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## NEW QUALITATIVE TESTS FOR RAPE AND TUNG OILS

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In the development of a new quantitative procedure for the estimation of peanut oil,<sup>2</sup> it was found that upon decomposition of the magnesium soaps from rape oil and dissolution of the resulting solid acids in 90% alcohol, an amount of less than 0.5% of acid insoluble in this solvent at 20° was obtained. This yield of insoluble acid, melting at 70°, is analogous to the so-called isolation of arachidic-lignoceric acid mixture from rapeseed oil by the "lead-salt-ether" method reported by Ponzio<sup>3</sup> and Archbutt.<sup>4</sup> Upon crystallization of this solid acid mixture *at* 25°, however, this precipitate was not obtained and hence would not interfere in the determination of peanut oil if the crystallization were carried out at this temperature.

Technique of the Test for Rapeseed Oil.—About 10 g. of rapeseed oil is saponified in a 300cc. Erlenmeyer flask under a reflux condenser by a mixture of 50 cc. of alcoholic potassium hydroxide<sup>5</sup> solution and 50 cc. of 95% alcohol. Upon the completion of saponification, the soap solution while still warm is neutralized with alcoholic acetic acid,<sup>6</sup> using phenolphthalein as indicator, and just enough alcoholic potassium hydroxide solution is then added to give a *permanent pink* color, whereupon 25 cc. of 0.8 M alcoholic magnesium acetate solution<sup>5</sup> is added. The mixture is heated to boiling, cooled and allowed to stand overnight in a refrigerator at about 10° for precipitation of the magnesium soaps.

The insoluble magnesium soaps are filtered off and washed with 30 cc. of 90% (by volume) alcohol at  $20^{\circ}$ . They are then transferred back into the flask and decomposed with 100 cc. of boiling 5 *M* hydrochloric acid. The decomposition is completed in about 5 minutes, when a clear oily layer has formed. After cooling, the fatty acid cake is filtered off and is washed free from magnesium and chloride ions with warm water.

The acid cake is dissolved with 60 cc. of 90% alcohol in a covered 150 cc. beaker which is allowed to stand overnight in a thermostat at  $20^{\circ}$  or  $15^{\circ}$ . Any crystals of insoluble acids which may have formed are then filtered off and discarded. The filtrate and washings are collected, evaporated to dryness, and the melting point, or acid number or preferably iodine number of the residue determined.

Two samples of genuine rapeseed oils were examined by this procedure; they yielded about 25% of the final acid product which had the following properties: m. p.,  $35^{\circ}$  (erucic acid,  $32^{\circ}$ ); mean mol. wt. about 328 (erucic acid, 338.45); iodine number, 70-72 (erucic acid, 74.99).

From the above data and also from the fact that the magnesium soaps

<sup>1</sup> Adapted from the thesis submitted by Chai-Lan Yu in partial fulfilment of the requirements for the degree of Doctor of Philosophy, in the Faculty of Pure Science, Columbia University.

<sup>2</sup> See preceding paper.

- <sup>3</sup> Ponzio, J. prakt. Chem., 48, 487 (1893).
- <sup>4</sup> Archbutt, J. Soc. Chem. Ind., 17, 1009 (1898).
- <sup>5</sup> Reagents were prepared as described in the previous paper.

of rapic acid and of the greater part of the unsaturated acids remain in the solution in 90% alcohol, it is inferred that the acid isolated is undoubtedly erucic acid. A confirmatory test can be performed by reducing it to behenic acid with an active catalyst in a suitable solvent. This will be investigated later.

## Tung Oil

The magnesium soaps from all oils examined (in the preceding paper) are either entirely or almost entirely soluble in 90% alcohol at the temperature of the *boiling* alcoholic soap solution. Tung oil is an exception, in that an abundance of white elastic magnesium soap may be prepared from it which is insoluble in 90% (by volume) alcohol even upon prolonged boiling under a reflux condenser. Since tung oil is said to contain mainly the glycerides of eleomargaric and oleic acids with small amounts of saturated acids, and since the magnesium soaps of oleic acid and small amounts of saturated acids are soluble in the boiling alcoholic solution, the insoluble substance is probably magnesium eleomargarate.

Technique of the Test.—The tung oil is saponified and the magnesium soaps are precipitated in the same manner as that described for rape oil. The mixture containing the insoluble magnesium soaps is filtered while almost boiling hot. The precipitate is washed with hot 90% alcohol and decomposed with dil. hydrochloric acid, preferably in the absence of air. The liberated acid has the strong odor characteristic of tung oil. The acid is soluble in cold, 90% alcohol and melts at about 44°. After 1 or 2 days' contact with the air, it absorbs oxygen and gradually changes to a dark brown, resinous mass. This change is greatly accelerated at elevated temperature. The yield of this acid (eleomargaric), obtained from the one sample of tung oil tested, was about 20%.

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## THE MECHANISM OF THERMAL DECOMPOSITION OF THE PENTANES

By G. Calingaert

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Normal and *iso*pentane, which constitute the lower-boiling fraction of petroleum ether, are at present of no technical use. The present writer has made a study of the product of their decomposition by heat, with a view to their possible utilization in other ways than as a fuel. If it were possible, by their partial decomposition, to obtain satisfactory yields of unsaturated hydrocarbons of 4 and 5 carbon atoms, then further treatment might produce acids, alcohols or other derivatives of higher commercial value than the original petroleum ether.

The pentanes, like organic compounds in general, are decomposed on being passed through a red-hot tube. The literature gives references to the products obtained in many cases. We find with hydrocarbons that the products of decomposition are generally the same, whatever hydrocarbon is tested. The percentage of hydrogen increases gradually, and