and 90 %, respectively. Above +35°C the amount of precipitate is more dependent on salt concentration than on temperature. At lower salt concentrations and at higher temperatures the β- and α-components are precipitated preferentially and preparative schemes may be worked out on that basis. Collagen which had been renatured at +4°C overnight behaved like native collagen on salt precipitation.

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The Crystal and Molecular Structure of a C,N-Disubstituted Oxaziridine

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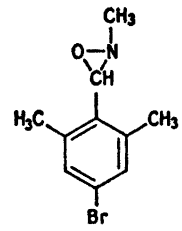
By UV-irradiation of *anti*-4-bromo-2,6-dimethyl-N-methylbenzaldoxime an isomeric compound was obtained.[1] This compound was first considered to be the stereoisomeric *syn*-compound, but on this basis the NMR-spectrum as well as the chromatographic behaviour was difficult to explain. From a paper on photochemical rearrangements of methyl substituted quinoline-N-oxides [2] we got the idea, that our compound might be a 2-methyl-3-(4-bromo-2,6-dimethylphenyl)-oxaziridine (I). A search of the literature [3] showed this to



(I)

be in accordance with the NMR-spectrum and the hydrolytic behaviour of the compound, and now the proposed molecular structure has been unambiguously established by the two-dimensional X-ray study described below.

The compound (m.p. 88—89°) crystallizes in exceedingly thin, long needles, which have a strong tendency to split in hairthin fibres parallel to the needle axis. It is rather volatile, and crystals having cross-sections as large as 0.06 × 0.02 mm evaporate completely in a couple of days when kept at room temperature on a microscope slide.

The crystals are monoclinic with the unique axis (*b*) in the direction of the needle axis. Weissenberg photographs *h0l*, *h1l* and *nk2n* made with Cu$K\alpha$-radiation showed the presence of reflections of all orders except *h0l* for $l = 2n + 1$ and *0k0* for $k = 2n + 1$. This indicates that the space-group is probably $P2_1/c$. The cell-dimensions found are $a = 11.5$ Å, $b = 4.18$ Å, $c = 21.6$ Å, $\beta = 96.3°$. There are 4 molecules per unit cell corresponding to a crystallographic density of 1.57 in good accordance with the value 1.62 found by flotation.

Reflections *h0l* were recorded by multiple-film technique, the intensities were estimated visually and Lorentz and polarization corrections were performed. The intensities of the diffracted beams fall off rapidly with increasing deviation angle and no spot with $\xi > 1.3$ was observed. 81 independent reflections were measured. Since only an approximate determination of the electron density projection was aimed at, no attempt to obtain better data was made.

A Patterson projection calculated and plotted on a GIER computer [4] gave the bromine co-ordinates and the general orientation of the molecule in the cell. Structure factors with signs based on the

bromine contribution (R = 31 %) were used for calculation of an electron density projection.[4] In this the bromine atom as well as the benzene ring with the three carbon atoms attached were clearly seen. These atoms were included in a new structure factor calculation (R = 26 %) with a subsequent synthesis of the electron density map. Now all the atoms of the molecule could be included in the structure factor calculation (R = 18.2 %) and a final refinement[5] of the positional parameters of the O, N and C-atoms (except the benzene carbons) lowered the R-index to 16.4 %. No further refinement was intitled by the given data.

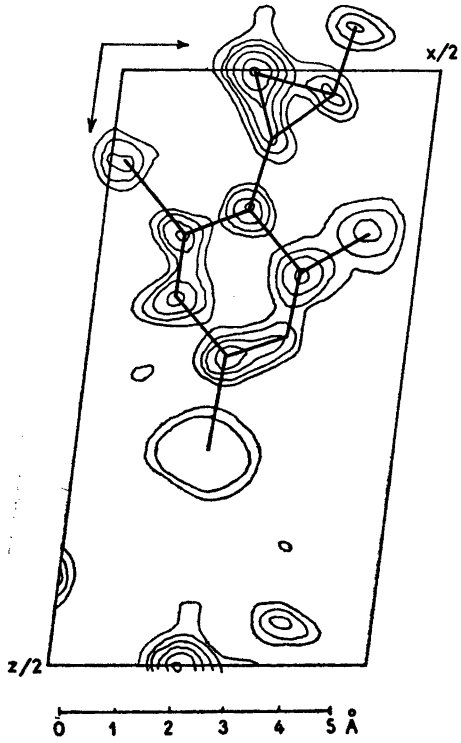


Fig. 1. Electron density projection of 2-methyl-3-(4-bromo-2,6-dimethylphenyl)-oxaziridine. Contours at arbitrary but equal intervals, except around the bromine atom where only the first and the fourth contour is drawn. Zero and negative contours are omitted.

Fig. 1 shows the molecule placed roughly parallel to the plane of projection. The presence of the three-membered ring is unequivocally established by the following reasoning: From chemical evidence it is known that at least two covalent bonds connect the three atoms in question; only under the assumption of a three-membered ring is this fact compatible with the observation that in the electron density projection the distances between each pair of the three atoms are rather close to expected single bond lengths. The CNO-ring is tilted a little out of the plane of the benzene ring, but the N—O-bond is approximately parallel to that plane. The configuration of the oxaziridine substituents is *trans* as might be expected.

A compound similar to (I) but containing chlorine instead of bromine has been prepared by reactions analogous to those leading to (I). This compound (m.p. 83—84°) has been shown to be isomorphous to the crystals of (I) and it is currently investigated in details by X-ray methods. The result is intended for publication in *Acta Crystallographica*.

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