

it was positioned in real space by obtaining the best fit to the axial reflexions, assuming *pgg* symmetry. A structure-factor calculation based on this benzene ring, followed by an electron-density map, yielded the positions of the remaining atoms. Further Fourier and least-squares refinement reduced the reliability index, defined as $R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$, from 0.37 to 0.12. Postulation of probable hydrogen positions reduced the reliability index to 0.10. The coordinates obtained at this stage are given in Table 2; the corresponding electron-density map (Fig. 1) gives clear verification that *p*-aminobenzoic acid molecules are associated as dimeric pairs.

On the basis of being able to obtain a structure and consideration of the density and unit-cell parameters it can be assumed that the twinning is such that the single-crystal constituents have identical rectangular projections down the twin *c* axis. The departure from *mmm* symmetry in the upper layer lines was attributed to each portion of the twin being present in differing amounts, and to the possible

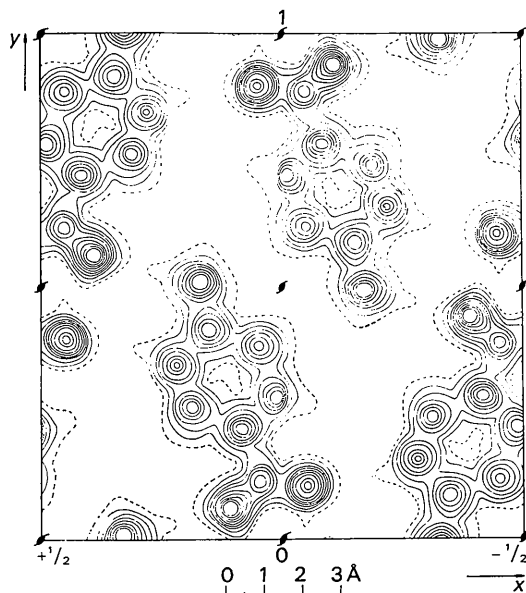


Fig. 1. Electron density projection perpendicular to *c* of form I. The contours are at intervals of $1 \text{ e.}\text{\AA}^{-2}$; the $1 \text{ e.}\text{\AA}^{-2}$ contour is shown dashed.

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The growth of crystals in silica gel, and the unit-cell dimensions of cadmium oxalate and copper tartrate.

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It has been known for at least fifty years that single crystals of some insoluble compounds can be grown by allowing suitable ions to diffuse slowly together in a suitable medium where they react. Johnson (1914) allowed the reactants to diffuse into a large volume of water and obtained crystals

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Table 2. Atomic coordinates and isotropic temperature factors

Atom	<i>x/a</i>	<i>y/b</i>	<i>B</i> (\AA^{-2})
C(1)	+0.147	+0.416	3.9
C(2)	+0.218	+0.351	3.9
C(3)	+0.186	+0.256	4.2
C(4)	+0.083	+0.226	3.7
C(5)	+0.013	+0.286	4.4
C(6)	+0.043	+0.384	3.9
C(7)	+0.051	+0.124	4.2
N(8)	+0.177	+0.513	4.1
O(9)	+0.109	+0.066	3.9
O(10)	-0.050	+0.102	3.9

monoclinic symmetry of the single crystallites, although on consideration of the unit-cell dimensions alone it is difficult to see how this could be the monoclinic form reported by Prasad *et al.* If the crystallites are monoclinic then this is an example of a crystal showing para-orthorhombic symmetry of the type discussed by Dunitz (1964).

Clearly under certain favourable conditions it is possible to elucidate details of a crystal structure with the use of a twinned crystal, but the fact of obtaining a rectangular projection with *mm* symmetry by no means guarantees that Fourier techniques can be applied to obtain a solution. If it is assumed that twinning is on a gross enough scale, then the intensities which arise at superimposed reciprocal lattice points will be proportional to the sum of the squares of the structure factors for each reciprocal lattice point. If the twinning is not on such a gross scale the relative phases of the structure factors will be important. Thus care must be exercised in applying the usual Fourier methods of crystal determination. In this case with the twinned crystal data available it is impossible to determine the *x* coordinates of the atoms in form I without recourse to an image seeking technique in Patterson space.

No further work is contemplated on the twinned crystals unless a single crystal is obtained.

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of calcium hydroxide, while Hatschek (1911) prepared small crystals of gypsum by allowing sulphate ions to diffuse into a gelatin gel which had been prepared from a dilute solution of calcium chloride. Dreaper (1913) obtained crystals of lead chloride by using a glass capillary tube, sealed at one end, to contain one of the reacting solutions and placing the other, open, end of the tube into a solution of the other reactant. Holmes (1917) has briefly reviewed previous work on crystallization in various media, including agar and gel-

atin gels and even fruit jam. He also described many of his own experiments which used silica gel. Interest has recently been revived in this method by 'Amateur Scientist' (1962) and by Vand, Henisch & McCauley (1963) who reported the growth of crystals of some salts of organic acids, some minerals including calcite, and some metals. There are many problems in solid-state physics which require the study of large single crystals and it seemed worth while to us to examine the possibilities of the method.

In our experiments the gels were prepared by adding either *N* acetic acid or *N* hydrochloric acid to a solution of waterglass, the density of which was varied between 1.02 and 1.08 g.cm⁻³. The mixture was poured into U-tubes and allowed to stand. The time required for the gel to set varied from a few seconds to a few hours; the gels used for the crystal-growing experiments were those that were nearly transparent. The gels were slightly acidic and formed plugs some four or five centimetres long in the bottom of the tubes. Copper tartrate crystals were prepared by placing a 0.5*N* solution of copper sulphate in one arm of a U-tube and a 0.5*N* solution of tartaric acid in the other arm. Several such tubes were prepared, and bright blue crystals with extremely well formed faces appeared in all of them after a few days, the dimensions of the largest crystal being 3 mm × 1.5 mm × 1.5 mm. Cadmium oxalate crystals were grown in a similar manner, the reacting solutions in this case being a 0.4*N* aqueous solution of cadmium iodide and a 2*N* alco-

holic solution of oxalic acid. Colourless crystals grew in all the specimen tubes, the largest crystals being about the same size as those of the copper tartrate.

Zero, first and second layer equi-inclination Weissenberg photographs were taken with the crystals rotating about the *c* axes. Cu *K*α radiation was used; the unit-cell dimensions were calculated from the diffraction patterns and are given in Table 1.

The densities of the crystals were measured in the standard way with a small density bottle. The observed values are compared with those calculated from unit-cell dimensions and composition of the crystals in Table 2. Copper tartrate crystals dehydrated easily when stored over calcium chloride, and by measuring the resulting loss of weight the degree of hydration was determined. The number of water molecules given in Table 2, for cadmium oxalate, is merely the number which gives best agreement between observed and calculated density.

In conclusion, it should be pointed out that, although the method works for some crystals, it does not follow that crystals of all insoluble salts may be grown in this way. Our own attempts to grow crystals of insoluble sulphides were failures and resulted only in finely divided precipitates appearing in the gels. However we do not yet know the optimum conditions for crystal growth and we feel that further research on the effect of such variables as concentration of the reactants, diffusion rates, temperature, and composition of the gel might be rewarding.

Table 1. Unit-cell dimensions

Cell parameter	Cadmium oxalate	Copper tartrate
α	135° 52' ± 30'	97° 21' ± 1°
β	132 29 ± 30	80 43 ± 45'
γ	68 22 ± 30	115 42 ± 30
<i>a</i>	7.36 ± 0.04 Å	8.42 ± 0.07 Å
<i>b</i>	9.39 ± 0.04	12.33 ± 0.10
<i>c</i>	9.06 ± 0.04	8.82 ± 0.07

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Table 2. Observed and calculated densities (g.cm⁻³)

Crystal	Observed	Composition	<i>N</i> *	Calculated
Copper tartrate	2.05 ± 0.05	Cu ₄ H ₄ O ₆ · 3H ₂ O	4	2.17 ± 0.06
Cadmium oxalate	2.79 ± 0.05	CdC ₂ O ₄ · 3H ₂ O	2	2.73 ± 0.05

*N** is the number of molecules per unit cell.

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The permissible rotations of a two-dimensional lattice. By EPHRAIM RAVID, *Scientific Department, Ministry of Defence, Box 7063, Tel-Aviv, Israel*

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There follows a simple proof of the well known fact that the only permissible rotations of a two-dimensional lattice are multiples of $2\pi/n$, where $n=1, 2, 3, 4$, and 6.

The proof rests on the obvious identity of the permissible values of n with the orders of those identical regular polygons which under rotation and translation can cover completely a finite plane surface. The angle between two

adjacent sides of a regular polygon of order n is $\pi(1-2/n)$. It is then required that an integral number m of such angles be equal to 2π , or $m\pi(1-2/n)=2\pi$, $0 \leq m \leq \infty$.

Dividing by $m\pi$ we have $2/n+2/m=1$. The only finite, integral solutions of this equation are $n=2, 3, 4, 6$. $n=1$ is of course the identity transformation.