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The Crystal Structure of β-Protactinium Pentabromide

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Protactinium pentabromide crystallizes in two distinct modifications. A three-dimensional structure analysis of the β form (final $R=9\cdot05\%$) shows it to be essentially isostructural with uranium pentachloride. The molecular Pa_2Br_{10} units are bis-octahedral with symmetric bromine bridging (in contrast to U_2Cl_{10}), and are formed by the protactinium atoms occupying one fifth of the octahedral holes of a close-packed bromine lattice. The monoclinic unit cell of β-$PaBr_5$ has $a=8\cdot385$, $b=11\cdot205$, $c=8\cdot950$ Å and $\beta=91\cdot1$°. The structural relationship of β-$PaBr_5$ with other pentahalides and with other protactinium compounds is discussed.

Introduction

Protactinium pentabromide, $PaBr_5$, has previously been reported (Maddock, 1961) on the basis of X-ray powder diffraction data, to possess orthorhombic symmetry with the lattice parameters $a=7\cdot25$, $b=12\cdot12$ $c=9\cdot13$ Å. Recent work at A.E.R.E., Harwell (Brown 1966), again powder studies, indicated that $PaBr_5$ was possibly dimorphic but the powder results were not satisfactorily interpreted. The present single-crystal investigation was undertaken in an attempt to clarify this situation and in order to provide structural data on protactinium pentabromide to permit a comparison with the information then available for niobium pentachloride and pentabromide (Zalkin & Sands, 1958) and protactinium pentachloride (Dodge, Smith, Johnson & Elson, 1967).

Experimental

Protactinium pentabromide was prepared as described previously (Brown & Jones, 1966), and purified by vacuum sublimation at 400°. The compound is highly sensitive to atmospheric moisture, and single crystals were obtained by slow sublimation of small quantities (*ca.* 10 μg) of the product sealed in evacuated thin-walled silica capillaries. Investigation of the resulting crystals confirmed the existence of two forms of this compound. Sublimation at approximately 400–410° resulted in the formation of β-$PaBr_5$ whereas at approximately 390–400° a low temperature modification, α-$PaBr_5$, was obtained. Capillaries invariably contained entirely one modification, not an equilibrium mixture, and complete transformation, *e.g.* $\alpha\rightarrow\beta$, could be achieved by varying the sublimation conditions. Although we have been able to obtain some preliminary results for α-$PaBr_5$ this modification is more difficult to crystallize than β-$PaBr_5$ and crystals suitable for a full structure determination have not yet been prepared. Preliminary investigations indicate that α-$PaBr_5$ possesses monoclinic symmetry with $a=12\cdot69$, $b=12\cdot82$, $c=9\cdot92$ Å; $\beta=108$°, the space group being $P2_1/c$.

The results of oscillation, Weissenberg and precession studies have shown that β-$PaBr_5$ possesses monoclinic symmetry, space group $P2_1/n$ with reflexions systematically absent for $h0l$ with $h+l=2n+1$ and $0k0$ with $k=2n+1$. The unit-cell parameters are $a=8\cdot385$, $b=11\cdot205$, $c=8\cdot950$ Å; $\beta=91\cdot1$° (all with an accuracy of $\pm0\cdot3\%$); the calculated density for four $PaBr_5$ molecules per unit cell is 4·98 g.cm^{-3}. The non-standard setting of the space group, $P2_1/n$, rather than the standard $P2_1/c$, was chosen to bring out the

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relationship with a close-packed arrangement of bromines and for consistency with the reported structure of uranium pentachloride.

Three-dimensional intensity data were collected, using a crystal approximately 0·1 × 0·1 × 0·1 mm., by the precession and multi-film, equi-inclination Weissenberg techniques with zirconium-filtered molybdenum *Kα* radiation $\lambda = 0{\cdot}7107$ Å). The reciprocal lattice nets 0*kl* to 5*kl* (Weissenberg) and *hk*0 (precession) were surveyed and a total of 281 unique reflexions measured. The intensities, estimated visually, were placed on the same absolute scale by the use of common reflexions. The appropriate Lorentz-polarization factors were applied and a correction for X-ray absorption was made by means of a 512-point Gaussian integration following essentially the method of Busing & Levy (1957) programmed for the Mercury computer (Program written by A. J. Smith). The mass absorption coefficient of protactinium for molybdenum *Kα* radiation was taken to be 118 $\text{cm}^2.\text{g}^{-1}$ by a linear interpolation from the values published for thorium and uranium (Roof, 1959). The linear absorption coefficient for the crystal, μ, is thus 15·6 cm^{-1}.

Determination of the structure

The positions of four protactinium atoms per cell (one per asymmetric unit) were unambiguously derived from a three-dimensional Patterson synthesis; subsequent Fourier syntheses, based on protactinium phasing, clearly revealed an octahedral arrangement of bromine atoms about each protactinium atom. At this stage details of the crystal structure of uranium pentachloride, UCl_5, became available (Smith, Johnson & Elson, 1967) and a comparison of the reported unit cell, space group and heavy atom position with those for β-$PaBr_5$ suggested that the two compounds were isostructural. The published chlorine coordinates were therefore inserted for the bromine positions in β-$PaBr_5$ and least-squares refinement, initially by the block-diagonal approximation (using the program of J. S. Rollett) and finally by full-matrix with individual anisotropic vibrational parameters, and allowance for anomalous scattering (using the program General SFLS by N. A. Bailey) reduced the *R* index to the final value of 9·05%. $\Delta f'$ and $\Delta f''$ values for protactinium, −15·75 and +13·65 electrons respectively, were obtained by interpolation from the thorium and uranium values measured by Roof (1961). The scattering factors (f, $\Delta f'$ and $\Delta f''$) given in *International Tables for* X-*ray Crystallography* (1962) were used for bromine. The shifts obtained in the final refinement cycle were less than one quarter of the corresponding standard errors for all positional and vibrational parameters. The final parameters are listed in Table 1 together with their standard errors. Observed and calculated structure factors are quoted in Table 2.

Discussion of the structure

Each protactinium atom is coordinated octahedrally by six bromine atoms; two such octahedra share an edge to form a Pa_2Br_{10} unit (Fig. 1) which is situated about a crystallographic centre of inversion. This dimeric configuration is very similar to that found for UCl_5 but, whereas the latter posesses a distinctly assymmetric chlorine bridge, bond lengths being 2·67 and 2·70 Å ($\sigma = 0{\cdot}01$ Å), β-$PaBr_5$ has symmetric bromine bridging, the bond lengths of 2·85 and 2·86 Å being identical within one standard deviation ($\sigma = 0{\cdot}013$ Å). The protactinium atoms are displaced from the centres of the octahedra by approximately 0·2 Å in directions away from each other along the line joining Pa and Pa′. As a result of this the bridging bromine atoms Br(1) and Br(1′) approach more closely than they would for regular octahedral coordination and the bond angles within the distorted octahedra deviate considerably from 90°. In particular the Br(1)–Pa–Br(1′) angle is compressed to 81·1°. The Pa–Br bond lengths to non-bridging bromines range from 2·54 to 2·59 Å. Bond lengths are shown in Fig. 1 and angles are listed in Table 3, together with their standard deviations. Intra-

Table 1. *Atomic parameters in* β-$PaBr_5$

(Space group $P2_1/n$, all atoms in general positions, e.s.d.'s in parentheses)

Positional parameters

	$10^4\ x/a$	$10^4\ y/b$	$10^4\ z/c$
Pa	−525 (9)	1377 (6)	1624 (8)
Br(1)	298 (35)	1157 (17)	−1452 (33)
Br(2)	−1356 (41)	1285 (36)	4384 (22)
Br(3)	2494 (42)	1233 (44)	2312 (35)
Br(4)	−3378 (41)	1210 (38)	639 (25)
Br(5)	−448 (41)	3671 (23)	1393 (33)

Vibrational parameters ($\times 10^4$)

	b_{11}	b_{22}	b_{33}	b_{23}	b_{31}	b_{12}
Pa	121 (12)	69 (5)	95 (8)	42 (26)	−23 (14)	−34 (32)
Br(1)	207 (50)	89 (21)	127 (34)	11 (57)	−52 (65)	109 (74)
Br(2)	279 (54)	172 (33)	90 (38)	32 (66)	100 (62)	372 (99)
Br(3)	222 (42)	125 (28)	204 (53)	27 (74)	29 (77)	1 (77)
Br(4)	264 (52)	183 (39)	120 (40)	−81 (68)	50 (66)	−294 (95)
Br(5)	252 (43)	89 (17)	197 (45)	35 (78)	60 (64)	152 (100)

Table 2. $|F_{\mathrm{obs}}|$ *and* $|F_{\mathrm{calc}}|$ *for* β-$PaBr_5$

Lines marked by black dots give *l* and *k* in that order. The remaining lines give, from left to right, *h*, $|F_o|$, $|F_c|$.

• 0	0	
2	10120	11416
4	4409	5064
6	1223	1565
8	2118	2620
10	4146	3113
• 0	1	
1	12824	9636
2	9667	7244
3	5536	5481
4	10045	9121
6	3769	3668
7	1498	1307
• 0	2	
1	6470	4804
3	10411	10344
5	5536	4027
7	2868	2960
• 0	3	
1	10411	9659
2	4736	3491
3	5259	4936
4	5330	4845
7	2288	2504
• 0	4	
0	33874	33418
2	9511	9089
5	2594	2029
10	2995	2811
• 0	5	
1	5990	5043
2	6174	6848
4	6515	6323
6	3668	3909
• 0	6	
0	4575	3630
2	3668	4068
3	5670	5673
• 0	7	
1	5468	5758
3	4055	2611
• 0	8	
0	13055	14053
2	4493	4926
3	3867	3643
• 0	9	
2	4055	3934
4	5399	4882
• 0	10	
0	4236	2867
• 0	12	
0	5468	4168
• 1	0	
3	29951	32441
• 1	1	
−4	3458	2866
4	7488	7986
• 1	2	
−4	4409	4678
−2	12227	12652
4	3867	4002
• 1	3	
−4	4323	4023
−3	3117	3558
−1	4323	5150
0	4409	4473
1	3962	2960
4	6971	7850
5	2868	2938
• 1	4	
−5	3769	3796
−3	2868	2363
0	2868	2908
1	7877	8978
3	21178	24376
• 1	5	
−3	6114	6979
−1	8646	10019
0	4575	3812
1	6235	7049
2	3962	3553
4	3668	4600
• 1	6	
−2	5041	6158
0	4575	6229
1	2734	2311
• 1	7	
0	1933	2376
2	4323	4813
4	4736	5061
• 1	8	
−2	2995	3667
0	2995	3190
1	4493	4752
3	11240	11589
• 1	9	
−3	5536	4610
−1	4814	5992
1	4146	4347
• 2	0	
−2	6541	7490
4	7285	6953
• 2	1	
−5	4055	4696
−3	6470	5124
−1	5188	5071
3	4146	2995
4	5330	3769
5	3117	3155
• 2	2	
−5	4409	4972
−1	5735	5593
1	16062	12913
• 2	3	
−5	4967	4723
−3	6863	6439
−1	5800	5367
0	6353	5631
2	2868	2781
• 2	4	
−4	20203	18483
−2	6052	6220
0	5399	4789
1	4236	3329
4	5864	5515
• 2	5	
−5	3117	3158
−3	3458	3682
−2	3349	3694
0	9109	9041
2	7587	7607
4	4146	4023
5	1933	1820
• 2	6	
−5	3235	3215
3	5330	4899
• 2	7	
−5	3565	3447
−3	4891	4608
−1	2995	3549
• 2	8	
−4	10120	9368
−2	2995	3564
−1	2288	1743
0	2995	2762
1	4575	3648
• 2	9	
0	5188	5483
2	6412	5167
• 2	10	
1	2594	1792
• 3	0	
−3	8603	9051
−1	36682	34612
1	10902	8775
3	7488	6446
• 3	1	
−5	5468	6247
−3	6808	7181
−2	5188	5725
0	8646	9604
1	3668	3412
2	5800	5377
3	9109	8294
5	3235	3507
• 3	2	
−4	9068	10153
−2	7130	6989
2	5536	4859
• 3	3	
−5	3349	3309
−3	4146	4242
−2	6235	6056
0	8902	9477
2	5468	5726
3	3962	4701
• 3	4	
−3	7636	7270
−1	25590	25286
1	6641	7217
3	4236	4959
• 3	5	
−5	6470	6119
−3	6294	6539
0	4891	5297
1	4656	3825
2	3668	2509
3	6114	7498
• 3	6	
−4	7182	6180
1	3565	3738
• 3	7	
0	5259	5827
2	4146	3634
• 3	8	
−3	5259	4186
−1	11792	11349
1	3458	4142
• 3	9	
−3	4055	3818
• 3	12	
−1	4656	3537
• 4	0	
−4	2734	2762
0	11140	10022
2	24909	25446
4	7024	6928
• 4	1	
−5	2594	2031
−4	3867	4103
−2	6808	6850
0	6917	7336
1	3962	4303
3	9933	8329
5	4736	3553
• 4	2	
−1	12998	12168
1	5115	5364
5	3117	3250
• 4	3	
−5	2734	3372
−3	4891	4489
−2	3458	3075
0	4656	3856
1	4409	4871
3	8338	8226
5	3349	4321
• 4	4	
−4	2118	2102
−3	2594	2268
0	8064	7922
2	18012	19495
• 4	5	
−4	3867	3993
0	6753	6974
1	2594	2509
3	4409	4808
• 4	6	
−1	6971	6644
1	3117	2774
• 4	7	
−5	2868	2765
−3	3117	3234
1	3235	3555
3	5399	5169
• 4	8	
−3	2288	2466
−1	3349	3231
0	4055	4361
1	3458	3674
• 4	9	
0	4236	4206
• 5	0	
−5	11980	11308
−3	3668	3239
−1	3962	4088
3	6412	6457
5	11438	11293
• 5	1	
−4	4146	3770
−2	3117	3160
−1	5115	5174
0	3117	3156
1	4146	4314
3	4575	4537
• 5	2	
2	9589	8596
• 5	3	
−4	4575	3845
−2	3769	4173
0	3867	4247
3	2118	2692
• 5	4	
−5	8689	9304
−3	3117	2965
−1	2734	3404
0	1729	1904
3	4967	5374
4	4967	3489
5	10375	9535
• 5	5	
−1	4891	4877
1	5259	5455
3	5188	4566
• 5	6	
2	5330	5127
• 5	7	
0	2868	3046
• 5	8	
−5	5259	5252
−3	2118	1975
0	2118	2353
• 5	9	
1	4656	3977
• 6	0	
−4	4814	4916
−2	14259	16083
0	4055	4380
2	4891	4830
• 6	1	
−4	3962	4418
−3	2868	2644
−1	6114	5861
1	3668	3663
2	4409	4446
• 6	2	
−5	6412	6566
−3	5670	5526
5	3867	4395
• 6	3	
−4	2995	3050
−3	3349	2773
−1	5864	5802
1	4146	4441
2	3349	2802
• 6	4	
−4	5188	4176
−2	11106	12541
0	3769	3952
2	3769	3833
• 6	6	
−5	4656	4361
−3	3769	3276
• 6	7	
1	3235	3310
• 6	8	
0	2118	2593
• 7	0	
−1	4967	5801
1	13366	13110
• 7	1	
−3	3769	3480
−1	4409	4823
0	2445	2441
1	1498	1529
2	4967	5634
• 7	2	
−4	1729	1865
−2	7636	7903
0	4409	4957
• 7	3	
−4	3349	3487
−1	3458	2887
2	5115	5597
• 7	4	
−1	4323	4756
1	10589	10448
• 7	5	
−1	4409	4635
• 7	6	
0	2995	2911
• 8	0	
4	7537	6622
• 8	1	
2	3962	3422
• 8	2	
1	5735	5985
• 8	4	
4	5864	5664
• 8	6	
1	3962	3726
• 9	0	
−3	5800	5577
• 9	4	
−3	4656	4579
• 10	0	
0	4891	4963
• 10	4	
0	4736	4082

molecular Br···Br contacts range from 3·63 to 3·95 Å. A few of these, including Br(1)···Br(1′), 3·71 Å, are significantly shorter than expected van der Waals distances (3·90 Å; Pauling, 1960). A projection of the structure down the *c* axis is illustrated in Fig. 2.

The crystal structure as a whole, like that of UCl_5, is based on a cubic closest packing of the halogen atoms, with the protactinium atoms occupying one fifth of the octahedral holes in adjacent pairs. This arrangement has previously been illustrated for UCl_5 (Smith, Johnson & Elson, 1967) and will not be repeated here. As these authors point out, an idealized structure of this type requires a repeat distance along the *b* axis of $4\sqrt{2}.r$ (r = packing radius of bromine) with *a*- and *c*-axis repeat distances both equal to $2\sqrt{5}.r$. Numerical values obtained for $r = 1{\cdot}95$ Å (Pauling, 1960) are $a = c = 8{\cdot}72$ Å, $b = 11{\cdot}03$ Å, compared with the experimental values of $a = 8{\cdot}39$, $c = 8{\cdot}95$, and $b = 11{\cdot}20$ Å. The protactinium atoms obviously distort the packing in the *ac* plane and the observed β angle, 91·1 °, deviates slightly from 90°.

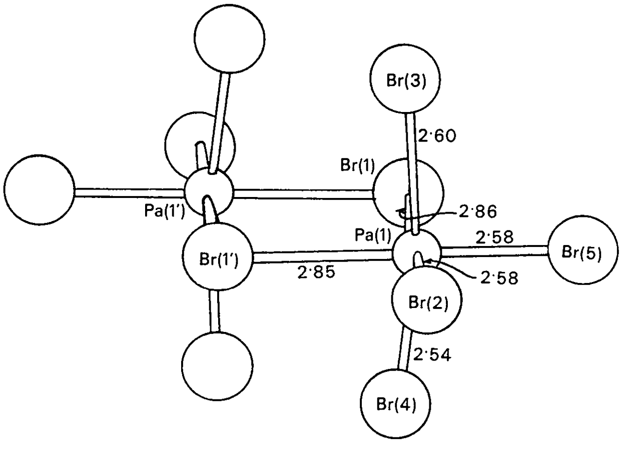

Fig. 1. The dimeric Pa_2Br_{10} unit.

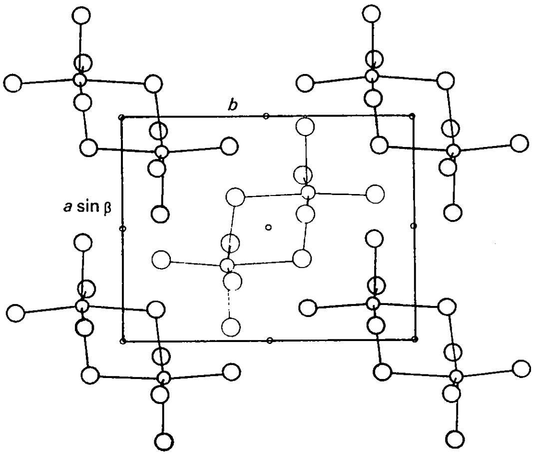

Fig. 2. A projection of the β-$PaBr_5$ structure along the *c* axis.

Table 3. *Bond lengths and angles for* β-$PaBr_5$

Bond lengths (e.s.ds in parentheses)

Pa(1)...Pa(1′)	4·341 (0·018) Å
Pa(1)...Br(1)	2·862 (0·013)
Pa(1)...Br(1′)	2·849 (0·013)
Pa(1)...Br(2)	2·581 (0·016)
Pa(1)...Br(3)	2·598 (0·018)
Pa(1)...Br(4)	2·541 (0·018)
Pa(1)...Br(5)	2·580 (0·018)

Angles (all e.s.ds. 0·5 °)

Br(1) -Pa(1)-Br(1′)	81·1 °
Br(1) -Pa(1)-Br(3)	88·3
Br(1) -Pa(1)-Br(4)	84·4
Br(1) -Pa(1)-Br(5)	90·1
Br(1′)-Pa(1)-Br(2)	91·8
Br(1′)-Pa(1)-Br(3)	83·4
Br(1′)-Pa(1)-Br(4)	88·4
Br(2) -Pa(1)-Br(3)	92·9
Br(2) -Pa(1)-Br(4)	93·4
Br(2) -Pa(1)-Br(5)	97·1
Br(3) -Pa(1)-Br(5)	93·2
Br(4) -Pa(1)-Br(5)	94·0
Pa(1) -Br(1)-Pa(1′)	98·9
Br(1) -Pa(1)-Br(2)	172·6
Br(1′)-Pa(1)-Br(5)	170·6
Br(3) -Pa(1)-Br(4)	169·8

The arrangement found for β-$PaBr_5$ and for UCl_5 differs from that observed for similar dimeric units of $NbCl_5$, $TaCl_5$ (Zalkin & Sands, 1958), and $MoCl_5$ (Sands & Zalkin, 1959) in which hexagonal closest packing of the chlorine atoms is reported with the metal atoms again occupying one fifth of the available octahedral holes. Protactinium pentachloride, $PaCl_5$, possesses a totally different structure (Dodge, Smith, Johnson & Elson, 1967) in which infinite chains of pentagonal bipyramids share edges, each protactinium atom being seven-coordinate. It will be interesting to see whether α-$PaBr_5$ follows the *d*-transition metal pentachlorides and shows hexagonal closest packing of the bromine atoms and also to search for both a possible low temperature form of $PaCl_5$, since the crystals used were grown by sublimation, and high temperature form of UCl_5 since the crystals of this compound were obtained from carbon tetrachloride solution.

The single-bond covalent radius of protactinium(V) derived from the six-coordinate β-$PaBr_5$ structure is $1{\cdot}44 \pm 0{\cdot}02$ Å, which can be compared with the mean single-bond values of $1{\cdot}45 \pm 0{\cdot}02$ Å and $1{\cdot}52 \pm 0{\cdot}02$Å for the seven-coordinate pentachloride (Dodge, Smith, Johnson & Elson, 1967) and nine-coordinate heptafluoro-complex, K_2PaF_7 (Brown, Kettle & Smith, 1967) respectively. The single-bond covalent radius of uranium (V) derived from the six-coordinate UCl_5 structure $1{\cdot}44 \pm 0{\cdot}01$ Å, is very close to these first two values.

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The Crystal Structure of *N*-Methyl-4-phenylisoxazolin-5-one

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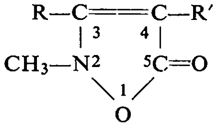
Crystals of *N*-methyl-4-phenylisoxazolin-5-one, $C_{10}H_9NO_2$, are monoclinic, $P2_1/c$, with $a=13{\cdot}716$, $b=10{\cdot}925$, $c=11{\cdot}333$ Å, $\beta=91{\cdot}51°$ and $Z=8$. X-ray analysis was based on layers 0–8 about the *b* axis. The structure was determined by trial-and-error methods and refined by an anisotropic least-squares analysis of 870 observed reflexions. The final *R* index including the contribution of hydrogen atoms was 0·053. The two independent molecules are arranged in the asymmetric unit according to a nearly orthorhombic symmetry. Bond distances and angles agree with accepted values. The phenyl and isoxazolinone rings of the two molecules are slightly tilted: the angles are 14° and 11°.

Introduction

Researches carried out (De Sarlo, Fabbrini & Renzi, 1966) on the series of aryl derivatives of 2-methylisoxazolin-5-one with the formula:



have established some differences in physical properties between the compounds with $R=C_6H_5$ and $R'=H$, CH_3, Br and those with $R=H$, CH_3 and $R'=C_6H_5$.

N-Methyl-4-arylisoxazolin-5-ones have higher melting points and lower solubilities in ether than the isomeric 3-aryl derivatives. Moreover, the $\nu_{C=O}$ in the infrared spectra is located before 1706 cm^{-1} for 4-aryl compounds and after 1730 cm^{-1} for all other *N*-methylisoxazolin-5-ones. It must be noted, however, that these differences are observed only in the solid phase, whereas comparable values are found in solution: *e.g.* the infrared spectra in carbon tetrachloride and the dipole moments in benzene solution. Analysis of the crystal structure of one compound for each of the two groups could account for these differences. In the present paper the crystal structure of *N*-methyl-4-phenylisoxazolin-5-one is described. In the following paper (Sabelli & Zanazzi, 1969) the crystal structure of *N*-methyl-3-phenyl-4-bromoisoxazolin-5-one is reported and the relations between the two types of compound are discussed.

Experimental

A sample of *N*-methyl-4-phenylisoxazolin-5-one was kindly supplied by Dr De Sarlo of the Istituto di Chimica Organica of the Florence University. Well formed, colourless crystals were obtained by recrystallization from an alcoholic solution.

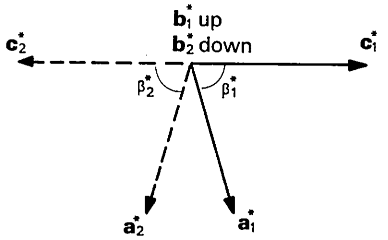


Fig. 1. Relationship between the axes in the twinned reciprocal lattices.