The hydrogen bonds among the tetraamine and perchlorate groups are N(1)—H(1)···O(2) and N(2)—H(2)···O(4) with corresponding lengths of 3·061 and 3·011 Å, respectively. These hydrogen bonds stabilize the crystal structure.

Comparing the structure of this crystal with that of perchlorato(Me_2-2,2,2-tet)copper(II) perchlorate monohydrate (Wu, Wang, Liou & Chung, 1989), we find that they are isomorphous. Both nickel(II) and copper(II) ions are six-coordinated, distorted square bipyramidal with the Me_2-2,2,2-tet ligand equatorial and the O atoms of the perchlorate ions axial. All of the five-membered rings for both complexes have the stable *gauche* conformation and the hydrogen bonds stabilize the crystal structures. The Ni—N_4 distances, 1·895 (7) to 1·908 (9) Å, are shorter than the Cu—N_4 distances, 1·990 (10) to 1·994 (14) Å; while the Ni—O distance, 2·840 (11) Å, is much longer than the Cu—O distance, 2·578 (8) Å. The N—*M*—N angles of the five-membered rings for the Ni^{II} complex are larger than those for the Cu^{II} complex.

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Structure of {Bis[(1-methylimidazol-2-yl)methyl]amine}(1-methylimidazole)copper(II) Diperchlorate

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Abstract. $[Cu(C_{14}H_{21}N_7)]^{2+}.2ClO_4^-$, $M_r = 549{\cdot}81$, monoclinic, $P2_1/n$, $a = 9{\cdot}171$ (1), $b = 8{\cdot}999$ (1), $c = 26{\cdot}677$ (3) Å, $\beta = 94{\cdot}08$ (1)°, $V = 2196{\cdot}1$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}66$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}70930$ Å, $\mu = 13{\cdot}0$ cm^{-1}, $F(000) = 1124$, $T = 295$ K, final $R = 0{\cdot}063$ for 2687 observed reflections. The geometry around the Cu^{II} ion is best described as a distorted octahedron with coordination to two imidazole N atoms and an amine N atom of the bis[(1-methylimidazol-2-yl)methyl]amine ligand (bmima), one imidazole N atom from a 1-methylimidazole ligand and two O atoms from the two disordered perchlorate anions. The N atoms occupy equatorial positions of the octahedron with Cu—N bond distances ranging from 1·954 (6)–2·056 (5) Å. The perchlorate O atoms are weakly coordinated and occupy axial positions with Cu—O bond distances ranging from 2·55 (1)–2·72 (1) Å. One of the coordinated perchlorates is also partially hydrogen bonded to the amine H atom of the bmima ligand.

Introduction. Imidazole complexes of copper are of interest as models of the histidine (imidazole) ligands known to be present at the active site of numerous copper proteins. For instance, imidazoles are found at the active sites of plastocyanin (Guss & Freeman, 1983), azurin from *Pseudomonas aeruginosa* (Norris, Anderson & Baker, 1988) and deoxyhemocyanin from *Panularis interruptus* (Linzen, Soeter, Riggs, Schneider, Schartau, Moore, Yokota, Behrens, Nakashima, Takagi, Nemoto, Vereifken, Bak, Beintema, Volbeda, Gaykema & Hol, 1985). Hemocyanin functions as an oxygen transport protein and is found in the hemolymph of several species of arthro-

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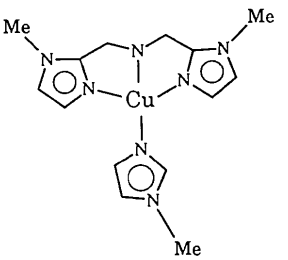
pods and mollusks (Solomon, Penfield & Wilcox, 1983). X-ray structural data on deoxyhemocyanin indicate that the two Cu^I ions are separated by 3·8 (4) Å, with each metal center coordinated to two imidazole rings of histidine, at 2·0 Å, and another at 2·7 Å (Volbeda & Hol, 1989). In oxyhemocyanin, the Cu^{II} centers are bridged by both a peroxide ion and an endogenous ligand which is thought to be either a hydroxide ion or water (Wilcox, Long & Solomon, 1984).

Surprisingly, few copper–imidazole complexes have been characterized crystallographically (Tolman, Rardin & Lippard, 1989; Sorrell & Borovik, 1987). Synthetic analogues of the proposed active sites of various metalloproteins have been useful in elucidating the relations between structure, electronic properties and chemical action of the enzymes (Ibers & Holm, 1980). As part of a continuing structural and chemical investigation of copper–imidazole complexes (Oberhausen, Richardson, Buchanan & Pierce, 1989; Doman, Richardson, Arar & Buchanan, 1989; Oberhausen, O'Brien, Richardson & Buchanan, 1990) we have determined the structure of the tri-imidazole complex $[Cu(bmima)(1\text{-MeIm})](ClO_4)_2$ {where bmima is bis[(1-methylimidazol-2-yl)methyl]amine and 1-MeIm is 1-methylimidazole}.



Experimental. The ligand bmima was synthesized by a previously reported procedure (Oberhausen, Richardson, Buchanan & Pierce, 1989). The copper complex was prepared by dissolving bmima (0·50 g, 2·4 mmol) and $[Cu(H_2O)_6](ClO_4)_2$ (0·90 g, 2·4 mmol) in 30 ml of methanol with stirring. After 0·5 h, the solution was filtered and 1-methylimidazole (0·40 g, 4·9 mmol) was added to the filtrate, resulting in the formation of an intense blue colored solution. The reaction mixture was allowed to set at room temperature overnight, whereupon large blue prisms precipitated (0·7110 g, 54%). Cut rectangular-shaped blue crystal, 0·45 × 0·30 × 0·22 mm, from methanol. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation, unit-cell parameters from least-squares refinement of 25 reflections with $15 < \theta < 18·6°$, space group $P2_1/n$ determined from intensity data and successful solution and refinement of the structure; 4042 reflections were collected, 3850 were unique and not systematically absent, $R_{int} = 0·013$; 2687 were observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics]; $\theta_{max} = 25°$, scan width $(0·8 + 0·340\tan\theta)°$, $\omega/2\theta$ scans, variable scan speed 1–3° min^{-1}; three standard reflections measured every 3600 s of X-ray exposure time, intensities of these standards remained constant throughout the data collection; data collected $-h$, $+k$, $\pm l$ to max. indices of 10, 10 and 31. Data corrected for background and Lp; empirical absorption correction based on a series of ψ scans, relative transmission coefficients ranged from 0·857 to 1·000 with average value of 0·959. Structure solved by direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), Cu atom located from E map, remaining non-H atoms from a series of difference Fourier syntheses on ΔF; model refined by full-matrix least squares based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma(F)^2 + (0·005F)^2 + 4·0]^{-1}$; *SDP/VAX* package of programs (Frenz, 1978) using VAX 11/750. All H-atom positions were calculated (C—H 0·97 Å) and added to the structure factor calculations with isotropic thermal parameters fixed at $1·3 \times B_{eq}$ of the bonded atom; H-atom parameters [except the positional parameters of H(N5)] were not refined; all non-H atoms refined using anisotropic thermal parameters. Model converged with 352 variables, $R = 0·063$, $wR = 0·066$, max. (Δ/σ) is 0·06, $S = 2·59$, max. residual electron density 0·71 (9), min. −0·57 (9) e $Å^{-3}$. Scattering factors were those of Cromer & Waber (1974); anomalous-dispersion corrections were included in F_c (Ibers & Hamilton, 1964), using values of f' and f'' from Cromer (1974). Positional and equivalent isotropic thermal parameters are listed in Table 1.

Discussion. An *ORTEP* (Johnson, 1965) view of the cation portion of the molecule is shown in Fig. 1. Selected bond distances and angles are listed in Table 2.* The Cu^{II} ion is in a tetragonally distorted octahedral environment coordinated to two imidazole ring N atoms [N(1) and N(3)], an amine N atom [N(5)] of the bmima ligand, an N atom [N(6)] from the 1-methylimidazole ligand and two O atoms from the perchlorate counter ions. The equatorial plane of the octahedron is formed from the four N atoms and is nearly planar to within 0·084 (5)°. The metal ion is not significantly displaced from the N_4 plane [0·021 (1) Å]. The two imidazole groups of the bmima ligand are nearly coplanar forming a dihedral angle of 8 (1)°. A similar arrangement of the bmima

* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54097 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and equivalent isotropic thermal parameters for non-H atoms*

$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}]$.

	x	y	z	B_{eq} (Å^2)	Occ.*
Cu	0·11145 (8)	0·23477 (9)	−0·12474 (3)	4·54 (2)	
N(1)	0·1033 (5)	0·4156 (6)	−0·0839 (2)	4·6 (1)	
N(2)	0·1892 (6)	0·5424 (6)	−0·0175 (2)	5·6 (1)	
N(3)	0·1705 (5)	0·0404 (6)	−0·1495 (2)	4·8 (1)	
N(4)	0·3101 (6)	−0·1577 (6)	−0·1378 (2)	5·7 (1)	
N(5)	0·2218 (5)	0·1585 (6)	−0·0600 (2)	4·4 (1)	
N(6)	−0·0021 (5)	0·2976 (7)	−0·1858 (2)	5·1 (1)	
N(7)	−0·1600 (5)	0·2899 (7)	−0·2518 (2)	5·0 (1)	
C(1)	0·1913 (6)	0·4140 (7)	−0·0431 (2)	4·4 (1)	
C(2)	0·0433 (7)	0·5588 (8)	−0·0843 (3)	6·4 (2)	
C(3)	0·0965 (8)	0·6354 (8)	−0·0446 (3)	6·7 (2)	
C(4)	0·2747 (9)	0·576 (1)	0·0299 (3)	8·1 (2)	
C(5)	0·2700 (6)	−0·0274 (7)	−0·1187 (2)	4·5 (1)	
C(6)	0·1449 (7)	−0·0514 (9)	−0·1908 (3)	6·4 (2)	
C(7)	0·2291 (8)	−0·1709 (9)	−0·1840 (3)	7·2 (2)	
C(8)	0·4196 (9)	−0·2603 (9)	−0·1162 (4)	8·3 (2)	
C(9)	0·2816 (7)	0·2820 (7)	−0·0294 (2)	5·0 (1)	
C(10)	0·3292 (7)	0·0418 (8)	−0·0717 (3)	5·1 (1)	
C(11)	−0·1239 (7)	0·2393 (9)	−0·2061 (2)	5·5 (2)	
C(12)	0·0401 (8)	0·3981 (9)	−0·2209 (3)	6·3 (2)	
C(13)	−0·0593 (8)	0·3927 (9)	−0·2616 (3)	6·0 (2)	
C(14)	−0·2836 (8)	0·243 (1)	−0·2852 (3)	7·3 (2)	
H(N5)	0·141 (7)	0·123 (9)	−0·047 (3)	5·7†	
Cl(1)	−0·1870 (2)	0·0051 (2)	−0·07413 (7)	6·14 (4)	
Cl(2)	0·4833 (2)	0·3474 (2)	−0·16481 (6)	5·46 (4)	
O(1)	−0·3034 (5)	0·0319 (7)	−0·0446 (2)	7·8 (1)	
O(2)	−0·060 (1)	−0·026 (1)	−0·0292 (4)	7·3 (3)‡	0·5
O(3)	−0·134 (1)	0·123 (1)	−0·0990 (4)	6·6 (2)‡	0·5
O(4)	−0·188 (2)	−0·126 (2)	−0·0988 (6)	12·6 (5)‡	0·5
O(5)	−0·275 (1)	−0·014 (2)	−0·1251 (5)	9·3 (3)‡	0·5
O(6)	−0·121 (2)	−0·124 (2)	−0·0671 (6)	11·7 (4)‡	0·5
O(7)	−0·106 (1)	0·142 (1)	−0·0801 (4)	5·9 (2)‡	0·5
O(8)	0·469 (1)	0·228 (1)	−0·1968 (4)	8·7 (3)‡	0·6
O(9)	0·529 (1)	0·355 (2)	−0·1135 (5)	11·5 (4)‡	0·6
O(10)	0·333 (1)	0·383 (1)	−0·1626 (4)	9·1 (3)‡	0·6
O(11)	0·5559 (9)	0·452 (1)	−0·1913 (3)	7·1 (2)‡	0·6
O(12)	0·544 (2)	0·197 (2)	−0·1633 (5)	8·2 (4)‡	0·4
O(13)	0·382 (2)	0·357 (2)	−0·1280 (5)	7·7 (3)‡	0·4
O(14)	0·621 (2)	0·411 (3)	−0·1493 (8)	13·3 (6)‡	0·4
O(15)	0·444 (2)	0·475 (3)	−0·1993 (8)	13·8 (7)‡	0·4

* Occupancies are listed if not 1·0. Groups fixed at averaged refined occupancies.

† H(N5) was refined with a fixed B.

‡ Atoms were refined isotropically.

Table 2. *Bond distances* (Å) *and bond angles* (°) *for selected non-H atoms*

Cu—N(1)	1·962 (5)	N(4)—C(5)	1·341 (8)
Cu—N(3)	1·960 (6)	N(4)—C(7)	1·397 (9)
Cu—N(5)	2·056 (5)	N(4)—C(8)	1·45 (1)
Cu—N(6)	1·954 (6)	N(5)—C(9)	1·463 (8)
Cu—O(3)	2·61 (2)	N(5)—C(10)	1·488 (8)
Cu—O(7)	2·55 (1)	N(6)—C(11)	1·316 (9)
Cu—O(10)	2·69 (2)	N(6)—C(12)	1·377 (9)
Cu—O(13)	2·72 (1)	N(7)—C(11)	1·321 (8)
N(1)—C(1)	1·308 (7)	N(7)—C(13)	1·346 (9)
N(1)—C(2)	1·402 (9)	N(7)—C(14)	1·454 (9)
N(2)—C(1)	1·343 (8)	C(1)—C(9)	1·479 (9)
N(2)—C(3)	1·363 (9)	C(2)—C(3)	1·33 (2)
N(2)—C(4)	1·48 (1)	C(5)—C(10)	1·469 (9)
N(3)—C(5)	1·332 (8)	C(6)—C(7)	1·33 (1)
N(3)—C(6)	1·383 (9)	C(12)—C(13)	1·37 (1)

N(1)—Cu—N(3)	161·7 (3)	Cu—N(5)—C(9)	111·0 (4)
N(1)—Cu—N(5)	81·2 (3)	Cu—N(5)—C(10)	110·7 (4)
N(1)—Cu—N(6)	100·6 (2)	Cu—N(6)—C(11)	127·9 (5)
N(3)—Cu—N(5)	81·7 (3)	Cu—N(6)—C(12)	126·7 (4)
N(3)—Cu—N(6)	96·9 (2)	C(1)—N(1)—C(2)	103·5 (5)
N(5)—Cu—N(6)	176·6 (2)	C(1)—N(2)—C(3)	106·8 (6)
O(3)—Cu—O(7)	12·7 (3)	C(1)—N(2)—C(4)	126·0 (6)
O(3)—Cu—O(10)	169·4 (3)	C(3)—N(2)—C(4)	127·1 (6)
O(3)—Cu—O(13)	166·6 (4)	C(5)—N(3)—C(6)	106·7 (6)
O(3)—Cu—N(1)	96·1 (3)	C(5)—N(4)—C(7)	105·5 (5)
O(3)—Cu—N(3)	90·5 (3)	C(5)—N(4)—C(8)	127·2 (6)
O(3)—Cu—N(5)	91·8 (3)	C(7)—N(4)—C(8)	127·2 (6)
O(3)—Cu—N(6)	85·1 (3)	C(9)—N(5)—C(10)	115·2 (5)
O(7)—Cu—O(10)	168·8 (3)	C(11)—N(6)—C(12)	104·7 (5)
O(7)—Cu—O(13)	153·9 (4)	C(11)—N(7)—C(13)	106·6 (5)
O(7)—Cu—N(1)	87·3 (3)	C(11)—N(7)—C(14)	126·6 (6)
O(7)—Cu—N(3)	96·3 (3)	C(13)—N(7)—C(14)	126·7 (6)
O(7)—Cu—N(5)	81·5 (2)	N(1)—C(1)—N(2)	112·6 (5)
O(7)—Cu—N(6)	95·5 (3)	N(1)—C(1)—C(9)	121·1 (6)
O(10)—Cu—O(13)	21·9 (4)	N(2)—C(1)—C(9)	126·4 (5)
O(10)—Cu—N(1)	81·7 (3)	N(1)—C(2)—C(3)	110·5 (6)
O(10)—Cu—N(3)	94·7 (3)	N(2)—C(3)—C(2)	106·6 (6)
O(10)—Cu—N(5)	98·2 (3)	N(3)—C(5)—N(4)	111·1 (5)
O(10)—Cu—N(6)	85·2 (3)	N(3)—C(5)—C(10)	122·0 (6)
O(13)—Cu—N(1)	75·7 (3)	N(4)—C(5)—C(10)	127·0 (5)
O(13)—Cu—N(3)	94·3 (4)	N(3)—C(6)—C(7)	108·1 (7)
O(13)—Cu—N(5)	76·5 (3)	N(4)—C(7)—C(6)	108·7 (7)
O(13)—Cu—N(6)	106·8 (3)	N(5)—C(9)—C(1)	107·0 (5)
Cu—N(1)—C(1)	114·0 (4)	N(5)—C(10)—C(5)	105·6 (5)
Cu—N(1)—C(2)	142·1 (4)	N(6)—C(11)—N(7)	113·1 (7)
Cu—N(3)—C(5)	113·2 (4)	N(6)—C(12)—C(13)	108·2 (6)
Cu—N(3)—C(6)	140·0 (5)	N(7)—C(13)—C(12)	107·3 (6)
Cu—O(3)—Cl(1)	146·0 (5)	Cu—O(10)—Cl(2)	129·6 (6)
Cu—O(7)—Cl(1)	139·6 (4)	Cu—O(13)—Cl(2)	136·9 (5)

ligand is observed for the complex [Cu(bmima)-(CH_3CO_2)]$^+$ (Oberhausen, Richardson, Buchanan & Pierce, 1989). The 1-methylimidazole ligand is twisted relative to the CuN_4 plane forming a dihedral angle of 68 (1)°.

The Cu^{II}—N(imidazole) bond distances of 1·962 (5), 1·960 (6) and 1·954 (6) Å are typical of other Cu^{II}—N(imidazole) bond lengths observed for complexes derived from bmima (Oberhausen, Richardson, Buchanan & Pierce, 1989) and other polyimidazole ligands (Doman, Richardson, Arar & Buchanan, 1989). Interestingly, the Cu—N(imidazole) lengths are longer for complexes containing more sterically constrained polyimidazole ligands, such as tris[2-(1-methylimidazolyl)methyl]-amine [2·062 (3), 2·021 (4) and 2·011 (2) Å] and bis[2-(1-methylimidazolyl)methyl][(2-pyridyl)methyl]-amine [2·009 (3) and 2·009 (3) Å] (Oberhausen, O'Brien, Richardson & Buchanan, 1990). The Cu—N(amine) bond distance in [Cu(bmima)(1-MeIm)]$^+$ is somewhat longer, 2·056 (5) Å, than those observed

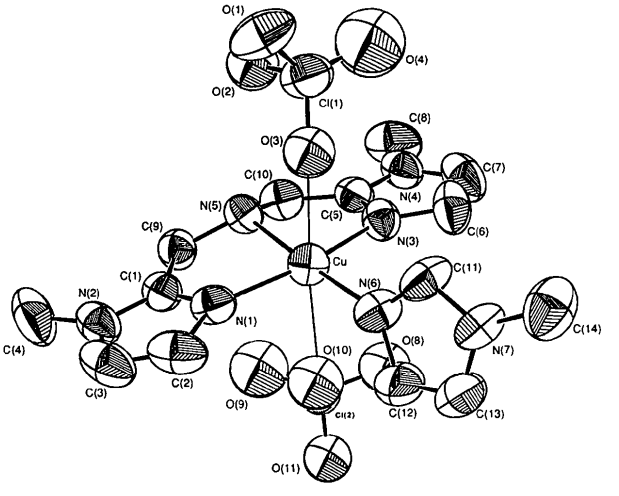


Fig. 1. *ORTEP* (Johnson, 1965) perspective view showing the title compound and the numbering scheme employed, 50% probability thermal ellipsoids. The H atoms and additional disorder models are omitted for clarity.

in other metal complexes of bmima. The N(5)—Cu—N(1), N(5)—Cu—N(3) and N(1)—Cu—N(3) bond angles of 81·2 (3), 81·7 (3) and 161·7 (3)°, respectively, reflect the strain imposed on the coordination environment of the copper center by the five-membered chelate rings of the bmima ligand. Similar angles have been observed in a structurally similar Cu–pyrazole complex (Driessen, de Graaff & Wiesmeijer, 1987). The C—C and C—N bond distances of the coordinated bmima and 1-MeIm ligands are similar to the lengths reported earlier for bmima (Richardson, Wilson-Blumenberg, Oberhausen, Mashuta & Buchanan, 1988).

The two perchlorate counter ions were found to be disordered. One perchlorate shows a rotational disorder about a common Cl(1)–O(1) axis in which two sets of three O atoms consisting of O(2), O(3), O(4) and O(5), O(6), O(7), respectively, were used to model the disorder problem. The Cl(1) and O(1) atoms were assigned occupancy factors of 1·0 during refinement while the remaining O-atom positions were given occupancies of 0·5 (these 'group' occupancies were fixed at the average of the individual occupancies, refined in least squares in earlier cycles, to obtain a reasonable physical and chemical model). Two of the disordered O atoms [O(3) and O(7)], were found to share a Cu coordination site with relatively long electrostatic contacts of 2·61 (2) and 2·55 (1) Å, respectively. These distances are within the range reported for such 'semi-coordination' as are the Cu—O—Cl angles describing the tilt of the ClO_4 group (Hathaway, Procter, Tomlinson, Brown, Lee & Melsom, 1967). O(2) is found to be hydrogen bonded to the amine N(5) H atom through both an intramolecular contact [N(5)—O(2) = 3·21 (1), O(2)—H(N5) = 2·11 (1) Å, N(5)—H(N5)—O(2) = 152·7 (4)°] and an intermolecular contact [O(2) at $-x, -y, 2-z$; N(5)—O(2)′ = 3·13 (1), O(2)′—H(N5) = 2·06 (1) Å, N(5)—H(N5)—O(2)′ = 147·9 (4)°]. The intermolecular hydrogen bond is indicated in Fig. 2. The disorder problem associated with the second perchlorate counter ion was successfully modeled using a common position for Cl(2) and two intersecting tetrahedral O_4 units [O(8) through O(11) and O(12) through O(15)] with the O atoms of one unit having occupancies fixed at 0·6 and the occupancies of the second unit fixed at 0·4 (average of refined individual atomic occupancies for each group). As with the first perchlorate, two of the O atoms O(10) and O(13) were found to be weakly associated with the copper ion, having Cu—O bond lengths of 2·69 (2) and 2·72 (1) Å, and Cu—O—Cl angles of 129·6 and 136·9°, respectively. This perchlorate was not found to be involved in any hydrogen bonding.

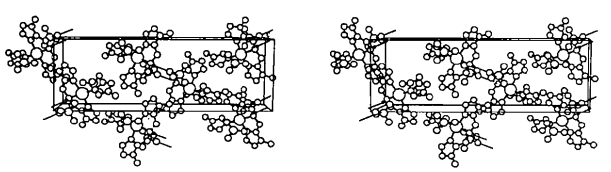


Fig. 2. Stereoview showing the molecular packing and the hydrogen bonding from H(N5) to O(2) of disordered ClO_4 related by the crystallographic center of symmetry. **a** is into the plane of the paper, **b** is vertical and **c** is horizontal.

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