

reported for the *Cc* refinement (the e.s.d.'s are about half as large). The geometry of the molecule was found to be close to that previously reported.

Category 3: centre of symmetry added and crystal class revised, no further refinement

Aquatetrakis(3,5-dimethylpyrazole)copper diperchlorate, $[\text{Cu}(\text{C}_5\text{H}_8\text{N}_2)_4(\text{H}_2\text{O})](\text{ClO}_4)_2$. The crystal structure of the complex was reported (Pervukhina, Podberezhskaya, Lavrenova, Larionov & Bakakin, 1986) in space group *C2* [$a = 19.783$ (5), $b = 16.271$ (6), $c = 13.994$ (6) Å, $\beta = 134.98$ (3)°, $Z = 4$]. The lattice vectors [001], [101] and [010] define an *I*-centred tetragonal cell ($a' = 13.994$, $b' = 13.994$, $c' = 16.217$ Å, $\alpha = 90.00$, $\beta = 90.00$, $\gamma = 89.96$ °, $Z = 4$). The corresponding coordinate transformations $x' = \bar{x} + z$, $y' = x$, $z' = y$ led to a structure that conforms quite closely to the symmetry of *I4/m* (No. 87). The *I4/m* coordinates have been deposited (Table 8A). Since in space group *I4/m* the Cl atom lies on a mirror with one of the Cl—O vectors almost perpendicular to this plane, the perchlorate group must be disordered. The structure is characterized by two types of hydrogen bonding both involving the ClO_4^- O atoms as acceptors with the water molecule and N(2) acting as donors [*i.e.* O(4)⋯N(2) = 2.808 Å and O(2)⋯O(9)(x, y, z) = 2.721 Å].

Category 4: change in Laue group

Tris(methyltrimethylsilylphosphino)phosphine, $\text{P}[\text{P}(\text{SiMe}_3)\text{Me}]_3$. The structure of this compound was described (Fritz, Stoll, Hönle & Schnering, 1986) in space group *Cc* [monoclinic, $a = 9.844$ (11), $b = 19.650$ (40), $c = 26.054$ (27) Å, $\beta = 100.91$ (9)°, $Z = 8$] and refined to $R_F = 0.09$ ($wR = 0.07$) using 2051

reflections with $I \geq 3\sigma(I)$. It is properly described as orthorhombic, space group *Fdd2* (No. 43). The lattice vectors $[0\bar{1}0]$, $[102]$ and $[\bar{1}00]$ define a cell with $a' = 19.650$, $b' = 51.166$, $c' = 9.844$ Å, $\alpha' = 89.98$, $\beta' = 90.00$, $\gamma = 90.00$ °, $Z = 16$. The corresponding transformations $x' = \bar{y}$, $y' = z/2 + 0.0665$, $z' = \bar{x} + z/2$ yield coordinates for atom pairs that are compatible (within three times the reported e.s.d.'s) with space group *Fdd2*. The *Fdd2* coordinates have been deposited (Table 9A).

We are grateful to Professor F. H. Herstein Technion) for bringing the structure of *N,N*-trimethylene(2-hydroxyacetophenone iminato)copper(II) to our attention and for helpful discussions.

References

- DEBAERDEMAEKER, T., BAUMGÄRTNER, R. & BRUNE, H.-A. (1987). *Z. Kristallogr.* **180**, 171–177.
- FRITZ, G., STOLL, K., HÖNLE, W. & SCHNERING, H. G. (1986). *Z. Anorg. Allg. Chem.* **544**, 127–136.
- IDA, K., OONISHI, I., NAKAHARA, A. & KOMIYAMA, Y. (1970). *Bull. Chem. Soc. Jpn.* **43**, 2347–2354.
- MARSH, R. E. & HERBSTEIN, F. H. (1988). *Acta Cryst.* **B44**, 77–88.
- MATVEEVA, R. G., VARFOLOMEEV, M. B., ŠAMRAJ, N. B. & LUNK, H.-J. (1986). *Z. Anorg. Allg. Chem.* **532**, 193–196.
- PERVUKHINA, N. V., PODBEREZSKAYA, N. V., LAVRENOVA, L. G., LARIONOV, S. V. & BAKAKIN, V. V. (1986). *J. Struct. Chem. (USSR)*, **27**, 434–438.
- SCHOMAKER, V. & MARSH, R. E. (1979). *Acta Cryst.* **B35**, 1933–1934.
- SHELDRIK, G. M. (1977). *SHELX77*. Program for crystal structure determination. Univ. of Cambridge, England.
- TAFEENKO, V. A., PROZOROVSKII, A. E., RYBAKOV, V. B. & KOVALEV, V. V. (1987). *J. Struct. Chem. (USSR)*, **28**(1), 158–163.
- YAMNOVA, N. A., PUSHCHAROVSKAYA, D. YU., BOGDANOVA, A. V. & LEONYUK, L. I. (1986). *Sov. Phys. Crystallogr.* **31**(5), 528–530.

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The Structure of Hexaaquanickel(II) Chlorate

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Abstract. Hexaaquanickel(II) chlorate, $[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_3)_2$, $M_r = 333.69$, cubic, $Pa\bar{3}$, $a = 10.3159$ (5) Å, $V = 1097.80$ (5) Å³, $Z = 4$, $F(000) = 680$, $D_x = 2.02$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $T = 296$ K, $\mu = 23.13$ cm⁻¹, $R = 0.026$ for 388 unique reflections having $I > \sigma_f$. The single type of Ni ion is coordi-

nated by six water-molecule O atoms, each at an observed distance 2.054 (1) Å, in an almost regular octahedral array. The single type of chlorate ion has a Cl—O bond length 1.487 (1) Å and O—Cl—O bond angle 106.45 (6)°. The Ni—O complex, but not the chlorate ion, manifested rigid-body behavior. The Ni—O distance corrected for rigid-body motion is 2.060 Å. Location and refinement of the two

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inequivalent H atoms permitted a detailed analysis of hydrogen bonding, which occurs principally between the oxygen octahedra and the chlorate groups.

Introduction. Among the oxyhalogen salts, whose structures generally have become of interest to us, hexaaquanickel(II) chlorate initially engaged our attention due to what proved to be erroneous data given for it in a standard chemical reference. As in our other recent studies, we hoped to determine a structure which included explicitly the protons of the waters of hydration.

Experimental. Nickel(II) chlorate was produced in solution by the metathetical reaction of aqueous analytical reagent grade nickel(II) sulfate with a saturated solution made from Alfa Products (152 Andover Street, Danvers, MA 01923) barium chlorate monohydrate. Following the mixing of these solutions in very nearly stoichiometric proportions the barium sulfate was removed by filtration. Slow evaporation of the filtrate at room temperature produced the experimental sample, a clear bluish-green compact crystal of irregular shape having 14 well defined faces and principal dimensions approximately $0.38 \times 0.46 \times 0.50$ mm. The sample was analyzed at 296 K with a Rigaku AFC5S diffractometer utilizing graphite-monochromated Mo $K\alpha$ radiation.

Unit-cell parameters were obtained from a symmetry-constrained least-squares fit of the setting angles for 25 well centered reflections with $29 \leq 2\theta \leq 30^\circ$. Intensity data were measured for 1868 reflections (exclusive of standards) with 2θ values in the range $4 \leq 2\theta \leq 60^\circ$. The ω - 2θ scan technique was employed with scan widths $(1.50 + 0.35 \tan \theta)^\circ$ in ω , and a background/scan time ratio of 0.5. Uncertainties were assigned to each reflection using the formula $\sigma_I^2 = \sigma_{CS}^2(I) + (0.03I)^2$, where σ_{CS} is based on counting statistics and I is the integrated intensity. Six standard reflections (022, 111, 222, 426, 515 and 624) were measured after every 150 reflections and showed a maximum non-systematic relative intensity variation of $\pm 1.5\%$. Accordingly, no decay correction was applied. The data were corrected for Lorentz and polarization effects. A ψ -scan absorption correction was applied based on ψ -scan data obtained at the conclusion of intensity data collection; the range of transmission factors was 0.846–1.000, with an average value 0.924.

The crystal was found to have Laue group $m\bar{3}$, then space group $Pa\bar{3}$ (No. 205) on the basis of systematic absences. The Ni atom was placed at the origin with site symmetry $\bar{3}$. Initial coordinates for the Cl atom in the special position with site symmetry 3 were obtained from a Patterson map, and Fourier methods were subsequently used to assign, in

general positions, the two inequivalent oxygens and the two inequivalent hydrogens. Full-matrix least-squares refinement was performed using the TEXSAN structure analysis package (Molecular Structure Corporation, 1988) to minimize the function $\sum \sigma_F^{-2}(|F_o| - |F_c|)^2$, in which $\sigma_F = \sigma_I/2FLP$. Neutral-atom scattering factors and anomalous-dispersion factors were taken from Cromer & Waber (1974) for Ni, Cl and O; the scattering factor for H was taken from Stewart, Davidson & Simpson (1965).

Following refinement to the isotropic stage for all atoms except hydrogen using the data having $I > 3\sigma_I$, the ψ -scan absorption correction was applied and the data were averaged ($R_{int} = 0.025$ for 620 triples of reflections). Then, using all the unique reflections with $I > \sigma_I$ and treating the secondary-extinction coefficient as an adjustable parameter, least-squares refinement proceeded to the anisotropic stage for all atoms except hydrogen and to the isotropic stage for the hydrogens.

The results for the final refinement cycle were: 388 independent observations having $I > \sigma_I$; 33 variables; $R = 0.026$; $wR = 0.031$; $S = 1.29$; $(\Delta/\sigma)_{max} < 0.01$. Maximum and minimum peaks on the final electron density difference map had values $+0.35$ and $-0.30 e \text{ \AA}^{-3}$, respectively. The maximum peak was located at $x = 0.29$, $y = 0.31$, $z = 0.29$, $\sim 0.8 \text{ \AA}$ from Cl; the minimum peak was located at $x = 0.78$, $y = 0.22$, $z = 0.21$, $\sim 0.6 \text{ \AA}$ from Cl. [For the final refinement cycle for 328 independent observations having $I > 3\sigma_I$: $R = 0.021$; $wR = 0.029$; $S = 1.33$. Similarly, for the 542 independent observations having $I > 0$: $R = 0.047$; $wR = 0.031$; $S = 1.11$.]

The final atomic coordinates and equivalent isotropic displacement parameters and their uncertainties are given in Table 1.* Selected interatomic distances and angles in the coordination polyhedron about nickel, in the chlorate ion, and in the water molecule are given in Table 2 together with their uncertainties. Hydrogen bonds involving the water-molecule oxygen and its two protons are delineated in Table 3, as discussed in detail below.

Rigid-body analysis of the Ni—O complex and the chlorate ion was performed using the program THMA11 (1986), based on the work of Schomaker & Trueblood (1968). The average magnitude of the differences in the mean square displacement amplitudes (MSDA) along the interatomic vectors for the two unique atom pairs of the chlorate ion was found

* Lists of structure factors and anisotropic displacement parameters and supplementary material relating to the rigid-body analysis have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52310 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to be $83(10) \times 10^{-4} \text{ \AA}^2$. The average magnitude of the differences in the MSDA along the interatomic vectors for the four unique atom pairs of the Ni—O complex was found to be $28(10) \times 10^{-4} \text{ \AA}^2$. On the basis of these values, we concluded that Hirshfeld's rigid-bond test, as described by Dunitz, Schomaker & Trueblood (1988), is satisfied for the Ni—O complex but not for the chlorate ion, and have applied corrections for the rigid-body motion in the Ni—O complex. The corrected bond lengths are given in Table 2; the corrected angles differed typically from the observed angles by less than their standard deviations and were therefore not tabulated.

Discussion. This structure can be described most simply as fluorite-like, with $\text{Ni}(\text{H}_2\text{O})_6$ replacing Ca and ClO_3 replacing F. Substantial hydrogen bonding occurs between the water molecules and the chlorate ions.

The nickel coordination polyhedron has, by symmetry, a single Ni—O(2) distance [observed value, 2.054 (1) Å] and thus departs only in an angular sense, but very slightly, from regular octahedral geometry, as documented in Table 2. Corrected for rigid-body motion the single Ni—O(2) distance becomes 2.060 Å.

The chlorate ion has, by symmetry, a single Cl—O(1) distance [observed value, 1.487 (1) Å] and an O(1)—Cl—O(1) angle of 106.45 (6)°.

Concerning the water molecule we note that the observed H—O—H angle (Table 2) is 106° with an e.s.d. of 3°. This value compares favorably with the value for water-molecule H—O—H angles determined by neutron diffraction and tabulated by Chiari & Ferraris (1982). For inorganic hydrates of class *J* (Chiari & Ferraris, 1982; Ferraris & Franchini-Angela, 1972) to which this salt belongs, the range is from 100.4 to 109.9° with an average value of 106.9°.

The occurrence of only a single type of virtually regular octahedra of water-oxygen atoms and a single type of chlorate groups leads to a rather simple hydrogen-bonding situation. Our analysis of these hydrogen bonds and their geometries is based partially upon treatments of hydrogen bonding by Jeffrey (1987) and Chiari & Ferraris (1982).

To account for the foreshortening of the water O—H bond distance as determined by X-ray diffraction, the position of each water proton was adjusted along the corresponding O—H bond direction (as determined by the X-ray data) until the O—H bond distance was 0.97 (3) Å, the mean value for the O—H bond distance as determined by neutron diffraction in inorganic hydrated crystals of class *J* (Chiari & Ferraris, 1982). The resulting adjusted proton coordinates were then used to calculate distances and angles involving potential hydrogen-

Table 1. Final positional parameters and equivalent isotropic and isotropic displacement parameters, B_{eq}/B (Å²), for hexaaquanickel(II) chlorate at 296 K

E.s.d.'s are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}/B^*
Ni	0	0	0	1.81 (2)
Cl	0.25378 (4)	0.25378	0.25378	2.11 (2)
O(1)	0.16904 (12)	0.67007 (12)	0.16536 (12)	2.87 (6)
O(2)	0.02884 (14)	-0.02775 (14)	0.19505 (13)	2.51 (6)
H(1)	0.0531 (25)	0.0343 (25)	0.2338 (27)	5.0 (7)
H(2)	-0.0343 (24)	-0.0489 (23)	0.2281 (25)	3.7 (6)

$$* B_{\text{eq}} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Table 2. Bond lengths (Å) and angles (°) for hexaaquanickel(II) chlorate

E.s.d.'s are in parentheses.

Coordination polyhedron	Observed distance (Å)	Rigid-body distance (Å)	Observed angle (°)
Ni—O(2)	2.054 (1)	2.060	O(2)—Ni—O(2) ⁽ⁱ⁾ 180
O(2)—O(2) ⁽ⁱ⁾	4.108 (3)	4.119	O(2)—Ni—O(2) ⁽ⁱⁱ⁾ 90.85 (6)
O(2)—O(2) ⁽ⁱⁱ⁾	2.926 (2)	2.936	O(2)—Ni—O(2) ⁽ⁱⁱⁱ⁾ 89.15 (6)
O(2)—O(2) ⁽ⁱⁱⁱ⁾	2.883 (2)	2.889	
Water molecule			
O(2)—H(1)	0.80 (3)		H(1)—O(2)—H(2) 106 (3)
O(2)—H(2)	0.77 (2)		
Chlorate ion			
Ni—Cl	4.5344 (6)		O(1 ^v)—Cl—O(1 ^v) 106.45 (6)
Ni—Cl ^(iv)	4.4449 (3)		
Cl—O(1 ^v)	1.487 (1)		

Code for symmetry-related atoms: none *x, y, z*; (i) $-x, -y, -z$; (ii) z, x, y ; (iii) $-z, -x, -y$; (iv) $-\frac{1}{2} + x, \frac{1}{2} - y, -z$; (v) $\frac{1}{2} - x, -\frac{1}{2} + y, z$; (vi) $z, \frac{1}{2} - x, -\frac{1}{2} + y$.

bond acceptor oxygen atoms. Since the adjusted O(water)—H—O(acceptor) angles typically differed from the observed angles (calculated directly from the X-ray determined coordinates) by less than one standard deviation, only the observed angles are tabulated together with observed O(water)—O(acceptor) distances and observed and neutron-adjusted H—O(acceptor) distances in Table 3. In the following material, the neutron-adjusted H—O(acceptor) distance is taken to be the hydrogen-bond length.

As shown in Table 3, each of the two inequivalent H atoms is involved in one hydrogen bond in the H—O(acceptor) distance range 1.90–1.92 Å and in which the acceptor is a chlorate oxygen. These values for the H—O(acceptor) distance lie approximately halfway between the mean value (1.777 Å) and the maximum value (2.069 Å) for H—O(acceptor) distances for type *J* hydrates as tabulated by Chiari & Ferraris (1982). Accordingly, we may properly describe these as strong hydrogen bonds. Since there are six of each of these two types of H atoms in the coordination polyhedron of a given Ni ion, we find a total of 12 strong hydrogen bonds linking that

Table 3. *Hydrogen-bond parameters for hexaaquanickel(II) chlorate*

E.s.d.'s are in parentheses. See text for details.

Water oxygen-acceptor oxygen	Distance (Å)	Proton-acceptor oxygen	Observed distance (Å)	Neutron-adjusted distance (Å)	Observed O—H—O angle (°)
O(2)—O(1 ⁱⁱⁱ)	2.838 (2)	H(1)—O(1 ⁱⁱⁱ)	2.07 (3)	1.90	O(2)—H(1)—O(1 ⁱⁱⁱ) 162 (3)
O(2)—O(1 ⁱⁱⁱⁱ)	2.841 (2)	H(2)—O(1 ⁱⁱⁱⁱ)	2.11 (2)	1.92	O(2)—H(2)—O(1 ⁱⁱⁱⁱ) 159 (2)
O(2)—O(2 ⁱⁱⁱ)	2.883 (2)	H(2)—O(2 ⁱⁱⁱ)	2.66 (3)	2.63	O(2)—H(2)—O(2 ⁱⁱⁱ) 99 (2)
O(2)—O(2 ^{iv})	2.926 (2)	H(1)—O(2 ^{iv})	2.81 (3)	2.82	O(2)—H(1)—O(2 ^{iv}) 90 (2)
O(2)—O(1 ^v)	3.226 (2)	H(1)—O(1 ^v)	2.88 (3)	2.83	O(2)—H(1)—O(1 ^v) 109 (2)
		H(2)—O(1 ^v)	2.87 (3)	2.80	O(2)—H(2)—O(1 ^v) 111 (2)

Code for symmetry-related atoms: none x, y, z ; (iii) $-z, -x, -y$; (vii) $-\frac{1}{2} + y, z, \frac{1}{2} - x$; (viii) $-z, -x, 1 - y$; (ix) y, z, x ; (x) $-x, -\frac{1}{2} + y, \frac{1}{2} - z$.

Table 4. *Ni^{II}—O distances in hydration polyhedra and Cl—O distances and angles in chlorate anions in various salts*

Salt	Observed Ni—O distance(s) (Å)	Mean observed Ni—O distance (Å)	Rigid-body-corrected mean Ni—O distance (Å)	Reference	
[Ni(H ₂ O) ₆] ₂ I ₂	6 × 2.064 (9)	2.064 (9)	—	Louër, Grandjean & Weigel (1973)	
[Ni(H ₂ O) ₆](BrO ₄) ₂	6 × 2.047 (2)	2.047 (2)	—	Gallucci, Gerkin & Reppart (1988)	
[Ni(H ₂ O) ₆] ₂ SO ₄ mono	2.047 (2), 2.055 (2), 2.039 (2), 2.066 (2), 2.042 (2), 2.040 (2)	2.048 (10)	—	Gerkin & Reppart (1988)	
[Ni(H ₂ O) ₆] ₂ SO ₄ mono	2 × : 2.045 (2), 2.043 (2), 2.051 (2), 2.068 (2), 2.036 (2), 2.045 (2)	2.048 (10)	—	Angel & Finger (1988)	
tetr	2 × : 2.014 (2), 2.053 (2), 2.087 (2)	2.051 (30)	—		
[Ni(H ₂ O) ₆](ClO ₃) ₂	6 × 2.054 (1)	2.054 (1)	2.060 (1)	This research	
Salt	Observed Cl—O distance(s) (Å)	Mean observed Cl—O distance (Å)	Rigid-body-corrected mean Cl—O distance (Å)	Mean observed O—Cl—O angle (°)	Reference
NaClO ₃	3 × 1.490 (3)	1.490 (3)	1.502 (3)	106.8 (2)	Burke-Laing & Trueblood (1977)
KClO ₃	2 × 1.474 (1), 1.479 (1)	1.476 (2)	1.497 (2)	106.4 (1)	Bats (1978)
Ba(ClO ₃) ₂ ·H ₂ O	1.493 (4), 1.480 (4), 1.483 (4)	1.485 (5)	—	106.2 (9)	Sikka, Momin, Rajagopal & Chidambaram (1968) (neutron)
[Ni(H ₂ O) ₆](ClO ₃) ₂	3 × 1.487 (1)	1.487 (1)	—	106.45 (6)	This research

polyhedron to the surrounding chlorate ions. These are shown in Fig. 1, a stereoview of the environment of a (central) Ni ion. As the figure makes apparent, these hydrogen bonds alone form a three-dimensional network.

In addition, we have tabulated two weaker hydrogen bonds in the distance range 2.63–2.82 Å which involve oxygen acceptors from within the same coordination polyhedron and a single bifurcated bond (see Jeffrey, 1987) to a chlorate-oxygen acceptor in the distance range 2.80–2.83 Å. We note that only one of the O(water)—O(acceptor) distances involved exceeds the 'maximum' value cited by Jeffrey and Chiari & Ferraris for hydrogen-bond formation in inorganic hydrates (3.15 Å), that being 3.226 Å in the case of the bifurcated bond.

Considering the hydrogen bonding from the viewpoint of the chlorate-oxygen acceptors we observe that each of the three equivalent oxygens of a chlorate ion is involved in two strong hydrogen bonds

(length 1.90 or 1.92 Å) and one bifurcated bond. It is noteworthy that the eight nearest-neighbor chlorate groups of a given (central) Ni ion fall into two classes with respect to their hydrogen bonding with waters associated with that given nickel ion: for what we shall call Type I chlorate ions, of which there are two, each chlorate-ion O atom is involved in one strong hydrogen bond with a water molecule associated with the central Ni ion and in one hydrogen bond with a water molecule not associated with the central Ni; for what we shall call Type II chlorate ions, of which there are six, only a single chlorate-ion O has one strong hydrogen bond with a water molecule associated with the central Ni ion, the remaining five strong hydrogen bonds involving water molecules not associated with the central Ni ion. These two types of chlorate ions are quite apparent in Fig. 1.

Although there is apparently no previously published structural work on transition-metal chlo-

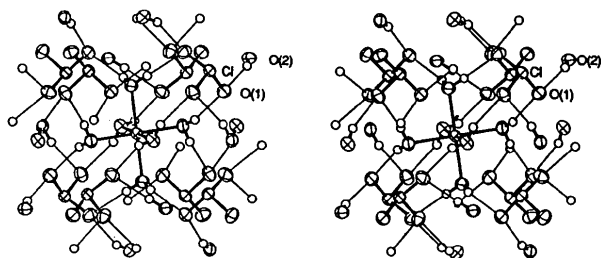


Fig. 1. Stereoview of the environment of a (central) nickel ion in the hexaaquanickel(II) chlorate structure from a direction nearly along the x axis (with the z axis vertical). The 12 strong hydrogen bonds to the chlorate ions from the six water molecules associated with this central nickel ion are shown. Drawn using ORTEPII (Johnson, 1971). Thermal ellipsoids are drawn at 50% probability for all atoms except H, for which they have been set artificially small.

rate hydrates, Yü & Beevers (1936) have determined the structure of $\text{Zn}(\text{BrO}_3)_2 \cdot 6\text{H}_2\text{O}$, which is isomorphic and strikingly similar (with respect to the heavy atoms) to that determined here for $[\text{Ni}(\text{H}_2\text{O})_6]-(\text{ClO}_3)_2$. A helpful view of a unit-cell projection is presented in their Fig. 3, while their Fig. 5 presents photographs of their three-dimensional model of a unit cell suitable for stereographic viewing.

In Table 4 we have tabulated previously published representative results for Ni—O coordination polyhedra and chlorate ions for comparison with the values determined in this study.

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References

- ANGEL, R. J. & FINGER, L. W. (1988). *Acta Cryst.* **C44**, 1869–1873.
- BATS, J. W. (1978). *Acta Cryst.* **B34**, 1679–1681.
- BURKE-LAING, M. E. & TRUEBLOOD, K. N. (1977). *Acta Cryst.* **B33**, 2698–2699.
- CHIARI, G. & FERRARIS, G. (1982). *Acta Cryst.* **B38**, 2331–2341.
- CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, pp. 71, 148. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- DUNITZ, J. D., SCHOMAKER, V. & TRUEBLOOD, K. N. (1988). *J. Phys. Chem.* **92**, 856–867.
- FERRARIS, G. & FRANCHINI-ANGELA, M. (1972). *Acta Cryst.* **B28**, 3572–3583.
- GALLUCCI, J. C., GERKIN, R. E. & REPPART, W. J. (1988). *Acta Cryst.* **C44**, 1345–1348.
- GERKIN, R. E. & REPPART, W. J. (1988). *Acta Cryst.* **C44**, 1486–1488.
- JEFFREY, G. A. (1987). In *Patterson and Pattersons*, edited by J. P. GLUSKER, B. K. PATTERSON & M. ROSSI, pp. 193–221. IUCr/Oxford Univ. Press.
- JOHNSON, C. K. (1971). ORTEPII. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- LOUËR, M., GRANDJEAN, D. & WEIGEL, D. (1973). *J. Solid State Chem.* **7**, 222–228.
- Molecular Structure Corporation (1988). TEXSAN; TEXRAY Structure Analysis Package, Version 2.1. Molecular Structure Corporation, The Woodlands, Texas, USA.
- SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63–76.
- SIKKA, S. K., MOMIN, S. N., RAJAGOPAL, H. & CHIDAMBARAM, R. (1968). *J. Chem. Phys.* **48**, 1883–1890.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- YÜ, S. H. & BEEVERS, C. A. (1936). *Z. Kristallogr.* **95**, 426–434.

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Untwinned Single Crystals of the High-Temperature Superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$

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Abstract. $\text{YBa}_2\text{Cu}_3-0.06(1)\text{O}_{7-0.18(2)}$, $M_r = 659.5$, orthorhombic, $Pmmm$, $a = 3.836(1)$, $b = 3.883(1)$, $c = 11.686(1)$ Å, $V = 174.07$ Å³, $Z = 1$, $F(000) = 290.82$, $D_x = 6.36$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 28.48$ mm⁻¹, $T = 295$ K, $R = 0.0495$, $wR = 0.0341$ for 1342 unique diffractometer data [$F^2 > 1\sigma(F^2)$] up

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