

pared to neutral Silica Gel G. Basic alumina exerted a powerful binding effect on the molecules. The introduction of more alkyl groups in the aromatic solvent gradually decreased the R_F values of the esters.

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Low temperature fluorescence detection of organic compounds on thin layer chromatograms

Low temperature detection of certain compounds by their fluorescence or phosphorescence was first considered by SZENT-GYÖRGYI¹. He found that many compounds which did not fluoresce at room temperature, would fluoresce at liquid nitrogen temperature. This phenomenon has been employed for the detection of some organic compounds on paper chromatograms¹⁻⁴. WEISS⁵ stated that intense fluorescence may be expected of compounds which are symmetrically conjugated or which do not yield stronger ionic structures. According to TOMASCHEK⁶, intensification of fluorescence is usually due to —OH, —OCH₃, =CH₂, —NH₂ and —CN groups, whereas its weakening is due to the —COOH radical. RADLEY AND GRANT⁷ found that although there may be weakening of fluorescence due to the presence of certain groups, the position of the main bands remained unaltered. Also, the presence of unsaturated groups in the side chain tends to intensify fluorescence.

Many workers have investigated the effect of chemical structure on fluorescence but no complete set of theoretical rules has so far been devised. The successful adaptation of the technique to thin layer chromatographic detections is now reported.

The separatory technique as such need not be altered, of course, but the soft glass plates generally distributed as thin layer supports shatter readily when subjected to liquid nitrogen. A pyrex support plate proved satisfactory at the low temperature

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experienced, although with the innovation of instant thin layer chromatograms*, it is possible to use these also.

1-3 μ l of a 1% w/v solution of a sample in a suitable volatile solvent was applied to the plate with the aid of a syringe. After the solvent had volatilized, the plate was placed in a shallow polystyrene box and viewed under a U.V. light. As a U.V. source, a miniera light U.V.S. 12** which provides an intensity of 2537 Å at 18 in. from its subject was used. After recording the results, liquid nitrogen was then poured upon the plate, completely submerging it, again the plate was observed under U.V. light and the results recorded. A number of different organic compounds were looked at and the results are tabulated in Table I. Since no chemical reaction occurs

TABLE I
COMPARATIVE FLUORESCENCE DATA

	<i>Compound</i>	<i>U.V. at room temp.</i>	<i>U.V. at liquid N₂ temp.†</i>
1	Acetophenone	c	f, g
2	Acetylsalicylic acid	a	b
3	Anisaldehyde	f	d
4	Anisole	b	c
5	Benzaldehyde	a	d, g
6	Benzalacetophenone	a	b
7	Benzimidazole	b	c
8	Benzoic acid	c	c
9	Benzophenone	b	c
10	Benzylic acid	c	c
11	Caffeic acid	b	c
12	<i>d</i> -Carvone	e	f
13	<i>l</i> -Carvone	e	f
14	Catechol	a	e
15	Cinnamic acid	f	f
16	Crotonaldehyde	a	d, g
17	Dibenzyl	a	c, g
18	Diphenylacetic acid	b	c
19	Ethyl isothiocyanate	a	b, g
20	Ethyl myristate	a	b, g
21	Ethyl oleate	b	c
22	Ethyl palmitate	a	b, g
23	Ethyl salicylate	d	d
24	Fluorene	b	d, g
25	Fumaric acid	e	e
26	Furfural	e	f, g
27	Hydrocinnamic acid	a	a
28	4-Hydroxybenzoic acid	b	c
29	Lactic acid	e	e
30	Lauric acid	a	a
31	Maleic acid	e	e
32	Malic acid	a	b
33	2-Methoxybenzoic acid	e	c
34	2-Methyl toluate	b	e

(continued on p. 281)

* I.T.L.C. Gelman Instrument Co., Ann Arbor, Mich. Eastman Chromatogram Set Distillation Products Industries, Rochester, N.Y.

** Ultraviolet Products Inc., San Gabriel, Calif.

TABLE I (continued)

Compound	U.V. at room temp.	U.V. at liquid N ₂ temp.†	
35	4-Methyl toluate	b	e
36	Naphthalene	a	c, g
37	1-Naphthol	e	f
38	2-Naphthol	e	f
39	Nitrobenzene	a	e
40	4-Nitrobenzoic acid	f	f
41	Oleic acid	c	c
42	Oxalic acid	a	b
43	Palmitic acid	b	c
44	Phellandrene	e	f
45	Phenanthrene	e	c, g
46	Phenol	a	e
47	Phloroglucinol	a	e
48	Phthalic acid	f	f
49	Phthalic anhydride	a	e, g
50	Resorcinol	a	e
51	Salicylic acid	d	d, g
52	Stearic acid	a	b
53	Stibene	c	d, g
54	Succinic acid	a	a
55	Salicylaldehyde	f	f
56	<i>d</i> -Tartaric acid	a	a
57	Thiomalic acid	b	c
58	Urea	a	b
59	Vanillin	e	c

† Key: a = not detectable; b = faint fluorescence; c = moderate fluorescence; d = strong fluorescence; e = weak absorption; f = strong absorption; g = phosphorescence.

during this detection, the spots found by this technique may be removed from developed plates and submitted to quantitative estimation. The authors feel that fluorescence and phosphorescence, owing to the closing of the energy levels of the organic compounds at these low temperatures, gives a more favourable method of detection than the technique of using a U.V. light at room temperature.

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