Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[$^2H_3$]Sarcosine

Monika Trzebiatowska-Gusowska[a] and Anna Gagor[b]*

[a]Wroclaw University of Technology, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland, and [b]W. Trzebiatowski Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Okólna str. 2, PO Box 1410, 50-950 Wrocław, Poland
Correspondence e-mail: a.gagor@int.pan.wroc.pl

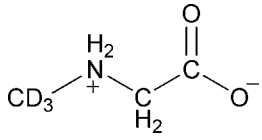
Received 6 November 2007; accepted 9 November 2007

Key indicators: single-crystal X-ray study; T = 286 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.075; data-to-parameter ratio = 9.5.

The crystal structure of [$^2H_3$]sarcosine or methyl[$^2H_3$]-ammonioacetate, $CD_3NH_2^+CH_2COO^-$ or $C_3H_4D_3NO_2$, was solved at room temperature. It is isostructural with nondeuterated sarcosine. The amino acid molecules exist as zwitterions. Each molecule is connected to four neighbouring molecules through N—H···O hydrogen bonds.

Related literature

Undeuterated sarcosine was reported by Mostad & Natarajan (1989).



Experimental

Crystal data

$C_3H_4D_3NO_2$
M_r = 92.11
Orthorhombic, $P2_12_12_1$
a = 6.7998 (13) Å
b = 7.9208 (16) Å
c = 8.5874 (18) Å
V = 462.52 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 286 (2) K
0.15 × 0.1 × 0.1 mm

Data collection

Kuma KM-4 diffractometer with CCD area detector
Absorption correction: none
5729 measured reflections
734 independent reflections
608 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.075
S = 0.96
734 reflections
77 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H4···O1^{i}	0.867 (18)	1.971 (18)	2.7886 (16)	156.9 (16)
N1—H3···O1^{ii}	0.905 (18)	1.860 (19)	2.7599 (17)	172.4 (15)
C3—D5···O2^{iii}	1.00 (2)	2.35 (2)	3.355 (2)	179 (2)

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

Data collection: *CrysAlis* (Mayer, 2006); cell refinement: *CrysAlis*; data reduction: *CrysAlis*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2085).

References

Brandenburg, K. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Mayer, M. (2006). *CrysAlis*. Version 1.171.30.3. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Mostad, A. & Natarajan, S. (1989). *Acta Chem. Scand.* **43**, 1004–1006.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Westrip, S. P. (2007). *publCIF*. In preparation.

  doi:10.1107/S1600536807057443

supplementary materials

Acta Cryst. (2007). E**63**, o4694 [**doi:10.1107/S1600536807057443**]

[$^2H_3$]Sarcosine

M. Trzebiatowska-Gusowska and A. Gagor

Comment

We determined the crystal structure of sarcosine-d3 (methyl-d3). It is isostructural with sarcosine – $P2_12_12_1$ (Mostad & Natarajan, 1989). The sarcosine molecules are in the form of zwitterions creating helices down the a, b and c axes through the network of N—H···O hydrogen bonds. There are also weak C(3)—D(5)···O(2) interactions with C(3)···O(2) = 3.355 (1) Å and a C(3)—D(5)···O(2) angle of 178.60 (1) °. Figure 1 shows the molecule of the sarcosine. Figure 2 shows the packing of sarcosine-d3 molecules in the crystal as seen down the *b* axis. Vibrational spectroscopy measurements corroborate the presence of the deuterated methyl group (–CD_3).

Experimental

The single crystals of sarcosine-d3 were obtained from Aldrich (99 atom % D).

Refinement

Positions of all hydrogen and deutrium atoms were taken from difference Fourier maps. They were refiened with isotropic displacement parameters correlated with the anisotropic displacement parameters of the atoms to which they were bonded [U_{iso}(H) = 1.2U_{eq}(C)]. Friedel pairs were merged prior to refinement

Figures

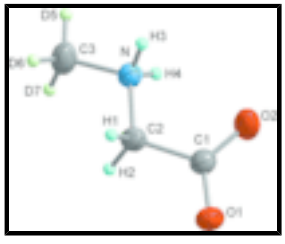


Fig. 1. The molecule of sarcosine-d3.

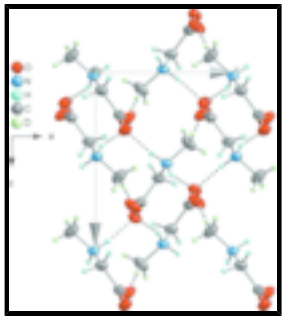


Fig. 2. The packing of sarcosine-d3 molecules in the crystal as seen down the *b* axis. The dashed lines represent the hydrogen bonds.

methyl[$^2H_3$]ammoniobenzoate

Crystal data

$C_3H_4D_3NO_2$	F_{000} = 192
M_r = 92.11	D_x = 1.323 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 734 reflections
a = 6.7998 (13) Å	θ = 3.8–29.4º
b = 7.9208 (16) Å	μ = 0.11 mm^{-1}
c = 8.5874 (18) Å	T = 286 (2) K
V = 462.52 (16) Å^3	Prism, colourless
Z = 4	0.15 × 0.1 × 0.1 mm

Data collection

Kuma KM-4 diffractometer with CCD area detector	734 independent reflections
Radiation source: fine-focus sealed tube	608 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
Detector resolution: 1024x1024 with blocks 2x2, 33.133pixel/mm pixels mm^{-1}	θ_{max} = 29.4º
T = 286(2) K	θ_{min} = 3.8º
ω–scan	h = −8→9
Absorption correction: none	k = −9→10
5729 measured reflections	l = −11→11

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.053P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Least-squares matrix: full	$(\Delta/\sigma)_{max}$ = 0.002
$R[F^2 > 2\sigma(F^2)]$ = 0.028	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$wR(F^2)$ = 0.075	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
S = 0.96	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
734 reflections	Extinction coefficient: 0.077 (15)
77 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Only H-atom coordinates refined	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.21502 (16)	0.18017 (13)	0.32862 (12)	0.0404 (3)
O2	0.2292 (2)	−0.09212 (14)	0.26347 (14)	0.0525 (4)
N1	−0.01173 (18)	−0.06244 (15)	0.01915 (14)	0.0293 (3)
H4	−0.046 (3)	−0.141 (2)	0.084 (2)	0.035*
H3	0.092 (2)	−0.102 (2)	−0.036 (2)	0.035*
C1	0.1734 (2)	0.05453 (18)	0.24312 (17)	0.0314 (3)
C2	0.0441 (2)	0.09257 (18)	0.10361 (17)	0.0320 (3)
H2	−0.074 (3)	0.146 (2)	0.135 (2)	0.038*
H1	0.104 (2)	0.163 (2)	0.030 (2)	0.038*
C3	−0.1714 (3)	−0.0354 (2)	−0.0949 (2)	0.0467 (4)
D5	−0.199 (3)	−0.146 (3)	−0.147 (3)	0.056*
D6	−0.134 (3)	0.056 (3)	−0.170 (2)	0.056*
D7	−0.285 (3)	0.003 (2)	−0.043 (2)	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0485 (6)	0.0358 (6)	0.0370 (6)	0.0047 (5)	−0.0085 (5)	−0.0113 (5)
O2	0.0736 (8)	0.0307 (6)	0.0532 (7)	0.0042 (5)	−0.0275 (6)	−0.0011 (5)
N1	0.0361 (6)	0.0255 (6)	0.0263 (6)	−0.0012 (5)	−0.0016 (5)	0.0013 (5)
C1	0.0352 (6)	0.0307 (7)	0.0282 (7)	−0.0019 (6)	0.0001 (6)	−0.0012 (6)
C2	0.0417 (7)	0.0256 (7)	0.0289 (7)	0.0003 (6)	0.0001 (6)	−0.0011 (6)
C3	0.0532 (10)	0.0388 (9)	0.0481 (10)	−0.0016 (8)	−0.0219 (8)	0.0011 (8)

Geometric parameters (Å, °)

O1—C1	1.2687 (17)	C1—C2	1.516 (2)
O2—C1	1.2343 (18)	C2—H2	0.949 (18)
N1—C2	1.4757 (18)	C2—H1	0.931 (18)
N1—C3	1.4782 (19)	C3—D5	1.00 (2)
N1—H4	0.867 (18)	C3—D6	1.00 (2)
N1—H3	0.905 (18)	C3—D7	0.94 (2)

C2—N1—C3	113.22 (12)	C1—C2—H2	111.1 (11)
C2—N1—H4	110.9 (11)	N1—C2—H1	106.0 (11)
C3—N1—H4	109.1 (11)	C1—C2—H1	113.5 (10)
C2—N1—H3	110.2 (11)	H2—C2—H1	106.9 (15)
C3—N1—H3	106.2 (10)	D5—C3—D6	113.5 (17)
H4—N1—H3	107.0 (16)	D5—C3—D7	109.8 (16)
O2—C1—O1	126.00 (14)	D6—C3—D7	105.7 (18)
O2—C1—C2	118.49 (12)	D5—C3—N1	107.8 (13)
O1—C1—C2	115.51 (12)	D6—C3—N1	110.2 (12)
N1—C2—C1	111.84 (11)	D7—C3—N1	109.8 (13)
N1—C2—H2	107.0 (11)		
C1—C2—N1—C3	167.20 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O1^{i}	0.867 (18)	1.971 (18)	2.7886 (16)	156.9 (16)
N1—H3···O1^{ii}	0.905 (18)	1.860 (19)	2.7599 (17)	172.4 (15)
C3—D5···O2^{iii}	1.00 (2)	2.35 (2)	3.355 (2)	178.6 (17)

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) −*x*+1/2, −*y*, *z*−1/2; (iii) *x*−1/2, −*y*−1/2, −*z*.

Fig. 1

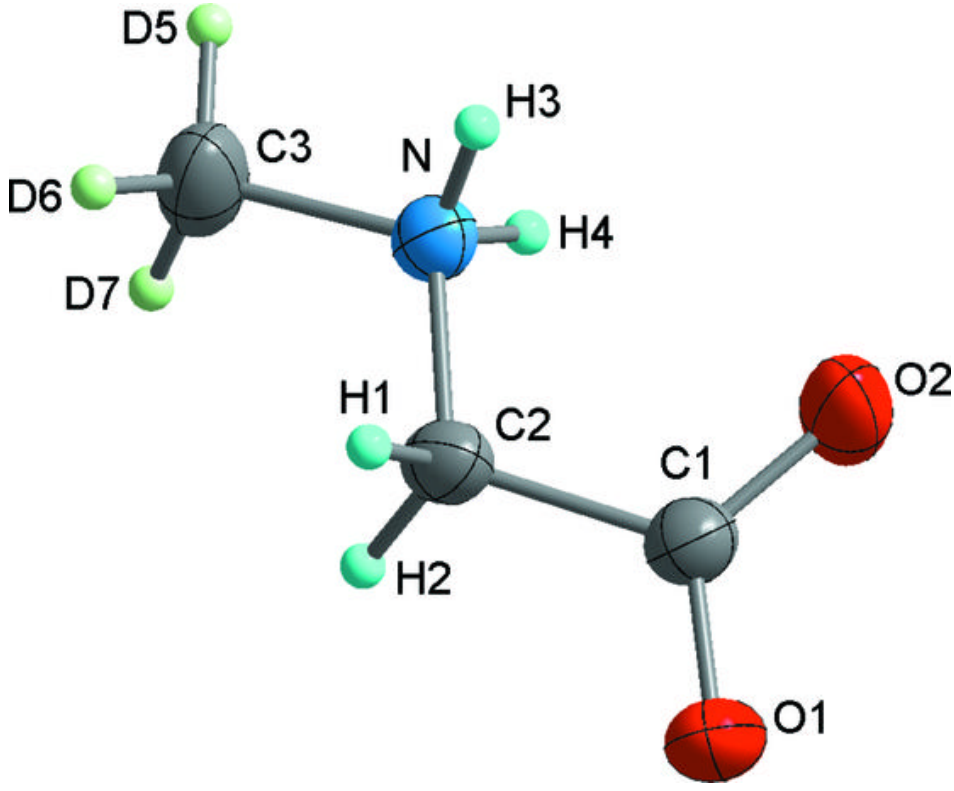


Fig. 2