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[CONTRIBUTIONS FROM THE ANALYTICAL LABORATORIES OF THE SCHOOL OF MINES, COLUMBIA COLLEGE.—NO. 5.]

THE QUANTITATIVE SEPARATION OF ROSIN OIL FROM MINERAL OILS.

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These two oils being both unsaponifiable are usually determined together as "unsaponifiable material" in the analysis of oils. They may be distinguished from one another by several methods.

1. *The Specific Gravity*.—Rosin oil has a much higher gravity than mineral oils, varying from 0.960 to 1.000 while heavy mineral oils range from 0.850 to 0.920.

2. *Valenta's Test*.—*Dingler's Poly. J.*, **252**, 297, and **253**, 418, *J. Chem. Soc.*, **48**, 93. Glacial acetic acid dissolves 2.67–6.50 per cent. by weight of mineral oil while rosin oil is soluble to the extent of 16.87 per cent. Mixtures, however, do not dissolve in proportion to the amount of rosin oil present.

3. *The Stannic Bromide Test*.—Allen, *Comm. Org. Anal.*, **2**, 463. A solution of stannic bromide in carbon disulphide gives, with small quantities of rosin oil in carbon disulphide solution, a purple coloration, while mineral oils do not.

4. *Solubility in Acetone*.—Demski and Morawski, *Dingler's Poly. J.*, **258**, 39. Acetone is miscible with rosin oil in all proportions while mineral oils require several volumes for solution.

5. *The Elaïdin Test*.—Hager, *Ztschr. anal. Chem.*, **19**, 116. Rosin oil gives a dark red clear liquid while mineral oils remain unchanged.

6. *Ammonia Emulsion Test*.—Hager, *Muspratt's Tech. Chemie.*, 1893, **4**, 127, mixes two cc. of the oil to be tested with two cc. of petroleum benzine and four cc. of water and after shaking adds one cc. of ten per cent. ammonia; he then shakes violently and allows to stand for one to two hours. A persistent milky layer indicates rosin oil.

7. *Action of Sulphuric Acid*.—Hager, *Ztschr. anal. Chem.*, agitates a portion of the oil with an equal volume of concentrated sulphuric acid for five minutes and then pours into several volumes of cold water. Mineral oil gives a white milky liquid which separates into two clear light-colored layers. Rosin oil gives a gray or brownish milky liquid which separates an upper layer of yellow-brown color full of opaque flocks.

8. *The Color Produced with Sulphuric and Acetic Acids*.—Storch, *Analyst*, **13**, 71. Two cc. of the oil is shaken up with one cc. of anhydrous acetic acid and warmed gently. After cooling, the acetic liquid is removed with a pipette and a drop of strong sulphuric acid added which immediately produces a brilliant red color if any rosin oil is present. Cholesterol in many fatty oils gives a similar reaction. The same author determines rosin oil

quantitatively by its solubility in alcohol which is greater than that of mineral oils.

9. *The Hübl Figure*.—Valenta, *Dingler's Poly. J.*, **253**, 420. The Hübl figure of rosin oil is forty-three to forty-eight, while that of mineral oils is below fifteen.

10. *The Refractive Index*.—Holde, *Mittheil d. chem. tech. Vers. Anst.*, 1890, **8**, 269. With the Abbe refractometer the indices are as follows:

Rosin oil.....	1.5344
Mineral oils.....	1.4923

11. *Maumene's Test*.—*Muspratt Tech. Chem.*, 1893, **4**, 128. Rosin oil gives a rise of temperature of 42° C. Mineral oils only a slight rise. Allen, *Comm. Org. Anal.*, **2**, 462, says the rise in temperature of rosin oil is 18°-20° C.

12. *The Action of Nitric Acid*.—*Muspratt*, **4**, 127. Nitric acid of 1.185 sp. gr. when heated with rosin oil reacts violently with it giving off copious red fumes while mineral oils are but slightly effected.

Allen, *Comm. Org. Anal.*, **2**, 462, says that cold nitric acid is sometimes without immediate action on rosin oil but on warming a violent reaction often very suddenly ensues and after cooling, the rosin oil is found to have been converted into a more or less brittle red resin.

This process was investigated with a view to making it quantitative. Attempts were made to use nitric acid of 1.42 sp. gr. at a boiling temperature, but the frothing of the mass proved a serious difficulty, the liquid in every instance frothing out of the flask. Acid of 1.2 sp. gr. was found more manageable, the frothing in this case being slight. It was thought that the red resin might be dissolved in alkali, in which it appears to be quite soluble, and leave behind the mineral oil which could be dissolved in benzine, but this process could not be made to work satisfactorily. It was found, however, that the red resin produced from the rosin oil was insoluble in petroleum ether while mineral oil dissolves easily.

The process was therefore altered by diluting the products of the reaction with water and extracting with petroleum ether.

The following process gave satisfactory results:

Fifty cc. of nitric acid of 1.2 sp. gr. are heated to boiling in a flask of 700 cc. capacity. The source of heat is removed and five grams of the oil to be analyzed added. The flask is then heated on the water-bath, with frequent shaking, for fifteen to twenty minutes, and about 400 cc. of cold water added. After the liquid has become entirely cold, fifty cc. of petroleum ether are added and the flask agitated. The oil which remains unacted upon dissolves in the ether, while the resin remains in suspension. The liquid is poured into a tapped separator, leaving the lumps of solid resin as far as possible behind in the flask. After settling, the aqueous liquid is drawn off and the ethereal layer poured into a tared flask. Another portion of petroleum ether is added to the resin remaining in the flask and allowed to act upon it for about ten minutes, when it is added to that in the tared flask. After distilling off the ether the oil is weighed. Mineral oils lose about ten per cent. in this way, and hence the weight of oil found must be divided by 0.9 in order to find the amount present in the sample analyzed.

Allen found mineral oils to lose ten to twelve per cent. on treatment with nitric acid. *Pharm. Jour.*, [3], 11, 266.

A mixture of seventy-six per cent. of mineral oil with twenty-four per cent. of rosin oil gave, by this method, 76.8 per cent. of mineral oil.

RUTHENIUM AND ITS NITROSOCHLORIDES.¹

BY JAMES LEWIS HOWE.

A FEW years ago A. Joly² published the results of an investigation on the supposed tetrachloride of ruthenium of Claus, in which he showed that the double salts of this compound, the "red salt" of Claus, are in reality double salts of a nitrosochloride, RuCl_2NO . When his work came to my notice I was engaged in studying the compounds of ruthenium, especially seeking to discover a simple and certain way of forming the supposed tetrachloride. Being engaged at present on other compounds of ruthenium, I present in this paper results reached,

¹ Read before the Cincinnati Section, March 15, 1894.

² *Compt. rend.*, 107, 998; 108, 854.