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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1991). C**47**, 1774–1775

Structure of bis(diphenylboron-dimethylglyoximato)nickel(II). Corrigendum.

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Abstract

The structure of $Ni(C_{32}H_{32}B_2N_4O_4)$ has been described as triclinic, space group $P\bar{1}$ [Xu, Lei, Cheng, Xu, Chen & Tang (1990). *Acta Cryst.* C**46**, 1447–1450]. It is properly described as monoclinic, space group $C2/c$. Revised coordinates are given.

The triclinic cell dimensions were reported as $a = 8{\cdot}385\ (2)$, $b = 14{\cdot}068\ (3)$, $c = 14{\cdot}234\ (3)$ Å, $\alpha = 75{\cdot}20\ (2)$, $\beta = 72{\cdot}74\ (2)$, $\gamma = 72{\cdot}53°$, $Z = 2$. The lattice vectors $[1\bar{2}0]$, $[\bar{1}00]$ and $[01\bar{1}]$ define a C-centered cell with $a' = 26{\cdot}838$, $b' = 8{\cdot}385$, $c' = 17{\cdot}269$ Å, $\alpha' = 90{\cdot}00$, $\beta' = 129{\cdot}29$, $\gamma' = 89{\cdot}87°$, $Z = 4$. The corresponding coordinate transformations are: $x' = \frac{1}{2}(-y-z) + 0{\cdot}25$, $y' = -x - \frac{1}{2}(y+z) + 0{\cdot}25$, $z' = -z$. After averaging over pairs of symmetry-related atoms, the $C2/c$ coordinates in Table 1 result. Included in Table 1 are the shifts in the coordinates necessary to attain the higher symmetry; none of these shifts is greater than the corresponding e.s.d. Since the shifts are so small, there are no significant changes in the bond lengths or angles. Xu, Lei, Cheng, Xu, Chen & Tang (1990) noted that 'the molecule has approximate $C2$ symmetry' and also noted 'the approximate systematic absence $k + l = 2n + 1$ for $0kl$ reflections in the triclinic system.' These absences are characteristic of the c-glide plane in $C2/c$, and the molecule lies on a crystallographic twofold axis.

The revised angle γ' appears to differ from the expected value of 90° by several e.s.d.'s. However, the atomic coordinates leave no doubt that the monoclinic description is correct, and it seems clear that the accuracies in the triclinic cell dimensions are appreciably worse than represented by the reported e.s.d.'s. It is the usual procedure – and, presumably, the one followed by Xu *et al.* (1990) – to report the precision values obtained during an automated centering routine on a computer-controlled diffractometer; such values need have little relationship to the true accuracies, and it is misleading to quote them as values of 'estimated standard deviations'. Many factors – such as absorption, mis-centering of the crystal, misalign-

Table 1. *Coordinates* ($\times 10^4$), *space group* $C2/c$

Numbers in square brackets are shifts in the $P\bar{1}$ coordinates necessary to attain the symmetry of $C2/c$.

	x'	y'	z'
Ni	0 [·3]	−2126	−2500 [·1]
O(1)	1260 [0]	−1928 [0]	−1929 [1]
O(2)	−348 [0]	−1998 [1]	−1244 [0]
N(1)	897 [1]	−1951 [2]	−1635 [1]
N(2)	128 [0]	−1968 [1]	−1313 [0]
C(1)	1166 [0]	−1616 [2]	−716 [0]
C(2)	710 [1]	−1629 [0]	−516 [0]
C(11)	1869 [1]	−1268 [4]	55 [0]
C(21)	891 [3]	−1290 [3]	478 [4]
C(101)	1439 [1]	−2772 [1]	−3140 [2]
C(102)	1981 [1]	−1779 [1]	−2638 [2]
C(103)	2382 [1]	−1762 [0]	−2888 [0]
C(104)	2251 [0]	−2713 [1]	−3642 [0]
C(105)	1715 [2]	−3690 [1]	−4160 [2]
C(106)	1322 [0]	−3723 [2]	−3900 [0]
C(107)	805 [2]	−4662 [2]	−2766 [1]
C(108)	1249 [0]	−5510 [3]	−1886 [0]
C(109)	1160 [2]	−7110 [0]	−1802 [2]
C(110)	620 [0]	−7908 [2]	−2594 [2]
C(111)	168 [2]	−7088 [1]	−3461 [3]
C(112)	260 [1]	−5498 [4]	−3544 [0]
B	958 [2]	−2877 [0]	−2888 [3]

* Contribution No. 8224.

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

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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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