

collected using  $\text{CuK}\alpha$  radiation by the multiple-film equi-inclination Weissenberg technique. Spot-shape, absorption, Lorentz and polarisation corrections were applied in the usual way. From three-dimensional Patterson synthesis and a minimum function based on the position of the phosphorus atom, the positions of the atoms in the phosphate group were located. Thereafter, successive Fourier and difference Fourier syntheses revealed the rest of the structure. Least-squares refinement was carried out using the LALS program (originally written by Gantzel, Sparks and Trueblood and modified later by Liminga of Uppsala and also in the University of Madras) on the IBM 370/155 computer at the Indian Institute of Technology, Madras. With anisotropic thermal parameters for all atoms, the residual index at present is 0.10 for 1000 observed reflections. Further refinement is under progress. The present fractional atomic coordinates are presented in Table I. From bond distances and bond angles, it is concluded that the formula of the crystal structure may be written as glycine dihydrogen phosphate ( $\text{NH}_3^+ \text{CH}_2 \text{COOH} \cdot \text{H}_2\text{PO}_4^-$ ). Hydrogen bonds of the type  $\text{N}-\text{H} \cdots \text{O}$  and  $\text{O}-\text{H} \cdots \text{O}$  exist in the structure.

TABLE I

Fractional atomic coordinates for glycine orthophosphate

| Atom            | x      | y     | z     |
|-----------------|--------|-------|-------|
| P               | 0.400  | 0.154 | 0.223 |
| O <sub>1P</sub> | 0.279  | 0.019 | 0.144 |
| O <sub>2P</sub> | 0.546  | 0.077 | 0.358 |
| O <sub>3P</sub> | 0.450  | 0.249 | 0.110 |
| O <sub>4P</sub> | 0.342  | 0.293 | 0.307 |
| O <sub>1</sub>  | 0.017  | 0.106 | 0.136 |
| O <sub>2</sub>  | 0.111  | 0.019 | 0.389 |
| C <sub>1</sub>  | 0.009  | 0.080 | 0.271 |
| C <sub>2</sub>  | -0.138 | 0.135 | 0.275 |
| N               | -0.253 | 0.200 | 0.118 |

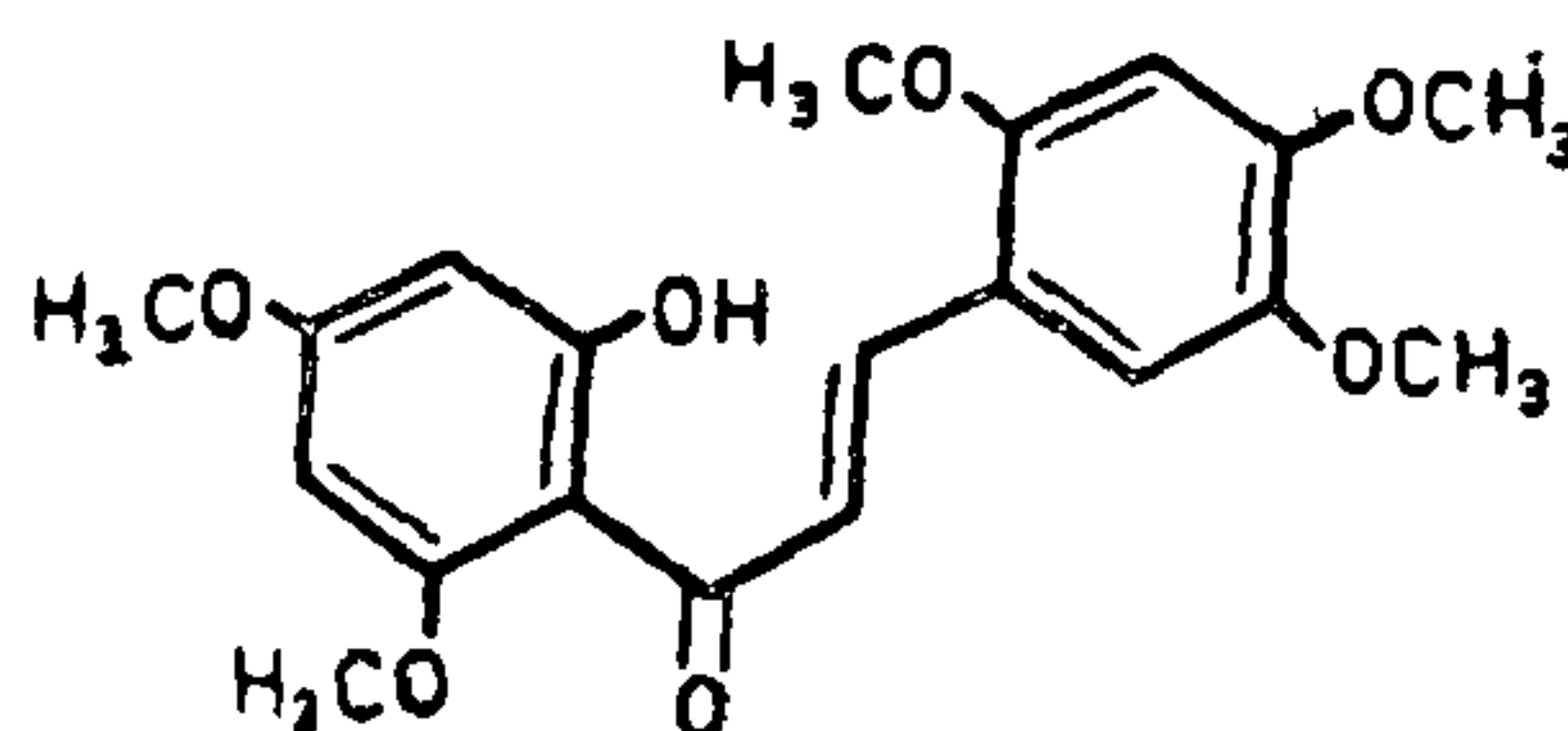
The authors are thankful to Prof. K. S. Chandrasekaran and Prof. S. Balasubramanian for their interest in this work. One of the authors (R. T.) is thankful to the U.G.C. for the award of a fellowship under the Faculty Improvement Programme.

School of Physics, R. THULASIDHASS,  
Madurai-Kamaraj University, J. K. MOHANA RAO,  
Madurai 625 021,  
India, April 27, 1979.

## SYNTHESIS OF RUBONE

SYNTHESIS of rubone (I), a naturally occurring chalcone, is being reported for the first time. This confirms the structure 2'-hydroxy-4',6',2,4,5-pentamethoxychalcone for rubone, assigned on the basis of chemical and spectral data.

Recently we have reported<sup>1</sup> the isolation of rubone (I) from petrol extract of the seed shells of *Derris robusta*. The structure for rubone as 2'-hydroxy-4',6',2,4,5-pentamethoxychalcone was proposed on the basis of its chemical and spectral data. This compound has now been synthesized, thereby providing synthetic confirmation for the constitution proposed for it



I

Condensation of 2-hydroxy-4,6-dimethoxyacetophenone with 2,4,5-trimethoxybenzaldehyde<sup>2</sup> in the presence of potassium hydroxide afforded I which agreed with natural sample in m.p., m.m.p., Co-TLC and co-IR. A mixture of 2-hydroxy-4,6-dimethoxyacetophenone (50 mg), 2,4,5-trimethoxybenzaldehyde (50 mg) was dissolved in 50 ml of ethanol and 10 ml of 10% KOH added to it. The reaction mixture was kept at room temperature for 48 hr followed by acidification in cold with conc. HCl when yellow solid separated out. It was found to be a mixture on TLC and hence subjected to column chromatography over silica gel when crystalline yellow compound (35 mg) was obtained on elution with benzene: pet. ether (1:1), m.p. 184-86°.  $\nu_{\text{max}}^{\text{KBr}}$  3450, 1618, 1608, 1442, 1035 and 828  $\text{cm}^{-1}$ . NMR ( $\text{CDCl}_3$ ,  $\delta$ ): 3.81 (s, 3H,  $\text{OCH}_3$ ), 3.87 (s, 9H, 3  $\times$   $\text{OCH}_3$ ), 3.93 (s, 3H,  $\text{OCH}_3$ ), 5.92 (d,  $J=2.5$  Hz, 1H,  $\text{H}_3$ ), 6.07 (d,  $J=2.5$  Hz,  $\text{H}_6$ ), 6.49 (s, 1H,  $\text{H}_5$ ), 7.09 (s, 1H,  $\text{H}_4$ ), 7.80 (d,  $J=16.5$  Hz,  $\text{H}_a$ ) and 8.1 (d,  $J=16.5$  Hz,  $\text{H}_b$ ).

Department of Chemistry,  
University of Delhi,  
Delhi 110 007,  
April 23, 1979.

S. S. CHIBBER,  
R. P. SHARMA,  
S. K. DUTT

1. Chibber, S. S., Sharma, R. P. and Dutt, S. K., *Phytochemistry*, Communicated.
2. Govindachari, T. R., Nagarajan, K. and Parthasarathy, P. C., *J. Chem. Soc.*, 1957, 1, 548.