COMMUNICATIONS TO THE EDITOR

LACTONE FORMATION IN THE SULFONATION OF HEAT TREATED ROSIN

Sir:

Some time ago Ruzicka and Meyer [*Helv. Chim. Acta*, **5**, 333 (1922)] described the formation of a lactone obtained on treating dihydroabietic acid with strong mineral acids. Apparently we have been able to obtain a similar, if not identical, lactone by sulfonation of heat treated rosin or heat treated abietic acid.

Fifty grams by weight of partially refined pseudopimaric acid [Brennan, Cairncross, Hasselstrom and Hull, U. S. Patent 2,072,628] melting point 167–169° (corr.), $(\alpha)^{31}D$ + 46.3°, and 250 g. of sulfuric acid, sp. gr. 1.84, were mixed at −5 to + 5°, and stirred for about twenty minutes. The brownish precipitate was collected, washed with cold water until the washing clouded when mixed with the original liquor, and the washed precipitate then extracted three times with boiling water, leaving a brownish insoluble, semi-solid rosin. The aqueous extracts on cooling deposited crystalline sulfonic acid in a yield of 25.5 g. After repeated crystallization from glacial acetic acid, it melted with decomposition at about 222–223° (uncorr.).

The brownish insoluble semi-solid rosin amounted to 30 g. When an acetone solution of this by-product was allowed to stand, it deposited 4 g. of a saturated lactone, a crystalline, white solid which, after recrystallization, melted at 130–131° (corr.). Calcd. for $C_{20}H_{32}O_2$: C, 78.9; H, 10.5. Found: C, 79.36, H, 10.51; C, 78.9, H, 10.79; C, 79.52, H, 10.73; C, 79.08, H, 10.53. The melting point of Ruzicka and Meyer's lactone was 130–131°.

The above lactone was saponified with 10% alcoholic potassium hydroxide solution for thirty-six hours. After evaporating the alcohol, the potassium salt of the hydroxy acid was dissolved in a large quantity of water and the hydroxy acid liberated with dilute acetic acid. The precipitated acid was purified through crystallization first from methanol–acetone, then from methanol and finally from hexane. The acid, fine white needles, melted at 161–162° (uncorr.), 165° (corr.) (dec.). Calcd. for $C_{20}H_{34}O_3$: C, 74.5, H, 10.6. Found: C, 74.73; H, 10.77.

The hydroxytetrahydroabietic acid of L. Ruzicka, H. Waldmann, Paul J. Meier and H. Hösli [*Helv. Chim. Acta*, **16**, 139–181 (1933)], obtained by saponification of the corresponding lactone, melted at 162–163°. Ruzicka and co-workers mention that the hydroxy acid is easily transformed into the lactone, which observation fully agrees with the properties of the hydroxytetrahydroabietic acid obtained by us. We have observed that the hydroxytetrahydroabietic acid when treated with acetyl chloride in the usual manner, yielded the lactone quantitatively which now melted at 131–131.5 (corr.); it did not lower the melting point when mixed with the lactone isolated from the original sulfonation product.

This investigation is being continued. Further information will be published at a later date.

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VITAMIN B-6

Sir:

We have reported the isolation of vitamin B-6 as its hydrochloride, m.p. 204–206° (dec.) elsewhere [*Proc. Exp. Biol. & Med.*, **38**, 64–65] (1938). Elementary analysis of the substance gives the empirical formula $C_8H_{12}NO_3Cl$. Calcd. for $C_8H_{12}NO_3Cl$: C, 46.70; H, 5.88; N, 6.81; Cl, 17.25. Found: C, 46.89, 46.79; H, 6.12, 6.10; N, 6.81, 6.94; Cl, 17.03, 17.13. Determinations of O—CH_3, N—CH_3 and water of crystallization were negative. It is optically inactive. The base itself, m.p. 160°, was isolated from the hydrochloride. *Anal.* Calcd. for $C_8H_{11}NO_3$: C, 56.80; H, 6.56. Found: C, 56.54, 56.84; H, 6.35, 6.15. Both the base and the hydrochloride readily sublime and final purification of the vitamin can be effected in this way. With ferric chloride the vitamin gives a reddish-brown coloration. The pure substance is stable to concentrated hydrochloric acid at elevated temperatures. It is not affected by heating with alkalies, nitrous acid, ethyl nitrate or Fehling's solution.

Since electrometric titration studies of the