SPECTROPHOTOMETRIC DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS IN AIRBORNE PARTICULATES

by

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ABSTRACT

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The separation and identification of polycyclic aromatic hydrocarbons in airborne particulates is important because some of these hydrocarbons have been found to be carcinogenic to man. They are present in combustion sources, cigarette smoke and in coal-tar pitch.

The polycyclic hydrocarbons were extracted from dust with benzene. These air particulates were collected from heating unit filters in a Youngstown building. The hydrocarbons in the benzene-soluble fraction were then separated by two-dimensional dual-band thin-layer chromatography. All these steps were done in the dark to avoid the photochemical decomposition of the compounds.

The locations of the hydrocarbons in the chromatoplate were detected by their fluorescence in ultraviolet light. The hydrocarbons were then extracted with methanol. The ultraviolet-visible absorption spectra of the methanol extracts were determined from 220 to 460 nm with a Cary 14 recording spectrophotometer. The absorption spectra of the

isolated polycyclic aromatic hydrocarbons were compared with available standards.

The R_f values of both standards and samples using 26 per cent acetylated cellulose as the adsorbent were also determined.

Benzo(a)pyrene which is a strong carcinogen has been identified. Benz(a)anthracene, a weak carcinogen, is also present. The other polycyclic aromatic hydrocarbons identified are coronene, benzo(g,h,i)perylene, perylene, benzo-(b)fluoranthene and, possibly, a pyrene derivative.

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The author gratefully acknowledges the polycyclic aromatic hydrocarbons that were given by Dr. E. Sawicki and J. Meeker of the Air Pollution Engineering Research, Robert A. Taft Sanitary Engineering Center, U. S. Department of Health, Welfare and Education in Cincinnati, Ohio.

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CHAPTER I

INTRODUCTION

Polynuclear aromatic hydrocarbons are of interest in air pollution research because of their carcinogenic activity and their presence in combustion sources, soot, cigarette smoke and engine exhausts.[1-3] Benzo(a)pyrene, a strong carcinogen, and benz(a)anthracene, a weak carcinogen, have been isolated from coal-tar pitch and have caused cancer of the skin and of the scrotum in chimney sweeps and coal-tar workers.[4]

The metabolism of polycyclic hydrocarbons has not been fully understood. Boyland[5] has discussed three hypotheses concerning the mechanism of action of carcinogenic polycyclic compounds. It is possible that aromatic carcinogens complex with purines. This is shown by the shift in bands toward longer wavelengths in the ultraviolet absorption of benzo(a)pyrene when it complexes with purines. Several experiments show that polycyclic hydrocarbon can combine with DNA. Many of the carcinogenic hydrocarbons are about the the same size and shape as the purine-pyrimidine pairs. It is also possible that carcinogenesis by chemicals is due to destruction of suppressors which control function and synthesis in normal cells so that these become malignant. These hypotheses need further investigation.

Benzo(a)pyrene and benz(a)anthracene with pyrene, benz(e)pyrene, fluoranthene, benzo(a)fluorene and/or benzo-(b)fluorene, chrysene, benzo(k)fluoranthene, perylene, benzo(g,h,i)perylene, anthanthrene and coronene are found consistently in airborne particulates of some 100 communities.[6]

A simple standardized analytical procedure is needed for the separation and identification of polynuclear aromatic hydrocarbons in airborne particulates. Benzene, methylene chloride and cyclohexane are the commonly used solvents for extraction of organic materials from these particulates. The hydrocarbons are then separated by column chromatography and thin-layer chromatography and identified by spectrophotometric and spectrophotofluorometric methods.

In this research, polynuclear aromatic hydrocarbons in particulates obtained from Youngstown air were separated and identified by two-dimensional dual-band thin-layer chromatography and ultraviolet-visible absorption spectroscopy.

REVIEW OF THE LITERATURE

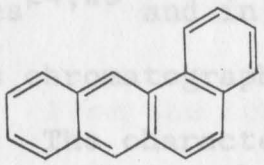
The benzo(a)pyrene content of the atmosphere has been the main interest of environmental studies because of the fact that it is carcinogenic to experimental animals[1] and is suspected of being carcinogenic to man. It may be used as a semi-quantitative index of the presence in air of other polynuclear hydrocarbons and has been used as a

measure of carcinogenic potential for comparison with other hydrocarbons.[1] Figure 1 shows the structures of benzo(a)-pyrene and some of the polycyclic hydrocarbons commonly found in airborne particulates.

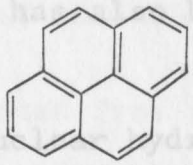
Many solvents and methods have been used to isolate organic fractions of particulates. Benzene, cyclohexane and acetone were nearly 100 per cent efficient in the extraction of benzo(a)pyrene in a study of solvent effects by Stanley, et al.[7] Benzene is used as the extracting solvent in the method of analysis for polynuclear aromatic hydrocarbon content of atmospheric particulate matter proposed by Sawicki and his group to the Intersociety Committee for a Manual of Methods for Ambient Air Sampling and Analysis.[8] Dubois and his coworkers[9] prefer cyclohexane to benzene because the latter, although it is a good solvent, seems to extract a considerable amount of non-aromatic material which may cause some interference at later stages of the analysis.

Thin-layer chromatography has proved a valuable tool for the separation and determination of the polycyclic hydrocarbons.[10-22] Some of the commonly used adsorbents are alumina, silica gel, cellulose and cellulose acetate. The cellulose acetate adsorbent system is best for the separation of the benzpyrene fraction obtained from column chromatography of the dust sample while the cellulose adsorbent system is best for the separation of the polynuclear aromatic hydrocarbons.[10]

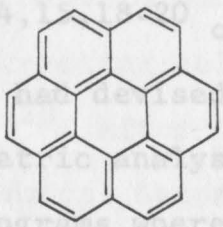
Benz(a)anthracene



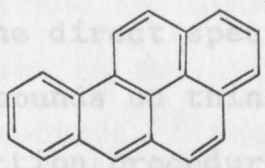
Pyrene



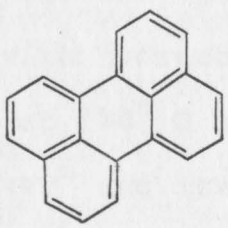
Coronene



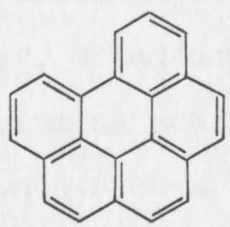
Benzo(a)pyrene



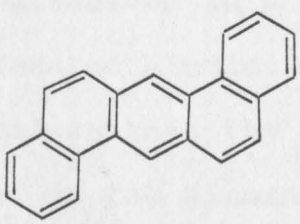
Perylene



Benzo(g,h,i)perylene



1,2,5,6-Dibenzanthracene



Chrysene

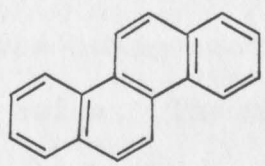


Fig. 1. Structures of polynuclear aromatic hydrocarbons.

White and Howard[23] separated polycyclic hydrocarbons from vegetable oil by reverse phase thin-layer chromatography. Analysis of polycyclic aromatic hydrocarbons in soot samples[24,25] and in cigarette smoke[26] has also been performed by gas chromatography.

The characterization of polynuclear hydrocarbons on thin-layer chromatograms is either by ultraviolet absorption spectroscopy[6,17,18,21,22,27] or by fluorescence spectroscopy[8,14,15,18-20] or usually both. Sawicki, Stanley and Johnson had devised a procedure for the direct spectrophotofluorometric analysis of aromatic compounds on thin-layer chromatograms where the tedious extraction procedure is eliminated.[21]

CHAPTER II

MATERIALS AND EXPERIMENTAL METHODS

From the dust particles collected from filters in heating units in the Ward Beecher Science Hall in Youngstown State University, approximately 20 g were used for Soxhlet extraction with benzene. Benzene has been found to be a good extracting solvent for benzo(a)pyrene and other hydrocarbons.[28] After 10 hours of extraction in the dark to avoid photochemical decomposition of the compounds,[29] the benzene extract was evaporated. About 700 mg of the benzene-soluble fraction of the air particulate sample were obtained.

For the chromatographic work, aluminum oxide was washed with hydrochloric acid and ether, dried and heated in an oven at 130° C for 30 hours. The alumina contained 12 per cent water[6] and its final concentration was then adjusted to 13.7 per cent.

150 mg of the benzene-soluble fraction of the sample were dissolved in a small volume of chloroform. One gram of the treated alumina was then added. The chloroform was evaporated so that the organic material was homogeneously dispersed in the alumina. This was done twice. The material is dispersed in alumina prior to chromatography because it is only slightly soluble in the primary eluting solvent.[6] The mixture was then added to a 0.5 x 15 inch column which

contained the treated alumina. 0.5 g of silica gel was added to the column.

The column was eluted with successive 100-ml volumes of redistilled n-pentane containing 0, 3, 6, 9 and 12 per cent of ether, respectively. The column was protected from the light.[6,29] Fractions of 25 ml were collected and the solvent was evaporated in the dark.

The residues were dissolved in 3 ml of n-pentane. The ultraviolet-visible absorption spectrum of each solution was then determined from 220 to 460 nm with the Cary 14 recording spectrophotometer.

The polycyclic hydrocarbons were separated by two-dimensional dual-band thin-layer chromatography.[30] Merck's aluminum oxide G (type E) for thin-layer chromatography and 26 per cent acetylated cellulose were used as the adsorbents.

The 26 per cent acetylated cellulose was prepared by the acetylation of cellulose powder.[30] Camag's cellulose powder DF for thin-layer chromatography was dried at 110° C for 30 minutes and cooled in a dessicator containing concentrated sulfuric acid. 30 g of the dried cellulose powder was acetylated in a mixed solution of benzene (675 ml), acetic anhydride (225 ml), and concentrated sulfuric acid (0.9 ml). Twenty-six per cent acetylated cellulose was obtained by the reaction at 70° C for 9 hours. The cellulose was washed with methanol and ether and dried completely in an oven at about 80° C.

An applicator[30] as shown in Figure 2 was used to prepare the chromatoplates with two adsorbent layers of aluminum oxide and acetylated cellulose.

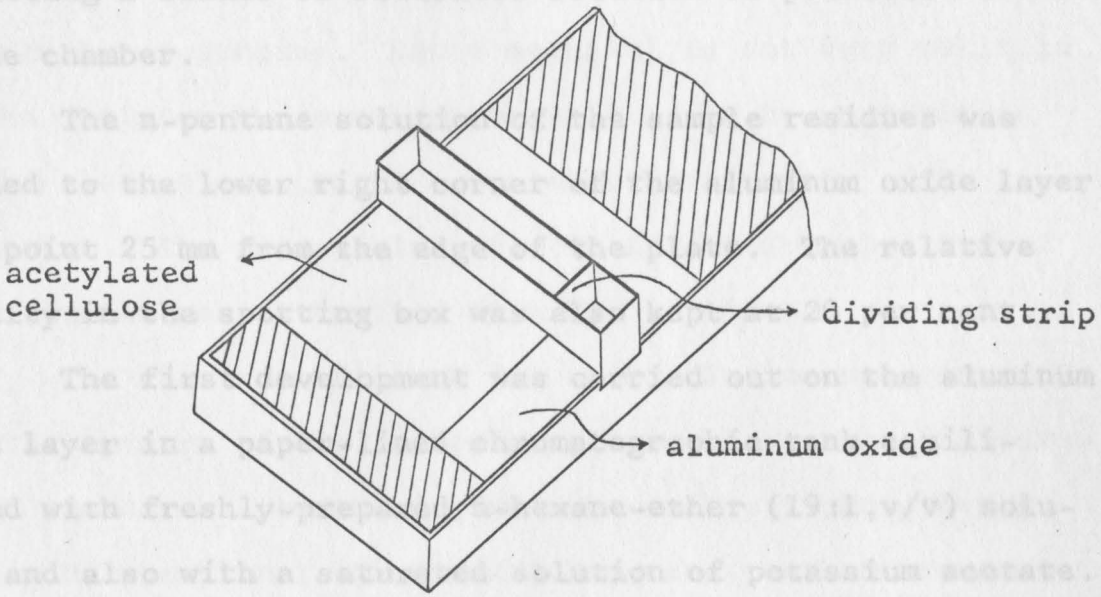


Fig. 2. Applicator for preparing dual band thin-layer chromatoplates.

Slurries of the adsorbents were prepared by mixing 10.0 g of the acetylated cellulose with 43.0 ml of 10 per cent aqueous methanol solution in a blender for one minute and 10.0 g of aluminum oxide with 13.0 ml of the methanol solution. The aluminum oxide slurry was poured into the smaller part of the trough and the slurry of acetylated cellulose into the larger part. They were immediately spread over glass plates (20 cm x 20 cm) to give a thickness of 250 μ.

The chromatoplates were dried for 3 hours at room temperature and were activated at 110° C for one hour. The activated plates were stored in a chamber kept at a relative humidity of about 20 per cent. The humidity was controlled by putting a beaker of saturated solution of potassium acetate in the chamber.

The n-pentane solution of the sample residues was applied to the lower right corner of the aluminum oxide layer at a point 25 mm from the edge of the plate. The relative humidity in the spotting box was also kept at 20 per cent.

The first development was carried out on the aluminum oxide layer in a paper-lined chromatographic tank equilibrated with freshly-prepared n-hexane-ether (19:1,v/v) solution and also with a saturated solution of potassium acetate. The development was continued until the solvent front had traveled 150 mm. The developing time was about 20 minutes. The plate was removed from the chamber, air-dried for 5 minutes, rotated 90° and placed in another chromatographic tank which had been equilibrated with freshly-prepared ether-methanol-water (4:4:1,v/v) mixture.

Separation on the acetylated cellulose layer was carried out by developing the plate three times to the 130 mm mark in the second solvent system with intermittent air-drying for 10 minutes. The total developing time was about 5 hours. Both developments were done in the dark.[6,29]

The polycyclic hydrocarbons separated on the chromatoplate were detected by means of their fluorescence under

ultraviolet light. The exposure to ultraviolet light was as brief as possible to avoid the photochemical decomposition of the hydrocarbons.[29,30]

The spots were then scraped off from the glass plate and extracted with methanol which was a better extraction solvent than n-pentane. Since methanol is not very volatile and the isolated hydrocarbons were found to be unstable in it, the absorption spectra were taken immediately after extraction. The ultraviolet-visible absorption spectrum of each solution was determined from 220 to 460 nm.

The spectrum obtained for each solution was compared with the spectra of the following polynuclear aromatic hydrocarbon standards: benzo(a)pyrene; benzo(r,s,t)pentaphene; benzo(g,h,i)perylene; 1,2,3,4-dibenzpyrene; anthanthrene; 5-methyl-3,4,8,9-dibenzpyrene; 20-methyl cholanthrene; pyrene; 1,2,4,5-dibenzopyrene; chrysene; 1,2,5,6-dibenzanthracene; coronene; benzo(b)fluoranthene; benz(a)anthracene; perylene; 1,2,3,4-dibenzanthracene and 2,3,6,7-dibenzanthracene.

The absorption spectra of the hydrocarbon standards in n-pentane were determined from 220 to 460 nm. n-Pentane was used since it readily dissolves aromatic hydrocarbons and it has a low ultraviolet cutoff.[18]

Some of the hydrocarbon standards were spotted on the chromatoplates and were then extracted with methanol. The spectra of the methanol extracts were recorded at the same wavelength range. There was a very small change in the spectra of the hydrocarbons in methanol compared with that

in n-pentane so that the latter were used for identification of the isolated hydrocarbons.

The R_f values of the standards and the isolated polycyclic hydrocarbons in 26 per cent acetylated cellulose were also determined.

CHAPTER III

RESULTS AND DISCUSSION

Column Chromatography

The amount of water in the alumina and the percentage of ether in the eluent affect the chromatography of the benzene-soluble fraction of the dust sample. Sawicki, et al.[6,8] found that alumina containing 12-13 per cent water is best for aliphatic and tetracyclic hydrocarbons while 14-15 per cent water works best for penta-, hexa- and heptacyclic aromatic hydrocarbons when the eluent is a pentane-ether mixture. Therefore alumina containing 13.7 per cent water was used for the fractionation.

Thin-Layer Chromatography

Figures 3 to 6 show the chromatograms of the organic fractions after the second development of the chromatoplates.

The R_f values of the isolated hydrocarbons in acetylated cellulose and their fluorescence color when exposed to long-wave ultraviolet light (365 nm) are given in Table 1.

Table 2 lists the R_f values and fluorescence color of the polynuclear aromatic hydrocarbon standards in acetylated cellulose.

TABLE 1

R_f VALUES AND FLUORESCENCE COLOR OF ISOLATED HYDROCARBONS IN ACETYLATED CELLULOSE

Isolated Hydrocarbon	R_f[a]	Fluorescence Color
Benzo(a)pyrene	0.11	Blue
Benz(a)anthracene	0.31	Violet
Benzo(b)fluoranthene	0.13	Light blue
Perylene	0.34	Blue-green
Benzo(g,h,i)perylene	0.48	Violet
Pyrene derivative	0.40	Dull yellow
Coronene[b]		Violet

[a] R_f values were mean of 3 determinations.

[b] A different type of adsorbent for thin-layer chromatography was used.

TABLE 2

R_f VALUES AND FLUORESCENCE COLOR OF POLYNUCLEAR AROMATIC HYDROCARBON STANDARDS IN ACETYLATED CELLULOSE

Polynuclear Aromatic Hydrocarbon	R_f [a]	Fluorescence Color
Pyrene	0.46	Violet
Benzo(a)pyrene	0.13	Blue
Chrysene	0.12	Dull violet
Coronene	0.53	Blue-green
Benzo(r,s,t)pentaphene	0.49	Blue violet
1,2,3,4-Dibenzpyrene	0.56	Yellow
20-Methyl cholanthrene	0.39	Blue
1,2,4,5-Dibenzpyrene	0.48	Violet
Benzo(g,h,i)perylene	0.51	Blue
1,2,5,6-Dibenzanthracene	0.38	Dull violet
Benzo(b)fluoranthene	0.16	Light blue
Perylene	0.36	Blue-green
1,2,3,4-Dibenzanthracene	0.52	Violet
Benz(a)anthracene	0.33	Violet
2,3,6,7-Dibenzanthracene	0.36	Dull yellow

[a] R_f values were mean of 3 determinations.

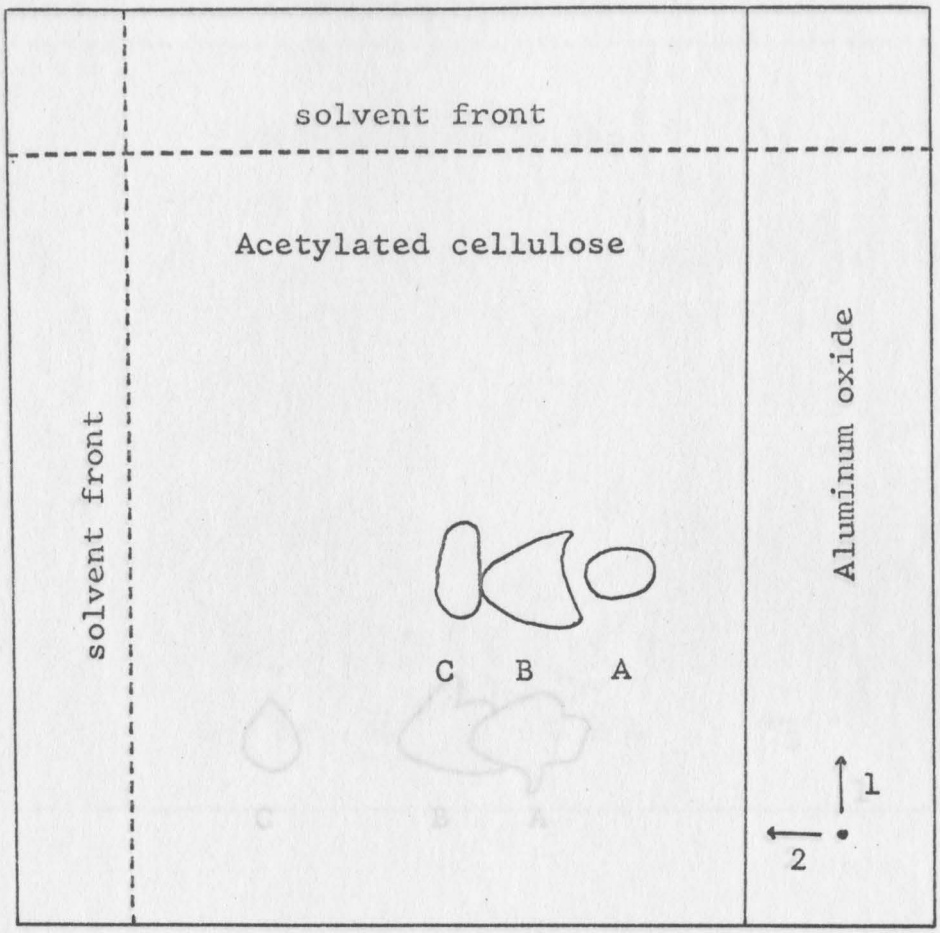


Fig. 3. Two-dimensional dual band thin-layer chromatogram of the pyrene fraction.

Spot A - dull violet fluorescence
Spot B - light blue fluorescence and has been identified as a possible pyrene derivative
Spot C - violet fluorescence

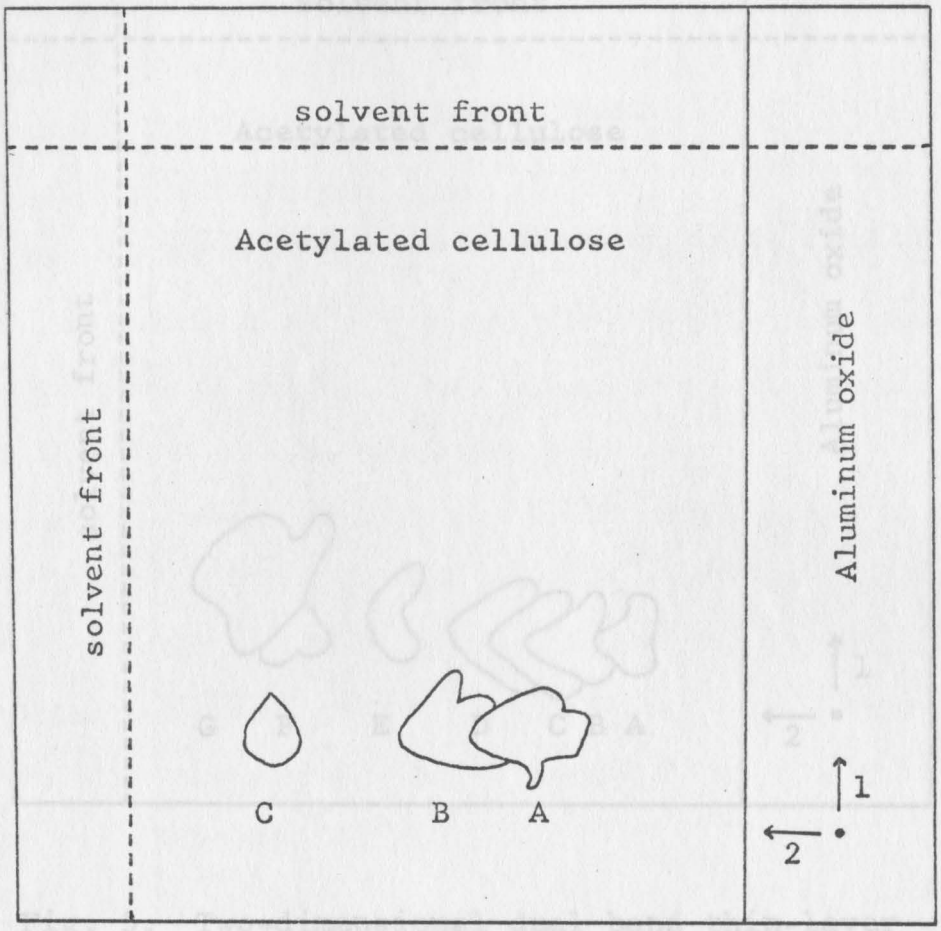


Fig. 4. Two-dimensional dual band thin-layer chromatogram of the benzanthracene fraction.

Spot A - violet fluorescence and has been identified as benz(a)anthracene
Spot B - dull light blue fluorescence
Spot C - blue fluorescence

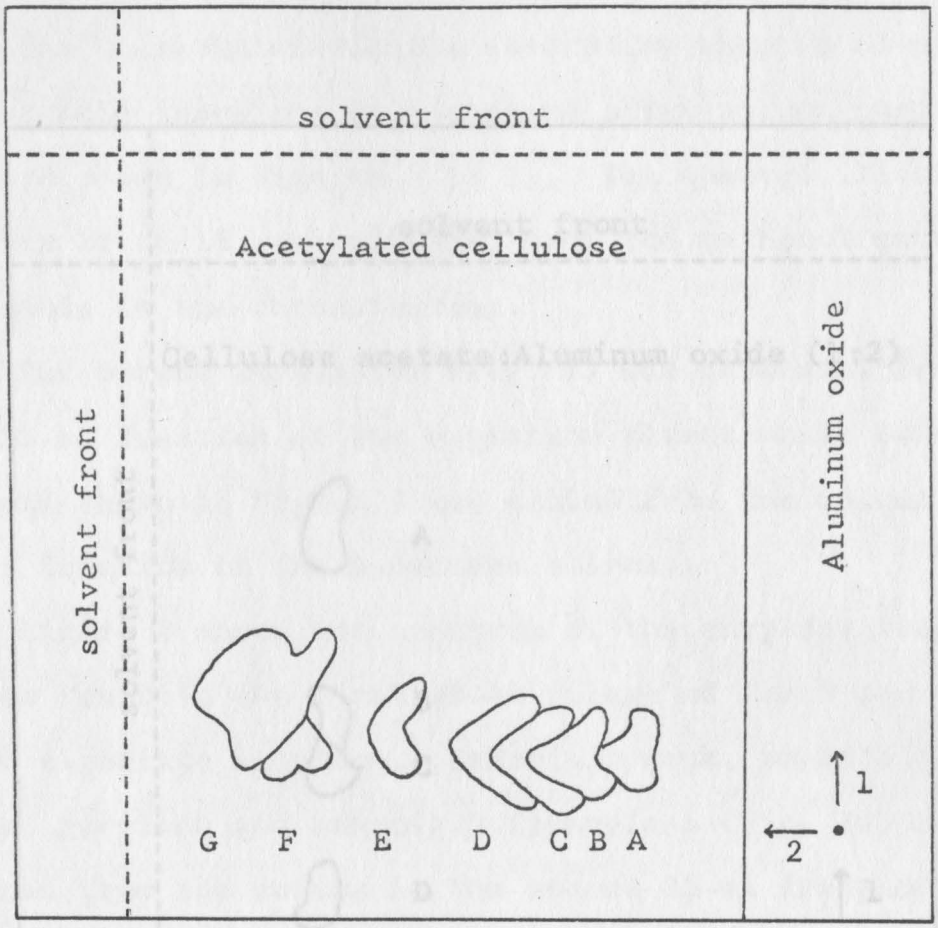


Fig. 5. Two-dimensional dual band thin-layer chromatogram of the benzopyrene fraction.

Spot A - blue fluorescence and has been identified as benzo(a)pyrene
Spot B - light blue fluorescence and has been identified as benzo(b)-fluoranthene
Spot C - yellow fluorescence
Spot D - blue fluorescence
Spot E - blue-green fluorescence and has been identified as perylene
Spot F - light violet fluorescence and has been identified as benzo-(g,h,i)perylene
Spot G - dull light blue fluorescence

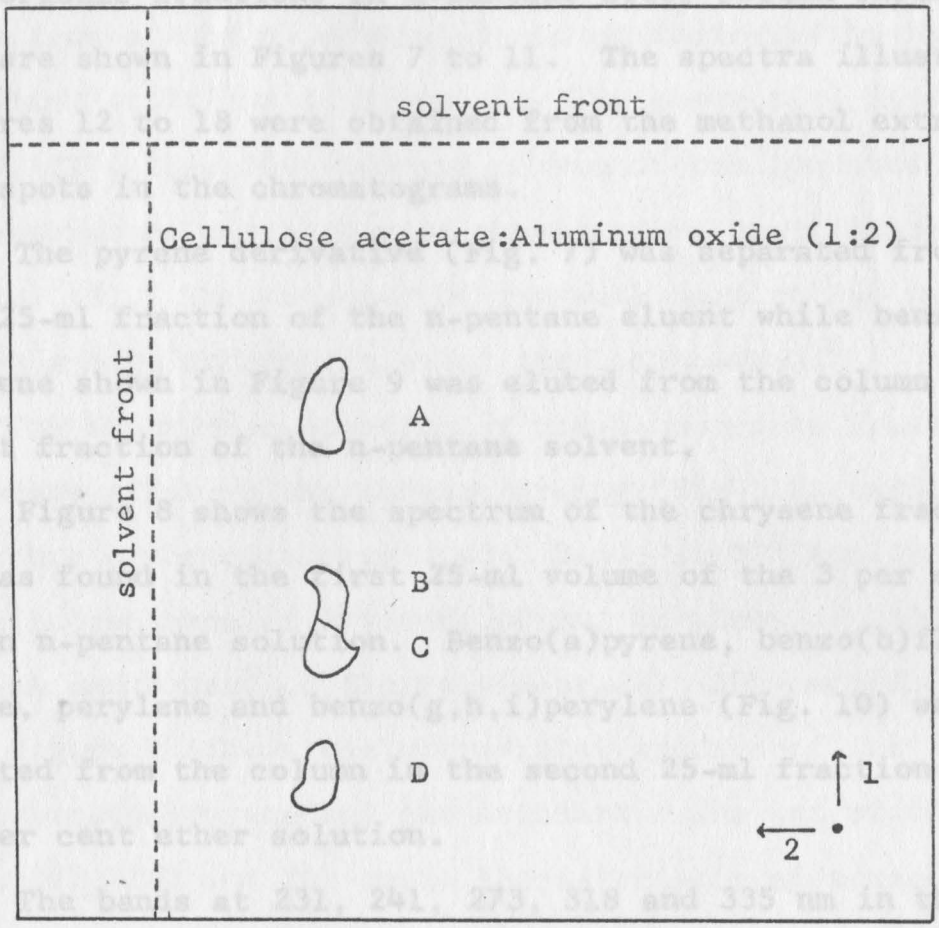


Fig. 6. Two-dimensional thin-layer chromatogram of the coronene fraction.

Spot A - dull violet fluorescence
Spot B - violet fluorescence and has been identified as coronene
Spot C - blue fluorescence
Spot D - greenish fluorescence

Ultraviolet - Visible Spectrophotometry

The ultraviolet-visible absorption spectra of some of the organic fractions in n-pentane after column chromatography are shown in Figures 7 to 11. The spectra illustrated in Figures 12 to 18 were obtained from the methanol extracts of the spots in the chromatograms.

The pyrene derivative (Fig. 7) was separated from the second 25-ml fraction of the n-pentane eluent while benz(a)-anthracene shown in Figure 9 was eluted from the column in the last fraction of the n-pentane solvent.

Figure 8 shows the spectrum of the chrysene fraction which was found in the first 25-ml volume of the 3 per cent ether in n-pentane solution. Benzo(a)pyrene, benzo(b)fluoranthene, perylene and benzo(g,h,i)perylene (Fig. 10) were all eluted from the column in the second 25-ml fraction of the 3 per cent ether solution.

The bands at 231, 241, 273, 318 and 335 nm in the spectrum of the pyrene fraction (Fig. 7) are attributed to pyrene. The spectrum of the purified fraction (after thin-layer chromatography) shows extraneous peaks at 282 and 286 nm (Fig. 12) compared with the pyrene standard. These bands may be due to a derivative of pyrene. The intense band at 286 nm may also indicate the interference of fluoranthene.[6]

The R_f value of the isolated hydrocarbon is close to pure pyrene but its fluorescent color is very different (Tables 1 and 2). It could be some derivative of pyrene but

unfortunately standards of pyrene derivatives were not available for comparison.

Benz(a)anthracene was eluted from the column in the last fraction of the n-pentane eluate as indicated by the intense band at 287 nm in Figure 9. The absorption bands at 256, 267, 277, 287, 300, 340 and 358 nm of the isolated benz(a)anthracene match that of the standard benz(a)anthracene (Fig. 13). The presence of the weak band at 384 nm indicates a high concentration of the hydrocarbon.[6]

The absorption spectrum of the benzopyrene fraction in n-pentane after column chromatography (Fig. 10) indicates the presence of benzo(a)pyrene with bands at 377 and 383 nm, benzo(b)fluoranthene at 289 and 302 nm, perylene at 253, 428 and 434 nm and benzo(g,h,i)perylene at 289, 301 and 313 nm.

Figure 14 shows the ultraviolet spectra of the isolated benzo(a)pyrene and the standard. The set of three peaks at 377, 380 and 383 nm is characteristic of benzo(a)-pyrene. A comparison of their absorption bands at 255, 265, 272, 284, 296, 345, 363, 402, 420 and 428 shows that benzo-(a)pyrene was completely separated from other hydrocarbons present in airborne particulates. The difficulty of isolating benzo(a)pyrene from hydrocarbons such as benzo(k)-fluoranthene, benzo(e)pyrene and benzo(g,h,i)perylene has been reported by Sawicki[10,17] and Dubois.[27]

Benzo(b)fluoranthene (Fig. 15) has a maximum absorption peak at 302 nm and two characteristic pairs of peaks. One pair occurs at 289 and 293 nm and the other at 240 and

246 nm. At lower wavelengths, between 240 and 310 nm, the absorption bands of the isolated benzo(b)fluoranthene correspond to that of the standard. At higher wavelength the difference between the two spectra is pronounced. The presence of peaks at 421 and 429 nm in the spectrum of the benzo(b)fluoranthene fraction may due to interference of benzo(a)pyrene since they were eluted from the column in the same fraction of the 3 per cent ether in n-pentane eluent. The R_f value and the fluorescent color indicate that this fraction is benzo(b)fluoranthene.

A typical spectrum of perylene is shown in Figure 16 where intense bands occur at 253, 407, 428 and 434 nm. The absorption spectrum of the isolated perylene is not very similar to that of the standard but its R_f value and fluorescent color agree with the perylene standard.

The ultraviolet spectrum of benzo(g,h,i)perylene illustrated in Figure 17 is similar to benzo(a)pyrene (Fig. 14). They have common peaks at 345 and 383 nm but benzo(a)pyrene has a peak at 402 nm which is absent from the spectrum of benzo(g,h,i)perylene.

The isolated benzo(g,h,i)perylene and the standard have identical absorption bands at 288, 300, 330, 345, 363 and 383 nm.

The separation of coronene by thin-layer chromatography from the other polycyclic hydrocarbons was different in the sense that another type of adsorbent and solvent systems were used. The adsorbents were aluminum oxide and

cellulose acetate (2:1). The first development was by cyclohexane saturated with N,N-dimethylformamide followed by aqueous N,N-dimethylformamide saturated with ether in the second dimension.[15] These adsorbents and solvent systems were discarded because no other hydrocarbons were identified.

Coronene has a very intense band at 302 nm as can be seen in Figure 18. The other absorption bands common to both the standard and the isolated coronene occur at 289, 296, 317, 322, 333, 338 and 344 nm.

There were other characteristic ultraviolet absorption spectra that were recorded but they could not be identified because only a few polycyclic aromatic hydrocarbon standards were available.

This preliminary investigation of the polycyclic hydrocarbons present in Youngstown air should be followed by a more extensive analysis of the air pollutants secreted by the industrial plants located in the city as well as those emitted by vehicles. A quantitative determination of the amount of these hydrocarbons is imperative considering that some of them are carcinogenic.

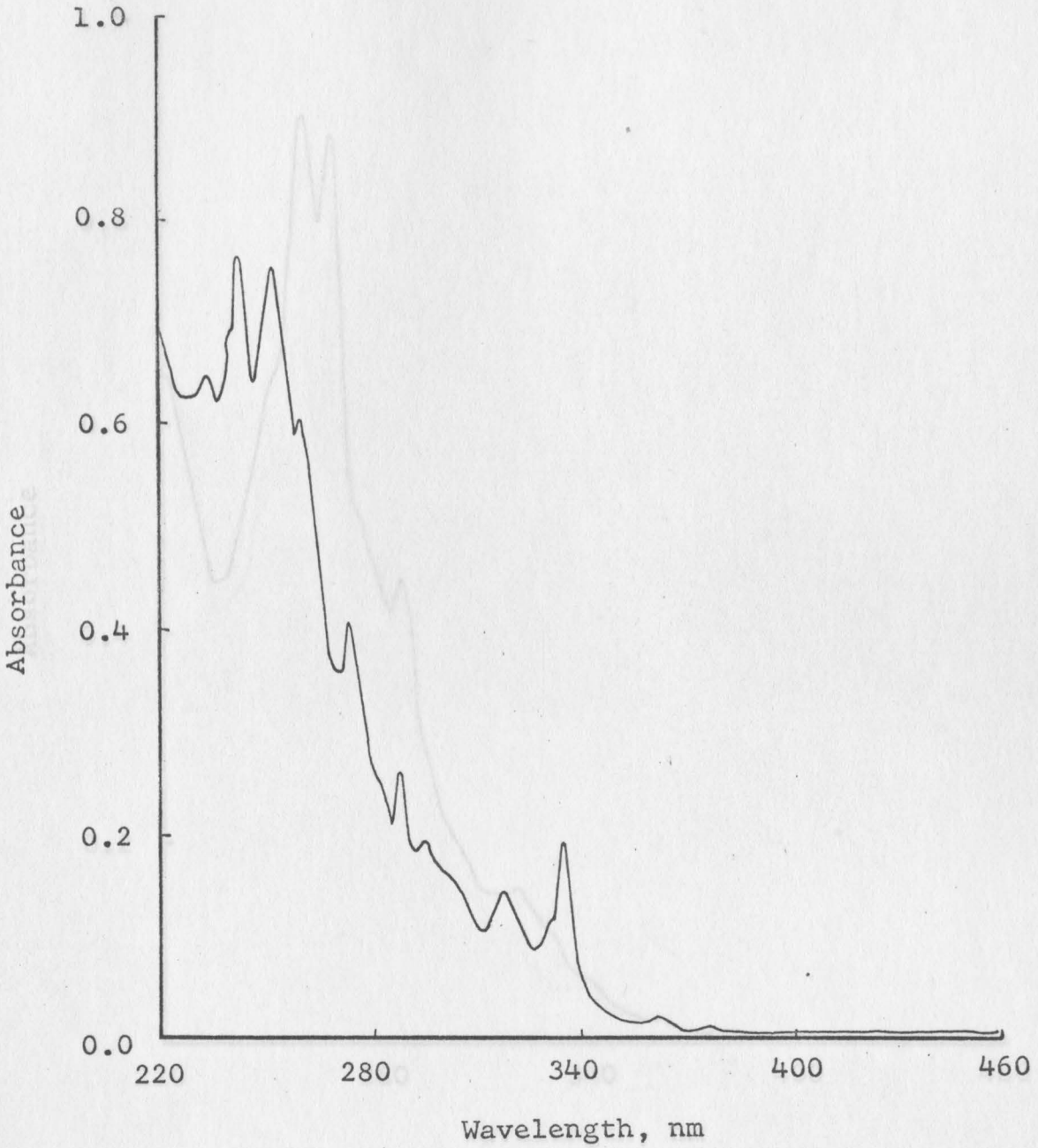


Fig. 7. Ultraviolet spectrum of the pyrene fraction in n-pentane after column chromatography.

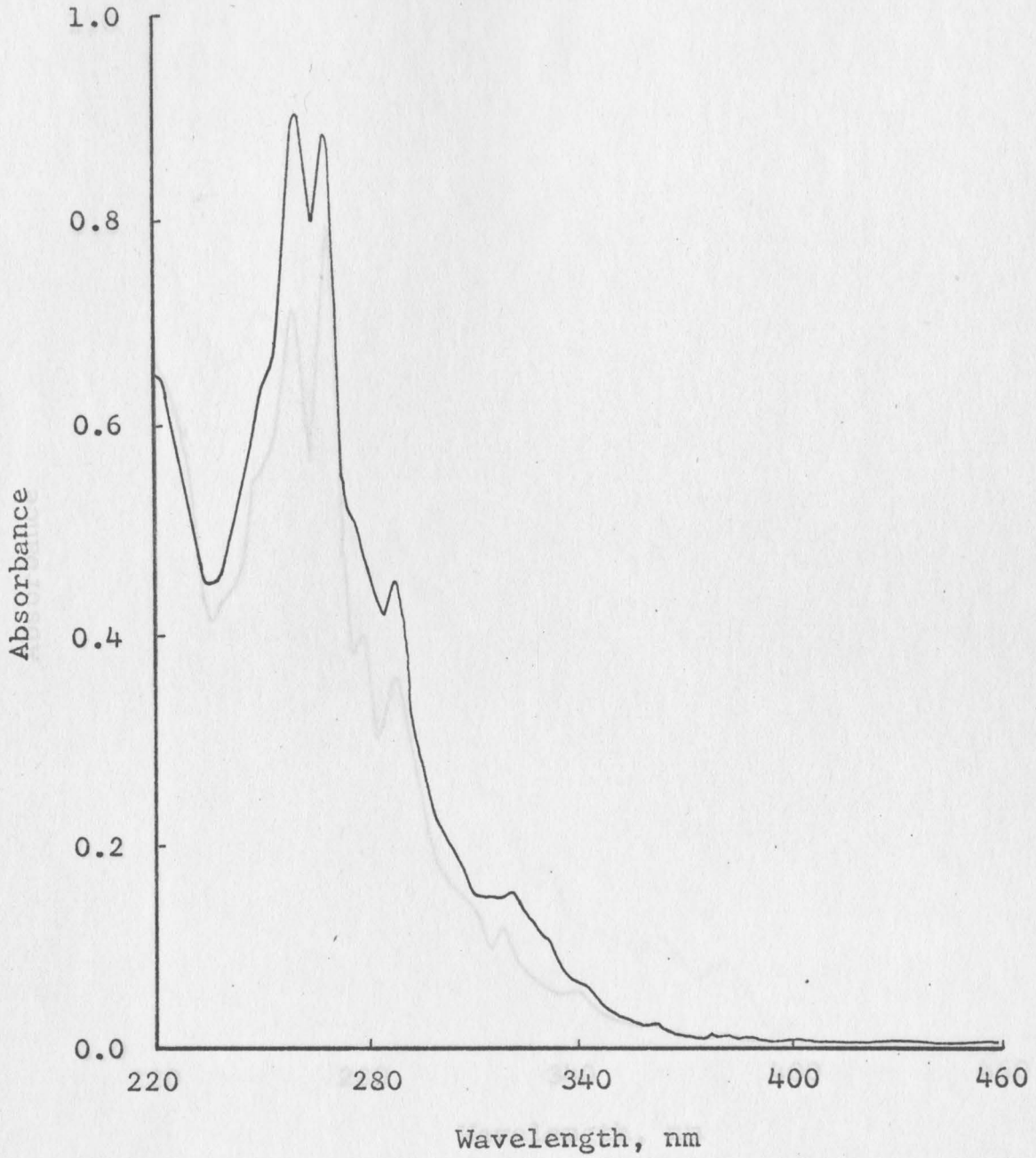


Fig. 8. Ultraviolet spectrum of the chrysene fraction in n-pentane after column chromatography.

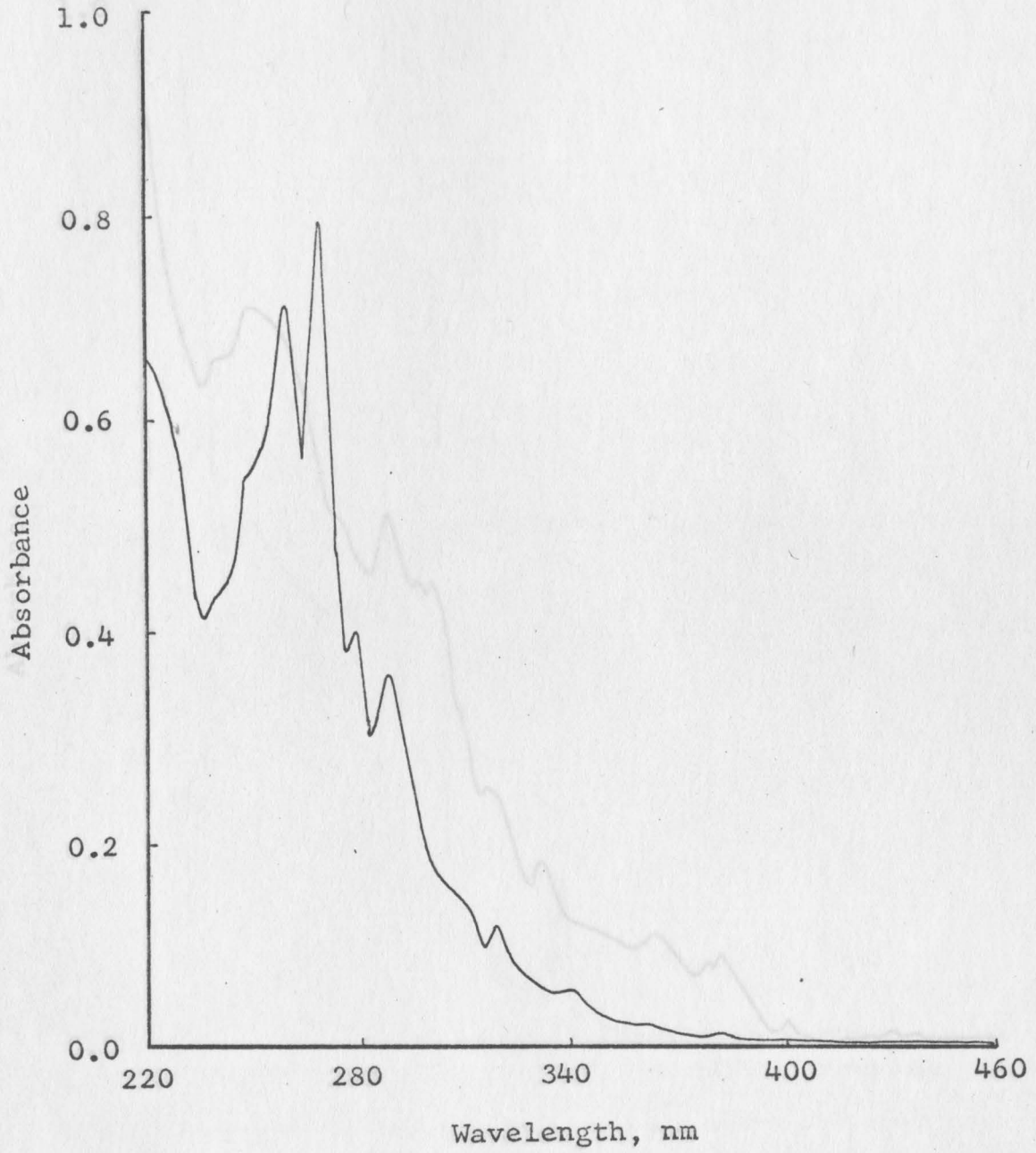


Fig. 9. Ultraviolet spectrum of the benzanthracene fraction in n-pentane after column chromatography.

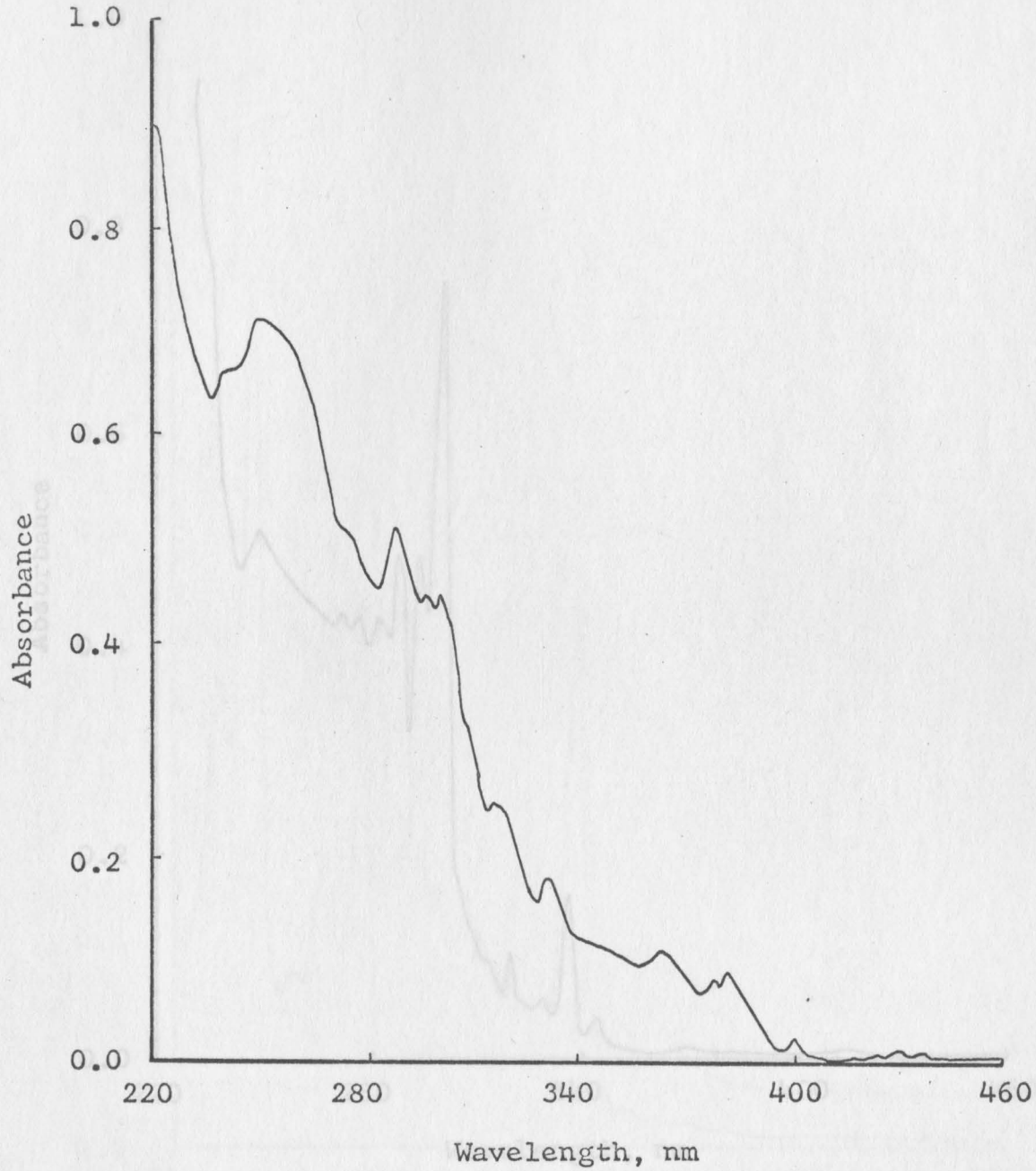


Fig. 10. Ultraviolet spectrum of the benzopyrene fraction in n-pentane after column chromatography.

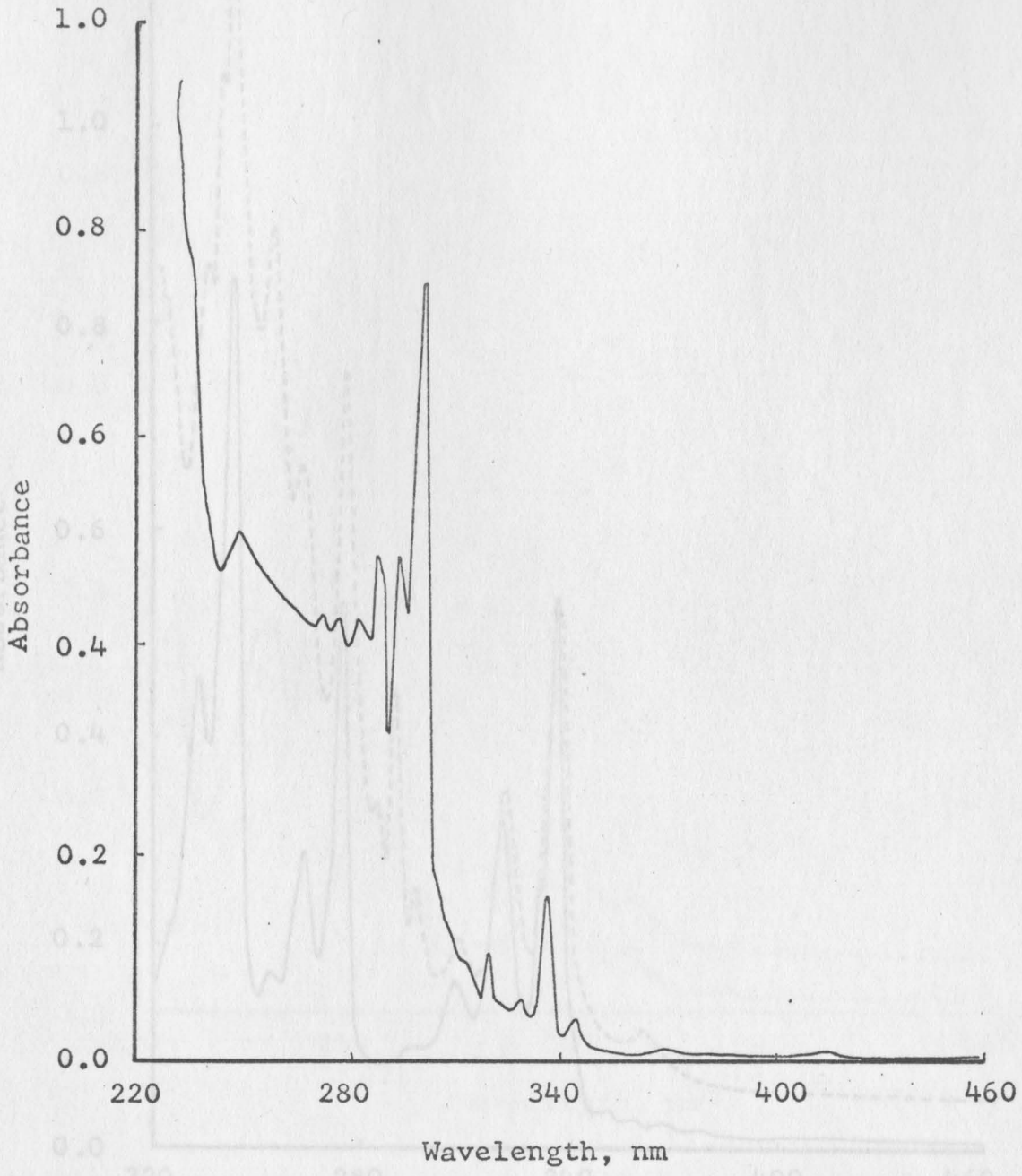


Fig. 11. Ultraviolet spectrum of the coronene fraction in n-pentane after column chromatography.

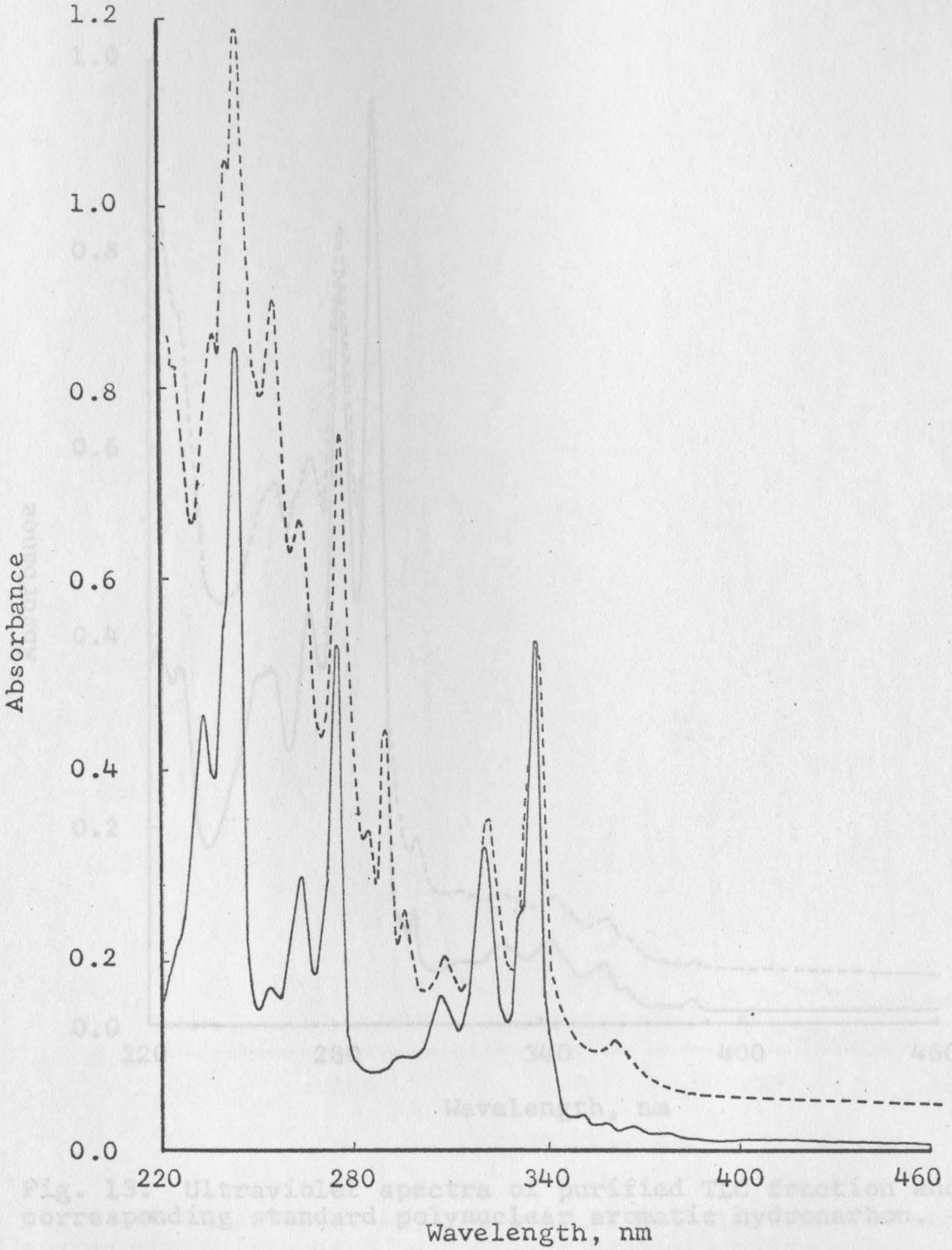


Fig. 12. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) pyrene fraction
(———) pyrene standard

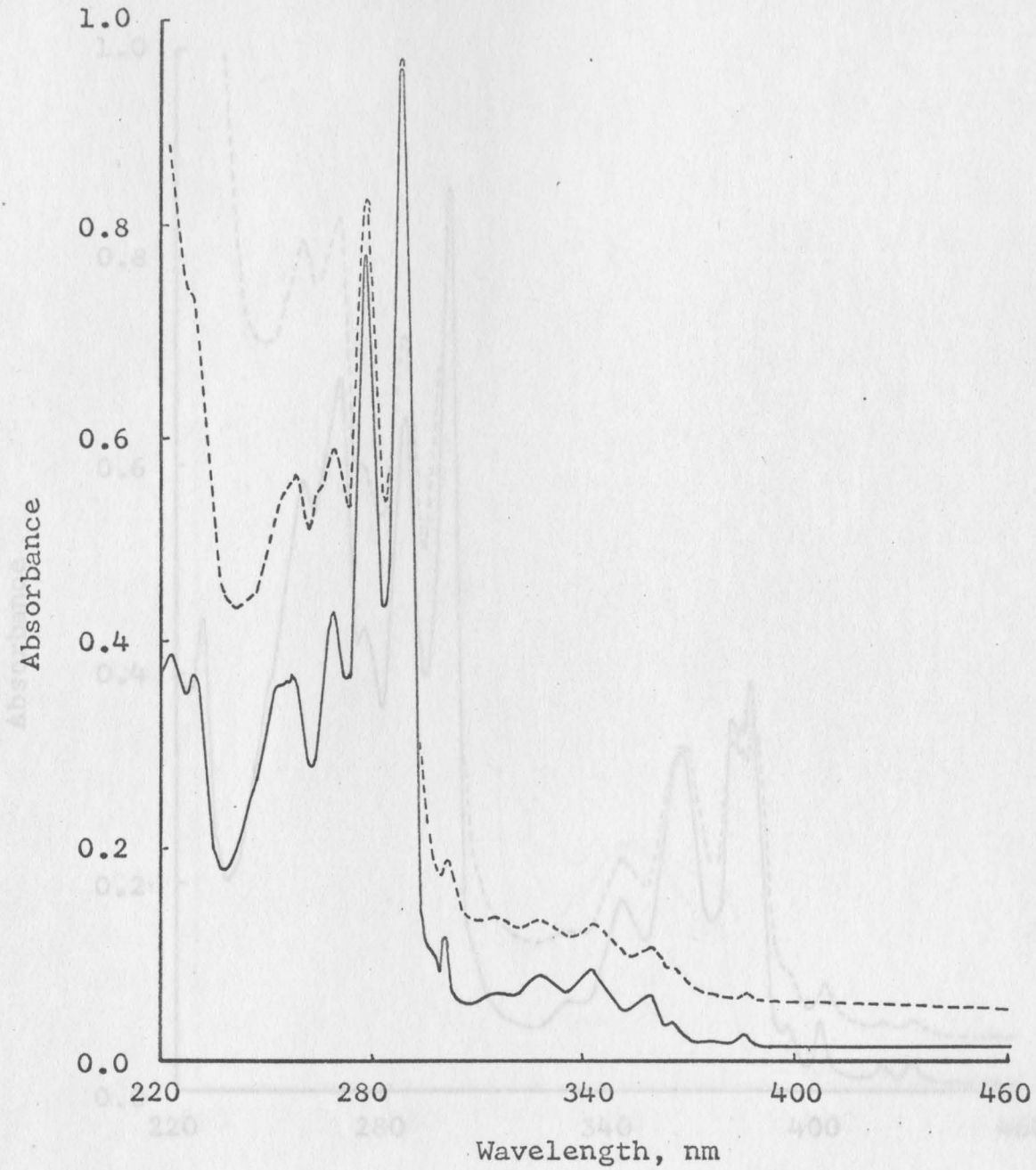


Fig. 13. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) benz(a)anthracene fraction
(———) benz(a)anthracene standard

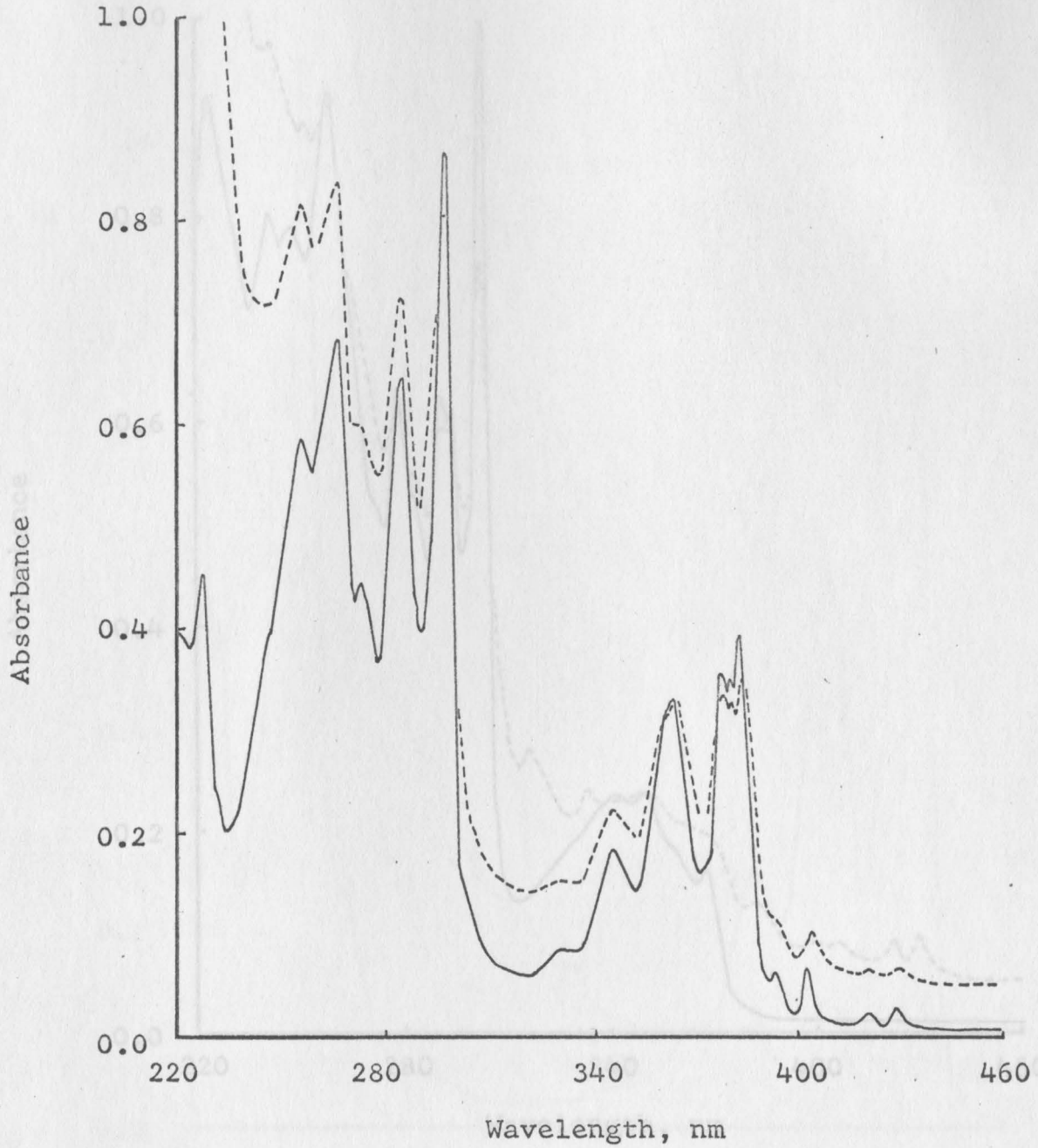


Fig. 14. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbons.

(-----) benzo(a)pyrene fraction
(———) benzo(a)pyrene standard

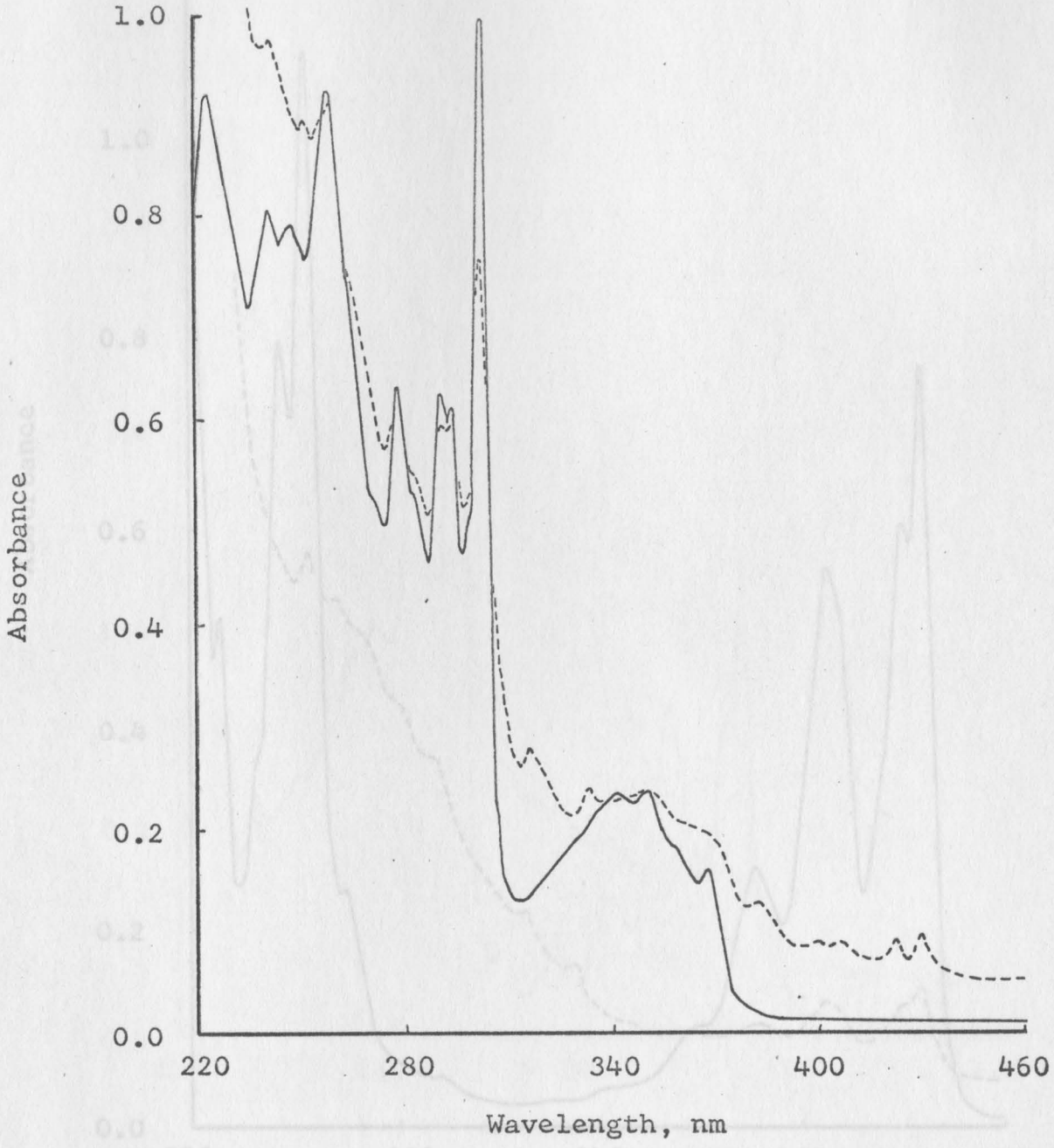


Fig. 15. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) benzo(b)fluoranthene fraction
(———) benzo(b)fluoranthene standard

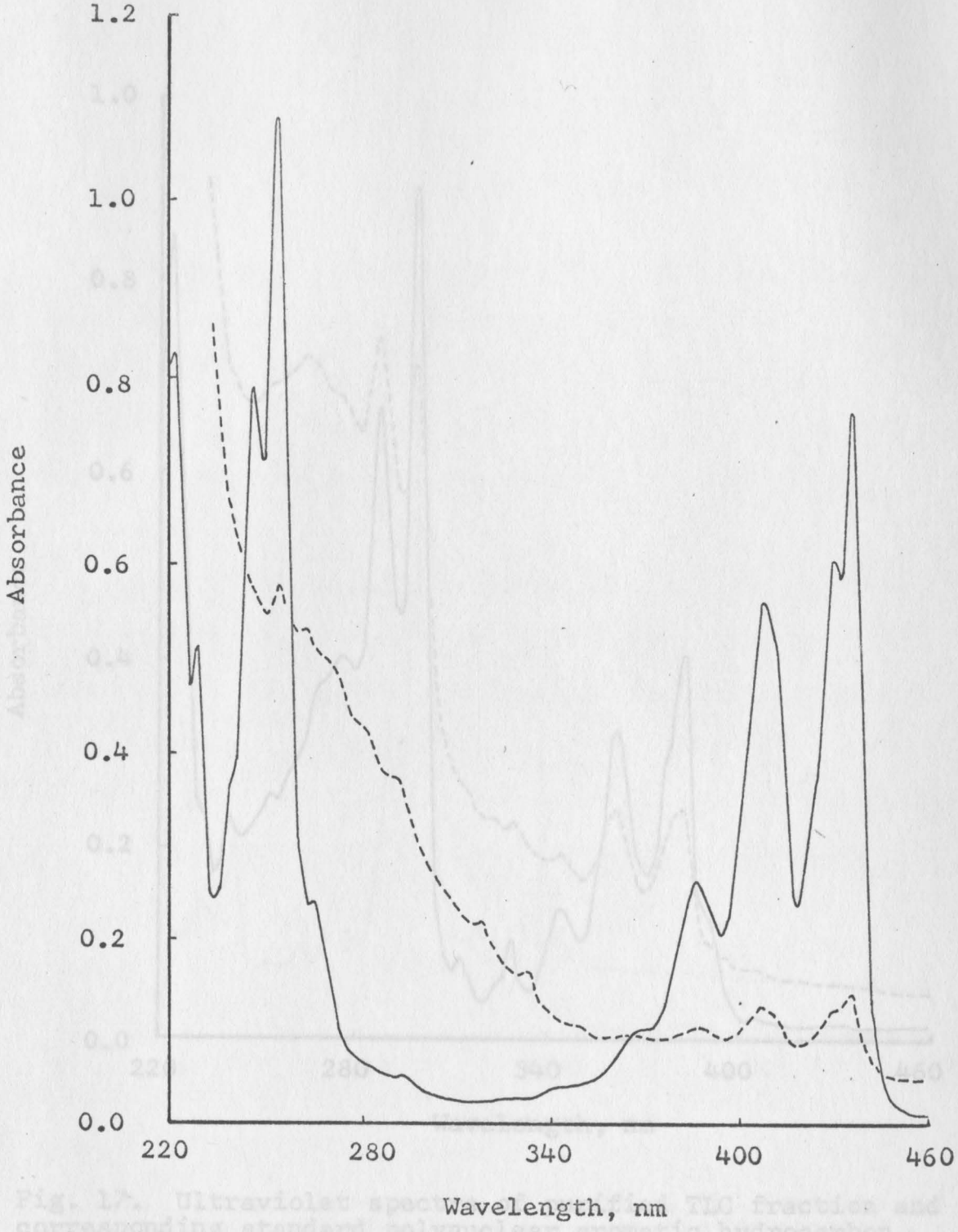


Fig. 16. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) perylene fraction
(———) perylene standard

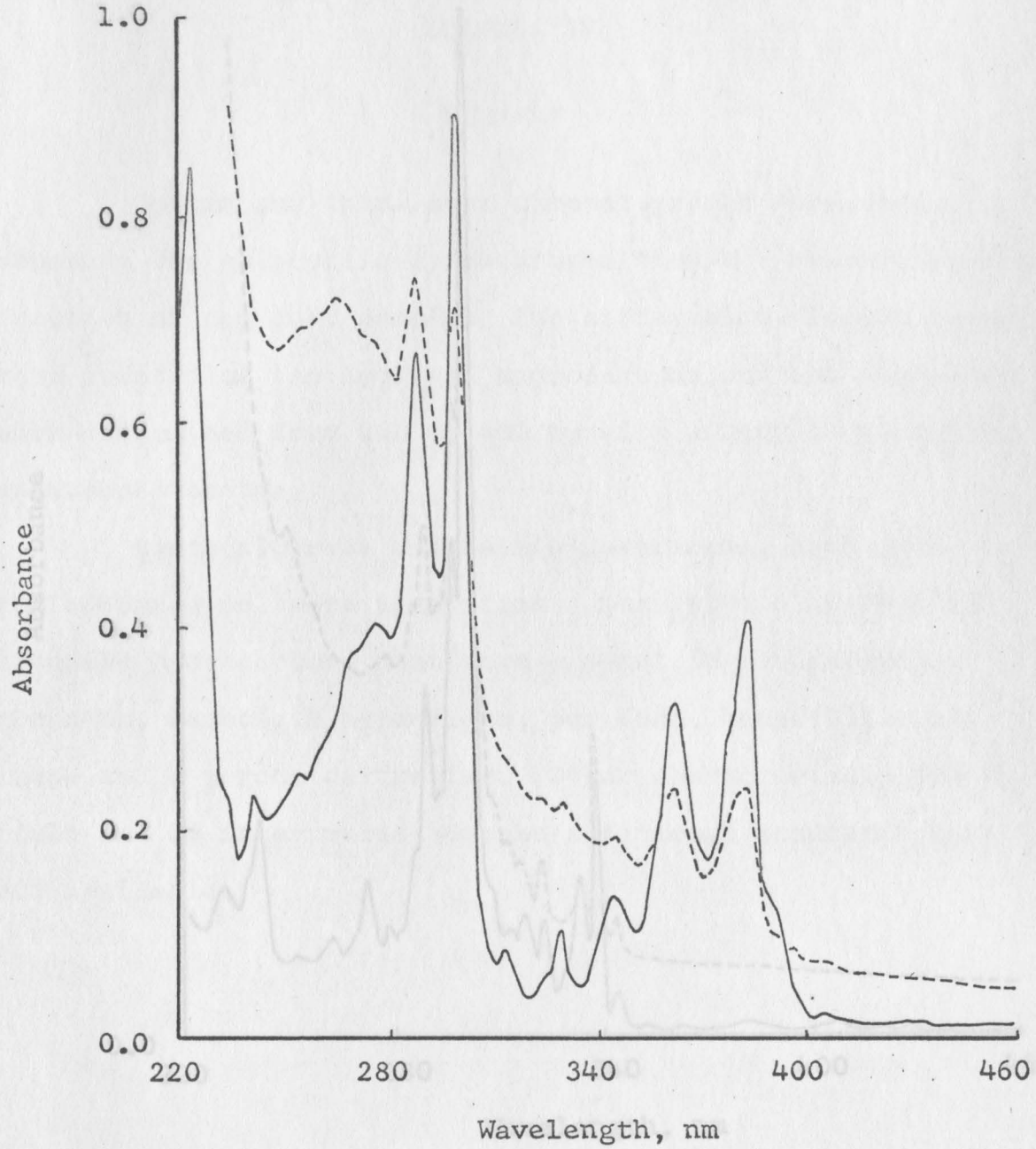


Fig. 17. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) benzo(g,h,i)perylene fraction
(———) benzo(g,h,i)perylene standard

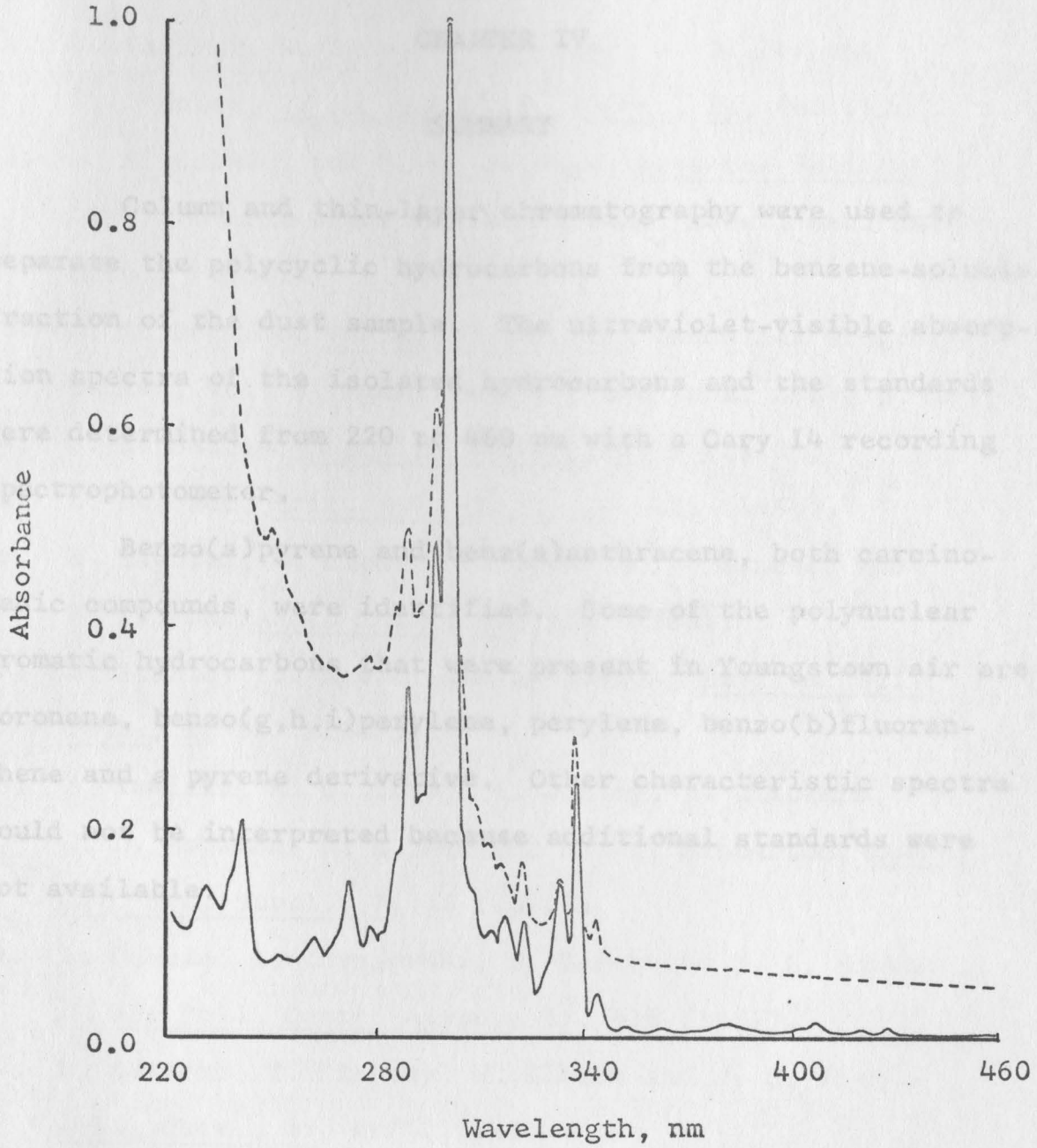


Fig. 18. Ultraviolet spectra of purified TLC fraction and corresponding standard polynuclear aromatic hydrocarbon.

(-----) coronene fraction
(———) coronene standard

CHAPTER IV

SUMMARY

Column and thin-layer chromatography were used to separate the polycyclic hydrocarbons from the benzene-soluble fraction of the dust sample. The ultraviolet-visible absorption spectra of the isolated hydrocarbons and the standards were determined from 220 to 460 nm with a Cary 14 recording spectrophotometer.

Benzo(a)pyrene and benz(a)anthracene, both carcinogenic compounds, were identified. Some of the polynuclear aromatic hydrocarbons that were present in Youngstown air are coronene, benzo(g,h,i)perylene, perylene, benzo(b)fluoranthene and a pyrene derivative. Other characteristic spectra could not be interpreted because additional standards were not available.

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