## A STUDY OF THE CHEMICAL COMPONENTS OF THE ROOTS OF DECALEPIS HAMILTONII

Part V. A Note on the Use of 4-o-Methylresorcylic Aldehyde as a Preservative

BY P. BHASKARA RAMA MURTI AND T. R. SESHADRI

(From the Departments of Chemistry and Chemical Technology, Andhra University, Waltair)

Received July 21, 1942

In the preservation of canned and stored food, from the attack of bacterial and fungal organisms, certain preservatives like sulphur dioxide and benzoic acid were at one time freely employed. Since they have undesirable effects on health, laws have been enacted in various countries restricting their use. Recent trend has been in the direction of utilising natural preservatives which are not toxic to larger animals and which inhibit the growth of microorganisms. Spices have been examined in this connection. Though they are not very good as such, volatile oils obtained from them have been found to be very satisfactory. Essential oil of mustard has proved itself to be a good preservative being even better than sulphur dioxide and benzoic acid. Oils of cinnamon, clove, thyme and bay leaves are also stated to be good.

It was noticed in the course of our work on the roots of *Decalepis Hamiltonii* and of *Hemidesmus indicus*, that the air dried roots when kept for a long time, were uneffected by fungus and insects. Even the fresh roots were remarkably free from micro-organisms in the course of several days. It appeared that this was due to the presence of the sweet smelling volatile compound, 4-o-methylresorcylicaldehyde to the extent of 0.8% in the former and about 0.12% in the latter. It was therefore felt desirable to investigate the properties of this aldehyde as a preservative. The substance has a sweet and agreeable smell and a mild pungent and aromatic taste. It can be easily isolated by steam distillation of the powdered roots. A better yield can be obtained by extracting the material with alcohol and after distilling off the solvent, subjecting the residue to steam distillation. It can also be obtained in large quantities synthetically by the methylation of  $\beta$ -resorcylicaldehyde, using dimethyl sulphate and sodium hydroxide.

Experiments were conducted to test roughly its toxicity to fish and bacteria. Small fresh-water fish known as  $Haplochilus\ panchax$  were employed for the former and B. Coli for the latter. It has been found that at a concentration of 0.021 per cent., a solution of it is toxic to fish and that they die within 4 minutes. The growth of B. Coli was arrested effectively in a

concentration of 0.041 per cent. for more than seven days. Two hydroxyaldehydes isomeric with this, namely, vanillin and isovanillin were also studied with a view to understand the effect of constitutional factors. Vanillin was toxic to fish at a strength of 0.029 per cent.; but they died only after 85 minutes. Iso-vanillin of the same concentration killed them in 2 hours. For bacteriostatic effect 0.231 per cent. solution of vanillin and 0.270 per cent. solution of iso-vanillin were required. Thus it is clear that the methyl ether of resorcylic aldehyde is far more effective than the other two. One disadvantage with it is that it is less soluble. However, in favour of its use as food preservative may be mentioned the fact that the *Decalepis* root is used largely in South India as a pickle and no untoward toxic effects on human beings have been noticed.

For purposes of characterisation of 4-o-methylresorcylical dehyde the bromo-derivatives are useful (for their preparation see Rao et al.3). In the course of our work we have noticed that all the three products of bromination, (1) 5-bromo-aldehyde; (2) 3:5-dibromoaldehyde and (3) 2:4:6-tribromoresorcinol monomethyl ether could be obtained simultaneously by the following method. An ether solution of the aldehyde was treated with excess of bromine (in ether solution) at 0° and allowed to stand for 2 hours. The 5-bromoderivative (m.p. 120-121°) crystallised out by this time almost completely (yield 75%). It was filtered and the ether solution evaporated The residue was dissolved in alcohol and the under reduced pressure. solution allowed to concentrate. 3-5 dibromoderivative separated out in a small amount and could be purified by recrystallisation from aqueous It melted at 98°. The mother liquor on treatment with water, gave a precipitate of impure 2: 4:6-tribromoresorcinol monomethyl ether. Repeated recrystallisation from aqueous alcohol yielded the pure compound melting at 104-105° (yield 15%). It gave no colour with ferric chloride, did not exhibit any of the reactions of the aldehyde group and was found to be identical with a sample obtained from resorcinol monomethyl ether by treatment with excess of bromine in acetic acid solution. The mixed melting point was undepressed. (Found: C, 23.8; H, 1.5; and Br, 65.9.  $C_7H_5O_2Br_3$  requires C, 23·3, H, 1·4 and Br,  $68\cdot4\%$ .)

Similar results were obtained when acetic acid was used as solvent in the place of ether.

## REFERENCES

- 1. Murti and Seshadri .. Proc. Ind. Acad. Sci. (A), 1941, 13, 221.
  - ------ .. *Ibid.*, 1941, **13**, 399.
- Ott and Nauen
  Ber., 1922, 55 B, 920.
  Rao, Srikantiah and Iyengar
  J. C. S., 1929, 1578.