

Changes in Space and Laue Groups of Some Published Crystal Structures*

BY FRANK H. HERBSTEIN† AND RICHARD E. MARSH

Arthur Amos Noyes Laboratory of Chemical Physics, California Institute of Technology, Pasadena, California 91125, USA

(Received 15 June 1981; accepted 12 October 1981)

Abstract

It is noted that the crystal structures of eight compounds can almost surely be described in space groups having higher Laue symmetry than reported in the original papers. In three instances the lattice symmetry remains unchanged; there is one change from triclinic to monoclinic and four from monoclinic to rhombohedral. Of special significance are the structures of benzene. Cl₂ and benzene. Br₂, where the earlier monoclinic descriptions were not compatible with spectroscopic evidence; the evidence should now be re-examined in terms of the rhombohedral descriptions.

We report here some additional (see Schomaker & Marsh, 1979a; Marsh & Schomaker, 1979, 1980) examples of crystal structures which can be better described in space groups having higher Laue symmetry than originally reported. In three of the examples no change of crystal system is involved; there is one change from triclinic to *C*-centered monoclinic and four from *C*-centered monoclinic to rhombohedral.

Group I: no changes in crystal system

Methylene oxalate

This compound crystallizes in the tetragonal system with cell dimensions (at 153 K) $a = 6.124(4)$, $c = 10.381(8)$ Å (Kvick & Liminga, 1980; KL). The structure was described in space group $P4_1$ (Laue group $4/m$) and was refined to $R = 0.173$ for 393 reflections 'having positive net intensities'. The structure can probably be better described in space group $P4_12_12$, with Laue symmetry $4/mmm$.

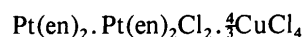
The intensity data, obtained as Supplementary Publication No. SUP 34887, comprise F_o , F_c , and $\sigma(F_o)$ values for 393 reflections. Of the reflections

* Contribution No. 6467 from the Arthur Amos Noyes Laboratory of Chemical Physics.

† On sabbatical leave from Technion – Israel Institute of Technology, Haifa, Israel.

which should be systematically extinguished because of the 2_1 axis in $P4_12_12$, only one, 070, is included in the table, with F_o approximately one-third as large as $\sigma(F_o)$. Included are 94 pairs of reflections hkl and khl which would be equivalent in the higher Laue symmetry $4/mmm$. The goodness-of-fit $\{\sum [\Delta F/\sigma(\Delta F)]^2/94\}^{1/2}$, where $\Delta F = |F_o(hkl) - F_o(khl)|$, is 0.73 for these 94 pairs. [Included is one outlying pair, 525 and 255, for which the deviation $\Delta F/\sigma(\Delta F)$ is 4.9.] Thus, these 94 pairs of reflections conform to the higher Laue symmetry considerably better than the assigned σ 's (which include a lack-of-confidence factor $0.025F$) would predict.

In Table 1 are listed the atomic coordinates symmetrized so as to correspond to $P4_12_12$ (this involves a shift in the origin by $\Delta z = -0.1312$). We include the shifts necessary to achieve the higher symmetry, and the corresponding standard deviations as obtained from Table 1 of KL. The goodness-of-fit relating the necessary shifts to their e.s.d.'s is 1.2.



The structure of this mixed-valence Wolfram's salt was described in the hexagonal space group $P6_3/m$ [$a = 15.657(2)$, $c = 10.522(7)$ Å] and was refined to an R of 0.070 for 990 reflections with $I > 2.58\sigma(I)$

Table 1. Atomic coordinates ($\times 10^4$ for C and O; $\times 10^3$ for H) for methylene oxalate, symmetrized and averaged so as to correspond to space group $P4_12_12$

Numbers in parentheses are standard deviations, as obtained from Table 1 of KL; numbers in square brackets are the shifts necessary to achieve the higher symmetry.

	<i>x</i>	<i>y</i>	<i>z</i>
O(1,3)	6572 (12) 10	3556 (15) 32	-1117 (13) 5
O(2,4)	9342 (11) 26	5856 (11) 12	-864 (*) 2
C(1,2)	8153 (14) 8	4126 (13) 4	-590 (13) 2
C(3)	11053 (16) 10	6053	0 (22) 6
H(1,2)	1119 (9) 15	768 (12) 23	34 (6) 1

* The z parameter for O(2) was held fixed by KL to specify the origin in the polar space group $P4_1$.

(Endres, Keller, Martin & Traeger, 1979). With one exception, the reported coordinates correspond to the higher-symmetry space group $P6_3/mmc$ within the reported e.s.d.'s. The exception is in the z coordinates of the N atoms, reported as $-0.016(2)$ and $-0.010(3)$; for the space group to be $P6_3/mmc$, these values should be equal in magnitude but opposite in sign.

In order to check further on the situation, we obtained the F table (Supplementary Publication No. SUP 34635). To our surprise, we found that the table comprised only those reflections with $h \geq k \geq 0$ and $l \geq 0$, i.e. only one-half the unique data for Laue group $6/m$. The authors report that the crystal was treated as monoclinic during data collection and subsequently 'the observations were reindexed to hexagonal symmetry and equivalent reflections were averaged'. Apparently this averaging was based on the higher Laue symmetry $6/mmm$, and hence, with the data available to us, we are unable to distinguish between $6/mmm$ and $6/m$.

Besides the higher Laue symmetry, space group $P6_3/mmc$ would require that reflections hhl be absent for l odd. Thirteen such reflections appear in the F table. They are all very weak; the largest is the 225 reflection which, at $F_o = 44.1$ (no e.s.d.'s are reported), would have an intensity less than 1% as large as the strongest reported reflection (600, at $F_o = 507.6$). The $P6_3/m$ structure does little to explain the intensities of these reflections: the largest $|F_c|$ value for them is 15.5, and the R index for the 13 reflections is 0.76.

While we think it probable that the structure can be satisfactorily described in $P6_3/mmc$, a definitive decision is not possible with the data at hand.

La[Fe(CN)₆].4H₂O

This structure was described in the hexagonal space group $P6_3/m$ with $a = 7.541(3)$, $c = 13.955(9)$ Å, $Z = 2$ (Mullica, Milligan & Garner, 1980; MMG), and was refined to $R = 0.068$ for 190 reflections. The reported coordinates correspond closely to the higher-symmetry space group $P6_3/mmc$; indeed, none of the parameters needs to be shifted by more than 0.8σ to attain the $P6_3/mmc$ description in Table 2. Since the agreement with the higher symmetry is so good, we have not obtained the original structure factors to

Table 2. Atomic coordinates for La[Fe(CN)₆].4H₂O; $P6_3/mmc$ description

	Position	x	y	z
La	2(c)	$\frac{1}{3}$	$\frac{2}{3}$	$\frac{1}{4}$
Fe	2(a)	0	0	0
C	12(k)	0.120	2x	0.079
N	12(k)	0.196	\bar{x}	0.119
O(1)	4(f)	$\frac{1}{3}$	$\frac{2}{3}$	0.904
O(2)	6(h)	0.525	\bar{x}	$\frac{1}{4}$

verify the Laue symmetry and the additional absences (hhl with l odd).

Crystals of La[Fe(CN)₆].4H₂O were formed by the controlled dehydration of the pentahydrate, the transformation involving the change in color from red-orange to red-purple (MMG). One possibility was that the transformation was from the $P6_3/m$ structure of the pentahydrate to an orthorhombic structure found by Hulliger, Landolt & Vetsch (1976) for related cobalt compounds; however, MMG concluded that the loss of water led to only small structural changes within the same space group. It now appears that the transformation is well defined, and involves a change from one hexagonal structure to another hexagonal structure of higher Laue symmetry.

Group II: changes in crystal system

Bis(oxamide oximate)nickel(II)-oxamide oxime

The structure of this compound was described in the triclinic space group $P\bar{1}$, with two formula units in a cell of dimensions $a = 4.982(1)$, $b = 12.133(3)$, $c = 12.871(4)$ Å, $\alpha = 67.67(2)$, $\beta = 78.84(2)$, $\gamma = 78.14(2)^\circ$, and $Z = 2$ (Endres, Jannack & Prickner, 1980; EJP). The two bis(oxamide oximate)nickel molecules are crystallographically independent and lie on separate centers of symmetry at $(0,0,0)$ and $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$; similarly, the free-ligand oxamide oxime molecules lie on other independent centers at $(0, \frac{1}{2}, 0)$ and $(0, 0, \frac{1}{2})$. The final R was 0.050 for 2055 reflections above 3.0σ .

The structure can be better described in the monoclinic space group $C2/c$. The base vectors $[\bar{1}20]$, $[\bar{1}00]$, and $[0\bar{1}1]$ describe a C -centered cell with dimensions $a' = 23.748$, $b' = 4.982$, $c' = 13.936$ Å, $\alpha' = 89.99$, $\beta' = 122.08$, $\gamma' = 90.01^\circ$, $Z = 4$; the corresponding coordinate transformations $x' = \frac{1}{2}(y + z)$, $y' = -x - \frac{1}{2}(y + z)$, $z' = z$, followed by a change in origin to an alternative center of symmetry at $(-\frac{1}{4}, -\frac{1}{4}, 0)$, result in atomic positions that correspond to space group $C2/c$ within the experimental e.s.d.'s. In this description, all four nickel coordination-complex molecules are crystallographically equivalent, as are the four free oxamide oxime molecules.

Atomic parameters corresponding to space group $C2/c$ are listed in Table 3; they were symmetrized by averaging the transformed coordinates of equivalent atoms in the two pairs of molecular species. Also listed are the deviations of the individual atoms from the symmetrized positions. These deviations have an r.m.s. value of 0.005 Å for the C, N, and O atoms, to be compared with an experimental e.s.d. of about 0.004 Å (EJP) for the averaged coordinates of two atoms; for the H atoms, the numbers are 0.05 Å vs 0.07 Å.

The c glide plane in space group $C2/c$ should be manifested in the absence of reflections of the type hhl

Table 3. *Atomic coordinates* ($\times 10^4$ for Ni, O, N, and C; $\times 10^3$ for H) *for the C2/c description of bis(oxamide oximato)nickel(II)-oxamide oxime*

Numbers in square brackets are the deviations of the individual atoms from the averaged, symmetrized positions.

	Position	x	y	z
Ni(1,2)	4(c)	2500	2500	0
O(1,4)	8(f)	3696 [4]	2893 [8]	2218 [6]
O(2,3)	8(f)	2104 [4]	5790 [2]	-1984 [4]
N(1,6)	8(f)	3327 [3]	3856 [4]	1135 [1]
N(2,5)	8(f)	2576 [0]	5158 [2]	-860 [2]
N(3,8)	8(f)	4152 [7]	7062 [9]	1550 [7]
N(4,7)	8(f)	3197 [3]	8884 [6]	-808 [3]
C(1,4)	8(f)	3552 [1]	5879 [1]	852 [4]
C(2,3)	8(f)	3093 [2]	6714 [5]	-344 [2]
O(5,6)	8(f)	4021 [0]	4264 [10]	-916 [1]
N(9,11)	8(f)	4407 [4]	2202 [4]	-994 [4]
N(10,12)	8(f)	4812 [5]	1803 [8]	963 [4]
C(5,6)	8(f)	4789 [4]	1125 [10]	-23 [1]
H(1,6)	8(f)	175 [1]	454 [13]	-214 [4]
H(2,10)	8(f)	426 [0]	858 [16]	140 [2]
H(3,9)	8(f)	430 [0]	686 [7]	230 [2]
H(4,8)	8(f)	354 [6]	980 [10]	-54 [6]
H(5,7)	8(f)	294 [0]	931 [0]	-151 [4]
H(11,14)	8(f)	384 [2]	500 [13]	-157 [3]
H(12,15)	8(f)	522 [9]	141 [3]	161 [11]
H(13,16)	8(f)	465 [2]	339 [11]	96 [5]

with l odd in the triclinic indexing. We have not attempted to verify these absences.

Hexaaziridinylcyclotri(phosphazene) tris(carbon tetrachloride), N₃P₃(NC₂H₄)₆.3CCl₄

The structure of this compound was described as monoclinic, space group $C2/c$, with $a = 15.609$ (5), $b = 18.748$ (4), $c = 12.208$ (6) Å, $\beta = 106.40$ (3)°, $Z = 4$ (Galy, Enjalbert & Labarre, 1980; GEL).

The vectors $[\frac{1}{2}, -\frac{1}{2}, 0]$, $[\frac{1}{2}, \frac{1}{2}, 0]$, $[001]$ describe a unit cell with $a' = b' = 12.198$, $c' = 12.208$ Å, $\alpha' = \beta' = 100.41$, $\gamma' = 100.44$ °, $Z = 2$. The corresponding coordinate transformation $x' = \frac{1}{2} + x - y$, $y' = x + y$, $z' = z$ leads to a set of atomic positions that are compatible, well within the reported e.s.d.'s, with the symmetry of the rhombohedral space group $R\bar{3}c$ (see Table 4). (The increment of $\frac{1}{2}$ in x' is necessary to place the origin at the proper center of inversion.) Since the c glide plane is common to both space groups, the systematic absences are unchanged.

GEL noted that the 'crystal shapes were close to perfect hexagonal prisms more or less elongated', and that preliminary Weissenberg photographs 'allowed the assignment of a hexagonal cell to the crystal'. However, their study with a Stoe reciprocal-lattice explorer camera convinced GEL that the crystals were in fact monoclinic. Transformation of our rhombohedral cell to hexagonal axes gives a triply-primitive cell with $a = 18.75$, $c = 16.88$ Å, the same hexagonal cell as reported by GEL, and there can be no reasonable

doubt that the structure should be described in $R\bar{3}c$ rather than in $C2/c$.

Mo₃(OAc)₆(OCH₃)₂(H₂O)₃(CF₃SO₃)₂

Dr M. Kaftory has drawn our attention to this compound which was originally described (Bino, Ardon, Maor, Kaftory & Dori, 1976) as monoclinic, space group $C2/c$, with $a = 20.083$ (5), $b = 11.637$ (5), $c = 15.210$ (5) Å, $\beta = 116.3$ (5)°, $Z = 4$ [the chemical formula and cell dimensions have been corrected (Kaftory, private communication) since the original publication]. More recently, Kaftory has redetermined the structure using a Philips PW1100 four-circle diffractometer; he found a rhombohedral unit cell with $a = 15.215$ Å, $\alpha = 45.25$ °, and carried out a successful refinement in space group $R\bar{3}c$. A report of this improved refinement has not yet been published. We note that the vectors $[\frac{1}{2}, -\frac{1}{2}, 1]$, $[\frac{1}{2}, \frac{1}{2}, 1]$, and $[0, 0, 1]$ of the monoclinic cell define a rhombohedral unit cell with dimensions $a' = b' = 15.188$, $c' = 15.210$ Å, $\alpha' = \beta' = 44.89$, $\gamma' = 45.05$ °, $Z = 2$; the averaged rhombohedral cell ($a_R = 15.195$ Å, $\alpha_R = 44.94$ °) agrees well with the diffractometer results.

Benzene.Cl₂ and benzene.Br₂

Benzene.Cl₂ (m. p. 233 K) and benzene.Br₂ (m. p. 259 K) were reported to be isomorphous (monoclinic, space group $C2/m$). The structure of the Cl₂ compound was described in detail by Hassel & Strømme (1959; HS) and that of the Br₂ compound more briefly (Hassel & Strømme, 1958). These compounds have also been investigated by IR and Raman spectroscopy, with results that do not agree with the symmetry deduced by HS.

HS report cell dimensions for benzene.Cl₂ at 183 K as $a = 7.41$, $b = 8.61$, $c = 5.65$ Å, $\beta = 99.5$ °, $Z = 2$ (no e.s.d.'s were reported). The vectors $[\frac{1}{2}, -\frac{1}{2}, 0]$, $[\frac{1}{2}, \frac{1}{2}, 0]$, $[0, 0, 1]$ describe a cell with $a' = b' = 5.68$, $c' = 5.65$ Å, $\alpha' = \beta' = 96.2$, $\gamma' = 98.6$ °. While these latter values -

Table 4. *Atomic coordinates for N₃P₃(NC₂H₄)₆.3CCl₄, symmetrized and averaged so as to correspond to space group $R\bar{3}c$*

Numbers in parentheses are standard deviations estimated from Table 2 of GEL; numbers in square brackets are r.m.s. deviations of the individual atoms from the symmetrized positions.

	Position	x	y	z
P(1,2)	6(e)	0.33611 (5) [1]	0.16389	0.25000 (-) [2]
N(1,2)	6(e)	0.1667 (1) [1]	0.3333	0.2500 (-) [1]
N(3,4,5)	12(f)	0.1789 (2) [2]	0.3168 (2) [2]	0.0284 (2) [1]
C(1,4,5)	12(f)	0.0670 (2) [1]	0.2373 (3) [1]	-0.0078 (2) [1]
C(2,3,6)	12(f)	0.0743 (2) [1]	0.3605 (2) [1]	0.0072 (2) [3]
C(7,8)	6(e)	0.6393 (2) [0]	-0.1393	0.2500 (-) [2]
Cl(1,5,6)	12(f)	0.6755 (1) [1]	0.0063 (1) [1]	0.2481 (1) [0]
Cl(2,3,4)	12(f)	0.6685 (1) [0]	-0.2179 (1) [0]	0.1291 (1) [0]

Table 5. Final parameters for benzene.Cl₂, space group $R\bar{3}m$ (hexagonal setting)

The anisotropic Gaussian has the form $\exp(10^3)(-2\pi^2)(U_{11}h^2a^{*2} + \dots + U_{23}klb^*c^*)$.

	Position	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl	6(c)	0	0	0.1177 (7)	103 (3)	U_{11}	41 (4)	$\frac{1}{2}U_{11}$	0	0
C	18(g)	0	0.158 (2)	$\frac{1}{2}$	113 (6)	70 (10)	85 (15)	$\frac{1}{2}U_{11}$	-18 (6)	$\frac{1}{2}U_{13}$

especially the angles — are appreciably unequal, convincing proof of rhombohedral symmetry comes from the atomic coordinates (Table 1, HS): after the transformations $x' = x - y$, $y' = x + y$, $z' = z$, the coordinates conform to space group $R\bar{3}m$ within 0.04 Å for C and exactly for Cl.

HS include a listing of 75 F_o values (plus 23 'less thans') comprising 67 independent reflections in $C2/m$. In $R\bar{3}m$, there are 11 pairs of equivalent reflections; the agreement factor $\sum |F_1 - F_2|/\bar{F}$ for these 11 pairs is 0.093, which is slightly better than the agreement among the $C2/m$ -equivalent pairs. After averaging of equivalent reflections, there are 56 independent observations in $R\bar{3}m$. We have carried out a least-squares refinement of the coordinates and anisotropic U 's for Cl and C, and obtained an R of 0.089, somewhat better than the 0.104 obtained by HF in $C2/m$ (they used separate isotropic B 's for each of the three zones of data they collected). The quantity minimized was $\sum w(F_o^2 - F_c^2)^2$, with weights $w = 1/F_o^4$ for $F_o \geq 12.0$ and $w = 1/144F_o^2$ for $F_o \leq 12.0$. Our final parameters, relative to hexagonal axes ($a_H = 8.49$, $c_H = 8.54$ Å), are given in Table 5. Bond distances are Cl-Cl = 2.01 (1) Å, C-C = 1.34 (2) Å; the distance from Cl to the center of the benzene ring is 3.26 (1) Å. All these values agree, within their e.s.d.'s, with the average values calculated from the $C2/m$ parameters of HS. (The e.s.d.'s do not include allowance for cell-dimension errors, which are unknown but certainly not negligible.)

The $C2/m$ cell dimensions reported for benzene.Br₂ ($a = 7.75$, $b = 8.83$, $c = 5.95$ Å, $\beta = 99.3^\circ$; Hassel & Strømme, 1958) lead to the rhombohedral cell dimensions $a' = b' = 5.87$, $c' = 5.95$ Å, $\alpha' = \beta' = 96.1$, $\gamma' = 97.5^\circ$. Neither atomic parameters nor F_o values were reported.

The initial IR study of these systems was by Person, Cook & Friedrich (1967), who studied benzene.Br₂ at 77 K. Their results were inconsistent with the site symmetry C_{2h} required by the $C2/m$ model of HS, and they concluded that the more likely site symmetry was C_{3v} ; they also noted that the crystal structure 'is very close to one with a rhombohedral unit cell ... with benzene and bromine molecules both located on sites of C_{3v} symmetry'. Subsequently, Brown & Person (1977) re-examined the assignment of IR bands and concluded that, while C_{2h} site symmetry could still be ruled out, 'positive identification of the site symmetry of the benzene as C_{3v} is weakened considerably'.

The low-temperature (77 K) Raman spectra of benzene.Cl₂ and benzene.Br₂ have been reported by Anthonsen & Møller (1977), Abe & Ito (1978), and Bobrov, Kimel'fel'd, Mostovaya & Mostovoi (1978). Anthonsen & Møller deduced that the space group was $C2$ or Cm , rather than $C2/m$, and that the benzene molecules did not lie on symmetry centers. Abe & Ito decided that the highest site symmetry of benzene compatible with the Raman spectrum was $\bar{3}m$ (D_{3h}); however, after considering the IR results of Person and co-workers, they concluded that 'it is more reasonable to assume that the site symmetry of benzene is C_{3v} rather than D_{3h} '.

The spectroscopic studies relevant here were made on crystalline samples held at 77 K whereas the diffraction studies were made at considerably higher temperatures. However, NQR spectra measured over large temperature ranges for both compounds (Kadaba, O'Reilly, Peterson & Scheie, 1971; Gordeev, Grechishkin, Kyuntsel & Rozenberg, 1970) show no evidence of a phase transition at any intermediate temperature. Thus, we have no reason to doubt that the spectroscopic and the diffraction studies were made on the same phases. Further consideration of the spectroscopic interpretations seems desirable in light of our revision of the crystallographic results.

Concluding remarks

As a result of the suggestions of two referees we append the following comments:

(1) In all but one (Endres *et al.*, 1979) of these cases the reported structures conform to the higher Laue symmetries within the reported e.s.d.'s. There can be little doubt, then, that the primary data — the diffraction intensities — must also conform to the higher symmetry, and we have not always felt it necessary to inspect the F tables, even to check for systematic absences. Serious violations of these absences, or of the higher Laue symmetry, would necessarily be reflected in a major perturbation of the reported structure and minor violations could always be blamed on experimental problems of one sort or another. Whatever the result of such an inspection, we are confident that our conclusion would remain unchanged: that the structures as reported can, and should, be described in the higher symmetries.

(2) In all instances our suspicions that the structures could be described in higher-symmetry space groups were aroused by inspection of the tables of atomic coordinates, rather than the unit-cell dimensions (see also Marsh & Schomaker, 1979). While a systematic search for a reduced unit cell (see, for example, Azaroff & Buerger, 1958)* might suggest an alternative lattice type, it would not be of help in determining the correct Laue symmetry within the lattice type. Moreover, in all cases it is the symmetry of the structure rather than the shape of the unit cell that is crucial, and evaluation of the symmetry is best achieved by examining directly the atomic parameters. We know of no practical way of programming a computer to carry out this examination.

Finally, we emphasize that in these eight instances of structures which can be described in space groups of higher Laue symmetry than originally reported, there should have been no problem of singularity or near-singularity in the original refinement procedures (Schomaker & Marsh, 1979b). Accordingly, the original descriptions of the molecular geometries should remain valid within the reported e.s.d.'s; all that is changed is that exact symmetry elements, as required by the higher crystallographic symmetry, replace approximate ones. On the other hand, near-singularities and ensuing molecular distortions *will* arise when a center of symmetry has gone unrecognized. Examples of this latter sort of problem in the recent literature number far more than eight, and we are making only slow progress in cataloguing them.

We thank A. Zewail (CIT) and M. Kaftory (Technion) for helpful discussions. This investigation was supported in part by Public Health Service Research Grant No. GM 16966 from the National Institute of General Medical Sciences, National Institutes of Health.

* A convenient method to search for a reduced cell is to use ORTEP (Johnson, 1965) or another suitable program to obtain distances from the cell origin to surrounding lattice points, and the angles between these vectors.

References

- ABE, H. & ITO, M. (1978). *J. Raman Spectrosc.* **7**, 35–40.
- ANTHONSEN, J. W. & MØLLER, C. K. (1977). *Spectrochim. Acta Part A*, **33**, 987–993.
- AZAROFF, L. V. & BUEGER, M. J. (1958). *The Powder Method in X-ray Crystallography*, Ch. 11. New York: McGraw-Hill.
- BINO, A., ARDON, M., MAOR, I., KAFTORY, M. & DORI, Z. (1976). *J. Am. Chem. Soc.* **98**, 7093–7095.
- BOBROV, A. V., KIMEL'FEL'D, YA. M., MOSTOVAYA, L. M. & MOSTOVOI, A. B. (1978). *J. Raman Spectrosc.* **7**, 88–89.
- BROWN, K. G. & PERSON, W. B. (1977). *J. Chem. Phys.* **66**, 876–877.
- ENDRES, H., JANNACK, T. & PRICKNER, B. (1980). *Acta Cryst.* **B36**, 2230–2233.
- ENDRES, H., KELLER, H. J., MARTIN, R. & TRAEGER, U. (1979). *Acta Cryst.* **B35**, 2880–2882.
- GALY, J., ENJALBERT, R. & LABARRE, J.-F. (1980). *Acta Cryst.* **B36**, 392–397.
- GORDEEV, A. D., GRECHISHKIN, V. S., KYUNTSSEL, I. A. & ROZENBERG, YU. I. (1970). *J. Struct. Chem. USSR*, **11**, 717–719.
- HASSEL, O. & STRØMME, K. (1958). *Acta Chem. Scand.* **12**, 1146–1147.
- HASSEL, O. & STRØMME, K. (1959). *Acta Chem. Scand.* **13**, 1781–1786.
- HULLIGER, F., LANDOLT, M. & VETSCH, H. (1976). *J. Solid State Chem.* **18**, 307–312.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.
- KADABA, P. K., O'REILLY, D. E., PETERSON, E. M. & SCHEIE, C. E. (1971). *J. Chem. Phys.* **55**, 5289–5291.
- KVICK, Å. & LIMINGA, R. (1980). *Acta Cryst.* **B36**, 734–736.
- MARSH, R. E. & SCHOMAKER, V. (1979). *Inorg. Chem.* **18**, 2331–2336.
- MARSH, R. E. & SCHOMAKER, V. (1980). *Acta Cryst.* **B36**, 219–220.
- MULLICA, D. F., MILLIGAN, W. O. & GARNER, R. L. (1980). *Acta Cryst.* **B36**, 2561–2564.
- PERSON, W. B., COOK, C. F. & FRIEDRICH, H. B. (1967). *J. Chem. Phys.* **46**, 2521–2532.
- SCHOMAKER, V. & MARSH, R. E. (1979a). *Acta Cryst.* **B35**, 1094–1099.
- SCHOMAKER, V. & MARSH, R. E. (1979b). *Acta Cryst.* **B35**, 1933–1934.