Refinement

Refinement on F
$R = 0.041$
$wR = 0.053$
$S = 1.033$
1075 reflections
138 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0020F^2]$

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

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

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

	x	y	z	$U_{\rm eq}$
O1	0.1267 (3)	0.4131 (1)	0.8735 (1)	0.0371 (4)
C2	−0.0253 (4)	0.3361 (1)	0.8335 (2)	0.0354 (6)
C21	0.0798 (4)	0.2447 (1)	0.8683 (2)	0.0369 (6)
C22	0.3126 (4)	0.2266 (1)	0.9404 (2)	0.0378 (6)
C23	0.3948 (4)	0.1286 (1)	0.9641 (2)	0.0348 (6)
O231	0.6363 (3)	0.1196 (1)	1.0300 (2)	0.0483 (5)
O232	0.2561 (3)	0.0613 (1)	0.9265 (1)	0.0449 (5)
C3	−0.2612 (4)	0.3638 (1)	0.7643 (2)	0.0410 (7)
C4	−0.2601 (4)	0.4626 (1)	0.7606 (2)	0.0424 (7)
C5	−0.0234 (4)	0.4876 (1)	0.8269 (2)	0.0357 (6)
N50	0.0889 (3)	0.5777 (1)	0.8549 (2)	0.0414 (5)
O501	0.3145 (4)	0.5830 (1)	0.9144 (2)	0.0640 (7)
O502	−0.0488 (3)	0.6453 (1)	0.8155 (2)	0.0584 (7)

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

O1—C2	1.372 (2)	C23—O232	1.221 (2)
O1—C5	1.355 (2)	C3—C4	1.409 (2)
C2—C21	1.434 (2)	C4—C5	1.345 (2)
C2—C3	1.364 (2)	C5--N50	1.419 (2)
C21—C22	1.330 (2)	N50—O501	1.225 (2)
C22—C23	1.469 (2)	N50—O502	1.226 (2)
C23—O231	1.323 (2)	O231—H231	0.90 (2)
C2—O1—C5	104.8 (2)	C2—C3—C4	107.3 (2)
O1—C2—C3	110.0 (2)	C3—C4—C5	105.0 (2)
O1—C2—C21	118.5 (2)	O1—C5—C4	113.0 (2)
C21—C2—C3	131.5 (2)	C4—C5—N50	130.5 (2)
C2—C21—C22	125.9 (2)	O1—C5—N50	116.6 (2)
C21—C22—C23	119.2 (2)	C5—N50—O502	116.7 (2)
C22—C23—O232	123.8 (2)	C5—N50—O501	118.7 (2)
C22—C23—O231	113.5 (2)	O501—N50—O502	124.6 (2)
O231—C23—O232	122.7 (2)	C23—O231—H231	110 (2)

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON*93 (Spek, 1993). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

The authors thank American Cyanamid Company (Pearl River, New York) for providing the crystals. One of them (TO) is indebted to the Research Council of the K. U. Leuven (Belgium) for Senior Fellowship No. F/92/32.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, bond distances and angles involving H atoms and torsion angles have been deposited with the IUCr (Reference: NA1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Burke-Laing, M. & Laing, M. (1976). *Acta Cryst.* B**32**, 3216–3224
Kusa, A., Polynova, T. N., Porai-Koshits, M. A., Kovach, Ya. & Vegkh, D. (1979). *Zh. Strukt. Khim.* **20**, 556
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1989). *CRYSRULER*. Version 2.0. Univ. degli Studi di Parma, Italy.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Spek, A. L. (1993). *PLUTON93. Molecular Graphics Program.* Univ. of Utrecht, The Netherlands.
Stoe & Cie (1992*a*). *DIF4. Diffractometer Control Program.* Version 7.0. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1992*b*). *REDU4. Data Reduction Program.* Version 7.03. Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (1994). C**50**, 1931–1933

2,6-Bis(dimethylamino)-3,5-pyridinedicarbaldehyde

LONG-LI LAI, LING-KANG LIU, MING-JEN SHIAO AND YUH-SHENG WEN

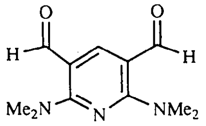
Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan

Abstract

The title molecule, $C_{11}H_{15}N_3O_2$, sits on a crystallographic twofold axis. This highly substituted pyridine compound has a very distorted pyridine plane (±0.1 Å) with the aldehyde O atoms displaced from the plane by *ca* 1.0 Å and the dimethylamino groups (planar within 0.06 Å) tilted away from the pyridine plane by 20.5 (1)°. The C1—C2 bond length [1.438 (3) Å] is much longer than that normally found in pyridine [1.379 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19], as reasonably expected from consideration of steric effects.

Comment

The structure determination of the title compound (I) was carried out in order to obtain more structural parameters for 2,6-diaminopyridine derivatives and to see how a tetra-substituted pyridine skeleton would reflect its steric crowdedness in the bonding geometry. The effect of substituents on the conformation is important because 2,6-diaminopyridine derivatives form strong hydrogen-bonding interactions with nucleotide base pairs (Hamilton, Geib & Dixon, 1992). Such a skeleton forms part of many heterocycles which function as DNA intercalators (Mitcher & Rao, 1984).



(I)

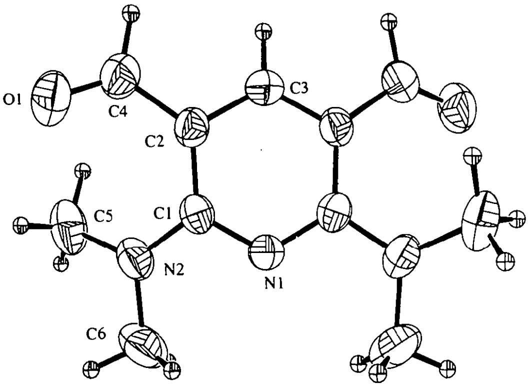


Fig. 1. View of $C_{11}H_{15}N_3O_2$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels. H atoms are shown as small circles of arbitrary radii.

Experimental

The title compound was obtained by reaction of 2,6-dimethoxypyridine with Vilsmeier reagent following a literature procedure (Shiao, Shyu & Tarng, 1990). Suitable crystals for this study were obtained from dichloromethane–hexane (1:2) solution as colorless rods (m.p. 412–414 K).

Crystal data

$C_{11}H_{15}N_3O_2$
$M_r = 221.26$
Monoclinic
$C2/c$
$a = 18.316\ (1)$ Å
$b = 8.270\ (1)$ Å
$c = 7.438\ (1)$ Å
$\beta = 92.358\ (3)°$
$V = 1125.7\ (2)$ Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8.04–18.52°$
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Rod
$0.63 \times 0.38 \times 0.38$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.908$, $T_{\max} = 0.999$
839 measured reflections
733 independent reflections
616 observed reflections $[F > 2\sigma(F)]$

$R_{\rm int} = 0.003$
$\theta_{\max} = 22.5°$
$h = -19 \rightarrow 19$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 7$
3 standard reflections
frequency: 60 min
intensity variation: 6%

Refinement

Refinement on F
$R = 0.033$
$wR = 0.041$
$S = 2.28$
616 reflections
105 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.100$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}
Extinction correction: secondary
Extinction coefficient: 0.151 (17)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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

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

	x	y	z	$U_{\rm eq}$
N1	0	0.1135 (3)	1/4	0.041 (1)
N2	0.1239 (1)	0.1030 (2)	0.2384 (2)	0.050 (1)
O1	0.1656 (1)	0.4129 (2)	0.0199 (2)	0.065 (1)
C1	0.0626 (1)	0.1932 (2)	0.2303 (2)	0.039 (1)
C2	0.0631 (1)	0.3653 (2)	0.2025 (3)	0.040 (1)
C3	0	0.4436 (4)	1/4	0.043 (2)
C4	0.1170 (1)	0.4606 (3)	0.1118 (3)	0.050 (2)
C5	0.1962 (1)	0.1701 (4)	0.2817 (4)	0.067 (1)
C6	0.1176 (2)	−0.0729 (3)	0.2419 (5)	0.072 (2)

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

N1—C1	1.336 (2)	O1—C4	1.211 (3)
N2—C1	1.348 (2)	C1—C2	1.438 (3)
N2—C5	1.459 (3)	C2—C3	1.383 (4)
N2—C6	1.460 (3)	C2—C4	1.451 (3)
C1—N1—C1$^{\rm i}$	120.81 (19)	N2—C1—C2	122.94 (16)
C1—N2—C5	123.13 (19)	C1—C2—C3	114.60 (17)
C1—N2—C6	119.06 (19)	C1—C2—C4	127.80 (17)
C5—N2—C6	116.53 (21)	C3—C2—C4	117.11 (19)
N1—C1—N2	116.06 (17)	C2—C3—C2$^{\rm i}$	124.15 (24)
N1—C1—C2	121.00 (16)		

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Intensity data were corrected for Lorentz, polarization and absorption effects. Data collection was carried out using *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction and structural refinements were performed using the *NRCVAX* package (Gabe, Lee & Le Page, 1985). The structure was solved by direct methods and refined with full-matrix least-squares cycles. All non-H atoms were refined with anisotropic displacement parameters. All H atoms were refined with isotropic displacement parameters. Fig. 1 was produced using *ORTEP*II (Johnson, 1976) and shows the molecular structure and atomic labelling.

We thank the National Science Council and Academia Sinica for the financial support.

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

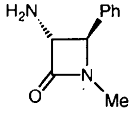
(*Received* 10 *June* 1994; *accepted* 1 *July* 1994)

Abstract

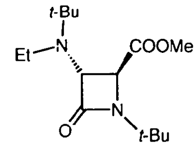
The central four-membered azetidinone ring of the title compound is puckered significantly with an average ring torsion angle of 5.1 (1)°. Molecules form infinite two-dimensional hydrogen-bonded networks *via* N—H···N [N···N 3.379 (3) Å] and N—H···O [N···O 3.056 (3) Å] interactions.

Comment

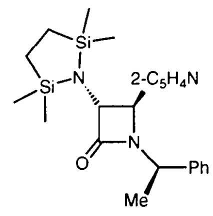
The title compound (I) belongs to a class of compounds called monobactams. Its core is a principal building block of natural and synthetic β-lactam antibiotics (*Chemistry and Biology of β-Lactam Antibiotics*, 1982). Its molecular structure (Fig. 1) comprises a four-membered cyclic amide with the phenyl 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

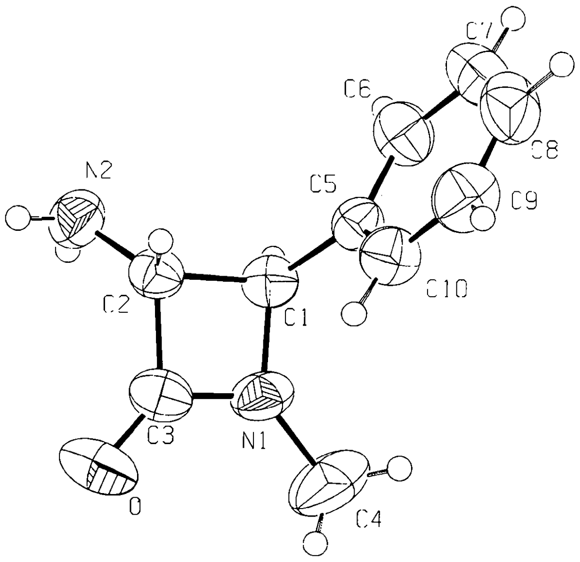


Fig. 1. Structure of (I) with displacement ellipsoids drawn at the 50% level.

Acta Crystallographica Section C
ISSN 0108-2701 ©1994