

THE X-RAY STUDY OF MONOBROMODUCLAUXIN

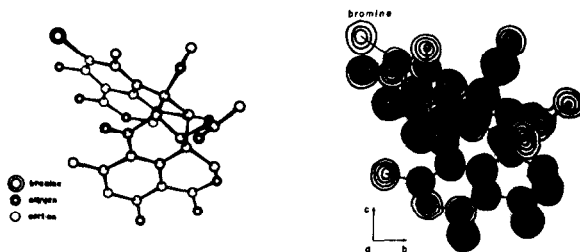
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AS several chemical approaches to determine the structure of duclauxin,  $C_{29}H_{22}O_{11}$ ,<sup>1)</sup> were unsuccessful, an X-ray study has been carried out to solve the problem.

Crystals of monobromoduclauxin, m.p. 260° (decomp.) were examined with Cu-K $\alpha$  radiation to give the following data:  $C_{29}H_{21}O_{11}Br$ , M=612 (Calcd. M=625.4), orthorhombic, space group:  $P 2_1 2_1 2_1$ , a=15.01, b=18.83, c=9.15 Å, D<sub>m</sub>=1.57 g/cm<sup>3</sup> (by floatation method), Z=4. Three dimensional data were collected for 1811 independent reflections of  $0kl \sim 7kl$ ,  $h0l$ ,  $hk0 \sim hk7$  with Weissenberg camera using heavy atom method in seven three-dimensional electron-density syntheses and three difference syntheses. Refinement was made with six cycles of least-square analysis on an IBM 7090 computer using diagonal approximations. The R-value finally stands at 0.16 excluding non-observed reflections. The absolute configuration of monobromoduclauxin which was elucidated by the anomalous dispersion method has now been represented as (I). A composite projection of the structure viewed along the a-axis is shown in Fig.1.



(I)

Fig.1

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REFERENCE

- 1) S. Shibata, Y. Ogihara, N. Tokutake, and O. Tanaka:  
Tetrahedron Letters, No. 18, 1287 (1965).