

greatest number of domains are simultaneously reflecting. The image recorded at each stationary setting would be of uniform intensity and would equal the area irradiated since there are, throughout that area, domains that simultaneously fulfill the Bragg condition. The bent lattices of Fig. 1 and the sequence in which their images

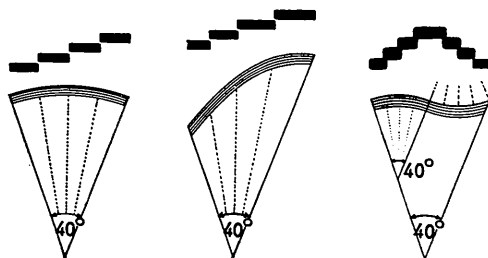


Fig. 1. Schematic representation of the correlation between lattice bending and X-ray images.

are recorded illustrate that, if the lattice is bent, it will not be possible to record the complete irradiated area in one setting. The minimum number of specimen settings required will equal the angular range of reflection divided by the horizontal divergence of the beam. Four settings would be required for each of the examples of Fig. 1 since the reflection range is 40 seconds of arc and the beam imperfection is 10 seconds of arc.

The ability of the technique to rule out the possibility of lattice bending and to distinguish between regions of slightly different orientation was established by data obtained from a fixed area of a zinc single crystal grown from the melt and subsequently quenched and annealed. The (0001) reflection from a face acid-cut parallel to the cleavage planes was examined. The correction due to dispersion was negligible in the $(n, -n)$ position. As grown, the rocking curve was Gaussian with a half-width of 40 seconds of arc and the image recorded with the specimen stationary at the rocking curve peak was uniform in intensity and identical in area and shape to the area irradiated and to the image recorded by con-

tinuously rotating the specimen throughout its angular range of reflection. Images recorded at the specimen orientations of half maximum intensity were of the same area as that recorded at the peak. The specimen condition is therefore similar to the random condition described, and there is no evidence of lattice bending.

The specimen showed evidence of damage when quenched from 400° C., for the rocking curve half-width was then 6 minutes of arc and the reflecting range 14 minutes of arc; such an increase was not observed for other zinc crystals when quenched. Annealing at 400° C. for 1 hr. broke this rocking curve up into one having several peaks and a total reflection range of 8 minutes of arc. Only seven stationary exposures were required to record images whose sums equal the area irradiated and that recorded by continuous rotation. More than seven sub-grains were observed since several of the images recorded simultaneous reflection from non-adjacent domains having the same orientation. The possibility of lattice bending was, therefore, eliminated and the possibility that the lattices of the individual sub-grains were bent was eliminated by the observation that there was, for each sub-grain, an orientation that gave an image of maximum area and that this area encompassed the images recorded for that particular sub-grain in slightly different angular positions.

The high resolving power of the technique gives detailed information about the lattice topography. Within the limitations imposed by the imperfection of the first crystal, the possibility that specimen lattices are bent can be eliminated by the observation that the image size and shape of one setting, or a number of settings less than that calculated by dividing the reflecting range by the imperfection of the monochromatized beam, is equal to the area irradiated.

References

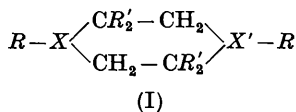
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The conformation of 1:4-diphenyl-1:4-diarsacyclohexane. By S. C. NYBURG and J. HILTON, *Department of Chemistry, University College of North Staffordshire, Keele, Staffordshire, England.*

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F. G. Mann and his co-workers (Beeby & Mann, 1951; Mann & Millar, 1952; Jones & Mann, 1955) have prepared and examined a number of heterocyclic compounds



of type (I), in particular those in which $X, X' = N, P$

or As (including those where $X \neq X'$). These compounds show interesting variations in their behaviour towards quaternizing agents, in that certain of them (e.g. (II) and (III)) will readily undergo diquaternization with simple alkyl halides but none will undergo diquaternization (with cyclization) with alkylene dibromides.

Thus 1:4-dimethylpiperazine ($X = X' = N, R = CH_3, R' = H$) reacts with ethylene dibromide to form a diquaternary salt bridged across the nitrogen atoms, but none of the following compounds will quaternize in this manner:

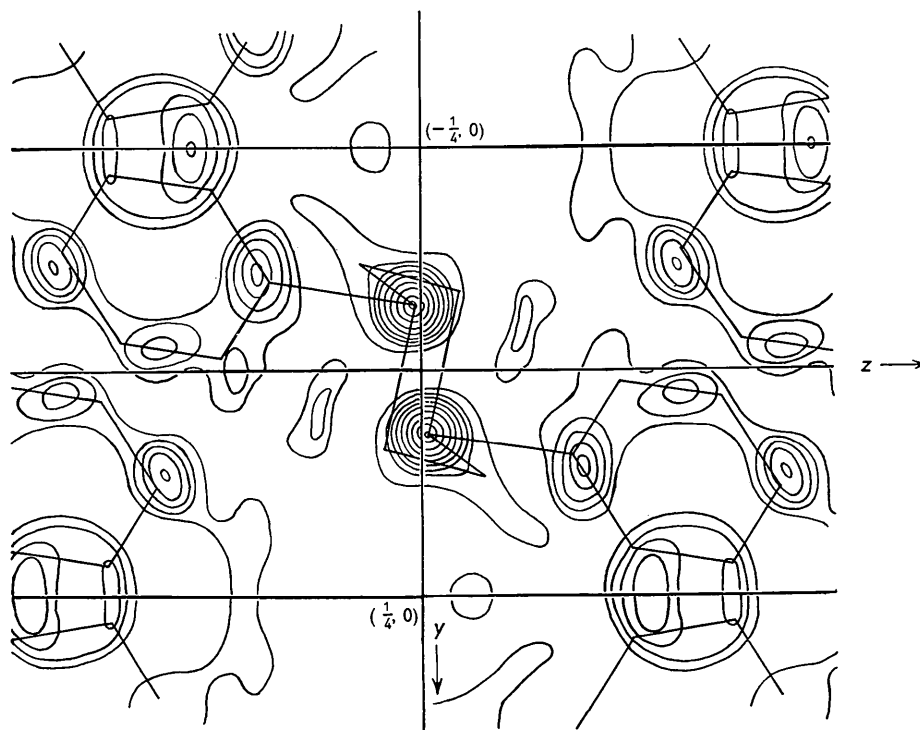


Fig. 1. The (100) electron-density projection. Contours at arbitrary levels with some omitted from As peaks.

	X	X'	R	R'
(II)	N	N	CH ₃	CH ₃
(III)	As	As	C ₆ H ₅	H
	N	P	C ₆ H ₅	H
	N	As	C ₆ H ₅	H

The molecule of the dinitrate of hexamethyl-piperazine (II) has been shown by X-rays to be centrosymmetric (Mann & Senior, 1954) and this means that the cation has the chair conformation. The molecule of the base may be 'locked' in this conformation by the *gem*-dimethyl groups and be prevented from bridging with ethylene dibromide.

We have examined crystals of 1:4-diphenyl-1:4-diarsa-cyclohexane (III) (Jones & Mann, 1955), and find that this molecule also has the chair conformation, although in this case, unlike (II), there are no alkyl groups which would prevent bridging.

The crystals belong to the orthorhombic system with

$$a = 7.41, b = 16.74, c = 11.90 \text{ \AA}; Z = 4.$$

The absences correspond to space group *Pmnc* but all (*Ok*l) with *k* odd are very weak.

The Patterson (100) projection reveals immediately that if the space group is *Pmnc* the arsenic atoms must be located very close to $(\frac{1}{2}, 0, \frac{1}{2})$ and $(\frac{1}{2}, 0, \frac{1}{2})$, together with the fourfold repeat positions of each. It proves, however, impossible to reconcile this space group with packing requirements whatever configuration is assumed for the molecule. If (*Ok*l) with *k* odd be discounted the

space group is *Pbcn* with arsenic atom coordinate close to $(x, 0, \frac{1}{2})$ and eightfold repeats. Assuming this to be the space group, the electron-density projection after two refinements was as shown in Fig. 1. Located as it is, the molecule must be centrosymmetric apart from possible slight deviations from the space-group requirements. This projection also shows clearly which of the two possible substitution positions on the arsenic atom the phenyl groups occupy. However, this was only to be expected in view of the almost certain steric conflict of the bulky phenyl groups with the central ring if the alternative substitution positions were occupied. The reason for the existence of faint (*Ok*l) reflexions with *k* odd remains obscure, but most probably arises from slight oxidation during examination to the corresponding asymmetric arsine oxide.

We are grateful to Dr F. G. Mann for suggesting this examination and supplying the crystals.

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