

amino group for developing a colorimetric method for the estimation of ampicillin. Ampicillin reacts with 4-dimethylaminocinnamaldehyde to form an orange coloured Schiff's base with an absorbance maximum at 480 nm. This forms the basis for the determination of ampicillin by the colorimetric method reported in this paper. The method is simple, accurate and has been successfully utilised for the estimation of ampicillin and its dosage forms.

Experimental

Reagent solution.—Dissolve 0.1 gm of 4-dimethylaminocinnamaldehyde (GR) in 100 ml isopropanol. The reagent can be preserved in a refrigerator for one week.

Standard and Sample Preparation.—All the samples of ampicillin and its formulations used in the analysis were diluted with a mixture of acetic acid and isopropanol (1:1:5) to give a concentration of 1 mg/ml.

Assay.—Pipette out 1 ml aliquots each of the standard and sample preparations into separate 25 ml standard flasks and make up to the mark with isopropanol. Transfer one ml of these solutions to 10 ml standard flasks, add 1 ml reagent followed by 1 ml acetic acid. Keep the flasks in the boiling water-bath for 2 minutes, allow to cool and dilute to the mark with isopropanol. Measure the absorbance at 480 nm within 15 minutes against reagent blank.

Results and Discussion

Ampicillin was found to react with 4-dimethylaminocinnamaldehyde to produce a Schiff's base. This aldehyde was preferred as a chromogenic agent over vanillin and 4-dimethylaminobenzaldehyde, as deeper colour was obtained. After several experiments isopropanol and glacial acetic acid were found to be the most suitable solvent and acid respectively for optimum colour development. To arrive at an absorbance with less photometric error the reagent requirement was fixed as 1 ml of 0.1% reagent for the range 0.5–12.5 μ g of ampicillin per ml.

The colour developed was stable for 15 minutes. The orange coloured chromogen had an absorbance maximum at 480 nm. The colour obeys Beer's law in the concentration range 0.5–12.5 μ g per ml of ampicillin. A standard was prepared simultaneously with each experiment to minimise the error.

Several samples of ampicillin, its tablet, capsule, injection and syrup were analysed by the present method and the official methods. The values obtained by the proposed method differ from those of the official methods by a maximum of $\pm 0.6\%$, whereas the results obtained by earlier colorimetric method³ for ampicillin differed by $\pm 2.0\%$.

The proposed method is accurate and is suitable for a quick analytical control of ampicillin and its dosage forms.

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1. Klaus Florey, *Analytical Profiles of Drug Substances*, Academic Press, New York and London, 1973, 2, 1.
2. Wilson, C. O., Gisvold, O. and Doerge, R. F., *Text Book of Organic Medicinal and Pharmaceutical Chemistry*, Pitman Medical Publishing Co. Ltd., London, Fifth Edition, 1966, p. 331.
3. Chowdhury, C., *Indian Journ. of Pharm.*, 1976, 38, 124.

MERCURIC NITRATE AS A SIMPLE AND NEW CHROMOGENIC SPRAY REAGENT FOR THE DETECTION OF OPIUM AND ITS ALKALOIDS

VARIOUS chromogenic spray reagents for the detection of opium and its alkaloids on thin layer plates have been described in literature¹⁻⁴, the most widely used being the iodoplatinate reagent and Dragendorff's reagent. The iodoplatinate reagent though effective and widely used involves the use of costly platonic chloride. The Dragendorff reagent does not give clear differentiation of the spot from background when the alkaloid is present in low quantities. During the present investigations, mercuric nitrate has proved to be a sensitive chromogenic spray reagent for the detection of opium alkaloids on t.l.c. plates.

Glass plates of size 20 \times 20 cm were coated with silica Gel G (E/M) to a thickness of 250 μ m and the plates after air drying, were activated in an air oven at 110° for one hour. Then plates were cooled in a desiccator. Methanolic solution of morphine and chloroform solution of codeine, narcotine, papaverine and thebaine were spotted on the plate, and the plate developed in a solvent mixture of benzene: methanol (8:2). After the solvent front had moved to a distance of 10-12 cm, the plate was taken out and dried and sprayed with 4% solution of mercuric nitrate in 3% dil HNO₃ and the plates were heated at 110° for 15 minutes.

Results and Discussion

Reddish-brown to yellow spots appeared after heating the plates. Morphine, thebaine, papaverine and narcotine could be detected even at 2 μ g level but the minimum detection limit for codeine is 10 μ g. The colours of the spots are given in Table I. Hg²⁺ is a well-known electron acceptor⁵. The alkaloids having a lone pair of electrons on the nitrogen atom function as electron

TABLE I

Detection of some opium alkaloids on TLC plates with aqueous mercuric nitrate spray

Alkaloid	Colour	Minimum detectable limit (μg)
Morphine	Reddish brown	2
Codeine	Yellow	10
Thebaine	Yellowish brown	2
Papaverine	Yellow	2
Narcotine	Yellow	2

donors and the formation of charge transfer complexes may be the very likely mechanism of colour formation. The reagent was applied for visualizing the alkaloids of opium in crude extracts as well as many other samples of forensic interest.

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1. Santavy, F., Stahl, E. (Editor), *Thin Layer Chromatography*, A Laboratory Hand-book, Springer, New York, 1969.
2. Rama Rao, N. V. and Murthy, H. R. K., *J. Indian Acad. For Sci.*, 1974, 13, p. 18.
3. Guven, K. C. and Aran, B., *Eczacilik Bull.*, 1973, 15, 28.
4. Rama Rao, N. V. and Tandon, S. N., *Forensic Science*, 1977 (in Press).
5. Mulliken, R. S. and Person, W. B., in "Molecular Complexes—A lecture and Reprint Volume", Wiley Interscience, 1969, p. 276.

PALLADIUM(II) CHELATES OF SOME o-NITROSOPHENOLS

THE literature survey shows that, not much work has been done on the metal complexes of ambidentate ligands containing -OH and -NO groups¹⁻³. Here we report the preparation and spectral study of Pd-complexes of the type Pd(qo)₂, where qOH represents one of the following:

4-Chloro-, 4-bromo-, 4-methyl-, 5-methoxy-2-nitrosophenols, 3-nitroso-4-hydroxy-coumarin, 2-nitroso-1-naphthol, 1-nitroso-2-naphthol, 3-chloro-, 3-bromo-1-nitroso-2-naphthols.

The complexes of 4-substituted phenols were prepared using the method described by Charalambous *et al.*¹. Other complexes were prepared in aqueous medium by reacting the metal salt with respective ligands. The pH was adjusted between 2.0 to 4.5. Excess ligand was removed by Soxhlet extraction with petroleum ether. The complexes were dried under vacuum over P₂O₅ and analysed. The elemental analysis shows that the ratio of metal to ligand is 1:2.

All complexes are non-electrolyte in DMSO. Infrared spectra were recorded on Nujol mull. The main features of spectra are as follows:

(i) The band characteristic of intramolecular hydrogen bonded OH which occurs at about 3100 cm⁻¹ in the ligand is not observed in spectra of complexes. The characteristic band attributable to co-ordinated water in the region 3400 cm⁻¹ is not observed in complexes.

(ii) The spectra of complexes show that there is decrease in C=O, C=N and an increase in N-O frequencies; this confirms the formation of complexes. The similarity of spectra of complexes and the ligands where O-H...O bond has been well established enables us to conclude oximic structure in the solid state.

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1. Charalambous, J., Frazer, M. I. and Taylor, F. B., *J. Chem. Soc.*, 1969, 18 A, 2787.
2. Patil, S. V. and Raju, J. R., *Indian J. Chem.*, 1974, 7, 270.
3. Gurrieri, S. and Siracusa, G., *Inorganica Chim. Acta*, 1971, 5, 650.

γ -RADIOLYSIS OF SOME REDOX SYSTEMS

WE had reported recently¹⁻³ results of our study on certain redox reactions, which though thermodynamically possible do not occur with any detectable rate, but go to completion on gamma irradiation at room temperature.

We present here results of our study on γ radiolysis of aqueous solutions of the following redox mixtures: (a) S₂O₃²⁻/NO₂⁻, (b) S₂O₃²⁻/BrO₃⁻, (c) S₂O₃²⁻/IO₃⁻, (d) As(III)/IO₃⁻, (e) Sb(III)/BrO₃⁻ and (f) Sb(III)/IO₃⁻ under conditions of neutral pH [(a) to (d)] and pH = 13 [(e) and (f)]. The γ -irradiations were effected