The more negative values of ΔG at 45° than at 35° or 25° and the positive values of ΔH in all the cases suggest that in the chelates steric strain exists around the rare-earth metal ion due to the presence of fused rings. The positive values of ΔS indicate that the entropy term is favourable for their formation.

Solid-chelates: The rare-earth chelates of H_2BE were prepared in the solid state by the method reported earlier[3] and the yields were found between 75–80%. Their molecular weights were determined ebullio-metrically (Table I) and 1:2 (metal-ligand) stoichio-metry was observed in these compounds.

Lanthanum chelate was found diamagnetic whereas the rest paramagnetic as shown in Table I. From the magnetic moments which are very close to spin-only values, it is apparent that in these compounds there is no metal-metal bonding and, hence, no spin-exchange occurs and they exist as monomers.

The results of I.R. studies are shown in Table II. Based on the data of this investigation, an octahedral stereochemistry of these compounds is suggested.

TABLE II

I.R. spectra of H_2BE and its rare-earth chelates

(in cm^{-1})

Compound	ν (SO_3H)	ν (C=N)	ν (OH)	ν (M–O)	ν (M–N)
H_2BE	1170	1630	3600	..	..
Rare-earth-H_2BE chelates	..	1610	..	650–630	600–580

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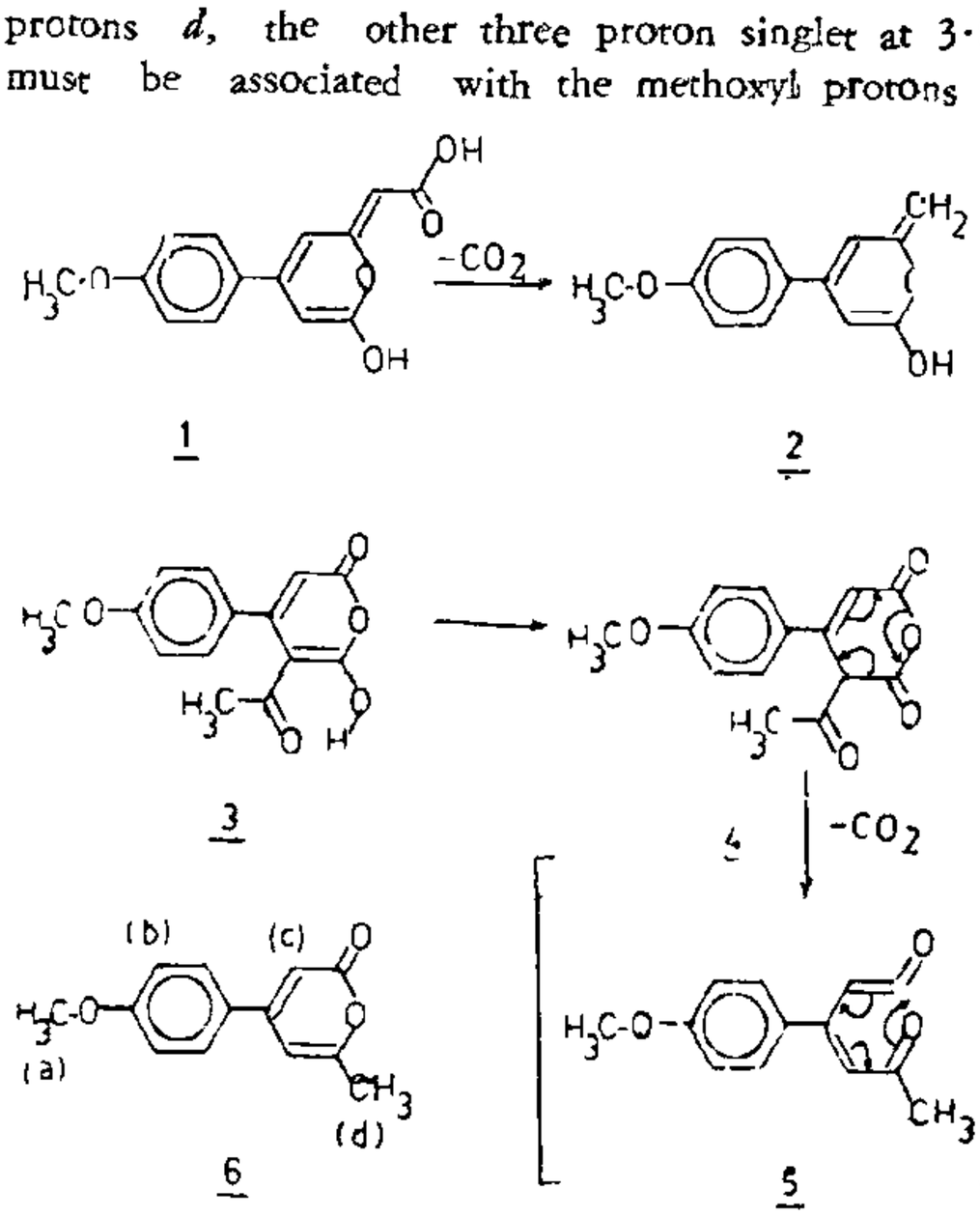
DECARBOXYLATION OF 4-ACETYL-3-ARYL PENT-2-ENE-1, 5-DIOIC ANHYDRIDE. FORMATION OF OXIN-2-ONES

THE condensation product of 3-aryl pent-2-ene-1, 5-dioic acids and fused sodium acetate-acetic anhydride, assigned structure *1* by Bhave and Nerurkar[1], was reported to decarboxylate on heating above its m.p. or on treatment with mineral acid to the lactone *2*.

The structure of the condensation product has been established by us on the basis of spectral studies to be a C-aiylated product *3*[2]. It is now the contention of the author that the decarboxylation observed by Bhave and Nerurkar proceeds by a retero Diels-Alder fragmentation of the anhydride ring of *3* to give the intermediate ketene *5*, which would readily undergo cyclization to form the 4-aryl-6-methyl oxin-2-one *6*. The structure of the lactone, thus became a moot problem. In the ir spectrum the strong band at 1700 cm^{-1} is most certainly due to the C = O stretching vibrations. The 1650, 1600 cm^{-1} bands are associated with C = C stretching vibrations of the diene system. The nmr spectrum exhibited a three proton singlet, at 2·33 which is due to the methyl protons *d*, the other three proton singlet at 3·88 must be associated with the methoxyl protons *a*.



The olefinic proton, *c* resonates at 6·3, and the 'quartet'—two sets of doublets centered at 7·0 and 7·85,—with additional weak lines surrounding the four main signals is evidently that of a *p*-disubstituted benzene. The spectral data, thus clearly substantiates our postulation for the structure and formation of the 4-aryl-6-methyl oxin-2-one.

Experimental

4-acetyl-3-(4'-methoxy phenyl) pent-2-ene-1,5-dioic anhydride 3 m.p. 132–133° C, lit.[1] 132° C was prepared as previously described[1] and repeated crystallization from benzene yielded a pure sample for spectral analyses.

4-(4'-methoxy phenyl)-6-methyl oxin-2-one 6, m.p. 113–114° C, lit[1] 112° C was prepared as previously described and charcoal treatment, followed by crystallization from alcohol, furnished pure sample for spectral analyses.

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ISOLATION AND CHARACTERISATION OF β-SITOSTEROL AND FLAVONOL GLYCOSIDE FROM THE TRUNK BARK OF *FICUS RUMPHII*

F. rumphii (N.O. Moraceae) is reputed for its medicinal importance[1]. In spite of this, practically no work is reported in literature on this plant. The air dried and crushed bark was extracted with 80% methanol. The extract after concentration deposited a yellow solid which on purification gave a white compound A. The supernatant liquid was extracted with ethylacetate. The ethylacetate soluble fraction on purification gave compound B.

Compound A, $C_{29}H_{50}O$, crystallised from ethanol as white plates was found to be β-sitosterol by TLC, mmp, IR, ms, colour reactions and derivatives formation.

Compound B, $C_{22}H_{22}O_9$, crystallised from ethylacetate and petroleum ether as light yellow solid, m.p. 210° (d). On hydrolysis with 7% ethanolic sulphuric acid it gave sugar and aglycone. The sugar was found to be glucose by TLC and osazone formation.

The aglycone gave colour reactions of flavonoids and showed absorption maxima at 268 nm and 376 nm. It contains only one methoxyl group (Ziesel Method). The aglycone was identified as 3'-methoxy flavonol. Thus the glycoside, $C_{22}H_{22}O_9$, will be 3'-methoxy flavone-3-glucoside.

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A THERMOSTABLE GLUCOAMYLASE FROM THE THERMOPHILIC FUNGUS *THERMOMYCES LANUGINOSUS*

HEAT stable enzymes have potential applications in industry. There has been increasing interest in the production of glucoamylase (exo $\alpha 1 \rightarrow 4$ glucan glucohydrolase) since it directly hydrolyses starch to glucose. This enzyme has been purified and characterised from some species of mesophilic fungi[1-3]. Subrahmanyam *et al.* have reported on the production of this enzyme from the thermophilic fungus, *Torula thermophile*[4]. We screened some species of thermophilic fungi[5] for the production of glucoamylase. These were isolated from compost or soil by the method described previously[6]. In this communication we report on the production, purification and some properties of an extracellular glucoamylase produced by the thermophilic fungus, *Thermomyces lanuginosus* ML-M.

The medium used for growing the organisms contained 2% soluble starch (Merck), 0·4% L-asparagine, 0·1% K_2HPO_4, 0·05% $MgSO_4.7H_2O$ and 0·01% (v/v) of a trace element solution[7]. The organisms were grown in 500 ml Erlenmeyer flasks containing 50 ml of the medium in static or in shake cultures at 50° C.

Glucoamylase was assayed by adding 0·1 ml of the culture filtrate to 3 ml of 0·1% soluble starch solution in 100 mM sodium acetate buffer, pH 5·0, and incubating the reaction mixture at 50° C for 30 min. Glucose produced was estimated by the glucose oxidase-peroxidase method[8].

In static cultures, of the four species of thermophilic fungi tested, *T. lanuginosus* produced the maximum glucoamylase of highest specific activity (Table I). In addition, the enzyme elaborated by this fungus was more heat stable than that of the other fungi. Therefore, *T. lanuginosus* was chosen for detailed studies.

The relationship between the growth of the organism, enzyme production, substrate utilisation and product formation was studied in shake cultures (Fig. 1). Growth was rapid in shake cultures and the maximum growth was obtained when the conversion of starch was complete. The appearance of the