

SCIENTIFIC SECTION

FLUECKIGER'S TEST FOR THYMOL AND CARVACROL.*

BY H. G. HEWITT.

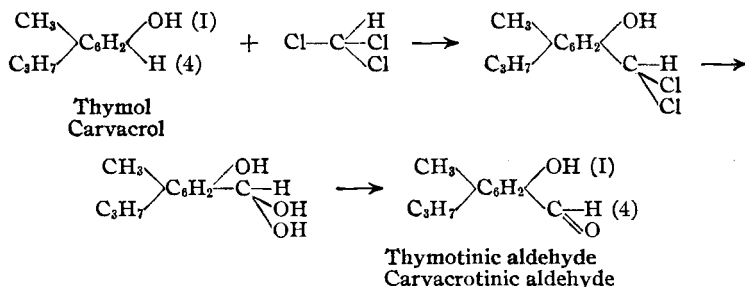
This test consists in acting on the substance to be tested, in chloroform solution, with solid sodium or potassium hydroxide and heating the reaction mixture gently. The presence of either phenol is indicated by the red coloration of the chloroform solution. The test will indicate 0.01 g. of thymol in 20 drops of chloroform with a fiery red color.¹

The color reaction is based on two distinct types of reactions:

(1.) The formation of thymotinic and carvacrotinic aldehyde, respectively; and

(2.) The reaction between these aldehydes and the excess of the respective phenol resulting in the formation of $\text{CH} : (\text{C}_{10}\text{H}_{12}\text{OH})_3$, one of the rosalic acid dyes.²

(ad 1.) That chloroform acts on phenols in the presence of sodium or potassium hydroxide with the synthesis of a hydroxy aldehyde was first observed by Reimer and Tiemann in 1876³, who thus prepared hydroxy benzoic aldehyde. It has since become a general method for the preparation of hydroxy aldehydes,⁴ as the corresponding use of CCl_4 has been employed as a general method for the preparation of the hydroxy⁵ acids since this reaction was first applied to the hydroxy benzoic acids by Reimer and Tiemann in 1876.⁶ The chloroform reaction was first applied by Hans Kobek to thymol in 1883⁷ and by Edward Nordmann to carvacrol in 1884.⁸ The several steps of the reaction become apparent from the following formulas:



(ad 2.) The second phase of the color reaction can be expressed by the following formula:

* Scientific Section, A. PH. A., St. Louis meeting, 1927.

¹ Flueckiger, "Reactionen" (1892), 156.

² Georgievics and Grandmougin, "A Textbook of Dye Chemistry" (1920), 182.

³ *Ber.*, 9, 1268.

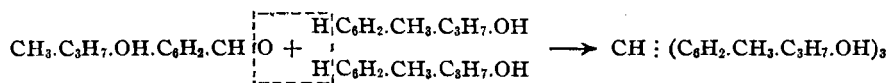
⁴ "Comp. Meyer-Jacobsen, *Org. Chem.*," Bd. 2, I, 516.

⁵ Same references.

⁶ *Ber.*, 9, 1285.

⁷ *Ibid.*, 16, 2096.

⁸ *Ibid.*, 17, 2632.



To use these reactions as a qualitative color test for either phenol appears to have been first suggested by Flueckiger¹ in 1888.

PHYTOCHEMICAL NOTES.*†

NO. 100. THE COROLLA OF *MONARDA FISTULOSA* L.

BY KARL H. RANG.

The corolla of this species has been subjected to chemical examination but once, *viz.* in 1903, and then only to a very partial and preliminary study.¹ Upon distillation, 340 Gm. of dried corollas yielded 9.24 Gm. or 2.71 p. c. of a dark reddish brown oil, the density of which (0.9586) indicated a high percentage of carvacrol and its oxidation products. Flueckiger's test gave a positive reaction for the monatomic phenol, the only substance indentified according to the report. The presence of quinhydrone, however, was assumed with little or no doubt. This assumption was later justified when quinhydrone crystals actually separated from the oil exposed to winter temperature. Upon keeping in a closed specimen bottle with cotton as a background for the blackish crystals, they changed color in the course of time. The thymoquinone part of the quinhydrone molecule apparently sublimed staining the cotton a light yellow, leaving the white crystals of hydrothymoquinone.²

In the summer of 1915 a considerable number of florets were collected and carefully garbled, but unfortunately no one was found to work with this valuable material.

During the summer of 1924 as much as 498 Gm. of dry florets were collected, for the most part about Madison. This material was used in the following experiments.

Moisture Determination.—The xylene method³ was employed using a sufficient amount of hydrocarbon (250 cc.) to leave in the distilling flask enough solvent to cover the florets after all of the moisture had been driven over. Ten-Gm. samples of fresh florets, pickled in xylene as rapidly as the material was collected, yielded 7.5 cc., 7.8 cc. and 7.8 cc., respectively of water, hence 75 p. c., 78 p. c. and 78 p. c., respectively of moisture. This is appreciably higher than the moisture content of the corollas of *Monarda punctata* L. as found by Hewitt.

The air-dried material yielded 0.45 cc. of water in each of three experiments,

¹ Flueckiger, "Pharm. Chemie," 2, 101.

* Scientific Section, A. Ph. A., St. Louis meeting, 1927.

† From the laboratory of Edward Kremers.

¹ J. J. Beck, "Oil from the Corolla of *Monarda Fistulosa*," *Ph. Rev.*, 21, 111.

² Unpublished results.

³ Bulletin No. 134, Forest Service. A. S. Dean, "The Estimation of Moisture in Creosoted Wood."