X-ray Determination of the Molecular Weight of Luteoskyrin

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A chemical study on luteoskyrin, one of the toxic components produced by penicillium islandicum, has been carried out by T. Yamamoto, Y. Yamamoto, Kanatomo, Tanimichi and Kikui[1]. According to their results, this substance exists in two different crystal forms; one is needle-like and the other cube-shaped. The former is obtained by recrystallization from an ethanolic solution, and the latter from a mixed solvent of aceton, petroleum benzine and water (2:1:1). Both forms of crystals have the same composition, $C_{2.45}H_{2.05}O_1$.

Since ordinary methods failed to give a reliable value for the molecular weight, we applied the single crystal X-ray diffraction method to its determination. The samples used in this study were supplied by the above-mentioned authors.

The unit-cell dimensions of each form were determined from oscillation and Sauter-Schiebold photographs about the *c* axis taken with Cu K_α radiation. The length of the *c* axis was measured from layer-line spacings. The other two axial lengths were first estimated roughly from Sauter-Schiebold photographs and accurately determined using the spectra on a set of oscillation photographs which were

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1) Y. Yamamoto, T. Yamamoto, S. Kanatomo, K. Tanimichi and H. Kikui, *J. Pharm. Soc. Japan*, (*Yakugaku Zassi*) **76**, 670, (1956).

completely indexed by the graphical method.

The densities were measured by the floatation and the pyknometer methods** at 20°C. In the former method, aqueous solution of zinc chloride was used as floatation liquid. The results are tabulated in Table I.

TABLE I
CRYSTAL DATA

Crystal form	needle-like	cube-shaped
Crystal system & lattice type	orthorhombic primitive lattice	monoclinic primitive lattice
Lattice constants		
a	14.8_4 Å	15.1_7 Å
b	15.0_3	12.6_4
c	12.5_0	21.5_0
α	90°	90°
β	90°	135°***
γ	90°	90°
Volume of cell	2.79×10^3 Å^3	2.91×10^3 Å^3
Density	1.48 g.cm^{-3}.	1.47 g.cm.$^{-3}$

From the volume of the unit cell V and the density ρ, the molecular weight M can be calculated according to the formula

$$M=\rho VN/Z,$$

where N is the Avogadro number and Z the number of molecules in the unit cell, which is necessary to calculate M. From symmetry consideration, Z should be 2^n. However, the value of Z greater than eight can be excluded on account of the fact that the molecular weight of this substance has been supposed to be of the order of several hundreds[2], and this is also supported by its high melting point (273°C decomp.). It is highly improbable that Z equals to unity. The value of Z is four or two, the former being more probable.

When Z is assumed to be four, the molecular weight is found to be 621±30 for needle-like crystals and 644±20 for cube-shaped.

The difference, if it is significant, between these two values may be due to the fact that one or both forms crystallize with solvent molecules, but this can not be concluded from this experiment alone.

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** In order to lower interfacial tension, a small amount of surface-active substance was added.

*** This value was estimated using layer-line reflections on the oscillation photographs.

2) T. Yamamoto, Private communication.