

The molecular structure thus established substantiates an intramolecular hydrogen bond of the type O—H... π . The present results are consistent with the observations based on the IR spectrum, and also with the inferred location of the H atom in the O—H... π interaction (Öki & Iwamura, 1961, 1967).

The crystal structure is illustrated in Fig. 2. No close intermolecular contact involving the O—H group was found in the crystal structure [H(1)...H > 2.86, H(1)...C > 3.00, O(1)...H > 2.96, and O(1)...C > 3.43 Å].

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(2*RS*,4*RS*)-2-Hydroxy-4-aminovaleric Acid Hydrochloride, a Structural Analogue of GABA

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Abstract. C₉H₁₁NO₃·HCl, $M_r = 169.61$, m.p. 175.5–176.5°C (dec.), triclinic, $P\bar{1}$, $a = 6.368$ (2), $b = 8.111$ (2), $c = 9.590$ (2) Å, $\alpha = 70.83$ (2), $\beta = 67.67$ (3), $\gamma = 98.64$ (3)°, $V = 406.4$ Å³, $Z = 2$, D_m (flotation) = 1.38, $D_x = 1.386$ g cm⁻³. The structure was refined to $R = 0.055$. The crystal structure is stabilized by hydrogen bonds.

Introduction. 2-Hydroxy-4-aminovaleric acid (Honoré, Hjeds, Krogsgaard-Larsen & Christiansen, 1978) and 2-hydroxy-4-aminovalerohydroxamic acid (Hjeds & Honoré, 1978) were synthesized as part of an investigation of the relationship between structure and biological activity of analogues of γ -aminobutyric acid (GABA). Two diastereomeric racemates of each compound were obtained and the relative stereochemistries of the hydroxamic acids and the amino acids were synthetically related (Hjeds & Honoré, 1978). This X-ray analysis of the title compound has been carried out in order to establish the relative stereochemistries of these racemates.

Platelet crystals of the title compound were grown from acetic acid. Intensities were measured on a

Nonius three-circle automatic diffractometer with graphite-monochromated Mo $K\alpha$ ($\lambda = 0.71069$ Å) radiation. The ω -scan technique with a scan speed of 1.2° min⁻¹ was employed. Background counts were taken for half the scanning time at each of the scan-range limits. The crystal (0.34 × 0.37 × 0.12 mm) was sealed in a glass capillary and oriented with c parallel to the ϕ axis of the goniostat. Of the 2346 independent reflections measured in the range $2.5 \leq \theta \leq 30.0^\circ$, 1652 had net intensities $> 3.0\sigma(I)$, where $\sigma(I)$ is the standard deviation from counting statistics. These were regarded as observed and used in the refinement. Lorentz and polarization corrections were applied but no absorption corrections were made [$\mu(\text{Mo } K\alpha) = 4.07$ cm⁻¹]. Cell dimensions were obtained by least-squares refinement from the 2θ values of 30 reflections measured on the diffractometer. The coordinates of the Cl⁻ ion were deduced from a Patterson function, and an electron density map phased on the contribution of the Cl⁻ ion gave the positions of all other non-hydrogen atoms. Refinement, first with isotropic, then anisotropic thermal parameters, proceeded smoothly. The coordinates of all 12 H atoms were located in a

Table 1. Final positional parameters ($\times 10^4$, for H $\times 10^3$)

Numbers in parentheses here and throughout this paper are the e.s.d.'s of the last significant digits.

	x	y	z
Cl	1583 (1)	10603 (1)	1598 (1)
O(1)	7401 (5)	2073 (3)	2065 (3)
O(2)	7094 (4)	1683 (3)	4556 (3)
O(3)	2939 (4)	2543 (3)	5395 (2)
C(1)	6402 (5)	2139 (3)	3522 (3)
C(2)	4357 (5)	2940 (4)	3698 (3)
C(3)	5452 (5)	5031 (3)	2725 (3)
C(4)	3584 (5)	6023 (4)	2853 (3)
C(5)	2308 (8)	5832 (6)	1845 (6)
N	4842 (5)	8029 (3)	2266 (3)
H(21)	335 (5)	249 (4)	326 (4)
H(31)	651 (6)	546 (4)	312 (4)
H(32)	663 (7)	539 (5)	154 (5)
H(41)	224 (7)	563 (5)	402 (5)
H(51)	113 (9)	646 (7)	199 (6)
H(52)	366 (10)	639 (7)	52 (7)
H(53)	145 (9)	458 (8)	221 (7)
H(81)	883 (9)	167 (6)	191 (6)
H(82)	160 (9)	160 (7)	591 (6)
H(71)	375 (8)	874 (6)	219 (5)
H(72)	609 (8)	854 (6)	118 (6)
H(73)	555 (7)	824 (5)	293 (5)

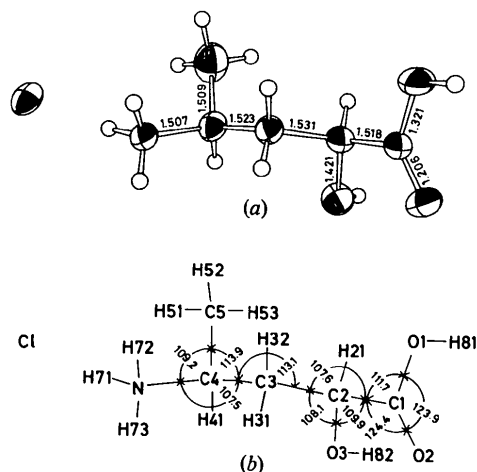


Fig. 1. (a) Bond lengths (Å) between non-hydrogen atoms. E.s.d.'s are 0.004–0.008 Å. Thermal ellipsoids for non-hydrogen atoms are scaled to 50% probability; H atoms are represented as spheres of arbitrary radius. (b) The numbering of the atoms and bond angles ($^{\circ}$) between non-hydrogen atoms. E.s.d.'s are 0.2–0.3 $^{\circ}$.

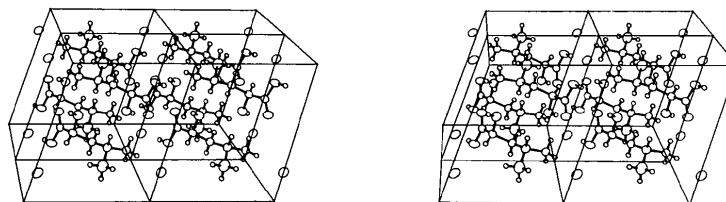


Fig. 2. Stereodiagram illustrating the molecular packing. The view is down **a**, with **b** \leftarrow and **c** \leftarrow .

difference map (0.4–0.8 e \AA^{-3}). In subsequent full-matrix least-squares calculations an overall scale factor, atomic coordinates, anisotropic thermal parameters for the non-hydrogen atoms and isotropic thermal parameters for H were refined. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 1/|1 + [(|F_o| - 5)/10]^2$. The final R was 0.055 ($R_w = 0.066$). Scattering factors were those of Cromer & Mann (1968) for Cl $^-$, O, N and C, and Stewart, Davidson & Simpson (1965) for H. The real part of the anomalous-dispersion correction for Cl used in the last stages of refinement was taken from *International Tables for X-ray Crystallography* (1974). The programs used were a local version of the *NRC 2A* Picker data-reduction program (Ahmed, 1968), the *XRAY* system (1972) and *ORTEP II* (Johnson, 1971). Table 1 lists the final atomic coordinates.*

Discussion. The relative stereochemistry of the title compound is (2*RS*,4*RS*). The thermal ellipsoids and atomic numbering for the molecule with the (2*S*,4*S*) configuration are shown in Fig. 1, with bond lengths and angles involving non-hydrogen atoms. The values are in good agreement with those found, for example, in GABA and β -hydroxy-GABA (Tomita, Higashi & Fujiwara, 1973; Steward, Player & Warner, 1973*a,b*; Tomita, Harada & Fujiwara, 1973).

A list of selected torsion angles is given in Table 2. The cation can be characterized by two approximately planar groups: (i) the *trans* zigzag chain of atoms C(1)–C(2)–C(3)–C(4)–N [plane (I)] and (ii) O(1), O(2), C(1) and C(2) [plane (II)]. O(3) lies 0.37 Å from plane (II). The angle between planes (I) and (II) is 88.4 $^{\circ}$. The intramolecular distances from the N atom to O(1), O(2) and O(3) are 5.364 (4), 5.528 (4) and 4.138 (4) Å respectively.

The packing of the molecules is illustrated in Fig. 2. The dimensions of the hydrogen bonds are summarized in Table 3. The crystals are built up of dimers of cations situated between infinite layers of Cl $^-$ ions parallel to (010) at about $y = 0$. The dimers result from the formation of two hydrogen bonds [N–H(73)⋯O(3)]

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33477 (51 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected torsion angles

O(3)–C(2)–C(1)–O(1)	±164.5 (2)°
O(3)–C(2)–C(1)–O(2)	∓18.2 (4)
O(3)–C(2)–C(3)–C(4)	∓59.2 (4)
C(1)–C(2)–C(3)–C(4)	∓177.7 (3)
C(2)–C(3)–C(4)–C(5)	∓74.2 (3)
C(2)–C(3)–C(4)–N	±164.6 (3)

Table 3. Selected interatomic distances (Å) and angles (°)

Symmetry code				
(i)	$x,$	$y,$	z	(iv) $1-x, 2-y, -z$
(ii)	$1+x,$	$-1+y,$	z	(v) $1-x, 1-y, 1-z$
(iii)	$-x,$	$1-y,$	$1-z$	
$A-H \cdots B$	$A-H$	$H \cdots B$	$A \cdots B$	$\angle AHB$
O(1)–H(81) \cdots Cl ⁽ⁱⁱⁱ⁾	0.99 (6)	2.03 (6)	3.019 (3)	175 (5)
O(3)–H(82) \cdots Cl ⁽ⁱⁱⁱ⁾	0.89 (5)	2.33 (4)	3.126 (3)	149 (6)
N–H(71) \cdots Cl ⁽ⁱ⁾	0.98 (5)	2.27 (5)	3.242 (4)	172 (3)
N–H(72) \cdots Cl ^(iv)	0.94 (4)	2.29 (5)	3.206 (3)	165 (5)
N–H(73) \cdots O(3) ^(v)	0.95 (6)	2.14 (6)	3.028 (5)	154 (4)
N–H(73) \cdots O(2) ^(v)	0.95 (6)	2.39 (5)	2.926 (4)	116 (3)

between the two cations related by a centre of symmetry at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. Furthermore, there is a short contact between H(73) and the carbonyl O(2) (Table 3). Each Cl[–] ion is an acceptor for four hydrogen bonds from

four different molecules in the crystal. Thus, of the five available H atoms per cation, all are utilized in the formation of hydrogen bonds. All other intermolecular contacts correspond to van der Waals interactions.

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Structure of the 1 : 1 Complex of Dimethyl Sulfoxide (DMSO) with 6-Hydroxy-1,3,7,9-tetranitroindazol[2,1-*a*]indazol-12-one

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Abstract. C₁₄H₆N₆O₁₀·C₂H₆OS, monoclinic, $P2_1/c$, $a = 8.354$ (3), $b = 11.750$ (5), $c = 21.489$ (9) Å, $\beta = 106.52$ (2)°, $V = 2022$ Å³, four formula units per unit cell. There is a strong hydrogen bond between the hydroxyl H and the DMSO oxygen: H \cdots O 1.68, O \cdots O 2.57 Å, $\angle O-H \cdots O$ 166°.

Introduction. Workers in this laboratory have been engaged in a study of the photolysis of aqueous solutions of 2,4,6-trinitrotoluene (α -TNT) (Burlinson, Kaplan & Adams, 1973; Kaplan, Burlinson & Sitzmann, 1975). The structure determination of the title complex was undertaken to identify a product obtained