

NOTE

Hydroxytriazenes as Metallochromic Indicators for Complexometric Determination of Iron in Pharmaceutical Preparations

C.P. JAIN, R.S. CHAUHAN, A.K. GOSWAMI* and D.N. PUROHIT

*Department of Chemistry
Mohanlal Sukhadia University, Udaipur-313 001, India*

The present paper deals with complexometric determination of Fe(III) using two hydroxytriazines as metallochromic indicators in two medicines Rubraplex (Sarabhai, India) and Fesovit (SKF, India) respectively. The hydroxytriazines used are 3-hydroxy-3-methyl-1-*m*-nitrophenyltriazine and 3-hydroxy-3-ethyl-1-*p*-nitrophenyltriazine.



Hydroxytriazines are compounds containing functional group —N—N=— . Their utility as spectrophotometric reagents as well as metallochromic indicators for complexometric determination of transition metals is evidenced by several reviews appearing on them during the last few years¹⁻⁵. Literature survey reveals that there are hardly any methods reported for the complexometric determination of Fe(III) in the iron containing medicines particularly using hydroxytriazines as indicators. In view of this some methods have been developed in our laboratories to determine Fe(III) in iron containing pharmaceutical samples complexometrically using hydroxytriazines⁶ as metallochromic indicators.

Synthesis of hydroxytriazenes: Both the hydroxytriazenes were synthesised using standard methods reported in literature⁶⁻¹⁰. The method involved coupling of aryl diazonium salt with aryl hydroxylamine at 0-5°C. The compounds thus prepared were crystallised and their compositions were verified by elemental analysis and melting point determinations.

The following general procedure was adopted to determine Fe(III) in Rubraplex and Fesovit:

- To convert Fe(II) into Fe(III) in the tonic to a titrable form by digestion of the pharmaceutical preparation.
- Complexometric determination of Fe(III) in the digested sample using 3-hydroxy-3-methyl-1-*m*-nitrophenyltriazine and 3-hydroxy-3-ethyl-1-*p*-nitrophenyltriazine as indicators.

In order to digest the capsule or tonic 5 mg/5 mL of it were taken and treated with a few drops of concentrated HNO₃ and heated till it was reduced to minimum quantity. Finally the residue was made up to 25 mL with distilled water, thus getting Fe(III) in solution.

Out of this 25 mL solution a 5 mL aliquot was drawn. Its pH was adjusted to the desired value using this buffer. A few drops of 0.1% solution of indicator 3-hydroxy-3-methyl-1-*m*-nitrophenyltriazine (HMNPT) were added and the solu-

tion was titrated with EDTA. The same procedure was repeated using the indicator 3-hydroxy-3-ethyl-1-*p*-nitrophenyltriazine (HENPT). Three replicates were performed in each case.

The results of the Fe(II) estimation in both the samples have been presented in Table-1. The results show that the values of Fe(III) contents, both theoretical and experimentally obtained, agree well. Thus the present studies establish two new metallochromic indicators for Fe(III) determination complexometrically in pharmaceutical preparations.

TABLE-1
RESULTS OF COMPLEXOMETRIC DETERMINATION OF IRON IN PHARMACEUTICAL PREPARATIONS USING HYDROXYTRIAZINES AS INDICATOR

| | Indicator | Vol of aliquots used for titration | pH | Vol of EDTA (1.0×10^{-2} M) consumed (mL) | Colour changed at the end point | Fe(III) contents in 5.0 mL of solution | |
|-----------|-----------|------------------------------------|-----|---|---------------------------------|--|--------|
| | | | | | | Labelled | Found |
| Rubraplex | HMNPT | 5.0 mL | 2.5 | (i) 8.6 | Dark green to brownish green | 4.9860 | 4.8160 |
| | | | | (ii) 8.6 | | | |
| | | | | (iii) 8.6 | | | |
| | HENPT | 5.0 mL | 7.0 | (i) 8.8 | Dark red to golden yellow | 4.9860 | 4.9840 |
| | | | | (ii) 8.9 | | | |
| | | | | (iii) 8.5 | | | |
| Fesovit | HMNPT | 5.0 mL | 2.5 | (i) 0.3 | Greyish green to golden yellow | 0.1711 | 0.1800 |
| | | | | (ii) 0.5 | | | |
| | | | | (iii) 0.5 | | | |
| | HENPT | 5.0 mL | 7.5 | (i) 0.4 | Brownish to golden yellow | 0.1711 | 0.1680 |
| | | | | (ii) 0.3 | | | |
| | | | | (iii) 0.3 | | | |

REFERENCES

1. D.N. Purohit, *Talanta*, **14**, 353 (1967).
2. D. Chakrovarty and A.K. Majumdar, *J. Indian Chem. Soc.*, **54**, 258 (1977).
3. R.L. Dutta and R. Sharma, *J. Sci. Ind. Res.*, **40**, 715 (1981).
4. D.N. Purohit, M.P. Tyagi, Rita Bhatnagar and I.R. Bishnoi, *Revs. Anal. Chem. (Israel)*, **11**, 269 (1992).
5. D.N. Purohit, A.K. Goswami, R.S. Chauhan and Deepika K. Gorji, *Revs. Anal. Chem. (Israel)*, **17**, 223 (1998).
6. D.N. Purohit, A.K. Goswami, R.S. Chauhan and Ratan C. Sharma, *Asian J. Chem.*, **7**, 207 (1995).
7. E. Bamberger, *Ber*, **29**, 102 (1896); **33**, 350 (1900).
8. E. Bamberger and F. Tshirner, *Ber*, **32**, 1675 (1899).
9. D.N. Purohit, *Nat. Acad. Sci. Letters*, **7**, 279 (1984).
10. N.C. Sogani and S.C. Bhattachariya, *J. Indian Chem. Soc.*, **36**, 563 (1959).