and the material prepared for publication with *SHELXL93* (Sheldrick, 1994).

This work was supported in part by the Office of Naval Research and the National Institutes of Drug Abuse.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71812 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BK1005]

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1992). *SHELXTL-Plus.* Release 4.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
Thornber, C. W., Shaw, J. S., Miller, L., Hayward, C. F., Morley, J. S., Timms, D. & Wilkinson, A. (1986). *Progress in Opioid Research*, edited by J. W. Holaday, P. Law & A. Herz, pp. 177–180. NIDA Research Monograph 75, National Institutes of Drug Abuse, USA.

Acta Cryst. (1994). C**50**, 1097–1099

Nitrilotriacetic Acid, $C_6H_9NO_6$

E. Skrzypczak-Jankun and Douglas A. Smith

Department of Chemistry, University of Toledo, Toledo, OH 43606, USA

H. Maluszynska

Institute of Physics, A. Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland

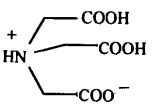
(*Received* 8 *September* 1993; *accepted* 5 *January* 1994)

Abstract

Nitrilotriacetic acid participates as a multidentate ligand in many metal chelation compounds of Al, B, Bi, Ca, Co, Cr, Cu, Fe, Mo, Nd, Ni, Pb, Ti, W, Zn and Zr. The structure of this popular ligand has been refined and compared with that of calcium nitrilotriacetate dihydrate [Whitlow (1972). *Acta Cryst.* B**28**, 1914–1919], which is another structure where this ligand is not affected by chelation.

Comment

The structure of nitrilotriacetic acid, which exists in the zwitterionic form (I) in the crystal, has been published previously (Stanford, 1967); it was described as well as possible on the basis of visually estimated photographic data (R = 0.093). As part of our continuing studies of amide derivatives of nitrilotriacetic acid and their chelation complexes with metals (Smith, Sucheck & Pinkerton, 1992; Smith, Cramer, Sucheck & Skrzypczak-Jankun, 1992; Skrzypczak-Jankun & Smith, 1994*a*,*b*), we have re-examined this ligand and report here the structure refined to a higher degree of accuracy.



(I)

Nitrilotriacetic acid (NTA) or its derivatives are present in many complex structures in which a metal atom is bonded to several atoms of the polydentate complexing agent. However, only a few structures of unchelated acid, anion or NTA derivatives are known: nitrilotriacetic acid (Stanford, 1967); calcium nitrilotriacetate dihydrate (Whitlow, 1972); 2,2′,2″-nitrilotriethanol (Mootz, Brodalla & Wiebcke, 1989); *N*-methylnitrilotriacetamide (Skrzypczak-Jankun & Smith, 1994*a*). In the last two of these structures, the central N atom is not protonated and the N—C bonds are shorter (1.467 and 1.463 Å, respectively) and the C—N—C angles smaller (110.7 and 110.9°, respectively) than in the protonated compounds. In $CaNTA.2H_2O$, five of the six NTA O atoms are bound to metal ions, but each O atom is joined to a different Ca ion, so that the NTA zwitterion is *not* the chelating ligand, but part of a three-dimensional ionic network similar to that seen in NTA itself. The structure described in this paper agrees very well with that observed in $CaNTA.2H_2O$ [mean values for N—C and C—N—C: 1.500 (2) Å and 113.0 (7)°, respectively, in NTA, and 1.496 (5) Å and 112.0 (15)°, respec-

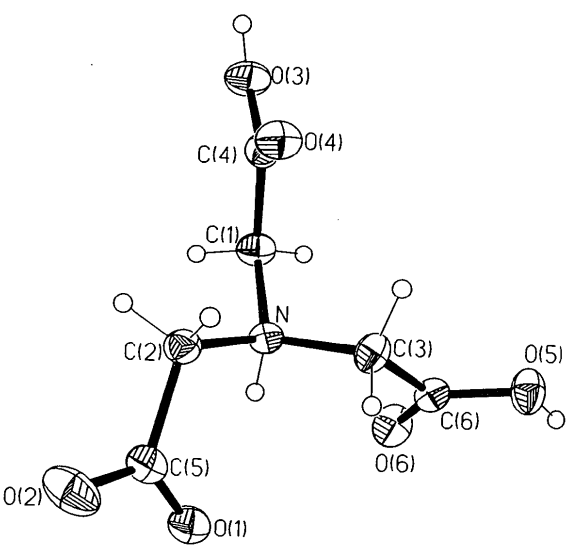


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the molecule with 50% displacement ellipsoids.

tively, in CaNTA.$2H_2O$]. The ammonium H atom is involved in bifurcated intramolecular hydrogen bonds and in one long-range intermolecular bond (see Table 3) to the carbonyl group containing O(6). Such a configuration could be described as a four-centered bond (Jeffrey & Saenger, 1991). The molecules are held together tightly in an extended hydrogen-bonding network. Each OH group interacts with the carboxylate ion from a different neighboring molecule; the interconnected zwitterions form channels parallel to the *c* axis. The carbonyl atom O(4) does not participate in the hydrogen-bonding lattice.

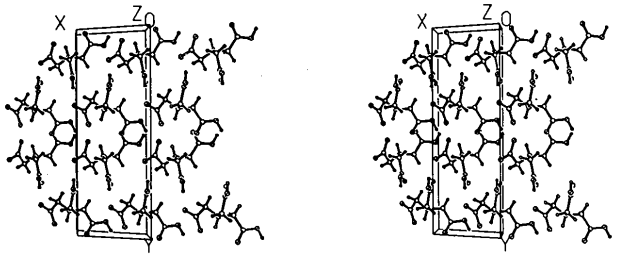


Fig. 2. Stereopacking diagram of the unit cell.

Experimental

Crystal data

$C_6H_9NO_6$
M_r = 191.14
Monoclinic
Cc
a = 7.961 (2) Å
b = 18.560 (3) Å
c = 6.501 (1) Å
β = 127.47 (3)°
V = 762.4 (7) Å^3
Z = 4
D_x = 1.67 Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–19°
μ = 0.142 mm^{-1}
T = 294 K
Prism
0.5 × 0.3 × 0.15 mm
Transparent
Crystal source: crystallized from water

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2646 measured reflections
1324 independent reflections
1311 observed reflections [$F > 3.0\sigma(F)$]

R_{int} = 0.0135
θ_{max} = 31.97°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 50 min
intensity variation: 0.5%

Refinement

Refinement on *F*
R = 0.0256
wR = 0.0330
S = 1.88
1311 reflections
152 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o) + 0.0001F_o^2]$

$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

U_{iso} for H atoms; $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	*y*	*z*	U_{iso}/U_{eq}
N	0.11265†	0.11512 (5)	0.19547†	0.0207 (4)
O(1)	0.33175 (24)	0.04415 (5)	0.08538 (30)	0.0304 (5)
O(2)	0.52603 (26)	0.13749 (6)	0.12368 (34)	0.0384 (7)
O(3)	0.05491 (25)	0.22778 (6)	0.61000 (29)	0.0310 (5)
O(4)	0.02272 (24)	0.25444 (5)	0.25132 (29)	0.0285 (5)
O(5)	−0.41780 (23)	0.05636 (5)	−0.31586 (29)	0.0300 (4)
O(6)	−0.13638 (25)	−0.00210 (6)	0.02632 (29)	0.0309 (5)
C(1)	0.09927 (27)	0.13275 (7)	0.41022 (31)	0.0236 (5)
C(2)	0.27989 (26)	0.15700 (7)	0.20781 (31)	0.0231 (5)
C(3)	−0.09740 (25)	0.11814 (7)	−0.06781 (29)	0.0239 (5)
C(4)	0.05393 (24)	0.21169 (7)	0.41284 (30)	0.0221 (5)
C(5)	0.38772 (26)	0.10888 (7)	0.13097 (31)	0.0242 (5)
C(6)	−0.21980 (25)	0.05018 (6)	−0.11187 (31)	0.0224 (5)
H(N1)	0.1546 (32)	0.0664 (11)	0.2203 (39)	0.029 (5)
H(1)	−0.0046 (32)	0.1024 (11)	0.3928 (44)	0.029 (5)
H(11)	0.2283 (40)	0.1229 (12)	0.5595 (50)	0.040 (6)
H(2)	0.2141 (33)	0.1994 (10)	0.0952 (39)	0.033 (5)
H(21)	0.3855 (39)	0.1744 (11)	0.3882 (48)	0.040 (6)
H(3)	−0.0708 (33)	0.1201 (10)	−0.1974 (43)	0.031 (5)
H(31)	−0.1746 (35)	0.1617 (11)	−0.0891 (43)	0.034 (5)
H(O3)	0.0483 (41)	0.2736 (14)	0.6120 (52)	0.044 (6)
H(O5)	−0.4852 (41)	0.0125 (16)	−0.3403 (55)	0.055 (7)

† Coordinate fixed to define origin.

Table 2. *Selected geometric parameters* (Å, °)

N—C(1)	1.499 (2)	O(6)—C(6)	1.210 (2)
N—C(2)	1.501 (2)	N—H(N1)	0.94 (2)
N—C(3)	1.499 (1)	C(1)—H(1)	0.95 (3)
C(1)—C(4)	1.511 (2)	C(1)—H(11)	0.90 (2)
C(2)—C(5)	1.519 (3)	C(2)—H(2)	0.98 (2)
C(3)—C(6)	1.511 (2)	C(2)—H(21)	0.99 (2)
O(1)—C(5)	1.253 (2)	C(3)—H(3)	0.99 (3)
O(2)—C(5)	1.249 (3)	C(3)—H(31)	0.97 (2)
O(3)—C(4)	1.311 (3)	O(3)—H(O3)	0.85 (3)
O(4)—C(4)	1.215 (2)	O(5)—H(O5)	0.93 (3)
O(5)—C(6)	1.309 (2)		
C(2)—N—C(1)	113.6 (1)	C(2)—C(5)—O(2)	116.8 (1)
C(2)—N—C(3)	112.3 (1)	O(1)—C(5)—O(2)	126.4 (2)
C(1)—N—C(3)	113.2 (1)	O(5)—C(6)—C(3)	111.0 (1)
N—C(1)—C(4)	112.0 (2)	O(5)—C(6)—O(6)	127.0 (2)
N—C(2)—C(5)	109.8 (1)	C(3)—C(6)—O(6)	122.0 (1)
N—C(3)—C(6)	109.2 (1)	C(2)—N—H(N1)	106.1 (17)
O(3)—C(4)—C(1)	111.9 (2)	C(1)—N—H(N1)	105.5 (18)
O(3)—C(4)—O(4)	124.9 (2)	C(3)—N—H(N1)	105.3 (10)
C(1)—C(4)—O(4)	123.2 (2)	C(4)—O(3)—H(O3)	106.2 (26)
C(2)—C(5)—O(1)	116.8 (2)	C(6)—O(5)—H(O5)	108.0 (14)
C(2)—N—C(1)—C(4)	58.6	N—C(1)—C(4)—O(3)	−176.2
C(3)—N—C(1)—C(4)	−70.9	N—C(1)—C(4)—O(4)	3.2
C(1)—N—C(2)—C(5)	141.4	N—C(2)—C(5)—O(1)	−3.6
C(3)—N—C(2)—C(5)	−88.6	N—C(2)—C(5)—O(2)	177.5
C(1)—N—C(3)—C(6)	−78.5	N—C(3)—C(6)—O(5)	167.8
C(2)—N—C(3)—C(6)	151.3	N—C(3)—C(6)—O(6)	−13.7

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

D	H	*A*	H···*A*	*D*···*A*	*D*—H···*A*
N	H(N)	O(1)	2.11	2.609	112
N	H(N)	O(6)	2.24	2.687	108

N	H(N)	O(1^{i})	2.79	3.582	143
O(3)	H(O3)	O(2^{ii})	1.67	2.518	176
O(5)	H(O5)	O(1^{iii})	1.61	2.512	160

Symmetry codes: (i) $x, -y, -\frac{1}{2}+z$; (ii) $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1+x, -y, \frac{1}{2}+z$.

Backgrounds were obtained from analysis of the scan profile (Blessing, Coppens & Becker, 1974). All H atoms were located in difference Fourier maps and refined isotropically. Data collection and cell refinement: *CAD*-4 (Enraf-Nonius, 1977). Data reduction: *MolEN* (Fair, 1990); *SHELX*76 (Sheldrick, 1976). Program(s) used to solve structure: *SHELX*76. Program(s) used to refine structure: *SHELX*76. Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1987); *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF* in *MolEN* (Fair, 1990).

We thank the College of Arts and Sciences of the University of Toledo for generous financial support of the X-ray diffraction facility.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BK1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Blessing, R. H., Coppens, P. & Becker, P. (1974). *J. Appl. Cryst.* 7, 488-492.

Enraf-Nonius (1977). *CAD-4 Operations Manual*. Enraf-Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.

Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*, p. 23. Berlin: Springer-Verlag.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Mootz, D., Brodalla, D. & Wiebcke, M. (1989). *Acta Cryst.* C**45**, 754-757.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Skrzypczak-Jankun, E. & Smith, D. A. (1994*a*). *Acta Cryst.* C**50**, 91-93.

Skrzypczak-Jankun, E. & Smith, D. A. (1994*b*). *Acta Cryst.* C**50**. In the press.

Smith, D. A., Cramer, S., Sucheck, S. & Skrzypczak-Jankun, E. (1992). *Tetrahedron Lett.* **33**, 7765-7768.

Smith, D. A., Sucheck, S. & Pinkerton, A. A. (1992). *J. Chem. Soc. Chem. Commun.* pp. 367-368.

Stanford, R. H. Jr (1967). *Acta Cryst.* **23**, 825-832.

Whitlow, S. H. (1972). *Acta Cryst.* B**28**, 1914-1919.

Acta Cryst. (1994). C**50**, 1099-1101

Bis(5-acetylamino-1,3,4-thiadiazole-2-sulfon)amide Dihydrate

MLADEN VINKOVIĆ* AND STJEPAN MUTAK

Research Institute, PLIVA-Pharmaceutical, Chemical, Food and Cosmetic Industry, Prilaz baruna Filipovića 89, 41000 Zagreb, Croatia

ANTE NAGL AND BORIS KAMENAR

Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Ul. kralja Zvonimira 8, 41000 Zagreb, Croatia

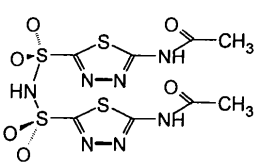
(*Received* 4 *January* 1993; *accepted* 19 *July* 1993)

Abstract

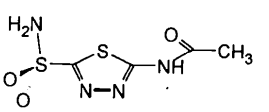
The molecules of the title compound [bis(5-acetylamino-1,3,4-thiadiazole-2-sulfonyl)amine dihydrate, $C_8H_9N_7O_6S_4.2H_2O$ (1)] consist of two nearly planar acetylaminothiadiazolesulfonyl units which are parallel to one another. Coplanarity of the acetylamino groups and the thiadiazole rings is achieved by π-electron delocalization over these groups and by non-bonded S···O interactions [S1···O1 2.667 (4), S1′···O1′ 2.661 (4) Å].

Comment

The title compound (1) was isolated as an impurity in the chemical synthesis of the commercial diuretic known by its generic name of acetazolamide (2) (Roblin & Clapp, 1950).



(1)



(2)

The title compound is a dimer containing two acetazolamide moieties (Fig. 1) and, as in acetazolamide itself (Mathew & Palenik, 1974), the acetylamino group is nearly coplanar with the thiadiazole ring [C2—N3—C3—O1 1.3 (8), C2′—N3′—C3′—O1′ −5.8 (7)°]. This coplanarity is most probably caused by π-electron delocalization over the acetylamino and thiadiazole groups as well as by non-bonded S···O interactions