C(11)—C(12)	1.409 (7)	C(11)—C(19)	1.536 (8)
C(12)—C(13)	1.393 (8)	C(13)—C(14)	1.404 (7)
C(13)—C(15)	1.549 (8)	C(15)—C(16)	1.530 (7)
C(15)—C(17)	1.541 (9)	C(15)—C(18)	1.526 (9)
C(19)—C(20)	1.537 (8)	C(19)—C(21)	1.518 (8)
C(19)—C(22)	1.547 (7)		
F(1)—C(1)—C(6)	119.3 (5)	F(1)—C(1)—C(2)	118.3 (5)
C(2)—C(1)—C(6)	122.3 (4)	C(1)—C(2)—C(3)	119.7 (6)
C(2)—C(3)—C(4)	121.2 (5)	C(3)—C(4)—C(7)	120.2 (6)
C(3)—C(4)—C(5)	117.6 (5)	C(5)—C(4)—C(7)	122.2 (6)
C(4)—C(5)—C(6)	121.0 (6)	C(1)—C(6)—C(5)	118.1 (6)
C(4)—C(7)—C(8)	127.5 (5)	C(7)—C(8)—C(9)	128.1 (6)
C(8)—C(9)—C(14)	122.8 (5)	C(8)—C(9)—C(10)	119.6 (5)
C(10)—C(9)—C(14)	117.6 (5)	C(9)—C(10)—C(11)	123.5 (5)
C(10)—C(11)—C(19)	121.4 (4)	C(10)—C(11)—C(12)	115.8 (5)
C(12)—C(11)—C(19)	122.7 (4)	O(1)—C(12)—C(11)	118.7 (5)
C(11)—C(12)—C(13)	123.2 (4)	O(1)—C(12)—C(13)	118.0 (4)
C(12)—C(13)—C(15)	121.5 (5)	C(12)—C(13)—C(14)	116.6 (5)
C(14)—C(13)—C(15)	121.9 (5)	C(9)—C(14)—C(13)	123.2 (5)
C(13)—C(15)—C(18)	110.1 (4)	C(13)—C(15)—C(17)	111.3 (4)
C(13)—C(15)—C(16)	110.5 (5)	C(17)—C(15)—C(18)	108.1 (5)
C(16)—C(15)—C(18)	110.7 (4)	C(16)—C(15)—C(17)	105.9 (4)
C(11)—C(19)—C(22)	111.0 (4)	C(11)—C(19)—C(21)	111.7 (5)
C(11)—C(19)—C(20)	111.3 (4)	C(21)—C(19)—C(22)	110.7 (5)
C(20)—C(19)—C(22)	105.7 (5)	C(20)—C(19)—C(21)	106.3 (4)

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55920 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1029]

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Structure of *N*-(2-Hydroxyethyl)ethylenediaminetriacetic Acid

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Abstract

The structure determination has shown that the title compound exists as a zwitterion in a *gauche* conformation about the central C—C bond. Both of the protonated N atoms participate in intramolecular hydrogen bonding. There are also two intermolecular hydrogen-bond interactions which link the molecules into chains extending parallel to **a**. One of these hydrogen bonds, acting between the carboxylic acid groups, is strong [O···O = 2.450 (2) Å] and close to symmetrical. The carboxyl groups assume the usual synplanar conformation. The results are compared with those obtained previously for closely related ethylenediaminetetraacetic acid.

Comment

This work is part of a more general study aiming at designing new metal-chelating agents. In this communication we report on the crystal structure of *N*-(hydroxyethyl)ethylenediaminetriacetic acid (H_3heedta) which is derived from the well known ligand ethylenediaminetetraacetic acid (H_4edta), by replacing one acetate group by a 2-hydroxyethyl substitutuent.

The present structure (Fig. 1) can be compared with that of the parent compound, H_4edta, which is known to exist in two crystalline modifications, α and β (Ladd, Povey & Stace, 1974; Cotrait, 1972; Ladd & Povey, 1973). The comparison has shown that the 2-hydroxyethyl substituent in place of the acetate group does not significantly affect the structure of the common ethylenediaminetriacetic portion. As in H_4edta, the molecule exists as a zwitterion in a *gauche* conformation with respect to the central C—C bond; the 2-hydroxethyl group is oriented *trans* to the central ethylene moiety (Table 2). Analogously to H_4edta, the H_3heedta molecule is also stabilized by two intramolecular hydrogen bonds between the protonated amino N atoms and the ionized carboxylate groups. The geometry of these hydrogen bonds (Table 3) is in keeping with that observed for similar bonds in the solid-state structures of amino acids and peptides (Görbitz, 1989).

The principal intermolecular interaction is the formation of a hydrogen bond between O(1)—H and O(3) of a translationally equivalent molecule (Table 3); this hydrogen bond fulfills all criteria to be classified as a strong hydrogen bond (Emsley, 1980; Novak, 1974), even though the acidic H atom is not precisely symmetrically placed between the donor and acceptor. That the H atom is more tightly bonded to O(1) than to O(3) is also evident from the difference (Δ) in the two carboxylic C—O distances which is much larger for the C(2), O(1), O(2) carboxyl group [$\Delta = 0.083$ (2) Å] than for the C(4), O(3), O(4) carboxyl group [$\Delta = 0.033$ (2) Å]. In this respect, H_3heedta more closely resembles β-H_4edta than α-H_4edta; in the latter, typical unsymmetrical hydrogen bonding was observed.

There is another intermolecular hydrogen-bond interaction between the O(7)—H hydroxyl and O(5) (at $x + 1, y, z$) (Table 3). The above two intermolecular contacts link the molecules into chains running parallel to the a axis. The chains are packed by van der Waals forces only.

As to the conformation of the carboxylic acid groups, the torsion angles $\chi_1 = $ O(2)—C(2)—C(1)—N(1) and $\chi_2 = $ O(4)—C(4)—C(3)—N(1) (Table 2) show that both partially ionized carboxyl groups (*i.e.* those involved in the strong hydrogen-bond interaction) follow the usual trend observed in α,β-saturated carboxylic acids, namely, a synplanar placement of the 'carbonyl' O atom [$\chi \simeq 0°$ (Leiserowitz, 1976)].

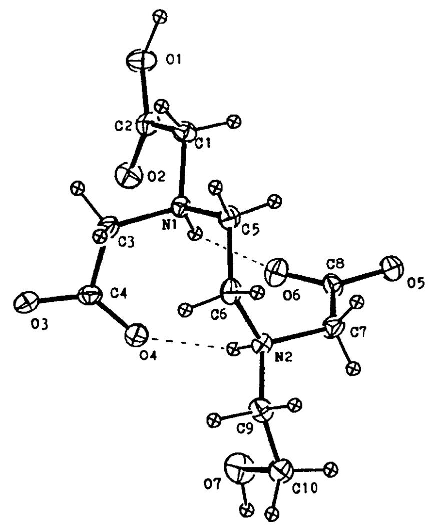

Fig. 1. Perspective drawing of the title compound and atom-numbering scheme. Thermal ellipsoids are drawn at the 40% probability level. H atoms are represented by spheres of arbitrary size. Intramolecular hydrogen bonds are indicated by dashed lines.

Experimental

Crystal data

$C_{10}H_{18}N_2O_7$	Mo $K\alpha$ radiation
$M_r = 278.3$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 15 reflections
Pc	$\theta = 6$-$20°$
$a = 7.076$ (5) Å	$\mu = 0.127$ mm^{-1}
$b = 8.218$ (7) Å	$T = 293$ K
$c = 10.353$ (8) Å	Prisms
$\beta = 105.20$ (4)°	$0.50 \times 0.25 \times 0.20$ mm
$V = 581$ (2) Å^3	Colourless
$Z = 2$	Crystal source: slow evaporation of aqueous solution at room temperature
$D_x = 1.592$ Mg m^{-3}	
$D_m = 1.58$ (1) Mg m^{-3}	
Density measured by flotation in bromoform-cyclohexane	

Data collection

Syntex $P2_1$ diffractometer	$\theta_{max} = 27.5°$
$\theta/2\theta$ scans	$h = 0 \rightarrow 9$
Absorption correction: none	$k = 0 \rightarrow 10$
1466 measured reflections	$l = -13 \rightarrow 12$
1351 independent reflections	2 standard reflections monitored every 100 reflections
1218 observed reflections [$I > 2\sigma(I)$]	intensity variation: <5%
$R_{int} = 0.023$	

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.09$
Final $R = 0.034$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$wR = 0.042$	$\Delta\rho_{min} = -0.19$ e Å^{-3}
$S = 0.86$	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
1218 reflections	
228 parameters	
$w = 1$ if $\lvert F_o \rvert < 10$	
$w = 10/\lvert F_o \rvert$ if $\lvert F_o \rvert \geq 10$	

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
C(1)	0.4238 (2)	0.6198 (2)	0.5529 (1)	2.07 (3)
C(2)	0.3881 (2)	0.5536 (2)	0.4125 (1)	2.16 (3)
C(3)	0.7682 (2)	0.5342 (2)	0.6152 (1)	2.12 (3)
C(4)	0.9244 (2)	0.5696 (2)	0.5436 (1)	2.16 (3)
C(5)	0.6550 (2)	0.7705 (2)	0.7278 (1)	2.11 (3)
C(6)	0.8578 (2)	0.8351 (2)	0.7839 (1)	2.19 (3)
C(7)	0.7899 (2)	1.1065 (2)	0.6719 (1)	2.42 (3)
C(8)	0.6319 (2)	1.0863 (2)	0.5416 (1)	2.01 (3)
C(9)	1.1259 (2)	1.0139 (2)	0.7811 (1)	2.30 (3)
C(10)	1.2228 (2)	1.1247 (2)	0.7048 (1)	2.56 (4)
N(1)	0.6303	0.6739 (1)	0.6021	1.69 (2)
N(2)	0.9241 (2)	0.9645 (1)	0.7037 (1)	1.74 (2)
O(1)	0.2195 (2)	0.4875 (1)	0.3639 (1)	2.94 (3)
O(2)	0.5092 (2)	0.5691 (1)	0.3505 (1)	2.75 (3)
O(3)	0.9990 (2)	0.4442 (1)	0.5079 (1)	2.99 (3)
O(4)	0.9671 (2)	0.7109 (1)	0.5267 (1)	2.87 (3)
O(5)	0.5150 (2)	1.2012 (1)	0.5138 (1)	2.76 (3)
O(6)	0.6342 (2)	0.9622 (1)	0.4743 (1)	2.66 (2)
O(7)	1.2129 (2)	1.0526 (2)	0.5808 (1)	3.77 (3)

Table 2. *Geometric parameters* (Å, °)

C(2)—O(1)	1.288 (2)	C(8)—O(5)	1.239 (2)
C(2)—O(2)	1.205 (2)	C(8)—O(6)	1.239 (2)
C(4)—O(3)	1.257 (2)	C(10)—O(7)	1.399 (2)
C(4)—O(4)	1.224 (2)		
C(1)—C(2)—O(1)	115.6 (1)	O(3)—C(4)—O(4)	126.7 (1)
C(1)—C(2)—O(2)	120.8 (2)	C(7)—C(8)—O(5)	114.6 (1)
O(1)—C(2)—O(2)	123.5 (1)	C(7)—C(8)—O(6)	118.3 (1)
C(3)—C(4)—O(3)	113.8 (1)	O(5)—C(8)—O(6)	127.1 (1)
C(3)—C(4)—O(4)	119.5 (1)		
N(1)—C(5)—C(6)—N(2)	68.1 (2)	O(6)—C(8)—C(7)—N(2)	−2.7 (2)
O(2)—C(2)—C(1)—N(1)	−9.6 (2)	N(2)—C(9)—C(10)—O(7)	−52.1 (2)
O(4)—C(4)—C(3)—N(1)	25.1 (2)		

Table 3. *Hydrogen-bond geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H···O(6)	0.90 (2)	1.94 (3)	2.717 (1)	144 (1)
N(2)—H···O(4)	0.85 (2)	2.08 (3)	2.843 (2)	152 (2)
O(1)—H···O(3^{i})	1.21 (2)	1.25 (2)	2.449 (1)	169 (2)
O(7)—H···O(5^{ii})	0.90 (2)	1.84 (3)	2.705 (2)	159 (2)

Symmetry code: (i) $x-1, y, z$; (ii) $x+1, y, z$.

Both positional and thermal parameters were refined for the polar H atoms but for the non-polar H atoms (bonded to C atoms), only the positional parameters were refined; isotropic *B* values were fixed at 0.5 $Å^2$ higher than the values of B_{eq} of the associated C atoms. The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All remaining calculations were performed using *NRC Crystallographic Programs for the IBM*360 *System* (1973). The refinement used a block-diagonal approximation. The weighting scheme (one of those available in the *NRC* program system) was chosen in order to make $w(\Delta F)^2$ approximately independent of $|F_o|$ and $\sin\theta/\lambda$. The origin was fixed by the *x* and *z* coordinates of the atom N(1).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55922 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1016]

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Structure of the Bis(7,7,8,8-tetracyano-*p*-quinodimethane)–4-(Benzylmethylaminomethyl)-2,2′,5,5′-tetrathiafulvalene Charge-Transfer Complex

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Abstract

The tetrathiafulvalenium (TTF) derivative [4-(benzylmethylaminomethyl)-2-(1,3-dithiol-2-ylidene)-1,3-dithiole cation] sublattice is built from one independent molecule which forms dimerized chains along the [100] direction. Short intra-dimer S···S contacts [3.532 (2) and 3.409 (2) Å] are observed. The anionic tetracyano-*p*-quinodimethane [TCNQ: 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile] sublattice is formed by three different molecules (*A*, *B* and *C*) which stack along the [001] direction perpendicular to the TTF chains.