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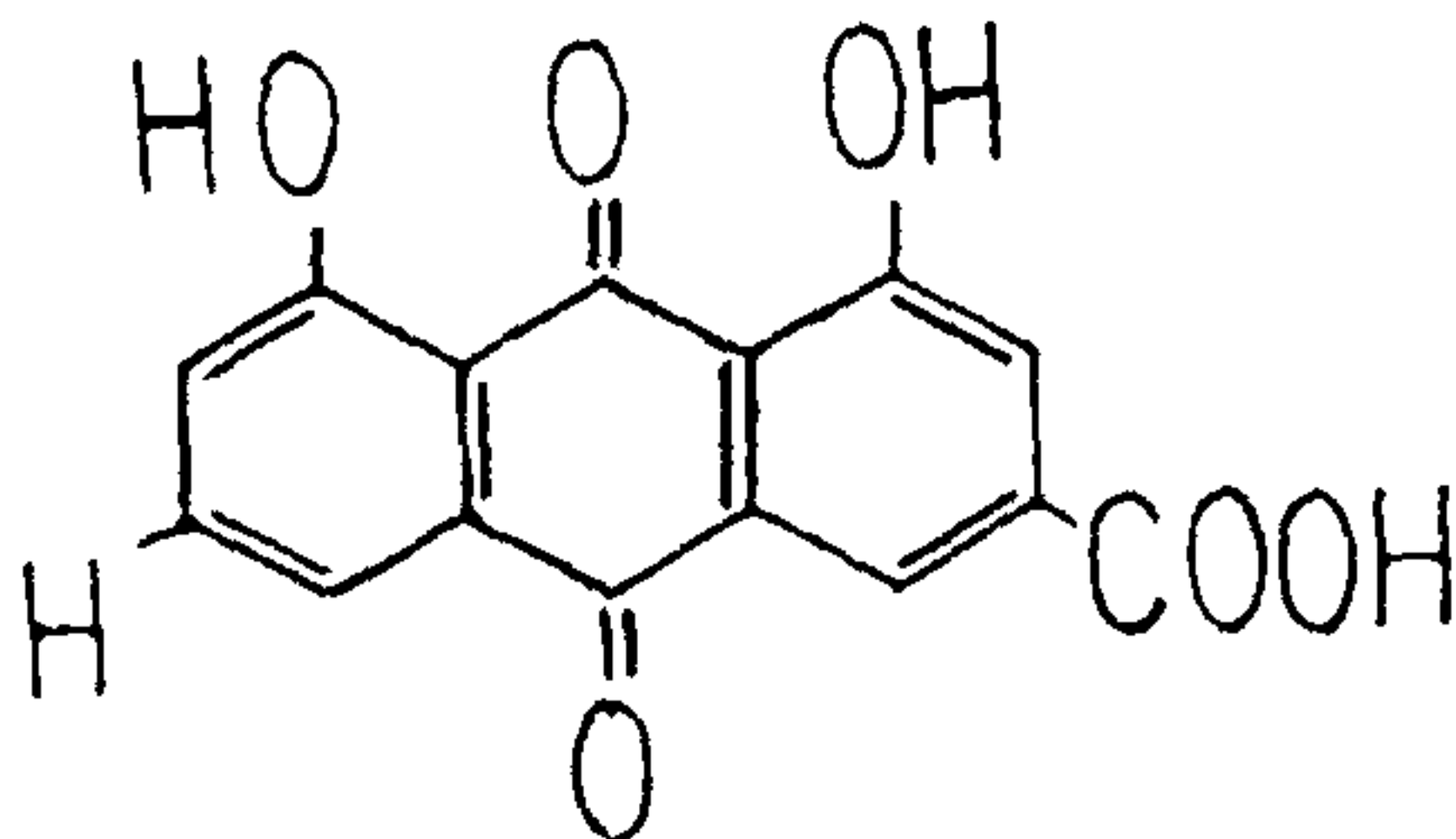
1. Atwal, M. S., Bauer, L., Dixit, S. N., Gearien, J. E. and Morris, R. W., *J. Med. Chem.*, 1965, 8 (5), 566.
2. Barton, N., Crowther, A. F., Hepworth, W., Richardson, D. N. and Driver, G. W., *Brit. Pat.*, 1960, 830, 823; *C.A.*, 1961, 55, 7442.
3. Chothia, D. S., Dike, S. Y., Engineer, A. B. and Merchant, J. R., *Indian J. Chem.*, 1976, 14B (5), 323.

RHEIN FROM THE LEAVES OF *CASSIA NODOSA*

Cassia nodosa (Fam. Ceasalpinaceae) is a tree of upto 30-40 feet high. Leaves are mentioned to function as purgatives¹ but yet there is no report of isolation of any active principle from leaves which may be responsible for its purgative action. This note describes the isolation and identification of the active principle rhein, which is found in free as well as in the combined state in leaves.

Experimental

Isolation of (I) Free rhein :—About 1 g of the dried powdered leaves was completely extracted with chloroform, (II) Combined rhein :—The marc after extraction of free rhein was heated with dilute HCl + FeCl₃ (20%) for 30 minutes on water-bath. Solution was cooled and the liberated aglycone (rhein) was extracted with chloroform.



RHEIN

Chloroform extracts from (I) and (II) were concentrated under reduced pressure and used for TLC. studies with silica gel G using solvent system benzene, chloroform, glacial acetic acid (90 : 10 : 0.5). Extracts (I) and (II) both produced single spots (R_f 0.15)

which were identical to the R_f of authentic sample of rhein developed on the same chromatogram.

15 mg of pure rhein was obtained by preparative TLC² (orange needle crystals), M.P.—321, I.R. spectra (cm^{-1}) in Nujol—1630; 1670; 1705; 1570 u.v. spectra max (in methanol)—(nm) 228; 260; 432.

Physical properties and the chromatographic studies confirmed the presence of rhein, the compound which is responsible for purgative action of the leaves.

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1. Irvine, F. R., *Woody Plants of Ghana*, Oxford University Press, 1961, p. 285.
2. Rai, P. P., Turner, T. D. and Greensmith, S. L., *J. Pharm. Pharmacol.*, 1974, 26, 722.

2-(2'-LEPIDYL AZO)-1-NAPHTHOL-4-AMMONIUM SULPHONATE (LANAS) AS VISUAL METALLO-CHROMIC INDICATOR IN COMPLEXOMETRIC DETERMINATION OF LEAD(II)

HETEROCYCLIC azo dyes introduced recently¹ find extensive use in complexometric²⁻⁴ and spectrophotometric⁵ determination of micro amounts of metal ions in solution. Only very few water soluble heterocyclic azo dyes have been used so far. We have synthesised 2-(2'-lepidyl azo)-1-naphthol-4-ammonium sulphonate (LANAS) and have used for micro determination of mercury(II) complexometrically⁶. In this present communication LANAS has been employed as metallo-chromic indicator for complexometric determination of lead(II) alone or in the presence of large number of other bivalent metal ions.

LANAS gives deep blue colour complex with lead(II) which is readily discharged on adding dilute solutions of EDTA. The observation has been made the basis for the present investigations. The formation of blue coloured Pb-LANAS complex is quantitative and completely discharged in the pH range 6.0 to 7.5 (hexamine-nitric acid buffer). The titration can be successfully performed in the temperature range 0 to 95°C with two to three drops of 0.01% (w/v) LANAS solution as indicator. The best results are obtained when solutions containing lead(II) 0.207 to 41.440 mg per 20 ml are titrated.

Recommended Procedure

To suitable aliquots containing 0.207 to 41.440 mg of lead(II), add two to three drops of 0.01% (w/v) LANAS solution. Add few drops of dilute nitric acid till yellow colour is obtained. Add 10% hexamine solution dropwise till blue colour is obtained. Raise the value to 20 ml and titrate with EDTA solution at room temperature till a sharp colour change blue to