Die intermolekularen Br–Br-Abstände sind merklich grösser: Abstände eines Bromatomes zu den zwei ihm benachbarten Bromatomen in der nächsten Zelle:

Br2–Br3′	4,16 Å	Br2–Br4′	4,23 Å

Abstände benachbarter Bromatome zweier Moleküle, die durch ein Symmetriezentrum miteinander verknüpft sind:

Br2–Br3″	4,14 Å
Br3–Br3″	4,25
Br4–Br3″	4,13

Damit besitzt $SnBr_4$ eine Kristallstruktur, die sich von der des kubischen SnJ_4 und der anderen Substanzen des SnJ_4-Typs wesentlich unterscheidet. Die Anordnung der Halogenatome im SnJ_4 stellt eine kubisch-dichteste Kugelpackung dar. Im $SnBr_4$ ist sie eine deformierte hexagonal-dichteste Kugelpackung.

Frau Prof. Dr. K. Boll-Dornberger und Herrn Dr. E. Höhne vom Institut für Strukturforschung der Deutschen Akademie der Wissenschaften zu Berlin danken wir für wertvolle Hinweise, welche die Arbeit förderten.

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Acta Cryst. (1963). **16**, 451

The Crystal Structure of Antimony at 4.2, 78 and 298 °K

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(*Received* 8 *June* 1962)

The lattice dimensions and structural parameter of high purity single crystals of antimony have been determined using low-extinction reflections of monochromated Ag *Kα* radiation for *z*, and Bond's precision technique with Cu *Kβ* radiation for *a* and *c*.

At 4·2 °K, $z=0{\cdot}23362$, $a=4{\cdot}3007$, $c=11{\cdot}222$ Å;
at 78 °K, $z=0{\cdot}23364$, $a=3{\cdot}3012$, $c=11{\cdot}232$ Å;
at 298 °K, $z=0{\cdot}23349$, $a=4{\cdot}3084$, $c=11{\cdot}274$ Å.

Comparison with another set of experiments, using Mo *Kα* radiation, suggests that an accuracy of $\pm 0{\cdot}00002$ in *z* was reached at the lower temperatures; the standard error in *a* and *c* values is about 1 part in 20,000.

Introduction

Recent redeterminations of the atomic positional parameter in Bi (Barrett, 1960; Cucka & Barrett, 1961, referred to as I and II, respectively) have shown the *z* coordinate to differ appreciably from the value usually accepted (James, 1921). The difference in the old and new *z* values (0·237 and 0·23389 respectively, at 298 °K) is sufficiently large to be of importance in theoretical considerations of the structure of Bi. Since Sb is isostructural with Bi, and the determination of its crystal structure also dates back some forty years (James & Tunstall, 1920), we have redetermined the *z* parameter of this element. Precise structure data for Sb are also needed in solid state research, for example in the interpretation of nuclear quadrupole coupling in this element, a matter of current interest as a method of studying the conduction electron density. Some unit cell dimensions of Sb at low temperatures were reported in II that were obtained by extrapolation of diffractometer readings taken on one side of the direct beam only. These have now been extended and redetermined with higher accuracy by the method of Bond (1960),

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using reflections at high diffraction angles on both sides of the direct beam and crystals of suitable perfection.

Experimental

The crystals used were of the extremely high purity that can be obtained with modern methods of distillation plus zone refinement. One set was prepared by zone melting, another by withdrawal from a temperature-controlled melt. Good (00·1) surfaces were prepared by cleavage; both (00·1) and (11·0) were also prepared by spark cutting and a combination of metallographic and chemical polishing.

The parameter z of the rhombohedral structure was first determined at 4·2, 78 and 298 °K by using accurately measured intensities of the 00·l reflections (hexagonal indexing) from the basal cleavage plane of a zone-melt grown single crystal, using crystal monochromatized Ag $K\alpha$ radiation. The equipment used is described in I and II. Of the 12 00·l reflections within the range of the diffractometer the 00·3, 00·6, 00·9 and 00·15 reflections were not measured because of the danger of appreciable error from extinction with these. The two outermost reflections, 00·33 and 00·36 were found to be systematically lower in intensity than expected on the basis of the others, probably because the broad peaks at high angles caused by crystal imperfection were too wide for the counter slits that were used. These reflections were therefore not used in the computation of z.

Results and discussion

Table 1 lists the parameters derived from the intensities of the remaining reflections by means of Fourier difference (F_o-F_c) syntheses as described in II. The scattering factors of Thomas & Umeda (1957) were used in the calculations with an anomalous dispersion correction, for the K electrons only, of $-1\cdot65$ electron units (James, 1948). It was found in our study of Bi that small alterations in the dispersion correction, although they caused appreciable variations in the derived temperature factor B, caused insignificant variations in z. As a check on the influence of the scattering factors assumed, z was also computed from James & Brindley's (1931) scattering factors, with K electron Hönl corrections; the individual results differed from the values reported here by only a few units in the fifth decimal place.

Table 1. *Temperature factors, B, (for vibrations parallel to c axes), reliability factors, R, in % $(\Sigma||F_o|-|F_c||/\Sigma|F_o|)$ and z parameters for antimony*

Crystal monochromatized Ag$K\alpha$ radiation

Temperature (°K)	z	B	R
4·2	0·233 62	0·021	1·37
78	0·233 59	0·097	1·28
	0·233 69	0·089	1·95
	0·233 64	0·100	1·33
	0·233 64	0·160	2·53
Average values at 78	*0·233 64*	*0·111*	*1·77*
298	0·233 45	0·691	4·46
	0·233 52	0·712	1·00
	0·233 58	0·620	1·01
	0·233 41	0·740	2·53
Average values at 298	*0·233 49*	*0·691*	*2·25*

It was felt desirable to repeat the determinations using different crystals, wavelengths, observers and methods of computing. Filtered Mo $K\alpha$ radiation was chosen, and the integrated intensities were determined for 00·15, 00·18, 00·21, 00·24, 00·27 and 00·30 reflections. Refinement was carried out both by Fourier difference synthesis and by the Busing–Levy (1959) least-squares program, based on F^2 values, on an IBM 704 computer. Again the scattering factors of Thomas & Umeda (1957) were used with an anomalous dispersion correction for the K, L and M electrons of $\Delta f''=2\cdot0$ and $-\Delta f'=0\cdot6$ (Dauben & Templeton, 1955). One run each was made at 2·4, 78 and 298 °K. The results are shown in Table 2.

Table 2. *Temperature factors, B, (for vibrations parallel to c axes), reliability factors, R, in % $(\Sigma||F_o|-|F_c||/\Sigma|F_o|)$ and z parameters for antimony*

Mo$K\alpha$ radiation

Temperature (°K)	z	B	R	Remarks
4·2	0·233 67	0*	3·31	(1)
	0·233 64	0*	6·20	(2)
78	0·233 66	0*	4·93	(1)
	0·233 64	0*	5·80	(2)
298	0·233 79	0·679	3·76	(1)
	0·233 80	0·671	3·70	(2)

(1) Computation by Fourier difference synthesis.
(2) Computation by least square IBM 704 computer.
* Temperature factor held at 0 for computations.

The small differences in the results obtained with the different computation methods are ascribed to the difference in the weighting schemes. For the IBM 704 computations, each intensity was given a weight $W=M/(I-Q)$ where I is the total integrated intensity, Q is the background count for the same angular range of integration and M is the number of times the readings were repeated; the difference synthesis effectively weighted each reflection according to $W=1/f$, where f is the scattering factor. The background intensity experienced with Mo radiation was several times as large in comparison with line intensities as was obtained with crystal monochromatized Ag radiation. Hence the z parameters obtained with Mo radiation are less reliable than those of Table 1, and this is evidenced by the higher R values. Nevertheless, the discrepancies between corresponding values in the two tables for the runs at 4·2 and 78 °K are of the order of 0·00002 in z (or 0·0002 Å). We conclude, therefore, that the accuracy attained for z at these low temperatures may be estimated as $\pm 0{\cdot}00002$. The room temperature values of Table 2, on the other hand, are much poorer because of the large background corrections and diverge from the Table 1 value by 0·00031 (corresponding to about 0·003 Å). Considering all of the data, it may be concluded that by increasing the temperature from 4·2 to 78 °K z does not change by a measurable amount; and that increasing the temperature further to room temperature decreases z by an amount that is significantly greater than estimated errors when monochromatized Ag radiation is used (though this cannot be confirmed by the less accurate filtered Mo radiation data).

Table 3. *Lattice constants c and a (hexagonal axes) and nearest and second nearest neighbor distances A_1, A_2, in antimony*

Temperature (°K)	a (Å)	c (Å)	A_1 (Å)	A_2 (Å)
4·2	4·3007	11·222	2·902	3·343
78	4·3012	11·232	2·903	3·344
298	4·3084	11·274	2·908	3·355

Table 3 lists unit cell dimensions obtained with Cu $K\beta$ radiation ($\lambda = 1{\cdot}39217$ Å) and the Bond (1960) technique, using 33·0 and 00·16 reflections respectively, for the a and c measurements. The equipment used in I and II was modified to provide a more highly collimated primary beam, and more care was taken in aligning the beam and the crystals. Crystals of high perfection were selected for these runs, the width at half height of 00·16 ($2\theta = 137°$) was 0·14°; and of 33·0 ($2\theta = 152°$) was 0·27°. No corrections for refraction and Lorentz-polarization effects were made. The standard error of the a and c values in Table 3, estimated from the several runs, was of the order of 1 part in 20,000. The interatomic distances in the table were computed from these a and c values and the average z values of Table 1. The thermal contractions indicated in Table 3 are in good agreement with Erfling's (1939) single-crystal measurements in the temperature range covered by him.

This work was suuported in part by contract Nonr 2121(11) and by the National Science Foundation grant to the Low Temperature Laboratory of the University of Chicago. We are indebted to the Applied Science Laboratory of the University and also to Dr Glen Everett for the samples used.

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Acta Cryst. (1963). **16**, 453

A Device for Taking X-ray Photographs of Single Crystals at High Temperatures*

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(*Received* 6 *June* 1962)

The paper describes a device for taking single-crystal X-ray photographs at temperatures up to 1000 °C. The furnace mount fits on a standard single-crystal goniometer and is therefore interchangeable between oscillation, Weissenberg and precession diffractometers. A modification of a Weissenberg diffractometer to take it is described. No cooling of the film cassette is needed.

Introduction

Though techniques for taking high-temperature powder photographs are highly developed, comparatively little has been published on ways of obtaining high-temperature single-crystal photographs. We were interested in temperatures up to 900 °C. The design chosen (described in detail below) uses as a furnace a small platinum wire carrying a very heavy current, the whole device being mounted on standard goniometer arcs. Since a Weissenberg diffractometer can be used for taking oscillation and rotation photographs

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* Supported in part by the European Research Office, U.S. Department of the Army.