PHYTOCHEMICAL NOTES.*

97. PRELIMINARY EXAMINATION OF THE COROLLA OF MONARDA PUNCTATA L.

BY H. G. HEWITT.

While the corolla of *Monarda fistulosa* L. has been subjected to chemical examination but once, *viz.*, in 1903, and then only to a very partial study,[1] the corolla of *Monarda punctata* L. has not as yet been studied as to its constituents.

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

During the summer of 1924 as much as 165 Gm. of dry florets were collected, for the most part about 3 miles west of Mazomanie. This material was used in the following experiments:

Moisture Determination.—The xylene method[2] was used, employing a sufficient amount of the hydrocarbon (250 cc.) to leave in the distilling flask enough solvent to cover the florets after all the moisture had been driven over. Ten Gm. of the florets, pickled in xylene in the field as rapidly as the material was being collected, yielded 6.8 cc., 7.0 cc. and 6.9 cc., respectively, of water, hence 68 p. c., 70 p. c., and 69 p. c., respectively, of moisture. This is appreciably lower than the moisture content of the corollas of *M. fistulosa* L. as found by K. H. Rang in a parallel experiment.

The air-dried material yielded 0.6 cc. of water in each of the three experiments, hence contains 6 p. c. of moisture. This is somewhat higher than the moisture content of the air-dried florets of *M. fistulosa* L. as found by Rang.

The distillation being ended, the xylene remaining in the flask was filtered while hot and the filtrate allowed to cool and to evaporate spontaneously. No crystalline deposits resulted whatsoever.

Flueckiger's Test.[3]—The distillate was separated into its aqueous portion and into its hydrocarbon portion. Neither of the separated distillation liquids, however, gave a test for thymol or carvacrol with Flueckiger's reagent. That the xylene does not interfere with this test for thymol and carvacrol was established by experiment. Neither did the xylene residue left upon evaporation of the hydrocarbon yield the characteristic Flueckiger reaction for these two phenols.

Ash Determination.—The air-dried florets were used in this experiment.

	Percentages of Ash.		
	I.	II.	III.
Total ash	10.92 p. c.	10.91 p. c.	10.49 p. c.
Insoluble ash	6.0 p. c.	6.11 p. c.	5.52 p. c.
Soluble ash	4.86 p. c.	4.80 p. c.	4.97 p. c.

I. 0.5078 Gm. of florets yielded 0.0600 Gm. of insoluble ash, 0.1092 Gm. of total ash, hence 0.0492 Gm. of water-soluble ash by difference.

II. 0.5020 Gm. of florets yielded 0.0611 Gm. of insoluble ash, 0.1091 Gm. of total ash, hence 0.0482 Gm. of water-soluble ash by difference.

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* From the laboratory of Edward Kremers.

[1] J. J. Beck, *Ph. Rev.*, 21 (1903), 111.

[2] A. L. Dean, *Bulletin* No. 134 of the Forest Service.

[3] "Flueckiger Reactionen" (1893), 147.

III. 0.5014 Gm. of florets yielded 0.0552 Gm. of insoluble ash, 0.1049 Gm. of total ash, hence 0.0497 Gm. of water-soluble ash by difference.

Comparison with the ash content of the florets of *Monarda fistulosa* L. reveals that whereas the total ash content of the florets of *M. punctata* L. is about two per cent higher, the water-soluble ash content is lower by one per cent and more. Taking into consideration the totally different types of soil on which these two species grow, *M. punctata* L. on sandy soil and *M. fistulosa* L. on clay soil, this difference may not be without significance. It seems all the more desirable, therefore, to make both a qualitative and quantitative examination of the inorganic constituents and to study them with reference to pigmentation, also to the formation of the monatomic phenols.

The Volatile Oil.—Seventy-two Gm. of air-dried florets were distilled with steam. The aqueous distillate was cohobated four times. The total oil, original plus that from the four cohobations, amounted to 2.7 Gm. or 3.45 p. c. The original oil was light reddish brown in color, that from the first cohobation was light yellow, the oils from the second and third cohobations were reddish brown, and that from the fourth was light brown. The amounts, however, were by far too small in each case for separate investigation, hence they were mixed. The mixed oil was light reddish brown in color and by no means as dark as that distilled from the florets of *Monarda fistulosa* L.[1] The density of the oil was 0.9652 at 22°. Comparison with the densities of original and cohobated leaf oils distilled by Sherk[2] reveals that the density of the floret oil is attained only by cohobated leaf oils, whereas in this case the bulk of the floret oil was not obtained by cohobation.

Dilution with heptane did not cause the precipitation of hydrothymoquinone. While this does not show the total absence of this diatomic phenol, it indicates that but little at most can be present. This is significant in connection with the absence of dark color in the oil.

The aqueous distillate from which the cohobated oils had been separated was fractionated. About 25 cc. of this came over between 73° and 99°. Refractionated in 5-cc. fractions these were tested with alkali and iodine. Four of the fractions yielded traces of a yellowish precipitate and the odor of iodoform.

DIMETHYLPHTHALATE AND OTHER ESTERS OF *O*-PHTHALIC ACID.*

BY J. A. HANDY AND L. F. HOYT.[1]

I. Introduction.—The authors' work on phthalates has been continued with special reference to the physical, chemical and pharmacological properties of dimethylphthalate and its comparison with diethylphthalate on whose properties, detection and estimation we have reported in papers Diethylphthalate I–V (1). The properties of some of the other esters of *o*-phthalic acid have also been determined and tabulated.

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[1] *Ph. Rev.*, 21 (1903), 111.

[2] D. C. L. Sherk, JOUR. A. PH. A., 10 (1921), 97.

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

[1] Larkin Co., Inc., Buffalo, N. Y.