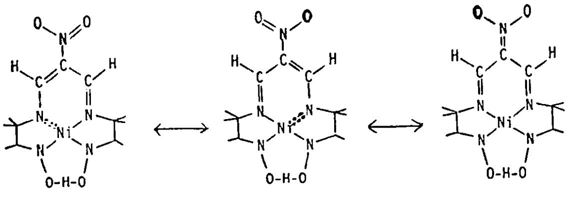
Ni—N(amine) distances [av. 1·856 (3) Å] in both forms are significantly shorter than the Ni—N(oxime) distances [av. 1·879 (4) Å] due to the aromaticity in the six-membered chelate ring, suggesting contributions from the three resonance forms (*a*) to (*c*).


(*a*) (*b*) (*c*)

Considerable multiple bonding, also consistent with aromaticity, in the N(amine)—C, C—C and C—N(nitro) bonds of the six-membered chelate ring is evident from the various bond distances, from the sum of the angles around N and C atoms, and from the planarity of the ring. The C(5) and C(7) atoms are sp^2 hybridized with coplanar H atoms, 1H(5) and 1H(7). In a related macrocyclic complex (Corfield, Mokren, Hipp & Busch, 1973) with a six-membered chelate ring in the boat configuration and a bonding scheme consistent with localized double bonds, the average N—C and C—C distances are 1·28 and 1·46 Å compared to 1·304 and 1·415 Å, respectively, in the present complex. The observed lengthening in the N—C distance and shortening in the C—C distance is expected on the basis of the charge delocalization in the chelate ring. Unusual chemical reactivity towards various electrophiles and NMR evidence also suggest aromatic nature of the six-membered ring.

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The Structure of Chlorobis(1,10-phenanthroline)copper(II) Perchlorate

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Abstract

$C_{24}H_{16}ClCuN_4^+.ClO_4^-$, $[Cu^{II}Cl(phen)_2]^+.ClO_4^-$, $M_r = 558·85$, monoclinic, $P2_1/c$, $a = 12·668\,(1)$, $b = 11·247\,(1)$, $c = 17·222\,(1)$ Å, $\beta = 111·50\,(1)°$, $V = 2283·0\,(1)$ Å^3, $Z = 4$, $D_m = 1·625$, $D_c = 1·635$ Mg m^{-3}. The structure was refined to $R = 0·072$ for 2573 reflections with $I > 2\sigma(I)$. The metal ion is pentacoordinated to the four N atoms of the bidentate chelates and to the chloride ion. The ligand atoms are arranged about the Cu atom in what can be best regarded as a distorted trigonal-bipyramidal con-

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figuration with bond lengths Cu—N, 2·004 (6), 2·136 (6), 1·986 (6) and 2·077 (6) Å, and Cu—Cl, 2·298 (2) Å. The perchlorate ion is not coordinated.

Introduction

Considerable attention has been focused on mechanistic schemes for complex formation. In order to provide more data on the configurations and structural properties of metallic coordination compounds, the crystal structure of the title compound is reported in this paper. This structural study is part of a series aimed at an examination of the coordination of bidentate ligands and various donor atoms around an oxidized metal ion.

Experimental

Single crystals were prepared and kindly supplied by Mrs E. Spodine of the Universidad de Chile. They are transparent, prismatic and bright deep green. The density was measured by the flotation method in a carbon tetrachloride/bromoform mixture at room temperature. The space-group symmetry and initial lattice parameters were determined from precession photographs, the lattice parameters being later refined by a least-squares fit using 34 reflections recorded on the diffractometer. A small crystal, with similar dimensions in all directions (approximate diameter 0·3 mm), was chosen for the intensity measurements taken on a Philips PW 1100 four-circle automatic diffractometer, in the $\omega/2\theta$ scan mode, with graphite-monochromated Cu $K\alpha$ radiation (λ = 1·54180 Å). 2680 independent reflections with $\theta < 55°$ were recorded at room temperature; of these, 2573 with $I > 2\sigma(I)$ were used in the structure refinement. Intensities were corrected for Lorentz and polarization effects in the usual manner. No absorption correction was made because of the small size of the crystal with a linear absorption coefficient μ(Cu $K\alpha$) of 3·88 mm^{-1} ($\mu R \sim$ 0·6). No correction for extinction was made.

Structure determination and refinement

The structure was solved using the multisolution tangent method with the *MULTAN* 76 system of programs (Main, Lessinger, Woolfson, Germain & Declercq, 1976). The molecular geometry of the phenanthroline and perchlorate groups was input to the program. The statistical distribution of $|E|$ values confirmed a centrosymmetric structure and 300 reflections with $|E| > 1·5$ were used for phase determination.

The phase set with the largest combined figure of merit and lowest residual gave the positions of 28 of the 35 non-hydrogen atoms of the asymmetric unit.

It was not possible to locate the four O atoms of the perchlorate ion. A structure factor calculation at this stage, based on these 28 atom coordinates, with the scale and overall temperature factors evaluated by the program, gave a value of $R = \sum||F_o| - |F_c||/\sum |F_o|$ of 0·31.

The missing non-hydrogen atoms were obtained from a difference Fourier synthesis, and isotropic refinement of the structure proceeded by block-diagonal least-squares methods with fixed H atoms. Their coordinates were generated by the program *BONDAT* (Kruger & Stewart, 1976) and they were given the isotropic temperature factors of their parent C atoms. Subsequent cycles of refinement included anisotropic temperature factors for all non-hydrogen atoms.

During the process of refinement, considerable motion of the O atoms in the perchlorate ion was revealed. On the basis of careful examinations of difference syntheses around the Cl atom, which refined well, occupational disorder for the O atoms was tried without any success. Refinement was thus considered complete when all shifts did not exceed 10% of the corresponding e.s.d.'s of the parameters. A final unweighted R index of 0·072 for all observed reflections was obtained.* A difference Fourier map, based on the last set of parameters, contained the highest residuals of electron density of heights +1·41 and +0·80 e Å^{-3} at distances of 1·2 and 1·0 Å distributed centrosymmetrically around the Cu^{2+} and Cl$^-$ ions

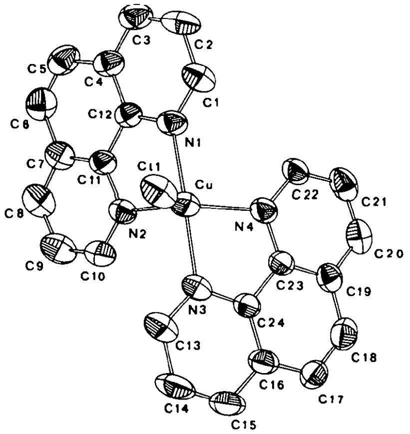

Fig. 1. Structure of the complex cation viewed along the b axis with the atom-numbering scheme and thermal ellipsoids at the 50% probability level. H atoms have been omitted for clarity.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35712 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

respectively. The next two highest peaks (less than 0·55 e Å^{-3}) were found in the vicinity of the perchlorate O atoms.

Scattering factors for non-hydrogen neutral atoms and for Cu^{2+} and Cl$^-$ were taken from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965). Values for the real and imaginary parts of the dispersion correction of the scattering factors for Cu and Cl atoms were taken from *International Tables for X-ray Crystallography* (1974). Crystallographic calculations involved in the structure refinement were performed with the NRC system of programs (Ahmed, Hall, Pippy & Huber, 1973). Computations were carried out on an IBM 370/145 computer at the Computer Centre of the Universidad de Chile.

Table 1. *Fractional coordinates* (×10^4), *and isotropic thermal parameters* (Å^2 × 10^3) *for non-hydrogen atoms, with e.s.d.'s in parentheses*

	x	y	z	$\bar{U}$*
Cu	7894 (1)	7673 (1)	5735 (1)	50
Cl(1)	7921 (2)	9635 (2)	5369 (1)	60
N(1)	9300 (5)	7347 (5)	5496 (4)	48
N(2)	7243 (5)	6373 (5)	4774 (4)	46
N(3)	6463 (5)	7887 (6)	5943 (4)	52
N(4)	8426 (5)	6960 (5)	6930 (4)	47
C(1)	10315 (6)	7827 (7)	5870 (5)	56
C(2)	11191 (6)	7667 (8)	5578 (6)	63
C(3)	11025 (7)	6969 (8)	4891 (5)	61
C(4)	9958 (6)	6440 (7)	4487 (5)	51
C(5)	9691 (8)	5718 (8)	3742 (5)	63
C(6)	8643 (8)	5263 (8)	3374 (5)	65
C(7)	7780 (6)	5431 (7)	3709 (5)	54
C(8)	6680 (7)	4968 (8)	3358 (6)	68
C(9)	5909 (7)	5199 (8)	3721 (6)	70
C(10)	6214 (6)	5937 (7)	4424 (5)	56
C(11)	8013 (6)	6138 (6)	4427 (4)	45
C(12)	9125 (6)	6642 (6)	4816 (4)	43
C(13)	5494 (7)	8339 (9)	5442 (5)	70
C(14)	4556 (6)	8482 (9)	5670 (6)	73
C(15)	4609 (6)	8077 (8)	6434 (6)	65
C(16)	5607 (6)	7535 (7)	6970 (5)	52
C(17)	5729 (6)	7015 (8)	7762 (5)	55
C(18)	6714 (7)	6515 (7)	8240 (5)	58
C(19)	7675 (6)	6454 (7)	7997 (4)	48
C(20)	8728 (7)	5998 (7)	8475 (5)	59
C(21)	9605 (7)	6028 (8)	8198 (5)	63
C(22)	9427 (6)	6550 (7)	7408 (5)	50
C(23)	7572 (6)	6953 (6)	7222 (4)	43
C(24)	6512 (5)	7446 (7)	6700 (4)	44
Cl(2)	7178 (2)	3196 (3)	6761 (1)	79
O(1)	8280 (6)	3035 (13)	6965 (6)	199
O(2)	6939 (8)	3690 (10)	7409 (6)	168
O(3)	6833 (13)	3855 (16)	6077 (8)	272
O(4)	6415 (12)	2293 (17)	6463 (9)	273

* $8\pi^2 \bar{U} = B_{eq}$, where B_{eq} is the equivalent isotropic temperature factor calculated from the anisotropic temperature coefficients (Hamilton, 1959).

Results and discussion

The final positional and isotropic thermal parameters of all non-hydrogen atoms are listed in Table 1. Interatomic distances and bond angles are given in Table 2.

The structure is made up of two separate entities: the [Cu^{II}Cl(phen)$_2$]$^+$ complex cation and the perchlorate anion. Fig. 1 presents the molecular configuration of the complex ion showing the main geometrical features

Table 2. *Intramolecular distances* (Å) *and bond angles* (°) *for non-hydrogen atoms, with e.s.d.'s in parentheses*

(*a*) Metal coordination

Cu—Cl(1)	2·298 (2)		
Cu—N(1)	2·004 (6)	Cu—N(3)	1·986 (6)
Cu—N(2)	2·136 (6)	Cu—N(4)	2·077 (6)
N(1)—Cu—N(2)	80·5 (2)	N(3)—Cu—N(4)	81·7 (2)
N(1)—Cu—Cl(1)	90·9 (2)	N(3)—Cu—Cl(1)	92·3 (2)
N(1)—Cu—N(4)	98·2 (2)	N(3)—Cu—N(2)	96·0 (2)

(*b*) Phenanthroline groups

N(1)—C(1)	1·324 (10)	N(3)—C(13)	1·316 (11)
N(1)—C(12)	1·363 (9)	N(3)—C(24)	1·375 (9)
N(2)—C(10)	1·314 (10)	N(4)—C(22)	1·317 (10)
N(2)—C(11)	1·344 (10)	N(4)—C(23)	1·350 (10)
C(1)—C(2)	1·388 (12)	C(13)—C(14)	1·392 (13)
C(2)—C(3)	1·371 (13)	C(14)—C(15)	1·370 (13)
C(3)—C(4)	1·406 (12)	C(15)—C(16)	1·402 (12)
C(4)—C(5)	1·449 (12)	C(16)—C(17)	1·439 (11)
C(4)—C(12)	1·388 (11)	C(16)—C(24)	1·390 (11)
C(5)—C(6)	1·345 (14)	C(17)—C(18)	1·341 (12)
C(6)—C(7)	1·423 (13)	C(18)—C(19)	1·427 (12)
C(7)—C(8)	1·400 (13)	C(19)—C(20)	1·385 (12)
C(7)—C(11)	1·408 (11)	C(19)—C(23)	1·409 (10)
C(8)—C(9)	1·364 (14)	C(20)—C(21)	1·359 (13)
C(9)—C(10)	1·400 (12)	C(21)—C(22)	1·422 (11)
C(11)—C(12)	1·436 (11)	C(23)—C(24)	1·427 (10)
C(1)—N(1)—C(12)	118·5 (6)	C(13)—N(3)—C(24)	117·9 (7)
C(10)—N(2)—C(11)	118·9 (7)	C(22)—N(4)—C(23)	119·0 (6)
N(1)—C(1)—C(2)	122·3 (7)	N(3)—C(13)—C(14)	123·2 (8)
C(1)—C(2)—C(3)	119·8 (8)	C(13)—C(14)—C(15)	119·1 (9)
C(2)—C(3)—C(4)	119·0 (8)	C(14)—C(15)—C(16)	119·5 (8)
C(3)—C(4)—C(12)	117·8 (7)	C(15)—C(16)—C(24)	117·8 (7)
C(5)—C(4)—C(12)	119·2 (7)	C(17)—C(16)—C(24)	118·2 (7)
C(4)—C(5)—C(6)	120·1 (8)	C(16)—C(17)—C(18)	120·4 (8)
C(5)—C(6)—C(7)	122·0 (8)	C(17)—C(18)—C(19)	122·8 (8)
C(6)—C(7)—C(11)	119·1 (7)	C(18)—C(19)—C(23)	117·8 (7)
C(8)—C(7)—C(11)	116·4 (7)	C(20)—C(19)—C(23)	116·4 (7)
C(7)—C(8)—C(9)	119·8 (8)	C(19)—C(20)—C(21)	121·0 (8)
C(8)—C(9)—C(10)	119·6 (8)	C(20)—C(21)—C(22)	118·9 (8)
C(9)—C(10)—N(2)	122·0 (8)	C(21)—C(22)—N(4)	121·4 (7)
N(2)—C(11)—C(7)	123·3 (7)	N(4)—C(23)—C(19)	123·2 (7)
C(7)—C(11)—C(12)	119·2 (7)	C(19)—C(23)—C(24)	119·4 (7)
N(1)—C(12)—C(4)	122·5 (7)	N(3)—C(24)—C(16)	122·3 (7)
C(4)—C(12)—C(11)	120·5 (7)	C(16)—C(24)—C(23)	121·2 (7)

(*c*) Perchlorate ion

Cl(2)—O(1)	1·322 (9)	Cl(2)—O(3)	1·324 (15)
Cl(2)—O(2)	1·376 (11)	Cl(2)—O(4)	1·365 (18)
O(1)—Cl(2)—O(2)	111·4 (7)	O(2)—Cl(2)—O(3)	113·4 (8)
O(1)—Cl(2)—O(3)	106·5 (9)	O(2)—Cl(2)—O(4)	105·6 (8)
O(1)—Cl(2)—O(4)	122·1 (9)	O(3)—Cl(2)—O(4)	97·2 (10)

of the coordination polyhedron together with the atom-labelling sequence used.

The $[Cu^{II}Cl(phen)_2]^+$ *cation*

As illustrated in Fig. 1, the complex cation assumes a distorted five-coordinate stereochemistry. The central Cu^{2+} ion binds the Cl^- ion and the two bidentate phenanthroline ligands (phen 1 and phen 2) *via* the N atoms, with bond distances and angles (Table 2) which agree well with those found in other Cu^{II} phenanthroline complexes (Anderson, 1973, 1975; Battaglia, Bonamartini Corradi, Marcotrigiano & Pellacani, 1977). N(2) and N(4), from phen 1 and phen 2 respectively, together with the Cl^- ion, are located in equatorial positions while the remaining two phenanthroline N atoms, N(1) and N(3) from phen 1 and phen 2 respectively, occupy the axial sites with a non-linear N(1)—Cu—N(3) angle of 176·2 (3)°. The main distortion in the bipyramidal configuration arises from the need of a N—Cu—N angle for the phenanthroline ligands of approximately 81° which removes the N atoms from the ideal axial positions approximately 9° in opposite directions. Also, the angles in the equatorial plane exhibit some distortion from the ideal trigonal angle of 120°. All relevant bond distances and angles in the coordination polyhedron are illustrated in Fig. 2.

Bond distances and angles involved in the phenanthroline ligands are normal (Table 2), with mean C—C distances of 1·400 (2) Å, mean C—N of 1·340 (3) Å, and mean intraligand angles: C—C—C = 119·3 (2), C—N—C = 118·6 (3), N—C—C = 122·5 (2)°. Phen 1 and phen 2 are reasonably planar and inclined at angles of 117·7 and 92·1° respectively, with respect to the equatorial mean plane. Details of the least-squares planes of the phenanthroline groups and the equatorial plane of the bipyramidal configuration are given in Table 3. Phen 2 exhibits major deviations from planarity, while the central metal ion and the three equatorial coordinating atoms are essentially coplanar, the maximum deviation from the least-squares plane being 0·005 (1) Å for the Cu^{2+} ion.

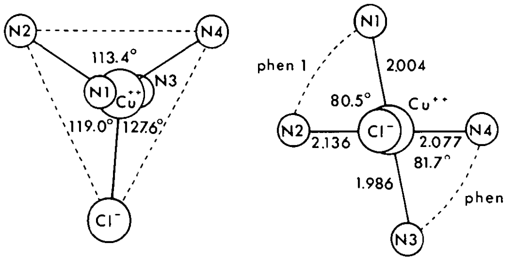

Fig. 2. Top and front views of the trigonal-bipyramidal coordination around the metal ion.

Table 3. *Least-squares planes and dihedral angles*

(*a*) Equations of the planes given by $lX + mY + nZ - p = 0$ where X, Y, Z are the orthogonal coordinates in Å along the *a*, *b*, and c^* axes

Plane 1 (phen 1)

$$-0{\cdot}0925X + 0{\cdot}8111Y - 0{\cdot}5775Z - 0{\cdot}8343 = 0$$

Plane 2 (phen 2)

$$-0{\cdot}1573X - 0{\cdot}8927Y - 0{\cdot}4223Z + 12{\cdot}6705 = 0$$

Plane 3 (trigonal base)

$$0{\cdot}9918X - 0{\cdot}1267Y - 0{\cdot}0155Z - 5{\cdot}0864 = 0$$

(*b*) Deviations (Å) of atoms from mean planes and dihedral angles (°). Daggers indicate atoms omitted from the calculations of the least-squares planes.

Plane 1		Plane 2		Plane 3	
N(1)	0·013 (6)	N(3)	0·032 (6)	Cu	0·005 (1)
N(2)	−0·008 (6)	N(4)	0·001 (6)	Cl(1)	−0·002 (2)
C(1)	0·008 (8)	C(13)	0·061 (9)	N(1)	1·974 (6)†
C(2)	0·012 (9)	C(14)	−0·028 (10)	N(2)	−0·002 (6)
C(3)	−0·010 (9)	C(15)	−0·072 (9)	N(3)	−1·958 (6)†
C(4)	−0·017 (8)	C(16)	−0·037 (8)	N(4)	−0·002 (6)
C(5)	0·002 (9)	C(17)	0·003 (8)		
C(6)	0·029 (9)	C(18)	0·033 (8)		
C(7)	−0·007 (8)	C(19)	0·044 (7)		
C(8)	0·004 (9)	C(20)	0·016 (8)		
C(9)	−0·010 (9)	C(21)	−0·029 (9)		
C(10)	0·018 (8)	C(22)	−0·062 (8)		
C(11)	−0·012 (7)	C(23)	0·010 (7)		
C(12)	−0·021 (7)	C(24)	0·028 (7)		
Cu	0·268 (1)†	Cu	0·082 (1)†		
Cl(1)	2·372 (2)†	Cl(1)	−1·681 (2)†		
N(3)	0·451 (6)†	N(1)	0·267 (6)†		
N(4)	−1·481 (6)†	N(2)	2·073 (6)†		

Planes	Dihedral angles
1–2	117·75
1–3	100·69
2–3	92·08

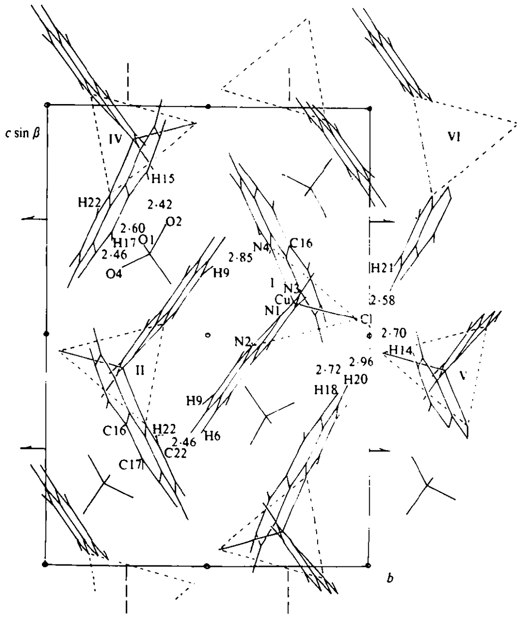

Fig. 3. Projection of the structure down the *a* axis. Broken lines indicate the trigonal base of the bipyramidal configuration.

The perchlorate ion

As pointed out before, the O atoms exhibit relatively high thermal motion but no definite indication of occupational disorder was found. The Cl—O distances and O—Cl—O angles, uncorrected for thermal motion, given in Table 2 have averages of 1·343 (6) Å and 109·9 (3)° respectively. The range of distances and angles of this highly distorted tetrahedron are comparable with those given by Pyżalska, Gawron & Borowiak (1979) and Fair & Schlemper (1978) respectively.

Intermolecular contacts

Besides the ionic forces, due to the ionic character of the structure, the cohesion of the crystal is achieved through van der Waals interactions.

Phen 1 (II) and phen 2 (III) (Fig. 3) exhibit several close contacts between their C atoms, the shorter distances being indicated in Table 4. The van der Waals interactions between the two phen 1 groups related by the centre of symmetry, and 3·56 (2) Å apart, are due to a partial overlapping of the C rings (estimated at 35%).

Considering the refined positions of the O atoms with appropriate caution, some O—C contacts, nevertheless, deserve attention. Three O atoms of the perchlorate ion have distances to the C atoms which are shorter than the sum of their usual van der Waals radii. The geometrically located H atom bonded to the C atom is not far from the line joining the latter with the O atom, indicating the occurrence of van der Waals C···O contacts *via* H atoms. The angles of deviation of the C—H bonds with respect to these lines, together with the distances involved in these contacts, are summarized in Table 4. Similar interactions are found between the chloride ion and some C atoms.

Table 4. *Selected intermolecular distances* (Å) *less than the sum of the van der Waals radii*

The angle (°) H—C··· is also given.

O(1)–C(22)^{iv}	3·18 (1)	O(1)–H(22)^{iv}	2·60	47·9
O(2)–C(15)^{iv}	3·34 (1)	O(2)–H(15)^{iv}	2·42	19·1
O(4)–C(17)^{iv}	3·45 (2)	O(4)–H(17)^{iv}	2·46	5·3
Cl(1)–C(21)^{vi}	3·56 (1)	Cl(1)–H(21)^{vi}	2·58	9·0
Cl(1)–C(18)^{iii}	3·65 (1)	Cl(1)–H(18)^{iii}	2·72	18·6
Cl(1)–C(14)^{v}	3·66 (1)	Cl(1)–H(14)^{v}	2·70	12·2
C(11)–C(18)^{iii}	3·37 (1)	C(4)–C(20)^{iii}	3·43 (1)	
C(12)–C(20)^{iii}	3·43 (1)			

Symmetry code

None x,y,z
(ii) $1-x, 1-y, 1-z$
(iii) $x, \frac{3}{2}-y, -\frac{1}{2}+z$
(iv) $1-x, -\frac{1}{2}+y, \frac{3}{2}-z$
(v) $1-x, 2-y, 1-z$
(vi) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$

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