member (36.32) and three for the 16-member (19.40). The fiber period of the copolyester (17.83) was taken from one sharp fiber pattern obtained at 6 cm.

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BELL TELEPHONE LABORATORY
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Some Halogenated Naphthoxyacetic Acids[1]—A Confirmation

BY C. ROBERT GEISER AND HOKE S. GREENE

The preparation of 2,4-dichloro-1-naphthoxyacetic acid, 1,6-dibromo-2-naphthoxyacetic acid, 4-chloro-1-naphthoxyacetic acid, and 2,4-dibromo-1-naphthoxyacetic acid reported by Haskelberg[2] has been independently confirmed by us using similar methods. Templeman and Sexton[3] had previously reported the melting point of 2,4-dichloro-1-naphthoxyacetic acid as 135° but we have found it to be 178° in agreement with Haskelberg.[2]

The aforementioned compounds showed selectivity as weed killers in 0.1% solution as a triethanolamine salt. However, at that concentration they were not as effective as 2,4-dichlorophenoxyacetic acid.

(1) From the M.S. thesis of C. Robert Geiser, University of Cincinnati, June, 1947.

(2) L. Haskelberg, *J. Org. Chem.*, **12**, 426 (1947).

(3) Templeton and Sexton, *Proc. Roy. Soc.* (*London*), **B133**, 300–313 (1946).

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF CINCINNATI
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Separation of Tropic and Atropic Acids by Partition Chromatography

BY SIDNEY GOTTLIEB

In the course of a study on the hydrolysis of alkaloids of the atropine group, it became necessary to separate tropic from atropic acids in a large number of hydrolysates. Although the solubility characteristics of these two acids permit their separation from one another by several steps involving fractional crystallization, a much more rapid and convenient method was developed using partition chromatography.

This technique, originally developed by Martin and Synge,[1] consists essentially of passing one of a pair of immiscible solvents over a film of the other, the latter solvent being adsorbed onto an inert lattice. The substances dissolved in the mobile solvent will then partition themselves between the two phases according to their partition coefficients in such a way as to form distinct bands. Since tropic and atropic acids differ significantly in their partition coefficients between water and chloroform, this pair of solvents was used, with precipitated silicic acid as the supporting lattice. An acid–base indicator in the form of an azo dye was incorporated into the aqueous phase and, as the acids moved down the column, their positions were clearly indicated by dark blue bands against a red background.

In several parallel determinations, the identity of the first fraction to come through the column was established as atropic acid by evaporating the solvent and recrystallizing the residue once from hot water, which yielded monoclinic prisms, m. p. 106.0–106.5° (cor.). That the material comprising the upper band was tropic acid was proved by evaporating the solvent and recrystallizing the residue twice from boiling benzene to yield needes, m. p. 116.0–117.0° (cor.).

Experimental

A typical column was prepared by intimately mixing 10 cc. of a 0.1% aqueous solution of 3,6-disulfo-β-naphthaleneazo-N-phenyl-α-naphthylamine[2] with 20 g. of dry precipitated silicic acid. In this investigation Eimer and Amend C. P. silicic acid, batch #403320, was used. The mixing was done with a mortar and pestle until the mixture had uniformly taken on the red color of the dye, and no lumpiness remained. A slurry of this preparation was made with 50 cc. of chloroform and the slurry poured into a glass tube having a diameter of 24 mm., constricted and plugged with cotton at one end. When enough chloroform had run through the column so that the suspension acted like a stiff gel on shaking, a mixture of 5 mg. each of tropic and atropic acids dissolved in 3 cc. of chloroform was pipetted into the column. After this solution had just passed the top of the column, 5 cc. of chloroform was added to wash any acid remaining on the sides of the column into the narrow band. Then 50 cc. of chloroform was run through the column to separate and develop the bands. The atropic acid, being more soluble in chloroform, moved rapidly down in a sharp band, usually being completely washed out by 40 cc. of chloroform. The upper band, containing the slower-moving tropic acid, which had moved about one-fourth of the way down the column, was then washed through with a mixture of 10% butyl alcohol in chloroform. Titration of the residues with *N*/50 sodium hydroxide indicated essentially quantitative recovery and separation of the two acids.

(1) Martin and Synge, *Biochem. J.*, **35**, 1358 (1941).

(2) Liddel and Rydon, *Biochem. J.*, **38**, 68 (1944).

CHEMICAL SECTION, MEDICAL DIVISION
FOOD AND DRUG ADMINISTRATION
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The Existence of Beta Cristobalite at Room Temperature

BY ALEXANDER GRENALL

Due to some unexplained circumstance, overheating of pelleted clay catalyst occurred in the regenerator kiln of a catalytic cracking unit to such a degree that some of the pellets were fused and glassy in appearance. On breaking open these pellets it was found that while the surfaces were glassy, the centers were not.

Independent X-ray diffraction examinations were made of the surface and center material. A complete analysis of the diffraction data from Debye–Scherrer photographs revealed, in addition to other phases, the presence of β-cristobalite in