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THE DETERMINATION OF PHENOL BY THE RING-OVEN TECHNIQUE

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Abstract. A simple, quick, precise and sensitive method for the determination of phenol has been devised and the effect of interferences has been studied by Weisz ring-oven technique. The shelf-life of the standard scale has also been investigated.

Phenols are considered to be man-made contaminants. Recent trend towards more stringent restrictions on pollution has heightened the need for a simple, quick precise and sensitive method for the determination of phenolic compounds. These compounds possess unpleasant odour and taste especially in chlorinated civil water supplies and hence are considered pollutants.

The Weisz ring-oven technique, since its invention, has played an important role not only in qualitiative but also in quantititative analysis. Due to its being simple and convenient, the technique has gained wide importance in the field of analytical chemisty. Many colour reactions, studied till now by optical methods, requiring comparably costly apparatus associated with its maintenance and after sale service, have been studied with this technique and in most cases even the accuracy and sensitivity of the results has been increased to a resonable extent. In view of the aforesaid, we have tried upon devising a method for the determination of phenol with the help of this technique. The famous colour reaction of phenol with amino antipyrine and potassium ferricyanide[1–4] has been utilized and some of the results of this investigation are reported in this communication.

Experimental

Reagents

Phenol. An exactly weighed 1 g of phenol (Analar-BDH) was dissolved in 100 ml water and used.

Amino Antipyrine. 0.2% solution (KOCH- Light Labs, England) was prepared.

Potassium Ferricyanide. Two grams analytical grade substance was dissolved in water to which 5 ml of 1M NaOH solution was added and volume was made to 100 ml with water.

All other reagents used were also of analytical grade.

Apparatus

Weisz ring-oven, prepared in our precision Workshop according to the prescribed measurements, officially calibrated micropipettes alongwith other volumetric glassware and Whatman filter paper No. 41 were used for the experiments.

Procedure

At the centre of filter paper 1 µl of phenol test solution and 2 µl of amino antipyrine were applied one after the other and it was allowed to dry out in air for about 2 min to control the expansion of the applied solutions. After that 1 µl of potassium ferricyanide was added to the same point The colour compound produced was washed into the ring zone on ring-oven maintained at 110°. Ten washings with water were sufficient for complete washing of the reaction product to the ring zone. Three rings with various volumes of phenol test solution were prepared by this method and all were evaluated against a standard scale (comprised of 8 standard rings) prepared by taking 1, 2, 4, 6, 8 and 10 µl of standard phenol solution using the above method. The results were calculated according to the known methods[5].

Using the above procedure shelf-life of the standard scale was checked by evaluating against it the known solutions, after regular intervals of time.

Interferences

The following method[6] for the study of the effect of interferences on the determination of phenol was followed. A ring (I) containing 1 µg of the interfering substance and the other ring (II) containing 0.4 µg of phenol and 1 µg of interfering substance was prepared. The substance was confirmed as noninterfering when ring (I) was identical to the blank (III) and the second matched with the 0.4 µg phenol ring of the standard scale.

The strength of unknow nsolutions was found according to Weisz's method.[5]

Results and Discussion

The reaction between phenol, amino antipyrine and potassium ferricyanide was very quick. A red colour was immediately produced on the application of all the substances in question on the paper. Out of various tried solvents, 8–10 washings with water proved to be the best suited for washing the reaction product to the ring zone on the ring-oven. The sensitivity of the method, as apparent from some results reported in Table 1, is high and phenol in as low concentrations as 150 µg/ml can be determined with quite a good accuracy and the limits of determination are from 150 µg/ml to 2.0 mg/ml with a maximum error of —7.77%. The method reported is direct, simple, quick, accurate and sensitive. Moreover, the apparatus used is not so expensive and is convenient to handle.

Stability of the standard scale was also studied and is as evident from Table 2 it can be safely used for five days.

TABLE 1. DETERMINATION OF PHENOL.

Amount Taken	Amount Found	Error (%)
150 μg ml	145 μg/ml	—3·33
300 ,,	300 ,,	—0·00
500 ,,	480 ,,	—4·00
600 ,,	640 ,,	+6·66
700 ,,	664 ,,	—5·14
900 ,,	830 ,,	—7·77
1·20 mg/ml	1·152 mg/ml	+4·00
1·50 ,,	1·470 ,,	—2·00
1·80 ,,	1·770 ,,	—1·66
2·00 ,,	1·890 ,,	—5·50

TABLE 2. STABILITY OF THE STANDARD SCALE.

Days	Amount Taken	Amount Found	Error (%)
1	150 μg/ml	145 μg/ml	—3·33
	450 ,,	455 ,,	+1·11
2	180 ,,	175 ,,	—2·77
	300 ,,	306 ,,	+2·00
3	1·800 mg/ml	1·688 mg/ml	—6·22
	1·500 ,,	1·400 ,,	—6·66
4	600 μg/ml	555 μg/ml	—2·66
	750 ,,	730 ,,	—2·86
5	700 ,,	680 ,,	—2·85
	600 ,,	620 ,,	+3·33
6	200 ,,	165 ,,	—17·50
	250 ,,	210 ,,	—16·00

o-Cresol, resorcinol, pyrogallol phloroglucinol, hydroquinone and catechol interfere with phenol determination at all levels.

In view of the stablity of the standard scale, sensitivity, accuracy and simplicity of the method, it could be used for routine quality check of phenol and in pollution problems to determine phenol content. The method could also be used for a number of its applications to be developed by various users later on.

References

1. E. Emerson, J. Org. Chem., **8**, 1692 (1943).
2. S. Gottlieb and P.M. Marsh, Ind. Eng. Chem. Anal Ed., **18**, 16 (1946).
3. E.F. Mohler and L.N. Jacoles, Anal. Chem., **29**, 1369 (1957).
4. R.G. Locoste, S.H. Venalile and J.C. Stone, Anal. Chem., **31**, 1246 (1959).
5. H. Weisz, *Microanalysis by the Ring-Oven Technique* (Pergamon, Oxford, 1970), second edition.
6. V. Dharmarajan and Philip W. West, Anal Chem. Acta, **57**, 471 (1971).