BATTELLE, L. F., BAU, R., GOKEL, G. W., OYAKAWA, R. T. & UGI, I. K. (1973). *J. Am. Chem. Soc.* **95**, 482–486.
BÜRGI, H. B., DUNITZ, J. D. & SHEFTER, E. (1973). *Cryst. Struct. Commun.* **2**, 667–672.
BYE, E. (1974). *Acta Chem. Scand. Ser. B*, **28**, 5–12.
CHURCHILL, M. R. & KALRA, K. L. (1973). *Inorg. Chem.* **12**, 1650–1656.
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CULLEN, W. R. & YEH, E-S. (1977). *J. Organomet. Chem.* **139**, C13–C16.
CULLEN, W. R., YEH, E-S., EINSTEIN, F. W. B., HUANG, C.-H. & WILLIS, A. C. (1979). Unpublished results.
DALY, J. J. (1964). *J. Chem. Soc.* pp. 3799–3810.
DUNITZ, J. D., ORGEL, L. E. & RICH, A. (1956). *Acta Cryst.* **9**, 373–375.
EINSTEIN, F. W. B. & JONES, R. D. G. (1972). *Inorg. Chem.* **11**, 395–400.
FRYZUK, M. D. & BOSNICH, B. (1977). *J. Am. Chem. Soc.* **99**, 6262–6267.
HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
HAYASHI, T., YAMAMOTO, K. & KUMADA, M. (1974). *Tetrahedron Lett.* pp. 4405–4408.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
KRUKONIS, A. P., SILVERMAN, J. & YANNONI, N. F. (1972). *Acta Cryst.* B**28**, 987–990.
SINOU, D. & KAGAN, H. B. (1976). *J. Organomet. Chem.* **114**, 325–337.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Crystal Structure of $(-)_{589}$ Ammineglycinato(1,4,7-triazacyclononane)cobalt(III) Diiodide 0·84-Hydrate

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Abstract

Crystals of the title compound, $[Co(C_6H_{15}N_3)(C_2H_4NO_2)(NH_3)]I_2.0{\cdot}84H_2O$, are orthorhombic, $P2_12_12_1$, with $a = 15{\cdot}075$ (3), $b = 17{\cdot}674$ (4), $c = 12{\cdot}625$ (3) Å and $Z = 8$. The structure was refined by a block-diagonal least-squares method to a final R value of 0·0253 for 4675 independent reflections with $|F_o| > 3\sigma(|F_o|)$ collected by X-ray diffractometry. The absolute configuration of the complex ion $(-)_{589}[Co(gly)(NH_3)(tacn)]^{2+}$ (gly: glycinate; tacn: 1,4,7-triazacyclononane) can be designated as *a*-ammine-*bc*-glycinato-*def*-(1,4,7-triazacyclononane)cobalt(III). The water molecules exhibit positional disorder.

Introduction

The title compound was recently prepared and resolved into optical isomers (Shimba, Fujinami & Shibata, 1979). This complex has three different ligands, which are unidentate, bidentate and cyclic terdentate. The optical activity of this complex ion arises from the chiral disposition of the unidentate and the bidentate ligands with respect to the terdentate ligand as well as from the ring conformation of the terdentate ligand. The absolute configuration of this complex ion could not be assigned on the basis of its circular-dichroism spectrum, since no reference complex of known absolute configuration was available. The compound, therefore, was subjected to X-ray structure analysis in order to reveal the conformational details of the complex ion and to establish the empirical relation between the absolute configuration and the CD spectrum.

Experimental

The plate-like crystals are dark orange-red. The crystal data are: $[Co(C_6H_{15}N_3)(C_2H_4NO_2)(NH_3)]$-

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$I_2.0{\cdot}84H_2O$, $C_8H_{22}CoN_5O_2^{2+}.2I^-.0{\cdot}84H_2O$, M_r = 548·2, orthorhombic, $P2_12_12_1$, a = 15·075 (3), b = 17·674 (4), c = 12·625 (3) Å, U = 3364 (1) Å^3, Z = 8, D_m = 2·14, D_x = 2·16 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 4·8 mm^{-1}. A crystal was ground to a sphere 0·3 mm in diameter by Bond's (1951) method, and used for the intensity measurement on a Rigaku automated four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation, a θ–2θ scan technique being employed with a scan rate of 2° min^{-1} in θ. The scan range for a reflection was $\Delta\theta = (1{\cdot}2 + 0{\cdot}5 \tan \theta_B)°$, where θ_B is the Bragg angle of the reflection. Ni-foil attenuators were used for strong reflections to avoid coincidence loss; the attenuation factors were carefully determined by Fukamachi's (1969) method. Weak reflections were repeatedly measured to reduce the counting errors as follows: $\sigma(|F_o|) < 0{\cdot}03|F_o|$ where $\sigma(|F_o|)$ is the standard deviation based on counting statistics, with repetition limits of 3 for reflections with $2\theta_B < 55°$ and 4 for others. The background was measured at both ends of the first scan for 10 s. In the case of the subsequent scan, the counting time T_B for the background count was calculated according to $T_B = (T_P/2)[(T_P/2t_B)(B_1 + B_2)/P]^{1/2}$, where T_P is the scanning time, t_B = 10 s, B_1 and B_2 are the background counts at each end of the first scan, and P is the total count of the first scan. Four reference reflections were monitored every 50 reflections; they showed a very slight decrease in intensities, less than 1%. Among 5430 independent reflections of the $+h+k+l$ set, 4675 with $|F_o| > 3\sigma(|F_o|)$ were used for the structure determination. The intensities were corrected for Lorentz, polarization and absorption effects. The cell dimensions were obtained by a least-squares calculation based on the θ values of 20 high-angle reflections measured on the diffractometer with the same specimen.

Structure determination

The structure was solved by the conventional heavy-atom method, and refined by block-diagonal least squares with anisotropic thermal parameters for non-hydrogen atoms and isotropic ones for H atoms. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974); for H the values given by Stewart, Davidson & Simpson (1965) were used. The weighting scheme $w = [\sigma^2(|F_o|) + (0{\cdot}014|F_o|)^2]^{-1}$ was employed in the refinement. All the H atoms of the complex ions could be located at plausible positions on difference Fourier maps. At a late stage of the refinement, a peak of about 1·0 e Å^{-3} was found between the two water oxygen atoms, O(5) and O(6), on the difference maps. This was thought to be a H atom participating in an O—H···O hydrogen bond. After further refinement with an additional H atom at this position, the structure converged to R and R_2 values of 0·0261 and 0·0303, respectively, with a negative thermal parameter for this particular H atom as large as −20 Å^2 $\{R = \sum \Delta F/\sum |F_o|$ and $R_2 = [\sum w(\Delta F)^2/\sum w|F_o|^2]^{1/2}\}$. This unusual value of the thermal parameter strongly suggested positional disorder of the water molecules. Thus, the water molecules were thought to be statistically distributed at this site, O(7), as well as the other two sites O(5) and O(6) (see Fig. 1); the populations of the water O atoms were refined by the full-matrix least-squares program *LINUS* (Coppens & Hamilton, 1970). In view of the short distances between O(5) and O(7) (*ca* 1·0 Å) and between O(6) and O(7) (*ca* 1·7 Å), the sites O(5) and O(6) must be vacant if O(7) is occupied. Therefore their populations, $p[O(i)]$'s, were constrained in such a way that the sum of $p[O(7)]$ and $p[O(5)]$ or $p[O(6)]$ never exceeds 1·0. The disordered structure converged to the final R of 0·0253, R_2 of 0·0293 and S of 0·80 with populations of 0·68 (4), 0·71 (2) and 0·29 for O(5), O(6) and O(7) $\{S = [\sum w(\Delta F)^2/(m-n)]^{1/2}$, where m is the number of reflections and n is the number of parameters refined$\}$. In the last cycle of the refinement, the parameter shift/e.s.d. values were less than one fifth for all the non-hydrogen atoms and less than one half for all the H atoms. The ordered model could be safely rejected at the 0·005 significance level by Hamilton's (1965) test. This disordered distribution of the water molecules may be due to partial dehydration. The final difference maps showed no peak greater than 0·3 e Å^{-3} around the water molecules.

The absolute configuration was determined by anomalous-scattering techniques including the correction terms f' and f'' for Co and I. The enantiomeric

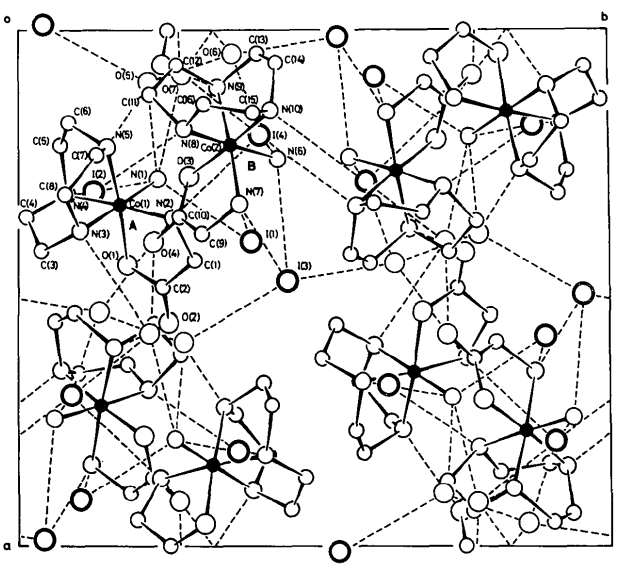

Fig. 1. A projection of the structure along **c**. The broken lines indicate hydrogen bonds (including possible ones).

Table 1. *Positional parameters* ($\times 10^4$; $\times 10^5$ *for* Co *and* I; $\times 10^3$ *for* H)

	x	y	z	
Co(1)	33997 (4)	17256 (3)	44869 (5)	
Co(2)	22306 (4)	36011 (3)	86739 (5)	
N(1)	2911 (3)	2371 (2)	3366 (4)	
N(2)	3671 (3)	2618 (2)	5346 (3)	
N(3)	3893 (3)	1046 (2)	5546 (3)	
N(4)	3197 (3)	824 (2)	3640 (3)	
N(5)	2252 (3)	1512 (2)	5127 (3)	
N(6)	2491 (3)	4402 (3)	7644 (4)	
N(7)	3400 (3)	3768 (3)	9335 (3)	
N(8)	1912 (3)	2811 (2)	9690 (3)	
N(9)	1105 (3)	3370 (2)	7984 (3)	
N(10)	1559 (3)	4284 (2)	9564 (4)	
C(1)	4486 (4)	2990 (3)	4926 (4)	
C(2)	4998 (3)	2439 (3)	4238 (4)	
C(3)	4296 (4)	403 (3)	4943 (5)	
C(4)	3638 (4)	144 (3)	4130 (5)	
C(5)	2224 (3)	721 (3)	3531 (4)	
C(6)	1792 (3)	864 (3)	4588 (4)	
C(7)	2426 (4)	1348 (3)	6272 (4)	
C(8)	3194 (4)	813 (3)	6331 (4)	
C(9)	3972 (3)	3110 (3)	9079 (4)	
C(10)	3659 (3)	2743 (3)	8075 (4)	
C(11)	1350 (4)	2255 (3)	9085 (5)	
C(12)	664 (4)	2702 (3)	8494 (5)	
C(13)	547 (3)	4062 (3)	8066 (5)	
C(14)	623 (4)	4383 (3)	9166 (5)	
C(15)	1605 (4)	3972 (4)	10679 (4)	
C(16)	1451 (4)	3144 (4)	10639 (4)	
O(1)	4553 (2)	1865 (2)	3901 (3)	
O(2)	5765 (3)	2576 (2)	3988 (3)	
O(3)	2853 (2)	2903 (2)	7798 (3)	
O(4)	4128 (3)	2303 (2)	7582 (3)	
I(1)	41005 (3)	39330 (2)	20297 (3)	
I(2)	31444 (2)	12786 (2)	7829 (3)	
I(3)	48638 (2)	45805 (3)	72715 (3)	
I(4)	20515 (3)	41085 (2)	48498 (3)	
O(5)	1012 (4)	2185 (5)	2990 (8)	0·68 (4)*
O(6)	459 (6)	3622 (5)	3235 (8)	0·71 (2)*
O(7)	895 (9)	2727 (9)	3233 (15)	0·29*
H(N1)1	307 (4)	279 (4)	326 (6)	
H(N1)2	293 (5)	217 (4)	272 (7)	
H(N1)3	232 (4)	241 (4)	327 (6)	
H(N2)1	376 (4)	247 (3)	606 (5)	
H(N2)2	325 (4)	295 (3)	528 (5)	
H(N3)	432 (3)	132 (3)	583 (4)	
H(N4)	345 (4)	89 (3)	296 (5)	
H(N5)	180 (3)	199 (3)	519 (4)	
H(N6)1	217 (5)	494 (4)	784 (7)	
H(N6)2	234 (4)	439 (4)	711 (5)	
H(N6)3	294 (4)	447 (3)	751 (5)	
H(N7)1	342 (4)	385 (4)	1012 (6)	
H(N7)2	367 (4)	417 (3)	900 (5)	
H(N8)	248 (4)	260 (3)	998 (5)	
H(N9)	121 (3)	320 (3)	724 (4)	
H(N10)	176 (3)	479 (3)	958 (4)	
H(C1)1	491 (4)	322 (3)	552 (5)	
H(C1)2	440 (3)	345 (3)	445 (4)	
H(C3)1	476 (5)	58 (4)	461 (6)	
H(C3)2	440 (4)	5 (3)	545 (5)	
H(C4)1	315 (4)	−19 (3)	443 (5)	
H(C4)2	398 (4)	−10 (3)	371 (5)	
H(C5)1	203 (4)	32 (3)	332 (5)	
H(C5)2	204 (3)	104 (2)	307 (3)	
H(C6)1	194 (3)	34 (2)	502 (3)	
H(C6)2	110 (4)	103 (3)	450 (5)	
H(C7)1	192 (3)	118 (3)	656 (4)	
H(C7)2	252 (4)	183 (3)	668 (5)	
H(C8)1	301 (3)	28 (3)	614 (4)	
H(C8)2	347 (3)	74 (2)	711 (4)	
H(C9)1	395 (3)	275 (3)	959 (4)	
H(C9)2	452 (4)	320 (3)	898 (5)	
H(C11)1	98 (4)	196 (3)	974 (5)	
H(C11)2	172 (4)	200 (3)	854 (5)	
H(C12)1	30 (5)	283 (4)	900 (6)	
H(C12)2	46 (3)	242 (3)	800 (4)	
H(C13)1	−5 (3)	391 (3)	791 (4)	
H(C13)2	68 (3)	437 (2)	754 (4)	
H(C14)1	25 (4)	418 (3)	967 (4)	
H(C14)2	46 (3)	483 (3)	913 (4)	
H(C15)1	224 (5)	415 (4)	1089 (7)	
H(C15)2	120 (4)	424 (3)	1106 (5)	
H(C16)1	87 (4)	302 (3)	1060 (5)	
H(C16)2	169 (5)	292 (4)	1125 (6)	

* Population parameters of the disordered water O atoms.

structure was refined separately. It converged to $R = 0{\cdot}0290$ and $R_2 = 0{\cdot}0344$, and could be rejected at the 0·005 significance level (Hamilton, 1965). To confirm this result, the observed structure amplitudes of several Bijvoet pairs were compared with the calculated values. The comparison certainly confirmed the correct absolute structure.* The absolute configuration of this complex ion can be designated as *a*-ammine-*bc*-glycinato-*def*-(1,4,7-triazacyclononane)cobalt(III), according to the IUPAC (1970) convention. In Table 1 the final positional parameters are listed.

* Lists of structure factors and thermal parameters, observed and calculated intensity relations between some Bijvoet pairs, bond lengths and angles within the complex ions, short contacts between non-bonded H atoms, and a table of the relevant interatomic distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34772 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Description of the structure and discussion

Fig. 1 shows a projection of the crystal structure along **c** and the numbering scheme of the atoms. The crystal consists of the complex cations, iodide ions and water molecules. There are two crystallographically independent but chemically equivalent complex ions in the unit cell which are labeled as *A* and *B* in Fig. 1. The observed geometries are similar. Fig. 2 shows a perspective view of the complex ion *A* along the pseudo triad axis; the absolute configuration is correctly

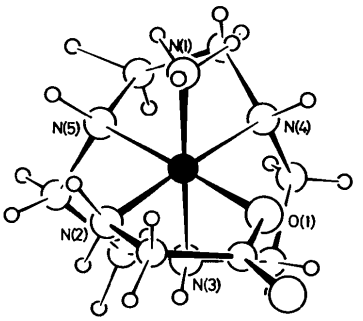

Fig. 2. A perspective drawing of the complex ion *A* projected on the plane defined by the three N atoms of the terdentate ligand.

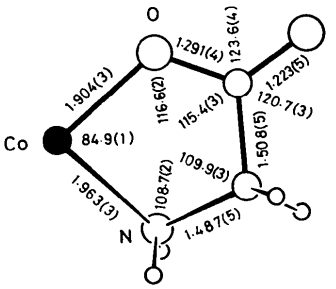

Fig. 3. A perspective drawing of the glycinatocobalt(III) ring of the complex ion *A*. The bond lengths (Å) and angles (°) are averaged over the two complex ions *A* and *B* with the e.s.d.'s of the average given in parentheses.

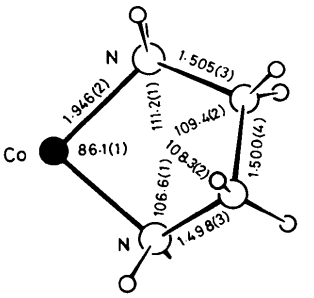

Fig. 4. A perspective drawing of a five-membered chelate ring, Co(1)—N(3)—C(3)—C(4)—N(4) (numbering: counterclockwise), projected on the coordination plane, of the terdentate ligand. The numerals are bond lengths (Å) and angles (°) averaged over the six rings of the complex ions *A* and *B*, the averaged e.s.d.'s being shown in parentheses.

represented. This triad axis is defined as a line through the Co atom and perpendicular to the plane formed by the three N atoms of the terdentate ligand. The Co atom is surrounded octahedrally by six ligating atoms. The cyclic terdentate ligand spans a face of an octahedron, being coordinated to the central Co atom with three secondary N atoms. The remaining coordination sites are occupied by the ammonia molecule and the glycinate ion. The conformations of the five-membered chelate rings of the tacn ligands are all δ.

The bond distances between Co and the ammine N atoms are both 1·961 (4) Å, being slightly shorter than the 1·972 (1) Å observed for the $[Co(NH_3)_6]^{3+}$ ion (Iwata & Saito, 1973).

The glycinato moieties in *A* and *B* are nearly planar with an average deviation of 0·36 (1) Å of the N atoms from the least-squares planes of the Co, C and O atoms.* All the bond lengths and angles are normal, as shown in Fig. 3. The N—C—C—O torsion angles are 18·6 (6) and −165·6 (5)° in complex ion *A*, and −19·0 (6) and 163·6 (5)° in *B*.

The average geometry of the five-membered chelate rings of the terdentate ligands is shown in Fig. 4. The bond lengths of Co and the secondary N atoms are in the range 1·935 (4) ~ 1·956 (4) Å, being shorter than other Co—N bonds. The chelate rings are highly strained. The two Co—N—C angles differ significantly from each other as shown in Fig. 4, as do the Co—N—C—C dihedral angles [−45·4 (2) and much strained −16·5 (2)° (averaged over the six rings)]; the two C atoms are unequally shifted by 0·71 (1) and 0·18 (1) Å on average to the same side of the coordination plane. This distortion may be attributed to repulsions between non-bonded H atoms. In fact, short contacts of about 2·2 Å occur between the two methylene H atoms in adjacent chelate rings and also between one methylene H atom and the glycinato amino H atom. The average N—C—C—N torsion angle is 40·4 (2)°. Other bond lengths and angles are normal.

The $[CoN_5O]$ chromophore is slightly distorted from a regular octahedron: the Co—N bonds of the terdentate ligands make an average angle of 52·1 (1)° with respect to the pseudo triad axis, in contrast to the 54·7° for a regular octahedron. The average dihedral angle between terdentate NCoN planes is 86·4 (1)°. A similar feature of the $[CoN_6]$ chromophore was observed in the $[Co\{(R)\text{-Metacn}\}_2]^{3+}$ ion (Mikami, Kuroda, Konno & Saito, 1977).

The complex cations, iodide ions and water molecules are held together by N—H···O, N—H···I and probably O—H···O and O—H···I hydrogen bonds to form a three-dimensional network. These hydrogen bonds are indicated by broken lines in Fig. 1.

The present compound will be of use as a reference complex to assign the absolute configurations of related complex ions, based on their CD spectra. In fact, the absolute configurations of several compounds of the type $[Co(a)(bc)(ddd)]^{2+}$ can be assigned (Shimba *et al.*, 1979). More structural information is necessary to establish the empirical rules relating the absolute configuration and the CD spectrum.

* The numeral in parentheses attached to the average value is the e.s.d. of the average.

The calculations were carried out on a FACOM 230-48 computer at this Institute with a local version of *UNICS* (1967). Some of the calculations were performed on a HITAC 8800/8700 computer at the Computer Center of the University of Tokyo. Part of the cost of this research was met by a Scientific Research Grant from the Ministry of Education, to which the authors' thanks are due.

References

BOND, W. L. (1951). *Rev. Sci. Instrum.* **22**, 344–345.
COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* **A26**, 71–83.
FUKAMACHI, T. (1969). *Jpn. J. Appl. Phys.* **8**, 851–854.
HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
IUPAC (1970). *Nomenclature of Inorganic Chemistry*, 2nd ed., pp. 75–83. London: Butterworths.
IWATA, M. & SAITO, Y. (1973). *Acta Cryst.* B**29**, 822–832.
MIKAMI, M., KURODA, R., KONNO, M. & SAITO, Y. (1977). *Acta Cryst.* B**33**, 1485–1489.
SHIMBA, S., FUJINAMI, S. & SHIBATA, M. (1979). Paper presented at the ACS/CSJ Chemical Congress (Symposium on Stereochemistry of Optically Active Transition Metal Compounds), Honolulu, Hawaii.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
UNICS (1967). *Universal Crystallographic Computation Program System*, edited by T. SAKURAI. The Crystallographic Society of Japan.

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The Crystal Structures of Rubidium and Sodium Acid Salts of α-Methoxy-α-phenylacetic Acid

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Abstract

The crystal structures of the rubidium, $RbH(C_9H_9O_3)_2$, and sodium, $NaH(C_9H_9O_3)_2$, acid salts of the racemate of α-methoxy-α-phenylacetic acid (Hmopa) have been determined. $RbH(mopa)_2$ is monoclinic, space group $C2/c$, with unit-cell parameters a = 26·770 (15), b = 6·979 (3), c = 9·496 (4) Å, β = 89·42 (2)°, Z = 4. $NaH(mopa)_2$ is orthorhombic, space group *Pbcn*, with a = 27·589 (14), b = 8·811 (5), c = 7·155 (5) Å, Z = 4. The hydroxyl H is on an inversion center in $RbH(mopa)_2$ and on the twofold axis in $NaH(mopa)_2$ and thus binds two identical mopa enantiomers in $NaH(mopa)_2$ and one mopa and its chiral mate in $RbH(mopa)_2$. The symmetrical hydrogen bonds have strong covalent character with O—H distances of 1·230 (4) Å in $RbH(mopa)_2$ and 1·226 (2) Å in $NaH(mopa)_2$. Both structures involve Rb—O and Na—O polyhedral chains along the c axis. In $NaH(mopa)_2$, carbonyl and methoxy O atoms are coordinated to the alkali-metal cations; in $RbH(mopa)_2$, hydroxyl, carbonyl and methoxy O atoms are coordinated to the cations.

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Introduction

The alkali acid salts of α-methoxy-α-phenylacetic acid (Hmopa) exhibit properties which are distinctly different from those of most other acid salts. Relatively high solvolytic and thermal stabilities of both small-sized (Na^+) and large-sized (K^+, Rb^+, Cs^+) alkali-metal acid salts with respect to the neutral salts and the apparent instabilities of the NH_4^+ and Li^+ acid salts with respect to salts of other acids are two such features. Compere (1970), in a detailed infrared study on these compounds, outlined several striking features: participation of the methoxy O in ionic bonding with the alkalis, two carboxyl O atoms in $NaH(mopa)_2$ and $KH(mopa)_2$ salts symmetrically bonded to H, and four equivalent carboxyl O atoms in the $RbH(mopa)_2$ and $CsH(mopa)_2$ salts bonded to the H. Interpretation of the infrared data proved to be incorrect both in the structural details (although some 'partial' structural resemblance to that of the X-ray analysis exists), and in the conclusion that there were four equivalent carboxyl O atoms in the