SHORT COMMUNICATIONS

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Structure of $MgCl_2.RbCl.6H_2O$. Corrigendum.[*]

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Abstract

The structure of $MgCl_2.RbCl.6H_2O$ was described and refined [Waizumi, Masuda, Ohtaki, Burkov & Scripkin (1991). *Acta Cryst.* C47, 251–254] in space group *P*1; it is properly described in *C*2/*c* ($R = 0.072$, $S = 1.74$ *vs* 0.078 and 1.92 previously). Revised coordinates and bond lengths are given; they lead to regular octahedral coordination about Mg, with Mg—O = 2.043 (2) Å, whereas the *P*1 model resulted in a large apparent spread of Mg—O distances, from 1.982 (12) to 2.115 (13) Å. The same change in space group – from *P*1 [Nakayasu, Suzukawa & Kobayashi (1983). *Denki Kagaku*, **51**, 419–422] to *C*2/*c* – is presumably applicable to the ammonium compound, $MgCl_2.NH_4Cl.6H_2O$, where once again the MgO_6 octahedron becomes regular [Mg—O = 2.053 (3) Å] rather than distorted.

Recently, the crystal structure of the double salt $MgCl_2.RbCl.6H_2O$ ('rubidium carnallite') was reported (Waizumi, Masuda, Ohtaki, Burkov & Scripkin 1991; hereinafter, WMOBS) as triclinic, space group *P*1, with $a = 6.672$ (5), $b = 13.282$ (15), $c = 6.639$ (5) Å, $\alpha = 89.83$ (8), $\beta = 91.72$ (6), $\gamma = 90.41$ (8)°, $Z = 2$. The crystals – and those of the isomorphous compound $MgCl_2.NH_4Cl.6H_2O$ – had originally been reported as monoclinic (Andress & Saffe, 1939); however, WMOBS chose the triclinic representation on the basis of a reinvestigation of the NH_4 salt by Nakayasu, Suzukawa & Kobayashi (1983). In fact, both compounds should be described as monoclinic, space group *C*2/*c*.

For the rubidium salt, the vectors [101], [$10\bar{1}$], [010] describe a *C*-centered cell with $a = 9.270$, $b = 9.553$, $c = 13.282$ Å, $\alpha = 90.40$, $\beta = 90.17$, $\gamma = 89.72°$, $Z = 4$. A listing of 2604 F_o [with $F_o > 3\sigma(F_o)$; WMOBS] and $\sigma^2(F_o)$ values was obtained from SUP 53400. When the indices were transformed and the F_o's averaged according to Laue symmetry 2/*m*, the value of $R_{\rm merge}$ was 0.063 for 1156 pairs of equivalent reflections and the value of '$\rm GOF_{merge}$' – that is, the r.m.s. value of the 1156 individual GOF's of averaging – was 1.79; the total resulting number of independent reflections was 1447. Of these, nine were of the type *h*0*l* with *l* odd, expected to be missing because of the *c*-glide plane in *C*2/*c*; seven of the nine showed $F_{\rm obs}$ values less than $5\sigma(F_o)$, while the 001 reflection was at 8.5σ, perhaps because of interference by the beam stop, and 203, though weak, was at 12.6σ for no apparent reason. Starting coordinates were from Table 1, WMOBS, again suitably transformed and averaged. Full-matrix minimization of the quantity $\sum w(F_o^2 - F_c^2)^2$, with $w = [4F_o^2\sigma^2(F_o) + 0.14F_o^4]^{-1}$ (see WMOBS), converged at $R = 0.072$ and GOF = 1.74 for 1438 reflections and 55 parameters. Included among the parameters was an isotropic extinction coefficient [final value, 2.8 (2) × 10^{-6}]; the H atoms were placed in assumed positions along the O···Cl lines, 0.85 Å from the O atoms. For the *P*1 structure, WMOBS report $R = 0.078$ and GOF = 1.92 for, apparently, 268 parameters (including coordinates of the H atoms, some of which were unreasonable in terms of O—H···Cl hydrogen bonds). The somewhat high residuals for both models probably result from absorption or other experimental errors: the most serious discrepancies $|F_o - F_c|$ seem to involve strong low-order reflections, especially the 00*l*'s, and the major features of a final difference map (reaching 2.5 e Å^{-3}) formed clover leaves around Rb and Cl. Such errors may also be responsible for the relatively large e.s.d.'s in the cell angles.

The final *C*2/*c* parameters are in Table 1, and some interatomic distances and angles in Table 2.[*] A noteworthy feature is that the three independent Mg—O distances are equal, well within experimental uncertainty. This is in contrast to the earlier results, where the apparent variations in the Mg—O bond lengths, from 1.982 (14) to 2.115 (13) Å, prompted WMOBS to classify them into two groups. This unusual spread of distances in the *P*1 model undoubtedly resulted from the refinement of a centrosymmetric structure in a non-centrosymmetric space group (Ermer & Dunitz, 1970; Schomaker & Marsh, 1979).

The structure of ammonium carnallite, $MgCl_2.NH_4Cl.6H_2O$, which was also described in *P*1 (Nakayasu *et al.*, 1983), can similarly be described in *C*2/*c*. In this case the transformation matrix is [101], [$10\bar{1}$] and [$0\bar{1}0$], leading to a unit cell with $a = 9.300$, $b = 9.550$, $c =$

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[*] Contribution No. 8427.

[*] Lists of structure factors, anisotropic U_{ij}'s and assumed hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54426 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Rubidium carnallite: final coordinates, $C2/c$ refinement; x, y, z and U_{eq} × 10^4.*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} (Å^2)
Rb	0	43 (.7)	2500	344 (2)
Mg	5000	0	5000	194 (3)
Cl(1)	2481 (1)	2621 (1)	2532 (1)	306 (2)
Cl(2)	0	0	0	314 (3)
O(1)	4098 (5)	135 (3)	3600 (3)	383 (8)
O(2)	3197 (4)	1008 (4)	512 (3)	374 (9)
O(3)	902 (4)	3125 (3)	4620 (3)	364 (8)

Table 2. *Rubidium carnallite: some distances and angles (Å, °), $C2/c$ refinement*

Rb—Cl(1)	3.369 (1)	O(1)···Cl(1)	3.144 (4)
Rb—Cl(1)	3.288 (1)	O(1)···Cl(1)	3.187 (4)
Rb—Cl(2)	3.321 (1)	O(2)···Cl(1)	3.166 (4)
Mg—O(1)	2.041 (4)	O(2)···Cl(2)	3.187 (4)
Mg—O(2)	2.047 (4)	O(3)···Cl(1)	3.177 (4)
Mg—O(3)	2.041 (3)	O(3)···Cl(2)	3.141 (3)

O(1)—Mg—O(2)	90.02 (14)	Cl(1)···O(1)···Cl(1)	97.98 (10)
O(1)—Mg—O(3)	90.16 (14)	Cl(1)···O(2)···Cl(2)	97.51 (10)
O(2)—Mg—O(3)	90.28 (14)	Cl(1)···O(3)···Cl(2)	96.88 (9)
O(1)—Mg—O(1)	180.0		
O(2)—Mg—O(2)	180.0		
O(3)—Mg—O(3)	180.0		

Table 3. *Ammonium carnallite: coordinates, space group $C2/c$;* x, y, z × 10^4*

	x	y	z
NH_4	0	16	2500
Mg	5000	0	5000
Cl(1)	2456	2620	2536
Cl(2)	0	0	0
O(1)	4084	138	3601
O(2)	3185	1004	515
O(3)	898	3114	4622

* These values were obtained by symmetrizing and averaging the $P1$ coordinates in Table 2, Nakaya *et al.* (1983).

13.301 Å, $\alpha = 89.95$, $\beta = 90.06$, $\gamma = 90.00°$. The resulting $C2/c$ coordinates are given in Table 3. Once again they lead to equal Mg—O distances, at 2.053 (3) Å, in contrast to the rather large variation 1.96 (1) – 2.14 (1) Å reported earlier.

A couple of final comments might be in order. First, the monoclinic unit cells for the Rb and NH_4 carnallites reported by Andress & Saffe (1939) – $a = 13.30$, $b \simeq c \simeq 6.65$ Å, $\beta \simeq 90°$ – correspond to a primitive cell rather than to the C-centered cell described here and the axis of symmetry was mislocated. This improper cell probably resulted from the strong pseudosymmetry: reflections with l odd are systematically weak, and the Mg, Cl and Rb (or NH_4) atoms alone describe an effectively tetragonal structure. Secondly, in the case of the Rb compound, the rather large deviations of the cell angles α and γ from 90°, plus the appearance of the 203 reflection with seemingly significant intensity, might cast doubt on the assignment of the monoclinic space group and Laue symmetry. However, there is a far larger body of evidence – the satisfactory agreement indexes for averaging in Laue group $2/m$, and the improved values of the final R and (most important) the over-all goodness-of-fit – that argues strongly in favor of the revised description. And the overwhelming disadvantage of attempting refinement in $P1$ is clearly shown in the wide range of apparent Mg—O distances when, in fact, the experimental data seem perfectly consistent with regular coordination.

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Structures de trois pyrroles substitués dérivés de munchnones. Erratum.

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Abstract

The *Abstract* of the paper by Toupet, Mazari, Texier & Carrié [*Acta Cryst.* (1991), C**47**, 1528–1531] is corrected. Methyl 2-methoxyphenyl-l-methyl-4,5-diphenylpyrrole-3-carboxylate (I), $C_{26}H_{23}NO_3$, $M_r = 397.5$, triclinic, $P\bar{1}$, $a = 10.525$ (2), $b = 10.719$ (1), $c = 10.965$ (1) Å, $\alpha = 71.84$ (1), $\beta = 82.09$ (1), $\gamma = 62.01$ (2)°, $V = 1038$ (1) Å^3, $Z = 2$, $D_x = 1.27$ g cm^{-3}, $\mu = 0.77$ cm^{-1}, $T = 293$ K, $F(000) = 420$, $wR = 0.033$ for 2612 observations. Methyl 5-methoxyphenyl-1-methyl-2,4-diphenylpyrrole-3-carboxylate (II), $C_{26}H_{23}NO_3$, $M_r = 397.5$, monoclinic, $P2_1/n$, $a = 10.940$ (2), $b = 13.649$ (5), $c = 14.418$ (4) Å, $\beta = 98.96$ (2)°, $V = 2126$ (1) Å^3, $Z = 4$, $D_x = 1.24$ g cm^{-3}, $\mu = 0.76$ cm^{-1}, $T =$

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