

FRACTIONATION OF WOOL WAX BY LIQUID THERMAL DIFFUSION*

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Wool wax consists of a mixture, mainly of long-chain esters of cholesterol, lanosterol, and their dihydro-derivatives, together with di-esters of hydroxy acids.¹ The presence of free acids, cholesterol, and higher long-chain alcohols has been demonstrated by liquid chromatography,² but attempts to fractionate the esters have not been successful. The composition of the hydrolysis products has been investigated extensively, and in a recent and comprehensive study Downing, Kranz, and Murray³ gave the distribution of the acids, hydroxy acids, alcohols, and diols in the hydrolysed wax, according to their carbon number.

This communication is a preliminary description of the fractionation of wool wax by liquid thermal diffusion, a technique which is being applied in these laboratories to the fractionation of a variety of complex mixtures of organic compounds. An apparatus consisting of a vertical stainless-steel column similar to that described by Washall and Melpolder⁴ was used in the treatment of a sample of crude commercial centrifuged wool wax. The inner and outer walls of the annular space (0.012 in.) were maintained at 70 and 140° respectively, and after 48 hr six separate fractions (5 ml each) were collected from ports spaced at equal distances down the length of the column.

The top fraction was a relatively hard, white, sharply melting solid and that from the bottom of the column was a very viscous, dark greenish brown substance. The melting points and saponification values of the top three fractions, which varied in colour from white to very pale brown, are given in Table 1. The variation in molecular weight was not regular, but proton magnetic resonance and infrared data clearly showed that some separation according to molecular type had occurred. It is known⁵ that separation by thermal diffusion is mainly determined by molecular shape and that compounds of smaller molecular volume concentrate at the bottom of the diffusion column. The data show that the content of steroid-type groups increased and the number of long-chain aliphatic groups decreased in the fraction order 1 to 6 from the top to the bottom of the column, assuming that all the molecules contain carboxylic ester groups. In respect of melting point and saponification number the three top fractions combined appear to meet if not surpass the B.P. specification.⁶

The authors are indebted to Dr. S. Sternhell for estimating proton distributions by proton magnetic resonance spectroscopy.

* Manuscript received November 14, 1963.

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³ Downing, D. T., Kranz, Z. H., and Murray, K. E., *Aust. J. Chem.*, 1960, **13**, 80.

⁴ Washall, T. A., and Melpolder, F. W., *Industr. Engng. Chem., Process Design & Dev.*, 1962, **1**, 26.

⁵ Jones, A. L., and Milberger, E. C., *Industr. Engng. Chem.*, 1953, **45**, 2689.

⁶ British Pharmacopoeia, p. 716. (Pharmaceutical Press: London 1958.)

