Quantitative Determination of Resorcinol in Presence of Phenol

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Abstract □ A simple and accurate method for the quantitative determination of resorcinol in the presence of phenol is reported. The method is based on the formation of indophenol by reacting resorcinol with 2,6-dibromoquinone-4-chlorimide. The concentration of indophenol can be measured spectrophotometrically. This method is recommended for the determination of resorcinol in resorcinol–phenol–boric acid solution and carbol-fuchsin solution. The decomposition products of resorcinol and phenol, *e.g.*, colored quinones, do not interfere with the assay procedure.

Keyphrases □ Resorcinol—spectrophotometric analysis in presence of phenol, effect of pH, decomposition products □ Carbol-fuchsin solution—spectrophotometric analysis of resorcinol, effect of pH, decomposition products □ Spectrophotometry—analysis, resorcinol in presence of phenol, effect of pH, decomposition products

In most quantitative methods for the determination of resorcinol, phenol interferes (1). A combination of a bromometric technique and NMR spectroscopy was recommended for the analysis of resorcinol in the presence of phenol (1). However, the method lacks sensitivity and may not be adaptable by many laboratories due to the lack of an NMR spectrometer.

This paper reports a very simple, sensitive, and accurate colorimetric method for the quantitative analysis of resorcinol in the presence of phenol. The method is based on the formation of a pink-colored indophenol by reacting resorcinol with 2,6-dibromoquinone-4-chlorimide (I), as recommended by Gibbs (2). The decomposition products, *e.g.*, colored quinones, and phenol do not interfere with the assay procedure.

This method may be considered more sensitive than a reported GLC method (3) since the latter requires a concentration of 3 mg/ml for analysis. Due to the small injection volume (1–2 μl), the total resorcinol required is less in the GLC method. Nevertheless, the sensitivity of the developed method can be improved further (see *Discussion*). The developed method is more sensitive than the 4-dimethylaminobenzaldehyde method (4), since the latter requires about four times more resorcinol for the same absorbance value.

EXPERIMENTAL

Chemicals and Reagents—All chemicals and reagents were USP, NF, or ACS grade. Compound I[1] (97%), phenol[2] loose crystals, and resorcinol[3] were used without further purification.

Preparation of Solutions—*Resorcinol–Phenol–Boric Acid Solution*—This solution was prepared as reported earlier (1).

Carbol-fuchsin Solution—This solution was prepared according to NF X (5).

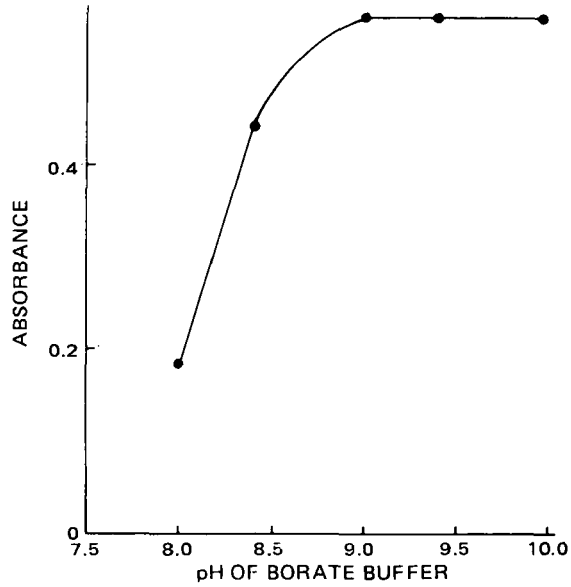

Figure 1—*Plot of pH* versus *absorbance.*

Compound I Solution—This solution was prepared fresh daily by dissolving 40.0 mg of I in 10.0 ml of methanol.

Sodium Borate Solution—A 1% aqueous solution was prepared fresh daily.

Borate Buffer Solution—The alkaline borate buffer solutions of various pH values (8.0, 8.4, 9.0, 9.4, and 10.0) were prepared according to USP XVIII (6).

Solutions of Potassium Ferricyanide—Four and 10% solutions in water were prepared fresh daily.

Standard Solutions of Resorcinol and Phenol for Colorimetric Analysis—The following were prepared in water every week: (*a*) a solution containing 10.0 μg of resorcinol/ml, (*b*) a solution containing 4.5 μg of phenol/ml, and (*c*) a solution containing 10.0 μg of resorcinol/ml and 4.5 μg of phenol/ml.

Wavelength of Maximum Absorption for Resorcinol—A 5.0-ml quantity of the standard aqueous solution of resorcinol was diluted to 10.0 ml with water. Then 2.0 ml of the borate buffer solution (pH 9.4) and 0.1 ml of the I solution were added and mixed. The solution was immediately scanned in the visible range. The maximum absorption was recorded at about 530 nm. This wavelength was selected for further investigations.

Effect of pH on Assay Sensitivity—The proposed procedure was followed to determine the absorbance values[4] at 530 nm using borate buffer solutions of various pH values (8.0, 8.4, 9.0, 9.4, and 10.0). A blank was prepared by substituting water for the resorcinol solution. All absorbance values were recorded after 90 sec of mixing (Fig. 1). For further investigations, a buffer solution of pH 9.4 was selected.

Calibration Curve—A 2.0-ml quantity of the borate buffer solution (pH 9.4) was mixed with 2.0, 3.0, 4.0, and 5.0 ml, respectively, of the standard aqueous resorcinol solution, and each mixture was brought to volume (12.0 ml) with water. Then 0.1 ml of the I solution was added, the solution was mixed, and the absorbance of each solution was measured within 50–120 sec against a reagent blank. Beer's law was followed.

[1] Aldrich Chemical Co., Milwaukee, Wis.
[2] Matheson, Coleman & Bell, Norwood, Ohio.
[3] Fisher Scientific Co., Fairlawn, N.J.

[4] A Bausch & Lomb Spectronic 20 was used.

Table I—Assay Results on Resorcinol

Product or Solution	Resorcinol Found, %
Standard aqueous solution of resorcinol and phenol	100.00
Standard aqueous solution of phenol	0.00
Resorcinol–phenol–boric acid solution	99.46
	99.65
	99.82
Average =	99.64
Average deviation =	±0.12
Carbol-fuchsin solution	100.25
	100.48
	100.25
Average =	100.33
Average deviation =	±0.10
All potassium ferricyanide-treated phenol solutions	0.00

Interference from Phenol—The procedure reported under *Calibration Curve* was repeated on a 5.0-ml sample of the standard aqueous solution of phenol and the standard solution containing both resorcinol and phenol. The results were calculated using a standard value (0.560) for resorcinol since Beer's law was followed (Table I).

Assay of Resorcinol–Phenol–Boric Acid Solution and Carbol-fuchsin Solution—Both solutions were diluted with water as follows: (*a*) 7.5 ml to 100.0 ml, (*b*) 5.0 ml of dilution (*a*) to 250.0 ml, and (*c*) 5.0 ml of dilution (*b*) to 250.0 ml.

To 10.0 ml of dilution (*c*), 2.0 ml of the borate buffer solution (pH 9.4) and 0.1 ml of the I solution were added and mixed. Within 50–120 sec, the absorbance values were recorded at 530 nm against a reagent blank made by substituting water for the assay solutions. The results for resorcinol were calculated using a standard value (0.336) since Beer's law was followed (Table I).

A minor interference from basic fuchsin was taken into consideration as follows. A 10.0-ml quantity of dilution (*c*) from the carbol-fuchsin solution was mixed with 2.0 ml of the borate buffer solution (pH 9.4). The absorbance of this solution was measured at 530 nm against a blank made by mixing 2.0 ml of the borate buffer solution with 10.0 ml of water. Before calculating the results on the carbol-fuchsin solution, this absorbance value (approximately 0.015) was subtracted from the absorbance value obtained in the assay procedure.

Interference from Decomposition Products of Resorcinol and Phenol—A 5.0-ml quantity of an aqueous solution of either resorcinol (0.5%) or phenol (0.225%) was transferred to a 50-ml volumetric flask. An appropriate quantity of potassium ferricyanide solution (1.0, 3.0, or 5.0 ml of 4% or 5.0 ml of 10% solution) was added and mixed. Then the mixture was brought to volume with 1% sodium borate solution. Then 5.0 ml of this mixture was diluted to 500 ml with water, and 10.0 ml of this mixture was mixed with 2.0 ml of the borate buffer solution (pH 9.4) and 0.1 ml of the I solution.

The absorbance of each solution (A_a) was measured after 50 sec at 530 nm against a blank made by substituting methanol for the I solution. The standard reading (A_s) for the resorcinol solution was determined similarly but without the addition of the potassium ferricyanide solution. The percent of resorcinol present in each solution was determined by:

$$\frac{A_a}{A_s} \times 100 = \% \text{ resorcinol} \qquad \text{(Eq. 1)}$$

The results on phenol solutions are presented in Table I. The results on resorcinol solutions are presented in Fig. 2. The percent of resorcinol oxidized was calculated by difference (100 − percent found).

DISCUSSION

The results (Table I) indicate that resorcinol can be assayed in the presence of phenol without any interference. Since phenol also reacts with I (2) to form a blue-colored indophenol, color must be measured within 50–120 sec of mixing the solutions. The full resorcinol color develops in about 50 sec, and phenol begins to react and cause interference after 120 sec. The method is simple and accurate. Beer's law is followed.

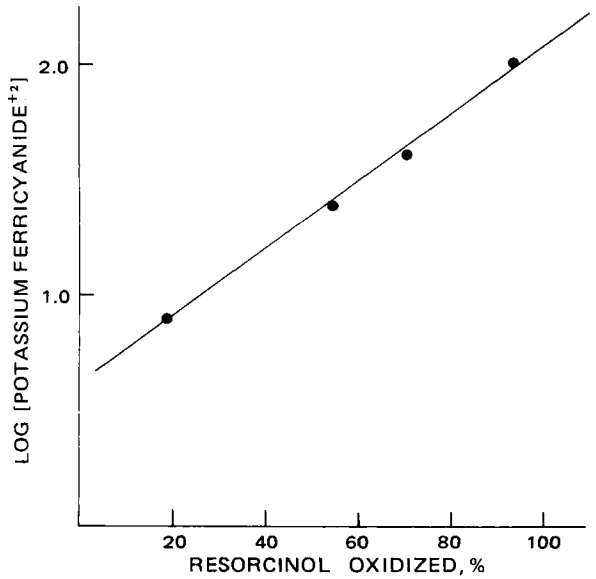

Figure 2—*Plot of percent resorcinol oxidized* versus *log of concentration (grams per 100 ml) of potassium ferricyanide.*

The effect of pH on the sensitivity of the assay method indicates (Fig. 1) that a buffer solution of pH 9–10 could be used. However, at pH 10 the phenol reaction proceeds faster, and at pH 9.0 the resorcinol reaction is slower; therefore, a pH value of 9.4 was preferred.

The decomposition products of resorcinol and phenol, *e.g.*, colored quinones, do not interfere with the assay method (Table I and Fig. 2), as was expected since two phenolic groups are required for this reaction. With one phenolic group, the reaction is delayed (2) and, therefore, does not interfere. If the benzene ring is ruptured, there is no reaction at all (2). When the resorcinol solution was treated with 5.0 ml of 10% solution of potassium ferricyanide in an alkaline sodium borate buffer solution, the decomposed solution was intensely colored (violet). On analysis with the method developed, the resorcinol contents were found to be only 6.5% of the original (Fig. 2).

By using smaller cells, such as a 1-cm cell of a spectrophotometer that requires approximately 3 ml of solution for measurement, the sensitivity of the method can be improved four times. Moreover, concentrations less than 3 μg/ml also can be easily measured since the absorbance value of a 3-μg/ml solution is fairly high (~0.336). This use will further improve the sensitivity of the developed method.

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