

TABLE II (Concluded)

Milking number	Glucose, %	Lactose, %	Yield, kg.	Fat, %	Solids, %	Specific gravity
21	.17	4.50	6.565	3.11	11.76	1.0335
22	— .04	4.78	5.641	3.87	12.82	1.0345
23	.00	4.74	6.444	3.01	11.86	1.0350
24	.01	4.77	5.680			1.0340

### Summary

A method for estimating small amounts of glucose in milk has been developed, which depends on selective fermentation of the glucose and measurement of the rotary power of fermented and unfermented milk.

Changes in rotary power corresponding to glucose percentages varying from zero to 0.35% of glucose have been found in normal cow's milk.

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[CONTRIBUTION FROM THE LABORATORY OF ORGANIC CHEMISTRY OF THE UNIVERSITY OF WISCONSIN]

## THE IDENTIFICATION OF PHENOLS

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RECEIVED NOVEMBER 3, 1930

PUBLISHED JANUARY 12, 1931

Reagents which have been recommended for the identification of phenols are diphenylcarbamine chloride,<sup>1</sup> *p*-nitrobenzyl bromide<sup>2</sup> and 3,5-dinitrobenzoyl chloride.<sup>3</sup> Since none of these reagents in the hands of the inexperienced student has given entirely satisfactory results, the characterization of phenols by the use of chloro-acetic acid has been developed in this Laboratory.

Chloro-acetic acid reacts smoothly with phenols in aqueous sodium hydroxide giving good yields of the sodium salts of aryloxyacetic acids. The acids themselves are crystalline solids easily purified by recrystallization from water. One gram of a phenol furnishes an amount of the derivative sufficient for the determination of its neutral equivalent, often a valuable aid in identification, in addition to its melting point.

The experimental procedure is quite simple. To a mixture of 1.0 g. of the phenol with 3.5 ml. of 33% sodium hydroxide is added 2.5 ml. of a 50% chloro-acetic acid solution; if necessary, a little water is added to dissolve the sodium salt of the phenol. The test-tube containing the solution is stoppered loosely and heated for one hour in a gently boiling water-bath. The solution is cooled, diluted, acidified to congo red with a mineral acid, and extracted once with ether. The ether extract is washed once with a little water, and the aryloxyacetic acid is removed by washing with dilute

<sup>1</sup> Herzog, *Ber.*, **40**, 1831 (1907).

<sup>2</sup> Reid, *THIS JOURNAL*, **39**, 304 (1917); Lyman and Reid, *ibid.*, **42**, 615 (1920).

<sup>3</sup> Brown and Kremers, *J. Am. Pharm. Assocn.*, **11**, 607 (1922).

sodium carbonate solution. Acidification of this extract gives the free acid, which is recrystallized from water.

The accompanying table contains the data which have been observed on the aryloxyacetic acids prepared in this Laboratory. Those acids for which no reference is given are new compounds. As was expected,<sup>4</sup> no nitrophenoxyacetic acids were isolated when *o*- or *p*-nitrophenols were used, and the yield was unsatisfactory when *m*-nitrophenol was used.

TABLE I

THE MELTING POINTS AND NEUTRAL EQUIVALENTS OF A FEW ARYLOXYACETIC ACIDS

	Phenol	Yield, g.	Neut. equiv.		M. p., °C. (uncorr.)	
			Obs.	Calcd.	Obs.	Literature
1	Phenol	0.38	155	152	98-99	98-99 <sup>b</sup>
2	<i>o</i> -Cresol	.65	166	166	151-152	151-152 <sup>c</sup>
3	<i>m</i> -Cresol	1.01	168	166	102-103	102 <sup>d</sup>
4	<i>p</i> -Cresol	0.75	167	166	134-136	135-136 <sup>e</sup>
5	<i>o</i> -Chlorophenol	.37	185.5	186.5	143-145	.....
6	<i>m</i> -Chlorophenol	.40	185	186.5	108-110	.....
7	<i>p</i> -Chlorophenol	.60	187	186.5	155-156.5	151-152 <sup>f</sup>
8	<i>o</i> -Bromophenol	.26	230	231	141-143	142.5-143 <sup>g</sup>
9	<i>m</i> -Bromophenol	.47	234	231	107-108.5	.....
10	<i>p</i> -Bromophenol	.60	228	231	157	153-154 <sup>h</sup>
11	<i>o</i> -Iodophenol	.20	277	278	134-135	.....
12	<i>m</i> -Iodophenol	.36	279	278	114-115.5	.....
13	<i>p</i> -Iodophenol	.48	278	278	154-156	155-156 <sup>i</sup>
14	<i>o</i> -Methoxyphenol	.41	180	182	116-116.5 <sup>a</sup>	121 <sup>j</sup>
15	<i>m</i> -Methoxyphenol	.63	182	182	111-113 <sup>a</sup>	115-118 <sup>k</sup>
16	<i>p</i> -Methoxyphenol	.56	182	182	110-112	.....
17	Thymol	.45	208	208	148-149	147-148 <sup>l</sup>
18	Carvacrol	.45	211	208	150-151	149 <sup>m</sup>
19	$\alpha$ -Naphthol	.50	199	202	191-192	190 <sup>n</sup>
20	$\beta$ -Naphthol	.50	203	202	153-154.5	156 <sup>o</sup>

<sup>a</sup> Despite alternate recrystallizations from water and from benzene, the melting point remained unchanged; <sup>b</sup> Hantzsch, *Ber.*, 19, 1296 (1886); <sup>c</sup> Oglialoro and Cannone, *Gazz. chim. ital.*, 18, 511 (1888); <sup>d</sup> Oglialoro and Forte, *ibid.*, 20, 508 (1890); <sup>e</sup> Gabriel, *Ber.*, 14, 923 (1881); <sup>f</sup> Peratoner, *Gazz. chim. ital.*, 28, I, 239 (1898); <sup>g</sup> Auwers and Haymann, *Ber.*, 27, 2799 (1894); <sup>h</sup> Fritzsche, *J. prakt. Chem.*, [2] 20, 295 (1879); <sup>i</sup> Marveli, Gambetta and Rimini, *Gazz. chim. ital.*, 50, I, 173 (1920); <sup>j</sup> Auwers and Haymann, *Ber.*, 27, 2804 (1894); <sup>k</sup> Gilbody, Perkin and Yates, *J. Chem. Soc.*, 79, 1409 (1901); <sup>l</sup> Spica, *Gazz. chim. ital.*, 10, 341 (1880); <sup>m</sup> Spica, *ibid.*, p. 345; <sup>n</sup> Spica, *ibid.*, 16, 438 (1886); <sup>o</sup> Spitzer, *Ber.*, 34, 3191 (1901).

The assistance of Professor S. M. McElvain is gratefully acknowledged.

### Summary

Aryloxyacetic acids, easily prepared from chloro-acetic acid and phenols are recommended as derivatives for the identification of the latter compounds.

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<sup>4</sup> Cf. Hewitt, Johnson and Pope, *J. Chem. Soc.*, 103, 1630 (1913).