This is in line with the increase in reactivity of electronegative groups which generally results from substitution of silicon for carbon.

Summary

1. Trimethylsilyl sulfate has been prepared by an improved method.
2. Reactions of trimethylsilyl sulfate have been studied.
3. A mechanism for the Flood reaction has been proposed.

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[CONTRIBUTION FROM THE DEPARTMENT OF AGRICULTURAL CHEMISTRY, THE OHIO STATE UNIVERSITY]

The Isolation of β-Amyrin and a Fatty Acid of High Molecular Weight from *Solidago leavenworthii* T. and G.

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In the usual process for obtaining rubber from goldenrod the leaf material is first exhaustively extracted with acetone to remove waxes and resinous materials. On evaporation of the acetone from this extract a dark green tar is obtained. Polhamus[1] reports a mean of 21.03% of tar from five samples of *Solidago minor*. At the Southern Regional Research Laboratory of the U. S. D. A., Guthrie, *et al.*,[2] obtained a good yield of quercitrin and its aglycone from such tar prepared from the leaves of *Solidago leavenworthii* T. and G. Through the kindness of Dr. Guthrie a sample of this tar was obtained by us and the benzene soluble fraction of it was subjected to further investigation.

Experimental Results

Isolation of β-Amyrin.—Three kg. of the above mentioned tar were extracted with 2 l. of boiling benzene followed by five extractions with one-liter portions of hot benzene. The combined extracts were filtered through paper pulp and the green pigments removed by treatment with activated charcoal. A dark brown solution resulted from which the benzene was distilled, thereby yielding 950 g. of a brown viscous oil. This oil was refluxed for two hours with 1500 cc. of 20% alcoholic potassium hydroxide. The mixture was cooled and 2 liters of water added. It was made slightly acid to litmus with 10 *N* sulfuric acid. Two layers were formed, the upper a brown oil and the lower a greenish colored water solution. The oily layer was separated and treated with one liter of 10% sodium hydroxide solution and 3 liters of water and shaken vigorously for an hour. This mixture was then repeatedly extracted with ether, the extracts united, washed and dried over anhydrous sodium sulfate.

The sodium hydroxide solution which remained after the ether extraction was set aside for further investigation.

The ether was distilled from the dried extract leaving a deep yellow greasy residue. This was dissolved in one liter of acetic anhydride and refluxed for two hours. After standing overnight a heavy deposit of yellow needles formed. The liquid was then decanted and the crystals washed twice with one-liter portions of hot 95% ethyl alcohol and finally with petroleum ether until colorless. Nineteen grams of vacuum dried crystals was obtained, amounting to a yield of about 0.13% of the dried leaf material.

These crystals (acetate) melted at 231–233°. The saponification equivalent was 467 to 471 and the specific rotation in chloroform was $[\alpha]^{28}D$ +78°. The saponified compound after recrystallization from a large volume of 95% ethyl alcohol gave long colorless needles which melted at 190–192°. The benzoate melted at 229–230°. The Liebermann–Burchard reaction was positive. These properties correspond quite closely to those recorded for β-amyrin.[3,4]

Isolation of a High Molecular Weight Fatty Acid.—The fraction of saponified oil that was soluble in 10% sodium hydroxide (see β-amyrin preparation above) was neutralized with 10 *N* sulfuric acid. A brown oily liquid rose to the surface. This oil was removed by extraction with ether, the ether extract was thoroughly washed with water and dried, the ether removed by distillation and the residue dissolved in 1500 cc. of hot 95% ethyl alcohol. This solution was partially cleared with activated charcoal. On cooling a slightly yellow flocculent precipitate formed. This was reprecipitated four times from 95% alcohol. The 5 g. of colorless, amorphous material thus obtained was saponified for six hours with 20% alcoholic potassium hydroxide. This solution was then cooled and diluted with an equal volume of water. A slightly yellowish soapy precipitate formed which was filtered off, washed with dilute sulfuric acid, dried over a steam-bath and dissolved in 30 cc. of hot glacial acetic acid. On slow cooling colorless rosets of crystals were deposited (dry wt. 1 g.) which melted at 82°. The molecular weight by the Rast method was 433. Due to the hydrocarbon-like properties of the compound neutralization equivalent values were erratic. The acid amide melted at 108–110°.

Anal. Calcd. for $C_{28}H_{56}O_2$ (424.4): C, 79.17; H, 13.29. Found: C, 79.27; H, 13.25.

Summary

The isolation of β-amyrin and a high molecular weight fatty acid from the acetone extract of leaves of *Solidago leavenworthii* T. and G. is described.

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(4) I. M. Heilbron, G. L. Moffet and F. S. Spring, *J. Chem. Soc.*, 1583 (1934).