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SPECTROPHOTOMETRIC DETERMINATION OF NITRITE

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SPECTROPHOTOMETRIC methods for the estimation of nitrite based on diazocoupling reactions are well known. When these methods are applied for the determination of nitrogen dioxide in air after fixing in alkali, they suffer from the interference of sulphur dioxide. The interference was overcome by the addition of acetone¹, by selective oxidation using hydrogen peroxide² or by coupling the diazotised amine in alkaline medium³. The present method is based on the diazotization of *p*-nitroaniline with nitrite and coupling the product with resorcinol in alkaline medium. The present method is also free from the interference of the most of the common ions associated with the determination of nitrogen dioxide in air after sampling in alkali or nitrite in water samples. Experimental variables have been investigated in detail and the optimum values obtained are incorporated in the working procedure.

The standard sodium nitrite solution had 10 µg NO₂⁻ per ml. *p*-nitroaniline (0.01%) in 0.05 N hydrochloric acid was prepared. The coupling agent was 0.1% solution of resorcinol in 2N sodium hydroxide. The trapping solution of sodium hydroxide (0.2 N) had 2 ml of *n*-butanol per litre. This was diluted twice with water for air sampling.

Aliquots containing 0–25 µg of nitrite were transferred to six test tubes each containing 5 ml of 0.2 N sodium hydroxide and diluted to 10 ml. These solutions were transferred to six 25 ml volumetric flasks kept in ice bath (0–5°C) each containing 5 ml of

p-nitroaniline reagent. The solutions were mixed well and allowed to stand for 5 min. Five ml of coupling agent were added to all the flasks, mixed well, diluted to the mark and allowed to attain room temperature. Absorbance, measured at 555 nm using 10 mm cells, was plotted against the concentration of nitrite which was a straight line passing through the origin (figure 1a).

Nitrogen dioxide in air

The samples of air containing NO₂ are passed through 10 ml of the trapping solution at 400 ml/minute and the nitrogen dioxide is fixed as nitrite. The trapping solution containing not more than 25 µg of nitrite is used to determine the concentration of the nitrite following the procedure described under the calibration graph.

Nitrite in water sample

Ten ml of the water sample containing not more than 25 µg of NO₂⁻ was transferred to a 25 ml beaker. Two ml of 0.05 M EDTA and 1 ml of 5% triethanolamine solution were added and mixed well. This solution was transferred to the 25 ml standard flask kept in ice bath containing 5 ml of *p*-nitroaniline reagent and the analysis was completed by following the procedure described under the calibration graph.

Results and discussion

Initial studies were carried out with sodium nitrite solution in 10 ml of 0.1 N alkali to fix the optimum acidity for the diazotization. Maximum colour was obtained when the overall acidity for diazotization was at least 0.125 N with respect to HCl and further increase in acidity upto 0.275 N did not affect the absorbance. Variation of the concentration of amine

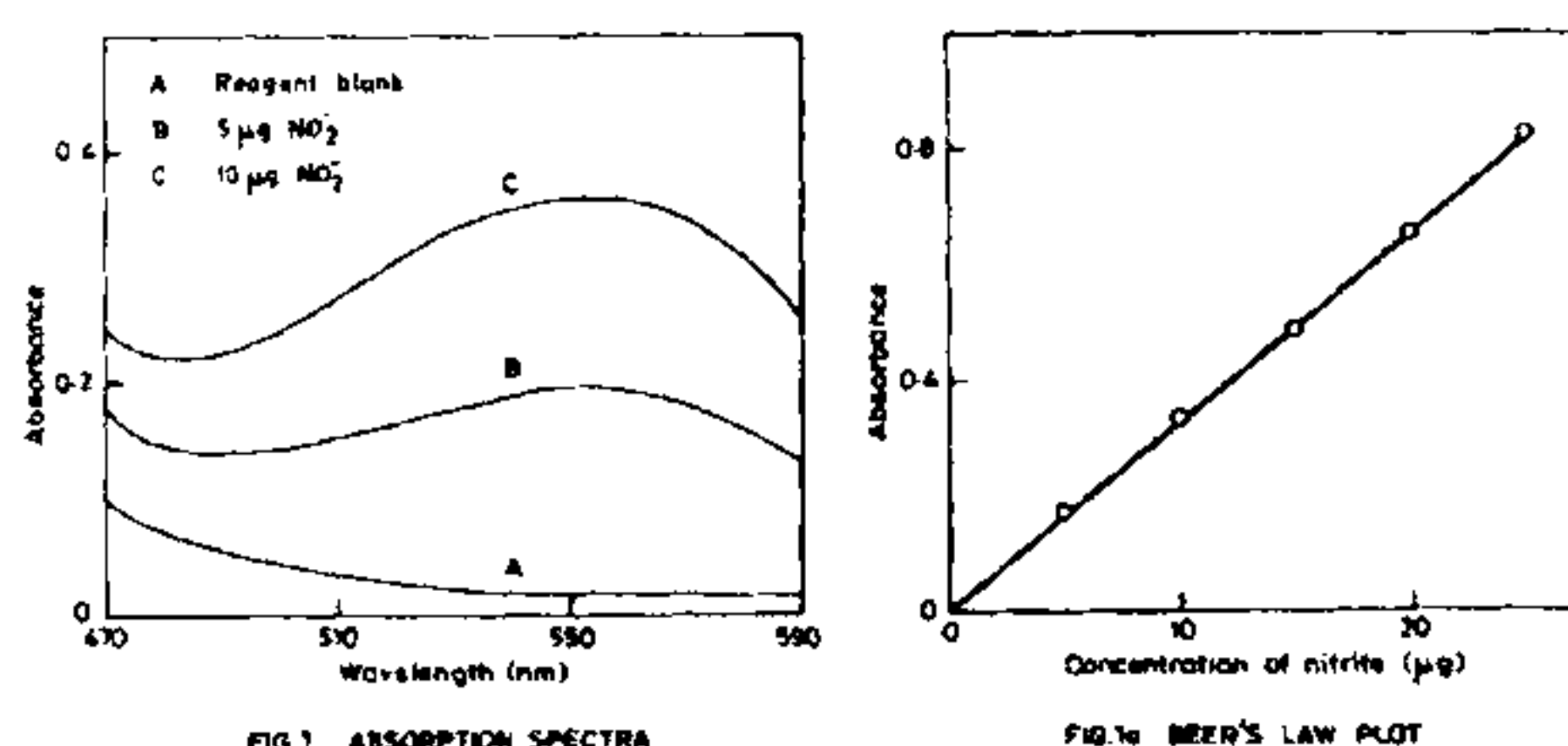


Figure 1. Absorption spectra, 1a. Beer's law plot.

indicated that a minimum of 1 ml of 0.01 % amine is required for the maximum colour development and higher concentration of amine had no effect on the colour system. Hence 5 ml of 0.01 % amine in 1 N hydrochloric acid is recommended. A similar study of variation of coupling agent concentration indicated that 5 ml of 0.10 % solution of resorcinol in 2 N alkali provided the maximum colour development. The absorption spectrum of the colour system for different concentrations of nitrite (figure 1) showed that absorption maximum is at 555 nm. The colour system obeyed Beer's law over the concentration range 0–25 μg of nitrite.

Interference studies

The interfering effect of several cations and anions in the determination of nitrite is shown in table 1. A deviation of more than ± 0.02 from the absorbance of the solution without any interfering ion was taken as a sign of interference.

The interference of Ca^{2+} , Mg^{2+} , Al^{3+} , Cd^{2+} , Zn^{2+} , Cu^{2+} was no longer observed in the presence of 2 ml of 0.05 M EDTA and 1 ml of 5 % triethanolamine solution. This mixture of solutions is recommended as a general masking agent to overcome the interference of most of the cations. There is no interference upto 1000 μg of $[\text{SO}_3]^{-2}$ and 400 μg of HCHO. However sulphide even at 10 μg level interfered. The interference of sulphide upto 100 μg can be overcome by the addition of 1 ml of 0.1 % HgCl_2 solution to the amine

reagent before the addition of nitrite and centrifuging the precipitated HgS before the absorbance measurement.

Ferric ion interfered by precipitating as $\text{Fe}(\text{OH})_3$ and the interference could not be overcome by the use of masking agents like EDTA, TEA, tartrate and citrate. However, this can be overcome by removing Fe^{3+} as the hydroxide and then subjecting the solution to colour development. The method suffers from the interference of Fe^{2+} and Mn^{2+} .

The molar absorptivity at 555 nm is 3.8×10^4 litre $\text{mole}^{-1} \text{cm}^{-1}$. The relative standard deviation for 10 μg of nitrite is $\pm 1\%$ for 10 determinations.

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CHEIROPOLYSHEMA FARMOSANA: AN ADDITION TO INDIAN FUNGI

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DURING one of the collection trips to the Western Ghats of India an interesting Hyphomycete was collected on dead fallen leaves of *Cinnamomum zeylanicum* which was identified as *Cheiropolyschema farmosana* Matsushima, hitherto not recorded from India. The genus *Cheiropolyschema* was first established by Matsushima¹ with a single species, *C. farmosana* collected on dead leaves of *Paulownia kawakamii* from Taiwan. No further collections of this has been reported so far. The present collection of this monotypic fungus differs from the type in the absence of chains of conidiogenous cells, slightly bigger conidiogenous cells and conidia which are smooth walled. The fungus is briefly described and illustrated below.

The fungus forms effuse colonies on the leaf surface with immersed mycelium. The conidiophores are micronematous, subhyaline to pale brown with a single terminal conidiogenous cell resembling a swollen vesicle and 6–8 μ in diameter. The conidia are en-

Table 1 Interference studies (NO_2^-) = 10 μg

Ions	Concentration (μg)	Remarks
Mg^{2+} , Ca^{2+}	2000	Interfered by a fall in the absorbance
Cd^{2+} , Hg^{2+} , Zn^{2+} Cu^{2+} , Al^{3+}	1000	Interfered by a fall in the absorbance
$\text{S}^{=}$	10	Interfered by a fall in the absorbance
CO_3^{2-} , PO_4^{3-} , NO_3^- SO_4^{2-} , Pb^{2+}	3000	No interference
SO_3^{2-}	1000	No interference
HCHO	400	No interference
Fe^{3+}	500	Interfered by a rise in the absorbance
Fe^{2+} , Mn^{2+}	100	Interfered by a rise in the absorbance