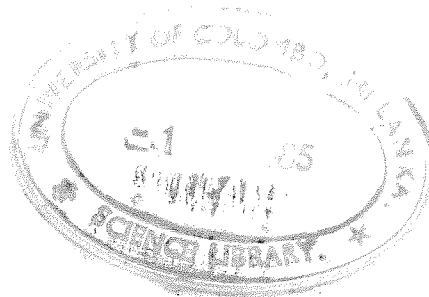


REINVESTIGATION OF THE EFFECT OF TETRASODIUM SALT OF
ETHYLENEDIAMINETETRA-ACETIC ACID IN THE DETERMINATION
OF LOW LEVELS OF NITRATE BY COPPER-CADMIUM
REDUCTION METHOD.



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SRI LANKA

BY

DON RUFUS ANTHONY RANATUNGA

CENTRE FOR ANALYTICAL RESEARCH AND DEVELOPMENT

DEPARTMENT OF CHEMISTRY

UNIVERSITY OF COLOMBO

SRI LANKA

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ABSTRACT

Many analytical methods for determination of nitrate have been reported. However several problems arise in the determinations due to numerous interfering substances.

Low levels of nitrate in natural waters are generally determined by copper-cadmium reduction to nitrite and the subsequent spectrophotometric determination of nitrite by a diazotization method. A determinate error was observed specially when nitrite standards are employed for calibration. It has been confirmed that the low recovery is caused by the tetrasodium salt of ethylenediaminetetraacetic acid (EDTA^{4-}) employed in the reduction process.

In order to determine the reasons for this interference and also to postulate a mechanism, an investigative programme was undertaken with sulphanilamide and β -naphthol. The effect of variables such as pH, concentration of EDTA^{4-} , concentration of nitrite was investigated to establish conditions for subsequent studies. The effect of concentration of EDTA^{4-} on absorbance was noticeably exponential.

Photometric titrations with standard magnesium(II) solution showed that nitrite has no effect on the consumption of EDTA^{4-} , whereas diazonium salt produced by nitrite was found to react with EDTA^{4-} . The study of consumption of EDTA^{4-} in the presence of cadmium(II) ions revealed that neither the cadmium(II) ions nor the $\text{Cd}(\text{EDTA})^{2-}$ complex had a catalytic effect leading to any reaction between nitrite and EDTA^{4-} .

A high rate of consumption of EDTA^{4-} was observed in the presence of excess of free (uncomplexed) EDTA^{4-} . The reduction of the amount of free EDTA^{4-} present in the reaction medium due to complexation of Cd(II) ions mitigates the reaction between diazonium salt and EDTA^{4-} . The Cd(EDTA)^{2-} complex was found to give no interfering effect in the development of colour.

Studies carried out with the system of SAA and NEDD with magnesium(II) ion solution as the titrant for the estimation of consumption of EDTA^{4-} showed that the detection of end point was very difficult. Difficulties arose in photometric titrations due to precipitation. To overcome them standard zinc(II) ion solution was selected as the titrant for further studies. The precipitation observed specially at high concentrations of nitrite was overcome by the introduction of an organic solvent, (i.e. ethanol. Detailed studies with this system at varying temperatures further confirmed the reaction between diazonium salt and EDTA^{4-} .

Thermodynamic feasibility of the reduction process by passage through the column is thoroughly explained using standard reduction potential values.

Studies on column effluents showed that the consumption of EDTA^{4-} was drastically higher than that with similar quantities of nitrite. The high consumption of EDTA^{4-} is due to cadmium(II) ions produced in the column due to the reduction of dissolved oxygen.

Though the reduction of dissolved oxygen assists in the production of cadmium(II) ions, it is obvious that the complete elimination of error observed can be achieved only by adding a sufficient quantity of cadmium(II) ions to complex all free forms of EDTA.

A method has been suggested for further studies of the effect of dissolved oxygen on the production of cadmium(II) ions.