Crystal structure of 3-methylamino-2,2-diphenyl-2H-azirine, (HNCH₃)C₂N(C₆H₅)₂

K. Peters*^I, E.-M. Peters^I, T. Hergenröther^{II} and H. Quast^{II}^I Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70506 Stuttgart, Germany^{II} Universität Würzburg, Institut für Organische Chemie, Am Hubland, D-97074 Würzburg, Germany

Received October 4, 1999, CCDC-No. 1267/304

Abstract

C₁₅H₁₄N₂, orthorhombic, *Pbca* (No. 61), *a* = 10.008(5) Å, *b* = 9.044(4) Å, *c* = 28.05(1) Å, *V* = 2539.0 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.068, *wR*(*F*) = 0.062, *T* = 293 K.

Source of material

The title compound was prepared, according to [1] by irradiation of a degassed solution of 5-methylamino-4,4-diphenyl-4H-1,2,3-triazole [2] in dry [D₃]acetonitrile at 293 K (*λ* > 280 nm, focussed 500W high-pressure mercury lamp HBO 500 W/2 from Osram, quartz optics). Cooling the irradiated solution at 243 K afforded colourless crystals, mp 451 K – 452 K.

Discussion

A strong intermolecular hydrogen bridge *d*(N4...H2) = 198 pm forms a zigzag chain of molecules parallel [010].

Table 1. Data collection and handling.

Crystal:	colourless rod, size 0.4 × 0.4 × 1.0 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	0.70 cm ⁻¹
Diffraction, scan mode:	Siemens R3m/V, Wyckoff
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3350, 2922
Criterion for <i>F</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>F</i> _{obs} > 3 σ(<i>F</i> _{obs}), 2306
<i>N</i> (<i>param</i>) _{refined} :	158
Program:	SHELXTL-plus [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1A)	8c	0.6068(3)	0.0592(3)	0.5535(1)	0.08
H(1B)	8c	0.4919(3)	−0.0296(3)	0.5784(1)	0.08
H(1C)	8c	0.5528(3)	−0.0867(3)	0.5303(1)	0.08
H(2)	8c	0.656(3)	−0.220(3)	0.5949(9)	0.067(9)
H(7A)	8c	0.9833(3)	−0.0570(3)	0.5387(1)	0.08
H(8A)	8c	1.1665(4)	−0.1558(4)	0.4995(1)	0.08
H(9A)	8c	1.3142(4)	−0.3127(4)	0.5398(1)	0.08
H(10A)	8c	1.2770(3)	−0.3674(4)	0.6203(1)	0.08
H(11A)	8c	1.0968(3)	−0.2602(3)	0.6607(1)	0.08
H(13A)	8c	1.0432(3)	0.0649(4)	0.6951(1)	0.08
H(14A)	8c	1.0334(4)	0.0537(4)	0.7783(1)	0.08
H(15A)	8c	0.8791(4)	−0.0990(4)	0.8156(1)	0.08
H(16A)	8c	0.7385(4)	−0.2495(4)	0.7697(1)	0.08
H(17A)	8c	0.7482(3)	−0.2398(4)	0.6863(1)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	8c	0.5722(3)	−0.0378(3)	0.5599(1)	0.073(2)	0.063(2)	0.101(3)	0.012(2)	−0.028(2)	0.001(2)
N(2)	8c	0.6706(3)	−0.1234(3)	0.58623(9)	0.061(2)	0.038(1)	0.077(2)	0.002(1)	−0.019(1)	0.006(1)
C(3)	8c	0.7790(3)	−0.0603(3)	0.60319(9)	0.061(2)	0.037(1)	0.053(2)	0.008(1)	−0.003(1)	0.004(1)
N(4)	8c	0.8416(2)	0.0627(2)	0.60547(8)	0.070(2)	0.034(1)	0.064(1)	0.003(1)	−0.007(1)	0.005(1)
C(5)	8c	0.9018(3)	−0.0799(3)	0.62883(9)	0.058(2)	0.034(1)	0.051(2)	−0.002(1)	−0.002(1)	0.004(1)
C(6)	8c	1.0213(3)	−0.1458(3)	0.60413(9)	0.054(2)	0.039(1)	0.050(2)	−0.006(1)	−0.001(1)	−0.003(1)
C(7)	8c	1.0444(3)	−0.1189(3)	0.5560(1)	0.083(2)	0.060(2)	0.056(2)	0.006(2)	0.006(2)	0.008(2)

* Correspondence author

(e-mail: karpet@vsibm1.mpi-stuttgart.mpg.de)

Table 3. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(8)	8c	1.1520(4)	−0.1785(4)	0.5326(1)	0.103(3)	0.075(2)	0.068(2)	−0.005(2)	0.030(2)	−0.001(2)
C(9)	8c	1.2391(4)	−0.2707(4)	0.5562(1)	0.067(2)	0.083(3)	0.095(3)	−0.006(2)	0.019(2)	−0.027(2)
C(10)	8c	1.2175(3)	−0.3020(4)	0.6037(1)	0.066(2)	0.085(3)	0.088(2)	0.018(2)	−0.011(2)	−0.016(2)
C(11)	8c	1.1098(3)	−0.2393(3)	0.6275(1)	0.065(2)	0.072(2)	0.056(2)	0.010(2)	−0.005(2)	−0.006(2)
C(12)	8c	0.8967(3)	−0.0862(3)	0.68250(9)	0.053(2)	0.045(2)	0.052(2)	0.008(1)	0.002(1)	−0.001(1)
C(13)	8c	0.9798(3)	0.0002(4)	0.7101(1)	0.088(2)	0.078(2)	0.059(2)	−0.018(2)	0.006(2)	−0.012(2)
C(14)	8c	0.9738(4)	−0.0058(4)	0.7595(1)	0.106(3)	0.097(3)	0.062(2)	−0.008(2)	−0.004(2)	−0.023(2)
C(15)	8c	0.8844(4)	−0.0961(4)	0.7814(1)	0.108(3)	0.100(3)	0.050(2)	0.023(3)	0.009(2)	−0.005(2)
C(16)	8c	0.8013(4)	−0.1848(4)	0.7544(1)	0.097(3)	0.110(3)	0.067(2)	−0.009(3)	0.022(2)	0.016(2)
C(17)	8c	0.8071(3)	−0.1791(4)	0.7049(1)	0.075(2)	0.079(2)	0.060(2)	−0.011(2)	0.002(2)	0.007(2)

References

1. Hergenröther, T.: Photolyse und Thermolyse von 1,2,3-Triazolen und Tetrazolen. Dissertation, Universität Würzburg, Germany 1992.
2. Quast, H.; Hergenröther, T.; Banert, K.; Peters, E.-M.; Peters, K.; von Schnering, H. G.: Zur Tautomerie von 5,5-Diphenyldihydro-4*H*-1,2,3-triazol-4-on und 5-Amino-4,4-diphenyl-4*H*-1,2,3-triazolen. Chem. Ber. **126** (1993) 103-108.
3. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.