

Neutron diffraction experiments were carried out by a diffractometer installed at the JRR-3 reactor of JAERI (10 MW, maximum thermal flux: 2×10^{13} n.cm⁻²sec⁻¹), with a Cu (111) transmission-type monochromator which reflects 0.98 Å neutrons. The usual θ - 2θ scanning was made along rekhas [110] and $[1\bar{1}0]$, over 2θ ranging from 18° to 68°, at room temperature and at 120°C, *i.e.* below and above the transition point (75°C) which was observed and discussed by Naito, Ishii, Hamaguchi & Oshima (1967). Integrated intensities of the fundamental reflexions 880; 16,16,0, 800, 16,0,0, and 24,0,0 observed at room temperature agreed well with those given by Willis (1964) for U₄O₉ single crystal.

Fig. 1(a) and (b) reproduces parts of the diffractometer records along the [110] rekha, at room temperature and at 120°C, where the peaks are indexed on the basis of the $4 \times a_0$ superlattice. In both of the figures small but distinct peaks are seen in between adjacent $4 \times a_0$ superlattice peaks. Since they can be indexed by half-integers, the structure of U₄O₉ is very probably with the $8 \times a_0$ superlattice both above and below the transition point. This result is in contrast with the conclusion accepted at present, that there exists only a $4 \times a_0$ superlattice in the structure of U₄O₉

(Belbeoch, Piekarski & Perio, 1961). Another $8 \times a_0$ superstructure peak, *viz.* 14.5, 14.5, 0, is also observed on the [110] rekha. Similar extra peaks are seen on the $[1\bar{1}0]$ diffractometer record as well, suggesting that the $8 \times a_0$ superlattice holds the cubic symmetry of the structure.

All of the observed $8 \times a_0$ superstructure reflexions showed the same behavior as most of the $4 \times a_0$ superstructure reflexions with respect to the phase transition (Naito *et al.*, 1967), *i.e.* their integrated intensities are stronger for the high-temperature phase than for the low-temperature phase.

Further analysis of the superstructure is in progress.

References

- BELBECH, B., LAREDO, E. & PERIO, P. (1964). *J. Nuclear Mat.* **13**, 100.
 BELBECH, B., PIEKARSKI, C. & PERIO, P. (1961). *Acta Cryst.* **14**, 837.
 NAITO, K., ISHII, T., HAMAGUCHI, Y. & OSHIMA, K. (1967). *Solid State Comm.* **5**, 349.
 WILLIS, B. T. M. (1964). *J. Physique*, **25**, 431.

Acta Cryst. (1968). B24, 1394

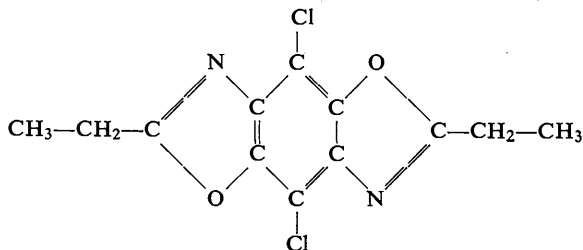
Unit cell and space group of 4,8-dichloro-2,6-diethylbenzo(1-2,4-5)bisoxazole. By L.G. ROLDAN and M.H. LITT,* *Allied Chemical Corporation, Central Research Laboratory, Morristown, New Jersey 07960, U.S.A.*

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The cell constants for 4,8-dichloro-2,6-diethylbenzo(1-2,4-5)bisoxazole are $a = 5.451 \pm 0.004$, $b = 13.398 \pm 0.006$, $c = 8.790 \pm 0.004$ Å; $\beta = 103.1 \pm 0.1^\circ$. The crystals belong to the space group $P2_1/a$. There are two molecules per unit cell.

In the course of the investigation of the synthesis of some dichlorobisoxazoles the 2,6-diethyl substituted compound was prepared. Details of its synthesis and postulated molecular structure have been published (Litt & Idelson, 1966).

The unit cell and space group have been found to be consistent with the postulated structure:



Single crystals were grown by recrystallization from benzene. They were found to be monoclinic and platelike, bounded principally by {110}. The cell constants determined from the zero layer of rotating crystal photographs

about the a and c axes with Cu $K\alpha$ radiation were refined by Bradley & Jay's (1932) extrapolation method. They are:

$$a = 5.451 \pm 0.004, b = 13.398 \pm 0.006, c = 8.790 \pm 0.004 \text{ \AA}; \\ \beta = 103.1 \pm 0.1^\circ.$$

The calculated density based on two molecules per unit cell is 1.545 g.cm⁻³, which is in agreement with an observed density of 1.55 g.cm⁻³. Precession photographs about the c axis and the rotation photographs showed the $h0l$ reflections to be absent when $h = 2n + 1$ and the $0k0$ reflections to be absent when $k = 2n + 1$; there were no other systematic extinctions; the space group was thereby established as $P2_1/a$. Since this space group shows four general positions in the unit cell, each molecule must lie with its center on a center of symmetry.

It may be noticed that a disordered structure with respect to N and O is possible because of the nearly equal electron density of these atoms and the symmetrical positions of the two heavier chlorine atoms.

No further work on this compound is planned.

References

- BRADLEY, A. J. & JAY, A. H. (1932). *Proc. Phys. Soc.* **44**, 563.
 LITT, M. H. & IDELSON, A. (1966). U.S. Patent 3,268,545.

* Present address: Division of Polymer Science, Case Western Reserve University, University Circle, Cleveland, Ohio 44106, U.S.A.