857 g., b. p. 290–292° (uncor.), was obtained or a yield of 90%. This too hydrolyzed completely when dissolved in water.

Anal. Calcd. for $C_{13}H_{24}O_8B_2$: C, 50.40; H, 8.46; neut. eq., 143.0. Found: C, 49.95; H, 8.42; neut. eq., 144.5.

Discussion.—These two esters, as might have been expected from their formulas, exhibited marked differences in their chemical reactions and physical properties. With metallic sodium, for example, the acid borate reacted vigorously on heating, whereas the orthoborate showed practically no reaction. In acetone solution, the acid borate appeared to form a true salt with ammonia, whereas the orthoborate yielded a gelatinous complex. The esters may be distilled at atmospheric pressure without decomposition. At room temperature, the orthoborate is a sirup of the consistency of glycerol, whereas the acid borate is so viscous that it hardly flows.

It should be pointed out that the above di-orthoborate, having been prepared from the *meso* form, may represent a new substance, since Dupire makes no mention as to whether in his preparation a single isomer or a mixture of the isomers was used.

Rippere and LaMer's[3] failure to obtain the Dupire-type[4] glycol borates may have been due to the fact that when the esterification is conducted azeotropically in benzene solution, a much more uniform reaction occurs at a lower temperature than when the reactants are merely mixed and heated to drive off the water.

(5) One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

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The Vapor Pressure of Trimethylamine from 0 to 40°

BY ELIJAH SWIFT, JR., AND HELEN PHILLIPS HOCHANADEL

In the course of a study of the properties of the simpler aliphatic amines near room temperature we have measured the vapor pressure of trimethylamine at a number of temperatures between 0 and 40°, since no published data could be found covering this range.

The purification of the compound has been described previously.[1] Since the chemical treatment does not take care of dissolved gases, each sample after desiccation was thoroughly degassed by repeated freezing and melting with evacuation. This process was continued until the sample had evaporated to approximately half of its original volume. As a check on the purity of the amine, its vapor pressure was measured at the ice-point, the vapor then being allowed to expand into a volume approximately ten times as great as before and the pressure again measured. If a significant difference in vapor pressure was

(1) E. Swift, Jr., THIS JOURNAL, 64, 115 (1942).

found, the degassing process was repeated until results concordant to at least 1 mm. were obtained.

The samples were transferred by vacuum distillation to an isotensiscope equipped with a magnetic stirrer, which served to break the surface of the liquid about twice a second. As a check on possible contamination during transfer, the vapor pressure at the ice-point was measured again, and in every case checked, within less than a millimeter, the value found when the sample was directly connected to the manometer.

At each temperature care was taken to allow sufficient time for equilibrium to be established, as evidenced by a steady reading for at least half an hour while the liquid was being agitated. The mercury levels in the isotensiscope were adjusted to within 0.5 mm. of each other, and the levels in the manometer then read with a Société Génévoise cathetometer. Since some of the preliminary measurements were made with a Gaertner cathetometer, the two instruments were checked against each other and found to agree within better than 0.5 mm. over the whole scale. For heights greater than 1000 mm., a meter stick was used, and later checked against the cathetometer scale.

The barometric pressure was taken from an open manometer which was read with the same cathetometer as above. The diameters of the reservoir and tube of this manometer were the same as those of the main manometer, to cancel out any bore correction. Brass scale corrections were applied to all readings.

Temperatures were maintained constant to $\pm 0.02°$, the absolute value being obtained to 0.01° by comparing the thermometers used with a platinum resistance thermometer calibrated at the ice-point.

The corrected observed pressures are shown in Table I, as averaged values for all readings.

TABLE I

VAPOR PRESSURES OF TRIMETHYLAMINE

T, °C.	$P_{obs.}$ mm.	$\frac{P_{obs.} - P_{calcd.}}{P_{obs.}}, \%^a$	$\frac{P_{obs.} - P_{calcd.}}{P_{obs.}}, \%^b$
0	683	0.4	0.6
15	1189	0.8	−0.3
20	1415	1.4	0
25	1663	1.2	−0.2
30	1946	1.4	−0.2
35	2266	1.5	0
40	2621	1.5	0.2

^{a} Equation (1), ref. 2. ^{b} Equation (1), this paper.

The largest difference between samples was less than 1%, the average deviation from the mean being 0.3%. The over-all accuracy is about 0.5%.

After these measurements were completed, the results of a very careful study by Aston and co-workers of the vapor pressure of trimethylamine at temperatures between −80 and +3° appeared,[2] together with an empirical equation

(2) J. G. Aston, M. L. Sagenkahn, G. J. Szasz, G. W. Moessen and H. F. Zuhr, *ibid.*, 66, 1171 (1944).

which reproduces their results with considerable accuracy in this range. However, their equation gives values which are too low when extrapolated to the higher temperature range reported here, as shown in the third column of Table I. Consequently, an equation was derived which fits the data in the range 0–40° within our limit of error.

$$\text{Log } P_{mm.} = -(2018.37/T) - 6.0303 \log T + 24.91300 \tag{1}$$

The deviations of the observed values from those calculated by means of this equation are shown in the fourth column of the table. The heat of vaporization calculated by means of this equation with suitable corrections[2] was found to be 5720 cal./mole at the normal boiling point, 276.03°K., as compared with the calorimetric value[2] of 5482. cal/mole.

The value found at the ice-point may be compared with other values in the literature. Wiberg and Sütterlin[3] report 681 mm., Simon and Huter[4] find a value of 679.1 (calcd.), Thompson and Linnett[5] 680 mm. The average is within our limit of error, equation (1) giving a value of 679 mm.

The authors wish to thank the Physics Department of Indiana University who loaned a cathetometer and to express their appreciation for a grant from the Graduate School Fund of Indiana University.

(3) E. Wiberg and W. Sütterlin, Z. Elektrochem., 41, 151 (1935).

(4) A. Simon and J. Huter, ibid., 41, 28 (1935).

(5) H. W. Thompson and J. W. Linnett, Trans. Faraday Soc., 32, 681 (1936).

CHEMISTRY DEPARTMENT
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Ursolic Acid and Paraffin Hydrocarbons from Cryptostegia Leaves[2]

BY JONATHAN W. WHITE, JR., AND F. R. SENTI

During an investigation of methods for the recovery of rubber from the leaves of Cryptostegia grandiflora, a brief study of some of the constituents of the leaf-cuticle wax was made. A fermentation pretreatment for rubber recovery resulted in a leaf fraction consisting of all the epidermis, veins, and latex ducts, and representing about 18% of the original leaf weight.[3] From this leaf fraction was isolated ursolic acid and a mixture of n-paraffin C_{29}, C_{31} and C_{33} hydrocarbons.

The leaf fraction was exhaustively extracted with hot acetone. When the extract was cooled to room temperature a voluminous crystalline precipitate formed, representing 7.1–8.2% of the weight of the material extracted. This would

correspond to 1.3–1.5% of the weight of the original leaves. After one recrystallization this crude wax had an acid number of 49.0, a saponification number of 79.3 and a melting range of 222–230°. Several components were isolated from this wax as described below.

Ursolic Acid.—Saponification by ethanolic potassium hydroxide yielded an acid fraction, representing 43% of the original wax. This was purified by the method of Sando[4] to yield a material identical with ursolic acid, as shown by the melting point (285°), X-ray powder patterns of the acid and of its sodium salt, and the optical properties of the crystals.[5] The sodium salt produced the sharper diffraction pattern; since this pattern has not been published, the interplanar spacings and relative intensities of reflection are presented in Table I.

TABLE I

X-RAY POWDER PATTERN OF SODIUM URSOLATE

Anal. Calculated for $C_{29}H_{47}OCO_2H$: C, 78.87; H, 10.60, neut. equiv., 465. Found: C, 78.94, 78.76; H, 10.44, 10.64; neut. equiv., 460.

Interplanar spacing, Å.	Intensity I/I_1	Interplanar spacing, Å.	Intensity, I/I_1
14.6	0.50	2.83	0.04
10.3	.03	2.71	.08
9.2	.50	2.63	.05
7.6	.03	2.54	.04
6.90	1.00	2.42	.05
6.49	0.50	2.35	.04
5.96	.50	2.28	.04
5.68	.50	2.23	.03
5.17	.08	2.18	.04
5.01	.18	2.13	.01
4.82	.13	2.10	.04
4.61	.14	2.05	.03
4.49	.12	2.00	.01
4.18	.10	1.98	.02
3.88	.07	1.95	.03
3.75	.03	1.91	.01
3.52	.09	1.87	.01
3.37	.06	1.79	.01
3.24	.03	1.71	.01
3.17	.06	1.66	.01
3.06	.04	1.59	.01
2.92	.06		

Ursolic acid has previously been reported to be a constituent in the skin of several fruits and of such leaves as Arbutus unedo, Epigaea asiatica, and mistletoe.[6]

(4) C. E. Sando, J. Biol. Chem., 90, 477 (1931).

(5) The crystals are blades which show biaxial positive interference figures with 2E approximately 130°. The index α, crosswise of the blades, was 1.551; β, along the blades, was 1.561; and γ, perpendicular to the blades, was undetermined. These values are in agreement with those reported by Sando.[4]

(6) K. S. Markley, S. B. Hendricks and C. E. Sando, J. Biol. Chem., 111, 133 (1935); K. S. Markley and C. E. Sando, ibid., 119, 641 (1937); W. A. Nealey, Western Canner and Packer, 33, No. 12, 22 (1941); A. Sanna, AHi IV Congr. Naz. chim. pura applicata (1932) 595 (1933); K. Fujii, N. Shimada and T. Sasaki, J. Pharm. Soc. Japan, 55, 650 (1935); E. I. van Itallie, Pharm. Weekblad, 58, 824 (1921).

(1) One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

(2) Natural Rubber from Domestic Sources, Paper.

(3) J. Naghski, J. W. White, Jr., S. R. Hoover and J. J. Willaman, in press.