

Table II summarises the effect of temperature on the dissociation constant of tribromide ion. The  $\Delta H^\ddagger$  for the reaction  $\text{Br}_3^- \rightleftharpoons \text{Br}_2 + \text{Br}^-$  works out to be  $11.0 (\pm 0.9) \text{ k. cal. mole}^{-1}$ . The values of  $\Delta S^\ddagger$  and  $\Delta G^\ddagger$  were calculated at three temperatures.

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November 17, 1979.

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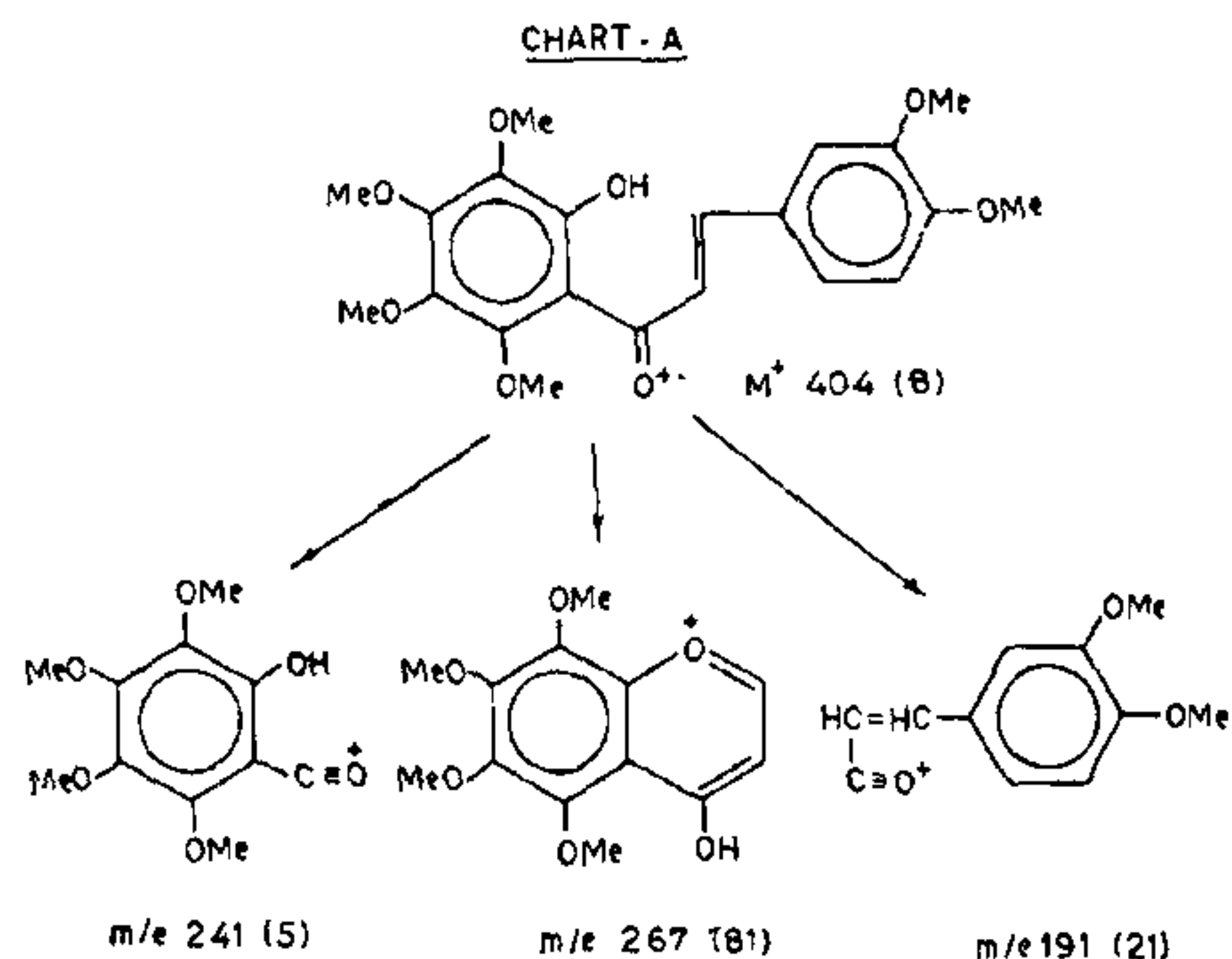
### ISOLATION OF 2'-HYDROXY 3', 4', 5', 6', 3, 4-HEXAMETHOXY CHALCONE FROM THE BARK OF *MACARANGA PELTATA* MUELL.

The isolation of 2'-hydroxy 3', 4', 5', 6', 3, 4-hexamethoxy chalcone for the first time in nature from the bark of *Macaranga peltata* Muell. and its characterisation by  $^1\text{H}$  NMR and mass spectra have been reported.

The methanolic extract of the bark (4.5 kg) of *Macaranga peltata* was concentrated to which a saturated solution of basic lead acetate in methanol was added to give yellow basic lead salt. From the filtrate isolation of berginin and three of its partial methyl ethers have already been reported<sup>1</sup>. The lead salt on decomposition gave a compound which was recrystallised from methanol as bright orange flakes m.p.  $174^\circ$  (55 mg). It analysed for  $\text{C}_{15}\text{H}_6\text{O}_2 (\text{OMe})_6$ : ( $M^+$  404) and gave positive ferric reaction for phenolic group, red colour with antimony trichloride in  $\text{CCl}_4$  characteristic of a chalcone<sup>2</sup>. The chelated carbonyl appeared at  $1620 \text{ cm}^{-1}$  in its I.R. and U.V.  $\lambda$  maxima nm ( $\log \epsilon$ ) at 382 (4.32), 288 (4.09) also supported the chalcone nature of the compound. Estimating for six methoxyls (Zeisel's) it can be regarded as 2'-hydroxy hexamethoxy chalcone.

The  $^1\text{H}$  NMR spectrum of chalcone in  $\text{CDCl}_3$  showed the chelated hydroxy proton at  $\delta$  13.2 as broad singlet and the six methoxyls as singlets at  $\delta$  3.74, 3.85, 3.86, 3.9, 3.96 and 4.06. The two chalcone protons appeared separately at  $\delta$  7.4 and 7.6 as doublets ( $J = 13 \text{ Hz}$ ). The three proton multiplet at  $\delta$  6.91, the pattern and the chemical shifts of which are reminiscent of veratryl nucleus<sup>3</sup>. It may therefore be considered that the chalcone is 2'-hydroxy, 3', 4',

5', 6', 3, 4-hexamethoxy chalcone. Selenium dioxide oxidation of the chalcone gave the flavone,  $\text{C}_{15}\text{H}_4\text{O}_2 (\text{OMe})_6$ , m.p.  $132-34^\circ$  which corresponded to robiletin (lit<sup>4</sup> m.p.  $134^\circ$ ). Mass fragmentation of the chalcone confirmed its structure further (Chart A).



This chalcone has not been reported to occur in nature so far, although this was reported as an isomerisation product of citromitin, the corresponding hexamethoxy flavanone, isolated from *Citrus mitis* previously by Row and Sastry<sup>5</sup>. However, this chalcone was not characterised by PMR and mass spectral studies previously. A direct comparison (m.m.p. and I.R.) of the chalcone and flavone obtained with the authentic samples supplied by Prof. Row proved their identity.

One of the authors (D. S. K.) thanks the C.S.I.R. for a fellowship. Our thanks are also due to Prof. L. R. Row for his encouragement and authentic samples.

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July 7, 1980.

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