

ditions previously shown to produce no fractionation.⁵

This indicates that the contribution of the cuprite oxygen to the density of the water formed from it is 5.5 p. p. m. less than that of atmospheric oxygen.

This is only 1 p. p. m. less than the difference between the density contributions of atmospheric and ordinary water oxygen⁵ and indicates therefore that the isotopic composition of the cuprite oxygen is practically the same as that of ordinary water.

This probably indicates that the original copper sulfide deposit was altered by reaction with water and not with atmospheric oxygen.

(5) Hall and Johnston, *THIS JOURNAL*, **58**, 1920-1922 (1936).

BOWLING GREEN STATE UNIVERSITY
BOWLING GREEN, OHIO RECEIVED SEPTEMBER 6, 1940

The Freezing Points and Low Temperature Viscosities of Diacetone Alcohol-Acetone Mixtures^{1a}

By V. LANTZ

In determining the viscosities of diacetone alcohol and its mixtures with acetone at temperatures near the freezing points of the solutions, it was observed that the commercially pure diacetone alcohol used^{1b} (d_{20}^{20} 0.9377, n_D^{20} 1.4235) had a freezing point considerably higher than the only published values that could be found. The published values are -55° ² and -57 to -54° ,³ whereas the value found was -45° . A second sample of commercial "pure diacetone" from the same source (d_{20}^{20} 0.9375, n_D^{20} 1.4235) had a freezing point of -45.9° . A portion of the second sample was distilled at 50 mm. pressure using a 20 to 1 reflux ratio in a two-meter vacuum-jacketed column packed with glass helices. The 80% heart cut (d_{20}^{20} 0.9382, n_D^{20} 1.4232) had a freezing point of -44.0° . Similar results (-44.9°) have recently been obtained by W. C. B. Smithuysen of the Bataafsche Petroleum Maatschappij, Amsterdam.⁴

The freezing points of acetone-diacetone alcohol solutions, shown in Table I, indicate that from 15

(1a) Original manuscript received July 19, 1939.

(1b) Supplied by Shell Chemical Company, San Francisco, California.

(2) T. H. Durrans, "Solvents," 4th ed., D. Van Nostrand Co., New York, N. Y., 1938, p. 112.

(3) "Handbook of Chemistry and Physics," 23rd ed., Chemical Rubber Publishing Company, Cleveland, Ohio, 1939, p. 750.

(4) Private communication.

to 20% of acetone, the most likely impurity in diacetone alcohol, would need to be present to lower the freezing point of diacetone alcohol to the previously published values. The especially purified acetone used to obtain the data in Table I had the following constants: d_{20}^{20} 0.7914, n_D^{20} 1.3587.

TABLE I
FREEZING POINT OF ACETONE-DIACETONE ALCOHOL SOLUTIONS

| | | | | | |
|-----------------|-------|-------|-------|-------|-------|
| Acetone, by wt. | 0 | 4.3 | 8.6 | 13.0 | 20.0 |
| F. p., °C. | -44.0 | -46.9 | -50.4 | -52.5 | -55.5 |

Freezing points were determined by noting the break in a temperature-time curve obtained by slowly warming a mush of crystals and liquid in an air jacketed test-tube held at a nearly constant temperature above the freezing point of the solution. This method was found to be more satisfactory for determining initial freezing points than the use of cooling curves because the high viscosities of the solutions at their freezing points led to excessive under-cooling. Temperatures were measured with an iron-constantin thermocouple, calibrated against a platinum resistance thermometer.

Kinematic viscosities of diacetone alcohol and of a solution containing 12.8% by weight of acetone were measured at temperatures near their freezing points, using a Ubbelohde type viscometer.⁵ Viscosities are shown in Table II.

TABLE II
LOW TEMPERATURE VISCOSITIES OF DIACETONE ALCOHOL AND OF A SOLUTION OF ACETONE IN DIACETONE

| Diacetone alcohol | | 12.8% acetone | |
|-------------------|-----------------------|------------------------------------|-----------------------|
| Temp., °C. | Viscosity centistokes | 87.2% diacetone alcohol Temp., °C. | Viscosity centistokes |
| -1.8 | 7.8 | -33 | 15.0 |
| -2.7 | 8.1 | -44 | 30.6 |
| -15.0 | 15.3 | -55.1 | 78.0 ^a |
| -30.9 | 43.6 | | |
| -42.7 | 128 | | |
| -49.5 | 298 ^a | | |

(5) Ubbelohde, *Ind. Eng. Chem., Anal. Ed.*, **9**, 85 (1937).

SHELL DEVELOPMENT CO.
EMERYVILLE, CALIFORNIA RECEIVED SEPTEMBER 9, 1940

The Stable Form of Sucrose Octaacetate

By R. P. LINSTAD, A. RUTENBERG, W. G. DAUBEN AND W. L. EVANS¹

In a recent preparation of sucrose octaacetate, a quantity of sucrose was heated in the usual way with sodium acetate and acetic anhydride. The only abnormal feature of the reaction was that the time of reacting was prolonged to twenty

(1) After the authors became aware that work was being done simultaneously in the chemical laboratories of Harvard University and The Ohio State University on the stable form of sucrose octaacetate, they agreed to publish a joint paper on their results concerning this very important compound.