

- Scand.*, 1966, 20, 597.
6. Nakamoto, K., *Infrared spectra of inorganic and coordination compounds.*, Wiley Interscience, New York, 1970.
 7. Piovesana, O. and Furlani, C., *J. Inorg. Nucl. Chem.*, 1970, 32, 879.
 8. Biradar, N. S. and Angadi, S. D., *J. Inorg. Nucl. Chem.*, 1976, 38, 1405.
 9. Moharana, S. N. and Dash, K. C., *Indian. J. Chem.*, 1970, 8, 1023.
 10. Jha, N. K., Prasad R. S. and Kumari, A., *J. Inorg. Nucl. Chem.*, 1981, 43, 3404.
 11. Drago, R. S., *Physical methods in inorganic chemistry*, Reinhold, New York, 1965.
 12. Lever, A. B. P., *Inorganic electronic spectroscopy*, Elsevier, Amsterdam, 1968.

ESTIMATION OF PALLADIUM(II) USING 4-AMINO-3-MERCAPTO-1, 2, 4-TRIAZINE -(4H)-5-ONE AS REPLACING REAGENT

B. NARAYANA and M. R. GAJENDRAGAD
 Department of Chemistry, Mangalore University,
 Mangalagangothri 574 199, India.

A simple, rapid, accurate and selective complexometric method is proposed for the determination of palladium(II). Palladium(II) with associated diverse metal ions can be first complexed with EDTA and the Pd-EDTA complex can be selectively decomposed using demasking agents¹⁻⁶. In some of these methods the liberation of EDTA is not rapid because it requires heating. Some other methods need extraction or involve precipitation during titration, the end point consequently being not distinct. In this paper, the use and advantages of 4-amino-3-mercapto-1,2,4-triazine -(4H)-5-one (AMT) as a selective demasking agent is reported for the complexometric determination of Pd(II).

The speciality of the reagent is that it does not form any precipitate with either Pd(II) the metal ion to be estimated, or Pb(II) the titrant for EDTA under the experimental conditions. This facilitates sharp detection of the end point without necessitating the use of other chemicals.

All chemicals used are of AR or chemically pure grade.

To an aliquot of the neutral solution (containing 0.5–5 mg) of Pd(II), an excess of EDTA solution is added. The pH of the solution is adjusted to between 5 and 6 by adding acetic acid–sodium acetate buffer. The excess of EDTA is back-titrated

against the standard lead nitrate solution to the sharp colour change of the xylenol orange indicator from yellow to red. To this is added 0.5% AMT solution in acetone-water mixture in slight excess over the molar ratio of 1:2. The contents are mixed well. The liberated EDTA is titrated with the lead nitrate solution to the same end point as before. The second titre value is equivalent to Pd(II) present in the aliquot. Reproducible and accurate results are obtained with a relative error of < 0.4% and standard deviation of < 0.01. Interference of many common metal ions and rare-earth metal ions is also studied.

The results of determination of palladium(II) from its solution show that reproducible and accurate values can be obtained. The effect of common metal ions on the quantitative determination of Pd(II) was studied with aliquots containing 2.753 mg of Pd(II). Cations like Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II), Fe(III), Bi(III), Al(III), La(III), Rh(III), Ru(III), Au(III), Cr(III), Zr(IV) and Ti(IV) show no interference. However cations like Ag(I), Fe(II), Sn(II), Hg(II) and Tl(III) show interference which can be overcome by using appropriate secondary masking agents.

The quantitative displacement of EDTA from Pd-EDTA complex by AMT indicates that the Pd-AMT complex is more stable than the Pd-EDTA complex at room temperature and that EDTA is released spontaneously. However for the complete release of EDTA, the AMT reagent is required to be added slightly in excess of the stoichiometric ratio of 1:2 for the metal:reagent. A large excess of the reagent does not show any adverse effects on the results of the estimation.

One of the authors (BN) thanks Mangalore University, Mangalore for a fellowship.

21 May 1987; Revised 6 October 1987

1. Raoot, K. N. and Raoot, S., *Indian J. Chem.*, 1974, 12, 1007.
2. Tikhonov, V. N. and Aleksandrova, E. A., *Zavod Lab.*, 1979, 43, 922.
3. Raoot, K. N. and Raoot, S., *Indian J. Chem.*, 1979, A18, 90.
4. Raoot, S. and Raoot, K. N., *Indian J. Technol.*, 1980, 18, 345.
5. Gadiyar, H. R. A., Gadag, R. V., Gajendragad, M. R. and Sudhakar Nayak, H. V., *J. Indian Chem. Soc.*, 1983, 50, 889.
6. Kumari, V. C., Raoot, K. N. and Raoot, S., *Analyst*, 1983, 108, 1148.