[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF KENTUCKY]

The Oil of the Rose Mallow (Hibiscus Moscheutos x H. Coccineus) Seed

BY CHARLES BARKENBUS AND SARAH T. THORN

The rose mallow is a common flowering perennial found in most gardens and produces a large number of seeds which are rather easily harvested. The oil from this seed has never been analyzed and, since a small amount of seed became available in 1931–1932, it was thought advisable to subject it to an analysis.

The botanical relationship of this common garden variety of hibiscus to that of the wild variety is rather obscure. Bobbink and Atkins[1] state that the Rose Mallow came from crosses between *Hibiscus Moscheutos*, a common plant growing wild in the marshes from Massachusetts to Florida and west to Michigan, and *Hibiscus Coccineus*, which is found in the swamp region of southern Georgia. The *Hibiscus* belongs to the Mallon family (*Malvaceae*) and should have an oil approximating that of the cotton seed and okra seed. Our results bear this out.

A search of the literature revealed that very little work had been done on the seed of this type of plant. Only one reference to a *Hibiscus* seed oil could be found. Rosselle seed oil (*Hibiscus sabdariffa*) was reported by C. D. V. Georgi[2] but only a few of the common constants were determined.

Experimental Part

An approximate analysis of the seed was made by the official methods and the results are listed in Table I.

TABLE I

APPROXIMATE ANALYSIS

Ether extract	20.23%	Pentosans	11.10%
Moisture	7.35%	Free invert sugar	0.00%
Protein (N × 6.25)	24.84%	Sugar by inversion	7.12%
Ash	4.15%	Starch (diastase)	0.06%
Crude fiber	21.64%		

The Physical and Chemical Examination of the Oil.—The oil was obtained by extracting 2290 g. of the seed with absolute ether. The ether was evaporated, the last traces being removed in a vacuum, and 262 g. of a clear brown oil with a decided greenish cast was obtained. The physical and chemical properties are given in Table II.

TABLE II

d^{20}	0.9262
n^{20}	1.4760
Iodine no. (Hanus)	107.8
Thiocyanogen iodine no.	68.29
Saponification value	186.55
Reichert–Meissl no.	1.8
Acid value	24.13
Acetyl value	22.92
Unsaponifiable matter, %	1.34
Soluble acids (% butyric acid)	0.34
Insoluble acids, %	95.0
Unsaturated acids (corr.), %	76.67
Saturated acids (corr.), %	14.13
Hexabromide no. of insoluble acids	0.0

The data above place this oil in the edible semi-drying class of oils and are very similar to those of cottonseed oil. The acetyl number, run by the official distillation method, indicates that some hydroxy acids are present while the hexabromide number shows the absence of glycerol linolenate at least in the so-called alpha form. The saturated and unsaturated acids were separated by the lead salt–ether method.

Unsaturated Acids.—The percentage of each of the common unsaturated acids present was calculated by the method of Kaufmann[3] using the iodine number and the thiocyanogen iodine number of the oil and the percentage of unsaturated acids. The results are listed in Table III.

TABLE III

UNSATURATED ACIDS IN THE OIL

Acid	Oleic	Linolic	Linolenic
Acid, %	31.70	43.56	0.06
Glycerol esters, %	33.12	45.53	0.06

These results indicate that linolenic acid is not present either in the alpha or beta form. This is also true of cotton seed oil.

Saturated Acids.—The amount of saturated acids available did not permit a quantitative fractional distillation of the methyl esters as outlined by Baughman and Jamieson.[4] The methyl esters were fractionated but the fractions were so small that each was hydrolyzed and the resulting acids

(1) Private communication, Bobbink and Atkins, Rutherford, N. J., 1931.

(2) C. D. V. Georgi, *Maylan Agri. J.*, **11**, 223 (1923).

(3) Kaufmann, *Z. angew. Chem.*, **42**, 73 (1929).

(4) Baughman and Jamieson, THIS JOURNAL, **42**, 155 (1920).

crystallized to constant melting point. The first fractions which made up most of the distillate gave acids melting at 62°, while the higher boiling fractions which were very small gave acids melting from 67 to 68°. The residue in the flask gave a very small amount of acid having a melting point of 76 to 78°. Though the datum is not quantitative the results indicate that palmitic acid is the main acid present, that stearic acid is in small amounts, and that arachidic acid is present in traces. This again is in agreement with cotton seed oil.

Summary

The oil from the seed of the Rose Mallow (*Hibiscus Moscheutos x H. coccineus*) has been analyzed. The composition of the oil is given in the table.

COMPOSITION OF THE OIL FROM THE SEED OF THE ROSE MALLOW

Glycerides of	
Oleic acid, %	33.12
Linolic acid, %	45.53
Satd. acids (chiefly palmitic with small amount of stearic and trace of arachidic), %	15.60
Unsaponifiable material, %	1.34

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Researches on Quinazolines. XXXIX. The Synthesis of Quinazoline Derivatives Structurally Analogous to the Angostura Alkaloids Galiopine and Galipine[1]

BY ELEANOR BEST MARR[2] AND MARSTON TAYLOR BOGERT

So many of our important alkaloids and other useful medicaments are derivatives of quinoline, isoquinoline or their hydrogenated cycles,[3] that it is surprising to find in the literature so little concerning the pharmacology of the closely related quinazoline derivatives, although quinazoline structurally may be considered as being simultaneously both a quinoline and an isoquinoline.

In fact, practically nothing appears to be known of the pharmacodynamics of quinazoline itself or its simple hydro derivatives, in spite of the fact that quinazoline, its 3,4-dihydro and 1,2,3,4-tetrahydro derivatives have been well known for many years. Further, these three bases are all soluble in water, the first forming a neutral solution and the other two giving alkaline ones.

In paving the way for a coöperative research in this field with some of our pharmacological friends, we have undertaken additional experimental work along the following lines: (1) Improvements in the preparation of quinazoline and its simple hydro derivatives, in order that these fundamental N-heterocycles may be available in sufficient quantities to justify a study of their physiological effects. (2) The synthesis and pharmacological examination of quinazoline derivatives of alkaloidal type, *i. e.*, structurally identical with familiar alkaloids, but containing the quinazoline in place of the quinoline or isoquinoline nucleus. This should give us a clue to whether similarity of molecular architecture is or is not a factor in determining physiological action in these types.

In the first of these two fields, we reported[4] a number of years ago, some modifications of the Riedel[5] process for the preparation of quinazoline. The present paper records a new and very satisfactory method for the production of the 3,4-dihydroquinazoline by the catalytic reduction of quinazoline. This is important, because 1,2,3,4-tetrahydroquinazoline is readily obtained by reducing the 3,4-dihydro derivative with sodium amalgam.[6]

The work in the alkaloidal field was prefaced by the synthesis of the 2-(β-phenethyl) derivatives, to learn what yields we might expect from the reactions contemplated and the most favorable conditions.

These syntheses are outlined below. In the introductory series, R was C_6H_5; in that which followed, it was $C_6H_3(OCH_3)_2$.

It will be observed that 2-(β-phenethyl)-4-quinazolone (III) was synthesized by two differ-

(1) Based upon the Dissertation submitted by Eleanor B. Marr, December, 1934, for the degree of Ph.D. under the Faculty of Pure Science, Columbia University, New York, N. Y., to which Dissertation the reader is referred for further experimental details and literature citations.—M. T. B.

(2) Ferguson Fellow in Chemistry, Columbia University, 1932–1934.

(3) See especially "Therapeutic Agents of the Quinoline Group," by W. V. von Oettingen, Chemical Catalog Co., N. Y., 1933.

(4) Bogert and McColm, THIS JOURNAL, **49**, 2651 (1927).

(5) Riedel, German Patent 174,941 (1905).

(6) Gabriel, *Ber.*, **36**, 811 (1903).