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DIMETHIRIMOL: QUANTITATIVE ESTIMATION

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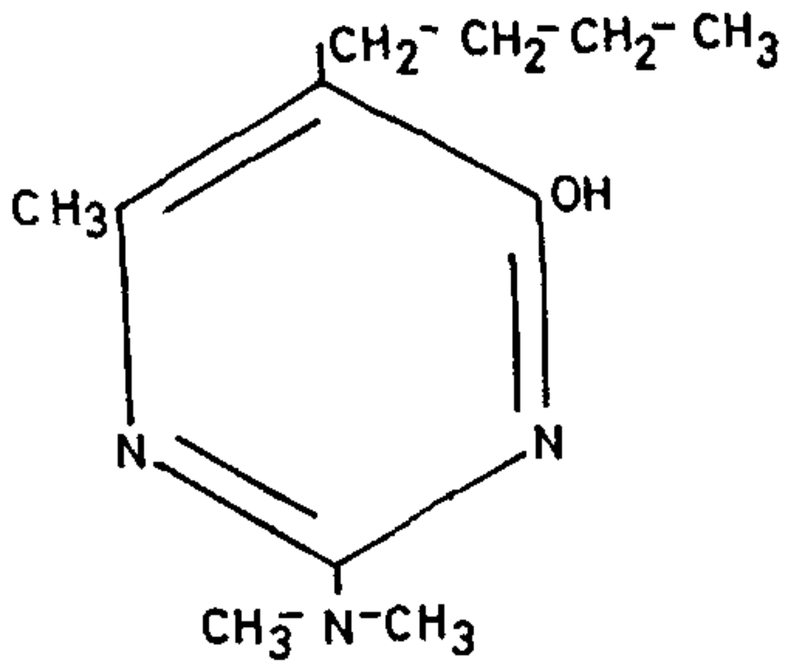
LITER ATURE survey on dimethirimol shows the lack of analytical procedure for the quantitative estimation of this fungicide. As proton resonance spectroscopy is one of the standard methods for the quantitative estimations[1,2], we have employed this technique and the results are discussed. Organic nitrogen is also estimated but it gives a lower percentage.

Dimethirimol, a pyrimidine[2] compound, is 5-*n*-butyl-2-dimethylamine-4 hydroxy-6-methyl pyrimidine. Its dimethylamino group gives a sharp singlet at 3·2 ppm without any overlapping of the other proton signals. Therefore, this peak is utilized for quantitative estimation. As the signal of chloroform (7·2 ppm) does not interfere with the analytical signal of dimethirimol, it is found suitable for use as internal standard.

Quantitative estimation is carried out by integrating two peaks, one at 3·2 ppm (dimethirimol) and the other at 7·2 ppm (chloroform) and the amount of substance found is calculated[2-4].

Twenty estimations were carried out, starting from 0·10 g to 0·02 g and the percentage was found between 98.5 and 99·8.

Dimethirimol of 99·6% purity was obtained from National Physical Laboratory, Teddington, England and the spectra were recorded on Varian T-60 NMR spectrometer, using spectroscopic grade carbontetrachloride as solvent to which a known amount of chloroform is added as internal standard.



STRUCTURE OF DIMETHIRIMOL

The integration values are the average of five integrations for each set. Organic nitrogen is also determined by standard methods[5] but it taken about 20 hr and gives 97-98% purity.

From experimental data, the per cent variation is found to be between −0·88 and +0·16 which is within the limits of experimental error. Therefore, this method can be used for quantitative estimation with a smaller quantity of compound.

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A NEW FLAVONOID GLYCOSIDE FROM THE LEAVES OF *IPOMOEA FISTULOSA*

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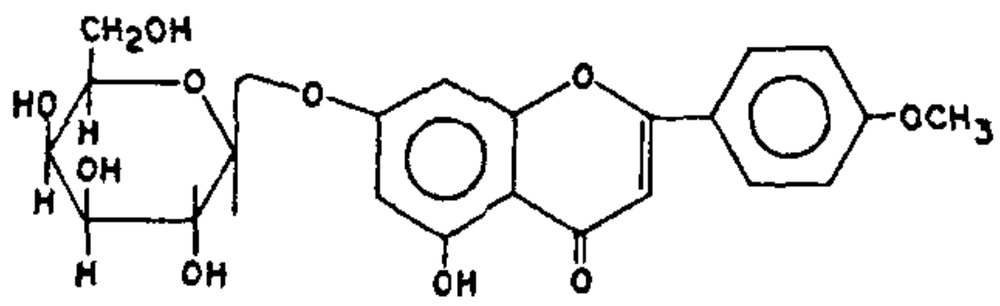
IPOMOEA fistulosa (I. carnaea N. O. Convolvulaceae) is known as Behaya[1] in Hindi and is a wildly growing poisonous straggling shrub. The plant is not only used to protect the crops from cattle[2], but as a hedge, purgative and laxative. An ester and two other compounds[3] have already been reported but the presence of the flavone glycoside and the phytosteroline has not been reported.

From the ethylacetate extract of leaves a new flavone glycoside (A) and a phytosteroline (B) have

been isolated. Homogeneity and purity of both the compounds were established by paper and thin layer chromatography.

The compound A, $C_{22}H_{22}O_{11}$, m.p. 310–12° gave the characteristic colour reactions of flavone and it is glycosidic in nature. On acid hydrolysis (7% ethanolic H_2SO_4), an glycone $C_{16}H_{12}O_5$, m.p. 180° and a sugar D-galactose was obtained. It was confirmed by its derivatives such as phenyl osazone, m.p. 196° (lit. 198°) and p-nitrophenyl D-galactosylamine m.p. 218° (lit. 220°). The aglycone is found to contain one methoxyl group by Ziesel's method[4] (IR at 2830 cm^{-1}, 1180 cm^{-1}). The presence of –OH group at C-7 was confirmed by the bathocromic shift of 25 nm is band II on addition of NaOEt and shift of 47 in band I confirmed the substitution at C-4′. Position of OH group ethanolic at C-5 was confirmed by the bathochromic shift of 50 nm is band I on addition of 1% ethanolic $AlCl_3$. KOH degradation and $KMnO_4$ oxidation gave phloroglucinol and anisic acid respectively which confirmed the positions of OH group and OCH at C5; C7 and C4′.

The sugar moiety is attached to aglycone at position C-7 because glycoside did not give positive test with the reagent vanillin hydrochloride[5]. This position was also confirmed by the UV spectra. On periodate oxidation it consumed 2·2 moles of periodate with the liberation of 1·2 mole of formic acid showing that one mole of sugar is present in pyranose form. It was also supported by a peak at 822 cm^{-1} in the IR spectrum[6].



ACACETIN-7-O-β-D-GALACTOSIDE

On methylation, and subsequent acid hydrolysis of the compound 2, 3, 4, 6 tetra-O-methyl D-galactose was found which showed that sugar moiety is attached through C_1 to C_7 of the aglycone. Emulsion hydrolysis of the glycoside gave galactose showing β-linkage. Thus the compound has been identified as acacetin-7-0-β-D-galactopyranoside.

The compound B, $C_{31}H_{60}O_6$, m.p 271°, $[\alpha]^{26}_D$ + 114° (in pyridine) was obtained as a residue settled in ethylacetate extract of leaves of this plant. The compound was insoluble in most of the organic solvents but highly soluble in pyridine. It gave positive Molisch's test showing it to be a glycoside. On hydrolysis with 10% methanolic HCl, an aglycone, m.p. 137–38°, $[\alpha]_D^{30}$ –34·6° (in $CHCl_3$) and a sugar D-galactose was obtained. The aglycone was identified as β-sitosterol as it gave all characteristic tests[7–10].

Methylation of the compound showed that sugar is attached at C-3 position of the aglycone. The compound was hydrolysed with enzyme taka-diastase showing α-linkage between sugar and aglycone.

The positive optical rotation of the phytosteroline suggested the α-glycosidic linkage. Hence from the above discussion the phytosteroline has been represented as 24-b-ethyl cholest 5-ene-3-O-α-D-galactopyranoside.

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KINETICS OF POLYMERISATION OF METHYL-METHACRYLATE IN PRESENCE OF CRYSTAL VIOLET

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THE retarding effect of 2,6-dichloro-phenol indophenol on polymerisation of methyl-methacrylate(MMA) has been studied[1]. In this paper we report the kinetics of polymerisation of MMA using crystal violet as a retarder.

The monomer has been polymerised under nitrogen atmosphere employing a modified dilatometric apparatus[2]. The monomer was purified by the method given by Overberger[3]. The initiator azobisisobutyronitrile (AIBN) was recrystallised twice from ethanol. The solution of the dye (BDH) was prepared dimethyl-formamide (DMF) at 25°C. The