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SAGITTOL. A NEW SESQUITERPENE ALCOHOL

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During the course of a systematic investigation of the carbohydrates of a number of uncultivated plants which are indigenous in the United States, attention was directed to *Balsamorrhiza sagittata* (Pursh) Nutt because of the peculiarly pleasant camphor-like odor of its roots and the fact that it and related species (*B. helianthoides*, *B. deltoidea*, etc.), have been used medicinally and for food purposes by the Indians of the Northwest.[1]

B. sagittata is a low plant, 12 to 18 inches high, with yellow flowers similar to the sunflower, and grows abundantly in Montana, Idaho, Oregon, Utah and Nevada.[2]

The root was eaten by the Indians only after peeling off the cortex which, according to Havard,[1] contains a "terebinthine balsam." Search of the literature has revealed no record of an investigation of this substance.

Upon distilling the roots with steam a crystalline substance separated from the distillate as fine, white needles, the yield being a little less than 0.5% based on the weight of the dry cortex. One recrystallization from alcohol was sufficient for purification, and the substance obtained was quite soluble in alcohol, ether and benzene. It has a pleasant odor, distinctly resembling that of camphor.

G. L. Keenan, of the Food, Drug and Insecticide Administration, has kindly furnished me with the following description of the crystals.

> "This substance crystallizes in very fine needles which are soluble in the organic liquids commonly used in the immersion method but are insoluble in solutions of potassium mercuric iodide in glycerol and water. The needles were found to have a negative elongation, that is, to exhibit their lowest index of refraction lengthwise. The extinction is parallel. The lowest value which it is possible to find is n_D 1.520, and this may be taken as α for the substance. A higher value, n_D 1.540, is shown on the needles crosswise and may be considered as γ for the substance."

The melting point of the pure substance is 77–78° (uncorr.). The specific rotation, determined in 95% alcohol solution, is $[\alpha]^{20}_{D} + 25.8°$ for $C = 2.080$, and $+26.5°$ for $C = 2.424$ g. per 100 cc. Combustion analyses gave the following results.[3]

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[1] Report Commiss. of Agr. for 1870, p. 404; J. Lindley and T. Moore, "The Treasury of Botany," **1870**, Vol. I, p. 120; V. Havard, *Bull. Torrey Bot. Club*, **22**, 98 (1895); F. V. Coville, Contrib. U. S. Nat. Herbar., **5**, 62 (1897); J. W. Blankinship, Mont. Agr. Coll. Expt. Sta. Bull. No. 56 (1905); R. V. Chamberlin, *Proc. Acad. Nat. Sci. Phila.*, **63**, 24 (1911); G. B. Grinnell, "The Cheyenne Indians," **1923**, Vol. II, pp. 166–191; J. R. Anderson, "Trees and Shrubs of British Columbia," Victoria, B. C., **1925**.

[2] Grateful acknowledgment is made of the courtesy of R. H. Rutledge, District Forester, U. S. Forest Service, Ogden, Utah, in furnishing a supply of roots.

[3] These and subsequent combustion analyses were performed by E. K. Nelson of this Bureau, to whom I am also grateful for general interest in this work.

(1) Subs., 0.0538: H_2O, 0.0560; CO_2, 0.1604; H, 11.65; C, 81.33
(2) Subs., 0.0709: H_2O, 0.0741; CO_2, 0.2101; H, 11.69; C, 80.85
(3) Subs., 0.0779: H_2O, 0.0827; CO_2, 0.2314; H, 11.88; C, 81.03
(4) Subs., 0.0727: H_2O, 0.0766; CO_2, 0.2155; H, 11.79; C, 80.86

Average values: H, 11.75; C, 81.02. Calcd. for $C_{15}H_{26}O$: H, 11.79; C, 81.01; O, 7.20.

The molecular weight of the substance was determined by the depression of the freezing point (Beckmann's method), benzene being used as a solvent. Two determinations resulted in values for the molecular weight of 220 and 224. The molecular weight was also determined by Rast's method:[4] 0.012 g. of substance in 0.112 g. of camphor caused a 20° depression in the melting point. Several determinations gave an average molecular weight of 214. The calculated molecular weight for the compound $C_{15}H_{26}O$ is 222.

Having established the empirical formula, the nature of the oxygen atom was determined by acetylation. Two grams of the substance was gently boiled with 5 cc. of acetic anhydride and a little fused sodium acetate for about an hour on a sand-bath. The resulting liquid was then poured into water, where it separated as a sirup but did not crystallize on standing. After being washed several times with water, the sirup was dissolved in ether, dried with anhydrous sodium sulfate, and the solvent evaporated *in vacuo*. It was again dissolved in alcohol and the solvent evaporated. After being thoroughly dried in a vacuum desiccator over both sodium hydroxide and calcium chloride, the sirup was analyzed with the following results.

(1) Subs., 0.0866: H_2O, 0.0819; CO_2, 0.2446; H, 10.58; C, 77.05
(2) Subs., 0.0908: H_2O, 0.0868; CO_2, 0.2572; H, 10.70; C, 77.27

Average values: H, 10.64; C, 77.16. Calcd. for $C_{15}H_{25}OCOCH_3$: H, 10.68; C, 77.21; O, 12.11.

The compound, which we have named "sagittol," is therefore a sesquiterpene alcohol. It is not identical in properties with any of the sesquiterpene alcohols described in the literature. Upon standing in a desiccator for several months sagittol became somewhat sticky and upon attempted recrystallization yielded a sirup instead of crystals. This compound will be investigated further as soon as additional plant material is available.

Summary

A new compound named "sagittol" has been isolated from *Balsamorrhiza sagittata* (Pursh) Nutt, and some of its properties determined.

From combustion and molecular weight determinations, the compound was found to have a molecular weight of 222, with the empirical formula $C_{15}H_{26}O$. The compound contains an alcohol group and is, therefore, a sesquiterpene alcohol.

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[4] Rast, *Ber.*, **55**, 1051 (1922).

The melting point of sagittol is 77–78° (uncorr.); its average specific rotation in 95% alcohol is +26.15° at an average concentration of of 2.252 g. per 100 cc. Sagittol crystallizes as very fine needles, the optical properties of which have been determined.

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[COMMUNICATION FROM THE DEPARTMENT OF INDUSTRIAL AND CELLULOSE CHEMISTRY, McGILL UNIVERSITY]

STUDIES ON REACTIONS RELATING TO CARBOHYDRATES AND POLYSACCHARIDES.

XXXI. THE BEHAVIOR OF CELLULOSE TOWARD SOLUTIONS OF ALUMINUM SALTS

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In view of the important role played by aluminum sulfate in the process of the rosin sizing of paper, many investigations have been carried out to ascertain the mechanism of the adsorption by the cellulose fiber. Schwalbe and Robsahm,[2] using sulfite pulps, claimed that cellulose decomposed aluminum sulfate up to 3% of its weight, but Schwalbe[3] in a re-investigation of the problem states that neither cotton nor pulp takes up appreciable amounts of aluminum hydroxide from aluminum sulfate solutions. Sutermeister[4] found that the amount adsorbed was never greater than 0.3% of the weight of the fiber and was independent of the concentration of the aluminum sulfate solution employed.

It was concluded by Tingle[5] that the observed withdrawal of aluminum hydroxide from solutions of aluminum sulfate in the presence of cellulose is due to the non-cellulosic constituents of the fiber. He could detect no adsorption by ordinary analytical methods where the cellulose was of a reasonable degree of purity, and concluded that within the limits imposed pure cellulose will adsorb no aluminum hydroxide from the solution.

In the case of aluminum acetate, and also in some cases with aluminum sulfate, Haller[6] found "negative adsorptions" by analysis of the residual solutions, but actually in every case, some fixation of aluminum hydroxide by the fiber had occurred. Durst[7] also found that "negative adsorption" took place with wood pulps from aluminum acetate solutions. The ash

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[1] Research Fellow, Pulp and Paper Research Institute, Montreal, Canada. The authors desire to express their thanks to the Canadian Pulp and Paper Association for financial assistance rendered in connection with this investigation.

[2] Schwalbe and Robsahm, *Wochenblatt*, **43**, 1454 (1912).

[3] Schwalbe, *Z. angew. Chem.*, **37**, 125 (1924).

[4] Sutermeister, *Pulp and Paper Mag. of Canada*, **11**, 803 (1922).

[5] Tingle, *J. Ind. Eng. Chem.*, **14**, 198 (1922).

[6] Haller, *Chem.-Ztg.*, **42**, 597 (1918).

[7] Durst, *ibid.*, **43**, 373 (1919).