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MEAN AMPLITUDES OF VIBRATIONS OF NITRYL FLUORIDE AND NITRYL CHLORIDE

THE authors have earlier subjected nitryl fluoride and nitryl chloride to normal co-ordinate treatment by using the most general quadratic form of potential energy expression.[1] The molecules were assumed to have planar configuration with a symmetry of the point group C_{2v}. The computations have now been extended to calculate the mean amplitudes of vibrations of the valence linkages N–F, N–O and N–Cl. The authors have sketched the theory of the evaluation of mean amplitudes of vibrations of bonds elsewhere.[2] Applying those methods the results obtained in the cases of the first two nitryl halides are given in Tables I and II. The percentage contribution of each of the normal co-ordinates to the mean square amplitudes have also been given.

TABLE I
Nitryl fluoride

Normal co ordinate Q	Frequency cm.$^{-1}$	m.s.a. of N–F bond $\times 10^{-4}$ A^2	% contribution	m.s.a of N–O bond $\times 10^{-4}$ A^2	% contribution
Q_1	1312	0·6431	2	4·6287	34
Q_2	822	23·4414	84	0·3967	3
Q_3	460	3·7976	14	0·0328	..
Q_4	1793	..	..	8·6629	63
Q_5	570	..	..	0·0767	..
Mean square amplitude		27·8821		13·9978	

Mean amplitude of N–F bond = 0·053 Å
Mean amplitude of N–O bond = 0·037 Å

TABLE II
Nitryl chloride

Normal co-ordinate Q	Frequency cm.$^{-1}$	m.s.a. of N–Cl bond $\times 10^{-4}$ A^2	% contribution	m.s.a. of N–O bond $\times 10^{-4}$ A^2	% contribution
Q_1	1293	4·2955	21	3·0655	25
Q_2	794	13·4366	65	0·2257	2
Q_3	411	2·8742	14	0·0306	..
Q_4	1685	..	..	8·7144	72
Q_5	367	..	..	0·0228	..

Department of Physics, P. G. PURANIK.*
Univ. College of Science, E. V. RAO.
Osmania University,
Hyderabad (A.P.), *July* 19, 1965.

* Present Address : Nizam College, Hyderabad.

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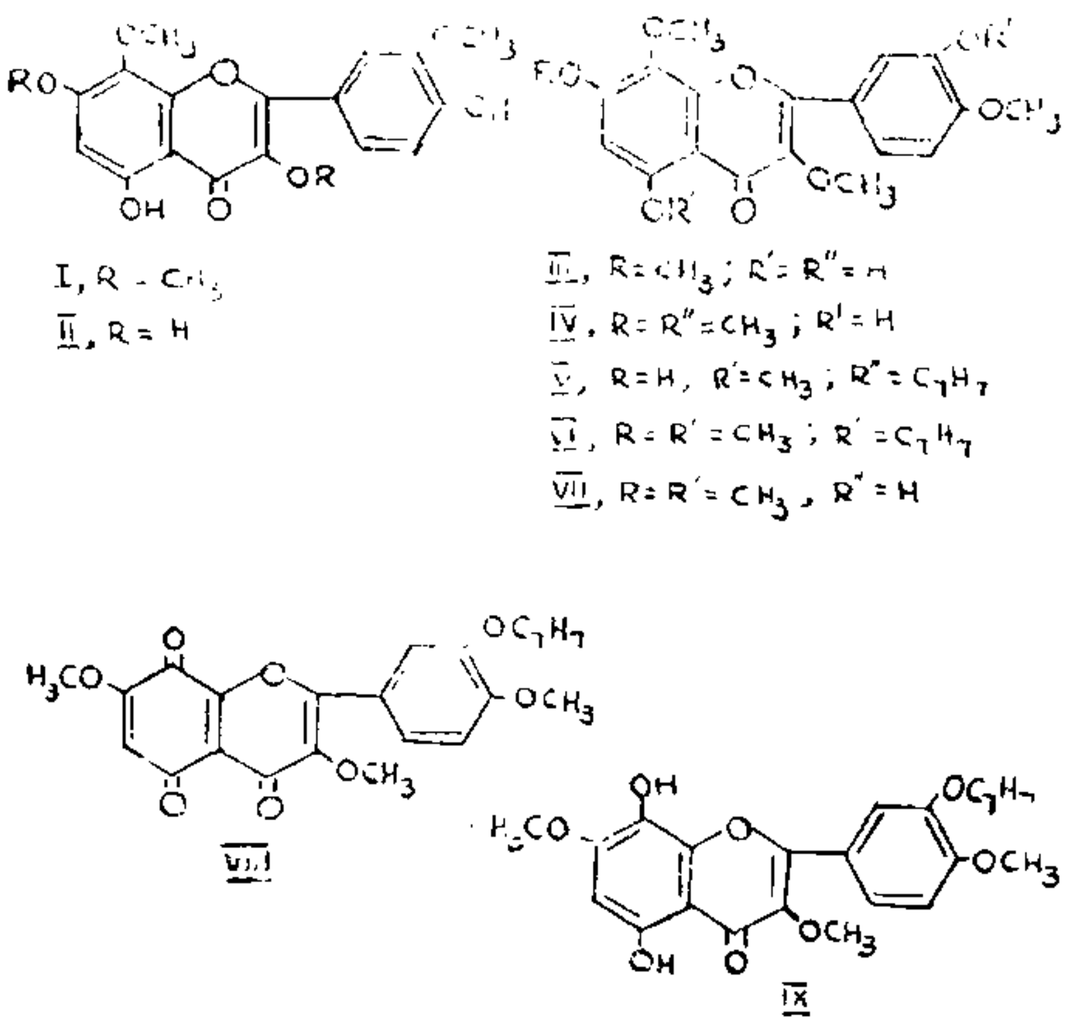
SYNTHESIS OF 3, 7, 8, 4′-O-TETRAMETHYL GOSSYPETIN

A NUMBER of ethers of gossypetin occur in nature besides one which has methylenedioxy group. Four partial methyl ethers are known : ternatin (I),[1] limocitrin (II),[2] gossypetin tetramethyl (III) and pentamethyl (IV) ethers.[3] Of these the tetramethyl ether (III) is a recent discovery and was found to be one of the components of *Ricinocarpus stylosus* by Henrick and Jefferies.[3] The structure was given on the basis of methylation studies and degradation reactions. Further support was provided by U.V. and N.M.R. studies. No synthetic confirmation was, however, provided. As part of a programme of syntheses of the partial methyl ethers of gossypetin we have been able to effect the synthesis of (III) by two methods. The synthetic sample agrees in its m.p., colour reactions and U.V. spectral characteristics with those reported for (III) obtained from natural source and hence provides confirmation for the constitution proposed.

Method I.—The synthesis starts from 2, 4-dihydroxy-ω, 3, 6-trimethoxy acetophenone[4] which was subjected to Allan-Robinson condensation with the anhydride and sodium salt of benzyl isovanillic acid. The flavonol (V) obtained, m.p. 215-17°, was methylated and the resulting methyl ether (VI), m.p. 165–67°, was subjected to catalytic debenzylation. The product (VII), m.p. 214–15°, was partially demethylated using aluminium chloride in acetonitrile to give the required flavonol (III), m.p. 184–85° (reported value[3] 184–85°), mixed m.p. with the natural sample undepressed.

Method II.—In this method compound (VI) was subjected to oxidative demethylation to yield the quinone (VIII), m.p. 205–06°. This was reduced to the quinol (IX), m.p. 184–85°, which on partial methylation at the 8-position followed by catalytic debenzylation gave (III). The acetate had m.p. 176–77° (reported

value[3] 179–80°); mixed melting point with the diacetate of the natural sample was undepressed.



Our thanks are due to Dr. P. R. Jefferies for an authentic sample of 3, 7, 8, 4′-O-tetramethyl gossypetin and its acetate.

Department of Chemistry, University of Delhi, Delhi-7, *June* 14, 1965.

M. KRISHNAMURTI.
T. R. SESHADRI.
P. R. SHANKARAN.

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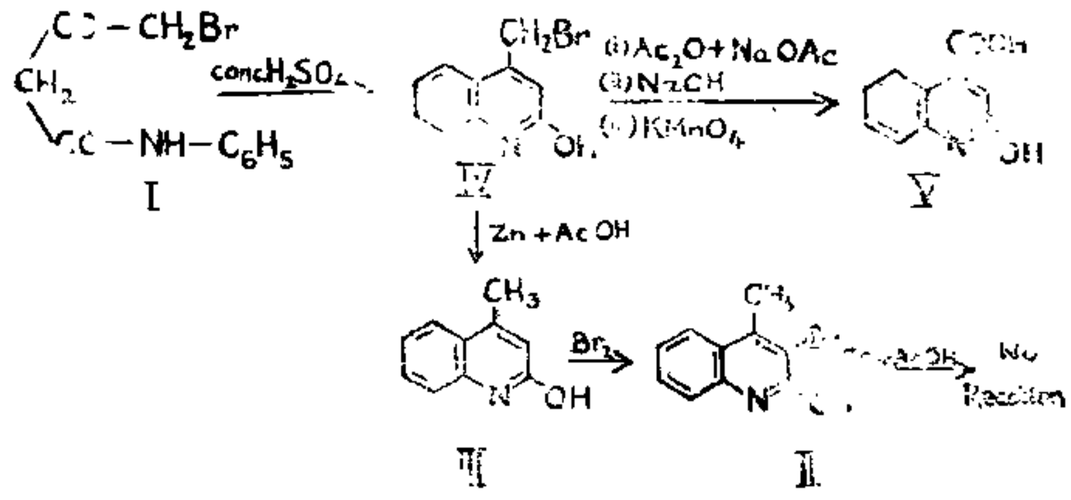
BROMINATION OF ACETOACETANILIDE : A REVISION OF STRUCTURE

KNORR[1] brominated acetoacetanilide in chloroform solution and claimed to have obtained α-bromoacetoacetanilide. Hasegawa[2] and Cook *et al.*[3] repeated the above experiment in chloroform and gave ω-bromoacetoacetanilide (I) structure to this product. Mehta and coworkers[4,5] brominated acetoacetanilide in acetic acid solution and claimed to have obtained α-bromoacetoacetanilide. They further reported that this product on cyclisation gave 3-bromo-4-methyl-2-hydroxyquinoline (II) which was identical with the product obtained on bromination of 4-methyl-2-hydroxyquinoline (III). In view of these contradictory reports, it was thought of interest to repeat the work by both the procedures. In both the cases, the same product (Found : N = 5·84%, Br = 31·69%; $C_{10}H_{10}O_2BrN$ requires: N = 5·46%, Br = 31·25%) with m.p. and mixed m.p. 135–36° was obtained. The product is assigned ω-bromoacetoacetanilide structure on the basis of the following series of reactions. It gave on cyclisation with concentrated sulphuric acid 4-bromomethyl-2-hydroxyquinoline (IV), m.p. 254–56° which is different from the 3-bromo-4-methyl-2-hydroxyquinoline (II) (Found : N = 5·84%, Br = 33·97%; $C_{10}H_8ONBr$ requires: N = 5·88%, Br = 33·61%), m.p. 274° obtained by the bromination of 4-methyl-2-hydroxyquinoline. 4-Bromomethyl-2-hydroxyquinoline (IV) is converted to known 2-hydroxy cinchoninic acid[6] (V) (Found : N = 7·14% ; $C_{10}H_7O_3N$ requires : N = 7·4%) by treatment with acetic anhydride and fused sodium acetate followed by hydrolysis and oxidation with $KMnO_4$. 4-Bromomethyl-2-hydroxyquinoline (IV) on reduction with zinc and acetic acid gave 4-methyl-2-hydroxyquinoline (III) while 3-bromo-4-methyl-2-hydroxyquinoline (II) remained unaffected under similar conditions.



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Chemistry Department, Faculty of Science, M.S. University of Baroda, Baroda-2, *March* 8, 1965.

R. J. CHUDGAR.
K. N. TRIVEDI.

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