

## NOTES

**Polarographic Determination of Nitrite with Phenol**

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A simple, rapid and fairly accurate polarographic method is developed for the determination of nitrite by diazotization with phenol. The diazotization is carried out at pH 1 and at 0°C.

The determination of nitrite in water and waste waters is ecological importance since it is reported that nitrite compounds caused bladder cancer associated with schistosomiasis and urinary infections<sup>1</sup>, gastroduodenal ulcer<sup>2</sup>. Sometimes nitrate is reduced to nitrite and hydrogenate proteins to secondary amines by mycobacteria and these in turn promote the formation of carcinogenic nitroso amines<sup>3</sup>. Further aromatic amines are poisonous cause bladder cancer. Nitrite, generally enter surface waters through industrial effluents from dye industry, coal mining, petrochemicals, fertilizers, leather tanning industry and paper and pulp industry.

Nitrite is generally estimated by UV-visible spectrophotometric methods<sup>6-11</sup>. But most of these methods lack selectivity and sensitivity. Literature survey reveals that not much work has been carried out on the polarographic method of estimation of nitrite at low concentrations. The polarographic methods offer advantageous over spectrophotometric methods in the sense that the limitations mentioned earlier do not figure in this technique. Therefore, a rapid and a simple polarographic method is developed for the determination of nitrites in small quantities and the results are communicated in this paper.

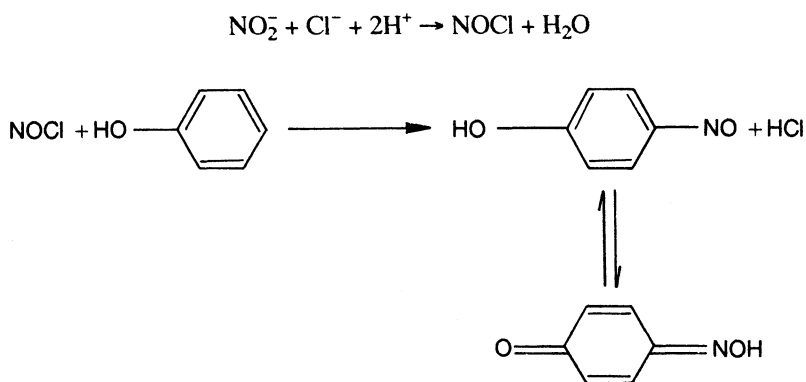
The chemicals employed in the studies are of analytical reagent grade. Polarograms are recorded with DC pen recording polarograph supplied by ELICO Pvt. Limited, Hyderabad, India. The capillary with the characteristics  $2.040 \text{ mg}^{2/3} \text{ sec}^{-1/2}$  and saturated calomel electrode are used as the indicator and reference electrode respectively. The pH measurements are made with digital pH meter.

2 Ml of the stock solution of aqueous phenol ( $10^{-2}$  M) is added to 2 ml of solution of pH1 and cooled to 0°C in ice bath. An aliquot of the separately cooled solution of  $\text{NaNO}_2(10^{-3})$  solution is added to the above mixture. The volume of reaction mixture is then raised to 20 ml with buffer solution. The resulting mixture is kept for 30 min. at 0°C in ice bath. The cooled mixture is

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de-aerated by passing pure nitrogen gas and taken in the polarographic cell for draw the polarogram.

The position of the wave on the potential areas ( $E_{1/2} = -1.20$  V SCE) suggests that the wave is due to the reduction of nitrosophenol or the isomeric form of it shown. Reaction mechanism is reported in the literature<sup>12</sup> and is shown below:



Hydrogen and chloride ions are available in the buffer solution.

The influence of mercury column height on the wave height and the semilogarithmic analysis of the waves indicate that the wave is diffusion controlled but irreversible. The irreversible nature of the wave is also confirmed from the shift of half-wave potentials towards more negative values with rise in the concentration of the compound. The heights of polarograms recorded with different concentrations of nitrite are presented in Table 1. The height varies linearly with concentration and suggests that the nitrite can be determined accurately in the concentration range 3–25 mg/l. Presence of inorganic salts such as NaCl, CdCl<sub>2</sub>, MgCl<sub>2</sub>, MgSO<sub>4</sub> do not interfere in the determination.

TABLE 1  
WAVE HEIGHTS OF POLAROGRAMS

Amount of NO <sub>2</sub> <sup>-</sup> (mg/l)	Wave height (micro-amperes)
4.7	4.0
9.4	6.8
14.1	10.5
18.7	14.1
23.4	17.5

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