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Chromatography and 77°K luminescence of some hydrocarbons on thin layers of microcrystalline nylon–polytetrafluoroethylene (Aviamide-6–Fluoroglide 200)*

The separation, identification, and quantitative measurement of hydrocarbons is of particular interest in studies of air pollutants, tobacco-smoke components, and chemical carcinogenesis. Low-temperature luminescence has been shown to be a sensitive means to detect organic compounds on thin-layer chromatograms[1]. This technique was used to evaluate new layer materials for thin-layer chromatography (TLC). The evaluation of particulate polytetrafluoroethylene as a layer material for TLC and its usefulness for separating metal ions have been described[2,3]. A mixed layer that consists of the polytetrafluoroethylene Fluoroglide 200 and the new microcrystalline nylon Aviamide-6 has been found suitable for separating hydrocarbons.

Ten hydrocarbons (aromatic and heterocyclic) and a mixture that contained the ten were chromatographed on thin layers of Aviamide-6–Fluoroglide 200 (4:1) developed with *n*-propanol. The resulting chromatograms were observed with 254- and 366-nm UV light under liquid nitrogen (77°K). Eight of the ten hydrocarbons were resolved. Presumably, the separation is based on the differences in the solubilities of the hydrocarbons in *n*-propanol. The hydrocarbons show distinctive luminescent properties by which they can be identified; even the unresolved hydrocarbons were detectable in the presence of each other. The intensity of the phosphorescence of certain of the compounds is particularly striking and possibly may be a means to their sensitive quantitative measurement.

Materials

Fluoroglide 200, chromatography grade TWO218 from Chemplast, Inc., 150 Dey Road, Wayne, N.J. 07470.

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Aviamide-6, from Chemical Research and Development Center, FMC Corporation, Box 8, Princeton, N.J. 08540. The Aviamide-6 is available as an aqueous paste (40–45 % solids) and as a dry powder. The dry powder was found to be more satisfactory for TLC and was used in this work.

Hydrocarbons. The following hydrocarbons, from the sources indicated, were used:

Hydrocarbons	*Source*
o-Hydroxydiphenyl	Eastman Kodak Co.
Naphthalene	J. T. Baker Chemical Co. ("Baker's Analyzed")
Fluorene	V. F. RAAEN, ORNL Chemistry Division
Phenanthrene	Matheson, Coleman and Bell (reagent 3468)
Pyrene	Eastman Kodak Co.
Chrysene	Aldrich Chemical Co., Inc. (reagent 121971)
Anthracene	V. F. Raaen (scintillation grade; purified)
Fluoroanthene	Eastman Kodak Co.
Benzo-*e*-pyrene (1,2-benzopyrene)	Aldrich Chemical Co., Inc. (reagent 042071)
Benzo-*g,h,i*-perylene (1,12-benzoperylene)	Aldrich Chemical Co., Inc. (reagent 020981)

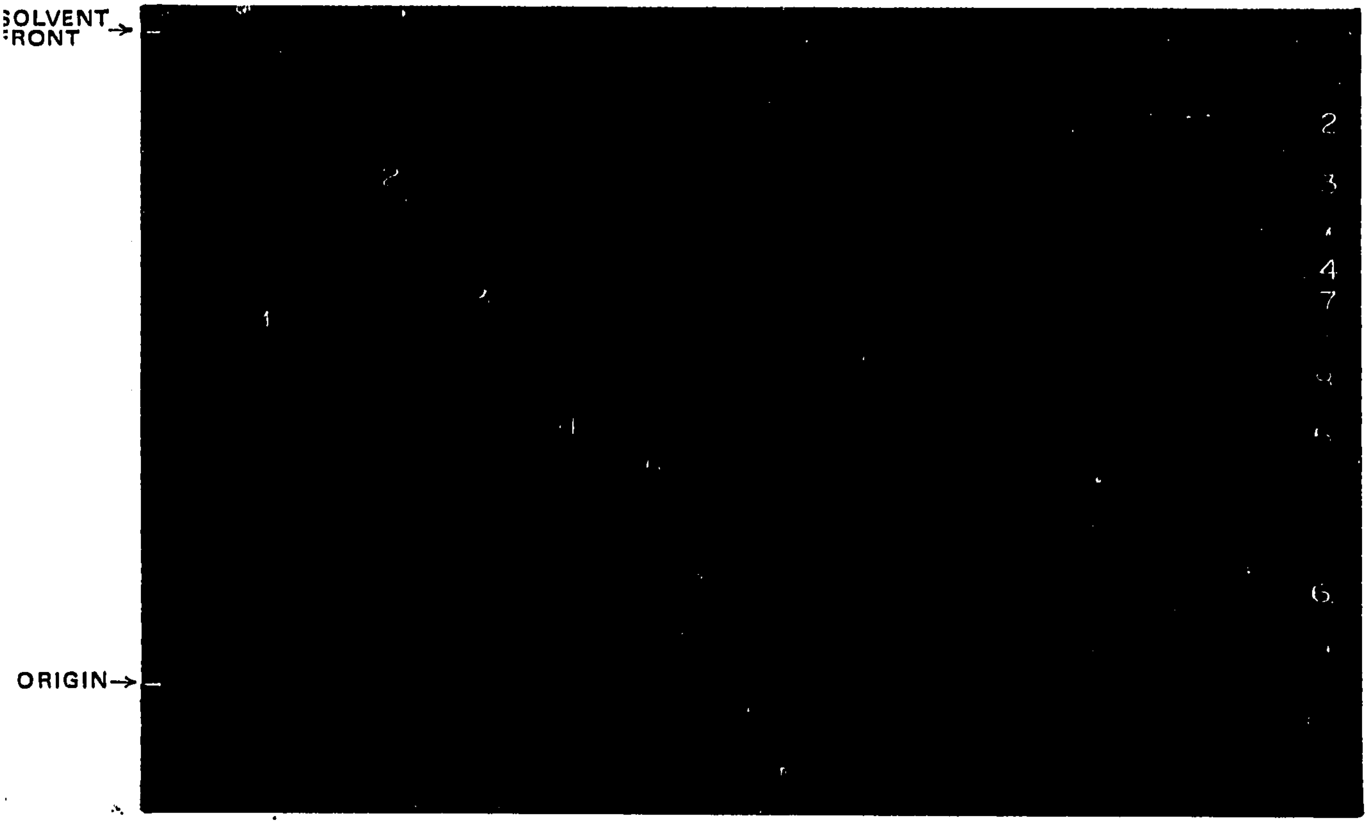

Fig. 1. Color photograph of chromatogram of hydrocarbons on Aviamide-6–Fluoroglide TWO218 (4:1) under liquid nitrogen (77°K) after excitation with 254-nm UV light. 1 = *o*-hydroxydiphenyl; 2 = naphthalene; 3 = fluorene; 4 = phenanthrene; 5 = pyrene; 6 = chrysene; 7 = anthracene; 8 = fluoranthene; 9 = benzo-*e*-pyrene; 10 = benzo-*g,h,i*-perylene.

For each of the hydrocarbons, a benzene solution was prepared. Its concentration was ~ 0.5 μg/ml except that of naphthalene, which was ~ 5 μg/ml. A benzene solution of a mixture of the hydrocarbons was also prepared that contained these same concentrations of the hydrocarbons. The other materials used either were reagent-grade chemicals or have been described earlier[1-3].

Procedures

Preparation of slurry. The slurry was prepared by adding 20.0 g of Aviamide-6 powder and 5.0 g of Fluoroglide 200 TWO218 to 75 ml of *n*-propanol and mixing the components on an orbital sander for about 1 min. This amount of slurry was suitable for preparing two 8 × 8 in. chromatofilms.

Preparation of chromatofilms. The slurry was layered onto two 8 × 8 in. sheets of 0.0075-in.-thick Mylar film as follows. The sheets were placed on a plate-glass support. To establish layer thickness, a row of three microscope slides was placed flush with each of two parallel sides of each film sheet. The slurry was poured onto the films and was spread manually with a 1/4-in.-diam. glass rod. The layers were air-dried at room temperature until they were evenly opaque.

Preparation of chromatograms. Volumes of the benzene solutions of the hydrocarbons were spotted on the layers with 1-μl Lang-Levy type pipets. The chromatograms were developed at room temperature with *n*-propanol in a standard Desaga developing tank, saturated with *n*-propanol, to a distance of about 16 cm from the origin. They were removed from the chamber and were dried at ambient conditions.

TABLE I

RESPONSE OF CHROMATOGRAPHED HYDROCARBONS TO UV LIGHT[a]

Hydrocarbon	*Ambient temperature*		*77° K*			
	Fluorescence		*Fluorescence*		*Phosphorescence*	
	254 nm	*366 nm*	*254 nm*	*366 nm*	*254 nm*[b]	*366 nm*
o-Hydroxy diphenyl	Brilliant violet	Violet	White	Pale violet	Pale yellow	Green white
Naphthalene	Dull purple	Dull purple	Purple (yellow ring)	Pale flesh	Deep yellow	Pale yellow
Fluorene	Orchid (yellow ring)	Dull orchid	White	Pale flesh	Blue	Pale yellow
Phenanthrene	Brilliant violet	Brilliant purple	Violet (yellow ring)	Violet	Bright yellow	Brilliant yellow orange
Pyrene	Brilliant white	Brilliant white	Brilliant white	Brilliant blue white	(Absorbs)	(Absorbs)
Chrysene	Brilliant purple	Brilliant purple	Orchid	Orchid	Yellow	Yellow
Anthracene	Violet	Bright violet	Pale orchid	Pale orchid	(Absorbs)	(Absorbs)
Fluoranthene	Brilliant blue white	Brilliant blue white	Brilliant white	Brilliant white	Red orange	Orange
Benzo-*e*-pyrene	Brilliant orchid white	Brilliant orchid white	Violet	Pale purple	Orange	Orange
Benzo-*g,h,i*-perylene	Brilliant green white	Brilliant green white	Brilliant green white	Brilliant blue white	(Absorbs)	(Not detectable)

[a] Under fluorescent room light, the chromatograms showed no spots, neither at ambient temperature nor at 77° K.

[b] See Fig. 1.

Analysis and photography of chromatograms. The chromatograms were observed with fluorescent room light and with 254- and 366-nm UV light. The observations were made both at room temperature and under liquid nitrogen[1]. To obtain an accurate record of the observation, the chromatograms were photographed under 254- and 366-nm UV light in air at room temperature and under liquid nitrogen[1]. Both the fluorescence and phosphorescence were photographed. The techniques for observing the chromatograms under liquid nitrogen and for photographing them have been described earlier[1].

Results and discussion

Table I is a chart of the responses of the hydrocarbons to the various conditions under which the chromatograms were observed. Fig. 1 is a photograph of the phosphorescence of the compounds under liquid nitrogen after exposure to 254-nm UV light. Unfortunately, the total brilliance of the phosphorescence was not captured in the photograph.

The chromatographic properties of the layers were excellent. They adhered well to the Mylar film, spotted easily, and gave straight solvent fronts. It may be of interest to note that, in experiments separate from those described above, gradient layers of Aviamide-6–Fluoroglide 200 were evaluated, both in the horizontal and vertical orientations. Separations of the hydrocarbons were also effected on the gradient layers.

Observation of the low-temperature (77° K) luminescence of the hydrocarbons permits the detection of unresolved hydrocarbons in the presence of one another.

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