Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-Methylimidazolidinetrione

Mark A. Rodriguez,[a]* Nicholas L. Andrews,[b] Timothy J. Boyle[b] and Colleen S. Frazer[a]

[a]PO Box 5800, MS 1411, Sandia National Laboratories, Albuquerque, NM 87185-1411, USA, and [b]Sandia National Laboratories, Advanced Materials Laboratory, 1001 University Blvd, Albuquerque, NM 87131, USA

Correspondence e-mail: marodri@sandia.gov

Received 19 May 2005
Accepted 22 June 2005
Online 30 June 2005

In the crystal structure of the title compound, $C_4H_4N_2O_3$, the packing is dominated by intermolecular carbonyl–carbonyl interactions and N—H$\cdots$O hydrogen bonds.

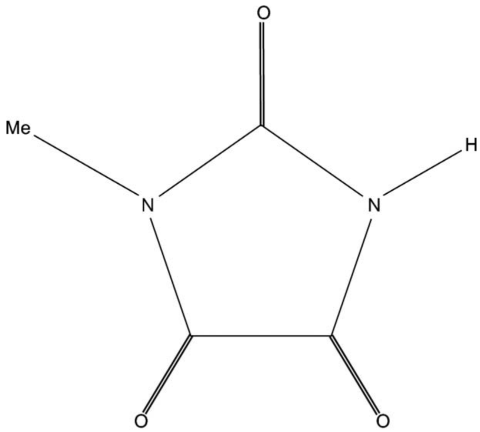
Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.081
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

N-Methylimidazolidinetrione, (I), is an alkyl derivative of panabanic acid (PBA) (Davies & Blum, 1955). The planar configuration of methyl derivative (I) is consistent with that of previously identified propyl and phenylethyl derivatives (Yoshihara *et al.*, 1999), with the alkyl group bound to one of the N atoms (N1) and a proton to the remaining N atom. In contrast, PBA possesses only H atoms in these positions.



(I)

Fig. 1 shows the molecular structure of (I) as a displacement ellipsoid plot. The C1—C2 bond bridges two of the carbonyl groups within the molecule and is somewhat elongated [1.545 (3) Å]. The unusually long Csp^2–Csp^2 single bond is worthy of comment. All other bonds within the ring are stronger N—C bonds with typical bond lengths ranging between 1.37 and 1.38 Å. This, coupled with the presence of a double-bonded terminal O atom on each of the atoms C1 and C2, has the effect of weakening the C1—C2 bond, thereby increasing its length. This elongated bond is consistent with those of PBA and other known alkyl derivatives, which have C—C bond lengths of 1.54 Å (Davies & Blum, 1955; Yoshihara *et al.*, 1999).

Fig. 2 shows a packing diagram of the crystal structure. Intermolecular carbonyl–carbonyl interactions propagate in a zigzag fashion along [100] and result in the following short intermolecular C$\cdots$O distances (Å): O1$\cdots$C2 = 2.805 (3), O3$\cdots$C1 = 2.892 (3), O1$\cdots$C1 = 2.959 (3), and O3$\cdots$C2 =3.006 (4). For clarity, only the shortest interaction distance (O1$\cdots$C2) is shown (dotted intermolecular bonds) in Fig. 2.

doi:10.1107/S1600536805019690

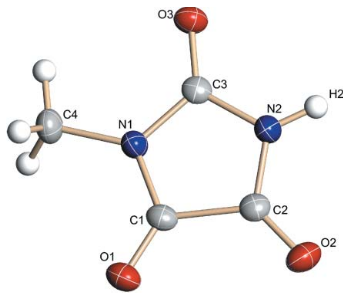


Figure 1
Displacement ellipsoid plot for (I), with ellipsoids shown at the 50% probability level.

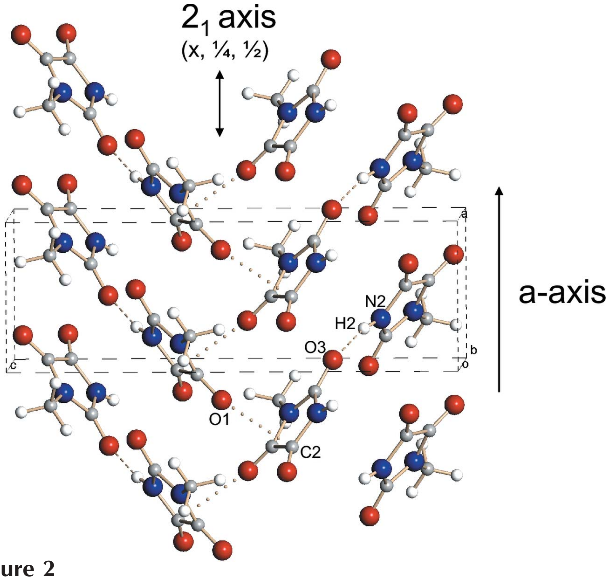


Figure 2
Packing diagram for (I). Dotted bonds between atoms O1 and C2 display the zigzag-like O1···C2 carbonyl interaction that generates chains along [100] by means of a 2_1 axis. Dashed lines show the N2—H2···O3 hydrogen bonding that links adjacent chains.

Such carbonyl–carbonyl interactions have been documented (Allen *et al.*, 1998) for other structures. The carbonyl–carbonyl interactions appear to dominate the intermolecular contacts and strongly affect the packing of (I). The carbonyl–carbonyl interactions form a spiral chain two molecules thick along [100] (by means of a 2_1 axis at $x, \frac{1}{4}, \frac{1}{2}$). These chains are linked to adjacent chains *via* an N2—H2···O3 hydrogen-bond interaction, which has a short intermolecular contact distance [N2···O3 = 2.810 (3) Å]. The hydrogen bonds (shown as dashed lines in Fig. 2) propagate along the [010] and [001] directions, thereby forming a three-dimensional structure by virtue of the intermolecular contacts.

Experimental

All compounds described below were handled with rigorous exclusion of air and water using standard Schlenk line and glove-box techniques. All reactions were run under an argon atmosphere in a glove-box. All reagents were stored under argon and used as received (Aldrich): tin(II) chloride ($SnCl_2$), lithium dimethylamide ($LiNMe_2$), diethyl ether (OEt_2) and sulfuryl chloride (SO_2Cl_2). Tin(II) dimethylamide [$Sn(NMe_2)_2$] was synthesized from the reaction of $SnCl_2$ and two equivalents of $LiNMe_2$ in Et_2O (Olmstead & Power, 1984). In a vial, $Sn(NMe_2)_2$ was added slowly to a stirred solution of an excess of SO_2Cl_2. Upon addition, smoke was emitted and the pale-yellow solution became dark yellow. After stirring for 12 h, the volatile portion of the reaction mixture was allowed to evaporate slowly. Colorless crystals were isolated and found to be *N*-methylimidazolidinetrione, (I). Yields were not determined or optimized and additional analytical data were not obtained.

Crystal data

$C_4H_4N_2O_3$
$M_r = 128.09$
Orthorhombic, $P2_12_12_1$
$a = 4.8966$ (8) Å
$b = 7.2049$ (12) Å
$c = 14.719$ (2) Å
$V = 519.27$ (15) Å^3
$Z = 4$
$D_x = 1.638$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 200 reflections
$\theta = 2.8$–27.5°
$\mu = 0.14$ mm^{-1}
$T = 168$ (2) K
Irregular fragment, colorless
0.21 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
$T_{min} = 0.94$, $T_{max} = 0.97$
4577 measured reflections

731 independent reflections
632 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$
$h = -6 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.05$
731 reflections
87 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.0879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.204 (2)	C3—N1	1.382 (3)
C1—N1	1.372 (3)	C4—N1	1.460 (3)
C1—C2	1.545 (3)	C4—H4*A*	0.98
C2—O2	1.202 (3)	C4—H4*B*	0.98
C2—N2	1.369 (3)	C4—H4*C*	0.98
C3—O3	1.210 (2)	N2—H2	0.86 (3)
C3—N2	1.381 (3)		
O1—C1—N1	128.1 (2)	H4*A*—C4—H4*B*	109.5
O1—C1—C2	126.9 (2)	N1—C4—H4*C*	109.5
N1—C1—C2	104.92 (17)	H4*A*—C4—H4*C*	109.5
O2—C2—N2	128.4 (2)	H4*B*—C4—H4*C*	109.5
O2—C2—C1	127.6 (2)	C1—N1—C3	111.25 (17)
N2—C2—C1	103.98 (18)	C1—N1—C4	124.16 (18)
O3—C3—N1	126.6 (2)	C3—N1—C4	124.52 (18)
O3—C3—N2	125.6 (2)	C2—N2—C3	111.97 (18)
N1—C3—N2	107.79 (18)	C2—N2—H2	125.2 (18)
N1—C4—H4*A*	109.5	C3—N2—H2	121.7 (18)
N1—C4—H4*B*	109.5		

Methyl H atoms were placed in idealized positions (C—H = 0.98 Å) and refined using a riding model, with $U_{iso}(H) = 1.5U_{eq}(C)$. The H atom bound to N2 was located in a difference Fourier map near the conclusion of the structure refinement process and was refined without restraints. Owing to the lack of heavy atoms (*i.e.*

heavier than Si), it was not possible to determine conclusively the absolute structure of the crystal. Therefore, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *XSHELL* (Bruker, 2000); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04–94 A L85000.

References

Allen, F. H., Ballham, C. A., Lommerse, J. P. M. & Raithby, P. R. (1998). *Acta Cryst.* B**54**, 320–329.

Bruker (1998). *SMART* (Version 5.054) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2000). *XSHELL*. Version 4.01. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2001). *SAINT-Plus*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.

Davies, D. R. & Blum, J. J. (1955). *Acta Cryst.* **8**, 129–136.

Olmstead, M. M. & Power, P. P. (1984). *Inorg. Chem.* **23**, 413.

Sheldrick, G. M. (1999). *SADABS*. Version 2.03. University of Göttingen, Germany.

Yoshihara, R., Hosomi, H., Aoyama, H. & Ohba, S. (1999). *Acta Cryst.* C**55**, 594–596.