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Unit cell and space group of sublimed fumaric acid.

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In the course of an investigation of the crystallization of fumaric acid, two different crystalline forms were found.

One is obtained when a water solution is allowed to crystallize. This form was studied by Reis & Schneider (1928) who claimed it to be monoclinic, whereas Yardley (1925) reported it as triclinic. Our experimental data from an identical type of crystallization agree with the Reis & Schneider work.

A second type of crystallization was obtained by sublimation of the previous form at 220–235 °C. This new crystalline form is predominantly composed of needlelike contact twins. The twins are formed by two enantiomorphic structures in which the (10$\bar{1}$) plane is the plane of composition and also the twin-plane. The new cell is triclinic and is not, so far as we know, covered by any publication. Its parameters are:

$a=4{\cdot}52\pm0{\cdot}02$, $b=7{\cdot}51\pm0{\cdot}02$, $c=5{\cdot}40\pm0{\cdot}04$ Å
$\alpha=136{\cdot}7\pm0{\cdot}3^\circ$, $\beta=110{\cdot}6\pm0{\cdot}3^\circ$, $\gamma=72{\cdot}8\pm0{\cdot}3^\circ$.

The number of molecules per unit cell is one. A calculated density of 1·63 g.cm^{-3} agrees with the observed density of 1·60 g.cm^{-3}. The space group $P\bar{1}$ is suggested by the absence of pyroelectric effect.

No further work on this compound is planned.

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Standard deviation of coordinates as a function of the residual and temperature coefficient.

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Tables of R, the residual, as a function of the standard deviation of coordinates, $\sigma(r)$, and temperature coefficient, B, have been calculated in two and three dimensions (Stanley, 1964, 1965). It has been suggested that tables in which the standard deviation of coordinates as a function of the residual were tabulated might be more use. These tables have now been prepared by interpolation from those previously published and give the values of $\sigma(r)$ in Å as a function of R and B for carbon atoms of the form suggested by Vand, Eiland & Pepinski (1957) within the limit of the sphere of reflexion of Cu $K\alpha$ radiation. From these tables it is probably more obvious how the data and the symmetry control the value of the residual. For three-dimensional data for a structure in a centrosymmetric space group with zero temperature coefficient, a residual of 5 % corresponds to a standard deviation of coordinates of 0·009 Å. The same residual for two-dimensional data for a structure in a non-centrosymmetric space group and a temperature coefficient of 4 Å^2 corresponds to a standard deviation of coordinates of 0·022 Å.

A comparison with other methods of estimating the standard deviation is interesting. The method of Booth (1947), as might be expected, gives estimates which agree very well with the present work. The [001] projection of triphenyl phosphate (Davies & Stanley, 1962) has a value of $R=14\,\%$ and a value of $B=4{\cdot}5$ Å^2. The value of $\sigma(r)$ calculated by Booth's method is 0·04 Å. This agrees very well with the value of 0·04 Å obtained from the tables. Comparison with estimates of standard deviations made from the values of the slopes of the difference map at atomic positions is less satisfactory because of the contribution made to the residual by all the deviations in the difference map and not merely the contribution from the atomic centres, and in all cases the error given by these tables is overestimated.

Table 1. *Values of* $\sigma(r)$ *as a function of* (1)*R and B in two dimensions*

B	0·0	2·0	4·0	6·0	8·0	10·0
R						
0·00	0·000	0·000	0·000	0·000	0·000	0·000
0·05	0·017	0·019	0·022	0·024	0·027	0·029
0·10	0·034	0·038	0·043	0·048	0·054	0·058
0·15	0·051	0·058	0·065	0·073	0·081	0·089
0·20	0·069	0·079	0·090	0·101	0·112	0·122
0·25	0·090	0·102	0·117	0·132	0·146	0·159
0·30	0·113	0·129	0·148	0·167	0·186	0·204
0·40	0·175	0·204	0·237	0·269	0·300	0·328
0·50	0·315	0·380	0·440	0·500	0·561	0·623

Table 2. *Values of* $\sigma(r)$ *as a function of* ($\bar{1}$)*R and B in two dimensions*

B	0·0	2·0	4·0	6·0	8·0	10·0
R						
0·00	0·000	0·000	0·000	0·000	0·000	0·000
0·05	0·011	0·012	0·014	0·015	0·017	0·018
0·10	0·021	0·024	0·027	0·031	0·034	0·037
0·15	0·033	0·037	0·042	0·047	0·052	0·057
0·20	0·045	0·051	0·057	0·064	0·071	0·078
0·25	0·057	0·065	0·074	0·083	0·092	0·100
0·30	0·071	0·080	0·091	0·103	0·114	0·125
0·40	0·100	0·116	0·132	0·149	0·165	0·181
0·50	0·138	0·160	0·184	0·209	0·232	0·254
0·60	0·192	0·225	0·262	0·298	0·331	0·361

Table 3. *Values of* $\sigma(r)$ *as a function of* ($\bar{1}$)*R and B in three dimensions*

B	0·0	2·0	4·0	6·0	8·0	10·0
R						
0·00	0·000	0·000	0·000	0·000	0·000	0·000
0·05	0·009	0·010	0·011	0·012	0·013	0·014
0·10	0·018	0·019	0·021	0·023	0·025	0·028
0·15	0·027	0·029	0·031	0·035	0·038	0·042
0·20	0·036	0·040	0·043	0·048	0·053	0·057
0·25	0·047	0·051	0·056	0·061	0·067	0·073
0·30	0·057	0·062	0·069	0·077	0·083	0·091
0·40	0·081	0·089	0·098	0·108	0·119	0·130
0·50	0·109	0·120	0·133	0·148	0·163	0·178
0·60	0·146	0·162	0·180	0·202	0·224	0·247

Table 4. *Values of* $\sigma(r)$ *as a function of* (1)*R and B in three dimensions*

B	0·0	2·0	4·0	6·0	8·0	10·0
R						
0·00	0·000	0·000	0·000	0·000	0·000	0·000
0·05	0·014	0·015	0·016	0·018	0·020	0·021
0·10	0·028	0·030	0·033	0·036	0·040	0·043
0·15	0·042	0·046	0·050	0·055	0·060	0·066
0·20	0·057	0·062	0·067	0·075	0·082	0·090
0·25	0·073	0·079	0·088	0·096	0·106	0·116
0·30	0·091	0·099	0·109	0·121	0·134	0·146
0·40	0·135	0·150	0·167	0·186	0·206	0·226
0·50	0·214	0·242	0·276	0·312	0·349	0·383

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The structure of metallic dodecaborides.

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The most common polyhedral grouping of boron atoms is that in which 12 boron atoms occupy the vertices of an icosahedron. The existence of such a group has been convincingly proven in a number of higher borides and boron structures such as boron carbide, B_4C (Zhdanov & Sevastianov, 1941; Clark & Hoard, 1943), boron phosphide, $B_{12}P_2$ (La Placa & Post, 1961), an aluminum borocarbide, AlC_4B_{24} (Matkovich, Economy & Giese, 1964), α-boron (Decker & Kasper, 1959), β-boron (Hughes, Kennard, Sullinger, Weakliem, Sands & Hoard, 1963) and tetragonal boron (Hoard, Hughes & Sands, 1958).

A different grouping of 12 boron atoms, *i.e.* a cubo-octahedral arrangement, was proposed by Bertaut & Blum (1949) for UB_{12} on the basis of steric considerations. Subsequently, Post & Glaser (1952) showed that the X-ray powder lines of ZrB_{12} are consistent with the cubo-octahedral boron grouping, and demonstrated the existence of an isotypic series of metal dodecaborides. In their calculations they assumed the structure proposed by Bertaut & Blum (1949) and did not consider other possibilities. (It should be noted that the icosahedral grouping is consistent with the observed space group extinctions.) Since the degree of refinement of the ZrB_{12} structure was relatively low, it was felt desirable to establish more accurately the form of the boron grouping in the cubic dodecaboride series.

Single crystals of ScB_{12} and YB_{12} were prepared by heating the metal oxides and boron with B:Me ratio of 7:1 in an induction coupled plasma torch apparatus similar to that described by Reed (1961). The samples were heated to a temperature of 2000–2500°C and then quenched by dropping directly into a beaker of water. After washing with concentrated hydrochloric acid, crystals of ScB_{12} and YB_{12} could be easily handpicked under the microscope.

Rotation and Weissenberg photographs of ScB_{12} crystals indicated a tetragonal unit cell with $a=5{\cdot}22$, $c=7{\cdot}35$ Å. The systematic extinctions are consistent with the space group *I*4/*mmm* and its subgroups. This cell is pseudo-cubic and dimensionally related to the cubic dodecaboride series. The examined crystals were twinned on the (111) plane. The tetragonal cell is in disagreement with the findings of