VI. THE PREPARATION OF PURE BEHENIC ACID.

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As already pointed out (pp. 40, 49, 58 and 66) the behenic acid obtained from the hardened oils of rape, mustard, jamba and *Tropæoleum majus* seeds melts at 79·3°–79·8°. This does not agree with the melting point given by recent authorities and attempts have therefore been made to prepare behenic acid by different methods and see whether the melting points of different specimens of the acid melted much above 79·8°.

As early as 1846 Walter (*Annalen*, 1846, **60**, 271) gave the melting point at 52–53°, but in 1848 Völcker (*Ibid.*, 1848, **64**, 342) gave the value 76° and this was confirmed by Goldschmidt (*Wien Acad.*, 1874, **70**, 451). In 1894 Talanzeff (*J. pr. Chem.*, 1894, [II], **50**, 72) prepared the acid by the addition of hydrogen iodide to erucic acid and subsequent reduction in alcoholic solution with zinc and hydrogen chloride. The melting point is given as 84° in a capillary tube. Meyer, Brod and Skita (*Monatsh.*, 1913, **34**, 1128) prepared behenic acid by reducing pure erucic acid with hydrogen in the presence of nickel as catalyst and gave the melting point at 82–84°, but state that with a catalyst which has been used several times a product melting at 75° is obtained and that it is impossible to get a product melting at 84° from this. Flecker and Taylor (*J. Chem. Soc.*, 1922, **121**, 1102) used an acid melting at 81–82° and prepared by reducing erucic acid and subsequent crystallisation from alcohol, and Toyama (*J. Chem. Ind. Japan*, 1922, **25**, 1053) describes an acid obtained by the same method as melting at 81–82°.

It is thus clear that the acid obtained from the seeds melts at a lower temperature than the acid prepared by the reduction of erucic acid, although the values given for this reduced acid vary from 81–84°.

Behenic acid has been prepared by the following methods :—

1. The reduction of brassidic acid melting at 59·5–60·0° and with an equivalent weight 337 (theory) but unknown iodine value by three per cent. of nickel deposited on kieselguhr. After four hours at 180° the iodine value fell to 4 and the product was repeatedly crystallised from acetone. After two crystallisations the melting point was 79·3–79·8° and remained constant after further crystallisation.

2. Kahlbaum's pure erucic acid was twice crystallised from 95 per cent. alcohol and was once crystallised by cooling to 13° to remove saturated acids. About 10 per cent. of the product was precipitated as lithium salt, also with the object of removing saturated acids, and the residue was crystallised again from alcohol. The acid melted at 33·5–34·0° and had an equivalent 337. It was reduced at 180° using a nickel catalyst and on crystallisation from acetone gave a product melting at 79·3–79·8°.

3. Attempts were made to obtain from Kahlbaum's acid a product with the correct iodine value (75) by Holde's (*Angew. Chem.*, 1922, **35**, 290) method of fractional precipitation with lithium acetate. The iodine value obtained for the more soluble portions was only 71·5, and the behenic acid obtained from this by hydrogenation melted at 79·3–79·8°.

4. A fresh specimen of erucic acid obtained from Kahlbaum had the iodine value 75·4 and the solidifying point 33·0°. This was completely hardened, extracted with petroleum ether to remove nickel soap and the acid crystallised from 95 per cent. alcohol and acetone in succession. It melted at 80° and the solidifying point was 79·2°.

5. A sample of brassidic acid with an iodine value 73·8 (theory 75) and a melting point 59·5–60·0° gave on reduction an acid melting at 79·3–79·8° after two crystallisations from acetone.

6. As already stated, the acid obtained by hydrogenating trierucin to tribehenin, and subsequent hydrolysis melted at 79·5–80·0°.