

The static and microwave values of dipole moments agree fairly well except for small divergencies in the case of molecules particularly with rather large dipole moments, such as *p*-methyl acetophenone. The higher moments obtained for unsaturated 'acrolein' and 'crotonaldehyde' compared to that of saturated 'propionaldehyde' seem to be due to the effect of conjugation¹ in the former.

The higher relaxation of 'crotonaldehyde' as compared to 'acrolein' may indicate that the heavier methyl substituent probably causes more hindrance to rotation in the former. The relaxation times of the aromatic ketones differ, and this will indicate that the rotation of the whole molecule rather than the group common to them leads to absorption.

The fact that the activation energies for dipole orientation E_r are less than those for viscous flow E_η is in conformity with the established fact that the process of dipole orientation involving only rotation is easier for the molecule than the combined motion of rotation and translation involved in viscous flow.

Dept. of Physics, D. K. DESHPANDE.
Karnatak University, K. SURYANARAYANA RAO.
Dharwar-3, June 19, 1967.

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DETERMINATION OF H₂S PRODUCED BY SULPHATE REDUCING BACTERIA

ON using the chemical formula given in the ASTM method D 993-58 on "Tests for Sulphate Reducing Bacteria in Industrial Water and Water-formed Deposits",¹ it was observed that the sulphuretted hydrogen, calculated by this formula, gave very low values. The opalescence of the culture medium, formation of enough black ferrous sulphide and the strong smell of sulphuretted hydrogen in the culture medium of sulphate reducing bacteria under study, led me to check the formula given in the ASTM method.

It was found that the formula was incorrect and also it appeared to be rather complicated by the use of normality of both iodine and sodium thiosulphate solution.

Before applying the correct formula, my views were communicated to the Editor,

American Society for Testing and Materials, Philadelphia, U.S.A. The ASTM Sub-Committee considered the formula given by me as correct.²

The correct formula for determining sulphuretted hydrogen in ppm is therefore given below :

$$H_2S = (X_1 - X_2) \times P \times 3408$$

where, X_1 = cc of sodium thiosulphate that would react with a known volume of iodine solution, added.

X_2 = cc of sodium thiosulphate solution that would react with the unreacted iodine (i.e., cc of sodium thiosulphate used in back titration).

P = Normality of sodium thiosulphate solution.

It would appear that whoever has made use of the ASTM formula has only reported 10% of the actual amount of H₂S produced in the culture medium. Such values of H₂S will be still valid provided they are multiplied ten times.

Defence Laboratory, J. C. CHAUDHURI.*
Jodhpur, April 15, 1967.

*Present Address: Naval Chemical and Metallurgical Laboratory, Naval Dockyard, Bombay.

1. ASTM, D., 993-58 In *The 1965 Book of ASTM Standards, Part 23*, published by the American Society for Testing and Materials.
2. Communication to J. C. Chaudhuri by Professor James B. Lackey, Professor of Sanitary Science, College of Engineering, University of Florida, U.S.A., letter dated 26 July 1965.

SYNTHESIS OF 2-(2-FURYL) CHROMONES BY SELENIUM DIOXIDE OXIDATION OF FURFURYLIDENE CHALCONES

2-FURYL chromones have hitherto been prepared by three general methods: (a) the Baker-Venkataraman transformation starting from an appropriate o-hydroxy acetophenone by treatment with furoyl chloride¹; (b) the thermal cyclisation between a phenol and an ethyl furoyl acetate² and (c) the conversion of a 2-(2-furyl) chromanone into the chromone by treatment with alkali.³

The conversion of a chromanone or a chalcone to the corresponding chromone by oxidation with selenium dioxide has first been reported by Venkataraman and co-workers⁴ for the preparation of a number of flavone derivatives. Although this method forms one of the most widely-applied and important route to the synthesis of a chromone or flavone ring system, it has not been applied to heterocyclic chalcones with the exception of the chalcone, 1-(5-nitro

TABLE I

Aldehyde	Ketone	Chalcone		2-Fural chromone	
		M.P. (°C.)	Yield (%)	M.P. (°C.)	Yield (%)
Furfural	4-Benzoyloxy resacetophenone	143	60	161	45
"	2-Acetyl resorcinol 6-methyl ether	105	60	168	55
"	4-Chloro-2-hydroxy acetophenone	104	60	205	55
"	4-Acetamino-2-hydroxy acetophenone	175	60	205	55
"	5-Acetamino-2-hydroxy acetophenone	193	60	225	55

pyrrolyl)-3-(o-hydroxyphenyl) propen-1-3-one, obtained from 5-nitro pyrrole 2-aldehyde and o-hydroxy acetophenone, from which the 2-pyrrolyl chromone was isolated by oxidation with selenium dioxide.⁵

The present communication deals with the application of the above synthetic method to the synthesis of a few 2-(2-furyl) chromones. Five 2-furylidene chalcones have been synthesised starting from furfural and 4-benzoyloxy resacetophenone, 2-acetyl resorcinol 6-methyl ether, 4-acetamino-2-hydroxy acetophenone, 5-acetamino-2-hydroxy acetophenone and 4-chloro-2-hydroxy acetophenone. The condensations were carried out in alcohol solution employing one molar proportion of 40-50% sodium hydroxide solution. These chalcones were oxidised employing 1:1 molar proportion of selenium dioxide in isoamyl alcohol medium by refluxing for about 3 hours and isolating the 2-furyl chromone from the residue after steam distillation to remove isoamyl alcohol and any unreacted 2-hydroxy acetophenone. The compounds prepared along with the yields and melting points are included in Table I.

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Dept. of Chem., S. SUBHADRA KUMARI.
Osmania Univ., K. S. R. KRISHNA MOHAN RAO.
June 10, 1967. A. V. SUBBA RAO.
N. V. SUBBA RAO.

TRANSFORMATION PRODUCTS OF CITRAL*

CITRAL is employed for the preparation of the widely used perfumes such as the ionones and methylionones.¹ In a recent communication² we have described the preparation of citral epoxide, an intermediate in the synthesis of (±) linalool.³

Conjugate addition of lithium methyl to citral† in the presence of cuprous iodide⁴ furnished methyl citronellol‡ (I),

n_D^{27} 1.4475, ν_{\max} 2717, 1727, 833 cm^{-1}

$\delta_{\text{TMS}}^{\text{CCl}_4}$ 1.07 (singlet, 6 H; gem dimethyl group), 1.61 and 1.68 (6 H; methyls on the isopropylidene group),

5.06 (1 H; vinyl proton) and 10.05 (triplet, 1 H; aldehyde proton)

which on sodium borohydride reduction in ethanol furnished methyl citronellol‡ (II)

ν_{\max} 3500, 1050 cm^{-1}

as a pleasant smelling liquid.

$\text{Me}_2\text{C}=\text{CH} \cdot (\text{CH}_2)_2 \cdot \text{C}(\text{Me}_2) \cdot \text{R}$

(I) $\text{R} = -\text{CH}_2 \cdot \text{CHO}$

(II) $\text{R} = -\text{CH}_2 \cdot \text{CH}_2\text{OH}$

Reduction of citral† with iron and 50% acetic acid on steam bath in the presence of nickel chloride furnished citronellol and mixture of hydrocarbons⁵ 2, 6-Dimethyl-2, trans-6-octadiene (III) and 2, 6-dimethyl-2, cis-6-octadiene (IV). Citronellol⁶ was identified through its boiling point, refractive index, infra-red spectrum and comparison of its vapour phase chromatography behaviour with that of an authentic sample. The structure assigned to the hydrocarbon mixture (n_D^{23} 1.4450) is consistent with its refractive index and IR spectrum and has been confirmed by its conversion to levulinic acid on ozonolysis. Reduction of citral† with sodium borohydride in ethanol furnished a mixture of geraniol and nerol, free from citronellol. The mixture of geraniol and nerol so obtained was reduced with iron and aqueous acetic acid to a mixture of hydrocarbons III and IV having

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