of the charge-transfer interaction for the present complex has been carried out.

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N-Methylaminoethanolatobis(ethylenediamine)cobalt(III) Perchlorate Monohydrate

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Abstract. Rac-[Co(*N*-Me-eta) (en)$_2$] (ClO$_4$)$_3$.H$_2$O, triclinic, $P\bar{1}$, $a=9·091$ (3), $b=13·689$ (3), $c=8·665$ (3) Å, $\alpha=96·34$ (3), $\beta=125·52$ (2), $\gamma=76·72$ (3)°, $Z=2$, $D_m=1·75$, $D_x=1·75$ g cm^{-3}. The cobalt atom is surrounded nearly octahedrally by five nitrogen atoms and an oxygen atom. Average Co–N distance is 1·976 (4) Å and Co–O is 1·879 (4) Å. The Co–N–C(methyl) angle of 121·4 (4)° is largely deviated from the normal tetrahedral angle owing to the repulsion between the methyl carbon atom and the amino nitrogen atoms.

Introduction. Red-purple platy crystals were grown from an aqueous solution. Intensities and cell dimensions were measured on a Rigaku automated four-circle diffractometer with a crystal of dimensions 0·1 × 0·3 × 0·3 mm. Data were collected by the ω–2θ scan technique to a maximum 2θ value of 55° with Mo $K\alpha$ radiation ($\lambda=0·7107$ Å) monochromated by a graphite plate. 5223 reflexions were measured of which 3663 ($|F|>3\sigma$) were classified as 'observed'. The intensities were corrected for Lorentz–polarization factors. No correction was made for absorption [μ(Mo $K\alpha$) = 13·5 cm^{-1}]. All the positions of non-hydrogen atoms were located by routine Patterson and Fourier syntheses assuming space group $P\bar{1}$. Refinement of the structure was carried out by block-diagonal least-squares techniques. Positional parameters of the non-hydrogen atoms were refined with anisotropic thermal factors. The structure converged at $R=0·073$. A difference synthesis gave the positions of all the hydrogen atoms: during further refinement the atomic parameters of the hydrogen atoms were allowed to vary by assuming isotropic thermal motion. The final discrepancy index (3663 reflexions) became 0·054. A final difference map showed no peaks greater than 0·6 e Å^{-3}. The atomic scattering factors for non-hydrogen atoms were those listed in *International Tables for X-ray Crystallography* (1962). For hydrogen atoms the values given by Stewart, Davidson & Simpson (1965) were used. Atomic coordinates and thermal parameters are listed in Table 1.‡

The bond lengths and angles are listed in Tables 2 and 3. A projection of the structure along the *a* axis is shown in Fig. 1, together with the labelling of the atoms. Table 2 lists the interatomic distances and

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‡ A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31468 (22 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Positional and thermal parameters*

(*a*) Fractional atomic coordinates (× 10^4) and thermal parameters (× 10^3) for the non-hydrogen atoms, with estimated standard deviations in parentheses. Thermal parameters are of the form $T=\exp[-2\pi^2(a^{*2}h^2U_{11}+b^{*2}k^2U_{22}+c^{*2}l^2U_{33}+2a^*b^*hkU_{12}+2a^*c^*hlU_{13}+2b^*c^*klU_{23})]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co	4226 (1)	2049 (0)	4123 (1)	22 (0)	17 (0)	25 (0)	−3 (0)	13 (0)	2 (0)
Cl(1)	8452 (2)	4703 (1)	1242 (2)	37 (1)	32 (1)	84 (1)	−3 (0)	31 (1)	2 (1)
Cl(2)	1660 (2)	979 (1)	7470 (2)	38 (1)	42 (1)	33 (1)	−11 (0)	20 (0)	−2 (0)
O(0)	4980 (4)	1200 (2)	6074 (4)	39 (2)	22 (1)	25 (1)	−6 (1)	15 (1)	2 (1)
N(1)	4801 (5)	3158 (3)	5916 (5)	33 (2)	21 (2)	36 (2)	−7 (1)	21 (2)	−3 (1)
N(2)	3536 (5)	2912 (3)	2048 (5)	30 (2)	24 (2)	34 (2)	−1 (1)	18 (2)	6 (1)
N(3)	6603 (5)	1777 (3)	4459 (5)	26 (2)	27 (2)	34 (2)	0 (1)	17 (2)	4 (1)
N(4)	1803 (5)	2280 (3)	3603 (5)	28 (2)	29 (2)	36 (2)	−3 (1)	18 (2)	3 (2)
N(5)	3676 (4)	852 (3)	2626 (5)	29 (2)	22 (2)	25 (2)	−4 (1)	13 (1)	2 (1)
C(1)	3648 (7)	4181 (3)	5458 (8)	57 (3)	18 (2)	57 (3)	−2 (2)	31 (3)	−6 (2)
C(2)	5010 (7)	2757 (4)	7578 (7)	57 (3)	31 (2)	34 (2)	−13 (2)	28 (2)	−7 (2)
C(3)	5919 (7)	1667 (3)	7731 (6)	44 (3)	32 (2)	26 (2)	−9 (2)	15 (2)	1 (2)
C(4)	5158 (7)	3128 (4)	2262 (8)	40 (3)	45 (3)	60 (3)	−4 (2)	30 (3)	21 (2)
C(5)	6529 (7)	2183 (4)	2897 (8)	39 (3)	52 (3)	57 (3)	2 (2)	34 (3)	17 (3)
C(6)	775 (6)	1689 (4)	2021 (7)	28 (2)	40 (3)	40 (3)	−7 (2)	12 (2)	3 (2)
C(7)	1958 (6)	684 (4)	2232 (7)	34 (2)	33 (2)	41 (3)	−15 (2)	16 (2)	0 (2)
O(1)	10075 (6)	4256 (4)	1405 (8)	55 (3)	75 (3)	114 (4)	26 (2)	54 (3)	39 (3)
O(2)	8057 (11)	4102 (5)	2160 (13)	202 (8)	79 (4)	282 (10)	−28 (5)	208 (8)	9 (5)
O(3)	7122 (8)	4760 (4)	−684 (9)	75 (4)	72 (4)	117 (5)	−5 (3)	−19 (3)	−5 (3)
O(4)	8451 (7)	5699 (3)	1926 (8)	96 (4)	47 (3)	120 (5)	−26 (3)	63 (4)	−27 (3)
O(5)	2584 (14)	190 (5)	7056 (18)	244 (10)	78 (5)	428 (17)	−57 (6)	280 (12)	−101 (7)
O(6)	408 (7)	755 (4)	7817 (7)	72 (3)	114 (4)	84 (3)	−41 (3)	50 (3)	14 (3)
O(7)	2821 (9)	1510 (5)	8910 (8)	143 (6)	142 (6)	78 (4)	−101 (5)	66 (4)	−53 (4)
O(8)	718 (8)	1609 (7)	5920 (9)	67 (4)	292 (11)	93 (5)	−15 (5)	29 (3)	111 (6)
O(*W*)	8424 (7)	3426 (4)	7155 (8)	72 (3)	88 (4)	126 (5)	−47 (3)	59 (3)	−44 (3)

bond angles within the complex cation. The complex cations, the perchlorate ions and water molecules are arranged to form layers parallel to the plane (100). In the layers the complex cations lie between the planes formed by perchlorate ions and water molecules. The layers are held together by hydrogen bonding. They are listed in Table 3. The crystals cleave easily along this plane in conformity with such a layer structure. A cobalt atom is surrounded nearly octahedrally by five nitrogen atoms and an oxygen atom. The *N*-Me-eta molecule is coordinated to the cobalt atom with its nitrogen atom and oxygen atom at 1·955 (4) and 1·879 (3) Å, respectively. The resulting five membered chelate ring is puckered. The ethylenediamine molecules assume *gauche* conformation with a mean azimuthal angle of 49·6° between the planes determined by C–C and C–N bonds. This angle is smaller than the value of 55·1° observed in $[Co(en)_3]Cl_3.H_2O$ (Iwata, Nakatsu & Saito, 1969). The geometry of the

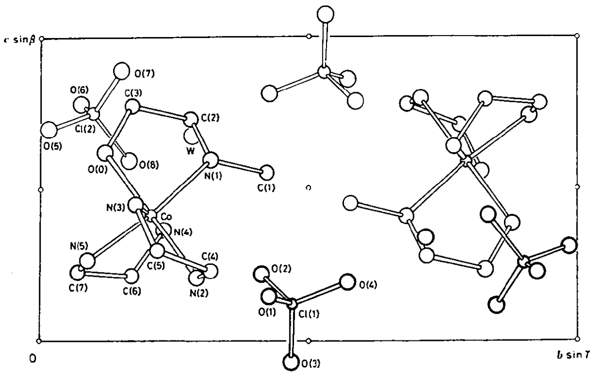

Fig. 1. A projection of the structure viewed along the *a* axis.

Table 1 (*cont.*)

(*b*) Positional parameters for the hydrogen atoms (× 10^3) with estimated standard deviations. Mean isotropic temperature factor of the hydrogen atoms is 4·5 (1·4) $Å^2$.

	x	y	z
H(1)	235 (8)	409 (4)	515 (8)
H(2)	424 (8)	452 (6)	471 (11)
H(3)	420 (8)	460 (4)	645 (8)
H(4)	594 (6)	328 (4)	629 (7)
H(5)	579 (6)	314 (4)	871 (6)
H(6)	354 (8)	286 (4)	727 (8)
H(7)	726 (6)	161 (4)	817 (7)
H(8)	617 (8)	131 (4)	874 (8)
H(9)	292 (6)	261 (3)	97 (6)
H(10)	249 (9)	340 (5)	166 (9)
H(11)	472 (8)	334 (5)	88 (8)
H(12)	542 (9)	375 (5)	332 (9)
H(13)	784 (7)	223 (4)	332 (7)
H(14)	595 (9)	165 (5)	169 (9)
H(15)	745 (7)	200 (4)	556 (7)
H(16)	697 (8)	125 (4)	456 (8)
H(17)	195 (7)	215 (4)	462 (7)
H(18)	154 (9)	286 (5)	377 (9)
H(19)	−53 (8)	161 (5)	193 (8)
H(20)	48 (7)	201 (4)	89 (7)
H(21)	232 (7)	36 (4)	334 (7)
H(22)	151 (7)	24 (4)	108 (7)
H(23)	370 (6)	82 (4)	170 (7)
H(24)	471 (7)	32 (4)	321 (8)
H(25)	886 (9)	372 (5)	800 (9)
H(26)	862 (8)	342 (5)	651 (9)

Table 2. *Interatomic distances and bond angles in the complex ion and the perchlorate ions with their estimated standard deviations in parentheses*

Co···O(0)	1·879 (3) Å	C(2)–C(3)	1·513 (6) Å
Co···N(1)	1·955 (4)	C(4)–C(5)	1·492 (7)
Co···N(2)	1·990 (4)	C(6)–C(7)	1·496 (7)
Co···N(3)	1·974 (4)		
Co···N(4)	1·957 (4)	Cl(1)–O(1)	1·421 (6)
Co···N(5)	1·962 (3)	Cl(1)–O(2)	1·406 (12)
O(0)–C(3)	1·400 (5)	Cl(1)–O(3)	1·454 (6)
N(1)–C(1)	1·494 (5)	Cl(1)–O(4)	1·426 (5)
N(1)–C(2)	1·500 (8)	Cl(2)–O(5)	1·347 (11)
N(2)–C(4)	1·482 (8)	Cl(2)–O(6)	1·421 (8)
N(3)–C(5)	1·482 (9)	Cl(2)–O(7)	1·402 (6)
N(4)–C(6)	1·472 (6)	Cl(2)–O(8)	1·412 (8)
N(5)–C(7)	1·480 (8)		

O(0)–Co···N(1)	86·7 (1)°	C(1)–N(1)–C(2)	109·9 (4)°
N(2)–Co···N(3)	85·1 (2)	N(1)–C(2)–C(3)	105·5 (4)
N(4)–Co···N(5)	85·3 (1)	O(0)–C(3)–C(2)	108·8 (2)
Co···O(0)–C(3)	110·9 (3)	N(2)–C(4)–C(5)	107·6 (3)
Co···N(1)–C(1)	121·4 (2)	N(3)–C(5)–C(4)	108·4 (5)
Co···N(1)–C(2)	105·6 (3)	N(4)–C(6)–C(7)	106·2 (3)
Co···N(2)–C(4)	107·2 (3)	N(5)–C(7)–C(6)	108·7 (3)
Co···N(3)–C(5)	110·3 (4)		
Co···N(4)–C(6)	109·7 (3)		
Co···N(5)–C(7)	109·5 (3)		

Table 3. *Interatomic distances less than* 3·5 Å *outside the complex ions*

Key to symmetry operations

(i)	x,	y,	z	(v)	x,	y,	$-1+z$
(ii)	$1-x$,	$1-y$,	$1-z$	(vi)	$-1+x$,	y,	z
(iii)	$1-x$,	$-y$,	$1-z$	(vii)	x,	y,	$1+z$
(iv)	$1+x$,	y,	z				

O(0^{i})–N(5^{iii})	2·887 (4) Å*	O(1^{i})—C(6^{iv})	3·473 (7) Å
O(0^{i})–C(7^{iii})	3·135 (5)	O(2^{i})—N(4^{iv})	3·422 (8)
N(3^{i})–O(5^{iii})	2·973 (10)*	O(W^{i})–O(8^{iv})	3·323 (11)
N(5^{i})–C(3^{iii})	3·379 (6)	N(2^{i})—O(7^{v})	3·012 (7)*
N(5^{i})–O(5^{iii})	3·248 (14)	N(5^{i})—O(7^{v})	3·077 (9)*
C(3^{i})–C(7^{iii})	3·345 (6)	C(4^{i})—O(7^{v})	3·488 (8)
C(5^{i})–O(5^{iii})	3·162 (9)	C(7^{i})—O(6^{v})	3·318 (8)
N(3^{i})–O(6^{iv})	3·247 (5)	O(1^{i})—O(W^{v})	3·345 (9)
N(3^{i})–O(8^{iv})	3·219 (8)	O(3^{i})—N(1^{v})	3·483 (7)
C(5^{i})–O(8^{iv})	3·260 (7)	O(3^{i})—C(1^{v})	3·360 (7)
O(1^{i})–N(2^{iv})	3·054 (7)*	O(3^{i})—C(2^{v})	3·456 (8)
O(1^{i})–N(4^{iv})	3·076 (6)*	O(3^{i})—O(W^{v})	2·970 (11)*
O(1^{i})–C(1^{iv})	3·300 (6)		

* An asterisk indicates hydrogen bonding.

whole complex ion can be designated as *lel*$_3$, since all the three central C–C bonds are nearly parallel to the pseudo-threefold axis of the complex ion. One remarkable feature is that the angle Co–N(1)–C(1) is as large as 121·4°. This distortion of the N–C(methyl) bond may be due to the repulsion between the methyl carbon atom C(1) and the amino nitrogen atoms N(2) and N(4) of the adjacent chelate rings.

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