Tetrahedron Letters No.52, pp. 4795-4796, 1965. Pergamon Press Ltd.
Printed in Great Britain.

X-RAY STRUCTURE DETERMINATION OF BROMONORANISATINONE, A DERIVATIVE OF ANISATIN.

N.Sakabe, Y.Hirata.
Chemical Institute, Faculty of Science,
Nagoya University, Nagoya, Japan.
and
A.Furusaki, Y.tomiie, I.Nitta.
Faculty of Science, Kwansei Gakuin University,
Nishinomiya, Japan.

(Received 1 November 1965)

Bromonoranisatinone ($C_{14}H_{15}O_7Br$) is a derivative of a toxic substance, anisatin ($C_{15}H_{20}O_8$) isolated from seeds of Illicium religiosum Siebold et Zuccarini (Japanese star anise). The properties of anisatin and its derivatives are discussed in the preceding paper (1).

The space group of bromonoranisatinone is $P2_1$ with the cell constants a=10.10, b=7.51, c=10.05 A, β=110°, and there are two molecules per unit cell.

Three-dimensional X-ray data were collected with Cu Kα radiation on integrating Weissenberg photographs around b and c axes. Relative values of the observed structure factors of 1542 reflections were converted into absolute scale by Wilson's method (2).

Positions of the bromine atoms were derived from the three-dimensional Patterson function. Three-dimensional minimum function method (3) was carried out for the elucidation of the positions of light atoms. The structure thus obtained was refined by successive three-dimensional Fourier syntheses and the leastsquares methods. The R factor is 17.1 per cent at the present stage.

Fig. 1 and Fig. 2 show the molecular framework projected along

the b axis and the complete chemical formula of bromonoranisatinone, respectively.

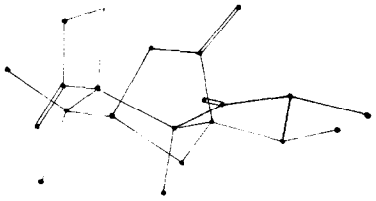

Fig. 1

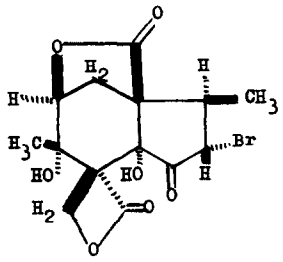

Fig. 2

The calculations were performed on the NEAC-2206 and the IBM 7090 computers using our programs for the former and those of Van den Hende (1961) and Sly, Shomaker, Van den Hende (1962) for the latter. The authors are grateful to Takeda Chemical Industries, LTD. for making the computer available, and are indebted to the National Institute of Health which supported this work through Grant RG-7969 and GM-7969. The sample used in this investigation was supplied by Mr. S.Takada Nagoya University, to whom the authors are indebted.

REFERENCES

1. K. Yamada, S. Takada, S. Nakamura and Y. Hirata, Tetrahedron Letters 52, 4785 (1965).
2. A. J. C. Wilson, Nature 150, 152 (1942).
3. M. J. Buerger, Acta Cryst. 4, 531 (1951).