

pure phthalone could be recovered. In absence of zinc chloride, the effort to get the phthalone also proved futile.

2-Methyl-3-acetamino-4-quinazolone¹ and phthalic anhydride condensed at 220° with evolution of water, forming a violet-brown substance which dissolved in alkalis to a brown solution, and from which it was reprecipitated by acetic or dilute sulfuric acid. Not enough of the material was obtained for further investigation.

NEW YORK CITY.

THE LEAF-OIL OF THE WASHINGTON CEDAR (*THUJA PLICATA*).

BY ROBERT EVSTAFIEFF ROSE AND CARL LIVINGSTON.

Received December 12, 1911.

The leaf oil of the Washington cedar (*Thuja plicata*) has not been examined chemically save by I. W. Brandel who published a short note on its composition.² The investigation referred to being only in the nature of an approximate analysis, the authors thought it advisable to undertake a more detailed study of the oil, the results of which follow: It may be mentioned that the conclusions reached differ very appreciably from those of Brandel.³

On steam distillation at 100°, the leaves and twigs of *Thuja plicata* yield about 1% of a clear, light yellow oil which possesses the characteristic odor of cedar boughs.

The following constants were found: D_{20}° , 0.913; n_D^{20} , 1.4552; $[\alpha]_D^{20}$, -4.77°; acid number, 0.518; ester number, 2.28; saponification number, 2.8; acetylation number, 8.8.

An elementary analysis showed the absence of sulfur and nitrogen, while the percentage composition (C = 78.6; H = 10.4) agreed very closely with that of a bicyclic ketone, $C_{10}H_{16}O$ (calc. C = 78.04; H = 10.52). The oil contained no phenols and was soluble in all proportions in anhydrous organic solvents and in 70% ethyl alcohol.

A fractional distillation under reduced pressure showed that 85% of the oil boiled between 100 and 110° under 40 mm. The small fraction (about 4%) boiling below 100°/40 mm. gave D_{20}° 0.851, n_D^{20} 1.4609, $[\alpha]_D^{20}$ +36.8° and was chiefly composed of pinene, which was identified by the preparation of the nitrosochloride melting at 103°. The main fraction was repeatedly distilled and yielded thus an oil boiling at 103-104° under 40 mm., which was found to be thujone by the following constants and derivatives:

D_{20}° , 0.9152; n_D^{20} , 1.4530; $[\alpha]_D^{20}$, -11.58°.

¹ Bogert and Gortner, THIS JOURNAL, 31, 946 (1909).

² Pharm. Rev., 26, 248.

³ Loc. cit.

Derivatives: tribromide, melting point $121-122^{\circ}$;¹ semicarbazone,² melting point $186-188^{\circ}$ (from methyl alcohol); tanacetone keto carbonic acid,³ melting point $75-76^{\circ}$.

The reaction with hydroxylamine gave an oily oxime which crystallized only partially after standing for several months, which fact, together with the observed laevo rotation—a rotation, it may be noted, slightly higher than any previously recorded—further characterized the substance as α -thujone.

The fraction boiling from $100-103^{\circ}/40$ mm. ($D_{20^{\circ}}$, 0.8975; $n_D^{20^{\circ}}$, 1.4549; $[\alpha]_D^{20^{\circ}}$, -0.62°) was tested for fenchone, since that ketone has been isolated from the oil of *thuja occidentalis*. Using Wallach's method—oxidation with potassium permanganate, steam distillation, further oxidation with concentrated nitric acid, and recovery of the unaltered fenchone by distillation in steam—only a few drops of oil were obtained which were heavier than water and in which no fenchone was found. An attempt to prepare the oxime of the ketone after removal of the thujone by oxidation was equally unsuccessful. The authors conclude, therefore, that fenchone is not present as stated by Brandel.⁴

The residue (about 5%) boiling above $110^{\circ}/40$ mm. ($D_{20^{\circ}}$, 0.980) was dark brown in color and had an odor of stewed prunes. This was hydrolyzed with alcoholic potash, steam distilled, and fractionated. A light yellow oil was obtained whose constants show it to be tanacetyl alcohol ($D_{25^{\circ}}$, 0.9266; $n_D^{25^{\circ}}$, 1.46207; $[\alpha]_D^{25^{\circ}}$, $+29.8$; boiling point at 757 mm., $210-220^{\circ}$), probably present as acetate in the original oil.

From the above results, the authors conclude that the volatil oil of *thuja plicata* is composed of 80-85% thujone, 3-5% pinene, 1-2% tanacetyl acetate, 1-3% tanacetyl alcohol, leaving about 10% to be accounted for by loss due to formation of resin during distillation and experimental losses.

DEPARTMENT OF CHEMISTRY,
UNIVERSITY OF WASHINGTON, SEATTLE.

[CONTRIBUTION FROM THE CONTRACTS LABORATORY, BUREAU OF CHEMISTRY, UNITED STATES DEPARTMENT OF AGRICULTURE.]

THE UNIFICATION OF REDUCING SUGAR METHODS. (A CORRECTION.)

By PERCY H. WALKER.

Mr. M. N. Straughn, of the sugar laboratory of this bureau, has recently called attention to certain errors in the tables for lactose given in the

¹ Wallach, *Ann.*, 275, 179; 286, 109.

² Wallach, *Ibid.*, 336, 251.

³ Wallach, *Ibid.*, 272, 113; 275, 164. Semmler, *Ber.*, 25, 3307. Tiemann and Semmler, *Ber.*, 30, 431.

⁴ *Loc. cit.*