ment of the instrument or a poor choice of reference reflections – can lead to errors far larger than represented by the apparent precisions.

References

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Structure of magnesium chlorite hexahydrate. Corrigendum.

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Abstract

The structure of $Mg(ClO_2)_2.6H_2O$ was described and refined [Okuda, Ishihara, Yamanaka, Ohba & Saito (1990). *Acta Cryst.* C**46**, 1755–1759] in the space group $P4_2mc$ [tetragonal, $a = 7{\cdot}471\ (8)$, $c = 9{\cdot}980\ (2)$ Å]; it is better described in $P4_2/nmc$ ($R = 0{\cdot}024$, *versus* 0·041 previously). Revised coordinates and bond lengths are given.

Recently, Okuda, Ishihara, Yamanaka, Ohba & Saito (1990; hereafter OIYOS) carried out crystal structure determinations of $Pb(ClO_2)_2$ and $Mg(ClO_2)_2.6H_2O$ and an improved refinement of $AgClO_2$. For the magnesium compound they reported systematic absences ($hk0$ with $h + k$ odd, hhl with l odd) characteristic of space group $P4_2/nmc$; however, they could not find a satisfactory solution in that space group ($R = 0{\cdot}066$), and presumed that the first set of absences was only 'pseudo-systematic'. Their resulting structure in $P4_2mc$ showed surprisingly irregular features: two independent ClO_2^- groups with Cl—O bond lengths of 1·537 (4) and 1·607 (5) Å and Mg—O distances ranging from 1·961 (11) to 2·121 (12) Å. Since such disparities often result from attempts to refine a centrosymmetric structure in a non-centrosymmetric space group, a further attempt to describe the structure in $P4_2/nmc$ seemed appropriate, and was successful.

The starting $P4_2/nmc$ model was obtained by incrementing the x and y coordinates (OIYOS, Table 1) by $\frac{1}{4}$ so as to place the Mg atom at a site of $\bar{4}2m$ symmetry (origin at a center of symmetry). Refinement was based on the 313 F_o values recovered from SUP 52951; the quantity minimized was $\sum w(F_o^2 - F_c^2)^2$, with weights w equal to $1/F_o^2$ for $F_o > 15{\cdot}0$ or to $1/15{\cdot}0F_o$ otherwise (Hughes, 1941). Initial refinement of the heavy-atom positions and U_{ij}'s led to an R of 0·048; the H atoms were then recovered as the largest peaks on a difference map. Final full-matrix refinement was based on 30 parameters: coordinates for seven independent atoms, anisotropic U_{ij}'s for the five Cl, Mg and O atoms, isotropic B's for the two H atoms, a scale factor and an isotropic extinction coefficient [final value $11{\cdot}8\ (4) \times 10^{-6}$]. Coordinates are given in Table 1.† Bond lengths and angles are given in Table 2. They are considerably more reasonable than reported earlier: there is but a single independent ClO_2^- ion with equal (by symmetry) Cl—O distances, and the two independent Mg—O distances are very nearly equal.

Table 1. *Coordinates and thermal parameters* (Å^2), *space group* $P4_2/nmc$; x,y,z *and* $U_{eq} \times 10^4$

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

	Site	x	y	z	U_{eq}/B
Cl	4(*d*)	2500	2500	5041 (1)	488 (2)
Mg	2(*a*)	2500	7500	2500	396 (2)
O(1)	8(*g*)	2500	4228 (2)	5954 (1)	437 (3)
O(2)	4(*c*)	2500	7500	450 (2)	681 (9)
O(3)	8(*f*)	550 (3)	5550	2500	689 (4)
H(2)	8(*g*)	3379 (32)	7500	13 (25)	7·0 (9)†
H(3)	16(*h*)	541 (23)	4679 (21)	2057 (14)	4·9 (5)†

† Isotropic displacement parameter, B.

Table 2. *Distances* (Å) *and angles* (°), *space group* $P4_2/nmc$

Cl—O(1)	1·580 (2)	H(2)···O(1)	2·03 (3)
Mg—O(2)	2·046 (2)	O(3)—H(3)	0·79 (2)
Mg—O(3)	2·061 (5)	O(3)···O(1)	2·757 (5)
O(2)—H(2)	0·79 (3)	H(3)···O(1)	1·97 (2)
O(2)···O(1)	2·818 (3)		

O(1)—Cl—O(1)	109·5 (1)	H(3)—O(3)—H(3)	107·7 (17)
O(2)—Mg—O(2)	180·0 (–)	Mg—O(2)—H(2)	123·6 (18)
O(2)—Mg—O(3)	90·0 (–)	Mg—O(3)—H(3)	126·2 (12)
O(3)—Mg—O(3)	90·0 (–)	O(2)—H(2)···O(1)	174·7 (25)
H(2)—O(2)—H(2)	112·8 (25)	O(3)—H(3)···O(1)	174·5 (16)

In their attempts to refine the structure in the space group $P4_2/nmc$, OIYOS reported that they placed the Cl atom at the special position $(-\frac{1}{4},\frac{1}{4},\frac{1}{4})$; this is a site of symmetry $\bar{4}2m$, unlikely for a ClO_2^- group. In the revised molecule, the site symmetry is mm.

* Contribution No. 8375.

† Lists of structure factors and U_{ij}'s have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54014 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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