pounds into line with the corresponding waters and ammonias.

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Chromatography of Some Common Indicators

BY J. V. KARABINOS[1] AND P. M. HYDE

Alumina,[2] calcium carbonate, talc, Super Cel and Hyflo Super Cel have been used successfully as adsorbents for the chromatography of coal tar dyes and indicators.[3] We wish to add to this list Silene EF[4] which has already found extensive use in the carbohydrate series.[5]

The relative degree of adsorption of the indicators on Silene EF–Celite 535[6] (5:1 by weight) was determined as follows. The indicator (0.75 mg.) dissolved in 0.4 ml. of 90% dioxane (90 ml. dioxane–10 ml. water) was put on a Tswett column (10 × 100 mm.) previously wetted with 0.5 ml. of the solvent and the indicator was developed with 5.0 ml. of 90% dioxane. The position of the colored zone was easily determined since the length of the column was calibrated in mm. (0, bottom; 100 mm., top). Checks were obtained in duplicate runs. Comparative data on the relative adsorption of the indicators on Silene EF–Alumina–Celite 535 (5:5:2 by weight) are also presented.

TABLE I

RELATIVE DEGREE OF ADSORPTION OF SOME INDICATORS

Indicator	Position of zone, mm. (color) Silene EF	Position of zone, mm. (color) Silene EF–Alumina
Aurin tricarboxylic acid, ammonium salt	99–100 (Br)	99–100 (Br)
Indigo carmine	99–100 (Bl)	99–100 (Bl)
Alizarin red S	98–100 (R)	98–100 (R)
Alizarin yellow R	98–100 (Y)	98–100 (Y)
Methyl violet	68–100 (Bl)	60–93 (Bl)
Dichlorofluorescein	91–96 (O)	97–98 (Y-O)
Congo red	85–90 (R)	97–100 (O)
Brom phenol blue	73–80 (P)	95–99 (P)
Brom cresol green	62–70 (Bl)	96–99 (Bl)
Chlor phenol red	60–70 (P)	95–98 (P)
Brom cresol purple	15–25 (P)	65–75 (P)
m-Cresolsulfonphthalein	15–25 (Y)	57–63 (O)
Methyl orange	5–20 (Y)	51–60 (Y)
Tropaeolin 00	8–18 (Y)	45–55 (Y)
o-Cresolphthalein	8–18 (Y)	47–55 (Y)
Thymol blue	0–10 (Y)	25–35 (Y)
Cresol red	8–15 (Y)	48–52 (Y)
Brom thymol blue	0–4 (Y)	0–2 (Y)
m-Nitrophenol	0	0
Phenolphthalein	0	0

It is interesting to note that the highly polar dyes, *e. g.*, the sodium salts are more strongly adsorbed.

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[2] C. P. grade, J. T. Baker Chemical Company, Phillipsburg, New Jersey.

[3] P. Ruggli and P. Jensen, *Helv. Chim. Acta*, **18**, 624 (1934); **19**, 64 (1936); P. Ruggli and M. Stauble, *ibid.*, **23**, 689 (1940); H. H. Strain, THIS JOURNAL, **61**, 1292 (1939); W. Rieman, *J. Chem. Ed.*, **18**, 131 (1941).

[4] A commercial hydrated calcium silicate generously supplied by the Columbia Chemical Division, Pittsburgh Plate Glass Co., Barberton, Ohio.

[5] L. W. Georges, R. S. Bower and M. L. Wolfrom, THIS JOURNAL, **68**, 2169 (1946).

[6] Johns–Manville Co., New York, N. Y.

Preparation of Resacetophenone

BY JOSEPH R. KILLELEA AND H. G. LINDWALL

The zinc chloride method commonly employed for the preparation of resacetophenone[1] involves a vacuum distillation and yields 65% of colored product. Since the vacuum distillation of high-melting solids is a troublesome process, we have adopted a boron fluoride method which eliminates the necessity of the distillation and gives superior yields of colorless product. Details are given below.

A mixture of resorcinol, 55.0 g. (0.5 mole), acetic anhydride, 54.5 g. (50.0 ml., 0.53 mole) and commercial anhydrous ether, 75 ml., is saturated with gaseous boron fluoride in an ice-bath. The mixture is then stored in a refrigerator for ninety-six hours. The heavy, yellow crystalline mass is broken up with a glass rod and the contents of the flask transferred to a beaker containing 200 g. of crushed ice. The ether is distilled off on a water-bath or allowed to evaporate in a good hood. The light yellow solid is washed with cold water. It weighs 73.0 g. (96%) and melts at 138–142°. It may be recrystallized from 1200 ml. of boiling water (Norit), the recovery being 69.0 g. of colorless solid (91%) melting at 144–145°.

A mixed melting point of this product with a sample prepared by the zinc chloride method showed no depression. The dibenzoate, prepared by Baker's method[2] melted at 80–81° mixed with the dibenzoate prepared from Cooper's product, 80–81°.

The boron fluoride method exemplified above has been employed, with slight modifications, in this Laboratory for the preparation of other polyhydroxyacetophenones.

[1] Cooper, "Org. Syn.," **21**, 103 (1941).

[2] Baker, *J. Chem. Soc.*, 1384 (1933).

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Vanadium Oxide, a Hydrogenation Catalyst[1]

BY V. I. KOMAREWSKY, L. B. BOS AND J. R. COLEY

Vanadium oxide is widely used as an oxidation catalyst. It has also been reported to be an effective catalyst for the dehydrogenation of hydro-

[1] Presented before the Division of Petroleum Chemistry at the Atlantic City Meeting of the American Chemical Society, April 14, 1947.