in the flavanone content of samples of *Eriodictyon* collected in various parts of this state. The material used in the procedure described was a commercially available product collected near Colusa, California, and marketed as "yerba santa."

The flavanones are being tested for "vitamin P" activity.

Experimental

Eriodictyol.—A total of 700 g. of powdered *Eriodictyon californicum* was extracted in about 70-g. portions with 500 ml. of ether in a Soxhlet extractor. The deep brown-green ether solution was filtered and shaken with successive portions of ammonium carbonate solution to remove acidic tarry material and then with 400 ml. of cold 10% sodium carbonate solution. A thick yellow precipitate of the sodium salt of homoeriodictyol formed and was collected by filtration on a large Büchner funnel, the filtrate being allowed to drop directly into dilute hydrochloric acid. The tarry precipitate from the filtrate was taken up in ether and again shaken with cold 10% sodium carbonate solution and the precipitate of sodium homoeriodictate collected by filtration. The filtrate, again dropping directly into dilute acid, deposited a tarry mass which was removed, transferred to a 2-liter round-bottomed flask and boiled with several 1.5-liter portions of water. The aqueous extracts were decanted from the tar and, after cooling, extracted with ether. The combined ether extracts were shaken with 300 ml. of ice-cold 10% sodium carbonate and the aqueous layer kept in an ice-bath for thirty minutes and then filtered from a rather small amount of precipitated sodium homoeriodictate. The filtrate was acidified with dilute hydrochloric acid, yielding a yellowish-brown tar. The tar was separated from the aqueous layer, transferred to a small beaker and rubbed with ether until the crystalline powder (A) which formed was no longer sticky. The product (about 8 g., moist) was collected and the filtrate allowed to evaporate slowly. A further amount (1 g.) of solid separated (B).

The first crop of material (A) was recrystallized from dilute alcohol, yielding 2.85 g. of colorless needles, m. p. 262–265°, dec. Dilution of the mother liquor yielded an additional 0.84 g. of product, m. p. 261–264° dec. Recrystallization of crop (B) yielded 0.78 g., m. p. 260–263° dec. The total yield was thus 4.47 g. of crude eriodictyol (0.64%). Recrystallized from dilute alcohol the substance formed tiny colorless needles, m. p. 265–266° dec. Descriptions of eriodictyol in the literature fail to record the fact that the m. p. of the compound depends upon the heating rate. Taken in the usual way, the m. p. may vary between 262 and 266°, with decomposition to a red liquid. If a sample is plunged into a preheated bath it melts at 267° (the usually recorded m. p.) to a pale yellow liquid.

Since the most probable impurity in the eriodictyol was homoeriodictyol a methoxyl determination was carried out but no methoxyl was found. The acetate, prepared with acetic anhydride–sodium acetate, formed colorless needles, m. p. 136–137° (literature[7] 137°).

The purified material dissolves in cold 10% sodium hydroxide solution to give a colorless solution which upon standing rapidly darkens. A solution of eriodictyol in 10% sodium hydroxide develops an intense blood-red color upon heating. Ferric chloride in methanol causes a transient green color which is immediately succeeded by brown-red.

Homoeriodictyol.—The various fractions of sodium salt collected as described above were combined and recrystallized from water. The moist salt (nearly colorless needles) was dissolved in about 400 ml. of boiling glacial acetic acid. Upon cooling there was obtained 40.5 g. of pale buff homoeriodictyol, m. p. 220°. Recrystallization raises the m. p. to the reported[7] value, 224°.

The acetate, prepared with sodium acetate-acetic anhydride, melted at 161–162° (reported[7] 163°).

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(7) Shinoda and Sato, *J. Pharm. Soc. Japan*, **49**, 71 (1929).

Isotopic Composition of Cuprite Oxygen

BY W. HEINLEN HALL AND CLARENCE HOCHANADEL

The isotopic composition of oxygen in carbonate rocks and iron oxide ores recently has been determined by Dole and Slobod.[1] We wish to report similar data for cuprite (Dana 224), one of the naturally occurring oxides of copper. The sample used by us was obtained from Ward's Natural Science Establishment and came from Bisbee, Arizona.

The technique which we used consisted in the reduction[2] of the oxide with commercial electrolytic hydrogen followed by a comparison of the density of the resulting water with that of water formed with the same hydrogen and atmospheric oxygen. The procedure used in purifying the water and determining the densities was the same as that used in earlier work.[3]

Densities were determined to within 1 p. p. m. by the buoyancy balance method in which temperature (measured with two Bureau of Standards calibrated Beckmann thermometers) was the observed variable. A completely submerged Pyrex float was used.[4]

After repeated purification of both samples, we observed that the floating temperature for water prepared from cuprite oxygen and commercial hydrogen was 0.021° lower than the floating temperature for water prepared by burning the same hydrogen in atmospheric oxygen under con-

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(1) Malcolm Dole and R. L. Slobod, THIS JOURNAL, **62**, 471–479 (1940).

(2) Reduction was carried out in such a way that the sample of hydrogen reacted completely, thus avoiding any fractionation.

(3) Hall and Johnston, THIS JOURNAL, **57**, 1515–1517 (1935).

(4) In this and other work extending over a period of five years, we have found variations in the floating temperature for any given float using ordinary water. We have attributed this to the dissolving of the float and/or to changes in the thermometers. It would appear from the observation made by Dole and Slobod that the thermometers may be responsible for the change since they found no appreciable effect when using a platinum resistance thermometer.

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 oxygen[5] 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.

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

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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}_4 0.9377, n^{20}_D 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}_4 0.9375, n^{20}_D 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}_4 0.9382, n^{20}_D 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 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}_4 0.7914, n^{20}_D 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 87.2% diacetone alcohol	
Temp., °C.	Viscosity centistokes	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]		

[a] Supercooled liquids

(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.

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

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The Stable Form of Sucrose Octaacetate

BY R. P. LINSTEAD, A. RUTENBERG, W. G. DAUBEN AND W. L. EVANS[1]

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

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(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.