Crystal structure of 3a*S**,4*S**,7a*R**-4-ethoxy-3a,7a-dihydro-3,7-diphenyl-4*H*-1,5-dioxa-2,6-diaza-indene, $C_{19}H_{18}N_2O_3$

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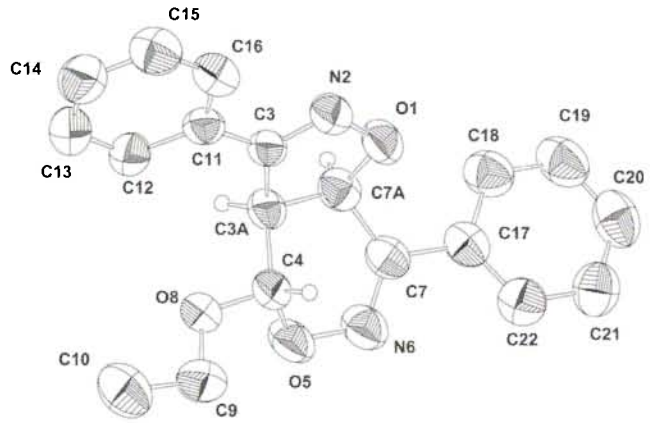
Received November 1, 1999, CCDC-No. 1267/326



Abstract

$C_{19}H_{18}N_2O_3$, orthorhombic, *Pbca* (No. 61), $a = 15.957(3)$ Å, $b = 7.958(2)$ Å, $c = 26.041(5)$ Å, $V = 3306.8$ Å^3, $Z = 8$, $R_{gt}(F) = 0.040$, $wR_{ref}(F^2) = 0.111$, $T = 293$ K.

Source of material

The title compound was obtained by 1,3-dipolar cycloaddition of in-situ generated benzonitrile oxide and 6-ethoxy-3-phenyl-6*H*-1,2-oxazine in diethyl ether (recrystallized from hexene/methylene chloride, 82 % yield) [1].

Table 1. Data collection and handling.

Crystal:	clear light yellow prism, size 0.6 × 0.8 × 0.8 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.89 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART1000, $\Delta\varphi = 0.3°$, 10 s/image
$2\theta_{max}$:	49.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5352, 2674
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1896
$N(param)_{refined}$:	218
Programs:	SHELXS-97 [2], SHELXL-97 [3], ZORTEP [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3A)	8*c*	0.6985	0.1880	0.3114	0.065
H(4)	8*c*	0.5281	0.1493	0.2794	0.067
H(7A)	8*c*	0.7422	0.1495	0.2316	0.069
H(9A)	8*c*	0.4316	0.2164	0.3399	0.100
H(9B)	8*c*	0.4588	0.4058	0.3440	0.100
H(10A)	8*c*	0.3986	0.3180	0.4207	0.144
H(10B)	8*c*	0.4926	0.3673	0.4298	0.144
H(10C)	8*c*	0.4670	0.1775	0.4259	0.144
H(12)	8*c*	0.6638	0.0725	0.3953	0.080
H(13)	8*c*	0.6592	–0.0597	0.4740	0.099
H(14)	8*c*	0.6425	–0.3459	0.4793	0.101
H(15)	8*c*	0.6275	–0.5013	0.4050	0.092
H(16)	8*c*	0.6284	–0.3712	0.3263	0.078
H(18)	8*c*	0.7556	0.1107	0.1434	0.090
H(19)	8*c*	0.7689	0.0887	0.0557	0.104
H(20)	8*c*	0.6690	0.1999	0.0021	0.099
H(21)	8*c*	0.5537	0.3349	0.0367	0.097
H(22)	8*c*	0.5395	0.3586	0.1244	0.085

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	8*c*	0.66391(9)	–0.0410(2)	0.21709(5)	0.092(1)	0.0527(9)	0.0569(8)	0.0044(8)	0.0002(7)	–0.0094(6)
N(2)	8*c*	0.6553(1)	–0.1439(2)	0.26076(6)	0.077(1)	0.051(1)	0.061(1)	0.0083(9)	–0.0017(8)	–0.0062(8)
C(3)	8*c*	0.6529(1)	–0.0541(2)	0.30149(7)	0.045(1)	0.052(1)	0.058(1)	0.0068(9)	–0.0054(8)	–0.0110(9)
C(3A)	8*c*	0.6560(1)	0.1319(2)	0.29024(7)	0.055(1)	0.048(1)	0.058(1)	–0.0023(9)	–0.0059(8)	–0.0118(8)
C(4)	8*c*	0.5722(1)	0.2179(2)	0.29503(6)	0.067(1)	0.047(1)	0.054(1)	0.005(1)	–0.0026(9)	–0.0080(8)
O(5)	8*c*	0.5771(1)	0.3802(2)	0.27030(5)	0.116(1)	0.047(1)	0.0645(9)	0.0145(8)	0.0115(8)	–0.0059(6)
N(6)	8*c*	0.5906(1)	0.3722(2)	0.21665(6)	0.102(2)	0.055(1)	0.063(1)	0.009(1)	0.011(1)	–0.0005(8)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	8*c*	0.6362(1)	0.2510(3)	0.20026(7)	0.062(1)	0.048(1)	0.065(1)	–0.005(1)	0.0066(9)	–0.005(1)
C(7A)	8*c*	0.6818(1)	0.1289(2)	0.23416(7)	0.053(1)	0.055(1)	0.065(1)	–0.006(1)	0.0004(9)	–0.0103(9)
O(8)	8*c*	0.55479(9)	0.2471(2)	0.34613(4)	0.0693(9)	0.065(1)	0.0551(7)	0.0156(7)	0.0000(6)	–0.0099(6)
C(9)	8*c*	0.4697(2)	0.2936(3)	0.35689(9)	0.076(2)	0.095(2)	0.080(2)	0.028(1)	0.006(1)	–0.007(1)
C(10)	8*c*	0.45574(7)	0.2887(2)	0.41334(4)	0.097(2)	0.106(2)	0.085(2)	0.005(2)	0.027(1)	–0.011(1)
C(11)	8*c*	0.64743(7)	–0.1354(2)	0.35219(4)	0.045(1)	0.055(1)	0.063(1)	0.0066(9)	–0.0031(8)	–0.0070(9)
C(12)	8*c*	0.65622(7)	–0.0432(2)	0.39695(4)	0.075(2)	0.062(2)	0.064(1)	0.008(1)	–0.016(1)	–0.007(1)
C(13)	8*c*	0.6538(2)	–0.1228(3)	0.44409(8)	0.101(2)	0.082(2)	0.064(1)	0.014(1)	–0.015(1)	–0.010(1)
C(14)	8*c*	0.6434(2)	–0.2931(3)	0.44743(9)	0.095(2)	0.088(2)	0.071(2)	0.014(2)	–0.000(1)	0.010(1)
C(15)	8*c*	0.6344(1)	–0.3854(3)	0.40304(8)	0.082(2)	0.066(2)	0.081(2)	0.000(1)	0.011(1)	0.006(1)
C(16)	8*c*	0.6356(1)	–0.3077(3)	0.35599(8)	0.066(1)	0.058(2)	0.071(1)	0.001(1)	0.007(1)	–0.008(1)
C(17)	8*c*	0.6461(1)	0.2383(2)	0.14362(7)	0.067(1)	0.048(1)	0.064(1)	–0.010(1)	0.010(1)	–0.0022(9)
C(18)	8*c*	0.7147(1)	0.1566(3)	0.12213(8)	0.074(2)	0.076(2)	0.074(1)	0.005(1)	0.014(1)	0.001(1)
C(19)	8*c*	0.7226(2)	0.1433(3)	0.06958(9)	0.097(2)	0.087(2)	0.075(2)	0.008(2)	0.028(1)	–0.003(1)
C(20)	8*c*	0.6631(2)	0.2093(3)	0.03748(9)	0.107(2)	0.078(2)	0.063(1)	–0.017(2)	0.017(1)	–0.003(1)
C(21)	8*c*	0.5945(2)	0.2899(3)	0.05826(8)	0.085(2)	0.090(2)	0.067(1)	–0.007(1)	0.002(1)	0.007(1)
C(22)	8*c*	0.5861(1)	0.3041(3)	0.11083(8)	0.068(1)	0.073(2)	0.071(1)	–0.003(1)	0.009(1)	0.003(1)

Acknowledgment. The authors thank Dr. Michael Ruf, BRUKER AXS; application and service center single crystal diffractometry, Karlsruhe for re-measurement the reflexion data with a SMART1000 diffraction system.

References

1. Schmidt, E.; Reißig, H.-U.: Unpublished results, Technische Universität Dresden, Germany 1999.
2. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Larger Structures. Acta Crystallogr. **A46** (1990)467-473.
3. Sheldrick, G. M.: SHELXL-97, a program for refining crystal structures. University of Göttingen, Germany 1997.
4. Zsolnay, L.: ZORTEP, PC version of ORTEP (C.K. Johnson). University of Heidelberg, Germany 1997.