

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXTL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

*Acta Cryst.* (1998). **C54**, 1644–1645

## 5-Methoxycarbonyl-6-methyl-4-phenyl-3,4-dihydro-2(1*H*)-pyridone†

JULIO DUQUE RODRÍGUEZ,<sup>a</sup> RAMÓN POMÉS HERNÁNDEZ,<sup>b</sup> ARIEL GÓMEZ GONZÁLEZ,<sup>b</sup> MARGARITA SUÁREZ NAVARRO,<sup>c</sup> YAMILA VERDECIA REYES,<sup>c</sup> ESTHAEL OCHOA RODRÍGUEZ<sup>c</sup> AND YVONNE MASCARENHAS<sup>d</sup>

<sup>a</sup>X-ray Laboratory National Center for Scientific Research, PO Box 6990, Havana, Cuba, <sup>b</sup>X-ray Laboratory, National Center for Scientific Research, PO Box 6990, Havana, Cuba, <sup>c</sup>Laboratory of Organic Synthesis, Havana University, Havana, Cuba, and <sup>d</sup>Institute of Physics, University of San Carlos, San Carlos, Brazil. E-mail: xray@infomed.sld.cu

(Received 18 February 1997; accepted 27 November 1997)

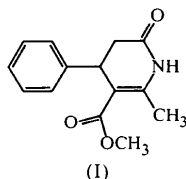
### Abstract

Crystals of the title compound, C<sub>14</sub>H<sub>15</sub>NO<sub>3</sub> are stabilized by intermolecular hydrogen bonds between the NH group and one O atom of the carbonyl group. The six-membered ring adopts a twist conformation and the lactone ring a planar conformation.

### Comment

Much effort has been devoted to the synthesis of 1,4-dihydropyridines (1,4-DHPs) because their calcium antagonist effect is useful in the treatment of cardiovascular diseases (Bossert & Vater, 1989). The presence of an aryl group on C-4 and ester groups on C-3 and C-5 of the 1,4-DHP ring has proved to be a basic requirement for pharmacological activity (Goldmann & Stoltefuss, 1991).

There are no unusual intermolecular or intramolecular distances or angles in the title compound, (I).



† Alternative name: methyl 1,4,5,6-tetrahydro-2-methyl-6-oxo-4-phenylpyridine-3-carboxylate.

There is an intramolecular N—H···O hydrogen bond with O1···H1(−*x*, 1−*y*, 1−*z*) = 2.05 (4) Å. The six-membered ring adopts a twist conformation with asymmetry parameters (Nardelli, 1983*a*)  $\Delta C_2(\text{C10—N1}) = 0.005$  (2) and  $\Delta C_2(\text{C11}) = 0.073$  (2).

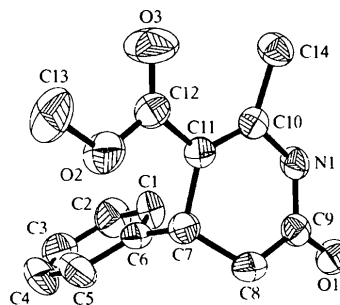


Fig. 1. A view of (I) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

### Experimental

The title compound was prepared in a two-step procedure from readily available 3,4-dihydro-2(1*H*)-pyridones by reaction with the Vilsmeier–Haack reagent (Verdecia *et al.*, 1995). Crystals suitable for X-ray analysis were obtained by slow evaporation from ethanol.

#### Crystal data

C<sub>14</sub>H<sub>15</sub>NO<sub>3</sub>  
*M<sub>r</sub>* = 245.28  
 Orthorhombic  
*Pbca*  
*a* = 8.983 (2) Å  
*b* = 21.125 (4) Å  
*c* = 13.196 (3) Å  
*V* = 2504.1 (9) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.301 Mg m<sup>−3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.71069 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 3–25°  
 $\mu$  = 0.0918 mm<sup>−1</sup>  
*T* = 293 (2) K  
 Prismatic  
 0.15 × 0.11 × 0.05 mm  
 Yellow

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$ –2 $\theta$  scans  
 Absorption correction: none  
 2870 measured reflections  
 2200 independent reflections  
 960 reflections with  $I > 2\sigma(I)$

*R<sub>int</sub>* = 0.041  
 $\theta_{\text{max}}$  = 25.0°  
 $h = -1 \rightarrow 10$   
 $k = -1 \rightarrow 25$   
 $l = -15 \rightarrow 1$   
 2 standard reflections every 100 reflections  
 intensity decay: <5%

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.050  
 $wR(F^2)$  = 0.10  
*S* = 1.165

$(\Delta/\sigma)_{\text{max}}$  = 0.059  
 $\Delta\rho_{\text{max}}$  = 0.203 e Å<sup>−3</sup>  
 $\Delta\rho_{\text{min}}$  = −0.158 e Å<sup>−3</sup>  
 Extinction correction: none

2200 reflections  
223 parameters  
All H-atom parameters  
refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.2855P]$   
where  $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from  
*International Tables for  
X-ray Crystallography*  
(Vol. C, Tables 4.2.6.8 and  
6.1.1.4)

Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N1—C9	1.362 (6)	C4—C5	1.393 (8)
N1—C10	1.403 (6)	C5—C6	1.385 (6)
O1—C9	1.227 (6)	C6—C7	1.522 (6)
O2—C12	1.323 (6)	C7—C8	1.524 (7)
O2—C13	1.445 (7)	C7—C11	1.522 (6)
O3—C12	1.189 (6)	C8—C9	1.493 (7)
C1—C2	1.380 (8)	C10—C11	1.339 (6)
C1—C6	1.384 (7)	C10—C14	1.491 (7)
C2—C3	1.366 (8)	C11—C12	1.469 (6)
C3—C4	1.34 (1)		
C9—N1—C10	125.5 (4)	C7—C8—C9	114.7 (4)
C12—O2—C13	116.6 (4)	O1—C9—C8	124.1 (4)
C2—C1—C6	121.7 (5)	N1—C9—C8	115.2 (4)
C1—C2—C3	120.0 (5)	N1—C9—O1	120.7 (4)
C2—C3—C4	119.3 (6)	N1—C10—C14	111.9 (4)
C3—C4—C5	121.9 (6)	N1—C10—C11	119.4 (4)
C4—C5—C6	119.7 (4)	C11—C10—C14	128.7 (4)
C1—C6—C5	117.5 (4)	C7—C11—C10	120.4 (4)
C5—C6—C7	120.8 (4)	C10—C11—C12	121.0 (4)
C1—C6—C7	121.7 (4)	C7—C11—C12	118.6 (4)
C6—C7—C11	112.8 (3)	O3—C12—C11	127.0 (4)
C6—C7—C8	111.8 (4)	O2—C12—C11	112.0 (4)
C8—C7—C11	110.0 (4)	O2—C12—O3	121.0 (4)

Table 2. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H $\cdots$ O1 <sup>i</sup>	0.86 (4)	2.05 (4)	2.903 (5)	179 (4)

Symmetry code: (i)  $-x, 1 - y, 1 - z$ .

The structure was solved by direct methods

Data collection: *CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *CRYSDA (DIRDIF; Beurskens et al., 1992)*. Data reduction: *REFLEX* (local program). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *PARST* (Nardelli, 1983*b*); *PARSTCIF* (Nardelli, 1992).

The authors thank the Third World Academy of Science for financial support through the TWAS research grant 94-001 RG/CHE/LA.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1040). Services for accessing these data are described at the back of the journal.

## References

- Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
- Bossert, F. & Vater, W. (1989). *Med. Res. Rev.* **9**, 291–324.
- Enraf–Nonius (1992). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
- Goldmann, S. & Stoltefuss, J. (1991). *Angew. Chem. Int. Ed. Engl.* **30**, 1559–1578.

- Nardelli M. (1983*a*). *Acta Cryst.* **C39**, 1141–1142.
- Nardelli, M. (1983*b*). *Comput. Chem.* **7**, 95–98.
- Nardelli, M. (1992). *PARSTCIF. Program for the Creation of a CIF from the Output of PARST*. University of Parma, Italy.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Verdecia, Y., Suarez, M., Morales, A., Rodriguez, E., Ochoa, E., Gonzalez, L., Martin, N., Quintero, M., Seoane, C & Soto, J. L. (1995). *J. Chem. Soc. Perkin Trans. 1*, pp. 947–951.

*Acta Cryst.* (1998). **C54**, 1645–1647

## *N*-Borane-*N*-(trimethylsilyl)morpholine

RAMON HUERTAS, JESUS R. MEDINA, JOHN A. SODERQUIST  
AND SONGPING D. HUANG

*Department of Chemistry, University of Puerto Rico, PO Box 23346, San Juan, PR 00931, USA. E-mail: huang@zintl.chem.uprr.pr*

(Received 2 February 1998; accepted 27 May 1998)

## Abstract

The title compound,  $C_7H_{20}BNOSi$ , features an *N,N*-disubstituted six-membered morpholine ring in a chair conformation, with the trimethylsilyl group in the equatorial position and the borane group in the axial position. The least-squares plane formed by the four C atoms of the morpholine ring has a mean deviation of 0.013 (2)  $\text{\AA}$ . The O and N atoms are 0.672 (2) and 0.650 (2)  $\text{\AA}$  above and below the plane, respectively.

## Comment

Borane–THF and borane dimethyl sulfide (BMS) are the most frequently used laboratory borane sources for hydroboration and other borane conversions (Brown, 1975; Soderquist, 1994). Both are safe and more convenient to handle than gaseous diborane. However, there are disadvantages of the use of either borane–THF or BMS as a borane source. For instance, concentrated solutions ( $>2M$ ) of borane–THF are unstable and can lose diborane. Moreover, the dimethyl sulfide complex is a foul-smelling liquid and dimethyl sulfide can contaminate the reaction solvents making their recovery difficult. As part of our efforts at developing new hydroborating agents, we carried out a reaction between gaseous diborane and *N*-(trimethylsilyl)morpholine in diethyl ether at 233 K yielding the title compound, (I), as the only product. In principle, two conformational isomers may exist with either one of the two different *N*-substituents occupying the axial or equatorial positions.