	120.1	C18F—C17F—H17F	120.0
C17B—C18B—C19B	120.6 (2)	C19F—C18F—C17F	120.6 (2)
C17B—C18B—C21B	119.7 (2)	C19F—C18F—C21F	119.9 (2)
C19B—C18B—C21B	119.6 (2)	C17F—C18F—C21F	119.5 (2)
C20B—C19B—C18B	119.3 (2)	C18F—C19F—C20F	119.6 (2)
C20B—C19B—H19B	120.3	C18F—C19F—H19F	120.2
C18B—C19B—H19B	120.3	C20F—C19F—H19F	120.2
C19B—C20B—C15B	120.5 (2)	C19F—C20F—C15F	120.3 (2)
C19B—C20B—H20B	119.8	C19F—C20F—H20F	119.9
C15B—C20B—H20B	119.8	C15F—C20F—H20F	119.9
F2B—C21B—F1B	107.4 (2)	F1F—C21F—F3F	106.2 (2)
F2B—C21B—F3B	107.0 (2)	F1F—C21F—F2F	106.2 (2)
F1B—C21B—F3B	105.8 (2)	F3F—C21F—F2F	106.6 (2)
F2B—C21B—C18B	113.1 (2)	F1F—C21F—C18F	113.2 (2)
F1B—C21B—C18B	110.4 (2)	F2F—C21F—C18F	112.9 (2)
C14C—N1C—C1C	121.91 (19)	C14G—N1G—C1G	122.86 (19)
C14C—N1C—H1C	119.0	C14G—N1G—H1G	118.6
C1C—N1C—H1C	119.0	C1G—N1G—H1G	118.6
N1C—C1C—C2C	112.5 (2)	N1G—C1G—C2G	113.79 (19)
N1C—C1C—H1CA	109.1	N1G—C1G—H1GA	108.8
C2C—C1C—H1CA	109.1	C2G—C1G—H1GA	108.8

N1C—C1C—H1CB	109.1	N1G—C1G—H1GB	108.8
C2C—C1C—H1CB	109.1	C2G—C1G—H1GB	108.8
H1CA—C1C—H1CB	107.8	H1GA—C1G—H1GB	107.7
C7C—C2C—C3C	118.3 (2)	C7G—C2G—C3G	118.0 (2)
C7C—C2C—C1C	120.9 (2)	C7G—C2G—C1G	120.5 (2)
C3C—C2C—C1C	120.8 (2)	C3G—C2G—C1G	121.5 (2)
C4C—C3C—C2C	120.8 (2)	C4G—C3G—C2G	121.4 (2)
C4C—C3C—H3C	119.6	C4G—C3G—H3G	119.3
C2C—C3C—H3C	119.6	C2G—C3G—H3G	119.3
C3C—C4C—C5C	121.1 (2)	C3G—C4G—C5G	121.1 (2)
C3C—C4C—H4C	119.5	C3G—C4G—H4G	119.4
C5C—C4C—H4C	119.5	C5G—C4G—H4G	119.4
C6C—C5C—C4C	117.3 (2)	C6G—C5G—C4G	117.1 (2)
C6C—C5C—C8C	121.7 (2)	C6G—C5G—C8G	121.4 (2)
C4C—C5C—C8C	120.9 (2)	C4G—C5G—C8G	121.4 (2)
C7C—C6C—C5C	121.4 (2)	C7G—C6G—C5G	121.6 (2)
C7C—C6C—H6C	119.3	C7G—C6G—H6G	119.2
C5C—C6C—H6C	119.3	C5G—C6G—H6G	119.2
C6C—C7C—C2C	121.0 (2)	C6G—C7G—C2G	120.8 (2)
C6C—C7C—H7C	119.5	C6G—C7G—H7G	119.6
C2C—C7C—H7C	119.5	C2G—C7G—H7G	119.6
C9C—C8C—C13C	117.9 (2)	C9G—C8G—C13G	118.0 (2)
C9C—C8C—C5C	121.3 (2)	C9G—C8G—C5G	121.0 (2)
C13C—C8C—C5C	120.8 (2)	C13G—C8G—C5G	121.0 (2)
C10C—C9C—C8C	121.3 (2)	C10G—C9G—C8G	121.2 (2)

C10C—C9C—H9C	119.4	C10G—C9G—H9G	119.4
C8C—C9C—H9C	119.4	C8G—C9G—H9G	119.4
C9C—C10C—C11C	120.1 (2)	C9G—C10G—C11G	120.1 (2)
C9C—C10C—H10C	120.0	C9G—C10G—H10G	120.0
C11C—C10C—H10C	120.0	C11G—C10G—H10G	120.0
C12C—C11C—C10C	119.4 (2)	C12G—C11G—C10G	119.3 (2)
C12C—C11C—H11C	120.3	C12G—C11G—H11G	120.4
C10C—C11C—H11C	120.3	C10G—C11G—H11G	120.4
C11C—C12C—C13	120.5 (2)	C11G—C12G—C13G	120.7 (2)
C11C—C12C—H12C	119.7	C11G—C12G—H12G	119.7
C13C—C12C—H12C	119.7	C13G—C12G—H12G	119.7
C12C—C13C—C8C	120.8 (2)	C12G—C13G—C8G	120.8 (2)
C12C—C13C—H13C	119.6	C12G—C13G—H13G	119.6
C8C—C13C—H13C	119.6	C8G—C13G—H13G	119.6
O1C—C14C—N1C	123.3 (2)	O1G—C14G—N1G	123.2 (2)
O1C—C14C—C15C	121.2 (2)	O1G—C14G—C15G	121.4 (2)
N1C—C14C—C15C	115.46 (19)	N1G—C14G—C15G	115.40 (19)
C16C—C15C—C20C	119.8 (2)	C16G—C15G—C20G	120.3 (2)
C16C—C15C—C14C	122.9 (2)	C16G—C15G—C14G	123.1 (2)
C20C—C15C—C14C	117.3 (2)	C20G—C15G—C14G	116.5 (2)
C15C—C16C—C17C	120.6 (2)	C15G—C16G—C17G	120.1 (2)
C15C—C16C—H16C	119.7	C15G—C16G—H16G	120.0
C17C—C16C—H16C	119.7	C17G—C16G—H16G	120.0
C16C—C17C—C18C	119.2 (2)	C18G—C17G—C16G	119.4 (2)
C16C—C17C—H17C	120.4	C18G—C17G—H17G	120.3

C18C—C17C—H17C	120.4	C16G—C17G—H17G	120.3
C19C—C18C—C17C	120.5 (2)	C19G—C18G—C17G	120.9 (2)
C19C—C18C—C21C	119.7 (2)	C19G—C18G—C21G	119.3 (2)
C17C—C18C—C21C	119.7 (2)	C17G—C18G—C21G	119.7 (2)
C18C—C19C—C20C	119.9 (2)	C18G—C19G—C20G	119.4 (2)
C18C—C19C—H19C	120.1	C18G—C19G—H19G	120.3
C20C—C19C—H19C	120.1	C20G—C19G—H19G	120.3
C19C—C20C—C15C	119.9 (2)	C15G—C20G—C19G	119.8 (2)
C19C—C20C—H20C	120.0	C15G—C20G—H20G	120.1
C15C—C20C—H20C	120.0	C19G—C20G—H20G	120.1
F1C—C21C—F3C	107.4 (2)	F1G—C21G—F2G	106.8 (2)
F1C—C21C—F2C	106.3 (2)	F1G—C21G—F3G	106.2 (2)
F3C—C21C—F2C	104.9 (2)	F2G—C21G—F3G	106.1 (2)
F1C—C21C—C18C	113.3 (2)	F1G—C21G—C18G	113.5 (2)
F3C—C21C—C18C	112.1 (2)	F2G—C21G—C18G	112.9 (2)
F2C—C21C—C18C	112.3 (2)	F3G—C21G—C18G	111.0 (2)
C14D—N1D—C1D	120.9 (2)	C14H—N1H—C1H	121.73 (19)
C14D—N1D—H1D	119.6	C14H—N1H—H1H	119.1
C1D—N1D—H1D	119.6	C1H—N1H—H1H	119.1
N1D—C1D—C2D	115.5 (2)	N1H—C1H—C2H	113.6 (2)
N1D—C1D—H1DA	108.4	N1H—C1H—H1HA	108.9
C2D—C1D—H1DA	108.4	C2H—C1H—H1HA	108.9
N1D—C1D—H1DB	108.4	N1H—C1H—H1HB	108.9
C2D—C1D—H1DB	108.4	C2H—C1H—H1HB 1	08.9
H1DA—C1D—H1DB	107.5	H1HA—C1H—H1HB	107.7

C3D—C2D—C7D	117.9 (2)	C7H—C2H—C3H	118.1 (2)
C3D—C2D—C1D	123.7 (2)	C7H—C2H—C1H	121.1 (2)
C7D—C2D—C1D	118.4 (2)	C3H—C2H—C1H	120.8 (2)
C4D—C3D—C2D	121.4 (2)	C4H—C3H—C2H	121.2 (2)
C4D—C3D—H3D	119.3	C4H—C3H—H3H	119.4
C2D—C3D—H3D	119.3	C2H—C3H—H3H	119.4
C3D—C4D—C5D	121.3 (2)	C3H—C4H—C5H	120.9 (2)
C3D—C4D—H4D	119.3	C3H—C4H—H4H	119.6
C5D—C4D—H4D	119.3	C5H—C4H—H4H	119.6
C4D—C5D—C6D	117.3 (2)	C6H—C5H—C4H	117.5 (2)
C4D—C5D—C8D	121.0 (2)	C6H—C5H—C8H	121.9 (2)
C6D—C5D—C8D	121.7 (2)	C4H—C5H—C8H	120.6 (2)
C7D—C6D—C5D	121.3 (2)	C5H—C6H—C7H	121.3 (2)
C7D—C6D—H6D	119.4	C5H—C6H—H6H	119.3
C5D—C6D—H6D	119.4	C7H—C6H—H6H	119.3
C6D—C7D—C2D	120.8 (2)	C2H—C7H—C6H	121.0 (2)
C6D—C7D—H7D	119.6	C2H—C7H—H7H	119.5
C2D—C7D—H7D	119.6	C6H—C7H—H7H	119.5
C9D—C8D—C13D	117.4 (2)	C9H—C8H—C13H	118.2 (2)
C9D—C8D—C5D	121.3 (2)	C9H—C8H—C5H	121.5 (2)
C13D—C8D—C5D	121.3 (2)	C13H—C8H—C5H	120.3 (2)
C10D—C9D—C8D	121.6 (2)	C8H—C9H—C10H	121.4 (2)
C10D—C9D—H9D	119.2	C8H—C9H—H9H	119.3
C8D—C9D—H9D	119.2	C10H—C9H—H9H	119.3
C9D—C10D—C11D	120.2 (2)	C11H—C10H—C9H	119.9 (3)

C9D—C10D—H10D	119.9	C11H—C10H—H10H	120.0
C11D—C10D—H10D	119.9	C9H—C10H—H10H	120.0
C12D—C11D—C10D	118.8 (2)	C12H—C11H—C10H	119.6 (2)
C12D—C11D—H11D	120.6	C12H—C11H—H11H	120.2
C10D—C11D—H11D	120.6	C10H—C11H—H11H	120.2
C11D—C12D—C13D	121.1 (2)	C11H—C12H—C13H	120.6 (3)
C11D—C12D—H12D	119.5	C11H—C12H—H12H	119.7
C13D—C12D—H12D	119.5	C13H—C12H—H12H	119.7
C12D—C13D—C8D	120.8 (3)	C12H—C13H—C8H	120.2 (3)
C12D—C13D—H13D	119.6	C12H—C13H—H13H	119.9
C8D—C13D—H13D	119.6	C8H—C13H—H13H	119.9
O1D—C14D—N1D	122.7 (2)	O1H—C14H—N1H	122.6 (2)
O1D—C14D—C15D	120.4 (2)	O1H—C14H—C15H	121.0 (2)
N1D—C14D—C15D	116.9 (2)	N1H—C14H—C15H	116.4 (2)
C16D—C15D—C20D	119.5 (2)	C16H—C15H—C20H	120.6 (2)
C16D—C15D—C14D	118.9 (2)	C16H—C15H—C14H	122.3 (2)
C20D—C15D—C14D	121.4 (2)	C20H—C15H—C14H	117.2 (2)
C17D—C16D—C15D	120.5 (2)	C15H—C16H—C17H	119.6 (2)
C17D—C16D—H16D	119.7	C15H—C16H—H16H	120.2
C15D—C16D—H16D	119.7	C17H—C16H—H16H	120.2
C16D—C17D—C18D	119.6 (2)	C18H—C17H—C16H	120.0 (2)
C16D—C17D—H17D	120.2	C18H—C17H—H17H	120.0
C18D—C17D—H17D	120.2	C16H—C17H—H17H	120.0
C19D—C18D—C17D	120.3 (2)	C17H—C18H—C19H	120.7 (2)
C19D—C18D—C21D	119.2 (2)	C17H—C18H—C21H	119.2 (2)

C17D—C18D—C21D	120.5 (2)	C19H—C18H—C21H	120.0 (2)
C18D—C19D—C20D	119.9 (2)	C18H—C19H—C20H	119.2 (2)
C18D—C19D—H19D	120.1	C18H—C19H—H19H	120.4
C20D—C19D—H19D	120.1	C20H—C19H—H19H	120.4
C19D—C20D—C15D	120.1 (2)	C15H—C20H—C19H	119.9 (2)
C19D—C20D—H20D	119.9	C15H—C20H—H20H	120.0
C15D—C20D—H20D	119.9	C19H—C20H—H20H	120.0
F3D—C21D—F2D	106.3 (2)	F1H—C21H—F3H	107.9 (2)
F3D—C21D—F1D	107.0 (2)	F1H—C21H—F2H	105.9 (2)
F2D—C21D—F1D	104.7 (2)	F3H—C21H—F2H	105.3 (2)
F3D—C21D—C18D	112.5 (2)	F1H—C21H—C18H	112.7 (2)
F2D—C21D—C18D	112.5 (2)	F3H—C21H—C18H	111.7 (2)
F1D—C21D—C18D	113.2 (2)	F2H—C21H—C18H	112.9 (2)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2). **48**

	x	*y*	*U*iso*/*U*eq
N1A	0.39983 (3)	0.35563 (19)	0.0176 (4)
H1A	0.4034	0.2756	0.021*
C1A	0.38380 (3)	0.3699 (2)	0.0183 (5)
H1AA	0.3826	0.2867	0.022*
H1AB	0.3868	0.4424	0.022*
F1A	0.47800 (3)	0.2633 (3)	0.0748 (8)
O1A	0.40434 (2)	0.57666 (15)	0.0225 (4)
F2A	0.48635 (3)	0.4602 (2)	0.0966 (11)
C2A	0.36472 (3)	0.3994 (2)	0.0170 (5)
F3A	0.49211 (3)	0.3138 (4)	0.0875 (10)
C3A	0.36245 (3)	0.4061 (2)	0.0190 (5)
H3A	0.3733	0.3944	0.023*
C4A	0.34471 (3)	0.4295 (2)	0.0184 (5)
H4A	0.3437	0.4338	0.022*
C5A	0.32843 (3)	0.4467 (2)	0.0174 (5)
C6A	0.33077 (3)	0.4414 (2)	0.0211 (5)
H6A	0.3200	0.4535	0.025*
C7A	0.34856 (3)	0.4187 (2)	0.0198 (5)
H7A	0.3498	0.4163	0.024*
C8A	0.30944 (3)	0.4686 (2)	0.0192 (5)
C9A	0.30776 (3)	0.5345 (2)	0.0207 (5)
H9A	0.3189	0.5663	0.025*
C10A	0.29012 (3)	0.5542 (3)	0.0253 (5)
H10A	0.2893	0.5999	0.030*
C11A	0.27364 (3)	0.5075 (3)	0.0292 (6)
H11A	0.2615	0.5218	0.035*
C12A	0.27506 (4)	0.4399 (3)	0.0332 (6)
H12A	0.2639	0.4063	0.040*
C13A	0.29270 (4)	0.4212 (3)	0.0286 (6)
H13A	0.2934	0.3749	0.034*
C14A	0.40925 (3)	0.4618 (2)	0.0178 (5)
C15A	0.42668 (3)	0.4342 (2)	0.0178 (5)
C16A	0.42766 (3)	0.3323 (2)	0.0191 (5)
H16A	0.4168	0.2781	0.023*
C17A	0.44456 (3)	0.3090 (2)	0.0223 (5)
H17A	0.4452	0.2406	0.027*
C18A	0.46052 (3)	0.3870 (2)	0.0208 (5)
C19A	0.45956 (3)	0.4909 (2)	0.0245 (5)
H19A	0.4705	0.5448	0.029*
C20A	0.44268 (3)	0.5150 (2)	0.0222 (5)

H20A	0.4419	0.5871	0.027*
C21A	0.47925 (3)	0.3578 (3)	0.0243 (5)
N1B	0.39800 (3)	0.85628 (19)	0.0184 (4)
H1B	0.4016	0.7767	0.022*
C1B	0.38107 (3)	0.8697 (2)	0.0201 (5)
H1BA	0.3791	0.7860	0.024*
H1BB	0.3833	0.9416	0.024*
F1B	0.47700 (2)	0.78896 (19)	0.0419 (4)
O1B	0.40366 (2)	1.07717 (16)	0.0222 (4)
F2B	0.48948 (2)	0.96045 (17)	0.0437 (4)
C2B	0.36312 (3)	0.9008 (2)	0.0164 (4)
F3B	0.48897 (2)	0.77370 (17)	0.0343 (4)
C3B	0.35318 (3)	0.8012 (2)	0.0195 (5)
H3B	0.3579	0.7127	0.023*
C4B	0.33642 (3)	0.8288 (2)	0.0194 (5)
H4B	0.3300	0.7593	0.023*
C5B	0.32888 (3)	0.9578 (2)	0.0170 (5)
C6B	0.33900 (3)	1.0579 (2)	0.0191 (5)
H6B	0.3343	1.1464	0.023*
C7B	0.35596 (3)	1.0296 (2)	0.0190 (5)
H7B	0.3627	1.0992	0.023*
C8B	0.31041 (3)	0.9869 (2)	0.0186 (5)
C9B	0.30712 (3)	1.1102 (2)	0.0219 (5)
H9B	0.3167	1.1767	0.026*
C10B	0.28989 (4)	1.1373 (3)	0.0267 (5)
H10B	0.2879	1.2216	0.032*
C11B	0.27569 (3)	1.0415 (3)	0.0273 (6)
H11B	0.2639	1.0602	0.033*
C12B	0.27862 (3)	0.9185 (3)	0.0263 (5)
H12B	0.2689	0.8528	0.032*
C13B	0.29598 (3)	0.8914 (2)	0.0217 (5)
H13B	0.2980	0.8069	0.026*
C14B	0.40816 (3)	0.9626 (2)	0.0175 (5)
C15B	0.42592 (3)	0.9345 (2)	0.0179 (5)
C16B	0.42741 (3)	0.8268 (2)	0.0198 (5)
H16B	0.4168	0.7691	0.024*
C17B	0.44437 (3)	0.8039 (2)	0.0223 (5)
H17B	0.4453	0.7313	0.027*
C18B	0.45991 (3)	0.8874 (2)	0.0219 (5)
C19B	0.45845 (3)	0.9970 (2)	0.0228 (5)
H19B	0.4691	1.0547	0.027*
C20B	0.44144 (3)	1.0205 (2)	0.0200 (5)
H20B	0.4403	1.0957	0.024*
C21B	0.47880 (4)	0.8543 (3)	0.0271 (5)
N1C	0.34966 (3)	0.13351 (19)	0.0185 (4)

H1C	0.3462	0.0570	0.022*
F1C	0.27181 (2)	0.0933 (2)	0.0559 (6)
O1C	0.34366 (2)	0.35432 (16)	0.0229 (4)
C1C	0.36640 (3)	0.1378 (2)	0.0215 (5)
H1CA	0.3647	0.2095	0.026*
H1CB	0.3674	0.0525	0.026*
F2C	0.25972 (3)	0.26422 (19)	0.0547 (5)
C2C	0.38472 (3)	0.1617 (2)	0.0179 (5)
F3C	0.25813 (2)	0.0789 (2)	0.0505 (5)
C3C	0.39332 (3)	0.0596 (2)	0.0204 (5)
H3C	0.3875	−0.0257	0.024*
C4C	0.41027 (3)	0.0812 (2)	0.0200 (5)
H4C	0.4158	0.0108	0.024*
C5C	0.41945 (3)	0.2058 (2)	0.0183 (5)
C6C	0.41053 (3)	0.3074 (2)	0.0201 (5)
H6C	0.4163	0.3928	0.024*
C7C	0.39346 (3)	0.2861 (2)	0.0196 (5)
H7C	0.3876	0.3575	0.024*
C8C	0.43804 (3)	0.2269 (2)	0.0190 (5)
C9C	0.45061 (3)	0.1219 (2)	0.0228 (5)
H9C	0.4472	0.0349	0.027*
C10C	0.46797 (4)	0.1417 (3)	0.0274 (6)
H10C	0.4763	0.0686	0.033*
C11C	0.47328 (4)	0.2684 (3)	0.0288 (6)
H11C	0.4851	0.2823	0.035*
C12C	0.46116 (4)	0.3741 (3)	0.0289 (6)
H12C	0.4649	0.4613	0.035*
C13C	0.44371 (4)	0.3540 (2)	0.0255 (5)
H13C	0.4355	0.4275	0.031*
C14C	0.33946 (3)	0.2426 (2)	0.0167 (5)
C15C	0.32190 (3)	0.2202 (2)	0.0183 (5)
C16C	0.32113 (3)	0.1270 (2)	0.0229 (5)
H16C	0.3321	0.0756	0.027*
C17C	0.30445 (3)	0.1080 (2)	0.0248 (5)
H17C	0.3041	0.0456	0.030*
C18C	0.28823 (3)	0.1815 (2)	0.0213 (5)
C19C	0.28894 (3)	0.2762 (2)	0.0240 (5)
H19C	0.2779	0.3270	0.029*
C20C	0.30585 (3)	0.2969 (2)	0.0231 (5)
H20C	0.3065	0.3634	0.028*
C21C	0.26967 (3)	0.1536 (3)	0.0260 (5)
N1D	0.35110 (3)	0.63926 (19)	0.0193 (4)
H1D	0.3459	0.5598	0.023*
C1D	0.36767 (3)	0.6581 (2)	0.0219 (5)
H1DA	0.3655	0.7380	0.026*

H1DB	0.3686	0.5809	0.026*
F1D	0.27200 (2)	0.7067 (2)	0.0421 (4)
O1D	0.35115 (2)	0.85200 (16)	0.0238 (4)
F2D	0.25939 (3)	0.7811 (2)	0.0580 (6)
C2D	0.38663 (3)	0.6739 (2)	0.0172 (5)
F3D	0.26243 (3)	0.5723 (2)	0.0549 (6)
C3D	0.38934 (3)	0.6467 (2)	0.0191 (5)
H3D	0.3789	0.6163	0.023*
C4D	0.40700 (3)	0.6627 (2)	0.0176 (5)
H4D	0.4084	0.6433	0.021*
C5D	0.42284 (3)	0.7069 (2)	0.0172 (5)
C6D	0.42010 (3)	0.7348 (2)	0.0219 (5)
H6D	0.4306	0.7651	0.026*
C7D	0.40239 (3)	0.7190 (2)	0.0214 (5)
H7D	0.4009	0.7390	0.026*
C8D	0.44170 (3)	0.7240 (2)	0.0186 (5)
C9D	0.44317 (3)	0.7603 (2)	0.0209 (5)
H9D	0.4318	0.7751	0.025*
C10D	0.46073 (3)	0.7753 (2)	0.0225 (5)
H10D	0.4613	0.7997	0.027*
C11D	0.47752 (4)	0.7549 (3)	0.0267 (6)
H11D	0.4896	0.7652	0.032*
C12D	0.47630 (4)	0.7193 (3)	0.0346 (6)
H12D	0.4877	0.7049	0.041*
C13D	0.45874 (4)	0.7044 (3)	0.0299 (6)
H13D	0.4583	0.6805	0.036*
C14D	0.34366 (3)	0.7410 (2)	0.0182 (5)
C15D	0.32523 (3)	0.7156 (2)	0.0184 (5)
C16D	0.32297 (3)	0.7672 (2)	0.0226 (5)
H16D	0.3334	0.8112	0.027*
C17D	0.30570 (3)	0.7551 (2)	0.0236 (5)
H17D	0.3043	0.7888	0.028*
C18D	0.29039 (3)	0.6934 (2)	0.0199 (5)
C19D	0.29249 (3)	0.6415 (2)	0.0235 (5)
H19D	0.2820	0.5993	0.028*
C20D	0.30996 (3)	0.6514 (2)	0.0212 (5)
H20D	0.3115	0.6142	0.025*
C21D	0.27121 (4)	0.6876 (3)	0.0256 (5)
N1E	0.39866 (3)	−0.03534 (19)	0.0199 (4)
H1E	0.4038	−0.1151	0.024*
C1E	0.38145 (3)	−0.0120 (3)	0.0228 (5)
H1EA	0.3801	−0.0850	0.027*
H1EB	0.3831	0.0718	0.027*
F1E	0.48051 (2)	0.0052 (2)	0.0467 (5)
O1E	0.39958 (2)	0.17549 (16)	0.0235 (4)

F2E	0.48560 (2)	−0.15479 (18)	0.0495 (5)
C2E	0.36314 (3)	−0.0029 (2)	0.0190 (5)
F3E	0.49333 (2)	0.0437 (2)	0.0567 (6)
C3E	0.35817 (3)	−0.1034 (2)	0.0209 (5)
H3E	0.3664	−0.1778	0.025*
C4E	0.34121 (3)	−0.0955 (2)	0.0207 (5)
H4E	0.3380	−0.1648	0.025*
C5E	0.32877 (3)	0.0124 (2)	0.0195 (5)
C6E	0.33383 (3)	0.1107 (2)	0.0239 (5)
H6E	0.3255	0.1844	0.029*
C7E	0.35078 (3)	0.1029 (2)	0.0233 (5)
H7E	0.3539	0.1716	0.028*
C8E	0.31074 (3)	0.0219 (2)	0.0203 (5)
C9E	0.30977 (3)	−0.0333 (2)	0.0220 (5)
H9E	0.3207	−0.0766	0.026*
C10E	0.29302 (4)	−0.0256 (2)	0.0268 (6)
H10E	0.2926	−0.0628	0.032*
C11E	0.27704 (4)	0.0365 (2)	0.0285 (6)
H11E	0.2656	0.0418	0.034*
C12E	0.27782 (4)	0.0905 (3)	0.0298 (6)
H12E	0.2669	0.1335	0.036*
C13E	0.29451 (3)	0.0827 (2)	0.0249 (5)
H13E	0.2948	0.1193	0.030*
C14E	0.40665 (3)	0.0628 (2)	0.0183 (5)
C15E	0.42530 (3)	0.0311 (2)	0.0180 (5)
C16E	0.42825 (3)	0.0715 (2)	0.0220 (5)
H16E	0.4182	0.1141	0.026*
C17E	0.44577 (3)	0.0503 (2)	0.0221 (5)
H17E	0.4476	0.0758	0.027*
C18E	0.46060 (3)	−0.0087 (2)	0.0207 (5)
C19E	0.45769 (3)	−0.0483 (3)	0.0257 (5)
H19E	0.4678	−0.0884	0.031*
C20E	0.44013 (3)	−0.0300 (2)	0.0230 (5)
H20E	0.4382	−0.0588	0.028*
C21E	0.47982 (3)	−0.0288 (2)	0.0252 (5)
N1F	0.39945 (3)	0.45722 (19)	0.0184 (4)
H1F	0.4025	0.3783	0.022*
C1F	0.38324 (3)	0.4695 (2)	0.0200 (5)
H1FA	0.3822	0.3870	0.024*
H1FB	0.3857	0.5437	0.024*
O1F	0.40600 (2)	0.67709 (16)	0.0240 (4)
F1F	0.47633 (2)	0.3871 (2)	0.0566 (6)
C2F	0.36434 (3)	0.4941 (2)	0.0173 (5)
F2F	0.49048 (3)	0.5436 (2)	0.0585 (6)
C3F	0.35925 (3)	0.4216 (2)	0.0208 (5)

H3F	0.3680	0.3590	0.025*
F3F	0.48853 (3)	0.3528 (2)	0.0664 (7)
C4F	0.34153 (3)	0.4396 (2)	0.0199 (5)
H4F	0.3384	0.3896	0.024*
C5F	0.32833 (3)	0.5298 (2)	0.0180 (5)
C6F	0.33350 (3)	0.6032 (2)	0.0203 (5)
H6F	0.3248	0.6657	0.024*
C7F	0.35136 (3)	0.5857 (2)	0.0186 (5)
H7F	0.3547	0.6371	0.022*
C8F	0.30906 (3)	0.5463 (2)	0.0181 (5)
C9F	0.29998 (3)	0.4383 (2)	0.0221 (5)
H9F	0.3063	0.3547	0.027*
C10F	0.28185 (3)	0.4514 (3)	0.0255 (5)
H10F	0.2758	0.3771	0.031*
C11F	0.27256 (3)	0.5730 (3)	0.0259 (5)
H11F	0.2602	0.5821	0.031*
C12F	0.28137 (3)	0.6813 (3)	0.0262 (5)
H12F	0.2751	0.7650	0.031*
C13F	0.29946 (3)	0.6677 (2)	0.0221 (5)
H13F	0.3053	0.7422	0.027*
C14F	0.40986 (3)	0.5624 (2)	0.0184 (5)
C15F	0.42717 (3)	0.5330 (2)	0.0192 (5)
C16F	0.42747 (3)	0.4323 (2)	0.0221 (5)
H16F	0.4163	0.3816	0.027*
C17F	0.44404 (3)	0.4063 (2)	0.0240 (5)
H17F	0.4442	0.3388	0.029*
C18F	0.46051 (3)	0.4787 (2)	0.0225 (5)
C19F	0.46028 (3)	0.5806 (2)	0.0255 (5)
H19F	0.4715	0.6312	0.031*
C20F	0.44353 (3)	0.6091 (2)	0.0234 (5)
H20F	0.4432	0.6809	0.028*
C21F	0.47889 (4)	0.4410 (3)	0.0274 (6)
N1G	0.34945 (3)	−0.16530 (18)	0.0170 (4)
H1G	0.3457	−0.0864	0.020*
C1G	0.36546 (3)	−0.1732 (2)	0.0193 (5)
H1GA	0.3641	−0.1017	0.023*
H1GB	0.3648	−0.2594	0.023*
F1G	0.27103 (2)	−0.08769 (17)	0.0373 (4)
O1G	0.34529 (2)	−0.38785 (15)	0.0205 (3)
F2G	0.26000 (2)	−0.27321 (17)	0.0464 (5)
C2G	0.38475 (3)	−0.1608 (2)	0.0180 (5)
F3G	0.25829 (2)	−0.09987 (19)	0.0410 (4)
C3G	0.38956 (3)	−0.2353 (2)	0.0206 (5)
H3G	0.3803	−0.2933	0.025*
C4G	0.40743 (3)	−0.2265 (2)	0.0211 (5)

H4G	0.4103	−0.2781	0.025*
C5G	0.42154 (3)	−0.1425 (2)	0.0191 (5)
C6G	0.41663 (3)	−0.0686 (2)	0.0197 (5)
H6G	0.4259	−0.0111	0.024*
C7G	0.39858 (3)	−0.0770 (2)	0.0190 (5)
H7G	0.3956	−0.0249	0.023*
C8G	0.44095 (3)	−0.1350 (2)	0.0205 (5)
C9G	0.44924 (3)	−0.2455 (2)	0.0234 (5)
H9G	0.4422	−0.3260	0.028*
C10G	0.46755 (4)	−0.2399 (3)	0.0277 (6)
H10G	0.4730	−0.3163	0.033*
C11G	0.47803 (4)	−0.1221 (3)	0.0270 (5)
H11G	0.4907	−0.1181	0.032*
C12G	0.46987 (3)	−0.0117 (3)	0.0263 (6)
H12G	0.4768	0.0693	0.032*
C13G	0.45158 (3)	−0.0176 (2)	0.0223 (5)
H13G	0.4462	0.0592	0.027*
C14G	0.34021 (3)	−0.2726 (2)	0.0162 (5)
C15G	0.32269 (3)	−0.2447 (2)	0.0166 (5)
C16G	0.32100 (3)	−0.1350 (2)	0.0184 (5)
H16G	0.3314	−0.0754	0.022*
C17G	0.30401 (3)	−0.1115 (2)	0.0202 (5)
H17G	0.3029	−0.0378	0.024*
C18G	0.28864 (3)	−0.1968 (2)	0.0196 (5)
C19G	0.29036 (3)	−0.3091 (2)	0.0225 (5)
H19G	0.2799	−0.3688	0.027*
C20G	0.30758 (3)	−0.3338 (2)	0.0210 (5)
H20G	0.3090	−0.4112	0.025*
C21G	0.26965 (3)	−0.1648 (3)	0.0254 (5)
N1H	0.35124 (3)	0.33006 (19)	0.0188 (4)
H1H	0.3475	0.4083	0.023*
C1H	0.36827 (3)	0.3215 (2)	0.0212 (5)
H1HA	0.3680	0.3955	0.025*
H1HB	0.3678	0.2372	0.025*
F1H	0.27216 (3)	0.3278 (3)	0.0670 (7)
O1H	0.34609 (2)	0.10876 (16)	0.0257 (4)
F2H	0.25782 (3)	0.20596 (18)	0.0503 (5)
C2H	0.38680 (3)	0.3275 (2)	0.0183 (5)
F3H	0.26134 (2)	0.41189 (17)	0.0414 (4)
C3H	0.39190 (3)	0.2260 (2)	0.0194 (5)
H3H	0.3835	0.1524	0.023*
C4H	0.40894 (3)	0.2301 (2)	0.0198 (5)
H4H	0.4121	0.1593	0.024*
C5H	0.42159 (3)	0.3378 (2)	0.0201 (5)
C6H	0.41638 (3)	0.4386 (2)	0.0255 (5)

H6H	0.4247	0.5125	0.031*
C7H	0.39931 (3)	0.4334 (2)	0.0235 (5)
H7H	0.3961	0.5036	0.028*
C8H	0.43977 (3)	0.3419 (2)	0.0220 (5)
C9H	0.44128 (3)	0.2867 (2)	0.0242 (5)
H9H	0.4304	0.2454	0.029*
C10H	0.45839 (4)	0.2906 (3)	0.0289 (6)
H10H	0.4591	0.2518	0.035*
C11H	0.47435 (4)	0.3513 (3)	0.0342 (7)
H11H	0.4860	0.3553	0.041*
C12H	0.47318 (4)	0.4059 (3)	0.0368 (7)
H12H	0.4841	0.4467	0.044*
C13H	0.45603 (3)	0.4018 (3)	0.0288 (6)
H13H	0.4554	0.4396	0.035*
C14H	0.34118 (3)	0.2219 (2)	0.0183 (5)
C15H	0.32307 (3)	0.2455 (2)	0.0187 (5)
C16H	0.32182 (3)	0.3406 (2)	0.0231 (5)
H16H	0.3326	0.3939	0.028*
C17H	0.30467 (3)	0.3574 (2)	0.0238 (5)
H17H	0.3038	0.4211	0.029*
C18H	0.28891 (3)	0.2813 (2)	0.0216 (5)
C19H	0.29006 (3)	0.1858 (2)	0.0212 (5)
H19H	0.2792	0.1342	0.025*
C20H	0.30740 (3)	0.1669 (2)	0.0196 (5)
H20H	0.3085	0.1003	0.024*

Table 5: Atomic displacement parameters (Å2).**48**

	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
N1A	0.0204 (9)	0.0120 (9)	0.0207 (11)	0.0011 (7)	0.0059 (8)	0.0013 (7)
C1A	0.0220 (11)	0.0147 (11)	0.0188 (12)	−0.0008 (9)	0.0063 (9)	0.0002 (9)
F1A	0.0332 (9)	0.1111 (19)	0.0679 (16)	−0.0051 (11)	−0.0076 (9)	0.0577 (15)
O1A	0.0300 (9)	0.0113 (8)	0.0251 (10)	0.0011 (6)	0.0054 (7)	0.0022 (7)
F2A	0.0732 (15)	0.0505 (13)	0.117 (2)	0.0130 (11)	−0.0596 (15)	−0.0301 (14)
C2A	0.0216 (11)	0.0099 (10)	0.0203 (12)	−0.0011 (8)	0.0069 (9)	−0.0011 (8)
F3A	0.0378 (10)	0.186 (3)	0.0408 (13)	0.0484 (15)	0.0150 (9)	0.0125 (15)
C3A	0.0194 (11)	0.0167 (11)	0.0191 (12)	−0.0026 (8)	0.0022 (9)	0.0027 (9)
C4A	0.0225 (11)	0.0162 (11)	0.0172 (12)	−0.0021 (9)	0.0063 (9)	−0.0008 (9)
C5A	0.0221 (11)	0.0079 (9)	0.0224 (13)	−0.0004 (8)	0.0061 (9)	−0.0005 (9)
C6A	0.0220 (11)	0.0207 (11)	0.0179 (12)	0.0014 (9)	0.0008 (9)	−0.0001 (9)
C7A	0.0267 (12)	0.0178 (11)	0.0148 (12)	0.0009 (9)	0.0056 (9)	−0.0012 (9)
C8A	0.0200 (11)	0.0133 (11)	0.0237 (14)	0.0007 (8)	0.0048 (9)	0.0048 (9)
C9A	0.0236 (11)	0.0145 (11)	0.0254 (14)	−0.0020 (9)	0.0088 (10)	0.0016 (9)
C10A	0.0307 (13)	0.0204 (12)	0.0282 (14)	0.0016 (10)	0.0136 (11)	0.0044 (10)
C11A	0.0234 (12)	0.0282 (13)	0.0378 (18)	0.0024 (10)	0.0114 (11)	0.0077 (11)
C12A	0.0196 (12)	0.0395 (15)	0.0364 (16)	−0.0006 (11)	0.0004 (11)	0.0018 (13)
C13A	0.0249 (12)	0.0347 (15)	0.0251 (14)	0.0007 (10)	0.0047 (10)	−0.0030 (11)
C14A	0.0212 (11)	0.0136 (11)	0.0213 (13)	−0.0003 (8)	0.0105 (9)	0.0020 (9)
C15A	0.0206 (11)	0.0124 (11)	0.0221 (13)	0.0012 (8)	0.0087 (9)	−0.0039 (9)
C16A	0.0202 (11)	0.0177 (11)	0.0203 (13)	−0.0009 (9)	0.0068 (9)	−0.0008 (9)
C17A	0.0250 (12)	0.0197 (11)	0.0229 (13)	0.0011 (9)	0.0077 (10)	0.0011 (9)
C18A	0.0218 (11)	0.0200 (12)	0.0217 (13)	0.0019 (9)	0.0078 (9)	−0.0048 (9)
C19A	0.0244 (11)	0.0224 (12)	0.0294 (16)	−0.0052 (10)	0.0120 (10)	−0.0011 (10)
C20A	0.0253 (11)	0.0146 (11)	0.0280 (15)	−0.0039	0.0092	0.0005 (9)

				(9)	(10)	
C21A	0.0220 (11)	0.0263 (13)	0.0248 (14)	0.0003 (10)	0.0069 (10)	−0.0023 (10)
N1B	0.0183 (9)	0.0127 (9)	0.0233 (11)	0.0016 (7)	0.0038 (8)	−0.0006 (8)
C1B	0.0184 (10)	0.0197 (11)	0.0216 (13)	0.0005 (9)	0.0043 (9)	0.0000 (9)
F1B	0.0340 (8)	0.0609 (11)	0.0294 (10)	0.0110 (8)	0.0059 (7)	0.0122 (8)
O1B	0.0251 (8)	0.0126 (8)	0.0268 (10)	0.0006 (6)	0.0032 (7)	0.0007 (7)
F2B	0.0359 (8)	0.0375 (9)	0.0434 (11)	−0.0009 (7)	−0.0139 (8)	−0.0089 (8)
C2B	0.0164 (10)	0.0175 (11)	0.0144 (11)	−0.0008 (8)	0.0024 (8)	−0.0012 (9)
F3B	0.0255 (7)	0.0447 (9)	0.0318 (9)	0.0116 (7)	0.0059 (6)	−0.0031 (7)
C3B	0.0232 (11)	0.0138 (11)	0.0202 (12)	0.0050 (9)	0.0037 (9)	0.0009 (9)
C4B	0.0213 (11)	0.0170 (11)	0.0200 (13)	−0.0009 (9)	0.0060 (9)	0.0048 (9)
C5B	0.0185 (10)	0.0176 (11)	0.0131 (12)	0.0000 (8)	0.0012 (8)	0.0005 (9)
C6B	0.0208 (11)	0.0143 (10)	0.0209 (13)	0.0014 (9)	0.0035 (9)	0.0024 (9)
C7B	0.0221 (11)	0.0172 (11)	0.0179 (13)	−0.0025 (9)	0.0055 (9)	0.0008 (9)
C8B	0.0209 (11)	0.0209 (12)	0.0134 (13)	0.0033 (9)	0.0037 (9)	0.0038 (9)
C9B	0.0226 (11)	0.0218 (12)	0.0208 (13)	0.0023 (9)	0.0049 (9)	−0.0003 (9)
C10B	0.0303 (13)	0.0279 (13)	0.0224 (14)	0.0116 (10)	0.0079 (10)	−0.0013 (10)
C11B	0.0222 (12)	0.0381 (15)	0.0232 (14)	0.0096 (10)	0.0089 (10)	0.0048 (11)
C12B	0.0212 (11)	0.0293 (13)	0.0278 (15)	−0.0014 (10)	0.0058 (10)	0.0050 (11)
C13B	0.0248 (11)	0.0222 (12)	0.0188 (13)	0.0022 (9)	0.0071 (9)	0.0020 (9)
C14B	0.0195 (10)	0.0143 (11)	0.0216 (13)	0.0010 (8)	0.0104 (9)	0.0015 (9)
C15B	0.0195 (11)	0.0142 (11)	0.0212 (13)	0.0019 (9)	0.0075 (9)	−0.0052 (9)
C16B	0.0215 (11)	0.0175 (11)	0.0225 (13)	0.0014 (9)	0.0094 (9)	−0.0038 (9)
C17B	0.0264 (12)	0.0199 (12)	0.0212 (13)	0.0053 (9)	0.0075 (10)	−0.0005 (9)
C18B	0.0223 (11)	0.0245 (12)	0.0184 (13)	0.0050 (9)	0.0044 (9)	−0.0067 (10)
C19B	0.0209 (11)	0.0217 (12)	0.0273 (16)	−0.0003 (9)	0.0089 (10)	−0.0062 (10)
C20B	0.0237 (11)	0.0129 (11)	0.0248 (14)	−0.0011 (9)	0.0089 (10)	−0.0030 (9)
C21B	0.0258 (12)	0.0281 (13)	0.0261 (14)	0.0051 (10)	0.0051 (10)	−0.0038 (11)
N1C	0.0186 (9)	0.0155 (9)	0.0217 (11)	−0.0013 (7)	0.0058 (8)	0.0025 (8)
F1C	0.0277 (8)	0.0993 (16)	0.0441 (11)	−0.0031 (9)	0.0156 (8)	0.0280 (11)
O1C	0.0315 (9)	0.0147 (8)	0.0249 (10)	−0.0017	0.0117 (7)	0.0026 (7)

				(7)		
C1C	0.0205 (11)	0.0211 (12)	0.0245 (13)	−0.0011 (9)	0.0089 (10)	−0.0020 (10)
F2C	0.0384 (9)	0.0419 (10)	0.0959 (17)	0.0066 (8)	0.0387 (11)	−0.0040 (10)
C2C	0.0176 (10)	0.0189 (11)	0.0204 (13)	0.0001 (9)	0.0106 (9)	−0.0019 (9)
F3C	0.0340 (9)	0.0687 (13)	0.0522 (12)	−0.0232 (9)	0.0174 (8)	−0.0272 (10)
C3C	0.0237 (11)	0.0158 (11)	0.0243 (13)	−0.0030 (9)	0.0108 (10)	0.0003 (9)
C4C	0.0231 (11)	0.0168 (11)	0.0216 (13)	−0.0011 (9)	0.0087 (9)	0.0024 (9)
C5C	0.0203 (11)	0.0175 (11)	0.0204 (13)	0.0002 (9)	0.0112 (9)	−0.0029 (9)
C6C	0.0215 (11)	0.0136 (11)	0.0286 (14)	−0.0006 (9)	0.0126 (10)	−0.0033 (9)
C7C	0.0211 (11)	0.0154 (11)	0.0243 (13)	0.0045 (9)	0.0092 (9)	0.0028 (9)
C8C	0.0197 (11)	0.0212 (12)	0.0187 (13)	−0.0035 (9)	0.0098 (9)	−0.0002 (9)
C9C	0.0282 (12)	0.0188 (12)	0.0218 (13)	−0.0021 (9)	0.0071 (10)	0.0017 (9)
C10C	0.0250 (12)	0.0276 (13)	0.0287 (15)	−0.0013 (10)	0.0056 (10)	0.0038 (11)
C11C	0.0219 (12)	0.0381 (15)	0.0264 (15)	−0.0065 (11)	0.0062 (10)	−0.0037 (11)
C12C	0.0281 (13)	0.0240 (13)	0.0353 (16)	−0.0063 (10)	0.0098 (11)	−0.0109 (11)
C13C	0.0257 (12)	0.0202 (12)	0.0315 (15)	0.0012 (10)	0.0096 (11)	−0.0057 (10)
C14C	0.0212 (11)	0.0165 (11)	0.0107 (11)	−0.0012 (9)	0.0015 (9)	0.0010 (8)
C15C	0.0218 (11)	0.0137 (11)	0.0191 (12)	−0.0003 (8)	0.0050 (9)	−0.0010 (9)
C16C	0.0203 (11)	0.0229 (12)	0.0249 (13)	0.0034 (9)	0.0047 (10)	0.0044 (10)
C17C	0.0285 (13)	0.0225 (12)	0.0245 (14)	0.0016 (10)	0.0091 (10)	0.0041 (10)
C18C	0.0227 (12)	0.0204 (12)	0.0213 (13)	−0.0006 (9)	0.0064 (9)	−0.0055 (10)
C19C	0.0214 (11)	0.0228 (12)	0.0260 (14)	0.0073 (9)	0.0029 (10)	0.0028 (10)
C20C	0.0256 (12)	0.0172 (11)	0.0260 (14)	0.0027 (9)	0.0059 (10)	0.0037 (10)
C21C	0.0236 (12)	0.0311 (14)	0.0240 (14)	−0.0014 (10)	0.0074 (10)	−0.0035 (11)
N1D	0.0198 (9)	0.0146 (9)	0.0232 (11)	−0.0017 (7)	0.0052 (8)	−0.0006 (8)

C1D	0.0217 (11)	0.0243 (12)	0.0206 (13)	0.0019 (9)	0.0075 (10)	0.0015 (10)
F1D	0.0359 (8)	0.0653 (12)	0.0310 (10)	−0.0074 (8)	0.0192 (7)	−0.0035 (8)
O1D	0.0222 (8)	0.0143 (8)	0.0348 (11)	−0.0017 (6)	0.0076 (7)	0.0023 (7)
F2D	0.0340 (9)	0.0824 (15)	0.0612 (14)	0.0223 (9)	0.0194 (9)	0.0323 (11)
C2D	0.0207 (11)	0.0120 (10)	0.0193 (12)	−0.0010 (8)	0.0059 (9)	−0.0015 (9)
F3D	0.0456 (10)	0.0558 (12)	0.0759 (15)	−0.0299 (9)	0.0380 (10)	−0.0289 (10)
C3D	0.0216 (11)	0.0143 (10)	0.0240 (13)	0.0020 (8)	0.0108 (9)	0.0023 (9)
C4D	0.0234 (11)	0.0138 (11)	0.0178 (12)	0.0015 (8)	0.0095 (9)	0.0011 (9)
C5D	0.0235 (11)	0.0103 (10)	0.0191 (13)	0.0002 (8)	0.0081 (9)	−0.0015 (9)
C6D	0.0231 (11)	0.0217 (12)	0.0239 (13)	−0.0014 (9)	0.0115 (10)	0.0004 (10)
C7D	0.0267 (12)	0.0223 (12)	0.0169 (12)	−0.0001 (9)	0.0087 (10)	0.0007 (9)
C8D	0.0221 (11)	0.0103 (10)	0.0243 (13)	−0.0001 (8)	0.0079 (10)	−0.0001 (9)
C9D	0.0237 (11)	0.0163 (11)	0.0245 (13)	0.0007 (9)	0.0098 (10)	0.0013 (9)
C10D	0.0294 (12)	0.0177 (12)	0.0208 (13)	0.0011 (9)	0.0075 (10)	−0.0009 (9)
C11D	0.0228 (12)	0.0260 (13)	0.0277 (15)	−0.0041 (10)	0.0005 (10)	−0.0005 (11)
C12D	0.0204 (12)	0.0514 (17)	0.0335 (16)	−0.0028 (12)	0.0101 (11)	−0.0078 (13)
C13D	0.0247 (12)	0.0403 (15)	0.0260 (15)	−0.0032 (11)	0.0088 (11)	−0.0066 (12)
C14D	0.0188 (11)	0.0162 (11)	0.0171 (12)	0.0006 (9)	0.0006 (9)	0.0044 (9)
C15D	0.0218 (11)	0.0121 (10)	0.0198 (12)	0.0014 (8)	0.0033 (9)	0.0041 (9)
C16D	0.0228 (11)	0.0210 (12)	0.0219 (13)	−0.0039 (9)	0.0023 (9)	0.0002 (9)
C17D	0.0302 (13)	0.0229 (12)	0.0191 (13)	−0.0018 (10)	0.0090 (10)	−0.0027 (10)
C18D	0.0237 (12)	0.0172 (11)	0.0197 (13)	−0.0013 (9)	0.0071 (9)	0.0028 (9)
C19D	0.0224 (11)	0.0228 (12)	0.0245 (14)	−0.0053 (9)	0.0049 (10)	−0.0024 (10)
C20D	0.0251 (12)	0.0201 (12)	0.0195 (13)	−0.0026 (9)	0.0079 (9)	−0.0003 (9)
C21D	0.0252 (12)	0.0317 (14)	0.0213 (14)	−0.0014 (10)	0.0087 (10)	−0.0007 (10)
N1E	0.0192 (9)	0.0164 (10)	0.0233 (12)	0.0023 (7)	0.0044 (8)	−0.0010 (8)

C1E	0.0212 (11)	0.0258 (12)	0.0212 (14)	−0.0007 (9)	0.0051 (10)	−0.0019 (10)
F1E	0.0366 (9)	0.0728 (12)	0.0258 (10)	0.0139 (9)	−0.0002 (7)	−0.0066 (8)
O1E	0.0245 (8)	0.0143 (8)	0.0302 (10)	0.0023 (6)	0.0048 (7)	0.0011 (7)
F2E	0.0413 (9)	0.0426 (10)	0.0513 (12)	0.0200 (8)	−0.0107 (8)	−0.0072 (8)
C2E	0.0209 (11)	0.0199 (11)	0.0150 (13)	−0.0024 (9)	0.0025 (9)	−0.0053 (9)
F3E	0.0239 (8)	0.0821 (15)	0.0579 (13)	−0.0137 (8)	0.0004 (8)	0.0284 (11)
C3E	0.0210 (11)	0.0170 (11)	0.0215 (13)	0.0025 (9)	0.0000 (9)	−0.0008 (9)
C4E	0.0200 (11)	0.0195 (11)	0.0218 (13)	−0.0002 (9)	0.0044 (9)	0.0019 (9)
C5E	0.0189 (10)	0.0163 (11)	0.0217 (14)	−0.0013 (9)	0.0028 (9)	−0.0053 (9)
C6E	0.0246 (12)	0.0144 (11)	0.0308 (14)	0.0030 (9)	0.0043 (10)	−0.0003 (10)
C7E	0.0273 (12)	0.0172 (11)	0.0257 (14)	−0.0016 (9)	0.0075 (10)	0.0026 (10)
C8E	0.0205 (11)	0.0141 (11)	0.0251 (15)	−0.0025 (8)	0.0040 (9)	−0.0075 (9)
C9E	0.0225 (11)	0.0165 (11)	0.0249 (14)	0.0015 (9)	0.0030 (10)	−0.0013 (10)
C10E	0.0309 (12)	0.0220 (13)	0.0292 (16)	−0.0029 (10)	0.0112 (11)	−0.0056 (10)
C11E	0.0233 (12)	0.0220 (13)	0.0426 (18)	−0.0010 (9)	0.0130 (11)	−0.0096 (11)
C12E	0.0211 (12)	0.0253 (13)	0.0394 (17)	0.0041 (10)	0.0020 (11)	−0.0038 (12)
C13E	0.0253 (12)	0.0203 (12)	0.0270 (14)	0.0025 (9)	0.0031 (10)	−0.0021 (10)
C14E	0.0212 (11)	0.0153 (11)	0.0203 (13)	−0.0010 (9)	0.0087 (9)	0.0030 (9)
C15E	0.0207 (11)	0.0152 (11)	0.0195 (13)	−0.0020 (8)	0.0075 (9)	0.0007 (9)
C16E	0.0219 (11)	0.0193 (12)	0.0266 (14)	0.0025 (9)	0.0097 (10)	−0.0011 (10)
C17E	0.0250 (11)	0.0233 (12)	0.0190 (13)	0.0014 (9)	0.0074 (9)	−0.0015 (10)
C18E	0.0226 (11)	0.0165 (11)	0.0243 (15)	−0.0011 (9)	0.0085 (10)	0.0015 (9)
C19E	0.0240 (12)	0.0294 (13)	0.0258 (15)	0.0047 (10)	0.0102 (10)	−0.0003 (11)
C20E	0.0267 (12)	0.0232 (13)	0.0215 (14)	0.0034 (9)	0.0105 (10)	−0.0014 (10)
C21E	0.0261 (12)	0.0243 (13)	0.0257 (15)	−0.0005	0.0075	−0.0002 (10)

				(9)	(10)	
N1F	0.0197 (9)	0.0141 (9)	0.0207 (11)	0.0026 (7)	0.0041 (8)	0.0038 (8)
C1F	0.0206 (11)	0.0211 (12)	0.0181 (13)	0.0006 (9)	0.0049 (9)	−0.0008 (9)
O1F	0.0331 (9)	0.0154 (8)	0.0226 (9)	0.0004 (7)	0.0059 (7)	0.0035 (7)
F1F	0.0317 (9)	0.0944 (16)	0.0387 (11)	0.0063 (9)	0.0006 (8)	0.0284 (11)
C2F	0.0210 (11)	0.0155 (10)	0.0148 (13)	−0.0009 (9)	0.0038 (9)	−0.0041 (8)
F2F	0.0379 (9)	0.0521 (12)	0.0658 (14)	−0.0128 (8)	−0.0202 (9)	0.0036 (10)
C3F	0.0220 (11)	0.0197 (12)	0.0188 (13)	0.0036 (9)	0.0023 (9)	0.0017 (9)
F3F	0.0481 (11)	0.0923 (16)	0.0471 (12)	0.0445 (11)	−0.0076 (9)	−0.0268 (11)
C4F	0.0236 (11)	0.0194 (11)	0.0173 (12)	−0.0024 (9)	0.0067 (9)	0.0021 (9)
C5F	0.0208 (10)	0.0138 (11)	0.0193 (13)	−0.0013 (8)	0.0050 (9)	−0.0037 (9)
C6F	0.0219 (11)	0.0161 (11)	0.0211 (13)	0.0029 (9)	0.0023 (9)	0.0028 (9)
C7F	0.0230 (11)	0.0160 (11)	0.0177 (12)	−0.0022 (9)	0.0069 (9)	0.0015 (9)
C8F	0.0226 (11)	0.0198 (11)	0.0116 (12)	−0.0007 (9)	0.0038 (9)	−0.0030 (9)
C9F	0.0248 (12)	0.0208 (12)	0.0205 (13)	0.0018 (9)	0.0058 (9)	−0.0012 (9)
C10F	0.0262 (12)	0.0301 (13)	0.0213 (14)	−0.0057 (10)	0.0084 (10)	−0.0017 (11)
C11F	0.0206 (11)	0.0344 (14)	0.0242 (14)	−0.0007 (10)	0.0085 (10)	−0.0042 (11)
C12F	0.0253 (12)	0.0257 (13)	0.0255 (14)	0.0060 (10)	0.0035 (10)	−0.0021 (10)
C13F	0.0248 (12)	0.0202 (12)	0.0212 (13)	0.0000 (9)	0.0060 (9)	−0.0017 (9)
C14F	0.0231 (11)	0.0154 (11)	0.0203 (13)	0.0010 (9)	0.0122 (9)	0.0011 (9)
C15F	0.0199 (11)	0.0172 (11)	0.0220 (14)	0.0003 (9)	0.0082 (9)	−0.0020 (9)
C16F	0.0220 (11)	0.0214 (12)	0.0245 (14)	−0.0024 (9)	0.0090 (10)	0.0010 (10)
C17F	0.0258 (12)	0.0201 (12)	0.0257 (14)	0.0003 (9)	0.0060 (10)	0.0034 (10)
C18F	0.0219 (11)	0.0221 (12)	0.0235 (15)	0.0006 (9)	0.0060 (10)	−0.0067 (10)
C19F	0.0218 (11)	0.0260 (13)	0.0289 (15)	−0.0091 (10)	0.0073 (10)	−0.0058 (10)
C20F	0.0253 (12)	0.0188 (11)	0.0270 (14)	−0.0049 (9)	0.0084 (10)	0.0004 (10)
C21F	0.0274 (13)	0.0276 (13)	0.0275 (15)	0.0016 (10)	0.0077 (11)	−0.0033 (11)
N1G	0.0184 (9)	0.0101 (9)	0.0236 (11)	−0.0011 (7)	0.0074 (8)	−0.0006 (7)

C1G	0.0209 (11)	0.0200 (11)	0.0182 (12)	−0.0011 (9)	0.0074 (9)	−0.0018 (9)
F1G	0.0271 (7)	0.0497 (10)	0.0375 (10)	−0.0002 (7)	0.0126 (7)	−0.0157 (8)
O1G	0.0275 (8)	0.0120 (8)	0.0231 (9)	0.0021 (6)	0.0087 (7)	0.0000 (6)
F2G	0.0356 (9)	0.0360 (9)	0.0786 (14)	−0.0110 (7)	0.0340 (9)	−0.0052 (9)
C2G	0.0214 (11)	0.0128 (11)	0.0219 (13)	0.0014 (8)	0.0095 (9)	−0.0027 (9)
F3G	0.0232 (7)	0.0579 (11)	0.0393 (10)	0.0108 (7)	0.0039 (7)	0.0068 (8)
C3G	0.0211 (11)	0.0198 (11)	0.0238 (13)	−0.0035 (9)	0.0107 (10)	0.0023 (9)
C4G	0.0255 (12)	0.0212 (12)	0.0185 (13)	−0.0010 (9)	0.0094 (10)	0.0033 (9)
C5G	0.0220 (11)	0.0169 (11)	0.0213 (13)	0.0002 (9)	0.0109 (10)	−0.0040 (9)
C6G	0.0214 (11)	0.0158 (11)	0.0249 (13)	−0.0027 (9)	0.0113 (10)	−0.0002 (9)
C7G	0.0228 (11)	0.0152 (11)	0.0214 (13)	0.0011 (9)	0.0099 (9)	0.0014 (9)
C8G	0.0237 (11)	0.0225 (11)	0.0172 (12)	−0.0003 (9)	0.0088 (9)	−0.0044 (9)
C9G	0.0267 (12)	0.0242 (12)	0.0198 (13)	−0.0030 (10)	0.0072 (10)	−0.0005 (10)
C10G	0.0306 (13)	0.0292 (13)	0.0227 (14)	0.0029 (10)	0.0063 (11)	0.0024 (11)
C11G	0.0227 (12)	0.0351 (14)	0.0221 (13)	−0.0008 (10)	0.0043 (10)	−0.0036 (11)
C12G	0.0268 (12)	0.0292 (13)	0.0254 (15)	−0.0096 (10)	0.0111 (10)	−0.0090 (10)
C13G	0.0241 (11)	0.0216 (12)	0.0223 (14)	−0.0011 (9)	0.0082 (10)	−0.0033 (9)
C14G	0.0204 (11)	0.0136 (11)	0.0132 (12)	−0.0010 (8)	0.0020 (9)	0.0006 (8)
C15G	0.0199 (11)	0.0123 (10)	0.0164 (12)	0.0011 (8)	0.0028 (9)	0.0045 (8)
C16G	0.0193 (10)	0.0135 (10)	0.0212 (12)	−0.0020 (8)	0.0030 (9)	0.0012 (9)
C17G	0.0231 (11)	0.0164 (11)	0.0209 (13)	0.0000 (9)	0.0058 (9)	−0.0011 (9)
C18G	0.0216 (11)	0.0188 (11)	0.0184 (13)	0.0010 (9)	0.0052 (9)	0.0040 (9)
C19G	0.0202 (11)	0.0213 (12)	0.0246 (14)	−0.0072 (9)	0.0037 (10)	0.0014 (10)
C20G	0.0244 (12)	0.0152 (11)	0.0225 (13)	−0.0024 (9)	0.0050 (10)	−0.0007 (9)
C21G	0.0208 (11)	0.0259 (13)	0.0285 (15)	−0.0009 (10)	0.0046 (10)	−0.0018 (10)
N1H	0.0201 (9)	0.0109 (9)	0.0237 (11)	0.0007 (7)	0.0032 (8)	0.0005 (8)
C1H	0.0218 (11)	0.0183 (11)	0.0240 (13)	0.0003 (9)	0.0068	0.0022 (9)

					(10)	
F1H	0.0408 (10)	0.139 (2)	0.0240 (10)	0.0213 (12)	0.0125 (8)	−0.0065 (11)
O1H	0.0286 (9)	0.0108 (8)	0.0395 (11)	0.0016 (7)	0.0122 (8)	0.0008 (7)
F2H	0.0438 (10)	0.0393 (10)	0.0808 (15)	−0.0100 (8)	0.0391 (10)	−0.0031 (10)
C2H	0.0202 (11)	0.0166 (11)	0.0206 (12)	0.0025 (9)	0.0096 (9)	−0.0014 (9)
F3H	0.0297 (8)	0.0359 (9)	0.0616 (12)	0.0113 (7)	0.0173 (8)	0.0124 (8)
C3H	0.0214 (11)	0.0147 (11)	0.0242 (14)	−0.0016 (9)	0.0100 (9)	−0.0016 (9)
C4H	0.0221 (11)	0.0162 (11)	0.0233 (13)	0.0043 (9)	0.0100 (10)	0.0032 (9)
C5H	0.0191 (11)	0.0190 (11)	0.0242 (13)	0.0016 (9)	0.0096 (10)	−0.0031 (9)
C6H	0.0235 (12)	0.0208 (12)	0.0322 (15)	−0.0061 (9)	0.0076 (10)	0.0014 (10)
C7H	0.0264 (12)	0.0181 (12)	0.0257 (14)	−0.0023 (9)	0.0063 (10)	0.0047 (10)
C8H	0.0201 (11)	0.0172 (11)	0.0287 (14)	0.0036 (9)	0.0065 (10)	−0.0040 (10)
C9H	0.0236 (11)	0.0201 (12)	0.0288 (15)	0.0046 (9)	0.0069 (10)	−0.0023 (10)
C10H	0.0312 (13)	0.0270 (13)	0.0242 (14)	0.0071 (10)	−0.0001 (11)	−0.0066 (10)
C11H	0.0238 (12)	0.0316 (14)	0.0391 (17)	0.0077 (11)	−0.0057 (11)	−0.0106 (12)
C12H	0.0211 (13)	0.0415 (16)	0.0470 (19)	−0.0017 (11)	0.0077 (12)	−0.0095 (14)
C13H	0.0215 (12)	0.0327 (14)	0.0320 (15)	−0.0008 (10)	0.0071 (10)	−0.0034 (11)
C14H	0.0202 (11)	0.0133 (11)	0.0194 (13)	0.0006 (8)	0.0019 (9)	0.0021 (9)
C15H	0.0214 (11)	0.0136 (10)	0.0200 (13)	0.0039 (9)	0.0033 (9)	0.0062 (9)
C16H	0.0238 (11)	0.0184 (11)	0.0244 (14)	−0.0024 (9)	0.0016 (10)	−0.0024 (10)
C17H	0.0282 (12)	0.0227 (12)	0.0200 (13)	0.0027 (10)	0.0052 (10)	−0.0014 (10)
C18H	0.0237 (12)	0.0223 (12)	0.0181 (13)	0.0048 (9)	0.0045 (9)	0.0078 (10)
C19H	0.0216 (11)	0.0181 (11)	0.0219 (13)	−0.0024 (9)	0.0024 (9)	0.0031 (9)
C20H	0.0231 (11)	0.0145 (11)	0.0211 (13)	0.0006 (9)	0.0057 (9)	0.0015 (9)
C21H	0.0272 (12)	0.0279 (13)	0.0209 (14)	0.0011 (10)	0.0068 (10)	0.0042 (10)

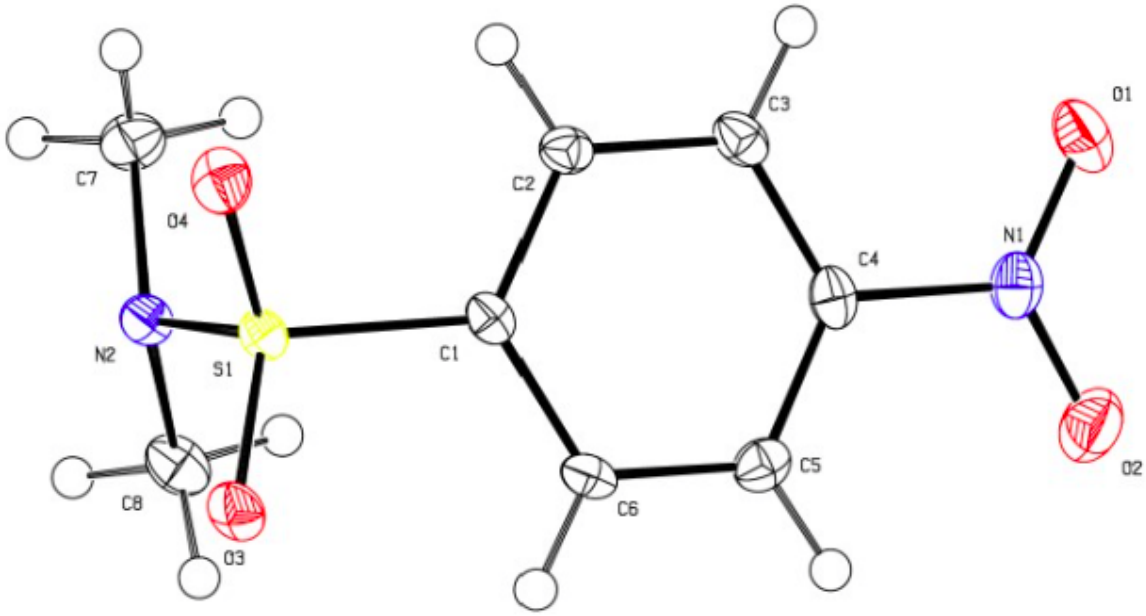

Figure 182: X-Ray Crystal Structure of *N*,*N*-dimethyl-4-nitrobenzenesulfonamide **6**

Table 1. Experimental details. **6**

	Prosp14mz122_0m
Crystal data	
Chemical formula	$C_8H_{10}N_2O_4S$
M_r	230.24
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, b, c (Å)	Cell setting: monoclinic 6.0899 (6), 16.1054 (16), 9.9594 (10)
β (°)	Cell setting: monoclinic 96.289 (5)
V (Å^3)	970.94 (17)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.99
Crystal size (mm)	0.33 × 0.27 × 0.05
Data collection	
Diffractometer	Bruker AXS Prospector CCD diffractometer
Absorption correction	Multi-scan Apex2 v2014.1-0 (Bruker, 2014)
T_{min}, T_{max}	0.421, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4711, 1651, 1565
R_{int}	0.067
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.147, 1.12
No. of reflections	1651
No. of parameters	138
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.56, −0.70

Table 2. Bond Lengths (Å). **6**

S1—O4	1.432(2)	C3—C4	1.386(4)
S1—O3	1.435(2)	C3—H3	0.9500
S1—N2	1.634(2)	C4—C5	1.381(3)
S1—C1	1.768(2)	C5—C6	1.384(4)
O1—N1	1.226(3)	C5—H5	0.9500
O2—N1	1.224(3)	C6—H6	0.9500
N1—C4	1.481(3)	C7—H7A	0.9800
N2—C7	1.475(3)	C7—H7B	0.9800
N2—C8	1.479(3)	C7—H7C	0.9800
C1—C2	1.396(3)	C8—H8A	0.9800
C1—C6	1.402(3)	C8—H8B	0.9800
C2—C3	1.393(4)	C8—H8C	0.9800
C2—H2	0.9500		

Table 3. Bond Angles (deg).**6**

O4—S1—O3	120.13 (12)	C5—C4—C3	123.4 (2)
O4—S1—N2	107.28 (11)	C5—C4—N1	118.7 (2)
O3—S1—N2	107.16 (10)	C3—C4—N1	117.9 (2)
O4—S1—C1	107.89 (11)	C4—C5—C6	18.5 (2)
O3—S1—C1	107.47 (11)	C4—C5—H5	120.7
N2—S1—C1	106.13 (11)	C6—C5—H5	120.7
O2—N1—O1	124.7 (2)	C5—C6—C1	119.2 (2)
O2—N1—C4	117.6 (2)	C5—C6—H6	120.4
O1—N1—C4	117.8 (2)	C1—C6—H6	120.4
C7—N2—C8	113.00 (19)	N2—C7—H7A	109.5
C7—N2—S1	117.03 (17)	N2—C7—H7B	109.5
C8—N2—S1	115.17 (16)	H7A—C7—H7B	109.5
C2—C1—C6	121.4 (2)	N2—C7—H7C	109.5
C2—C1—S1	119.53 (19)	H7A—C7—H7C	109.5
C6—C1—S1	118.84 (18)	H7B—C7—H7C	109.5
C3—C2—C1	119.2 (2)	N2—C8—H8A	109.5
C3—C2—H2	120.4	N2—C8—H8B	109.5
C1—C2—H2	120.4	H8A—C8—H8B	109.5
C4—C3—C2	118.2 (2)	N2—C8—H8C	109.5
C4—C3—H3	120.9	H8A—C8—H8C	109.5
C2—C3—H3	120.9	H8B—C8—H8C	109.5
O4—S1—N2—C7	41.2 (2)	S1—C1—C2—C3	−175.08 (18)
O3—S1—N2—C7	171.46 (18)	C1—C2—C3—C4	0.7 (3)

C1—S1—N2—C7	−73.9 (2)	C2—C3—C4—C5	−0.9 (4)
O4—S1—N2—C8	177.63 (16)	C2—C3—C4—N1	178.0 (2)
O3—S1—N2—C8	−52.1 (2)	O2—N1—C4—C5	5.6 (3)
C1—S1—N2—C8	62.49 (19)	O1—N1—C4—C5	−174.2 (2)
O4—S1—C1—C2	−34.1 (2)	O2—N1—C4—C3	−173.3 (2)
O3—S1—C1—C2	−164.99 (19)	O1—N1—C4—C3	6.8 (3)
N2—S1—C1—C2	80.6 (2)	C3—C4—C5—C6	0.7 (4)
O4—S1—C1—C6	151.0 (2)	N1—C4—C5—C6	−178.2 (2)
O3—S1—C1—C6	20.1 (2)	C4—C5—C6—C1	−0.3 (4)
N2—S1—C1—C6	−94.3 (2)	C2—C1—C6—C5	0.1 (4)
C6—C1—C2—C3	−0.3 (4)	S1—C1—C6—C5	174.90 (18)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2).**6**

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.22617 (10)	0.24884 (3)	0.14831 (6)	0.0133 (3)
O1	−0.1540 (3)	−0.02164 (12)	−0.32327 (19)	0.0273 (5)
O2	−0.4392 (3)	−0.01335 (13)	−0.2111 (2)	0.0297 (5)
O3	0.1278 (3)	0.24719 (10)	0.27284 (19)	0.0190 (5)
O4	0.4599 (3)	0.23871 (11)	0.1491 (2)	0.0216 (5)
N1	−0.2497 (4)	0.00464 (12)	−0.2299 (2)	0.0192 (5)
N2	0.1605 (3)	0.33768 (13)	0.0754 (2)	0.0150 (5)
C1	0.0948 (4)	0.17151 (15)	0.0420 (2)	0.0138 (5)
C2	0.1908 (4)	0.14594 (15)	−0.0721 (2)	0.0151 (5)
H2	0.3313	0.1666	−0.0889	0.018*
C3	0.0781 (4)	0.08975 (15)	−0.1610 (2)	0.0169 (6)
H3	0.1404	0.0708	−0.2389	0.020*
C4	−0.1275 (4)	0.06218 (14)	−0.1327 (2)	0.0159 (6)
C5	−0.2247 (4)	0.08661 (15)	−0.0202 (2)	0.0169 (6)
H5	−0.3653	0.0657	−0.0040	0.020*
C6	−0.1128 (4)	0.14221 (15)	0.0686 (2)	0.0150 (5)
H6	−0.1760	0.1603	0.1466	0.018*
C7	0.2694 (5)	0.35887 (17)	−0.0453 (3)	0.0227 (6)
H7A	0.1933	0.3308	−0.1244	0.034*

H7B	0.4239	0.3408	−0.0321	0.034*
H7C	0.2634	0.4191	−0.0594	0.034*
C8	−0.0780 (4)	0.35759 (17)	0.0602 (3)	0.0210 (6)
H8A	−0.0977	0.4179	0.0539	0.031*
H8B	−0.1450	0.3367	0.1387	0.031*
H8C	−0.1495	0.3314	−0.0220	0.031*

Table 5: Atomic displacement parameters (Å2).**6**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S1	0.0154 (4)	0.0143 (4)	0.0100 (4)	0.0011 (2)	−0.0003 (3)	−0.00029 (18)
O1	0.0371 (12)	0.0235 (11)	0.0211 (10)	0.0007 (9)	0.0027 (9)	−0.0073 (8)
O2	0.0275 (11)	0.0312 (11)	0.0299 (11)	−0.0100 (9)	0.0006 (9)	−0.0018 (8)
O3	0.0272 (11)	0.0202 (10)	0.0098 (9)	−0.0008 (7)	0.0022 (8)	−0.0003 (6)
O4	0.0171 (10)	0.0230 (10)	0.0234 (10)	0.0015 (7)	−0.0036 (8)	−0.0025 (7)
N1	0.0233 (13)	0.0135 (11)	0.0200 (11)	−0.0002 (10)	−0.0012 (10)	0.0022 (9)
N2	0.0180 (11)	0.0138 (10)	0.0135 (10)	0.0016 (8)	0.0028 (8)	0.0017 (8)
C1	0.0172 (12)	0.0123 (12)	0.0116 (11)	0.0027 (10)	0.0008 (9)	0.0003 (9)
C2	0.0159 (12)	0.0140 (12)	0.0160 (12)	0.0024 (10)	0.0043 (10)	0.0017 (9)
C3	0.0233 (13)	0.0145 (13)	0.0133 (12)	0.0033 (10)	0.0035 (10)	0.0007 (9)
C4	0.0209 (13)	0.0102 (11)	0.0156 (12)	0.0022 (10)	−0.0020 (10)	0.0014 (9)
C5	0.0162 (12)	0.0156 (12)	0.0191 (12)	0.0007 (10)	0.0026 (10)	0.0043 (10)
C6	0.0173 (12)	0.0155 (12)	0.0128 (11)	0.0039 (10)	0.0049 (9)	0.0008 (9)
C7	0.0283 (14)	0.0224 (14)	0.0186 (13)	−0.0019 (11)	0.0077 (11)	0.0042 (10)
C8	0.0233 (14)	0.0197 (13)	0.0199 (13)	0.0077 (11)	0.0021 (11)	0.0007 (10)

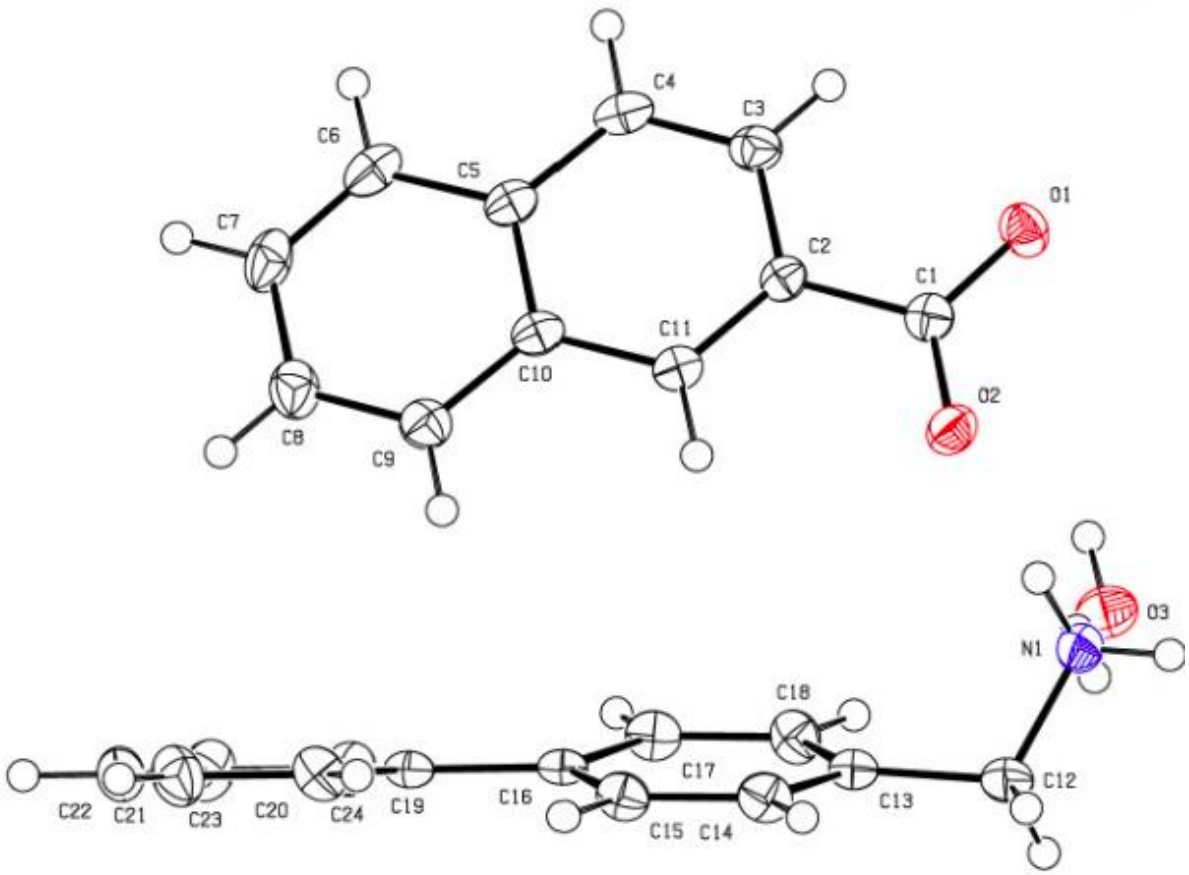

Figure 183: X-Ray Crystal Structure of [1,1'-biphenyl]-4-ylmethanaminium 2-naphthoate hydrate **53**

Table 1. Experimental details.**53**

	Prosp14mz160_0m
Crystal data	
Chemical formula	$C_{13}H_{14}N \cdot C_{11}H_7O_2 \cdot H_2O$
M_r	373.43
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	Cell setting: monoclinic 22.6186 (14), 11.3771 (7), 7.6355 (5)
β (°)	Cell setting: monoclinic 98.890 (3)
V (Å^3)	1941.3 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.67
Crystal size (mm)	0.21 × 0.09 × 0.03
Data collection	
Diffractometer	Bruker AXS X8 Prospector CCD diffractometer
Absorption correction	Multi-scan Apex2 v2014.1-0 (Bruker, 2014)
T_{min}, T_{max}	0.629, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29767, 3351, 2962
R_{int}	0.042
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.083, 1.09
No. of reflections	3351
No. of parameters	261
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.16

Table 2. Bond Lengths (Å).**53**

C1—O2	1.2459(16)
C1—O1	1.2831(16)
C1—C2	1.5058(18)
C2—C11	1.3720(19)
C2—C3	1.4221(19)
C3—C4	1.362(2)
C3—H3	0.9500
C4—C5	1.414(2)
C4—H4	0.9500
C5—C6	1.4209(19)
C5—C10	1.4243(19)
C6—C7	1.365(2)
C6—H6	0.9500
C7—C8	1.413(2)
C7—H7	0.9500
C8—C9	1.366(2)
C8—H8	0.9500
C9—C10	1.4159(19)
C9—H9	0.9500
C10—C11	1.4207(19)
C11—H11	0.9500
C12—N1	1.4944(17)
C12—C13	1.5054(19)
C12—H12A	0.9900
C12—H12B	0.9900
C13—C14	1.3886(19)
C13—C18	1.3938(19)
C14—C15	1.3843(19)
C14—H14	0.9500
C15—C16	1.3980(18)
C15—H15	0.9500
C16—C17	1.4020(19)
C16—C19	1.4871(18)
C17—C18	1.382(2)
C17—H17	0.9500
C18—H18	0.9500
C19—C24	1.3976(19)
C19—C20	1.4006(19)
C20—C21	1.385(2)
C20—H20	0.9500
C21—C22	1.382(2)
C21—H21	0.9500
C22—C23	1.382(2)
C22—H22	0.9500
C23—C24	1.387(2)
C23—H23	0.9500
C24—H24	0.9500
N1—H1A	0.9100
N1—H1B	0.9100
N1—H1C	0.9100
O3—H3A	0.892(14)
O3—H3B	0.857(15)

Table 3. Bond Angles (deg).**53**

O2—C1—O1	123.78 (12)	C18—C13—C12	121.91 (12)
O2—C1—C2	119.48 (12)	C15—C14—C13	121.03 (12)
O1—C1—C2	116.73 (12)	C15—C14—H14	119.5
C11—C2—C3	119.42 (12)	C13—C14—H14	119.5
C11—C2—C1	120.23 (12)	C14—C15—C16	121.72 (12)
C3—C2—C1	120.35 (12)	C14—C15—H15	119.1
C4—C3—C2	120.71 (13)	C16—C15—H15	119.1
C4—C3—H3	119.6	C15—C16—C17	116.72 (12)
C2—C3—H3	119.6	C15—C16—C19	121.34 (12)
C3—C4—C5	121.05 (13)	C17—C16—C19	121.94 (12)
C3—C4—H4	119.5	C18—C17—C16	121.53 (12)
C5—C4—H4	119.5	C18—C17—H17	119.2
C4—C5—C6	122.47 (13)	C16—C17—H17	119.2
C4—C5—C10	118.87 (12)	C17—C18—C13	121.11 (12)
C6—C5—C10	118.67 (13)	C17—C18—H18	119.4
C7—C6—C5	120.81 (13)	C13—C18—H18	119.4
C7—C6—H6	119.6	C24—C19—C20	117.03 (13)
C5—C6—H6	119.6	C24—C19—C16	121.90 (12)
C6—C7—C8	120.39 (13)	C20—C19—C16	121.07 (12)
C6—C7—H7	119.8	C21—C20—C19	121.44 (13)
C8—C7—H7	119.8	C21—C20—H20	119.3
C9—C8—C7	120.28 (14)	C19—C20—H20	119.3
C9—C8—H8	119.9	C22—C21—C20	120.60 (14)

C7—C8—H8	119.9	C22—C21—H21	119.7
C8—C9—C10	120.83 (13)	C20—C21—H21	119.7
C8—C9—H9	119.6	C21—C22—C23	118.95 (13)
C10—C9—H9	119.6	C21—C22—H22	120.5
C9—C10—C11	122.18 (12)	C23—C22—H22	120.5
C9—C10—C5	119.01 (12)	C22—C23—C24	120.68 (14)
C11—C10—C5	118.80 (12)	C22—C23—H23	119.7
C2—C11—C10	121.14 (12)	C24—C23—H23	119.7
C2—C11—H11	119.4	C23—C24—C19	121.30 (14)
C10—C11—H11	119.4	C23—C24—H24	119.3
N1—C12—C13	112.72 (11)	C19—C24—H24	119.3
N1—C12—H12A	109.0	C12—N1—H1A	109.5
C13—C12—H12A	109.0	C12—N1—H1B	109.5
N1—C12—H12B	109.0	H1A—N1—H1B	109.5
C13—C12—H12B	109.0	C12—N1—H1C	109.5
H12A—C12—H12B	107.8	H1A—N1—H1C	109.5
C14—C13—C18	117.86 (12)	H1B—N1—H1C	109.5
C14—C13—C12	120.18 (12)	H3A—O3—H3B	109.3 (17)
O2—C1—C2—C11	−2.70 (18)	N1—C12—C13—C14	−121.26 (13)
O1—C1—C2—C11	177.54 (12)	N1—C12—C13—C18	61.27 (17)
O2—C1—C2—C3	177.38 (12)	C18—C13—C14—C15	1.6 (2)
O1—C1—C2—C3	−2.39 (18)	C12—C13—C14—C15	−175.98 (12)
C11—C2—C3—C4	−0.31 (19)	C13—C14—C15—C16	−0.3 (2)
C1—C2—C3—C4	179.62 (12)	C14—C15—C16—C17	−1.24 (19)
C2—C3—C4—C5	0.1 (2)	C14—C15—C16—C19	178.47 (12)

C3—C4—C5—C6	−178.83 (13)	C15—C16—C17—C18	1.46 (19)
C3—C4—C5—C10	0.64 (19)	C19—C16—C17—C18	−178.25 (12)
C4—C5—C6—C7	179.44 (13)	C16—C17—C18—C13	−0.2 (2)
C10—C5—C6—C7	−0.03 (19)	C14—C13—C18—C17	−1.4 (2)
C5—C6—C7—C8	0.7 (2)	C12—C13—C18—C17	176.15 (12)
C6—C7—C8—C9	−0.7 (2)	C15—C16—C19—C24	−166.36 (13)
C7—C8—C9—C10	−0.2 (2)	C17—C16—C19—C24	13.34 (19)
C8—C9—C10—C11	−178.24 (12)	C15—C16—C19—C20	13.24 (19)
C8—C9—C10—C5	0.87 (19)	C17—C16—C19—C20	−167.06 (13)
C4—C5—C10—C9	179.74 (12)	C24—C19—C20—C21	−0.4 (2)
C6—C5—C10—C9	−0.77 (18)	C16—C19—C20—C21	−179.98 (13)
C4—C5—C10—C11	−1.12 (18)	C19—C20—C21—C22	−0.1 (2)
C6—C5—C10—C11	178.37 (12)	C20—C21—C22—C23	0.4 (2)
C3—C2—C11—C10	−0.21 (19)	C21—C22—C23—C24	−0.2 (2)
C1—C2—C11—C10	179.87 (11)	C22—C23—C24—C19	−0.3 (2)
C9—C10—C11—C2	−179.97 (12)	C20—C19—C24—C23	0.6 (2)
C5—C10—C11—C2	0.92 (18)	C16—C19—C24—C23	−179.80 (13)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2). **53**

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.10870 (6)	0.43938 (12)	0.72956 (17)	0.0212 (3)
C2	0.17272 (6)	0.40034 (11)	0.77898 (16)	0.0204 (3)
C3	0.18634 (6)	0.29153 (12)	0.86775 (17)	0.0234 (3)
H3	0.1548	0.2436	0.8965	0.028*
C4	0.24410 (6)	0.25528 (12)	0.91198 (17)	0.0247 (3)
H4	0.2523	0.1823	0.9711	0.030*
C5	0.29207 (6)	0.32460 (12)	0.87128 (17)	0.0222 (3)
C6	0.35274 (6)	0.28839 (13)	0.91268 (18)	0.0276 (3)
H6	0.3620	0.2154	0.9708	0.033*
C7	0.39783 (6)	0.35722 (14)	0.86999 (19)	0.0307 (3)
H7	0.4381	0.3313	0.8973	0.037*
C8	0.38491 (6)	0.46677 (14)	0.78540 (19)	0.0294 (3)
H8	0.4166	0.5145	0.7573	0.035*
C9	0.32709 (6)	0.50427 (12)	0.74396 (17)	0.0238 (3)
H9	0.3189	0.5782	0.6874	0.029*
C10	0.27915 (6)	0.43443 (11)	0.78423 (16)	0.0201 (3)
C11	0.21844 (6)	0.46948 (11)	0.73849 (16)	0.0196 (3)
H11	0.2093	0.5421	0.6788	0.024*
C12	0.08001 (6)	0.65082 (12)	0.21530 (19)	0.0264 (3)
H12A	0.0684	0.7288	0.2564	0.032*

H12B	0.0668	0.6467	0.0856	0.032*
C13	0.14708 (6)	0.63927 (12)	0.25297 (17)	0.0221 (3)
C14	0.18176 (6)	0.73077 (12)	0.33353 (18)	0.0244 (3)
H14	0.1627	0.7985	0.3715	0.029*
C15	0.24363 (6)	0.72477 (12)	0.35935 (17)	0.0230 (3)
H15	0.2662	0.7889	0.4144	0.028*
C16	0.27379 (6)	0.62682 (11)	0.30658 (16)	0.0194 (3)
C17	0.23829 (6)	0.53381 (12)	0.22936 (18)	0.0235 (3)
H17	0.2571	0.4649	0.1943	0.028*
C18	0.17653 (6)	0.54001 (12)	0.20307 (18)	0.0250 (3)
H18	0.1538	0.4755	0.1501	0.030*
C19	0.34026 (6)	0.62240 (11)	0.33082 (16)	0.0202 (3)
C20	0.37505 (6)	0.70409 (13)	0.43876 (19)	0.0271 (3)
H20	0.3558	0.7632	0.4973	0.032*
C21	0.43698 (7)	0.70062 (14)	0.4620 (2)	0.0322 (3)
H21	0.4595	0.7572	0.5358	0.039*
C22	0.46631 (7)	0.61558 (14)	0.3789 (2)	0.0317 (3)
H22	0.5088	0.6130	0.3956	0.038*
C23	0.43287 (7)	0.53438 (14)	0.2711 (2)	0.0326 (3)
H23	0.4526	0.4757	0.2130	0.039*
C24	0.37083 (6)	0.53772 (13)	0.24692 (18)	0.0266 (3)
H24	0.3487	0.4813	0.1718	0.032*
N1	0.04866 (5)	0.55748 (10)	0.30360 (15)	0.0238 (3)

H1A	0.0535	0.4869	0.2518	0.036*
H1B	0.0090	0.5748	0.2922	0.036*
H1C	0.0644	0.5540	0.4206	0.036*
O1	0.06827 (4)	0.36827 (8)	0.76586 (13)	0.0269 (2)
O2	0.09769 (4)	0.53692 (8)	0.65768 (13)	0.0266 (2)
O3	0.04705 (5)	0.34857 (9)	0.11305 (14)	0.0304 (2)
H3A	0.0569 (8)	0.3478 (16)	0.004 (2)	0.046*
H3B	0.0555 (8)	0.2818 (14)	0.162 (2)	0.046*

Table 5: Atomic displacement parameters (Å2).**53**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C1	0.0234 (7)	0.0201 (7)	0.0194 (6)	−0.0020 (5)	0.0017 (5)	−0.0039 (5)
C2	0.0231 (7)	0.0197 (7)	0.0181 (6)	−0.0002 (5)	0.0019 (5)	−0.0029 (5)
C3	0.0286 (7)	0.0194 (7)	0.0216 (7)	−0.0027 (5)	0.0025 (5)	0.0008 (5)
C4	0.0338 (8)	0.0184 (7)	0.0207 (6)	0.0023 (6)	0.0006 (6)	0.0021 (5)
C5	0.0285 (7)	0.0214 (7)	0.0161 (6)	0.0035 (5)	0.0015 (5)	−0.0030 (5)
C6	0.0316 (8)	0.0272 (8)	0.0229 (7)	0.0096 (6)	0.0008 (6)	−0.0017 (6)
C7	0.0226 (7)	0.0399 (9)	0.0291 (8)	0.0105 (6)	0.0022 (6)	−0.0039 (7)
C8	0.0241 (7)	0.0369 (8)	0.0283 (7)	0.0008 (6)	0.0079 (6)	−0.0030 (6)
C9	0.0267 (7)	0.0242 (7)	0.0210 (7)	0.0019 (6)	0.0056 (5)	−0.0011 (5)
C10	0.0256 (7)	0.0189 (7)	0.0159 (6)	0.0022 (5)	0.0035 (5)	−0.0036 (5)
C11	0.0259 (7)	0.0170 (6)	0.0158 (6)	0.0023 (5)	0.0028 (5)	−0.0008 (5)
C12	0.0253 (7)	0.0230 (7)	0.0306 (8)	0.0005 (6)	0.0038 (6)	0.0067 (6)
C13	0.0247 (7)	0.0205 (7)	0.0213 (7)	−0.0001 (5)	0.0043 (5)	0.0047 (5)
C14	0.0290 (7)	0.0173 (7)	0.0280 (7)	0.0026 (5)	0.0081 (6)	0.0003 (6)
C15	0.0271 (7)	0.0174 (7)	0.0248 (7)	−0.0025 (5)	0.0048 (6)	−0.0020 (5)
C16	0.0251 (7)	0.0177 (6)	0.0156 (6)	−0.0009 (5)	0.0044 (5)	0.0026 (5)
C17	0.0279 (7)	0.0177 (7)	0.0258 (7)	0.0009 (5)	0.0069 (6)	−0.0025 (5)
C18	0.0274 (7)	0.0214 (7)	0.0261 (7)	−0.0050 (6)	0.0038 (6)	−0.0040 (6)
C19	0.0256 (7)	0.0190 (7)	0.0165 (6)	−0.0005 (5)	0.0047 (5)	0.0039 (5)
C20	0.0280 (7)	0.0262 (8)	0.0279 (7)	−0.0029 (6)	0.0072 (6)	−0.0041 (6)

C21	0.0275 (8)	0.0365 (9)	0.0323 (8)	−0.0086 (6)	0.0039 (6)	−0.0046 (7)
C22	0.0226 (7)	0.0412 (9)	0.0315 (8)	−0.0001 (6)	0.0050 (6)	0.0042 (7)
C23	0.0286 (8)	0.0373 (9)	0.0326 (8)	0.0077 (6)	0.0067 (6)	−0.0030 (7)
C24	0.0271 (7)	0.0266 (8)	0.0256 (7)	0.0016 (6)	0.0025 (6)	−0.0030 (6)
N1	0.0216 (6)	0.0220 (6)	0.0276 (6)	0.0007 (5)	0.0035 (5)	0.0007 (5)
O1	0.0227 (5)	0.0232 (5)	0.0351 (6)	−0.0034 (4)	0.0049 (4)	−0.0009 (4)
O2	0.0258 (5)	0.0220 (5)	0.0304 (5)	0.0020 (4)	−0.0007 (4)	0.0033 (4)
O3	0.0366 (6)	0.0222 (5)	0.0320 (6)	0.0035 (4)	0.0044 (5)	0.0004 (4)

Table 6: Hydrogen-bond geometry (Å, º). **53**

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3	0.91	1.89	2.7841 (15)	167
N1—H1B···O1i	0.91	1.85	2.7488 (15)	168
N1—H1C···O2	0.91	1.86	2.7690 (15)	176
O3—H3A···O1ii	0.89 (1)	1.89 (2)	2.7751 (15)	170 (2)
O3—H3B···O1iii	0.86 (2)	1.88 (2)	2.7395 (14)	176 (2)

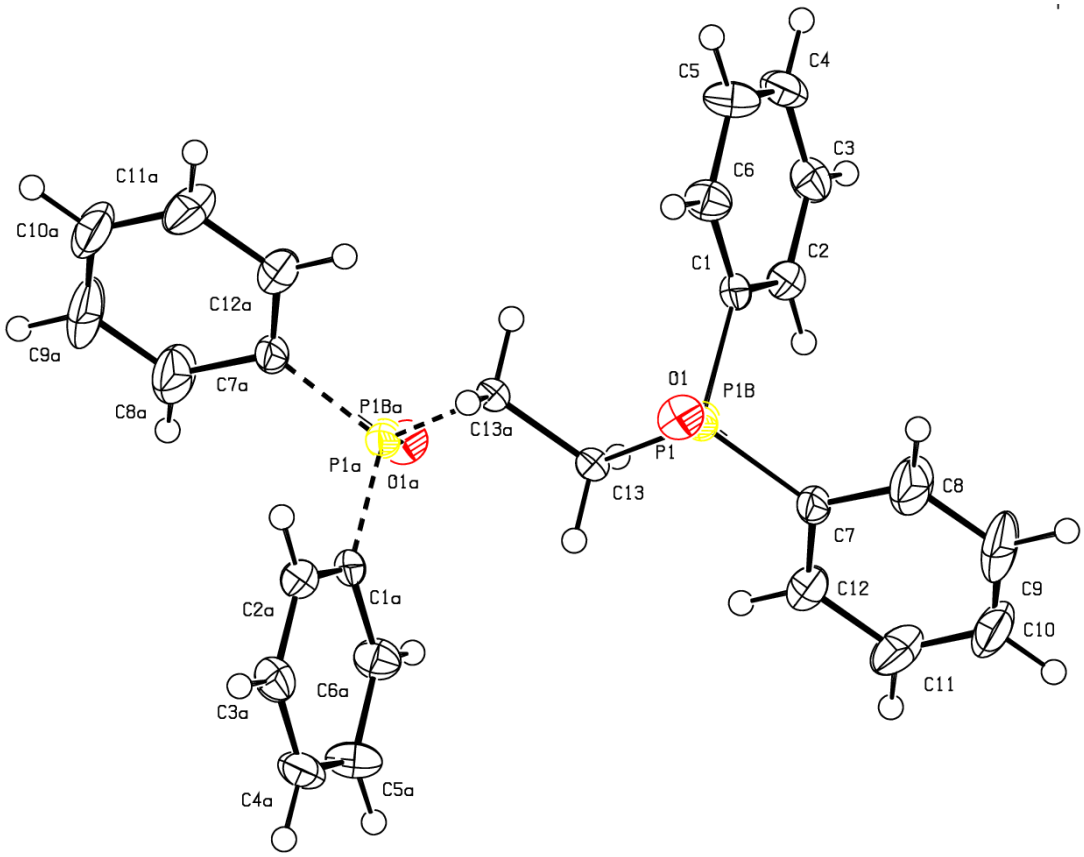

Figure 184: X-Ray Crystal Structure of ethane-1,2-diylbis(diphenylphosphine oxide) **5**

Table 1: Experimental details. **5**

Bond precision: C-C = 0.0033 A Wavelength=1.54178

Cell: a=12.9238(13) b=5.5132(6) c=16.1269(16)

alpha=90 beta=110.869(5) gamma=90 Temperature:100 K

	Calculated	Reported
Volume	1073.68 (19)	1073.68 (19)
Space group	P 21/n	P 21/n
Hall group	-p 2yn	-p 2yn
Moiety formula	C26 H24 O0.41 P2	C26 H24 O0.41 P2
Sum formula	C26 H24 O0.41 P2	C26 H24 O0.41 P2
Mr	404.95	404.74
Dx,g cm-3	1.253	1.252
Z	2	2
Mu (mm-1)	1.907	1.906
F000	426.6	426.0
F000'	428.68	428.68
h,k,lmax	15,6,19	15,6,19
Nref	1893	1879
Tmin	0.852	0.586
Tmax	0.963	0.753
Tmin'	0.683	-

Correction method= MULTI-SCAN

Data completeness= 0.993 Theta(max)= 66.371

R(reflections)= 0.0363(1649) wR2(reflections)= 0.0886(1879)

S = 1.034 Npar= 142

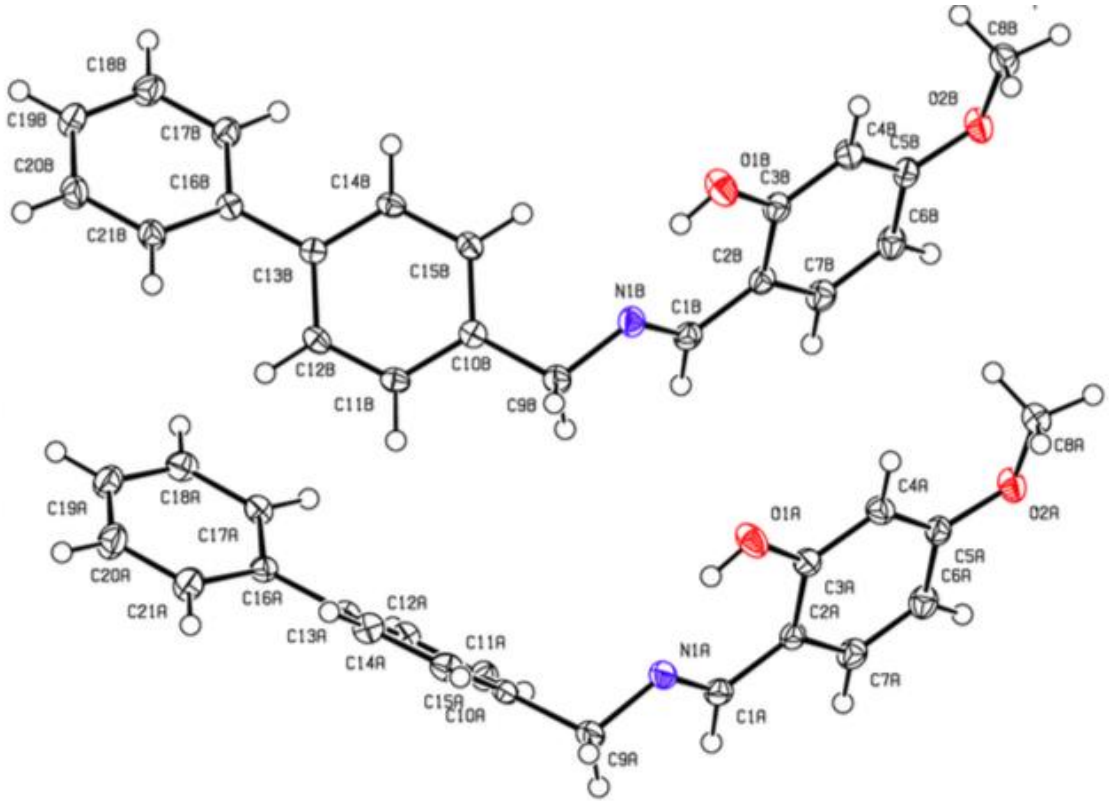

Figure 185: X-Ray Crystal Structure of **14**

Table 1: Experimental details.**14**

Crystal data	
Chemical formula	C21H19NO2
*M*r	317.37
Crystal system, space group	Triclinic, *P*1
Temperature (K)	100
a, *b*, *c* (Å)	Cell setting: triclinic 9.451 (2), 11.101 (3), 16.979 (4)
α, β, γ (°)	Cell setting: triclinic 84.470 (4), 77.258 (3), 69.773 (3)
V (Å3)	1630.1 (6)
Z	4
Radiation type	Mo *K*α
μ (mm−1)	0.08
Crystal size (mm)	0.64 × 0.32 × 0.10
Data collection	
Diffractometer	Bruker AXS APEXII CCD diffractometer
Absorption correction	Multi-scan Apex2 v2014.1 (Bruker, 2014)
*T*min, *T*max	0.649, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19761, 9810, 6736
*R*int	0.023
(sin θ/λ)max (Å−1)	0.717
Refinement	
$R[F2 > 2\sigma(F2)]$, *wR*(*F*2), *S*	0.050, 0.152, 1.03
No. of reflections	9810
No. of parameters	437
H-atom treatment	H-atom parameters constrained
Δρmax, Δρmin (e Å−3)	0.50, −0.18

Computer programs: Apex2 v2014.11 (Bruker, 2014), *SAINT* V8.30C (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2014), SHELXLE Rev714 (Hübschle *et al.*, 2011).

Table 2: Bond Lengths (Å).**14**

O1A—C3A	1.3472(13)	O1B—C3B	1.3505(13)
O1A—H1C	0.8400	O1B—H1D	0.8400
O2A—C5A	1.3563(13)	O2B—C5B	1.3578(13)
O2A—C8A	1.4310(14)	O2B—C8B	1.4328(14)
N1A—C1A	1.2775(15)	N1B—C1B	1.2790(14)
N1A—C9A	1.4611(14)	N1B—C9B	1.4586(14)
C1A—C2A	1.4485(15)	C1B—C2B	1.4462(15)
C1A—H1A	0.9500	C1B—H1B	0.9500
C2A—C7A	1.4009(16)	C2B—C7B	1.4020(15)
C2A—C3A	1.4119(15)	C2B—C3B	1.4093(15)
C3A—C4A	1.3951(15)	C3B—C4B	1.3951(16)
C4A—C5A	1.3839(16)	C4B—C5B	1.3858(16)
C4A—H4A	0.9500	C4B—H4B	0.9500
C5A—C6A	1.4054(16)	C5B—C6B	1.4018(16)
C6A—C7A	1.3747(16)	C6B—C7B	1.3752(16)
C6A—H6A	0.9500	C6B—H6B	0.9500
C7A—H7A	0.9500	C7B—H7B	0.9500
C8A—H8AA	0.9800	C8B—H8BA	0.9800
C8A—H8AB	0.9800	C8B—H8BB	0.9800
C8A—H8AC	0.9800	C8B—H8BC	0.9800
C9A—C10A	1.5116(15)	C9B—C10B	1.5115(15)
C9A—H9AA	0.9900	C9B—H9BA	0.9900
C9A—H9AB	0.9900	C9B—H9BB	0.9900

C10A—C11A	1.3876(16)	C10B—C15B	1.3920(15)
C10A—C15A	1.3932(15)	C10B—C11B	1.3943(15)
C11A—C12A	1.3859(16)	C11B—C12B	1.3843(15)
C11A—H11A	0.9500	C11B—H11B	0.9500
C12A—C13A	1.3973(15)	C12B—C13B	1.4043(15)
C12A—H12A	0.9500	C12B—H12B	0.9500
C13A—C14A	1.3971(15)	C13B—C14B	1.3989(15)
C13A—C16A	1.4820(15)	C13B—C16B	1.4854(15)
C14A—C15A	1.3864(15)	C14B—C15B	1.3901(15)
C14A—H14A	0.9500	C14B—H14B	0.9500
C15A—H15A	0.9500	C15B—H15B	0.9500
C16A—C17A	1.4002(15)	C16B—C21B	1.3988(15)
C16A—C21A	1.4004(15)	C16B—C17B	1.4026(15)
C17A—C18A	1.3876(16)	C17B—C18B	1.3860(16)
C17A—H17A	0.9500	C17B—H17B	0.9500
C18A—C19A	1.3866(17)	C18B—C19B	1.3818(16)
C18A—H18A	0.9500	C18B—H18B	0.9500
C19A—C20A	1.3915(17)	C19B—C20B	1.3891(17)
C19A—H19A	0.9500	C19B—H19B	0.9500
C20A—C21A	1.3846(16)	C20B—C21B	1.3864(16)
C20A—H20A	0.9500	C20B—H20B	0.9500
C21A—H21A	0.9500	C21B—H21B	0.9500

Table 3: Bond Angles (deg).**14**

C3A—O1A—H1C	109.5	C3B—O1B—H1D	109.5
C5A—O2A—C8A	117.70 (9)	C5B—O2B—C8B	118.07 (9)
C1A—N1A—C9A	118.90 (10)	C1B—N1B—C9B	118.61 (10)
N1A—C1A—C2A	121.36 (10)	N1B—C1B—C2B	121.93 (10)
N1A—C1A—H1A	119.3	N1B—C1B—H1B	119.0
C2A—C1A—H1A	119.3	C2B—C1B—H1B	119.0
C7A—C2A—C3A	118.25 (10)	C7B—C2B—C3B	118.11 (10)
C7A—C2A—C1A	120.95 (10)	C7B—C2B—C1B	120.59 (10)
C3A—C2A—C1A	120.80 (10)	C3B—C2B—C1B	121.28 (10)
O1A—C3A—C4A	118.09 (10)	O1B—C3B—C4B	118.20 (10)
O1A—C3A—C2A	121.32 (10)	O1B—C3B—C2B	121.00 (10)
C4A—C3A—C2A	120.58 (10)	C4B—C3B—C2B	120.80 (10)
C5A—C4A—C3A	119.54 (10)	C5B—C4B—C3B	119.38 (10)
C5A—C4A—H4A	120.2	C5B—C4B—H4B	120.3
C3A—C4A—H4A	120.2	C3B—C4B—H4B	120.3
O2A—C5A—C4A	124.33 (10)	O2B—C5B—C4B	124.20 (10)
O2A—C5A—C6A	114.97 (10)	O2B—C5B—C6B	115.06 (10)
C4A—C5A—C6A	120.71 (10)	C4B—C5B—C6B	120.74 (10)
C7A—C6A—C5A	119.31 (11)	C7B—C6B—C5B	119.36 (10)
C7A—C6A—H6A	120.3	C7B—C6B—H6B	120.3
C5A—C6A—H6A	120.3	C5B—C6B—H6B	120.3
C6A—C7A—C2A	121.55 (10)	C6B—C7B—C2B	121.57 (10)
C6A—C7A—H7A	119.2	C6B—C7B—H7B	119.2
C2A—C7A—H7A	119.2	C2B—C7B—H7B	119.2

O2A—C8A—H8AA	109.5	O2B—C8B—H8BA	109.5
O2A—C8A—H8AB	109.5	O2B—C8B—H8BB	109.5
H8AA—C8A—H8AB	109.5	H8BA—C8B—H8BB	109.5
O2A—C8A—H8AC 1	09.5	O2B—C8B—H8BC	109.5
H8AA—C8A—H8AC	109.5	H8BA—C8B—H8BC	109.5
H8AB—C8A—H8AC	109.5	H8BB—C8B—H8BC	109.5
N1A—C9A—C10A	111.08 (9)	N1B—C9B—C10B	112.17 (9)
N1A—C9A—H9AA	109.4	N1B—C9B—H9BA	109.2
C10A—C9A—H9AA	109.4	C10B—C9B—H9BA	109.2
N1A—C9A—H9AB	109.4	N1B—C9B—H9BB	109.2
C10A—C9A—H9AB	109.4	C10B—C9B—H9BB	109.2
H9AA—C9A—H9AB	108.0	H9BA—C9B—H9BB	107.9
C11A—C10A—C15A	118.52 (10)	C15B—C10B—C11B	117.97 (10)
C11A—C10A—C9A	121.06 (10)	C15B—C10B—C9B	122.14 (9)
C15A—C10A—C9A 1	20.42 (10)	C11B—C10B—C9B	119.89 (10)
C12A—C11A—C10A	120.73 (10)	C12B—C11B—C10B	121.09 (10)
C12A—C11A—H11A	119.6	C12B—C11B—H11B	119.5
C10A—C11A—H11A	119.6	C10B—C11B—H11B	119.5
C11A—C12A—C13A	121.08 (10)	C11B—C12B—C13B	121.27 (10)
C11A—C12A—H12A	119.5	C11B—C12B—H12B	119.4
C13A—C12A—H12A	119.5	C13B—C12B—H12B	119.4
C14A—C13A—C12A	118.01 (10)	C14B—C13B—C12B	117.39 (10)
C14A—C13A—C16A	121.68 (10)	C14B—C13B—C16B	120.65 (10)
C12A—C13A—C16A	120.30 (9)	C12B—C13B—C16B	121.96 (9)
C15A—C14A—C13A	120.69 (10)	C15B—C14B—C13B	121.03 (10)

C15A—C14A—H14A	119.7	C15B—C14B—H14B	119.5
C13A—C14A—H14A	119.7	C13B—C14B—H14B	119.5
C14A—C15A—C10A	120.96 (10)	C14B—C15B—C10B	121.24 (10)
C14A—C15A—H15A	119.5	C14B—C15B—H15B	119.4
C10A—C15A—H15A	119.5	C10B—C15B—H15B	119.4
C17A—C16A—C21A	117.93 (10)	C21B—C16B—C17B	117.58 (10)
C17A—C16A—C13A	121.05 (9)	C21B—C16B—C13B	121.37 (10)
C21A—C16A—C13A	120.99 (10)	C17B—C16B—C13B	121.04 (9)
C18A—C17A—C16A	120.90 (10)	C18B—C17B—C16B	121.05 (10)
C18A—C17A—H17A	119.5	C18B—C17B—H17B	119.5
C16A—C17A—H17A	119.5	C16B—C17B—H17B	119.5
C19A—C18A—C17A	120.49 (11)	C19B—C18B—C17B	120.61 (11)
C19A—C18A—H18A	119.8	C19B—C18B—H18B	119.7
C17A—C18A—H18A	119.8	C17B—C18B—H18B	119.7
C18A—C19A—C20A	119.25 (11)	C18B—C19B—C20B	119.21 (11)
C18A—C19A—H19A	120.4	C18B—C19B—H19B	120.4
C20A—C19A—H19A	120.4	C20B—C19B—H19B	120.4
C21A—C20A—C19A	120.38 (11)	C21B—C20B—C19B	120.42 (10)
C21A—C20A—H20A	119.8	C21B—C20B—H20B	119.8
C19A—C20A—H20A	119.8	C19B—C20B—H20B	119.8
C20A—C21A—C16A	121.04 (11)	C20B—C21B—C16B	121.13 (11)
C20A—C21A—H21A	119.5	C20B—C21B—H21B	119.4
C16A—C21A—H21A	119.5	C16B—C21B—H21B	119.4
C9A—N1A—C1A—C2A	−178.98 (9)	C9B—N1B—C1B—C2B	178.25 (10)
N1A—C1A—C2A—C7A	−178.04 (10)	N1B—C1B—C2B—C7B	−177.28 (10)

N1A—C1A—C2A—C3A	2.15 (17)	N1B—C1B—C2B—C3B	1.07 (17)
C7A—C2A—C3A—O1A	178.60 (10)	C7B—C2B—C3B—O1B	178.06 (10)
C1A—C2A—C3A—O1A	−1.57 (17)	C1B—C2B—C3B—O1B	−0.33 (17)
C7A—C2A—C3A—C4A	−2.09 (16)	C7B—C2B—C3B—C4B	−1.97 (17)
C1A—C2A—C3A—C4A	177.74 (10)	C1B—C2B—C3B—C4B	179.64 (10)
O1A—C3A—C4A—C5A	−179.51 (10)	O1B—C3B—C4B—C5B	−178.95 (11)
C2A—C3A—C4A—C5A	1.16 (17)	C2B—C3B—C4B—C5B	1.08 (17)
C8A—O2A—C5A—C4A	7.92 (16)	C8B—O2B—C5B—C4B	8.67 (16)
C8A—O2A—C5A—C6A	−172.18 (10)	C8B—O2B—C5B—C6B	−171.74 (10)
C3A—C4A—C5A—O2A	−179.03 (10)	C3B—C4B—C5B—O2B	−179.51 (10)
C3A—C4A—C5A—C6A	1.07 (17)	C3B—C4B—C5B—C6B	0.93 (17)
O2A—C5A—C6A—C7A	177.76 (10)	O2B—C5B—C6B—C7B	178.40 (10)
C4A—C5A—C6A—C7A	−2.34 (18)	C4B—C5B—C6B—C7B	−1.99 (18)
C5A—C6A—C7A—C2A	1.37 (18)	C5B—C6B—C7B—C2B	1.06 (18)
C3A—C2A—C7A—C6A	0.80 (17)	C3B—C2B—C7B—C6B	0.89 (17)
C1A—C2A—C7A—C6A	−179.02 (11)	C1B—C2B—C7B—C6B	179.29 (11)
C1A—N1A—C9A—C10A	−113.87 (12)	C1B—N1B—C9B—C10B	−113.91 (11)
N1A—C9A—C10A—C11A	−107.38 (12)	N1B—C9B—C10B—C15B	−1.62 (15)
N1A—C9A—C10A—C15A	72.44 (13)	N1B—C9B—C10B—C11B	178.57 (9)
C15A—C10A—C11A—C12A	0.44 (16)	C15B—C10B—C11B—C12B	−0.61 (16)
C9A—C10A—C11A—C12A	−179.73 (10)	C9B—C10B—C11B—C12B	179.21 (10)
C10A—C11A—C12A—C13A	−0.08 (17)	C10B—C11B—C12B—C13B	−0.30 (16)
C11A—C12A—C13A—C14A	−0.17 (16)	C11B—C12B—C13B—C14B	0.78 (15)
C11A—C12A—C13A—C16A	−178.67 (10)	C11B—C12B—C13B—C16B	−178.98 (10)
C12A—C13A—C14A—C15A	0.06 (16)	C12B—C13B—C14B—C15B	−0.35 (15)

C16A—C13A—C14A—C15A178.53 (10)	C16B—C13B—C14B—C15B 179.42 (10)
C13A—C14A—C15A—C10A0.31 (16)	C13B—C14B—C15B—C10B−0.57 (16)
C11A—C10A—C15A—C14A−0.56 (16)	C11B—C10B—C15B—C14B1.04 (16)
C9A—C10A—C15A—C14A179.61 (10)	C9B—C10B—C15B—C14B −178.77 (10)
C14A—C13A—C16A—C17A−35.99 (15)	C14B—C13B—C16B—C21B−150.71 (11)
C12A—C13A—C16A—C17A142.45 (11)	C12B—C13B—C16B—C21B 29.05 (15)
C14A—C13A—C16A—C21A146.03 (11)	C14B—C13B—C16B—C17B27.84 (15)
C12A—C13A—C16A—C21A−35.53 (15)	C12B—C13B—C16B—C17B−152.41 (11)
C21A—C16A—C17A—C18A 0.88 (16)	C21B—C16B—C17B—C18B−0.36 (17)
C13A—C16A—C17A—C18A−177.17 (10)	C13B—C16B—C17B—C18B−178.95 (11)
C16A—C17A—C18A—C19A−0.49 (17)	C16B—C17B—C18B—C19B−0.18 (18)
C17A—C18A—C19A—C20A−0.13 (18)	C17B—C18B—C19B—C20B0.75 (19)
C18A—C19A—C20A—C21A0.34 (19)	C18B—C19B—C20B—C21B−0.80 (18)
C19A—C20A—C21A—C16A0.06 (19)	C19B—C20B—C21B—C16B0.26 (18)
C17A—C16A—C21A—C20A−0.67 (17)	C17B—C16B—C21B—C20B0.31 (17)
C13A—C16A—C21A—C20A177.38 (11)	C13B—C16B—C21B—C20B 178.90 (10)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2)**.14**

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	0.95301 (10)	0.32316 (9)	0.16154 (5)	0.0285 (2)
H1C	0.9517	0.2724	0.2015	0.043*
O2A	0.71132 (10)	0.46517 (8)	−0.06551 (5)	0.02450 (19)
N1A	0.87391 (11)	0.15279 (9)	0.25651 (6)	0.0203 (2)
C1A	0.78057 (13)	0.15315 (11)	0.21226 (6)	0.0202 (2)
H1A	0.7220	0.0970	0.2258	0.024*
C2A	0.76224 (12)	0.23735 (11)	0.14182 (6)	0.0177 (2)
C3A	0.85124 (13)	0.31884 (11)	0.11835 (7)	0.0188 (2)
C4A	0.83705 (13)	0.39646 (11)	0.04887 (7)	0.0204 (2)
H4A	0.8986	0.4499	0.0328	0.024*
C5A	0.73270 (13)	0.39502 (11)	0.00349 (6)	0.0189 (2)
C6A	0.63925 (14)	0.31792 (12)	0.02740 (7)	0.0231 (2)
H6A	0.5651	0.3197	−0.0029	0.028*
C7A	0.65619 (13)	0.23988 (12)	0.09508 (7)	0.0225 (2)
H7A	0.5945	0.1864	0.1105	0.027*
C8A	0.81599 (14)	0.53285 (12)	−0.09873 (7)	0.0244 (3)
H8AA	0.9218	0.4726	−0.1069	0.037*
H8AB	0.8042	0.5993	−0.0614	0.037*
H8AC	0.7937	0.5730	−0.1506	0.037*
C9A	0.88942 (15)	0.06420 (11)	0.32602 (7)	0.0235 (2)
H9AA	0.9990	0.0112	0.3223	0.028*
H9AB	0.8307	0.0059	0.3254	0.028*
C10A	0.83068 (13)	0.13616 (10)	0.40429 (6)	0.0180 (2)
C11A	0.93143 (13)	0.14444 (11)	0.45023 (7)	0.0210 (2)
H11A	1.0391	0.1046	0.4322	0.025*
C12A	0.87668 (13)	0.21013 (11)	0.52210 (7)	0.0209 (2)
H12A	0.9475	0.2146	0.5527	0.025*
C13A	0.71901 (12)	0.26987 (10)	0.55026 (6)	0.0160 (2)
C14A	0.61800 (13)	0.26174 (11)	0.50367 (6)	0.0187 (2)
H14A	0.5103	0.3016	0.5215	0.022*
C15A	0.67335 (13)	0.19611 (11)	0.43175 (6)	0.0200 (2)
H15A	0.6030	0.1920	0.4007	0.024*
C16A	0.66284 (12)	0.34207 (10)	0.62636 (6)	0.0172 (2)
C17A	0.53623 (13)	0.45578 (11)	0.63437 (7)	0.0193 (2)
H17A	0.4826	0.4852	0.5913	0.023*
C18A	0.48806 (13)	0.52612 (11)	0.70440 (7)	0.0230 (2)
H18A	0.4024	0.6034	0.7086	0.028*
C19A	0.56399 (14)	0.48444 (12)	0.76828 (7)	0.0247 (3)
H19A	0.5310	0.5328	0.8161	0.030*
C20A	0.68910 (15)	0.37095 (13)	0.76150 (7)	0.0279 (3)
H20A	0.7414	0.3414	0.8051	0.033*

C21A	0.73773 (14)	0.30079 (12)	0.69158 (7)	0.0250 (3)
H21A	0.8232	0.2234	0.6878	0.030*
O1B	0.96224 (10)	0.81163 (9)	0.16471 (5)	0.0294 (2)
H1D	0.9594	0.7610	0.2047	0.044*
O2B	0.71533 (10)	0.96488 (8)	−0.06009 (5)	0.02442 (19)
N1B	0.87514 (11)	0.64412 (9)	0.26093 (6)	0.0198 (2)
C1B	0.78123 (13)	0.64818 (11)	0.21601 (6)	0.0192 (2)
H1B	0.7189	0.5950	0.2296	0.023*
C2B	0.76713 (12)	0.73134 (11)	0.14509 (6)	0.0177 (2)
C3B	0.85869 (13)	0.81049 (11)	0.12149 (7)	0.0193 (2)
C4B	0.84473 (13)	0.88982 (11)	0.05271 (7)	0.0209 (2)
H4B	0.9081	0.9418	0.0367	0.025*
C5B	0.73758 (13)	0.89208 (11)	0.00800 (6)	0.0192 (2)
C6B	0.64202 (13)	0.81712 (12)	0.03182 (7)	0.0226 (2)
H6B	0.5666	0.8212	0.0019	0.027*
C7B	0.65853 (13)	0.73770 (12)	0.09896 (7)	0.0219 (2)
H7B	0.5948	0.6859	0.1145	0.026*
C8B	0.82222 (14)	1.03041 (12)	−0.09408 (7)	0.0244 (3)
H8BA	0.9269	0.9683	−0.1045	0.037*
H8BB	0.8154	1.0943	−0.0561	0.037*
H8BC	0.7974	1.0735	−0.1449	0.037*
C9B	0.87938 (14)	0.56045 (11)	0.33270 (7)	0.0210 (2)
H9BA	0.9861	0.5015	0.3309	0.025*
H9BB	0.8137	0.5077	0.3327	0.025*
C10B	0.82436 (12)	0.63538 (10)	0.40976 (6)	0.0164 (2)
C11B	0.81753 (12)	0.57067 (10)	0.48433 (6)	0.0180 (2)
H11B	0.8464	0.4797	0.4861	0.022*
C12B	0.76937 (12)	0.63680 (10)	0.55581 (6)	0.0177 (2)
H12B	0.7653	0.5904	0.6058	0.021*
C13B	0.72638 (11)	0.77120 (10)	0.55586 (6)	0.0148 (2)
C14B	0.73231 (12)	0.83570 (10)	0.48089 (6)	0.0176 (2)
H14B	0.7034	0.9267	0.4788	0.021*
C15B	0.77982 (12)	0.76875 (11)	0.40934 (6)	0.0183 (2)
H15B	0.7819	0.8149	0.3592	0.022*
C16B	0.67688 (12)	0.84346 (10)	0.63198 (6)	0.0163 (2)
C17B	0.57577 (13)	0.97060 (11)	0.63593 (7)	0.0217 (2)
H17B	0.5370	1.0107	0.5894	0.026*
C18B	0.53151 (14)	1.03873 (12)	0.70658 (7)	0.0257 (3)
H18B	0.4631	1.1248	0.7079	0.031*
C19B	0.58592 (14)	0.98256 (12)	0.77511 (7)	0.0236 (2)
H19B	0.5544	1.0292	0.8236	0.028*
C20B	0.68728 (14)	0.85711 (12)	0.77220 (7)	0.0246 (3)
H20B	0.7263	0.8181	0.8188	0.030*
C21B	0.73185 (13)	0.78848 (11)	0.70170 (7)	0.0215 (2)
H21B	0.8009	0.7026	0.7007	0.026*

Table 5: Atomic displacement parameters (Å2)**.14**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1A	0.0350 (5)	0.0354 (5)	0.0265 (5)	−0.0230 (4)	−0.0170 (4)	0.0126 (4)
O2A	0.0266 (4)	0.0293 (5)	0.0193 (4)	−0.0103 (4)	−0.0082 (3)	0.0037 (3)
N1A	0.0233 (5)	0.0176 (5)	0.0171 (4)	−0.0054 (4)	−0.0001 (4)	−0.0006 (4)
C1A	0.0225 (6)	0.0177 (5)	0.0186 (5)	−0.0079 (4)	0.0027 (4)	−0.0034 (4)
C2A	0.0191 (5)	0.0178 (5)	0.0155 (5)	−0.0067 (4)	0.0000 (4)	−0.0030 (4)
C3A	0.0195 (5)	0.0191 (5)	0.0189 (5)	−0.0075 (4)	−0.0048 (4)	0.0000 (4)
C4A	0.0224 (6)	0.0208 (6)	0.0206 (5)	−0.0104 (5)	−0.0053 (4)	0.0017 (4)
C5A	0.0204 (5)	0.0188 (5)	0.0160 (5)	−0.0046 (4)	−0.0030 (4)	−0.0020 (4)
C6A	0.0223 (6)	0.0283 (6)	0.0213 (6)	−0.0104 (5)	−0.0059 (4)	−0.0022 (5)
C7A	0.0233 (6)	0.0252 (6)	0.0220 (6)	−0.0125 (5)	−0.0023 (4)	−0.0035 (5)
C8A	0.0299 (6)	0.0231 (6)	0.0197 (6)	−0.0092 (5)	−0.0046 (5)	0.0028 (4)
C9A	0.0320 (6)	0.0157 (5)	0.0187 (5)	−0.0050 (5)	−0.0019 (5)	0.0008 (4)
C10A	0.0227 (5)	0.0133 (5)	0.0160 (5)	−0.0056 (4)	−0.0014 (4)	0.0022 (4)
C11A	0.0168 (5)	0.0180 (5)	0.0256 (6)	−0.0041 (4)	−0.0022 (4)	0.0005 (4)
C12A	0.0171 (5)	0.0213 (6)	0.0245 (6)	−0.0054 (4)	−0.0058 (4)	−0.0009 (4)
C13A	0.0175 (5)	0.0131 (5)	0.0173 (5)	−0.0052 (4)	−0.0042 (4)	0.0025 (4)
C14A	0.0152 (5)	0.0207 (5)	0.0192 (5)	−0.0054 (4)	−0.0027 (4)	0.0005 (4)
C15A	0.0203 (5)	0.0220 (6)	0.0190 (5)	−0.0082 (4)	−0.0056 (4)	0.0008 (4)
C16A	0.0172 (5)	0.0164 (5)	0.0186 (5)	−0.0065 (4)	−0.0030 (4)	−0.0004 (4)
C17A	0.0185 (5)	0.0190 (5)	0.0189 (5)	−0.0053 (4)	−0.0031 (4)	0.0019 (4)
C18A	0.0206 (6)	0.0196 (6)	0.0244 (6)	−0.0044 (4)	0.0011 (4)	−0.0017 (4)
C19A	0.0273 (6)	0.0254 (6)	0.0214 (6)	−0.0094 (5)	−0.0014 (5)	−0.0059 (5)
C20A	0.0317 (7)	0.0294 (7)	0.0232 (6)	−0.0069 (5)	−0.0106 (5)	−0.0041 (5)
C21A	0.0252 (6)	0.0225 (6)	0.0248 (6)	−0.0011 (5)	−0.0103 (5)	−0.0027 (5)
O1B	0.0331 (5)	0.0405 (6)	0.0267 (5)	−0.0241 (4)	−0.0164 (4)	0.0113 (4)
O2B	0.0258 (4)	0.0309 (5)	0.0189 (4)	−0.0119 (4)	−0.0075 (3)	0.0047 (3)
N1B	0.0203 (5)	0.0209 (5)	0.0167 (4)	−0.0056 (4)	−0.0020 (4)	−0.0012 (4)
C1B	0.0189 (5)	0.0183 (5)	0.0189 (5)	−0.0059 (4)	0.0003 (4)	−0.0039 (4)
C2B	0.0175 (5)	0.0190 (5)	0.0163 (5)	−0.0062 (4)	−0.0015 (4)	−0.0033 (4)
C3B	0.0189 (5)	0.0223 (6)	0.0187 (5)	−0.0082 (4)	−0.0048 (4)	−0.0020 (4)
C4B	0.0221 (6)	0.0235 (6)	0.0201 (5)	−0.0112 (5)	−0.0050 (4)	0.0006 (4)
C5B	0.0200 (5)	0.0208 (6)	0.0157 (5)	−0.0051 (4)	−0.0032 (4)	−0.0026 (4)
C6B	0.0213 (6)	0.0299 (6)	0.0200 (5)	−0.0113 (5)	−0.0065 (4)	−0.0019 (5)
C7B	0.0213 (6)	0.0257 (6)	0.0219 (6)	−0.0116 (5)	−0.0037 (4)	−0.0026 (5)
C8B	0.0263 (6)	0.0268 (6)	0.0201 (6)	−0.0107 (5)	−0.0029 (5)	0.0013 (5)
C9B	0.0241 (6)	0.0178 (5)	0.0181 (5)	−0.0036 (4)	−0.0036 (4)	−0.0002 (4)
C10B	0.0128 (5)	0.0174 (5)	0.0186 (5)	−0.0042 (4)	−0.0038 (4)	0.0001 (4)
C11B	0.0177 (5)	0.0143 (5)	0.0213 (5)	−0.0042 (4)	−0.0054 (4)	0.0019 (4)
C12B	0.0167 (5)	0.0176 (5)	0.0183 (5)	−0.0056 (4)	−0.0052 (4)	0.0042 (4)
C13B	0.0112 (4)	0.0154 (5)	0.0178 (5)	−0.0038 (4)	−0.0042 (4)	0.0002 (4)
C14B	0.0184 (5)	0.0136 (5)	0.0204 (5)	−0.0045 (4)	−0.0057 (4)	0.0025 (4)

C15B	0.0195 (5)	0.0185 (5)	0.0171 (5)	−0.0068 (4)	−0.0052 (4)	0.0031 (4)
C16B	0.0148 (5)	0.0178 (5)	0.0166 (5)	−0.0058 (4)	−0.0037 (4)	0.0003 (4)
C17B	0.0233 (6)	0.0199 (6)	0.0205 (5)	−0.0029 (4)	−0.0088 (4)	−0.0007 (4)
C18B	0.0276 (6)	0.0192 (6)	0.0271 (6)	−0.0012 (5)	−0.0082 (5)	−0.0042 (5)
C19B	0.0255 (6)	0.0250 (6)	0.0199 (6)	−0.0065 (5)	−0.0048 (5)	−0.0059 (5)
C20B	0.0287 (6)	0.0245 (6)	0.0190 (5)	−0.0051 (5)	−0.0083 (5)	0.0007 (4)
C21B	0.0236 (6)	0.0193 (6)	0.0184 (5)	−0.0028 (4)	−0.0056 (4)	0.0013 (4)

Table 6: Hydrogen-bond geometry (Å,º).**14**

D—H···A	*D*—H	H···A	D···A	D—H···A
O1A-H1C···N1A	0.84	1.82	2.5679 (13	148
O1B-H1D···N1B	0.84	1.84	2.5853 (13)	148

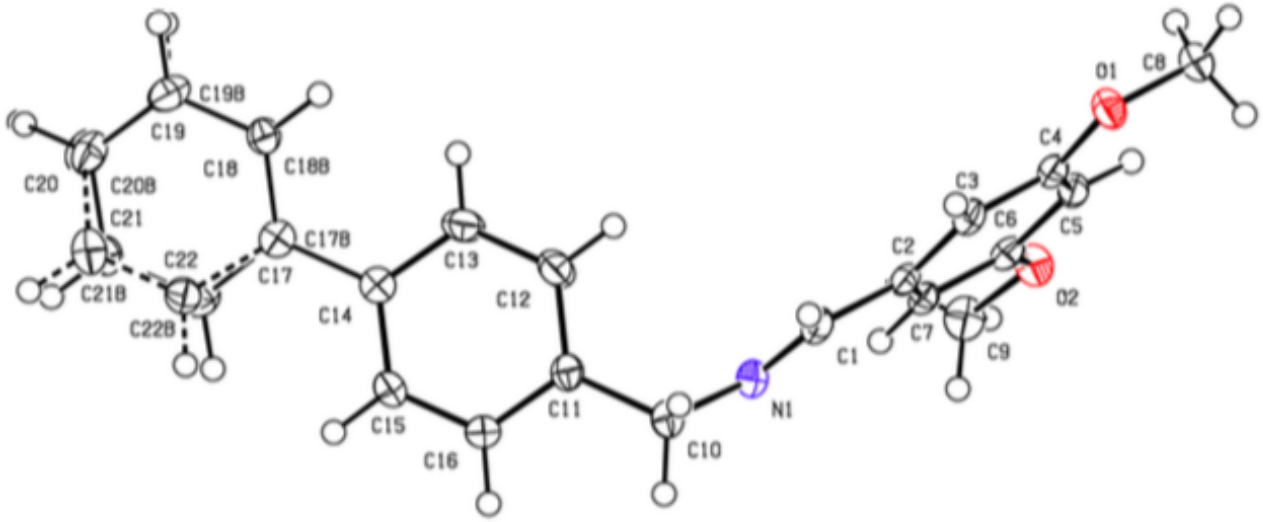

Figure 186: X-Ray Crystal Structure of **12**

Table 1. Experimental details.**12**

	Quest15mz225_0m
Crystal data	
Chemical formula	C22H21NO2
*M*r	331.40
Crystal system, space group	Orthorhombic, *P*212121
Temperature (K)	100
a, *b*, *c* (Å)	Cell setting orthorhombic 5.0309 (2), 8.6580 (4), 39.2775 (18)
V (Å3)	1710.83 (13)
Z	4
Radiation type	Mo *K*α
μ (mm−1)	0.08
Crystal size (mm)	0.55 × 0.11 × 0.08
Data collection	
Diffractometer	Bruker AXS D8 Quest CMOS diffractometer
Absorption correction	Multi-scan Apex2 v2014.11 (Bruker, 2014)
*T*min, *T*max	0.706, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15556, 4654, 3653
*R*int	0.032
(sin θ/λ)max (Å−1)	0.732
Refinement	
$R[F2 > 2\sigma(F2)]$, *wR*(*F*2), *S*	0.044, 0.085, 1.03
No. of reflections	4654
No. of parameters	269
No. of restraints	84
H-atom treatment	H-atom parameters constrained
Δρmax, Δρmin (e Å−3)	0.26, −0.22
Absolute structure	A phenyl ring is disordered over two orientations by slight rotation around the phenyl axis. Atoms of the two moieties at the 1 and 4 positions were each constrained to have identical ADPs. Atoms within the same moiety were subjected to a rigid bond restraint (RIGU), and the two moieties were restrained to have similar geometries. Subject to these conditions theoccupancy ratio refined to 0.475(15) to 0.526(15). Refined as an inversion twin.
Absolute structure parameter	0.3 (14)

Computer programs: Apex2 v2014.11 (Bruker, 2014), *SAINT* V8.34A (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2014), SHELXLE Rev714 (Hübschle *et al.*, 2011).

Table 2: Bond lengths (Å).**12**

C1—N1 1.263(2)	C13—C14 1.393(3)
C1—C2 1.474(2)	C13—H13 0.9500
C1—H1 0.9500	C14—C15 1.382(3)
C2—C3 1.385(2)	C14—C17B 1.491(2)
C2—C7 1.400(2)	C14—C17 1.491(2)
C3—C4 1.398(3)	C15—C16 1.386(3)
C3—H3 0.9500	C15—H15 0.9500
C4—O1 1.366(2)	C16—H16 0.9500
C4—C5 1.380(2)	C17—C18 1.400(8)
C5—C6 1.404(3)	C17—C22 1.454(6)
C5—H5 0.9500	C18—C19 1.382(9)
C6—O2 1.367(2)	C18—H18 0.9500
C6—C7 1.384(2)	C19—C20 1.373(18)
C7—H7 0.9500	C19—H19 0.9500
C8—O1 1.431(2)	C20—C21 1.38(2)
C8—H8A 0.9800	C20—H20 0.9500
C8—H8B 0.9800	C21—C22 1.390(7)
C8—H8C 0.9800	C21—H21 0.9500
C9—O2 1.427(2)	C22—H22 0.9500
C9—H9A 0.9800	C17B—C22B 1.366(5)
C9—H9B 0.9800	C17B—C18B 1.374(7)
C9—H9C 0.9800	C18B—C19B 1.390(8)
C10—N1 1.465(2)	C18B—H18B 0.9500
C10—C11 1.517(2)	C19B—C20B 1.381(17)

C10—H10A	0.9900	C19B—H19B	0.9500
C10—H10B	0.9900	C20B—C21B	1.383(19)
C11—C12	1.376(3)	C20B—H20B	0.9500
C11—C16	1.383(3)	C21B—C22B	1.401(7)
C12—C13	1.390(3)	C21B—H21B	0.9500
C12—H12	0.9500	C22B—H22B	0.9500

Table 3: Bond angles (deg**).12**

N1—C1—C2	122.60 (18)	C15—C14—C17B	121.63 (16)
N1—C1—H1	118.7	C13—C14—C17B	121.53 (17)
C2—C1—H1	118.7	C15—C14—C17	121.63 (16)
C3—C2—C7	120.60 (16)	C13—C14—C17	121.53 (17)
C3—C2—C1	118.73 (16)	C14—C15—C16	121.59 (18)
C7—C2—C1	120.67 (16)	C14—C15—H15	119.2
C2—C3—C4	120.12 (17)	C16—C15—H15	119.2
C2—C3—H3	119.9	C11—C16—C15	121.34 (18)
C4—C3—H3	119.9	C11—C16—H16	119.3
O1—C4—C5	124.70 (16)	C15—C16—H16	119.3
O1—C4—C3	115.31 (16)	C18—C17—C22	113.7 (4)
C5—C4—C3	119.98 (16)	C18—C17—C14	124.3 (4)
C4—C5—C6	119.41 (16)	C22—C17—C14	122.0 (3)
C4—C5—H5	120.3	C19—C18—C17	124.3 (7)
C6—C5—H5	120.3	C19—C18—H18	117.9
O2—C6—C7	124.51 (16)	C17—C18—H18	117.9
O2—C6—C5	114.25 (15)	C20—C19—C18	119.9 (10)
C7—C6—C5	121.24 (16)	C20—C19—H19	120.1
C6—C7—C2	118.63 (16)	C18—C19—H19	120.1
C6—C7—H7	120.7	C19—C20—C21	119.9 (12)
C2—C7—H7	120.7	C19—C20—H20	120.0
O1—C8—H8A	109.5	C21—C20—H20	120.0
O1—C8—H8B	109.5	C20—C21—C22	120.5 (9)
H8A—C8—H8B	109.5	C20—C21—H21	119.8
O1—C8—H8C	109.5	C22—C21—H21	119.8

H8A—C8—H8C	109.5	C21—C22—C17	121.8 (5)
H8B—C8—H8C	109.5	C21—C22—H22	119.1
O2—C9—H9A	109.5	C17—C22—H22	119.1
O2—C9—H9B	109.5	C22B—C17B—C18B	121.1 (4)
H9A—C9—H9B	109.5	C22B—C17B—C14	119.5 (3)
O2—C9—H9C	109.5	C18B—C17B—C14	119.3 (3)
H9A—C9—H9C	109.5	C17B—C18B—C19B	119.6 (6)
H9B—C9—H9C	109.5	C17B—C18B—H18B	120.2
N1—C10—C11	111.11 (16)	C19B—C18B—H18B	120.2
N1—C10—H10A	109.4	C20B—C19B—C18B	120.3 (9)
C11—C10—H10A	109.4	C20B—C19B—H19B	119.9
N1—C10—H10B	109.4	C18B—C19B—H19B	119.9
C11—C10—H10B	109.4	C19B—C20B—C21B	119.4 (11)
H10A—C10—H10B	108.0	C19B—C20B—H20B	120.3
C12—C11—C16	117.62 (17)	C21B—C20B—H20B	120.3
C12—C11—C10	121.22 (17)	C20B—C21B—C22B	120.4 (8)
C16—C11—C10	121.13 (17)	C20B—C21B—H21B	119.8
C11—C12—C13	121.17 (18)	C22B—C21B—H21B	119.8
C11—C12—H12	119.4	C17B—C22B—C21B	119.1 (5)
C13—C12—H12	119.4	C17B—C22B—H22B	120.4
C12—C13—C14	121.43 (18)	C21B—C22B—H22B	120.4
C12—C13—H13	119.3	C1—N1—C10	117.26 (17)
C14—C13—H13	119.3	C4—O1—C8	116.90 (14)
C15—C14—C13	116.84 (16)	C6—O2—C9	117.12 (14)
N1—C1—C2—C3	175.77 (17)	C13—C14—C17—C18	13.6 (5)
N1—C1—C2—C7	−4.0 (3)	C15—C14—C17—C22	16.5 (5)

C7—C2—C3—C4	0.2 (3)	C13—C14—C17—C22	−162.6 (5)
C1—C2—C3—C4	−179.51 (16)	C22—C17—C18—C19	−1.3 (6)
C2—C3—C4—O1	178.22 (16)	C14—C17—C18—C19	−177.8 (4)
C2—C3—C4—C5	−1.3 (3)	C17— C18—C19—C20	−1 (3)
O1—C4—C5—C6	−177.60 (16)	C18—C19—C20—C21	3 (7)
C3—C4—C5—C6	1.9 (3)	C19—C20—C21—C22	−2 (7)
C4—C5—C6—O2	178.80 (15)	C20—C21—C22—C17	0 (3)
C4—C5—C6—C7	−1.4 (3)	C18—C17—C22—C21	1.9 (6)
O2—C6—C7—C2	−179.91 (16)	C14—C17—C22—C21	178.5 (4)
C5—C6—C7—C2	0.4 (2)	C15—C14—C17B—C22B	−14.1 (5)
C3—C2—C7—C6	0.2 (2)	C13—C14—C17B—C22B	166.8 (5)
C1—C2—C7—C6	179.99 (15)	C15—C14—C17B—C18B	167.6 (5)
N1—C10—C11—C12	−50.1 (2)	C13—C14—C17B—C18B	−11.5 (5)
N1—C10—C11—C16	131.83 (19)	C22B—C17B—C18B—C19B	5.3 (7)
C16—C11—C12—C13	1.1 (3)	C14—C17B—C18B—C19B	−176.4 (4)
C10—C11—C12—C13	−177.0 (2)	C17B—C18B—C19B—C20B	−3 (3)
C11—C12—C13—C14	−0.2 (3)	C18B—C19B—C20B—C21B	0 (6)
C12—C13—C14—C15	−0.7 (3)	C19B—C20B—C21B—C22B	1 (6)
C12—C13—C14—C17B	178.46 (19)	C18B—C17B—C22B—C21B	−4.6 (6)
C12—C13—C14—C17	178.46 (19)	C14—C17B—C22B—C21B	177.1 (4)
C13—C14—C15—C16	0.5 (3)	C20B—C21B—C22B—C17B	1 (3)
C17B—C14—C15—C16	−178.60 (19)	C2—C1—N1—C10	−179.26 (15)
C17—C14—C15—C16	−178.60 (19)	C11—C10—N1—C1	113.79 (18)
C12—C11—C16—C15	−1.3 (3)	C5—C4—O1—C8	−1.4 (2)
C10—C11—C16—C15	176.86 (18)	C3—C4—O1—C8	179.02 (15)
C14—C15—C16—C11	0.4 (3)	C7—C6—O2—C9	7.3 (2)
C15—C14—C17—C18	−167.3 (5)	C5—C6—O2—C9	−172.91 (16)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2).**12**

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2554 (4)	0.8102 (2)	0.88961 (4)	0.0221 (4)	
H1	0.1520	0.9015	0.8876	0.027*	
C2	0.4701 (4)	0.8055 (2)	0.91512 (4)	0.0196 (4)	
C3	0.5249 (4)	0.9378 (2)	0.93367 (4)	0.0210 (4)	
H3	0.4238	1.0289	0.9299	0.025*	
C4	0.7287 (4)	0.9375 (2)	0.95787 (4)	0.0205 (4)	
C5	0.8720 (4)	0.8043 (2)	0.96380 (4)	0.0203 (4)	
H5	1.0070	0.8027	0.9807	0.024*	
C6	0.8165 (4)	0.6713 (2)	0.94464 (4)	0.0198 (4)	
C7	0.6167 (3)	0.6702 (2)	0.92042 (4)	0.0197 (4)	
H7	0.5798	0.5795	0.9077	0.024*	
C8	0.9750 (4)	1.0786 (2)	0.99936 (4)	0.0262 (4)	
H8A	0.9296	1.0070	1.0178	0.039*	
H8B	0.9908	1.1834	1.0085	0.039*	
H8C	1.1445	1.0477	0.9891	0.039*	
C9	0.9495 (4)	0.4143 (2)	0.93108 (5)	0.0287 (4)	
H9A	0.7700	0.3716	0.9333	0.043*	
H9B	1.0800	0.3365	0.9381	0.043*	
H9C	0.9817	0.4434	0.9073	0.043*	
C10	−0.0129 (4)	0.7175 (2)	0.84574 (4)	0.0257 (4)	
H10A	−0.1042	0.8168	0.8501	0.031*	
H10B	−0.1442	0.6334	0.8487	0.031*	
C11	0.0912 (4)	0.7158 (2)	0.80949 (4)	0.0203 (4)	
C12	0.3023 (4)	0.8071 (2)	0.79988 (5)	0.0298 (5)	
H12	0.3897	0.8686	0.8165	0.036*	
C13	0.3897 (4)	0.8108 (3)	0.76632 (5)	0.0340 (5)	
H13	0.5357	0.8752	0.7604	0.041*	

C14	0.2681 (4)	0.7223 (2)	0.74115 (4)	0.0197 (4)	
C15	0.0587 (4)	0.6297 (2)	0.75121 (5)	0.0292 (5)	
H15	−0.0282	0.5670	0.7348	0.035*	
C16	−0.0277 (4)	0.6260 (2)	0.78469 (5)	0.0324 (5)	
H16	−0.1714	0.5604	0.7907	0.039*	
C17	0.3572 (4)	0.7291 (2)	0.70498 (4)	0.0208 (4)	0.475 (15)
C18	0.5958 (16)	0.7964 (10)	0.6942 (2)	0.0247 (15)	0.475 (15)
H18	0.7112	0.8373	0.7111	0.030*	0.475 (15)
C19	0.6743 (19)	0.8072 (11)	0.66055 (19)	0.0299 (17)	0.475 (15)
H19	0.8376	0.8559	0.6549	0.036*	0.475 (15)
C20	0.516 (9)	0.747 (6)	0.6353 (5)	0.0280 (8)	0.475 (15)
H20	0.5730	0.7505	0.6123	0.034*	0.475 (15)
C21	0.2736 (17)	0.6829 (11)	0.64347 (17)	0.0320 (16)	0.475 (15)
H21	0.1616	0.6449	0.6259	0.038*	0.475 (15)
C22	0.1921 (16)	0.6731 (10)	0.67722 (14)	0.0247 (15)	0.475 (15)
H22	0.0241	0.6286	0.6823	0.030*	0.475 (15)
C17B	0.3572 (4)	0.7291 (2)	0.70498 (4)	0.0208 (4)	0.525 (15)
C18B	0.5299 (16)	0.8434 (9)	0.69501 (16)	0.0220 (13)	0.525 (15)
H18B	0.5997	0.9137	0.7113	0.026*	0.525 (15)
C19B	0.6019 (17)	0.8556 (10)	0.66095 (17)	0.0290 (15)	0.525 (15)
H19B	0.7152	0.9371	0.6538	0.035*	0.525 (15)
C20B	0.509 (8)	0.750 (6)	0.6375 (4)	0.0280 (8)	0.525 (15)
H20B	0.5572	0.7584	0.6142	0.034*	0.525 (15)
C21B	0.3459 (15)	0.6308 (9)	0.64827 (15)	0.0266 (14)	0.525 (15)
H21B	0.2840	0.5565	0.6323	0.032*	0.525 (15)
C22B	0.2712 (15)	0.6195 (9)	0.68253 (14)	0.0247 (13)	0.525 (15)
H22B	0.1622	0.5367	0.6901	0.030*	0.525 (15)
N1	0.2034 (3)	0.69748 (18)	0.87030 (4)	0.0252 (4)	
O1	0.7708 (3)	1.07509 (14)	0.97401 (3)	0.0252 (3)	
O2	0.9741 (3)	0.54747 (14)	0.95226 (3)	0.0255 (3)	

Table 5: Atomic displacement parameters (Å2). **12**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C1	0.0230 (10)	0.0263 (9)	0.0171 (8)	0.0009 (8)	0.0014 (7)	0.0038 (7)
C2	0.0196 (9)	0.0247 (9)	0.0145 (7)	−0.0025 (8)	0.0023 (7)	0.0034 (7)
C3	0.0223 (9)	0.0228 (9)	0.0178 (8)	0.0025 (8)	0.0009 (7)	0.0041 (7)
C4	0.0241 (10)	0.0218 (9)	0.0157 (8)	−0.0026 (8)	0.0027 (7)	0.0007 (7)
C5	0.0195 (9)	0.0251 (9)	0.0162 (8)	−0.0008 (8)	0.0001 (7)	0.0013 (7)
C6	0.0189 (9)	0.0220 (9)	0.0185 (8)	0.0012 (8)	0.0050 (7)	0.0032 (7)
C7	0.0225 (10)	0.0213 (9)	0.0154 (8)	−0.0026 (7)	0.0042 (7)	0.0004 (7)
C8	0.0282 (11)	0.0277 (10)	0.0228 (9)	−0.0036 (9)	−0.0025 (8)	−0.0037 (8)
C9	0.0342 (11)	0.0226 (10)	0.0294 (10)	0.0059 (9)	0.0005 (9)	−0.0014 (8)
C10	0.0252 (11)	0.0324 (10)	0.0194 (8)	−0.0038 (9)	−0.0038 (8)	0.0018 (8)
C11	0.0208 (10)	0.0207 (9)	0.0195 (8)	0.0030 (7)	−0.0034 (7)	0.0025 (7)
C12	0.0251 (11)	0.0380 (11)	0.0263 (10)	−0.0082 (10)	0.0007 (8)	−0.0106 (9)
C13	0.0295 (12)	0.0407 (12)	0.0319 (10)	−0.0166 (10)	0.0085 (9)	−0.0109 (9)
C14	0.0189 (9)	0.0169 (8)	0.0232 (8)	0.0049 (7)	−0.0007 (8)	0.0000 (7)
C15	0.0327 (11)	0.0337 (11)	0.0210 (9)	−0.0121 (9)	−0.0064 (9)	0.0004 (8)
C16	0.0354 (12)	0.0394 (11)	0.0223 (9)	−0.0190 (10)	−0.0018 (9)	0.0034 (8)
C17	0.0217 (9)	0.0175 (9)	0.0231 (8)	0.0046 (7)	0.0009 (7)	0.0003 (7)
C18	0.023 (3)	0.024 (4)	0.027 (2)	0.004 (3)	−0.002 (2)	−0.001 (3)
C19	0.032 (4)	0.031 (4)	0.026 (2)	−0.001 (3)	0.005 (2)	0.004 (3)
C20	0.0362 (19)	0.0275 (15)	0.0203 (17)	0.0025 (16)	0.0059 (18)	0.001 (2)
C21	0.037 (4)	0.036 (4)	0.023 (2)	0.000 (3)	−0.003 (2)	−0.004 (2)
C22	0.029 (3)	0.022 (3)	0.023 (2)	−0.003 (3)	−0.001 (2)	0.000 (2)
C17B	0.0217 (9)	0.0175 (9)	0.0231 (8)	0.0046 (7)	0.0009 (7)	0.0003 (7)

C18 B	0.026 (3)	0.021 (3)	0.0197 (18)	−0.001 (2)	0.002 (2)	−0.004 (2)
C19 B	0.031 (4)	0.027 (3)	0.029 (2)	−0.004 (3)	0.008 (2)	0.004 (2)
C20 B	0.0362 (19)	0.0275 (15)	0.0203 (17)	0.0025 (16)	0.0059 (18)	0.001 (2)
C21 B	0.033 (3)	0.027 (3)	0.020 (2)	−0.003 (2)	0.002 (2)	−0.006 (2)
C22 B	0.030 (3)	0.020 (3)	0.024 (2)	−0.001 (2)	0.005 (2)	0.0007 (18)
N1	0.0285 (9)	0.0292 (8)	0.0178 (7)	−0.0041 (8)	−0.0037 (7)	0.0036 (6)
O1	0.0318 (8)	0.0211 (7)	0.0227 (6)	−0.0008 (6)	−0.0066 (6)	−0.0020 (5)
O2	0.0263 (7)	0.0235 (7)	0.0267 (7)	0.0050 (6)	−0.0032 (6)	−0.0010 (5)

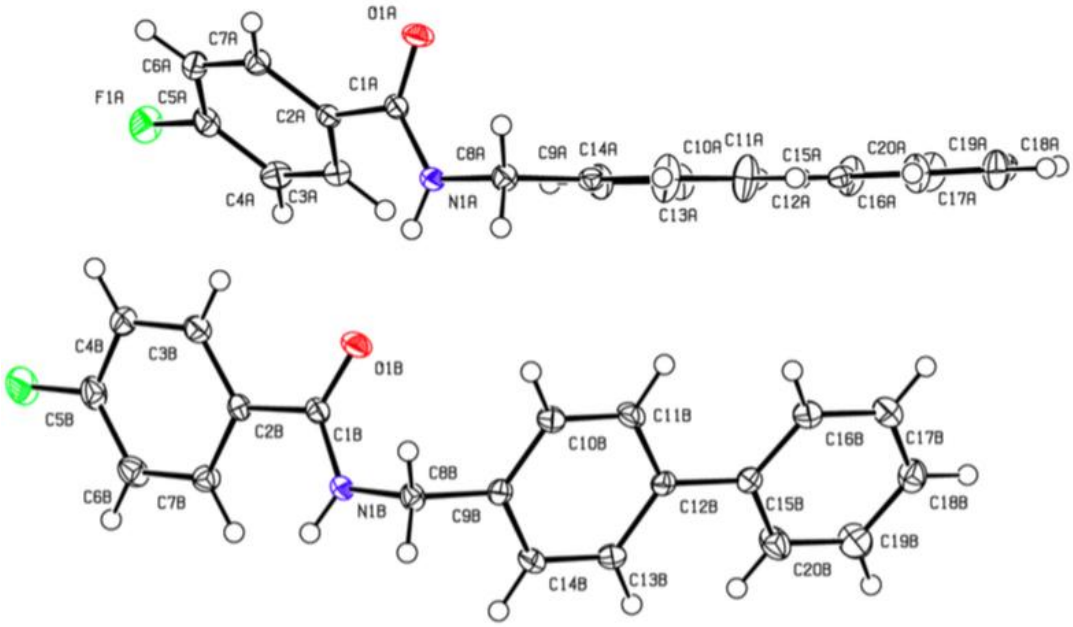

Figure 187: X-Ray Crystal Structure of **32**

Table 1. Experimental details.**32**

	Quest15mz227_0m
Crystal data	
Chemical formula	C20H16FNO
*M*r	305.34
Crystal system, space group	Triclinic, *P*1
Temperature (K)	100
a, *b*, *c* (Å)	Cell setting:triclinic 9.4862 (10), 9.8459 (11), 17.533 (2)
α, β, γ (°)	Cell setting:triclinic 104.269 (6), 96.928 (7), 103.868 (5)
V (Å3)	1512.4 (3)
Z	4
Radiation type	Mo *K*α
μ (mm−1)	0.09
Crystal size (mm)	0.55 × 0.12 × 0.08
Data collection	
Diffractometer	Bruker AXS D8 Quest CMOS diffractometer
Absorption correction	Multi-scan Apex2 v2014.1-1 (Bruker, 2014)
*T*min, *T*max	0.531, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23354, 8334, 5939
*R*int	0.056
(sin θ/λ)max (Å−1)	0.715
Refinement	
$R[F2 > 2\sigma(F2)]$, *wR*(*F*2), *S*	0.056, 0.155, 1.03
No. of reflections	8334
No. of parameters	421
No. of restraints	2

H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρmax, Δρmin (e Å−3)	0.38, −0.35

Computer programs: Apex2 v2014.1-1 (Bruker, 2014), *SAINT* V8.34A (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2014), SHELXLE Rev714 (Hübschle *et al.*, 2011).

Special details

Geometry

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Table 2: Bond lengths (Å). **32**

N1A—C1A 1.3362(17)	F1B—C5B 1.3570(16)
N1A—C8A 1.4538(16)	C1B—O1B 1.2379(16)
N1A—H1A 0.876(13)	C1B—N1B 1.3387(18)
F1A—C5A 1.3647(14)	C1B—C2B 1.5043(18)
C1A—O1A 1.2365(16)	N1B—C8B 1.4695(16)
C1A—C2A 1.5012(17)	N1B—H1B 0.875(13)
C2A—C3A 1.3941(19)	C2B—C3B 1.392(2)
C2A—C7A 1.3963(19)	C2B—C7B 1.3969(19)
C3A—C4A 1.3927(18)	C3B—C4B 1.3881(19)
C3A—H3A 0.9500	C3B—H3B 0.9500
C4A—C5A 1.379(2)	C4B—C5B 1.379(2)
C4A—H4A 0.9500	C4B—H4B 0.9500
C5A—C6A 1.378(2)	C5B—C6B 1.378(2)
C6A—C7A 1.3878(18)	C6B—C7B 1.3878(19)
C6A—H6A 0.9500	C6B—H6B 0.9500
C7A—H7A 0.9500	C7B—H7B 0.9500
C8A—C9A 1.522(2)	C8B—C9B 1.516(2)
C8A—H8AA 0.9900	C8B—H8BA 0.9900
C8A—H8AB 0.9900	C8B—H8BB 0.9900
C9A—C14A 1.382(2)	C9B—C14B 1.386(2)
C9A—C10A 1.3928(19)	C9B—C10B 1.3938(19)
C10A—C11A 1.382(2)	C10B—C11B 1.386(2)
C10A—H10A 0.9500	C10B—H10B 0.9500
C11A—C12A 1.399(2)	C11B—C12B 1.395(2)

C11A—H11A	0.9500	C11B—H11B	0.9500
C12A—C13A	1.3965(18)	C12B—C13B	1.406(2)
C12A—C15A	1.491(2)	C12B—C15B	1.491(2)
C13A—C14A	1.390(2)	C13B—C14B	1.388(2)
C13A—H13A	0.9500	C13B—H13B	0.9500
C14A—H14A	0.9500	C14B—H14B	0.9500
C15A—C16A	1.3986(18)	C15B—C20B	1.392(2)
C15A—C20A	1.404(2)	C15B—C16B	1.403(2)
C16A—C17A	1.390(2)	C16B—C17B	1.390(2)
C16A—H16A	0.9500	C16B—H16B	0.9500
C17A—C18A	1.385(2)	C17B—C18B	1.382(2)
C17A—H17A	0.9500	C17B—H17B	0.9500
C18A—C19A	1.390(2)	C18B—C19B	1.387(2)
C18A—H18A	0.9500	C18B—H18B	0.9500
C19A—C20A	1.385(2)	C19B—C20B	1.385(2)
C19A—H19A	0.9500	C19B—H19B	0.9500
C20A—H20A	0.9500	C20B—H20B	0.9500

Table 3: Bond angles (deg). **32**

C1A—N1A—C8A	121.28 (11)	O1B—C1B—N1B	122.12 (12)
C1A—N1A—H1A	121.5 (11)	O1B—C1B—C2B	121.01 (12)
C8A—N1A—H1A	117.1 (11)	N1B—C1B—C2B	116.86 (12)
O1A—C1A—N1A	122.90 (12)	C1B—N1B—C8B	120.89 (11)
O1A—C1A—C2A	120.61 (11)	C1B—N1B—H1B	120.7 (11)
N1A—C1A—C2A	116.48 (11)	C8B—N1B—H1B	117.1 (11)
C3A—C2A—C7A	119.65 (12)	C3B—C2B—C7B	118.98 (12)
C3A—C2A—C1A	121.58 (12)	C3B—C2B—C1B	117.60 (12)
C7A—C2A—C1A	118.64 (12)	C7B—C2B—C1B	123.41 (12)
C4A—C3A—C2A	120.48 (13)	C4B—C3B—C2B	121.11 (13)
C4A—C3A—H3A	119.8	C4B—C3B—H3B	119.4
C2A—C3A—H3A	119.8	C2B—C3B—H3B	119.4
C5A—C4A—C3A	117.82 (13)	C5B—C4B—C3B	118.03 (13)
C5A—C4A—H4A	121.1	C5B—C4B—H4B	121.0
C3A—C4A—H4A	121.1	C3B—C4B—H4B	121.0
F1A—C5A—C6A	118.05 (12)	F1B—C5B—C6B	118.55 (13)
F1A—C5A—C4A	118.43 (12)	F1B—C5B—C4B	118.65 (13)
C6A—C5A—C4A	123.52 (12)	C6B—C5B—C4B	122.80 (13)
C5A—C6A—C7A	117.95 (12)	C5B—C6B—C7B	118.42 (13)
C5A—C6A—H6A	121.0	C5B—C6B—H6B	120.8
C7A—C6A—H6A	121.0	C7B—C6B—H6B	120.8
C6A—C7A—C2A	120.54 (12)	C6B—C7B—C2B	120.65 (13)
C6A—C7A—H7A	119.7	C6B—C7B—H7B	119.7
C2A—C7A—H7A	119.7	C2B—C7B—H7B	119.7

N1A—C8A—C9A	113.57 (11)	N1B—C8B—C9B	113.17 (11)
N1A—C8A—H8AA	108.9	N1B—C8B—H8BA	108.9
C9A—C8A—H8AA	108.9	C9B—C8B—H8BA	108.9
N1A—C8A—H8AB	108.9	N1B—C8B—H8BB	108.9
C9A—C8A—H8AB	108.9	C9B—C8B—H8BB	108.9
H8AA—C8A—H8AB	107.7	H8BA—C8B—H8BB	107.8
C14A—C9A—C10A	117.08 (13)	C14B—C9B—C10B	117.77 (14)
C14A—C9A—C8A	123.19 (12)	C14B—C9B—C8B	120.61 (13)
C10A—C9A—C8A	119.73 (12)	C10B—C9B—C8B	121.62 (13)
C11A—C10A—C9A	121.57 (13)	C11B—C10B—C9B	121.39 (13)
C11A—C10A—H10A	119.2	C11B—C10B—H10B	119.3
C9A—C10A—H10A	119.2	C9B—C10B—H10B	119.3
C10A—C11A—C12A	121.81 (13)	C10B—C11B—C12B	121.41 (13)
C10A—C11A—H11A	119.1	C10B—C11B—H11B	119.3
C12A—C11A—H11A	119.1	C12B—C11B—H11B	119.3
C13A—C12A—C11A	116.19 (13)	C11B—C12B—C13B	116.82 (13)
C13A—C12A—C15A	121.98 (12)	C11B—C12B—C15B	122.26 (12)
C11A—C12A—C15A	121.82 (12)	C13B—C12B—C15B	120.91 (12)
C14A—C13A—C12A	121.74 (13)	C14B—C13B—C12B	121.52 (13)
C14A—C13A—H13A	119.1	C14B—C13B—H13B	119.2
C12A—C13A—H13A	119.1	C12B—C13B—H13B	119.2
C9A—C14A—C13A	121.60 (12)	C9B—C14B—C13B	121.09 (14)
C9A—C14A—H14A	119.2	C9B—C14B—H14B	119.5
C13A—C14A—H14A	119.2	C13B—C14B—H14B	119.5
C16A—C15A—C20A	116.79 (13)	C20B—C15B—C16B	117.29 (14)
C16A—C15A—C12A	121.59 (12)	C20B—C15B—C12B	121.18 (13)

C20A—C15A—C12A	121.60 (12)	C16B—C15B—C12B	121.52 (13)
C17A—C16A—C15A	121.56 (13)	C17B—C16B—C15B	121.06 (14)
C17A—C16A—H16A	119.2	C17B—C16B—H16B	119.5
C15A—C16A—H16A	119.2	C15B—C16B—H16B	119.5
C18A—C17A—C16A	120.61 (14)	C18B—C17B—C16B	120.71 (15)
C18A—C17A—H17A	119.7	C18B—C17B—H17B	119.6
C16A—C17A—H17A	119.7	C16B—C17B—H17B	119.6
C17A—C18A—C19A	118.88 (14)	C17B—C18B—C19B	118.76 (15)
C17A—C18A—H18A	120.6	C17B—C18B—H18B	120.6
C19A—C18A—H18A	120.6	C19B—C18B—H18B	120.6
C20A—C19A—C18A	120.42 (13)	C20B—C19B—C18B	120.68 (15)
C20A—C19A—H19A	119.8	C20B—C19B—H19B	119.7
C18A—C19A—H19A	119.8	C18B—C19B—H19B	119.7
C19A—C20A—C15A	121.74 (13)	C19B—C20B—C15B	121.49 (15)
C19A—C20A—H20A	119.1	C19B—C20B—H20B	119.3
C15A—C20A—H20A	119.1	C15B—C20B—H20B	119.3
C8A—N1A—C1A—O1A	−5.0 (2)	O1B—C1B—N1B—C8B	4.8 (2)
C8A—N1A—C1A—C2A	173.77 (12)	C2B—C1B—N1B—C8B	−174.13 (12)
O1A—C1A—C2A—C3A	136.70 (15)	O1B—C1B—C2B—C3B	−18.6 (2)
N1A—C1A—C2A—C3A	−42.09 (19)	N1B—C1B—C2B—C3B	160.34 (13)
O1A—C1A—C2A—C7A	−39.1 (2)	O1B—C1B—C2B—C7	162.52 (14)
N1A—C1A—C2A—C7A	142.13 (14)	N1B—C1B—C2B—C7B	−18.5 (2)
C7A—C2A—C3A—C4A	0.8 (2)	C7B—C2B—C3B—C4B	0.5 (2)
C1A—C2A—C3A—C4A	−174.93 (13)	C1B—C2B—C3B—C4B	−178.45 (13)
C2A—C3A—C4A—C5A	−1.7 (2)	C2B—C3B—C4B—C5B	−1.2 (2)
C3A—C4A—C5A—F1A	−179.02 (13)	C3B—C4B—C5B—F1B	−179.10 (14)

C3A—C4A—C5A—C6A	0.9 (2)	C3B—C4B—C5B—C6B	0.9 (2)
F1A—C5A—C6A—C7A	−179.14 (13)	F1B—C5B—C6B—C7B	−179.88 (14)
C4A—C5A—C6A—C7A	1.0 (2)	C4B—C5B—C6B—C7B	0.1 (2)
C5A—C6A—C7A—C2A	−1.9 (2)	C5B—C6B—C7B—C2B	−0.9 (2)
C3A—C2A—C7A—C6A	1.1 (2)	C3B—C2B—C7B—C6B	0.6 (2)
C1A—C2A—C7A—C6A	176.95 (13)	C1B—C2B—C7B—C6B	179.44 (13)
C1A—N1A—C8A—C9A	−92.20 (15)	C1B—N1B—C8B—C9B	−80.46 (16)
N1A—C8A—C9A—C14A	0.40 (19)	N1B—C8B—C9B—C14B	−75.56 (16)
N1A—C8A—C9A—C10A	−179.90 (13)	N1B—C8B—C9B—C10B	104.81 (14)
C14A—C9A—C10A—C11A	0.8 (2)	C14B—C9B—C10B—C11B	0.6 (2)
C8A—C9A—C10A—C11A	−178.87 (16)	C8B—C9B—C10B—C11B	−179.77 (12)
C9A—C10A—C11A—C12A	0.2 (3)	C9B—C10B—C11B—C12B	−0.2 (2)
C10A—C11A—C12A—C13A	−1.1 (2)	C10B—C11B—C12B—C13B	−0.2 (2)
C10A—C11A—C12A—C15A	178.16 (16)	C10B—C11B—C12B—C15B	−179.61 (12)
C11A—C12A—C13A—C14A	0.9 (2)	C11B—C12B—C13B—C14B	0.2 (2)
C15A—C12A—C13A—C14A	−178.27 (13)	C15B—C12B—C13B—C14B	179.59 (13)
C10A—C9A—C14A—C13A	−0.9 (2)	C10B—C9B—C14B—C13B	−0.6 (2)
C8A—C9A—C14A—C13A	178.76 (14)	C8B—C9B—C14B—C13B	179.74 (13)
C12A—C13A—C14A—C9A	0.0 (2)	C12B—C13B—C14B—C9B	0.2 (2)
C13A—C12A—C15A—C16A	179.07 (14)	C11B—C12B—C15B—C20B	−169.89 (13)
C11A—C12A—C15A—C16A	−0.1 (2)	C13B—C12B—C15B—C20B	10.76 (19)
C13A—C12A—C15A—C20A	0.5 (2)	C11B—C12B—C15B—C16B	10.64 (19)
C11A—C12A—C15A—C20A	−178.71 (15)	C13B—C12B—C15B—C16B	−168.71 (13)
C20A—C15A—C16A—C17A	−0.5 (2)	C20B—C15B—C16B—C17B	0.4 (2)
C12A—C15A—C16A—C17A	−179.13 (14)	C12B—C15B—C16B—C17B	179.90 (13)
C15A—C16A—C17A—C18A	0.7 (3)	C15B—C16B—C17B—C18B	−1.3 (2)

C16A—C17A—C18A—C19A	−0.5 (2)	C16B—C17B—C18B—C19B	1.4 (2)
C17A—C18A—C19A—C20A	0.0 (2)	C17B—C18B—C19B—C20B	−0.7 (2)
C18A—C19A—C20A—C15A	0.2 (2)	C18B—C19B—C20B—C15B	−0.2 (3)
C16A—C15A—C20A—C19A	0.0 (2)	C16B—C15B—C20B—C19B	0.3 (2)
C12A—C15A—C20A—C19A	178.68 (14)	C12B—C15B—C20B—C19B	−179.20 (14)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2). **32**

	x	*y*	*z*	*U*iso*/*U*eq
N1A	0.15032 (12)	0.69953 (13)	0.34308 (7)	0.0162 (2)
H1A	0.1227 (17)	0.6068 (15)	0.3406 (10)	0.019*
F1A	−0.50188 (9)	0.55159 (10)	0.41546 (6)	0.0275 (2)
C1A	0.07026 (14)	0.78935 (14)	0.37009 (8)	0.0149 (3)
O1A	0.11461 (11)	0.92320 (10)	0.38302 (7)	0.0215 (2)
C2A	−0.08060 (14)	0.71929 (14)	0.38283 (8)	0.0148 (3)
C3A	−0.17334 (15)	0.59203 (15)	0.32792 (9)	0.0179 (3)
H3A	−0.1383	0.5435	0.2834	0.021*
C4A	−0.31706 (15)	0.53547 (16)	0.33781 (9)	0.0200 (3)
H4A	−0.3820	0.4503	0.2999	0.024*
C5A	−0.36206 (14)	0.60709 (16)	0.40440 (9)	0.0188 (3)
C6A	−0.27325 (15)	0.73196 (16)	0.46067 (9)	0.0186 (3)
H6A	−0.3078	0.7774	0.5062	0.022*
C7A	−0.13180 (14)	0.78932 (15)	0.44873 (9)	0.0171 (3)
H7A	−0.0693	0.8770	0.4857	0.020*
C8A	0.29153 (14)	0.75346 (15)	0.32093 (9)	0.0174 (3)
H8AA	0.3581	0.6954	0.3340	0.021*
H8AB	0.3372	0.8560	0.3535	0.021*
C9A	0.27912 (14)	0.74636 (14)	0.23260 (9)	0.0159 (3)
C10A	0.40600 (15)	0.7943 (2)	0.20301 (10)	0.0298 (4)
H10A	0.4994	0.8294	0.2383	0.036*
C11A	0.39933 (15)	0.7921 (2)	0.12352 (10)	0.0295 (4)
H11A	0.4884	0.8255	0.1056	0.035*
C12A	0.26510 (13)	0.74203 (14)	0.06878 (8)	0.0145 (3)
C13A	0.13876 (15)	0.69220 (17)	0.09874 (9)	0.0219 (3)
H13A	0.0453	0.6558	0.0635	0.026*
C14A	0.14618 (15)	0.69449 (17)	0.17872 (9)	0.0218 (3)

H14A	0.0577	0.6595	0.1968	0.026*
C15A	0.25736 (14)	0.74369 (14)	−0.01641 (8)	0.0155 (3)
C16A	0.38425 (15)	0.79574 (18)	−0.04562 (9)	0.0237 (3)
H16A	0.4778	0.8296	−0.0105	0.028*
C17A	0.37671 (16)	0.79906 (19)	−0.12478 (10)	0.0265 (3)
H17A	0.4649	0.8340	−0.1431	0.032*
C18A	0.24182 (16)	0.75192 (16)	−0.17720 (9)	0.0214 (3)
H18A	0.2367	0.7549	−0.2312	0.026*
C19A	0.11416 (16)	0.70015 (18)	−0.14948 (10)	0.0264 (3)
H19A	0.0209	0.6674	−0.1848	0.032*
C20A	0.12203 (15)	0.69601 (18)	−0.07071 (9)	0.0253 (3)
H20A	0.0335	0.6600	−0.0530	0.030*
F1B	−0.47568 (10)	0.13072 (11)	0.47506 (7)	0.0380 (3)
C1B	0.04928 (14)	0.28124 (15)	0.35896 (8)	0.0158 (3)
O1B	0.09649 (11)	0.40727 (11)	0.35476 (7)	0.0228 (2)
N1B	0.12214 (12)	0.18074 (13)	0.33943 (7)	0.0161 (2)
H1B	0.0936 (17)	0.0962 (16)	0.3490 (10)	0.019*
C2B	−0.09261 (14)	0.23498 (15)	0.38795 (8)	0.0150 (3)
C3B	−0.14548 (15)	0.34429 (16)	0.43143 (9)	0.0197 (3)
H3B	−0.0929	0.4437	0.4404	0.024*
C4B	−0.27373 (17)	0.31051 (17)	0.46191 (10)	0.0241 (3)
H4B	−0.3086	0.3852	0.4924	0.029*
C5B	−0.34902 (16)	0.16524 (17)	0.44656 (10)	0.0238 (3)
C6B	−0.30110 (15)	0.05355 (16)	0.40352 (10)	0.0226 (3)
H6B	−0.3556	−0.0455	0.3939	0.027*
C7B	−0.17139 (14)	0.08920 (15)	0.37459 (9)	0.0184 (3)
H7B	−0.1358	0.0137	0.3454	0.022*
C8B	0.26851 (14)	0.21915 (16)	0.31787 (9)	0.0173 (3)
H8BA	0.3205	0.1464	0.3260	0.021*
H8BB	0.3272	0.3155	0.3542	0.021*

C9B	0.26113 (14)	0.22497 (15)	0.23194 (9)	0.0162 (3)
C10B	0.29588 (15)	0.35726 (15)	0.21328 (9)	0.0194 (3)
H10B	0.3253	0.4461	0.2554	0.023*
C11B	0.28844 (15)	0.36199 (15)	0.13455 (9)	0.0190 (3)
H11B	0.3126	0.4540	0.1238	0.023*
C12B	0.24624 (13)	0.23459 (14)	0.07072 (8)	0.0147 (3)
C13B	0.21232 (17)	0.10163 (16)	0.09013 (9)	0.0230 (3)
H13B	0.1835	0.0125	0.0483	0.028*
C14B	0.22002 (17)	0.09756 (16)	0.16909 (9)	0.0235 (3)
H14B	0.1967	0.0059	0.1802	0.028*
C15B	0.23843 (13)	0.23723 (15)	−0.01432 (8)	0.0151 (3)
C16B	0.29606 (17)	0.36648 (17)	−0.03408 (10)	0.0249 (3)
H16B	0.3410	0.4546	0.0075	0.030*
C17B	0.28841 (19)	0.36770 (18)	−0.11356 (10)	0.0288 (4)
H17B	0.3264	0.4568	−0.1256	0.035*
C18B	0.22617 (16)	0.24059 (18)	−0.17530 (10)	0.0240 (3)
H18B	0.2230	0.2412	−0.2296	0.029*
C19B	0.16850 (19)	0.11217 (19)	−0.15653 (10)	0.0303 (4)
H19B	0.1244	0.0242	−0.1984	0.036*
C20B	0.17452 (18)	0.11085 (17)	−0.07742 (10)	0.0271 (3)
H20B	0.1341	0.0217	−0.0659	0.032*

Table 5: Atomic displacement parameters (Å2). **32**

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N1A	0.0176 (5)	0.0130 (6)	0.0200 (6)	0.0047 (4)	0.0070 (5)	0.0065 (5)
F1A	0.0141 (4)	0.0292 (5)	0.0355 (6)	−0.0002 (3)	0.0086 (4)	0.0068 (4)
C1A	0.0165 (6)	0.0141 (7)	0.0142 (6)	0.0035 (5)	0.0028 (5)	0.0053 (5)
O1A	0.0213 (5)	0.0120 (5)	0.0326 (6)	0.0045 (4)	0.0089 (4)	0.0077 (4)
C2A	0.0159 (6)	0.0135 (6)	0.0170 (7)	0.0049 (5)	0.0042 (5)	0.0068 (5)
C3A	0.0192 (6)	0.0164 (7)	0.0182 (7)	0.0055 (5)	0.0040 (5)	0.0045 (6)
C4A	0.0174 (6)	0.0159 (7)	0.0213 (7)	0.0009 (5)	−0.0002 (5)	0.0010 (6)
C5A	0.0122 (6)	0.0206 (7)	0.0252 (8)	0.0030 (5)	0.0049 (5)	0.0104 (6)
C6A	0.0186 (6)	0.0207 (7)	0.0176 (7)	0.0061 (5)	0.0055 (5)	0.0058 (6)
C7A	0.0176 (6)	0.0161 (7)	0.0163 (7)	0.0039 (5)	0.0022 (5)	0.0039 (6)
C8A	0.0150 (6)	0.0188 (7)	0.0193 (7)	0.0052 (5)	0.0044 (5)	0.0065 (6)
C9A	0.0159 (6)	0.0141 (7)	0.0197 (7)	0.0064 (5)	0.0053 (5)	0.0056 (6)
C10A	0.0109 (6)	0.0529 (11)	0.0196 (8)	0.0005 (6)	−0.0002 (5)	0.0093 (7)
C11A	0.0112 (6)	0.0511 (11)	0.0220 (8)	0.0006 (6)	0.0049 (6)	0.0100 (7)
C12A	0.0132 (6)	0.0128 (6)	0.0171 (7)	0.0047 (5)	0.0030 (5)	0.0027 (5)
C13A	0.0112 (6)	0.0293 (8)	0.0227 (8)	0.0006 (5)	0.0012 (5)	0.0089 (6)
C14A	0.0132 (6)	0.0279 (8)	0.0243 (8)	0.0018 (5)	0.0057 (5)	0.0105 (6)
C15A	0.0151 (6)	0.0122 (6)	0.0187 (7)	0.0050 (5)	0.0040 (5)	0.0020 (5)
C16A	0.0130 (6)	0.0357 (9)	0.0217 (8)	0.0028 (6)	0.0022 (5)	0.0113 (7)
C17A	0.0186 (7)	0.0389 (9)	0.0240 (8)	0.0064 (6)	0.0071 (6)	0.0126 (7)
C18A	0.0256 (7)	0.0239 (8)	0.0161 (7)	0.0103 (6)	0.0045 (6)	0.0045 (6)
C19A	0.0174 (7)	0.0353 (9)	0.0209 (8)	0.0034 (6)	−0.0017 (6)	0.0044 (7)
C20A	0.0142 (6)	0.0363 (9)	0.0215 (8)	0.0016 (6)	0.0032 (5)	0.0066 (7)

F1B	0.0300 (5)	0.0414 (6)	0.0604 (7)	0.0168 (4)	0.0309 (5)	0.0293 (6)
C1B	0.0178 (6)	0.0148 (7)	0.0140 (6)	0.0026 (5)	0.0021 (5)	0.0051 (5)
O1B	0.0245 (5)	0.0147 (5)	0.0319 (6)	0.0046 (4)	0.0100 (4)	0.0099 (5)
N1B	0.0177 (5)	0.0134 (6)	0.0189 (6)	0.0035 (4)	0.0070 (4)	0.0069 (5)
C2B	0.0163 (6)	0.0172 (7)	0.0122 (6)	0.0047 (5)	0.0015 (5)	0.0062 (5)
C3B	0.0222 (7)	0.0184 (7)	0.0198 (7)	0.0059 (5)	0.0041 (6)	0.0075 (6)
C4B	0.0290 (8)	0.0256 (8)	0.0260 (8)	0.0145 (6)	0.0142 (6)	0.0114 (7)
C5B	0.0191 (7)	0.0306 (8)	0.0294 (8)	0.0096 (6)	0.0115 (6)	0.0168 (7)
C6B	0.0179 (7)	0.0210 (8)	0.0296 (8)	0.0026 (5)	0.0049 (6)	0.0110 (7)
C7B	0.0173 (6)	0.0167 (7)	0.0205 (7)	0.0042 (5)	0.0039 (5)	0.0045 (6)
C8B	0.0139 (6)	0.0190 (7)	0.0202 (7)	0.0035 (5)	0.0050 (5)	0.0081 (6)
C9B	0.0127 (6)	0.0169 (7)	0.0204 (7)	0.0045 (5)	0.0065 (5)	0.0057 (6)
C10B	0.0218 (7)	0.0137 (7)	0.0197 (7)	0.0014 (5)	0.0038 (5)	0.0029 (6)
C11B	0.0212 (7)	0.0131 (7)	0.0215 (7)	0.0016 (5)	0.0038 (5)	0.0057 (6)
C12B	0.0104 (5)	0.0151 (7)	0.0192 (7)	0.0041 (5)	0.0050 (5)	0.0045 (5)
C13B	0.0339 (8)	0.0124 (7)	0.0205 (8)	0.0040 (5)	0.0084 (6)	0.0014 (6)
C14B	0.0350 (8)	0.0136 (7)	0.0234 (8)	0.0053 (6)	0.0106 (6)	0.0074 (6)
C15B	0.0103 (5)	0.0178 (7)	0.0187 (7)	0.0056 (5)	0.0038 (5)	0.0057 (6)
C16B	0.0347 (8)	0.0173 (7)	0.0224 (8)	0.0062 (6)	0.0087 (6)	0.0044 (6)
C17B	0.0414 (9)	0.0232 (8)	0.0267 (9)	0.0108 (7)	0.0110 (7)	0.0122 (7)
C18B	0.0245 (7)	0.0328 (9)	0.0192 (7)	0.0129 (6)	0.0044 (6)	0.0105 (7)
C19B	0.0386 (9)	0.0243 (9)	0.0198 (8)	0.0008 (6)	−0.0006 (7)	0.0023 (7)
C20B	0.0334 (8)	0.0208 (8)	0.0215 (8)	−0.0012 (6)	0.0018 (6)	0.0064 (6)

Table 6: Hydrogen-bond geometry (Å, º).**32**

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O1B	0.88 (1)	2.00 (1)	2.8626 (16)	168 (2)
C6A—H6A···F1Bi	0.95	2.46	3.1954 (16)	134
N1B—H1B···O1Aii	0.88(1)	1.98(1)	2.8107 (16)	157(2)
C8B—H8BB···F1Aiii	0.99	2.39	3.3627 (17)	166

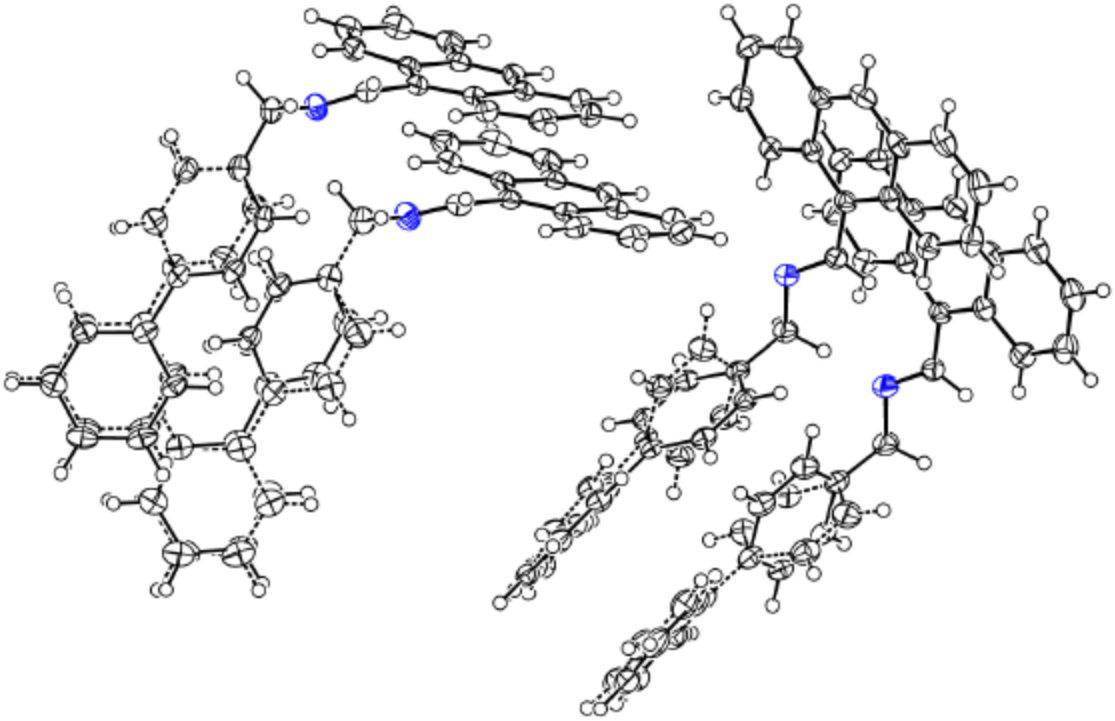

Figure 188: X-ray crystal structure of **27**

Table 1. Experimental details.**27**

	Prosp15mz088_0m
Crystal data	
Chemical formula	$C_{28}H_{21}N$
M_r	371.46
Crystal system, space group	Triclinic, *P*1
Temperature (K)	100
a, *b*, *c* (Å)	10.2208 (6), 19.4895 (12), 21.5312 (12)
α, β, γ (°)	101.121 (3), 103.659 (3), 105.145 (3)
V (Å^3)	3871.9 (4)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.56
Crystal size (mm)	0.12 × 0.10 × 0.07
Data collection	
Diffractometer	Bruker AXS X8 Prospector CCD diffractometer
Absorption correction	Multi-scan Apex2 v2014.11 (Bruker, 2014)
T_{min}, T_{max}	0.692, 0.753
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32198, 13290, 10673
R_{int}	0.029
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.597
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.115, 1.05
No. of reflections	13290
No. of parameters	1276
No. of restraints	624
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.16

Computer programs: Apex2 v2014.11 (Bruker, 2014), *SAINT* V8.34A (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014*/7 (Sheldrick, 2014), SHELXLE Rev714 (Hübschle *et al.*, 2011).

Special details

Refinement

The structure metrically resembles a double volume I-centered monoclinic unit cell with the parameters 24.042, 10.221, 31.707, beta = 96.31 and is twinned by the higher metric pseudosymmetry. The twin matrix "1 0 0, -1 -1 0, -1 0 -1" was applied, the twin ratio after completion of refinement with the final structural model refined to 0.743(1) to 0.257(1).

The structure exhibits pseudo-translation along the a-axis, relating molecules A and B as well as C and D with each other. The translational symmetry is broken by the torsion angle of the phenylene ring of the biphenyl moiety (the benzene unit closer to the imine group). The molecules also exhibit disorder of this phenylene group, with ca. 10% of all phenyl rings being oriented the "other way". i.e. with phenylene groups having the rotation angle of the pseudo-translated molecule.

In case of 1:1 disorder the structure would exhibit a diagonal glide plane, leading to a half the volume monoclinic primitive cell in P21/c with parameters 20.922, 5.110, 18.813, beta = 106.75 (transformation matrix -0.5 0 -1, 0.5 0 0, -0.5 -1 0). The resultant structure would feature one independent molecule with a disordered phenylene ring. The structure can be refined in this simpler setting, with reasonable quality indicators (R1 = 3.76%, residuals +0.23, -0.15, all atom positive definite with meaningful ADPs), but the disorder ratio for the phenyl ring refines to an essentially 1:1 ratio 0.510(1) to 0.490(1)), indicating that the major A and B moieties are averaged with the minor G and H moieties, and the major C and D with the minor E and F moieties.

The major and minor biphenyl units were each restrained to have similar geometries. The phenyl rings of the minor moieties were restrained to be flat and in plane with the first C-atom of the phenylene ring they are bonded to (C20). Carbon atoms of the major and minor moieties of the phenyl ring as well as ipso and para C atoms of the phenylene ring were constrained to have pairwise identical ADPs. The atoms of the phenylene ring of both moieties were restrained to have similar Uij components of their ADPs if closer than 1.7 Angstrom.

Subject to these conditions the disorder ratio refined to 0.906(2) to 0.094(2) for the A/B and E/F moieties, respectively, and 0.911(2) to 0.089(2) for the C/D and G/H units, respectively.

Table 2: Bond Lengths (Å).**27**

C1A—N1A 1.266(3)	C23C—C24C 1.388(4)
C1A—C2A 1.484(3)	C23C—C28C 1.402(4)
C1A—H1A 0.9500	C24C—C25C 1.383(4)
N1A—C16A 1.460(3)	C24C—H24C 0.9500
C2A—C3A 1.418(3)	C25C—C26C 1.388(4)
C2A—C15A 1.419(3)	C25C—H25C 0.9500
C3A—C4A 1.428(3)	C26C—C27C 1.382(4)
C3A—C8A 1.439(3)	C26C—H26C 0.9500
C4A—C5A 1.358(3)	C27C—C28C 1.396(4)
C4A—H4A 0.9500	C27C—H27C 0.9500
C5A—C6A 1.414(3)	C28C—H28C 0.9500
C5A—H5A 0.9500	C1D—N1D 1.270(3)
C6A—C7A 1.350(3)	C1D—C2D 1.473(3)
C6A—H6A 0.9500	C1D—H1D 0.9500
C7A—C8A 1.424(3)	N1D—C16D 1.461(3)
C7A—H7A 0.9500	C2D—C15D 1.420(3)
C8A—C9A 1.394(3)	C2D—C3D 1.428(3)
C9A—C10A 1.384(3)	C3D—C8D 1.424(3)
C9A—H9A 0.9500	C3D—C4D 1.431(3)
C10A—C11A 1.425(3)	C4D—C5D 1.370(3)
C10A—C15A 1.447(3)	C4D—H4D 0.9500
C11A—C12A 1.347(4)	C5D—C6D 1.402(4)
C11A—H11A 0.9500	C5D—H5D 0.9500

C12A—C13A 1.411(4)

C12A—H12A 0.9500

C13A—C14A 1.368(3)

C13A—H13A 0.9500

C14A—C15A 1.431(3)

C14A—H14A 0.9500

C16A—C17E 1.511(3)

C16A—C17A 1.511(3)

C16A—H16A 0.9900

C16A—H16B 0.9900

C17A—C18A 1.375(3)

C17A—C22A 1.398(3)

C18A—C19A 1.389(3)

C18A—H18A 0.9500

C19A—C20A 1.390(5)

C19A—H19A 0.9500

C20A—C21A 1.401(4)

C20A—C23A 1.488(3)

C21A—C22A 1.384(3)

C21A—H21A 0.9500

C22A—H22A 0.9500

C23A—C28A 1.398(4)

C23A—C24A 1.404(3)

C24A—C25A 1.389(4)

C6D—C7D 1.352(3)

C6D—H6D 0.9500

C7D—C8D 1.437(3)

C7D—H7D 0.9500

C8D—C9D 1.395(3)

C9D—C10D 1.392(3)

C9D—H9D 0.9500

C10D—C11D 1.431(3)

C10D—C15D 1.432(3)

C11D—C12D 1.357(3)

C11D—H11D 0.9500

C12D—C13D 1.412(4)

C12D—H12D 0.9500

C13D—C14D 1.369(3)

C13D—H13D 0.9500

C14D—C15D 1.436(3)

C14D—H14D 0.9500

C16D—C17H 1.510(3)

C16D—C17D 1.510(3)

C16D—H16G 0.9900

C16D—H16H 0.9900

C17D—C22D 1.376(3)

C17D—C18D 1.409(3)

C18D—C19D 1.383(3)

C24A—H24A 0.9500

C25A—C26A 1.387(4)

C25A—H25A 0.9500

C26A—C27A 1.389(4)

C26A—H26A 0.9500

C27A—C28A 1.389(4)

C27A—H27A 0.9500

C28A—H28A 0.9500

C1B—N1B 1.270(3)

C1B—C2B 1.485(3)

C1B—H1B 0.9500

N1B—C16B 1.467(3)

C2B—C3B 1.413(3)

C2B—C15B 1.418(3)

C3B—C4B 1.432(3)

C3B—C8B 1.439(3)

C4B—C5B 1.358(3)

C4B—H4B 0.9500

C5B—C6B 1.417(3)

C5B—H5B 0.9500

C6B—C7B 1.347(3)

C6B—H6B 0.9500

C7B—C8B 1.428(3)

C7B—H7B 0.9500

C18D—H18D 0.9500

C19D—C20D 1.398(3)

C19D—H19D 0.9500

C20D—C21D 1.403(3)

C20D—C23D 1.485(3)

C21D—C22D 1.390(3)

C21D—H21D 0.9500

C22D—H22D 0.9500

C23D—C28D 1.392(3)

C23D—C24D 1.398(3)

C24D—C25D 1.386(3)

C24D—H24D 0.9500

C25D—C26D 1.376(4)

C25D—H25D 0.9500

C26D—C27D 1.387(3)

C26D—H26D 0.9500

C27D—C28D 1.388(3)

C27D—H27D 0.9500

C28D—H28D 0.9500

C17E—C18E 1.318(14)

C17E—C22E 1.523(15)

C18E—C19E 1.371(16)

C18E—H18E 0.9500

C19E—C20E 1.379(17)

C8B—C9B 1.393(3)

C9B—C10B 1.385(3)

C9B—H9B 0.9500

C10B—C11B 1.425(3)

C10B—C15B 1.448(3)

C11B—C12B 1.347(3)

C11B—H11B 0.9500

C12B—C13B 1.414(4)

C12B—H12B 0.9500

C13B—C14B 1.368(3)

C13B—H13B 0.9500

C14B—C15B 1.428(3)

C14B—H14B 0.9500

C16B—C17F 1.509(3)

C16B—C17B 1.509(3)

C16B—H16C 0.9900

C16B—H16D 0.9900

C17B—C22B 1.385(3)

C17B—C18B 1.407(3)

C18B—C19B 1.382(3)

C18B—H18B 0.9500

C19B—C20B 1.392(3)

C19B—H19B 0.9500

C20B—C21B 1.410(3)

C19E—H19E 0.9500

C20E—C21E 1.401(18)

C20E—C23E 1.485(15)

C21E—C22E 1.397(17)

C21E—H21E 0.9500

C22E—H22E 0.9500

C23E—C28E 1.398(17)

C23E—C24E 1.403(17)

C24E—C25E 1.372(18)

C24E—H24E 0.9500

C25E—C26E 1.383(18)

C25E—H25E 0.9500

C26E—C27E 1.388(18)

C26E—H26E 0.9500

C27E—C28E 1.389(18)

C27E—H27E 0.9500

C28E—H28E 0.9500

C17F—C18F 1.353(15)

C17F—C22F 1.392(14)

C18F—C19F 1.380(17)

C18F—H18F 0.9500

C19F—C20F 1.397(17)

C19F—H19F 0.9500

C20F—C21F 1.388(17)

C20B—C23B 1.482(3)

C21B—C22B 1.382(3)

C21B—H21B 0.9500

C22B—H22B 0.9500

C23B—C28B 1.397(3)

C23B—C24B 1.399(3)

C24B—C25B 1.385(4)

C24B—H24B 0.9500

C25B—C26B 1.378(4)

C25B—H25B 0.9500

C26B—C27B 1.383(3)

C26B—H26B 0.9500

C27B—C28B 1.385(3)

C27B—H27B 0.9500

C28B—H28B 0.9500

N1C—C1C 1.268(3)

N1C—C16C 1.454(3)

C1C—C2C 1.474(3)

C1C—H1C 0.9500

C2C—C15C 1.425(3)

C2C—C3C 1.428(3)

C3C—C8C 1.425(3)

C3C—C4C 1.427(3)

C4C—C5C 1.371(3)

C20F—C23F 1.486(15)

C21F—C22F 1.378(16)

C21F—H21F 0.9500

C22F—H22F 0.9500

C23F—C24F 1.377(17)

C23F—C28F 1.398(17)

C24F—C25F 1.374(18)

C24F—H24F 0.9500

C25F—C26F 1.378(18)

C25F—H25F 0.9500

C26F—C27F 1.378(18)

C26F—H26F 0.9500

C27F—C28F 1.379(18)

C27F—H27F 0.9500

C28F—H28F 0.9500

C17G—C18G 1.329(15)

C17G—C22G 1.491(15)

C18G—C19G 1.381(16)

C18G—H18G 0.9500

C19G—C20G 1.391(17)

C19G—H19G 0.9500

C20G—C21G 1.405(18)

C20G—C23G 1.495(16)

C21G—C22G 1.394(17)

C4C—H4C 0.9500

C5C—C6C 1.402(4)

C5C—H5C 0.9500

C6C—C7C 1.352(3)

C6C—H6C 0.9500

C7C—C8C 1.435(3)

C7C—H7C 0.9500

C8C—C9C 1.391(3)

C9C—C10C 1.398(3)

C9C—H9C 0.9500

C10C—C15C 1.425(3)

C10C—C11C 1.430(3)

C11C—C12C 1.361(3)

C11C—H11C 0.9500

C12C—C13C 1.406(4)

C12C—H12C 0.9500

C13C—C14C 1.361(3)

C13C—H13C 0.9500

C14C—C15C 1.436(3)

C14C—H14C 0.9500

C16C—C17G 1.505(3)

C16C—C17C 1.505(3)

C16C—H16E 0.9900

C16C—H16F 0.9900

C21G—H21G 0.9500

C22G—H22G 0.9500

C23G—C24G 1.394(17)

C23G—C28G 1.400(17)

C24G—C25G 1.381(18)

C24G—H24G 0.9500

C25G—C26G 1.377(18)

C25G—H25G 0.9500

C26G—C27G 1.372(18)

C26G—H26G 0.9500

C27G—C28G 1.392(18)

C27G—H27G 0.9500

C28G—H28G 0.9500

C17H—C18H 1.364(15)

C17H—C22H 1.367(15)

C18H—C19H 1.380(17)

C18H—H18H 0.9500

C19H—C20H 1.390(17)

C19H—H19H 0.9500

C20H—C21H 1.407(17)

C20H—C23H 1.496(16)

C21H—C22H 1.401(17)

C21H—H21H 0.9500

C22H—H22H 0.9500

C17C—C18C 1.383(3)

C17C—C22C 1.385(3)

C18C—C19C 1.380(3)

C18C—H18C 0.9500

C19C—C20C 1.389(5)

C19C—H19C 0.9500

C20C—C21C 1.396(5)

C20C—C23C 1.492(3)

C21C—C22C 1.385(3)

C21C—H21C 0.9500

C22C—H22C 0.9500

C23H—C24H 1.375(17)

C23H—C28H 1.406(17)

C24H—C25H 1.402(18)

C24H—H24H 0.9500

C25H—C26H 1.385(18)

C25H—H25H 0.9500

C26H—C27H 1.369(18)

C26H—H26H 0.9500

C27H—C28H 1.371(18)

C27H—H27H 0.9500

C28H—H28H 0.9500

Table 3: Bond Angles (deg).**27**

N1A—C1A—C2A	123.99 (19)	C24C—C23C—C20C	121.4 (3)
N1A—C1A—H1A	118.0	C28C—C23C—C20C	120.7 (3)
C2A—C1A—H1A	118.0	C25C—C24C—C23C	121.3 (3)
C1A—N1A—C16A	116.99 (19)	C25C—C24C—H24C	119.4
C3A—C2A—C15A	120.32 (18)	C23C—C24C—H24C	119.4
C3A—C2A—C1A	121.65 (18)	C24C—C25C—C26C	120.5 (3)
C15A—C2A—C1A	118.00 (18)	C24C—C25C—H25C	119.7
C2A—C3A—C4A	123.96 (18)	C26C—C25C—H25C	119.7
C2A—C3A—C8A	119.16 (18)	C27C—C26C—C25C	119.2 (3)
C4A—C3A—C8A	116.82 (18)	C27C—C26C—H26C	120.4
C5A—C4A—C3A	121.5 (2)	C25C—C26C—H26C	120.4
C5A—C4A—H4A	119.2	C26C—C27C—C28C	120.3 (3)
C3A—C4A—H4A	119.2	C26C—C27C—H27C	119.8
C4A—C5A—C6A	121.2 (2)	C28C—C27C—H27C	119.8
C4A—C5A—H5A	119.4	C27C—C28C—C23C	120.7 (3)
C6A—C5A—H5A	119.4	C27C—C28C—H28C	119.6
C7A—C6A—C5A	119.5 (2)	C23C—C28C—H28C	119.6
C7A—C6A—H6A	120.2	N1D—C1D—C2D	123.66 (18)
C5A—C6A—H6A	120.2	N1D—C1D—H1D	118.2
C6A—C7A—C8A	121.4 (2)	C2D—C1D—H1D	118.2
C6A—C7A—H7A	119.3	C1D—N1D—C16D	116.52 (17)
C8A—C7A—H7A	119.3	C15D—C2D—C3D	119.37 (18)
C9A—C8A—C7A	120.9 (2)	C15D—C2D—C1D	118.77 (17)
C9A—C8A—C3A	119.60 (19)	C3D—C2D—C1D	121.83 (17)
C7A—C8A—C3A	119.43 (19)	C8D—C3D—C2D	119.73 (18)
C10A—C9A—C8A	122.1 (2)	C8D—C3D—C4D	117.30 (18)
C10A—C9A—H9A	118.9	C2D—C3D—C4D	122.92 (19)
C8A—C9A—H9A	118.9	C5D—C4D—C3D	120.9 (2)
C9A—C10A—C11A	121.2 (2)	C5D—C4D—H4D	119.5
C9A—C10A—C15A	119.43 (19)	C3D—C4D—H4D	119.5
C11A—C10A—C15A	119.3 (2)	C4D—C5D—C6D	121.4 (2)
C12A—C11A—C10A	121.8 (2)	C4D—C5D—H5D	119.3
C12A—C11A—H11A	119.1	C6D—C5D—H5D	119.3
C10A—C11A—H11A	119.1	C7D—C6D—C5D	119.76 (19)
C11A—C12A—C13A	119.8 (2)	C7D—C6D—H6D	120.1
C11A—C12A—H12A	120.1	C5D—C6D—H6D	120.1
C13A—C12A—H12A	120.1	C6D—C7D—C8D	121.0 (2)
C14A—C13A—C12A	121.0 (2)	C6D—C7D—H7D	119.5
C14A—C13A—H13A	119.5	C8D—C7D—H7D	119.5
C12A—C13A—H13A	119.5	C9D—C8D—C3D	119.92 (18)
C13A—C14A—C15A	121.6 (2)	C9D—C8D—C7D	120.5 (2)
C13A—C14A—H14A	119.2	C3D—C8D—C7D	119.52 (19)
C15A—C14A—H14A	119.2	C10D—C9D—C8D	121.3 (2)

C2A—C15A—C14A	124.31 (19)	C10D—C9D—H9D	119.4
C2A—C15A—C10A	119.19 (19)	C8D—C9D—H9D	119.4
C14A—C15A—C10A	116.50 (19)	C9D—C10D—C11D	120.4 (2)
N1A—C16A—C17E	111.43 (18)	C9D—C10D—C15D	119.98 (19)
N1A—C16A—C17A	111.43 (18)	C11D—C10D—C15D	119.6 (2)
N1A—C16A—H16A	109.3	C12D—C11D—C10D	121.0 (2)
C17A—C16A—H16A	109.3	C12D—C11D—H11D	119.5
N1A—C16A—H16B	109.3	C10D—C11D—H11D	119.5
C17A—C16A—H16B	109.3	C11D—C12D—C13D	119.8 (2)
H16A—C16A—H16B	108.0	C11D—C12D—H12D	120.1
C18A—C17A—C22A	118.5 (2)	C13D—C12D—H12D	120.1
C18A—C17A—C16A	122.54 (19)	C14D—C13D—C12D	121.2 (2)
C22A—C17A—C16A	118.95 (19)	C14D—C13D—H13D	119.4
C17A—C18A—C19A	121.3 (2)	C12D—C13D—H13D	119.4
C17A—C18A—H18A	119.4	C13D—C14D—C15D	121.0 (2)
C19A—C18A—H18A	119.4	C13D—C14D—H14D	119.5
C18A—C19A—C20A	121.1 (2)	C15D—C14D—H14D	119.5
C18A—C19A—H19A	119.4	C2D—C15D—C10D	119.54 (18)
C20A—C19A—H19A	119.4	C2D—C15D—C14D	123.22 (19)
C19A—C20A—C21A	117.3 (2)	C10D—C15D—C14D	117.24 (19)
C19A—C20A—C23A	121.7 (3)	N1D—C16D—C17H	111.48 (17)
C21A—C20A—C23A	120.9 (3)	N1D—C16D—C17D	111.48 (17)
C22A—C21A—C20A	121.6 (2)	N1D—C16D—H16G	109.3
C22A—C21A—H21A	119.2	C17D—C16D—H16G	109.3
C20A—C21A—H21A	119.2	N1D—C16D—H16H	109.3
C21A—C22A—C17A	120.2 (2)	C17D—C16D—H16H	109.3
C21A—C22A—H22A	119.9	H16G—C16D—H16H	108.0
C17A—C22A—H22A	119.9	C22D—C17D—C18D	117.39 (19)
C28A—C23A—C24A	117.9 (3)	C22D—C17D—C16D	122.41 (18)
C28A—C23A—C20A	120.9 (3)	C18D—C17D—C16D	120.20 (19)
C24A—C23A—C20A	121.0 (3)	C19D—C18D—C17D	120.95 (19)
C25A—C24A—C23A	120.8 (3)	C19D—C18D—H18D	119.5
C25A—C24A—H24A	119.6	C17D—C18D—H18D	119.5
C23A—C24A—H24A	119.6	C18D—C19D—C20D	121.49 (18)
C26A—C25A—C24A	120.7 (3)	C18D—C19D—H19D	119.3
C26A—C25A—H25A	119.6	C20D—C19D—H19D	119.3
C24A—C25A—H25A	119.6	C19D—C20D—C21D	117.33 (19)
C25A—C26A—C27A	119.0 (3)	C19D—C20D—C23D	121.28 (18)
C25A—C26A—H26A	120.5	C21D—C20D—C23D	121.37 (19)
C27A—C26A—H26A	120.5	C22D—C21D—C20D	120.64 (19)
C26A—C27A—C28A	120.7 (3)	C22D—C21D—H21D	119.7
C26A—C27A—H27A	119.7	C20D—C21D—H21D	119.7
C28A—C27A—H27A	119.7	C17D—C22D—C21D	122.15 (18)
C27A—C28A—C23A	120.9 (3)	C17D—C22D—H22D	118.9
C27A—C28A—H28A	119.5	C21D—C22D—H22D	118.9

C23A—C28A—H28A	119.5	C28D—C23D—C24D	117.7 (2)
N1B—C1B—C2B	123.79 (18)	C28D—C23D—C20D	121.0 (2)
N1B—C1B—H1B	118.1	C24D—C23D—C20D	121.3 (2)
C2B—C1B—H1B	118.1	C25D—C24D—C23D	121.1 (2)
C1B—N1B—C16B	116.48 (17)	C25D—C24D—H24D	119.4
C3B—C2B—C15B	120.30 (17)	C23D—C24D—H24D	119.4
C3B—C2B—C1B	121.59 (17)	C26D—C25D—C24D	120.3 (2)
C15B—C2B—C1B	118.05 (17)	C26D—C25D—H25D	119.8
C2B—C3B—C4B	123.89 (18)	C24D—C25D—H25D	119.8
C2B—C3B—C8B	119.44 (18)	C25D—C26D—C27D	119.6 (2)
C4B—C3B—C8B	116.62 (18)	C25D—C26D—H26D	120.2
C5B—C4B—C3B	121.72 (19)	C27D—C26D—H26D	120.2
C5B—C4B—H4B	119.1	C26D—C27D—C28D	120.1 (2)
C3B—C4B—H4B	119.1	C26D—C27D—H27D	119.9
C4B—C5B—C6B	121.1 (2)	C28D—C27D—H27D	119.9
C4B—C5B—H5B	119.4	C27D—C28D—C23D	121.1 (2)
C6B—C5B—H5B	119.4	C27D—C28D—H28D	119.5
C7B—C6B—C5B	119.4 (2)	C23D—C28D—H28D	119.5
C7B—C6B—H6B	120.3	C18E—C17E—C16A	131.1 (7)
C5B—C6B—H6B	120.3	C18E—C17E—C22E	110.7 (10)
C6B—C7B—C8B	121.7 (2)	C16A—C17E—C22E	118.1 (8)
C6B—C7B—H7B	119.2	C17E—C18E—C19E	126.0 (14)
C8B—C7B—H7B	119.2	C17E—C18E—H18E	117.0
C9B—C8B—C7B	121.11 (19)	C19E—C18E—H18E	117.0
C9B—C8B—C3B	119.52 (18)	C18E—C19E—C20E	124.0 (16)
C7B—C8B—C3B	119.35 (19)	C18E—C19E—H19E	118.0
C10B—C9B—C8B	121.90 (19)	C20E—C19E—H19E	118.0
C10B—C9B—H9B	119.0	C19E—C20E—C21E	116.6 (15)
C8B—C9B—H9B	119.0	C19E—C20E—C23E	121.4 (17)
C9B—C10B—C11B	121.20 (19)	C21E—C20E—C23E	121.8 (16)
C9B—C10B—C15B	119.62 (18)	C22E—C21E—C20E	118.2 (16)
C11B—C10B—C15B	119.2 (2)	C22E—C21E—H21E	120.9
C12B—C11B—C10B	121.7 (2)	C20E—C21E—H21E	120.9
C12B—C11B—H11B	119.2	C21E—C22E—C17E	124.2 (15)
C10B—C11B—H11B	119.2	C21E—C22E—H22E	117.9
C11B—C12B—C13B	120.0 (2)	C17E—C22E—H22E	117.9
C11B—C12B—H12B	120.0	C28E—C23E—C24E	118.2 (19)
C13B—C12B—H12B	120.0	C28E—C23E—C20E	121 (2)
C14B—C13B—C12B	120.7 (2)	C24E—C23E—C20E	121 (2)
C14B—C13B—H13B	119.7	C25E—C24E—C23E	120 (2)
C12B—C13B—H13B	119.7	C25E—C24E—H24E	119.9
C13B—C14B—C15B	121.7 (2)	C23E—C24E—H24E	119.9
C13B—C14B—H14B	119.1	C24E—C25E—C26E	122 (2)
C15B—C14B—H14B	119.1	C24E—C25E—H25E	119.1
C2B—C15B—C14B	124.24 (18)	C26E—C25E—H25E	119.1

C2B—C15B—C10B	119.03 (18)	C25E—C26E—C27E	119 (2)
C14B—C15B—C10B	116.73 (18)	C25E—C26E—H26E	120.7
N1B—C16B—C17F	110.75 (17)	C27E—C26E—H26E	120.7
N1B—C16B—C17B	110.75 (17)	C26E—C27E—C28E	120 (2)
N1B—C16B—H16C	109.5	C26E—C27E—H27E	119.8
C17B—C16B—H16C	109.5	C28E—C27E—H27E	119.8
N1B—C16B—H16D	109.5	C27E—C28E—C23E	121 (2)
C17B—C16B—H16D	109.5	C27E—C28E—H28E	119.6
H16C—C16B—H16D	108.1	C23E—C28E—H28E	119.6
C22B—C17B—C18B	117.22 (19)	C18F—C17F—C22F	122.8 (11)
C22B—C17B—C16B	122.18 (17)	C18F—C17F—C16B	119.4 (8)
C18B—C17B—C16B	120.59 (18)	C22F—C17F—C16B	115.9 (7)
C19B—C18B—C17B	121.26 (18)	C17F—C18F—C19F	117.6 (14)
C19B—C18B—H18B	119.4	C17F—C18F—H18F	121.2
C17B—C18B—H18B	119.4	C19F—C18F—H18F	121.2
C18B—C19B—C20B	121.40 (18)	C18F—C19F—C20F	119.5 (16)
C18B—C19B—H19B	119.3	C18F—C19F—H19F	120.3
C20B—C19B—H19B	119.3	C20F—C19F—H19F	120.3
C19B—C20B—C21B	117.3 (2)	C21F—C20F—C19F	121.8 (15)
C19B—C20B—C23B	121.43 (18)	C21F—C20F—C23F	118.8 (14)
C21B—C20B—C23B	121.2 (2)	C19F—C20F—C23F	118.7 (15)
C22B—C21B—C20B	120.9 (2)	C22F—C21F—C20F	117.6 (15)
C22B—C21B—H21B	119.6	C22F—C21F—H21F	121.2
C20B—C21B—H21B	119.6	C20F—C21F—H21F	121.2
C21B—C22B—C17B	121.90 (17)	C21F—C22F—C17F	118.7 (14)
C21B—C22B—H22B	119.1	C21F—C22F—H22F	120.6
C17B—C22B—H22B	119.1	C17F—C22F—H22F	120.6
C28B—C23B—C24B	117.3 (2)	C24F—C23F—C28F	119.3 (18)
C28B—C23B—C20B	120.8 (2)	C24F—C23F—C20F	121.6 (18)
C24B—C23B—C20B	121.8 (2)	C28F—C23F—C20F	119.2 (19)
C25B—C24B—C23B	121.1 (2)	C25F—C24F—C23F	120.6 (19)
C25B—C24B—H24B	119.4	C25F—C24F—H24F	119.7
C23B—C24B—H24B	119.4	C23F—C24F—H24F	119.7
C26B—C25B—C24B	120.5 (2)	C24F—C25F—C26F	120 (2)
C26B—C25B—H25B	119.7	C24F—C25F—H25F	120.0
C24B—C25B—H25B	119.7	C26F—C25F—H25F	120.0
C25B—C26B—C27B	119.5 (3)	C27F—C26F—C25F	120 (2)
C25B—C26B—H26B	120.3	C27F—C26F—H26F	119.8
C27B—C26B—H26B	120.3	C25F—C26F—H26F	119.8
C26B—C27B—C28B	120.2 (3)	C26F—C27F—C28F	120 (2)
C26B—C27B—H27B	119.9	C26F—C27F—H27F	120.1
C28B—C27B—H27B	119.9	C28F—C27F—H27F	120.1
C27B—C28B—C23B	121.4 (3)	C27F—C28F—C23F	120 (2)
C27B—C28B—H28B	119.3	C27F—C28F—H28F	120.0
C23B—C28B—H28B	119.3	C23F—C28F—H28F	120.0

C1C—N1C—C16C	117.04 (18)	C18G—C17G—C22G	112.0 (10)
N1C—C1C—C2C	124.38 (18)	C18G—C17G—C16C	129.5 (8)
N1C—C1C—H1C	117.8	C22G—C17G—C16C	118.1 (8)
C2C—C1C—H1C	117.8	C17G—C18G—C19G	126.8 (15)
C15C—C2C—C3C	119.33 (18)	C17G—C18G—H18G	116.6
C15C—C2C—C1C	118.40 (17)	C19G—C18G—H18G	116.6
C3C—C2C—C1C	122.27 (18)	C18G—C19G—C20G	120.5 (16)
C8C—C3C—C4C	117.00 (18)	C18G—C19G—H19G	119.8
C8C—C3C—C2C	119.67 (18)	C20G—C19G—H19G	119.8
C4C—C3C—C2C	123.28 (19)	C19G—C20G—C21G	117.7 (17)
C5C—C4C—C3C	121.2 (2)	C19G—C20G—C23G	116.6 (16)
C5C—C4C—H4C	119.4	C21G—C20G—C23G	123.9 (16)
C3C—C4C—H4C	119.4	C22G—C21G—C20G	118.9 (16)
C4C—C5C—C6C	121.4 (2)	C22G—C21G—H21G	120.6
C4C—C5C—H5C	119.3	C20G—C21G—H21G	120.6
C6C—C5C—H5C	119.3	C21G—C22G—C17G	123.1 (15)
C7C—C6C—C5C	119.55 (19)	C21G—C22G—H22G	118.5
C7C—C6C—H6C	120.2	C17G—C22G—H22G	118.5
C5C—C6C—H6C	120.2	C24G—C23G—C28G	117.9 (19)
C6C—C7C—C8C	121.2 (2)	C24G—C23G—C20G	119 (2)
C6C—C7C—H7C	119.4	C28G—C23G—C20G	123 (2)
C8C—C7C—H7C	119.4	C25G—C24G—C23G	122 (2)
C9C—C8C—C3C	119.97 (18)	C25G—C24G—H24G	119.0
C9C—C8C—C7C	120.4 (2)	C23G—C24G—H24G	119.0
C3C—C8C—C7C	119.63 (19)	C26G—C25G—C24G	120 (2)
C8C—C9C—C10C	121.3 (2)	C26G—C25G—H25G	120.0
C8C—C9C—H9C	119.3	C24G—C25G—H25G	120.0
C10C—C9C—H9C	119.3	C27G—C26G—C25G	119 (2)
C9C—C10C—C15C	119.90 (19)	C27G—C26G—H26G	120.7
C9C—C10C—C11C	120.3 (2)	C25G—C26G—H26G	120.7
C15C—C10C—C11C	119.82 (19)	C26G—C27G—C28G	123 (2)
C12C—C11C—C10C	121.1 (2)	C26G—C27G—H27G	118.7
C12C—C11C—H11C	119.5	C28G—C27G—H27G	118.7
C10C—C11C—H11C	119.5	C27G—C28G—C23G	119 (2)
C11C—C12C—C13C	119.6 (2)	C27G—C28G—H28G	120.6
C11C—C12C—H12C	120.2	C23G—C28G—H28G	120.6
C13C—C12C—H12C	120.2	C18H—C17H—C22H	124.2 (11)
C14C—C13C—C12C	121.1 (2)	C18H—C17H—C16D	117.6 (8)
C14C—C13C—H13C	119.4	C22H—C17H—C16D	115.1 (8)
C12C—C13C—H13C	119.4	C17H—C18H—C19H	114.4 (15)
C13C—C14C—C15C	121.7 (2)	C17H—C18H—H18H	122.8
C13C—C14C—H14C	119.2	C19H—C18H—H18H	122.8
C15C—C14C—H14C	119.2	C18H—C19H—C20H	122.6 (16)
C2C—C15C—C10C	119.67 (18)	C18H—C19H—H19H	118.7
C2C—C15C—C14C	123.64 (19)	C20H—C19H—H19H	118.7

C10C—C15C—C14C	116.69 (19)	C19H—C20H—C21H	120.3 (15)
N1C—C16C—C17G	111.79 (18)	C19H—C20H—C23H	122.1 (16)
N1C—C16C—C17C	111.79 (18)	C21H—C20H—C23H	116.0 (14)
N1C—C16C—H16E	109.3	C22H—C21H—C20H	116.5 (15)
C17C—C16C—H16E	109.3	C22H—C21H—H21H	121.8
N1C—C16C—H16F	109.3	C20H—C21H—H21H	121.8
C17C—C16C—H16F	109.3	C17H—C22H—C21H	119.0 (15)
H16E—C16C—H16F	107.9	C17H—C22H—H22H	120.5
C18C—C17C—C22C	118.3 (2)	C21H—C22H—H22H	120.5
C18C—C17C—C16C	119.67 (19)	C24H—C23H—C28H	119.1 (17)
C22C—C17C—C16C	122.0 (2)	C24H—C23H—C20H	125.0 (18)
C19C—C18C—C17C	120.8 (2)	C28H—C23H—C20H	116.0 (18)
C19C—C18C—H18C	119.6	C23H—C24H—C25H	120.6 (19)
C17C—C18C—H18C	119.6	C23H—C24H—H24H	119.7
C18C—C19C—C20C	121.8 (2)	C25H—C24H—H24H	119.7
C18C—C19C—H19C	119.1	C26H—C25H—C24H	119 (2)
C20C—C19C—H19C	119.1	C26H—C25H—H25H	120.5
C19C—C20C—C21C	117.0 (2)	C24H—C25H—H25H	120.5
C19C—C20C—C23C	121.3 (3)	C27H—C26H—C25H	120 (2)
C21C—C20C—C23C	121.6 (3)	C27H—C26H—H26H	119.8
C22C—C21C—C20C	121.2 (2)	C25H—C26H—H26H	119.8
C22C—C21C—H21C	119.4	C26H—C27H—C28H	121 (2)
C20C—C21C—H21C	119.4	C26H—C27H—H27H	119.6
C17C—C22C—C21C	120.9 (2)	C28H—C27H—H27H	119.6
C17C—C22C—H22C	119.6	C27H—C28H—C23H	120 (2)
C21C—C22C—H22C	119.6	C27H—C28H—H28H	120.0
C24C—C23C—C28C	117.9 (3)	C23H—C28H—H28H	120.0
C2A—C1A—N1A—C16A	179.31 (18)	C18C—C17C—C22C—C21C	2.7 (3)
N1A—C1A—C2A—C3A	28.2 (3)	C16C—C17C—C22C—C21C	−177.5 (2)
N1A—C1A—C2A—C15A	−153.3 (2)	C20C—C21C—C22C—C17C	−0.8 (4)
C15A—C2A—C3A—C4A	−172.90 (19)	C19C—C20C—C23C—C24C	−30.1 (4)
C1A—C2A—C3A—C4A	5.6 (3)	C21C—C20C—C23C—C24C	152.8 (4)
C15A—C2A—C3A—C8A	4.2 (3)	C19C—C20C—C23C—C28C	148.0 (3)
C1A—C2A—C3A—C8A	−177.34 (18)	C21C—C20C—C23C—C28C	−29.1 (4)
C2A—C3A—C4A—C5A	177.4 (2)	C28C—C23C—C24C—C25C	−1.4 (3)
C8A—C3A—C4A—C5A	0.2 (3)	C20C—C23C—C24C—C25C	176.7 (2)

C3A—C4A—C5A—C6A	1.9 (3)	C23C—C24C—C25C—C26C	0.9 (3)
C4A—C5A—C6A—C7A	−1.8 (4)	C24C—C25C—C26C—C27C	0.1 (4)
C5A—C6A—C7A—C8A	−0.5 (4)	C25C—C26C—C27C—C28C	−0.4 (4)
C6A—C7A—C8A—C9A	−175.8 (2)	C26C—C27C—C28C—C23C	−0.1 (4)
C6A—C7A—C8A—C3A	2.6 (3)	C24C—C23C—C28C—C27C	1.0 (3)
C2A—C3A—C8A—C9A	−1.3 (3)	C20C—C23C—C28C—C27C	−177.1 (2)
C4A—C3A—C8A—C9A	176.05 (19)	C2D—C1D—N1D—C16D	178.82 (17)
C2A—C3A—C8A—C7A	−179.71 (19)	N1D—C1D—C2D—C15D	148.20 (19)
C4A—C3A—C8A—C7A	−2.4 (3)	N1D—C1D—C2D—C3D	−34.0 (3)
C7A—C8A—C9A—C10A	176.2 (2)	C15D—C2D—C3D—C8D	−4.3 (3)
C3A—C8A—C9A—C10A	−2.2 (3)	C1D—C2D—C3D—C8D	177.91 (17)
C8A—C9A—C10A—C11A	−175.9 (2)	C15D—C2D—C3D—C4D	172.90 (18)
C8A—C9A—C10A—C15A	2.7 (3)	C1D—C2D—C3D—C4D	−4.9 (3)
C9A—C10A—C11A—C12A	177.9 (2)	C8D—C3D—C4D—C5D	0.1 (3)
C15A—C10A—C11A—C12A	−0.7 (3)	C2D—C3D—C4D—C5D	−177.17 (18)
C10A—C11A—C12A—C13A	1.2 (4)	C3D—C4D—C5D—C6D	−2.1 (3)
C11A—C12A—C13A—C14A	0.2 (4)	C4D—C5D—C6D—C7D	1.9 (3)
C12A—C13A—C14A—C15A	−2.2 (3)	C5D—C6D—C7D—C8D	0.2 (3)
C3A—C2A—C15A—C14A	176.40 (19)	C2D—C3D—C8D—C9D	0.9 (3)
C1A—C2A—C15A—C14A	−2.1 (3)	C4D—C3D—C8D—C9D	−176.44 (18)
C3A—C2A—C15A—C10A	−3.7 (3)	C2D—C3D—C8D—C7D	179.26 (17)
C1A—C2A—C15A—C10A	177.75 (18)	C4D—C3D—C8D—C7D	1.9 (3)
C13A—C14A—C15A—C2A	−177.5 (2)	C6D—C7D—C8D—C9D	176.3 (2)

C13A—C14A—C15A—C10A	2.7 (3)	C6D—C7D—C8D—C3D	−2.1 (3)
C9A—C10A—C15A—C2A	0.3 (3)	C3D—C8D—C9D—C10D	2.6 (3)
C11A—C10A—C15A—C2A	178.89 (19)	C7D—C8D—C9D—C10D	−175.72 (18)
C9A—C10A—C15A—C14A	−179.80 (19)	C8D—C9D—C10D—C11D	176.38 (19)
C11A—C10A—C15A—C14A	−1.2 (3)	C8D—C9D—C10D—C15D	−2.7 (3)
C1A—N1A—C16A—C17E	−126.9 (2)	C9D—C10D—C11D—C12D	−177.4 (2)
C1A—N1A—C16A—C17A	−126.9 (2)	C15D—C10D—C11D—C12D	1.6 (3)
N1A—C16A—C17A—C18A	10.4 (3)	C10D—C11D—C12D—C13D	−1.6 (3)
N1A—C16A—C17A—C22A	−170.24 (18)	C11D—C12D—C13D—C14D	−0.1 (3)
C22A—C17A—C18A—C19A	−2.0 (3)	C12D—C13D—C14D—C15D	1.8 (3)
C16A—C17A—C18A—C19A	177.3 (2)	C3D—C2D—C15D—C10D	4.2 (3)
C17A—C18A—C19A—C20A	0.6 (4)	C1D—C2D—C15D—C10D	−177.92 (17)
C18A—C19A—C20A—C21A	0.7 (3)	C3D—C2D—C15D—C14D	−175.64 (17)
C18A—C19A—C20A—C23A	−175.5 (2)	C1D—C2D—C15D—C14D	2.2 (3)
C19A—C20A—C21A—C22A	−0.6 (3)	C9D—C10D—C15D—C2D	−0.8 (3)
C23A—C20A—C21A—C22A	175.6 (2)	C11D—C10D—C15D—C2D	−179.86 (18)
C20A—C21A—C22A—C17A	−0.8 (3)	C9D—C10D—C15D—C14D	179.09 (17)
C18A—C17A—C22A—C21A	2.1 (3)	C11D—C10D—C15D—C14D	0.0 (3)
C16A—C17A—C22A—C21A	−177.30 (19)	C13D—C14D—C15D—C2D	178.17 (19)
C19A—C20A—C23A—C28A	−158.9 (4)	C13D—C14D—C15D—C10D	−1.7 (3)
C21A—C20A—C23A—C28A	25.0 (4)	C1D—N1D—C16D—C17H	131.00 (18)
C19A—C20A—C23A—C24A	25.4 (3)	C1D—N1D—C16D—C17D	131.00 (18)
C21A—C20A—C23A—C24A	−150.7 (2)	N1D—C16D—C17D—C22D	104.4 (2)

C28A—C23A—C24A—C25A	−0.6 (4)	N1D—C16D—C17D—C18D	−76.3 (2)
C20A—C23A—C24A—C25A	175.3 (2)	C22D—C17D—C18D—C19D	2.3 (3)
C23A—C24A—C25A—C26A	−1.3 (4)	C16D—C17D—C18D—C19D	−177.09 (19)
C24A—C25A—C26A—C27A	2.4 (4)	C17D—C18D—C19D—C20D	−0.7 (3)
C25A—C26A—C27A—C28A	−1.5 (4)	C18D—C19D—C20D—C21D	−1.2 (3)
C26A—C27A—C28A—C23A	−0.4 (4)	C18D—C19D—C20D—C23D	177.6 (2)
C24A—C23A—C28A—C27A	1.4 (4)	C19D—C20D—C21D—C22D	1.4 (3)
C20A—C23A—C28A—C27A	−174.5 (2)	C23D—C20D—C21D—C22D	−177.3 (2)
C2B—C1B—N1B—C16B	−178.63 (17)	C18D—C17D—C22D—C21D	−2.0 (3)
N1B—C1B—C2B—C3B	32.3 (3)	C16D—C17D—C22D—C21D	177.29 (19)
N1B—C1B—C2B—C15B	−150.28 (19)	C20D—C21D—C22D—C17D	0.2 (3)
C15B—C2B—C3B—C4B	−172.97 (19)	C19D—C20D—C23D—C28D	−145.8 (2)
C1B—C2B—C3B—C4B	4.4 (3)	C21D—C20D—C23D—C28D	32.9 (3)
C15B—C2B—C3B—C8B	4.4 (3)	C19D—C20D—C23D—C24D	32.9 (3)
C1B—C2B—C3B—C8B	−178.25 (18)	C21D—C20D—C23D—C24D	−148.5 (2)
C2B—C3B—C4B—C5B	177.4 (2)	C28D—C23D—C24D—C25D	−0.5 (3)
C8B—C3B—C4B—C5B	0.0 (3)	C20D—C23D—C24D—C25D	−179.19 (19)
C3B—C4B—C5B—C6B	1.9 (3)	C23D—C24D—C25D—C26D	1.2 (3)
C4B—C5B—C6B—C7B	−1.7 (4)	C24D—C25D—C26D—C27D	−0.7 (3)
C5B—C6B—C7B—C8B	−0.4 (3)	C25D—C26D—C27D—C28D	−0.5 (3)
C6B—C7B—C8B—C9B	−176.2 (2)	C26D—C27D—C28D—C23D	1.2 (3)
C6B—C7B—C8B—C3B	2.3 (3)	C24D—C23D—C28D—C27D	−0.7 (3)
C2B—C3B—C8B—C9B	−1.0 (3)	C20D—C23D—C28D—C27D	177.97 (19)

C4B—C3B—C8B—C9B	176.52 (19)	N1A—C16A—C17E—C18E	−105.2 (13)
C2B—C3B—C8B—C7B	−179.59 (19)	N1A—C16A—C17E—C22E	78.2 (11)
C4B—C3B—C8B—C7B	−2.0 (3)	C16A—C17E—C18E—C19E	−175.2 (15)
C7B—C8B—C9B—C10B	175.98 (19)	C22E—C17E—C18E—C19E	2 (3)
C3B—C8B—C9B—C10B	−2.6 (3)	C17E—C18E—C19E—C20E	−1 (4)
C8B—C9B—C10B—C11B	−176.2 (2)	C18E—C19E—C20E—C21E	3 (5)
C8B—C9B—C10B—C15B	2.7 (3)	C18E—C19E—C20E—C23E	176 (2)
C9B—C10B—C11B—C12B	178.7 (2)	C19E—C20E—C21E—C22E	−5 (5)
C15B—C10B—C11B—C12B	−0.2 (3)	C23E—C20E—C21E—C22E	−179 (2)
C10B—C11B—C12B—C13B	0.4 (3)	C20E—C21E—C22E—C17E	7 (4)
C11B—C12B—C13B—C14B	0.7 (3)	C18E—C17E—C22E—C21E	−5 (3)
C12B—C13B—C14B—C15B	−1.9 (3)	C16A—C17E—C22E—C21E	172.4 (18)
C3B—C2B—C15B—C14B	176.35 (18)	C19E—C20E—C23E—C28E	−30 (3)
C1B—C2B—C15B—C14B	−1.1 (3)	C21E—C20E—C23E—C28E	143 (4)
C3B—C2B—C15B—C10B	−4.2 (3)	C19E—C20E—C23E—C24E	150 (3)
C1B—C2B—C15B—C10B	178.33 (17)	C21E—C20E—C23E—C24E	−37 (4)
C13B—C14B—C15B—C2B	−178.6 (2)	C28E—C23E—C24E—C25E	0.0 (4)
C13B—C14B—C15B—C10B	2.0 (3)	C20E—C23E—C24E—C25E	180.0 (3)
C9B—C10B—C15B—C2B	0.7 (3)	C23E—C24E—C25E—C26E	0.0 (6)
C11B—C10B—C15B—C2B	179.64 (18)	C24E—C25E—C26E—C27E	0.0 (8)
C9B—C10B—C15B—C14B	−179.84 (18)	C25E—C26E—C27E—C28E	0.0 (9)
C11B—C10B—C15B—C14B	−0.9 (3)	C26E—C27E—C28E—C23E	0.0 (8)
C1B—N1B—C16B—C17F	−130.16 (19)	C24E—C23E—C28E—C27E	0.0 (6)

C1B—N1B—C16B—C17B	−130.16 (19)	C20E—C23E—C28E—C27E	180.0 (4)
N1B—C16B—C17B—C22B	−100.9 (2)	N1B—C16B—C17F—C18F	2.7 (11)
N1B—C16B—C17B—C18B	80.5 (2)	N1B—C16B—C17F—C22F	−161.9 (10)
C22B—C17B—C18B—C19B	−1.8 (3)	C22F—C17F—C18F—C19F	−15 (3)
C16B—C17B—C18B—C19B	176.93 (19)	C16B—C17F—C18F—C19F	−178.7 (15)
C17B—C18B—C19B—C20B	1.2 (3)	C17F—C18F—C19F—C20F	9 (3)
C18B—C19B—C20B—C21B	0.2 (3)	C18F—C19F—C20F—C21F	−4 (4)
C18B—C19B—C20B—C23B	−178.3 (2)	C18F—C19F—C20F—C23F	−174.8 (19)
C19B—C20B—C21B—C22B	−1.0 (3)	C19F—C20F—C21F—C22F	6 (4)
C23B—C20B—C21B—C22B	177.4 (2)	C23F—C20F—C21F—C22F	176.0 (18)
C20B—C21B—C22B—C17B	0.4 (3)	C20F—C21F—C22F—C17F	−11 (3)
C18B—C17B—C22B—C21B	0.9 (3)	C18F—C17F—C22F—C21F	17 (3)
C16B—C17B—C22B—C21B	−177.74 (19)	C16B—C17F—C22F—C21F	−179.3 (15)
C19B—C20B—C23B—C28B	147.6 (3)	C21F—C20F—C23F—C24F	−142 (3)
C21B—C20B—C23B—C28B	−30.8 (3)	C19F—C20F—C23F—C24F	29 (3)
C19B—C20B—C23B—C24B	−31.9 (3)	C21F—C20F—C23F—C28F	38 (3)
C21B—C20B—C23B—C24B	149.7 (2)	C19F—C20F—C23F—C28F	−151 (3)
C28B—C23B—C24B—C25B	1.4 (3)	C28F—C23F—C24F—C25F	0.0 (4)
C20B—C23B—C24B—C25B	−179.1 (2)	C20F—C23F—C24F—C25F	180.0 (3)
C23B—C24B—C25B—C26B	−1.2 (4)	C23F—C24F—C25F—C26F	0.1 (6)
C24B—C25B—C26B—C27B	0.0 (4)	C24F—C25F—C26F—C27F	−0.2 (9)
C25B—C26B—C27B—C28B	0.9 (4)	C25F—C26F—C27F—C28F	0.1 (9)
C26B—C27B—C28B—C23B	−0.7 (3)	C26F—C27F—C28F—C23F	0.0 (9)

C24B—C23B—C28B—C27B	−0.5 (3)	C24F—C23F—C28F—C27F	−0.1 (6)
C20B—C23B—C28B—C27B	−179.9 (2)	C20F—C23F—C28F—C27F	179.9 (4)
C16C—N1C—C1C—C2C	−179.54 (18)	N1C—C16C—C17G—C18G	102.5 (13)
N1C—C1C—C2C—C15C	154.58 (19)	N1C—C16C—C17G—C22G	−84.1 (11)
N1C—C1C—C2C—C3C	−26.5 (3)	C22G—C17G—C18G—C19G	5 (3)
C15C—C2C—C3C—C8C	−3.8 (3)	C16C—C17G—C18G—C19G	178.4 (17)
C1C—C2C—C3C—C8C	177.30 (17)	C17G—C18G—C19G—C20G	2 (5)
C15C—C2C—C3C—C4C	173.60 (18)	C18G—C19G—C20G—C21G	−10 (6)
C1C—C2C—C3C—C4C	−5.3 (3)	C18G—C19G—C20G—C23G	−176 (2)
C8C—C3C—C4C—C5C	0.0 (3)	C19G—C20G—C21G—C22G	10 (6)
C2C—C3C—C4C—C5C	−177.43 (19)	C23G—C20G—C21G—C22G	174 (3)
C3C—C4C—C5C—C6C	−1.9 (3)	C20G—C21G—C22G—C17G	−3 (4)
C4C—C5C—C6C—C7C	1.7 (3)	C18G—C17G—C22G—C21G	−5 (3)
C5C—C6C—C7C—C8C	0.5 (3)	C16C—C17G—C22G—C21G	−179.0 (19)
C4C—C3C—C8C—C9C	−176.10 (18)	C19G—C20G—C23G—C24G	36 (4)
C2C—C3C—C8C—C9C	1.4 (3)	C21G—C20G—C23G—C24G	−129 (5)
C4C—C3C—C8C—C7C	2.1 (3)	C19G—C20G—C23G—C28G	−144 (4)
C2C—C3C—C8C—C7C	179.60 (17)	C21G—C20G—C23G—C28G	51 (5)
C6C—C7C—C8C—C9C	175.8 (2)	C28G—C23G—C24G—C25G	0.0 (4)
C6C—C7C—C8C—C3C	−2.4 (3)	C20G—C23G—C24G—C25G	180.0 (3)
C3C—C8C—C9C—C10C	2.1 (3)	C23G—C24G—C25G—C26G	0.0 (6)
C7C—C8C—C9C—C10C	−176.01 (18)	C24G—C25G—C26G—C27G	0.0 (8)
C8C—C9C—C10C—C15C	−3.3 (3)	C25G—C26G—C27G—C28G	0.1 (9)

C8C—C9C—C10C—C11C	176.14 (19)	C26G—C27G—C28G—C23G	0.0 (8)
C9C—C10C—C11C—C12C	−178.9 (2)	C24G—C23G—C28G—C27G	0.0 (6)
C15C—C10C—C11C—C12C	0.6 (3)	C20G—C23G—C28G—C27G	−180.0 (4)
C10C—C11C—C12C—C13C	−1.5 (3)	N1D—C16D—C17H—C18H	2.8 (11)
C11C—C12C—C13C—C14C	0.8 (3)	N1D—C16D—C17H—C22H	163.9 (10)
C12C—C13C—C14C—C15C	0.9 (3)	C22H—C17H—C18H—C19H	19 (3)
C3C—C2C—C15C—C10C	2.6 (3)	C16D—C17H—C18H—C19H	178.7 (15)
C1C—C2C—C15C—C10C	−178.41 (17)	C17H—C18H—C19H—C20H	−12 (3)
C3C—C2C—C15C—C14C	−176.97 (18)	C18H—C19H—C20H—C21H	6 (4)
C1C—C2C—C15C—C14C	2.0 (3)	C18H—C19H—C20H—C23H	170.9 (18)
C9C—C10C—C15C—C2C	0.9 (3)	C19H—C20H—C21H—C22H	−6 (4)
C11C—C10C—C15C—C2C	−178.57 (18)	C23H—C20H—C21H—C22H	−171.4 (17)
C9C—C10C—C15C—C14C	−179.51 (17)	C18H—C17H—C22H—C21H	−20 (3)
C11C—C10C—C15C—C14C	1.0 (3)	C16D—C17H—C22H—C21H	−180.0 (16)
C13C—C14C—C15C—C2C	177.8 (2)	C20H—C21H—C22H—C17H	12 (3)
C13C—C14C—C15C—C10C	−1.8 (3)	C19H—C20H—C23H—C24H	−19 (2)
C1C—N1C—C16C—C17G	126.2 (2)	C21H—C20H—C23H—C24H	147 (2)
C1C—N1C—C16C—C17C	126.2 (2)	C19H—C20H—C23H—C28H	161 (2)
N1C—C16C—C17C—C18C	167.43 (18)	C21H—C20H—C23H—C28H	−33 (2)
N1C—C16C—C17C—C22C	−12.4 (3)	C28H—C23H—C24H—C25H	0.0 (4)
C22C—C17C—C18C—C19C	−2.7 (3)	C20H—C23H—C24H—C25H	−180.0 (3)
C16C—C17C—C18C—C19C	177.48 (19)	C23H—C24H—C25H—C26H	−0.1 (6)
C17C—C18C—C19C—C20C	0.8 (3)	C24H—C25H—C26H—C27H	0.1 (9)

C18C—C19C—C20C—C21C	1.2 (4)	C25H—C26H—C27H—C28H	−0.2 (9)
C18C—C19C—C20C—C23C	−176.1 (2)	C26H—C27H—C28H—C23H	0.2 (9)
C19C—C20C—C21C—C22C	−1.1 (4)	C24H—C23H—C28H—C27H	−0.1 (7)
C23C—C20C—C21C—C22C	176.1 (2)	C20H—C23H—C28H—C27H	179.9 (4)

Table 4: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å2) for (Prosp15mz088_0m).**27**

H-atom positions were not refined or were subject to constraints.

	x	y	z	Uiso*/Ueq	Occ. (<1)
C1A	0.4105 (2)	0.13873 (11)	0.29235 (9)	0.0293 (4)	
H1A	0.4860	0.1678	0.2805	0.035*	
N1A	0.3744 (2)	0.06918 (10)	0.27121 (9)	0.0359 (4)	
C2A	0.3421 (2)	0.17767 (11)	0.33458 (9)	0.0277 (4)	
C3A	0.2781 (2)	0.14484 (11)	0.37798 (9)	0.0286 (4)	
C4A	0.2837 (2)	0.07571 (12)	0.38938 (10)	0.0327 (5)	
H4A	0.3294	0.0484	0.3656	0.039*	
C5A	0.2253 (2)	0.04816 (13)	0.43350 (11)	0.0376 (5)	
H5A	0.2334	0.0027	0.4409	0.045*	
C6A	0.1525 (3)	0.08586 (14)	0.46860 (11)	0.0417 (5)	
H6A	0.1099	0.0652	0.4984	0.050*	
C7A	0.1441 (2)	0.15138 (13)	0.45949 (10)	0.0379 (5)	
H7A	0.0944	0.1763	0.4829	0.046*	
C8A	0.2080 (2)	0.18392 (12)	0.41559 (10)	0.0311 (4)	
C9A	0.2066 (2)	0.25374 (12)	0.40979 (10)	0.0345 (5)	
H9A	0.1572	0.2783	0.4336	0.041*	
C10A	0.2749 (2)	0.28840 (12)	0.37046 (10)	0.0330 (5)	
C11A	0.2801 (3)	0.36171 (12)	0.36859 (12)	0.0421 (5)	
H11A	0.2329	0.3865	0.3937	0.051*	
C12A	0.3500 (3)	0.39686 (13)	0.33212 (12)	0.0454 (6)	
H12A	0.3534	0.4461	0.3324	0.054*	
C13A	0.4182 (3)	0.36030 (14)	0.29350 (12)	0.0439 (6)	
H13A	0.4677	0.3853	0.2680	0.053*	
C14A	0.4142 (2)	0.28931 (13)	0.29230 (11)	0.0363 (5)	
H14A	0.4582	0.2654	0.2646	0.044*	
C15A	0.3453 (2)	0.25018 (12)	0.33169 (10)	0.0304 (4)	
C16A	0.4517 (2)	0.03982 (13)	0.22951 (11)	0.0368 (5)	
H16A	0.5097	0.0142	0.2536	0.044*	
H16B	0.5171	0.0811	0.2201	0.044*	
C17A	0.3515 (2)	−0.01345 (12)	0.16476 (10)	0.0316 (4)	0.906 (2)
C18A	0.2087 (2)	−0.02191 (14)	0.14448 (12)	0.0377 (5)	0.906 (2)
H18A	0.1700	0.0050	0.1728	0.045*	0.906 (2)
C19A	0.1201 (2)	−0.06910 (14)	0.08346 (12)	0.0364 (5)	0.906 (2)
H19A	0.0221	−0.0735	0.0706	0.044*	0.906 (2)
C20A	0.1724 (4)	−0.10999 (14)	0.04094 (13)	0.0287 (5)	0.906 (2)
C21A	0.3173 (2)	−0.10225 (12)	0.06255 (10)	0.0280 (5)	0.906 (2)
H21A	0.3559	−0.1299	0.0347	0.034*	0.906 (2)
C22A	0.4056 (2)	−0.05533 (12)	0.12339 (10)	0.0283 (5)	0.906 (2)
H22A	0.5032	−0.0516	0.1371	0.034*	0.906 (2)

C23A	0.0813 (3)	−0.15657 (16)	−0.02657 (12)	0.0300 (6)	0.906 (2)
C24A	−0.0376 (3)	−0.14052 (17)	−0.06031 (13)	0.0359 (6)	0.906 (2)
H24A	−0.0645	−0.1016	−0.0386	0.043*	0.906 (2)
C25A	−0.1162 (3)	−0.1808 (2)	−0.12513 (14)	0.0385 (7)	0.906 (2)
H25A	−0.1975	−0.1699	−0.1469	0.046*	0.906 (2)
C26A	−0.0775 (7)	−0.2369 (3)	−0.1584 (2)	0.0375 (6)	0.906 (2)
H26A	−0.1294	−0.2631	−0.2033	0.045*	0.906 (2)
C27A	0.0385 (8)	−0.2541 (3)	−0.1251 (3)	0.0359 (8)	0.906 (2)
H27A	0.0645	−0.2932	−0.1471	0.043*	0.906 (2)
C28A	0.1167 (7)	−0.2147 (3)	−0.0600 (3)	0.0312 (5)	0.906 (2)
H28A	0.1953	−0.2273	−0.0379	0.037*	0.906 (2)
C1B	0.9066 (2)	0.14488 (11)	0.28949 (9)	0.0256 (4)	
H1B	0.9852	0.1743	0.2798	0.031*	
N1B	0.86497 (19)	0.07545 (9)	0.26433 (8)	0.0305 (4)	
C2B	0.8394 (2)	0.18279 (11)	0.33321 (9)	0.0257 (4)	
C3B	0.7777 (2)	0.14877 (11)	0.37639 (9)	0.0259 (4)	
C4B	0.7825 (2)	0.07847 (11)	0.38536 (10)	0.0308 (4)	
H4B	0.8261	0.0515	0.3599	0.037*	
C5B	0.7264 (2)	0.04932 (12)	0.42935 (10)	0.0356 (5)	
H5B	0.7340	0.0032	0.4350	0.043*	
C6B	0.6566 (2)	0.08647 (13)	0.46702 (11)	0.0389 (5)	
H6B	0.6155	0.0648	0.4968	0.047*	
C7B	0.6489 (2)	0.15274 (13)	0.46031 (10)	0.0370 (5)	
H7B	0.6015	0.1773	0.4854	0.044*	
C8B	0.7104 (2)	0.18700 (12)	0.41638 (9)	0.0294 (4)	
C9B	0.7090 (2)	0.25740 (12)	0.41274 (10)	0.0323 (4)	
H9B	0.6610	0.2815	0.4379	0.039*	
C10B	0.7756 (2)	0.29330 (11)	0.37350 (10)	0.0301 (4)	
C11B	0.7805 (2)	0.36703 (12)	0.37336 (11)	0.0380 (5)	
H11B	0.7354	0.3915	0.4000	0.046*	
C12B	0.8474 (3)	0.40298 (12)	0.33637 (12)	0.0403 (5)	
H12B	0.8496	0.4522	0.3374	0.048*	
C13B	0.9143 (2)	0.36736 (12)	0.29605 (11)	0.0386 (5)	
H13B	0.9622	0.3931	0.2704	0.046*	
C14B	0.9107 (2)	0.29621 (12)	0.29357 (10)	0.0321 (4)	
H14B	0.9540	0.2730	0.2651	0.039*	
C15B	0.8437 (2)	0.25597 (11)	0.33266 (9)	0.0272 (4)	
C16B	0.9459 (2)	0.04723 (11)	0.22352 (10)	0.0302 (4)	
H16C	1.0016	0.0205	0.2474	0.036*	
H16D	1.0133	0.0891	0.2159	0.036*	
C17B	0.8474 (2)	−0.00411 (11)	0.15765 (10)	0.0272 (4)	0.906 (2)
C18B	0.7927 (2)	0.02422 (11)	0.10555 (10)	0.0269 (4)	0.906 (2)
H18B	0.8145	0.0761	0.1128	0.032*	0.906 (2)
C19B	0.7078 (2)	−0.02204 (12)	0.04397 (10)	0.0267 (4)	0.906 (2)
H19B	0.6740	−0.0012	0.0095	0.032*	0.906 (2)

C20B	0.6709 (3)	−0.09848 (14)	0.03135 (11)	0.0235 (6)	0.906 (2)
C21B	0.7231 (2)	−0.12690 (11)	0.08400 (10)	0.0248 (4)	0.906 (2)
H21B	0.6987	−0.1788	0.0773	0.030*	0.906 (2)
C22B	0.8094 (2)	−0.08021 (11)	0.14516 (10)	0.0255 (4)	0.906 (2)
H22B	0.8436	−0.1009	0.1796	0.031*	0.906 (2)
C23B	0.5832 (3)	−0.14807 (15)	−0.03520 (11)	0.0250 (5)	0.906 (2)
C24B	0.4791 (3)	−0.12935 (16)	−0.07719 (11)	0.0313 (6)	0.906 (2)
H24B	0.4647	−0.0834	−0.0631	0.038*	0.906 (2)
C25B	0.3968 (3)	−0.17675 (18)	−0.13889 (12)	0.0356 (6)	0.906 (2)
H25B	0.3255	−0.1634	−0.1663	0.043*	0.906 (2)
C26B	0.4175 (4)	−0.2432 (2)	−0.16086 (13)	0.0349 (7)	0.906 (2)
H26B	0.3608	−0.2755	−0.2032	0.042*	0.906 (2)
C27B	0.5215 (6)	−0.2623 (2)	−0.12078 (17)	0.0346 (6)	0.906 (2)
H27B	0.5373	−0.3077	−0.1359	0.042*	0.906 (2)
C28B	0.6027 (5)	−0.2156 (2)	−0.05866 (16)	0.0285 (6)	0.906 (2)
H28B	0.6730	−0.2296	−0.0314	0.034*	0.906 (2)
N1C	−0.0284 (2)	0.57310 (10)	0.27293 (9)	0.0364 (4)	
C1C	0.0298 (2)	0.64254 (11)	0.29437 (9)	0.0290 (4)	
H1C	−0.0256	0.6727	0.2819	0.035*	
C2C	0.1777 (2)	0.67956 (11)	0.33733 (9)	0.0271 (4)	
C3C	0.2506 (2)	0.64533 (11)	0.38062 (9)	0.0271 (4)	
C4C	0.1841 (2)	0.57573 (12)	0.39014 (10)	0.0325 (5)	
H4C	0.0875	0.5493	0.3655	0.039*	
C5C	0.2569 (3)	0.54635 (13)	0.43419 (11)	0.0380 (5)	
H5C	0.2089	0.5006	0.4405	0.046*	
C6C	0.4007 (3)	0.58239 (13)	0.47015 (11)	0.0382 (5)	
H6C	0.4501	0.5606	0.4996	0.046*	
C7C	0.4685 (2)	0.64839 (13)	0.46267 (10)	0.0362 (5)	
H7C	0.5661	0.6725	0.4868	0.043*	
C8C	0.3956 (2)	0.68261 (11)	0.41886 (9)	0.0292 (4)	
C9C	0.4643 (2)	0.75267 (12)	0.41502 (10)	0.0321 (4)	
H9C	0.5619	0.7764	0.4395	0.039*	
C10C	0.3928 (2)	0.78892 (11)	0.37580 (10)	0.0309 (4)	
C11C	0.4632 (3)	0.86273 (12)	0.37582 (11)	0.0386 (5)	
H11C	0.5596	0.8866	0.4021	0.046*	
C12C	0.3945 (3)	0.89929 (12)	0.33886 (11)	0.0402 (5)	
H12C	0.4418	0.9487	0.3402	0.048*	
C13C	0.2529 (3)	0.86342 (13)	0.29870 (11)	0.0394 (5)	
H13C	0.2055	0.8888	0.2725	0.047*	
C14C	0.1826 (3)	0.79294 (12)	0.29675 (10)	0.0349 (5)	
H14C	0.0874	0.7700	0.2687	0.042*	
C15C	0.2486 (2)	0.75245 (11)	0.33607 (9)	0.0285 (4)	
C16C	−0.1751 (2)	0.54604 (12)	0.23060 (11)	0.0350 (5)	
H16E	−0.2052	0.5884	0.2216	0.042*	
H16F	−0.2369	0.5209	0.2541	0.042*	

C17C	−0.1936 (2)	0.49305 (12)	0.16578 (10)	0.0314 (4)	0.911 (2)
C18C	−0.3290 (2)	0.45091 (12)	0.12501 (10)	0.0291 (5)	0.911 (2)
H18C	−0.4091	0.4543	0.1390	0.035*	0.911 (2)
C19C	−0.3490 (2)	0.40406 (12)	0.06420 (10)	0.0288 (5)	0.911 (2)
H19C	−0.4431	0.3762	0.0369	0.035*	0.911 (2)
C20C	−0.2352 (5)	0.39660 (15)	0.04185 (17)	0.0304 (6)	0.911 (2)
C21C	−0.0990 (3)	0.43767 (14)	0.08422 (12)	0.0382 (5)	0.911 (2)
H21C	−0.0186	0.4333	0.0709	0.046*	0.911 (2)
C22C	−0.0788 (2)	0.48468 (14)	0.14525 (12)	0.0390 (5)	0.911 (2)
H22C	0.0151	0.5115	0.1734	0.047*	0.911 (2)
C23C	−0.2584 (5)	0.34942 (17)	−0.02573 (16)	0.0322 (5)	0.911 (2)
C24C	−0.3780 (5)	0.2875 (2)	−0.0564 (2)	0.0341 (5)	0.911 (2)
H24C	−0.4441	0.2734	−0.0331	0.041*	0.911 (2)
C25C	−0.4028 (5)	0.2459 (3)	−0.1204 (2)	0.0401 (8)	0.911 (2)
H25C	−0.4861	0.2042	−0.1405	0.048*	0.911 (2)
C26C	−0.3069 (4)	0.26473 (17)	−0.15528 (17)	0.0406 (8)	0.911 (2)
H26C	−0.3241	0.2361	−0.1992	0.049*	0.911 (2)
C27C	−0.1860 (4)	0.32553 (16)	−0.12539 (18)	0.0428 (8)	0.911 (2)
H27C	−0.1194	0.3386	−0.1488	0.051*	0.911 (2)
C28C	−0.1613 (5)	0.3677 (2)	−0.0610 (2)	0.0419 (7)	0.911 (2)
H28C	−0.0777	0.4094	−0.0409	0.050*	0.911 (2)
C1D	0.5240 (2)	0.64278 (11)	0.28861 (9)	0.0264 (4)	
H1D	0.4625	0.6711	0.2796	0.032*	
N1D	0.47463 (19)	0.57335 (10)	0.26319 (8)	0.0313 (4)	
C2D	0.6713 (2)	0.68188 (11)	0.33125 (9)	0.0256 (4)	
C3D	0.7463 (2)	0.64928 (11)	0.37546 (9)	0.0267 (4)	
C4D	0.6815 (2)	0.57939 (11)	0.38540 (10)	0.0307 (4)	
H4D	0.5856	0.5518	0.3604	0.037*	
C5D	0.7558 (3)	0.55170 (12)	0.43045 (11)	0.0371 (5)	
H5D	0.7094	0.5059	0.4372	0.044*	
C6D	0.8988 (3)	0.58944 (14)	0.46684 (11)	0.0396 (5)	
H6D	0.9493	0.5686	0.4968	0.048*	
C7D	0.9646 (2)	0.65564 (14)	0.45907 (10)	0.0386 (5)	
H7D	1.0616	0.6810	0.4837	0.046*	
C8D	0.8902 (2)	0.68830 (12)	0.41408 (9)	0.0300 (4)	
C9D	0.9566 (2)	0.75849 (12)	0.40929 (10)	0.0345 (5)	
H9D	1.0538	0.7833	0.4338	0.041*	
C10D	0.8832 (2)	0.79291 (11)	0.36927 (10)	0.0315 (4)	
C11D	0.9508 (3)	0.86665 (12)	0.36800 (11)	0.0404 (5)	
H11D	1.0476	0.8913	0.3933	0.049*	
C12D	0.8789 (3)	0.90197 (12)	0.33118 (12)	0.0426 (6)	
H12D	0.9245	0.9513	0.3317	0.051*	
C13D	0.7360 (3)	0.86512 (13)	0.29210 (12)	0.0399 (5)	
H13D	0.6862	0.8902	0.2665	0.048*	
C14D	0.6679 (3)	0.79394 (12)	0.29036 (11)	0.0343 (5)	

H14D	0.5727	0.7700	0.2627	0.041*	
C15D	0.7383 (2)	0.75495 (11)	0.32981 (9)	0.0278 (4)	
C16D	0.3252 (2)	0.54341 (12)	0.22316 (10)	0.0313 (4)	
H16G	0.2893	0.5845	0.2156	0.038*	
H16H	0.2689	0.5163	0.2477	0.038*	
C17D	0.3055 (2)	0.49195 (11)	0.15704 (10)	0.0278 (4)	0.911 (2)
C18D	0.3415 (2)	0.52071 (12)	0.10587 (10)	0.0283 (5)	0.911 (2)
H18D	0.3824	0.5725	0.1139	0.034*	0.911 (2)
C19D	0.3182 (2)	0.47456 (12)	0.04406 (10)	0.0270 (4)	0.911 (2)
H19D	0.3425	0.4954	0.0103	0.032*	0.911 (2)
C20D	0.2598 (2)	0.39795 (12)	0.03019 (10)	0.0223 (5)	0.911 (2)
C21D	0.2280 (2)	0.36944 (11)	0.08190 (10)	0.0245 (4)	0.911 (2)
H21D	0.1901	0.3176	0.0746	0.029*	0.911 (2)
C22D	0.2514 (2)	0.41640 (12)	0.14374 (10)	0.0264 (4)	0.911 (2)
H22D	0.2292	0.3956	0.1779	0.032*	0.911 (2)
C23D	0.2297 (3)	0.34886 (13)	−0.03698 (11)	0.0240 (5)	0.911 (2)
C24D	0.3152 (3)	0.36646 (14)	−0.07740 (11)	0.0292 (5)	0.911 (2)
H24D	0.3948	0.4104	−0.0614	0.035*	0.911 (2)
C25D	0.2858 (3)	0.32100 (15)	−0.14040 (12)	0.0312 (6)	0.911 (2)
H25D	0.3462	0.3334	−0.1667	0.037*	0.911 (2)
C26D	0.1695 (3)	0.25794 (17)	−0.16497 (13)	0.0294 (6)	0.911 (2)
H26D	0.1489	0.2271	−0.2084	0.035*	0.911 (2)
C27D	0.0825 (3)	0.23959 (18)	−0.12601 (14)	0.0288 (7)	0.911 (2)
H27D	0.0016	0.1963	−0.1429	0.035*	0.911 (2)
C28D	0.1134 (3)	0.28438 (16)	−0.06242 (13)	0.0257 (6)	0.911 (2)
H28D	0.0542	0.2708	−0.0358	0.031*	0.911 (2)
C17E	0.3515 (2)	−0.01345 (12)	0.16476 (10)	0.0316 (4)	0.094 (2)
C18E	0.311 (2)	−0.0861 (8)	0.1441 (8)	0.033 (3)	0.094 (2)
H18E	0.3437	−0.1112	0.1750	0.039*	0.094 (2)
C19E	0.226 (2)	−0.1287 (9)	0.0819 (8)	0.029 (3)	0.094 (2)
H19E	0.2054	−0.1807	0.0729	0.035*	0.094 (2)
C20E	0.169 (4)	−0.1007 (12)	0.0317 (11)	0.0287 (5)	0.094 (2)
C21E	0.206 (2)	−0.0237 (10)	0.0458 (9)	0.040 (3)	0.094 (2)
H21E	0.1769	−0.0007	0.0123	0.048*	0.094 (2)
C22E	0.287 (2)	0.0183 (11)	0.1103 (9)	0.046 (3)	0.094 (2)
H22E	0.3030	0.0700	0.1210	0.055*	0.094 (2)
C23E	0.085 (3)	−0.1495 (14)	−0.0358 (10)	0.0300 (6)	0.094 (2)
C24E	−0.025 (3)	−0.1322 (17)	−0.0759 (12)	0.0359 (6)	0.094 (2)
H24E	−0.0458	−0.0887	−0.0600	0.043*	0.094 (2)
C25E	−0.101 (3)	−0.178 (2)	−0.1382 (13)	0.0385 (7)	0.094 (2)
H25E	−0.1750	−0.1653	−0.1648	0.046*	0.094 (2)
C26E	−0.074 (6)	−0.242 (3)	−0.163 (2)	0.0375 (6)	0.094 (2)
H26E	−0.1283	−0.2728	−0.2066	0.045*	0.094 (2)
C27E	0.034 (8)	−0.260 (3)	−0.124 (3)	0.0359 (8)	0.094 (2)
H27E	0.0541	−0.3034	−0.1407	0.043*	0.094 (2)

C28E	0.113 (6)	−0.214 (3)	−0.061 (2)	0.0312 (5)	0.094 (2)
H28E	0.1862	−0.2269	−0.0347	0.037*	0.094 (2)
C17F	0.8474 (2)	−0.00411 (11)	0.15765 (10)	0.0272 (4)	0.094 (2)
C18F	0.7060 (16)	−0.0153 (11)	0.1431 (9)	0.038 (3)	0.094 (2)
H18F	0.6677	0.0093	0.1733	0.046*	0.094 (2)
C19F	0.619 (2)	−0.0638 (11)	0.0830 (9)	0.039 (3)	0.094 (2)
H19F	0.5215	−0.0680	0.0681	0.047*	0.094 (2)
C20F	0.676 (2)	−0.1066 (15)	0.0441 (10)	0.0235 (6)	0.094 (2)
C21F	0.8204 (19)	−0.0983 (11)	0.0622 (8)	0.034 (3)	0.094 (2)
H21F	0.8597	−0.1245	0.0336	0.041*	0.094 (2)
C22F	0.9045 (19)	−0.0506 (10)	0.1231 (8)	0.034 (3)	0.094 (2)
H22F	0.9995	−0.0496	0.1410	0.041*	0.094 (2)
C23F	0.585 (2)	−0.1537 (13)	−0.0230 (9)	0.0250 (5)	0.094 (2)
C24F	0.476 (3)	−0.1350 (16)	−0.0593 (10)	0.0313 (6)	0.094 (2)
H24F	0.4579	−0.0911	−0.0414	0.038*	0.094 (2)
C25F	0.392 (3)	−0.1794 (19)	−0.1213 (11)	0.0356 (6)	0.094 (2)
H25F	0.3175	−0.1661	−0.1461	0.043*	0.094 (2)
C26F	0.418 (5)	−0.243 (2)	−0.1476 (15)	0.0349 (7)	0.094 (2)
H26F	0.3596	−0.2739	−0.1904	0.042*	0.094 (2)
C27F	0.526 (6)	−0.263 (2)	−0.1121 (18)	0.0346 (6)	0.094 (2)
H27F	0.5433	−0.3070	−0.1304	0.042*	0.094 (2)
C28F	0.610 (5)	−0.218 (2)	−0.0499 (16)	0.0285 (6)	0.094 (2)
H28F	0.6852	−0.2318	−0.0253	0.034*	0.094 (2)
C17G	−0.1936 (2)	0.49305 (12)	0.16578 (10)	0.0314 (4)	0.089 (2)
C18G	−0.247 (2)	0.4198 (8)	0.1465 (9)	0.033 (3)	0.089 (2)
H18G	−0.2763	0.3964	0.1781	0.040*	0.089 (2)
C19G	−0.264 (2)	0.3740 (10)	0.0855 (9)	0.032 (3)	0.089 (2)
H19G	−0.3072	0.3222	0.0764	0.039*	0.089 (2)
C20G	−0.219 (6)	0.4033 (12)	0.0372 (15)	0.0304 (6)	0.089 (2)
C21G	−0.178 (3)	0.4804 (10)	0.0485 (9)	0.040 (3)	0.089 (2)
H21G	−0.1609	0.5023	0.0141	0.048*	0.089 (2)
C22G	−0.162 (3)	0.5242 (11)	0.1108 (9)	0.043 (3)	0.089 (2)
H22G	−0.1304	0.5764	0.1187	0.052*	0.089 (2)
C23G	−0.252 (4)	0.3503 (12)	−0.0289 (12)	0.0322 (5)	0.089 (2)
C24G	−0.379 (5)	0.292 (2)	−0.053 (2)	0.0341 (5)	0.089 (2)
H24G	−0.4427	0.2870	−0.0270	0.041*	0.089 (2)
C25G	−0.416 (5)	0.241 (2)	−0.114 (2)	0.0401 (8)	0.089 (2)
H25G	−0.5042	0.2015	−0.1288	0.048*	0.089 (2)
C26G	−0.326 (5)	0.2466 (18)	−0.1523 (18)	0.0406 (8)	0.089 (2)
H26G	−0.3508	0.2118	−0.1940	0.049*	0.089 (2)
C27G	−0.200 (5)	0.3039 (19)	−0.1291 (19)	0.0428 (8)	0.089 (2)
H27G	−0.1382	0.3081	−0.1557	0.051*	0.089 (2)
C28G	−0.160 (4)	0.356 (2)	−0.0682 (19)	0.0419 (7)	0.089 (2)
H28G	−0.0723	0.3952	−0.0536	0.050*	0.089 (2)
C17H	0.3055 (2)	0.49195 (11)	0.15704 (10)	0.0278 (4)	0.089 (2)

C18H	0.4230 (17)	0.4783 (12)	0.1454 (9)	0.036 (3)	0.089 (2)
H18H	0.5153	0.5004	0.1765	0.043*	0.089 (2)
C19H	0.397 (2)	0.4297 (11)	0.0846 (9)	0.036 (3)	0.089 (2)
H19H	0.4748	0.4249	0.0696	0.044*	0.089 (2)
C20H	0.260 (2)	0.3873 (13)	0.0445 (9)	0.0223 (5)	0.089 (2)
C21H	0.141 (2)	0.3978 (11)	0.0622 (9)	0.036 (3)	0.089 (2)
H21H	0.0467	0.3726	0.0337	0.044*	0.089 (2)
C22H	0.1689 (16)	0.4473 (11)	0.1239 (9)	0.033 (3)	0.089 (2)
H22H	0.0942	0.4499	0.1425	0.040*	0.089 (2)
C23H	0.231 (2)	0.3432 (10)	−0.0252 (9)	0.0240 (5)	0.089 (2)
C24H	0.322 (3)	0.3541 (13)	−0.0627 (11)	0.0292 (5)	0.089 (2)
H24H	0.4114	0.3922	−0.0443	0.035*	0.089 (2)
C25H	0.285 (3)	0.3097 (16)	−0.1278 (11)	0.0312 (6)	0.089 (2)
H25H	0.3481	0.3174	−0.1536	0.037*	0.089 (2)
C26H	0.154 (4)	0.2544 (17)	−0.1538 (15)	0.0294 (6)	0.089 (2)
H26H	0.1278	0.2239	−0.1979	0.035*	0.089 (2)
C27H	0.063 (3)	0.2434 (18)	−0.1166 (16)	0.0288 (7)	0.089 (2)
H27H	−0.0264	0.2054	−0.1351	0.035*	0.089 (2)
C28H	0.099 (3)	0.2866 (15)	−0.0529 (15)	0.0257 (6)	0.089 (2)
H28H	0.0346	0.2784	−0.0276	0.031*	0.089 (2)

Table 5: Atomic displacement parameters (Å^2).27

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0262 (11)	0.0344 (11)	0.0245 (9)	0.0075 (9)	0.0059 (8)	0.0073 (8)
N1A	0.0376 (10)	0.0376 (10)	0.0336 (9)	0.0120 (8)	0.0160 (8)	0.0057 (8)
C2A	0.0225 (10)	0.0310 (10)	0.0238 (9)	0.0064 (9)	0.0018 (8)	0.0041 (8)
C3A	0.0239 (10)	0.0316 (10)	0.0219 (9)	0.0052 (9)	0.0008 (8)	0.0012 (8)
C4A	0.0313 (12)	0.0323 (11)	0.0290 (10)	0.0069 (9)	0.0057 (9)	0.0051 (9)
C5A	0.0365 (13)	0.0346 (11)	0.0329 (11)	0.0014 (10)	0.0055 (10)	0.0091 (9)
C6A	0.0349 (13)	0.0500 (14)	0.0322 (11)	0.0009 (11)	0.0106 (10)	0.0101 (10)
C7A	0.0284 (12)	0.0483 (13)	0.0296 (11)	0.0062 (10)	0.0095 (9)	0.0014 (10)
C8A	0.0229 (10)	0.0370 (11)	0.0229 (9)	0.0051 (9)	−0.0002 (8)	−0.0001 (8)
C9A	0.0301 (11)	0.0363 (11)	0.0288 (10)	0.0109 (10)	0.0029 (9)	−0.0029 (9)
C10A	0.0268 (11)	0.0320 (11)	0.0289 (10)	0.0080 (9)	−0.0035 (9)	−0.0005 (8)
C11A	0.0379 (13)	0.0331 (12)	0.0437 (12)	0.0127 (10)	−0.0038 (10)	0.0020 (10)
C12A	0.0416 (14)	0.0312 (11)	0.0508 (13)	0.0088 (11)	−0.0065 (11)	0.0118 (10)
C13A	0.0338 (13)	0.0448 (13)	0.0444 (13)	0.0045 (11)	−0.0025 (10)	0.0215 (11)
C14A	0.0307 (12)	0.0423 (12)	0.0348 (11)	0.0115 (10)	0.0036 (9)	0.0159 (10)
C15A	0.0226 (10)	0.0333 (11)	0.0270 (10)	0.0069 (9)	−0.0025 (8)	0.0050 (8)
C16A	0.0358 (12)	0.0435 (12)	0.0348 (11)	0.0163 (10)	0.0155 (10)	0.0082 (9)
C17A	0.0327 (11)	0.0331 (10)	0.0340 (10)	0.0113 (9)	0.0159 (9)	0.0121 (9)
C18A	0.0321 (12)	0.0448 (13)	0.0403 (12)	0.0150 (11)	0.0184 (10)	0.0076 (10)
C19A	0.0251 (11)	0.0452 (13)	0.0408 (12)	0.0108 (10)	0.0145 (10)	0.0113 (10)
C20A	0.0297 (11)	0.0314 (12)	0.0325 (12)	0.0096 (10)	0.0159 (10)	0.0177 (9)
C21A	0.0320	0.0325	0.0287	0.0158 (9)	0.0143 (9)	0.0150 (9)

	(12)	(11)	(10)			
C22A	0.0285 (11)	0.0340 (11)	0.0305 (10)	0.0146 (9)	0.0128 (9)	0.0156 (9)
C23A	0.0254 (11)	0.0364 (12)	0.0321 (12)	0.0049 (10)	0.0138 (10)	0.0185 (10)
C24A	0.0293 (12)	0.0441 (14)	0.0398 (14)	0.0104 (11)	0.0164 (11)	0.0184 (11)
C25A	0.0243 (13)	0.0505 (14)	0.0433 (15)	0.0063 (11)	0.0114 (11)	0.0251 (13)
C26A	0.0307 (12)	0.0436 (16)	0.0324 (14)	−0.0004 (11)	0.0089 (11)	0.0151 (11)
C27A	0.0378 (14)	0.0358 (15)	0.0328 (11)	0.0050 (13)	0.0133 (10)	0.0124 (11)
C28A	0.0295 (12)	0.0348 (11)	0.0317 (10)	0.0067 (9)	0.0127 (9)	0.0152 (9)
C1B	0.0247 (10)	0.0277 (10)	0.0237 (9)	0.0080 (8)	0.0062 (8)	0.0074 (8)
N1B	0.0325 (10)	0.0288 (9)	0.0321 (9)	0.0096 (8)	0.0150 (8)	0.0067 (7)
C2B	0.0230 (10)	0.0263 (10)	0.0231 (9)	0.0067 (8)	0.0026 (8)	0.0033 (8)
C3B	0.0217 (10)	0.0270 (10)	0.0220 (9)	0.0051 (8)	0.0010 (8)	0.0012 (8)
C4B	0.0302 (11)	0.0284 (10)	0.0306 (10)	0.0060 (9)	0.0083 (9)	0.0065 (8)
C5B	0.0362 (12)	0.0334 (11)	0.0331 (11)	0.0045 (10)	0.0090 (9)	0.0105 (9)
C6B	0.0359 (13)	0.0462 (13)	0.0301 (11)	0.0036 (11)	0.0114 (10)	0.0113 (10)
C7B	0.0309 (12)	0.0472 (13)	0.0277 (10)	0.0089 (10)	0.0100 (9)	0.0025 (9)
C8B	0.0239 (10)	0.0357 (11)	0.0216 (9)	0.0078 (9)	0.0020 (8)	0.0009 (8)
C9B	0.0279 (11)	0.0355 (11)	0.0282 (10)	0.0123 (9)	0.0046 (9)	−0.0014 (8)
C10B	0.0247 (11)	0.0282 (10)	0.0275 (10)	0.0090 (9)	−0.0029 (8)	−0.0022 (8)
C11B	0.0345 (12)	0.0305 (11)	0.0412 (12)	0.0132 (10)	0.0013 (10)	0.0011 (9)
C12B	0.0378 (13)	0.0255 (10)	0.0468 (13)	0.0107 (10)	−0.0036 (10)	0.0054 (9)
C13B	0.0335 (12)	0.0328 (11)	0.0417 (12)	0.0050 (10)	−0.0005 (10)	0.0149 (10)
C14B	0.0313 (11)	0.0314 (11)	0.0308 (10)	0.0101 (9)	0.0041 (9)	0.0085 (9)
C15B	0.0229	0.0279	0.0240 (9)	0.0070 (8)	−0.0013 (8)	0.0037 (8)

	(10)	(10)				
C16B	0.0302 (11)	0.0297 (10)	0.0345 (10)	0.0119 (9)	0.0145 (9)	0.0084 (8)
C17B	0.0286 (10)	0.0275 (9)	0.0312 (10)	0.0118 (8)	0.0160 (8)	0.0082 (8)
C18B	0.0307 (11)	0.0228 (10)	0.0340 (10)	0.0107 (9)	0.0160 (9)	0.0126 (8)
C19B	0.0285 (11)	0.0291 (10)	0.0304 (10)	0.0132 (9)	0.0126 (9)	0.0161 (9)
C20B	0.0237 (10)	0.0299 (11)	0.0262 (11)	0.0131 (9)	0.0134 (9)	0.0149 (9)
C21B	0.0268 (11)	0.0235 (10)	0.0286 (10)	0.0104 (9)	0.0100 (8)	0.0120 (8)
C22B	0.0285 (11)	0.0284 (10)	0.0268 (10)	0.0143 (9)	0.0105 (8)	0.0139 (8)
C23B	0.0228 (10)	0.0331 (11)	0.0257 (11)	0.0094 (9)	0.0118 (9)	0.0168 (9)
C24B	0.0326 (12)	0.0417 (13)	0.0287 (13)	0.0188 (10)	0.0114 (11)	0.0176 (11)
C25B	0.0295 (12)	0.0549 (14)	0.0284 (13)	0.0167 (11)	0.0084 (11)	0.0214 (12)
C26B	0.0280 (12)	0.0481 (13)	0.0243 (15)	0.0049 (10)	0.0069 (13)	0.0113 (12)
C27B	0.0346 (13)	0.0361 (11)	0.0321 (15)	0.0090 (10)	0.0107 (13)	0.0096 (11)
C28B	0.0277 (12)	0.0323 (11)	0.0285 (14)	0.0098 (9)	0.0089 (12)	0.0148 (10)
N1C	0.0295 (10)	0.0342 (10)	0.0379 (9)	0.0091 (8)	−0.0004 (8)	0.0076 (8)
C1C	0.0324 (11)	0.0302 (10)	0.0260 (9)	0.0122 (9)	0.0084 (9)	0.0084 (8)
C2C	0.0267 (11)	0.0300 (10)	0.0238 (9)	0.0099 (9)	0.0082 (8)	0.0037 (8)
C3C	0.0288 (11)	0.0311 (10)	0.0223 (9)	0.0131 (9)	0.0086 (8)	0.0036 (8)
C4C	0.0324 (12)	0.0330 (11)	0.0293 (10)	0.0102 (9)	0.0062 (9)	0.0061 (9)
C5C	0.0487 (14)	0.0348 (11)	0.0343 (11)	0.0188 (11)	0.0115 (10)	0.0115 (9)
C6C	0.0455 (14)	0.0433 (13)	0.0303 (11)	0.0267 (11)	0.0067 (10)	0.0093 (9)
C7C	0.0321 (12)	0.0463 (13)	0.0276 (10)	0.0188 (10)	0.0030 (9)	0.0032 (9)
C8C	0.0302 (11)	0.0341 (11)	0.0219 (9)	0.0132 (9)	0.0084 (8)	0.0001 (8)
C9C	0.0270	0.0357	0.0274	0.0088 (9)	0.0062 (8)	−0.0010

	(11)	(11)	(10)			(8)
C10C	0.0329 (12)	0.0300 (10)	0.0272 (10)	0.0074 (9)	0.0141 (9)	−0.0002 (8)
C11C	0.0380 (13)	0.0320 (11)	0.0414 (12)	0.0053 (10)	0.0166 (10)	0.0021 (9)
C12C	0.0475 (14)	0.0300 (11)	0.0449 (12)	0.0081 (11)	0.0239 (11)	0.0078 (10)
C13C	0.0515 (15)	0.0356 (12)	0.0416 (12)	0.0190 (11)	0.0231 (11)	0.0161 (10)
C14C	0.0370 (12)	0.0380 (12)	0.0321 (11)	0.0132 (10)	0.0125 (9)	0.0108 (9)
C15C	0.0320 (11)	0.0301 (10)	0.0255 (9)	0.0112 (9)	0.0133 (9)	0.0050 (8)
C16C	0.0274 (11)	0.0326 (11)	0.0400 (11)	0.0077 (9)	0.0032 (9)	0.0096 (9)
C17C	0.0261 (11)	0.0303 (10)	0.0366 (10)	0.0100 (9)	0.0032 (8)	0.0128 (8)
C18C	0.0238 (11)	0.0319 (11)	0.0342 (11)	0.0093 (9)	0.0074 (9)	0.0163 (9)
C19C	0.0237 (11)	0.0320 (11)	0.0304 (10)	0.0079 (9)	0.0031 (8)	0.0154 (9)
C20C	0.0263 (17)	0.0309 (11)	0.0366 (11)	0.0117 (10)	0.0066 (9)	0.0149 (9)
C21C	0.0248 (12)	0.0452 (13)	0.0441 (12)	0.0151 (10)	0.0063 (10)	0.0110 (10)
C22C	0.0225 (11)	0.0413 (13)	0.0451 (13)	0.0084 (10)	0.0012 (10)	0.0072 (10)
C23C	0.0317 (12)	0.0372 (11)	0.0358 (11)	0.0202 (10)	0.0094 (9)	0.0167 (9)
C24C	0.0381 (13)	0.0386 (13)	0.0322 (12)	0.0181 (11)	0.0108 (10)	0.0159 (10)
C25C	0.0436 (17)	0.0413 (14)	0.0362 (15)	0.0174 (12)	0.0087 (13)	0.0110 (12)
C26C	0.046 (2)	0.050 (2)	0.0352 (12)	0.029 (2)	0.0132 (12)	0.0153 (14)
C27C	0.0452 (16)	0.056 (2)	0.0482 (14)	0.0335 (17)	0.0251 (13)	0.0238 (16)
C28C	0.0345 (13)	0.0486 (18)	0.0520 (16)	0.0226 (13)	0.0174 (12)	0.0169 (13)
C1D	0.0300 (11)	0.0290 (10)	0.0224 (9)	0.0116 (9)	0.0091 (8)	0.0078 (8)
N1D	0.0289 (9)	0.0310 (9)	0.0291 (8)	0.0094 (8)	0.0023 (7)	0.0055 (7)
C2D	0.0262 (10)	0.0287 (10)	0.0212 (9)	0.0094 (9)	0.0080 (8)	0.0036 (8)
C3D	0.0304 (11)	0.0281 (10)	0.0215 (9)	0.0113 (9)	0.0092 (8)	0.0019 (8)

C4D	0.0351 (12)	0.0288 (10)	0.0262 (10)	0.0117 (9)	0.0063 (9)	0.0046 (8)
C5D	0.0498 (14)	0.0324 (11)	0.0330 (11)	0.0193 (11)	0.0126 (10)	0.0096 (9)
C6D	0.0457 (14)	0.0497 (14)	0.0299 (11)	0.0292 (12)	0.0072 (10)	0.0114 (10)
C7D	0.0317 (12)	0.0513 (14)	0.0289 (10)	0.0179 (11)	0.0031 (9)	0.0036 (10)
C8D	0.0295 (11)	0.0347 (11)	0.0230 (9)	0.0121 (9)	0.0072 (8)	−0.0001 (8)
C9D	0.0274 (11)	0.0385 (12)	0.0284 (10)	0.0043 (10)	0.0071 (9)	−0.0015 (9)
C10D	0.0332 (12)	0.0285 (10)	0.0291 (10)	0.0061 (9)	0.0142 (9)	−0.0010 (8)
C11D	0.0425 (14)	0.0299 (11)	0.0421 (12)	0.0014 (10)	0.0196 (11)	0.0003 (9)
C12D	0.0559 (16)	0.0259 (10)	0.0514 (13)	0.0090 (11)	0.0323 (12)	0.0084 (10)
C13D	0.0584 (16)	0.0347 (11)	0.0426 (12)	0.0226 (11)	0.0305 (12)	0.0171 (10)
C14D	0.0412 (13)	0.0360 (11)	0.0332 (11)	0.0166 (10)	0.0182 (10)	0.0119 (9)
C15D	0.0352 (12)	0.0280 (10)	0.0230 (9)	0.0113 (9)	0.0147 (9)	0.0039 (8)
C16D	0.0256 (11)	0.0321 (11)	0.0332 (10)	0.0086 (9)	0.0048 (9)	0.0079 (9)
C17D	0.0194 (10)	0.0318 (10)	0.0299 (10)	0.0092 (8)	0.0024 (8)	0.0085 (8)
C18D	0.0234 (11)	0.0256 (10)	0.0344 (11)	0.0062 (9)	0.0046 (9)	0.0124 (9)
C19D	0.0243 (11)	0.0307 (11)	0.0293 (10)	0.0080 (9)	0.0084 (8)	0.0162 (9)
C20D	0.0192 (10)	0.0270 (11)	0.0246 (11)	0.0100 (8)	0.0049 (9)	0.0140 (8)
C21D	0.0240 (10)	0.0247 (10)	0.0263 (10)	0.0077 (8)	0.0072 (8)	0.0109 (8)
C22D	0.0244 (11)	0.0313 (11)	0.0250 (10)	0.0080 (9)	0.0066 (8)	0.0132 (8)
C23D	0.0258 (10)	0.0280 (10)	0.0250 (11)	0.0142 (9)	0.0074 (9)	0.0153 (9)
C24D	0.0297 (11)	0.0335 (12)	0.0291 (12)	0.0108 (10)	0.0102 (10)	0.0164 (9)
C25D	0.0362 (12)	0.0432 (14)	0.0274 (12)	0.0199 (11)	0.0167 (11)	0.0208 (10)
C26D	0.0362 (14)	0.0347 (11)	0.0240 (13)	0.0196 (11)	0.0097 (10)	0.0105 (10)

C27D	0.0284 (14)	0.0291 (11)	0.0319 (13)	0.0131 (10)	0.0101 (11)	0.0085 (10)
C28D	0.0275 (12)	0.0294 (10)	0.0264 (13)	0.0127 (9)	0.0119 (9)	0.0121 (9)
C17E	0.0327 (11)	0.0331 (10)	0.0340 (10)	0.0113 (9)	0.0159 (9)	0.0121 (9)
C18E	0.033 (4)	0.037 (4)	0.033 (4)	0.017 (4)	0.012 (4)	0.013 (4)
C19E	0.030 (4)	0.030 (4)	0.033 (4)	0.016 (4)	0.012 (4)	0.013 (4)
C20E	0.0297 (11)	0.0314 (12)	0.0325 (12)	0.0096 (10)	0.0159 (10)	0.0177 (9)
C21E	0.036 (5)	0.041 (5)	0.042 (5)	0.010 (5)	0.014 (5)	0.013 (5)
C22E	0.037 (5)	0.047 (5)	0.049 (5)	0.012 (5)	0.012 (5)	0.007 (5)
C23E	0.0254 (11)	0.0364 (12)	0.0321 (12)	0.0049 (10)	0.0138 (10)	0.0185 (10)
C24E	0.0293 (12)	0.0441 (14)	0.0398 (14)	0.0104 (11)	0.0164 (11)	0.0184 (11)
C25E	0.0243 (13)	0.0505 (14)	0.0433 (15)	0.0063 (11)	0.0114 (11)	0.0251 (13)
C26E	0.0307 (12)	0.0436 (16)	0.0324 (14)	−0.0004 (11)	0.0089 (11)	0.0151 (11)
C27E	0.0378 (14)	0.0358 (15)	0.0328 (11)	0.0050 (13)	0.0133 (10)	0.0124 (11)
C28E	0.0295 (12)	0.0348 (11)	0.0317 (10)	0.0067 (9)	0.0127 (9)	0.0152 (9)
C17F	0.0286 (10)	0.0275 (9)	0.0312 (10)	0.0118 (8)	0.0160 (8)	0.0082 (8)
C18F	0.036 (5)	0.039 (5)	0.039 (5)	0.012 (4)	0.014 (4)	0.006 (4)
C19F	0.036 (5)	0.038 (5)	0.040 (5)	0.006 (5)	0.012 (4)	0.010 (4)
C20F	0.0237 (10)	0.0299 (11)	0.0262 (11)	0.0131 (9)	0.0134 (9)	0.0149 (9)
C21F	0.037 (5)	0.034 (5)	0.033 (5)	0.010 (4)	0.012 (4)	0.010 (4)
C22F	0.035 (5)	0.032 (5)	0.037 (5)	0.012 (4)	0.010 (4)	0.012 (4)
C23F	0.0228 (10)	0.0331 (11)	0.0257 (11)	0.0094 (9)	0.0118 (9)	0.0168 (9)
C24F	0.0326 (12)	0.0417 (13)	0.0287 (13)	0.0188 (10)	0.0114 (11)	0.0176 (11)
C25F	0.0295 (12)	0.0549 (14)	0.0284 (13)	0.0167 (11)	0.0084 (11)	0.0214 (12)
C26F	0.0280 (12)	0.0481 (13)	0.0243 (15)	0.0049 (10)	0.0069 (13)	0.0113 (12)
C27F	0.0346 (13)	0.0361 (11)	0.0321 (15)	0.0090 (10)	0.0107 (13)	0.0096 (11)
C28F	0.0277 (12)	0.0323 (11)	0.0285 (14)	0.0098 (9)	0.0089 (12)	0.0148 (10)
C17G	0.0261	0.0303	0.0366	0.0100 (9)	0.0032 (8)	0.0128 (8)

	(11)	(10)	(10)			
C18G	0.027 (4)	0.037 (4)	0.033 (4)	0.003 (4)	0.008 (4)	0.014 (4)
C19G	0.026 (4)	0.034 (4)	0.037 (4)	0.007 (4)	0.012 (4)	0.010 (4)
C20G	0.0263 (17)	0.0309 (11)	0.0366 (11)	0.0117 (10)	0.0066 (9)	0.0149 (9)
C21G	0.031 (5)	0.042 (6)	0.045 (5)	0.008 (5)	0.007 (5)	0.018 (5)
C22G	0.032 (5)	0.041 (5)	0.049 (5)	0.007 (5)	0.005 (5)	0.009 (5)
C23G	0.0317 (12)	0.0372 (11)	0.0358 (11)	0.0202 (10)	0.0094 (9)	0.0167 (9)
C24G	0.0381 (13)	0.0386 (13)	0.0322 (12)	0.0181 (11)	0.0108 (10)	0.0159 (10)
C25G	0.0436 (17)	0.0413 (14)	0.0362 (15)	0.0174 (12)	0.0087 (13)	0.0110 (12)
C26G	0.046 (2)	0.050 (2)	0.0352 (12)	0.029 (2)	0.0132 (12)	0.0153 (14)
C27G	0.0452 (16)	0.056 (2)	0.0482 (14)	0.0335 (17)	0.0251 (13)	0.0238 (16)
C28G	0.0345 (13)	0.0486 (18)	0.0520 (16)	0.0226 (13)	0.0174 (12)	0.0169 (13)
C17H	0.0194 (10)	0.0318 (10)	0.0299 (10)	0.0092 (8)	0.0024 (8)	0.0085 (8)
C18H	0.027 (5)	0.037 (5)	0.036 (5)	0.005 (5)	0.005 (4)	0.007 (5)
C19H	0.029 (5)	0.036 (5)	0.038 (5)	0.008 (4)	0.003 (5)	0.009 (5)
C20H	0.0192 (10)	0.0270 (11)	0.0246 (11)	0.0100 (8)	0.0049 (9)	0.0140 (8)
C21H	0.030 (5)	0.033 (5)	0.037 (5)	0.003 (5)	−0.001 (5)	0.012 (4)
C22H	0.029 (5)	0.034 (5)	0.034 (5)	0.006 (4)	0.006 (4)	0.011 (4)
C23H	0.0258 (10)	0.0280 (10)	0.0250 (11)	0.0142 (9)	0.0074 (9)	0.0153 (9)
C24H	0.0297 (11)	0.0335 (12)	0.0291 (12)	0.0108 (10)	0.0102 (10)	0.0164 (9)
C25H	0.0362 (12)	0.0432 (14)	0.0274 (12)	0.0199 (11)	0.0167 (11)	0.0208 (10)
C26H	0.0362 (14)	0.0347 (11)	0.0240 (13)	0.0196 (11)	0.0097 (10)	0.0105 (10)
C27H	0.0284 (14)	0.0291 (11)	0.0319 (13)	0.0131 (10)	0.0101 (11)	0.0085 (10)
C28H	0.0275 (12)	0.0294 (10)	0.0264 (13)	0.0127 (9)	0.0119 (9)	0.0121 (9)