

removed. By fractional crystallization from alcohol, four portions were obtained. The melting point of each fraction was determined as follows: No. 1, m. p. 51-52°; No. 2, m. p. 50°; No. 3, m. p. 49°; No. 4, m. p. 50°. The melting point of myristic acid<sup>1</sup> is 53.8°, that of lauric acid<sup>2</sup> is 43.6° and that of palmitic acid<sup>3</sup> is 62.62°. Nothing definite can be stated about the identification of the above solid acids. Myristic acid would be indicated with probably lauric or palmitic acid.

An abstract from a part of the thesis submitted by Ralph M. Wilson to the Graduate faculty of the University of Oklahoma for the degree of Master of Science in Pharmacy, August 1930.

## SUGGESTED ASSAYS FOR SOME N. F. PREPARATIONS.\*

### 5. PHENOLATED WATER.

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Phenolated Water, N. F. V is wholly dependent upon the phenol which it contains for its therapeutic efficacy; and, as the required phenol content is low (approximately 2 Gm. of phenol to 100 cc. of finished product), it is essential that the full amount be present. The following assay for phenol is, therefore, recommended. It is essentially the method of the United States Pharmacopoeia, X, for Phenol *per se* and for Liquified Phenol.

*Reagents.*—1. 0.1 *N* Bromine solution (Koppeschaar's Solution, U. S. P. X)  
 2. 0.1 *N* Sodium thiosulphate solution, U. S. P. X  
 3. Hydrochloric acid, U. S. P. X  
 4. 20% Potassium iodide solution  
 5. Chloroform  
 6. Starch test solution, U. S. P. X.

*Procedure.*—Dilute 10 cc. of Phenolated Water to 100 cc. Introduce into a 250-cc. glass-stoppered Ehrlenmeyer flask 10 cc. of the diluted sample containing about 0.0206 Gm. of phenol. Add to the contents of the flask and likewise to the contents of a control flask prepared in the same way but containing none of the sample, 25 cc. of 0.1 *N* bromine. Quickly introduce into each flask 5 cc. of hydrochloric acid, insert the stopper and shake during half an hour. Add to each flask as quickly as possible, 5 cc. of potassium iodide solution (20%) and immediately replace the stoppers to prevent the escape of any bromine vapor. Shake well and add to each flask 1 cc. of chloroform to dissolve the precipitated tribromophenol. Titrate the liberated iodine with 0.1 *N* sodium thiosulphate, using starch T.S. as the indicator. The 0.1 *N* bromine remaining in the flask containing the sample subtracted from that remaining in the control flask represents the bromine consumed in the formation of tribromophenol. Each cc. of 0.1 *N* bromine consumed corresponds to 0.001568 Gm. of C<sub>6</sub>H<sub>5</sub>OH.

*Suggested Standard.*—Phenolated water contains in each 100 cc., not less than 2 Gm. and not more than 2.2 Gm. of C<sub>6</sub>H<sub>5</sub>OH.

<sup>1</sup> Lewkowitsch, 6th Edition, page 158.

<sup>2</sup> *Ibid.*, page 160. <sup>3</sup> *Ibid.*, page 162.

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## DISCUSSION.

Two samples of Phenolated Water were carefully prepared from an assayed sample of Liquified Phenol, U. S. P. X. In the assay of the sample, 10-cc. portions of a solution containing 3.768 Gm. of Liquefied Phenol in 1000 cc. were used and gave the following results:

TABLE I.—ASSAY LIQUEFIED PHENOL.

Cc. of Sample.	Cc. 0.1 N Br <sub>2</sub> Taken.	Cc. 0.1 N Na <sub>2</sub> S <sub>2</sub> O <sub>8</sub> Required.	Cc. 0.1 N Br <sub>2</sub> Consumed.	Gm. C <sub>6</sub> H <sub>5</sub> OH Found in 1000 cc. of Solution.
10	38.54	17.14	21.40	3.36—
10	38.54	17.09	21.45	3.36+
10	38.59	17.14	21.45	3.36+
10	38.60	17.14	21.46	3.36+
10	38.60	17.14	21.46	3.36+
10	38.60	17.14	21.46	3.36+
Average = 3.36				

Using the above figure, the percentage of phenol (C<sub>6</sub>H<sub>5</sub>OH) was calculated to be 89.17%, viz:  $\frac{3.36 \times 100}{3.768} = 89.17\% \text{ C}_6\text{H}_5\text{OH}$ .

Sample No. 1 of Phenolated Water was prepared by diluting 22 cc. of the Liquefied Phenol to 1000 cc. The sample was analyzed and the following results were obtained:

TABLE II.—ASSAY PHENOLATED WATER.

Cc. of Diluted Sample.	Cc. 0.1 N Br <sub>2</sub> in Control.	Cc. 0.1 N Br <sub>2</sub> in Unknown.	Cc. 0.1 N Br <sub>2</sub> Consumed.	Gm. of C <sub>6</sub> H <sub>5</sub> OH Found per 100	Gm. of C <sub>6</sub> H <sub>5</sub> OH Calc. per 100
				Cc. of Aqua Phenolata.	Cc. of Aqua Phenolata.
10	38.74	25.66	13.08	2.05+	2.09
10	38.74	25.56	13.18	2.07—	2.09
10	39.04	25.76	13.28	2.08+	2.09
10	39.04	25.76	13.28	2.08+	2.09
Average = 2.07				2.09	
Error based on calculated amount = 0.02					
Percentage error = $\frac{0.02 \times 100}{2.09}$ or 0.95%					

Sample No. 2 of Phenolated Water was prepared by diluting 22 cc. of the Liquefied Phenol to 1020 cc. In the assay of this sample, the following results were obtained:

TABLE III.—ASSAY PHENOLATED WATER, NO. 2.

Cc. of Diluted Sample.	Cc. 0.1 N Br <sub>2</sub> in Control.	Cc. 0.1 N Br <sub>2</sub> in Unknown.	Cc. 0.1 N Br <sub>2</sub> Consumed.	Gm. of C <sub>6</sub> H <sub>5</sub> OH Found per 100	Gm. of C <sub>6</sub> H <sub>5</sub> OH Calc. per 100
				Cc. of Aqua Phenolata.	Cc. of Aqua Phenolata.
10	39.04	26.06	12.98	2.04	2.05
10	39.04	25.96	13.08	2.05+	2.05
10	32.35	19.35	13.00	2.04	2.05
10	32.35	19.35	13.00	2.04	2.05
Average = 2.04				2.05	
Error based on calculated amount = 0.01					
Percentage error = $\frac{0.01 \times 100}{2.05}$ or 0.49%					