Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: CR1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J., Lee, F. L. & Le Page, Y. (1985). *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 167–174. Oxford Univ. Press.
Hamilton, A. D., Geib, S. J. & Dixon, R. P. (1992). *J. Am. Chem. Soc.* **114**, 365–367, and references therein.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Mitcher, L. M. & Rao, G. S. R. (1984). *Natural Products and Drug Development*, edited by P. Krogsgaard-arsen, S. B. Christensen & H. Kofod, pp. 193–212, and references therein. Alfred Benzon Symposium 20. Denmark: Munksgaard.
Shiao, M.-J., Shyu, I.-M. & Tarng, K.-Y. (1990). *Synth. Commun.* **20**, 2971–2977.

Acta Cryst. (1994). C**50**, 1933–1935

trans-3-Amino-1-methyl-4-phenyl-2-azetidinone, $C_{10}H_{12}N_2O$

ANTHONY L. SPEK

Bijvoet Center for Biomolecular Research,
Department of Crystal and Structural Chemistry,
Utrecht University, Padualaan 8, 3584 CH Utrecht,
The Netherlands

FRED H. VAN DER STEEN, JOHANN T. B. H. JASTRZEBSKI AND GERARD VAN KOTEN

Debye Institute, Department of Metal-Mediated
Synthesis, Utrecht University, Padualaan 8,
3584 CH Utrecht, The Netherlands

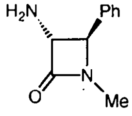
(*Received* 10 *June* 1994; *accepted* 1 *July* 1994)

Abstract

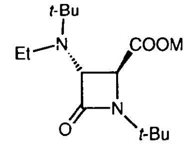
The central four-membered azetidinone ring of the title compound is puckered significantly with an average ring torsion angle of 5.1 (1)°. Molecules form infinite two-dimensional hydrogen-bonded networks *via* N—H···N [N···N 3.379 (3) Å] and N—H···O [N···O 3.056 (3) Å] interactions.

Comment

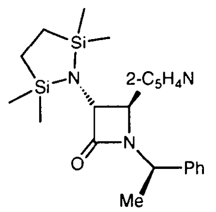
The title compound (I) belongs to a class of compounds called monobactams. Its core is a principal building block of natural and synthetic β-lactam antibiotics (*Chemistry and Biology of β-Lactam Antibiotics*, 1982). Its molecular structure (Fig. 1) comprises a four-membered cyclic amide with the phenyl and amino substituents in a *trans* arrangement. The geometry of the four-membered ring is similar to that reported for *trans*-1-(*N*-*tert*-butyl)-3-(*N*-*tert*-butyl-*N*-ethylamino)-4-methoxycarbonyl-2-azetidinone (II) (van Vliet, Jastrzebski, Klaver, Goubitz & van Koten, 1987) and *trans*-(3*R*,4*S*)-1-(*R*)-α-methylbenzyl-3-(2,2,5,5-tetramethyl-1-aza-2,5-disilacyclopentyl)-4-(2-pyridyl)-2-azetidinone (III) (van der Steen, Kleijn, Spek & van Koten, 1990).



(I)



(II)



(III)

The previously reported trend of relatively long C—N and short C═O amide bonds (compared to normal amides) being a common feature for monocyclic (Davis & Storr, 1984) as well as bicyclic (McGregor, 1984) β-lactams is not observed here. The significant puckering of the core four-membered ring is also in disagreement with earlier observations, but the value of the average

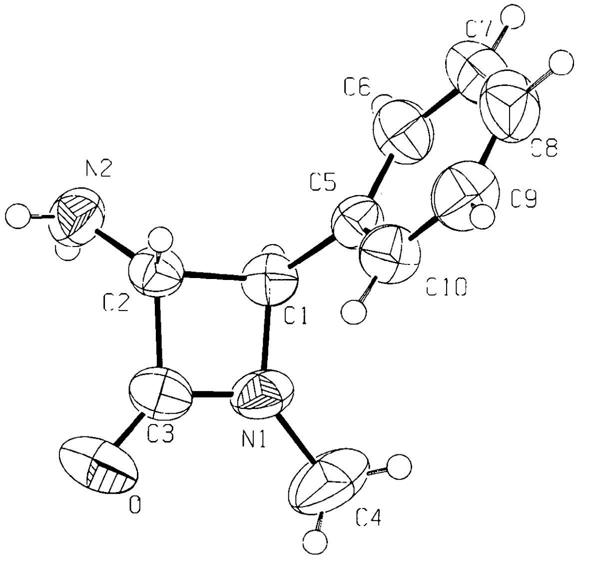


Fig. 1. Structure of (I) with displacement ellipsoids drawn at the 50% level.

ring torsion angle [5.1 (1)°] is consistent with that observed in the related structure (III) [5.9 (1)° (van der Steen, Kleijn, Spek & van Koten, 1990)].

The two amine H atoms are hydrogen bonded to the amine N and keto O atoms, respectively, of symmetry-related molecules [N2—H2*A*···N2($x-\frac{1}{2}$, $\frac{3}{2}-y$, $-z$): N···N = 3.379 (3), N—H = 0.92 (3), H···N = 2.51 (3) Å, N—H···N = 156 (2)°; N2—H2*B*···O($\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$): N···O = 3.056 (3), N—H = 0.87 (2), H···O = 2.26 (3) Å, N—H···O 151 (2)°]. Hydrogen bonding joins the molecules into two separate infinite symmetry-related layers perpendicular to the *c* axis. In addition, a short C—H···O contact [H1···O($1+x$, y, z) = 2.60 (2) Å] is observed.

Experimental

The preparation of (I) was performed according to the procedure described by van der Steen, Kleijn, Jastrzebski & van Koten (1991). Crystals were obtained from a diethyl ether/pentane solution.

Crystal data

$C_{10}H_{12}N_2O$
$M_r = 176.22$
Orthorhombic
$P2_12_12_1$
$a = 5.9478$ (5) Å
$b = 6.7797$ (3) Å
$c = 24.3482$ (11) Å
$V = 981.82$ (10) Å^3
$Z = 4$
$D_x = 1.192$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.0$–$16.0°$
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Needle
0.80 × 0.28 × 0.12 mm
Yellowish

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3749 measured reflections
1712 independent reflections
1360 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0381$
$\theta_{\rm max} = 24.95°$
$h = -7 \rightarrow 0$
$k = -8 \rightarrow 8$
$l = -28 \rightarrow 28$
3 standard reflections frequency: 60 min intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0391$
$wR(F^2) = 0.0971$
$S = 1.030$
1712 reflections
166 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.0151P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.030$
$\Delta\rho_{\rm max} = 0.125$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.130$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O	−0.2795 (3)	0.2702 (2)	0.04019 (8)	0.0788 (6)
N1	0.0561 (3)	0.2792 (2)	0.09043 (7)	0.0559 (6)
N2	0.0553 (4)	0.6317 (3)	−0.00017 (8)	0.0576 (6)
C1	0.2000 (3)	0.4537 (3)	0.08565 (8)	0.0467 (6)
C2	0.0040 (3)	0.5489 (3)	0.05266 (8)	0.0456 (6)
C3	−0.1093 (4)	0.3464 (3)	0.05810 (8)	0.0537 (7)
C4	0.1067 (9)	0.0819 (4)	0.1088 (2)	0.0918 (13)
C5	0.2648 (3)	0.5514 (3)	0.13878 (7)	0.0445 (6)
C6	0.4654 (4)	0.6515 (4)	0.14308 (10)	0.0605 (7)
C7	0.5196 (5)	0.7528 (4)	0.19060 (11)	0.0753 (11)
C8	0.3755 (5)	0.7536 (4)	0.23415 (11)	0.0770 (10)
C9	0.1764 (5)	0.6543 (4)	0.23043 (10)	0.0723 (10)
C10	0.1201 (4)	0.5529 (3)	0.18318 (8)	0.0572 (7)

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

O—C3	1.217 (3)	N2—C2	1.436 (3)
N1—C1	1.465 (2)	C1—C2	1.556 (3)
N1—C3	1.340 (3)	C1—C5	1.504 (3)
N1—C4	1.442 (3)	C2—C3	1.535 (3)
C1—N1—C3	96.18 (15)	N2—C2—C1	117.73 (17)
C1—N1—C4	130.7 (3)	N2—C2—C3	121.34 (17)
C3—N1—C4	130.6 (2)	C1—C2—C3	85.03 (15)
N1—C1—C2	86.46 (14)	O—C3—N1	132.59 (19)
N1—C1—C5	115.93 (16)	O—C3—C2	135.54 (19)
C2—C1—C5	116.97 (16)	N1—C3—C2	91.86 (16)

X-ray data were collected with the crystal mounted inside a Lindemann glass capillary. A collimator with a uniform primary beam greater than 1 mm was selected. The space group was derived from the observed systematic extinctions (*h*00: $h = 2n + 1$; 0*k*0: $k = 2n + 1$; 00*l*: $l = 2n + 1$). H atoms were located from a difference Fourier map and their positions and individual $U_{\rm iso}$'s refined. The structure contains no residual solvent-accessible areas (*PLATON*; Spek, 1990)

The adopted absolute configuration (Table 2, Fig. 1) was chosen to be consistent the configuration in related monobactams (*e.g.* van der Steen, Kleijn, Spek & van Koten, 1990). The starting material for (I) was achiral.

Data collection: locally modified CAD-4 software, version 5. Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *PLATON*.

This work was supported in part by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Scientific Research (NWO).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1021). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* **A40**, C-410.
Chemistry and Biology of β-Lactam Antibiotics (1982). Vols 1–3, edited by R. B. Morin & M. Gorman. New York: Academic Press.

Davis, D. E. & Storr, R. C. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 7, edited by W. Lwowski, p. 247. New York: Pergamon.
McGregor, D. N. (1984). *Comprehensive Heterocyclic Chemistry*, Vol. 7, edited by W. Lwowski, pp. 300–301. New York: Pergamon.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Spek, A. L. (1993). *HELENA. Program for Reduction of CAD-4 Data*. Utrecht Univ., The Netherlands.
Steen, F. H. van der, Kleijn, H., Jastrzebski, J. T. B. H. & van Koten, G. (1991). *J. Org. Chem.* **56**, 5147–5158.
Steen, F. H. van der, Kleijn, H., Spek, A. L. & van Koten, G. (1990). *J. Chem. Soc. Chem. Commun.* pp. 503–504.
Vliet, M. R. P. van, Jastrzebski, J. T. B. H., Klaver, W. J., Goubitz, K. & van Koten, G. (1987). *Recl Trav. Chim. Pays-Bas*, **106**, 132–134.

Acta Cryst. (1994). C**50**, 1935–1938

Two *N*-Aminoazolylmethylene Derivatives of Meldrum's Acid

Alexander J. Blake, Robert O. Gould, Iain Irving,
Hamish McNab and Mark Morrow

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

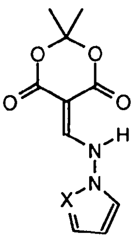
(*Received* 23 *May* 1994; *accepted* 23 *June* 1994)

Abstract

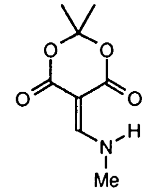
The structures of two derivatives of Meldrum's acid, 2,2-dimethyl-5-[*N*-(1-pyrrolyl)aminomethylene]-1,3-dioxane-4,6-dione, $C_{11}H_{12}N_2O_4$, (1), and 2,2-dimethyl-5-[*N*-(1-pyrazolyl)aminomethylene]-1,3-dioxane-4,6-dione, $C_{10}H_{11}N_3O_4$, (2), which differ only at one position in the five-membered ring, are described.

Comment

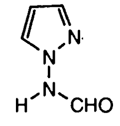
The title compounds (1) and (2) were synthesized as part of a pyrolysis study aimed at the preparation of novel heterocyclic compounds (Blake, McNab, Morrow & Rataj, 1993). The crystal structures were determined because of the current interest in the structures of *N*-aminoazoles (de la Concepcion Foces-Foces, Cano, Claramunt, Sanz, Catalan, Fabero, Fruchier & Elguero, 1990; Salazar, Espada, Sanz, Claramunt, Elguero, Garcia-Granda, Diaz & Gomez-Beltran, 1993) and because of our interest in hydrogen bonding in aminomethylene Meldrum's acid derivatives (Blake, Hunter & McNab, 1989).



(1) *X* = CH
(2) *X* = N



(3)



(4)

The *N*-aminopyrrolyl derivative (1) crystallized with two molecules in the asymmetric unit. One of these, designated by unprimed atoms, showed the effects of slight disorder, which gave less reliable molecular parameters for the region around C7. Fortunately, no such problem occurred in the other molecule, which is defined by primed atoms.

The aminomethylene Meldrum's acid moiety shows no significant differences between structures (1) and (2). The presence of hydrogen bonding between O4 and H8 [O4···N8 2.746 (5) and 2.710 (3) Å in (1) and (2), respectively] induces detectable asymmetry in the Meldrum's acid unit; C4—O4 is significantly longer than C6—O6 and C4—C5 is significantly shorter than C5—C6 in both cases. This asymmetry is also reflected in the bond angles around C5. [Note that the atomic coordinates and bond lengths for (2) should be compared with the equivalent primed atomic coordinates and bond lengths in (1) because of the disorder affecting the molecule defined by unprimed atoms in (1).] In methylaminomethylene Meldrum's acid (3) the quality of the diffraction data were generally inadequate to identify such deviations (Blake *et al.*, 1989). The bond lengths within the *N*-aminopyrazolyl unit are very similar to those reported for *N*-(pyrazol-1-yl)formamide (4) (Salazar *et al.*, 1993).

There are some significant differences between the valence angles involving the *N*-aminoazole units in (1) and (2), particularly at the ring heteroatom N9. The angles within the pyrazole ring in (2) are, however, very similar to those in the formamide (4) (Salazar *et al.*, 1993).

The atoms O4, C4, C5, C6, O6, C7 and N8 show small r.m.s. deviations from their best planes of 0.04 (5) and 0.013 (3) Å for (1) and (2), respectively. The corresponding deviations from the planes defined by N8, N9, C10, C11, C12 and C13 in (1) and N8, N9, N10, C11, C12 and C13 in (2) are 0.004 (6) and 0.003 (4) Å, respectively. The interplanar angles of 70.3 (2) and 41.26 (5)° in (1) and (2), respectively, are much smaller than the value of 85.5° seen in *N*-(pyrazol-1-yl)formamide (4) (Salazar *et al.*, 1993). These lower values may reflect the fact that the Meldrum's acid group promotes more extensive delocalization of the N8 lone pair than does the formyl group.

Acta Crystallographica Section C
ISSN 0108-2701 ©1994