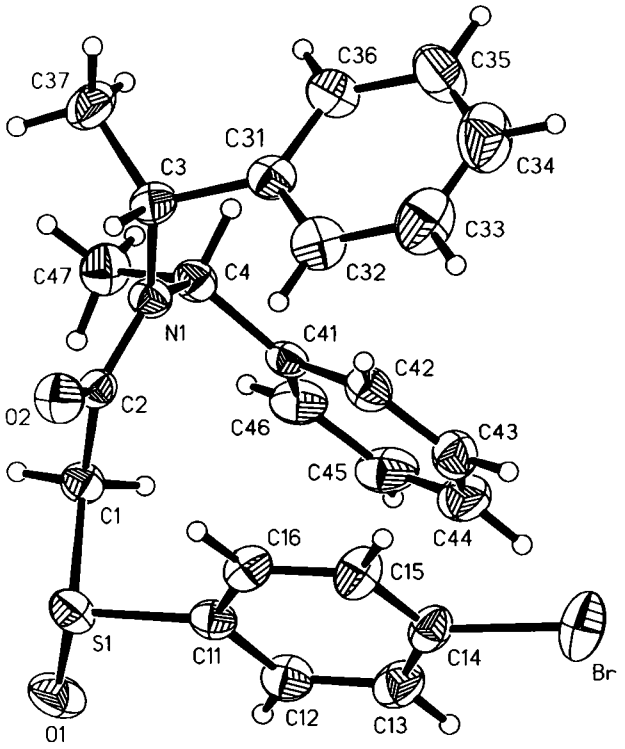
Crystal structure of ($R,R,R_S;S,S,S_S$)-4-bromobenzylsulfinylacetic acid *N,N*-bis-1-(phenylethyl)amide, $C_{24}H_{24}BrNO_2S$

T. Jablonska-Pikus, J. Kosior, K. Kurys and A. E. Koziol*

Maria Curie-Skłodowska University, Faculty of Chemistry, 20-031 Lublin, Poland

Received February 8, 2005, accepted and available on-line March 3, 2005; CCDC no. 1267/1479



Abstract

$C_{24}H_{24}BrNO_2S$, orthorhombic, *Pbca* (no. 61), $a = 12.388(2)$ Å, $b = 19.225(3)$ Å, $c = 18.534(3)$ Å, $V = 4414.0$ Å^3, $Z = 8$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.115$, $T = 293$ K.

Source of material

Synthesis – (4-bromophenylthio)acetyl chloride reacted with racemic (*RR*;*SS*)bis-1-(phenylethyl)amine at 0 °C in benzene. Resulting amide (m.p. 103-105 °C) was recrystallized from cyclohexane and next oxidized using H_2O_2 in acetic acid at 0 °C. Crystallization of the title compound (m.p. 131-132 °C) was performed from methanol/water solution.

Discussion

The title compound crystallizes as a racemic mixture. The absolute configuration of the chiral C3, C4 and S1 atoms of the presented enantiomer is R,R,R_S. The molecular conformation adopted in the solid-state results on *cisoidal* orientation of all three phenyl substituents in relation to a nearly coplanar fragment, i.e. the tertiary amide and methylenesulfoxide groups. Thus the exposition of the amide and sulfoxide oxygen atoms enables formation of the intermolecular C–H···O hydrogen bonds. Shortest C···O distances involving phenyl groups are: d(C36···O1′) = 3.389(6) Å, d(C44···O1′) = 3.396(6) Å, d(C16···O2′) = 3.267(5) Å and d(C15···O2′) = 3.285(6) Å.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.2 × 0.2 × 0.37 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	35.75 cm^{-1}
Diffractometer, scan mode:	Kuma KM4, $\omega/2\theta$
$2\theta_{max}$:	131.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4457, 3668
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1642
$N(param)_{refined}$:	265
Programs:	SHELXS-97 [1], SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	8c	0.4870	0.2245	0.0671	0.055
H(1B)	8c	0.5966	0.2339	0.0256	0.055
H(3)	8c	0.4354	0.0969	–0.1208	0.060
H(37A)	8c	0.4708	0.1918	–0.1925	0.088
H(37B)	8c	0.3508	0.2169	–0.1863	0.088
H(37C)	8c	0.3780	0.1480	–0.2278	0.088
H(4)	8c	0.2991	0.2433	–0.0764	0.056
H(47A)	8c	0.4633	0.2981	–0.1073	0.076
H(47B)	8c	0.4778	0.3168	–0.0255	0.076
H(47C)	8c	0.3796	0.3468	–0.0689	0.076
H(12)	8c	0.4766	0.1735	0.2296	0.069
H(13)	8c	0.3203	0.1230	0.2774	0.076
H(15)	8c	0.3327	–0.0225	0.1230	0.069
H(16)	8c	0.4891	0.0266	0.0770	0.060
H(32)	8c	0.3271	0.0268	–0.0420	0.080
H(33)	8c	0.1617	–0.0261	–0.0258	0.110
H(34)	8c	0.0099	0.0175	–0.0782	0.122
H(35)	8c	0.0209	0.1150	–0.1485	0.107
H(36)	8c	0.1850	0.1698	–0.1664	0.080
H(42)	8c	0.2534	0.1490	0.0372	0.062
H(43)	8c	0.1647	0.1565	0.1441	0.073
H(44)	8c	0.1635	0.2585	0.2090	0.086
H(45)	8c	0.2463	0.3547	0.1615	0.087
H(46)	8c	0.3351	0.3499	0.0523	0.073

* Correspondence author (e-mail: akoziol@hermes.umcs.lublin.pl)

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}
Br	8c	0.17966(5)	0.01007(3)	0.23496(4)	0.0838(4)	0.0709(4)	0.1029(5)	−0.0058(3)	0.0397(4)	0.0117(3)
S(1)	8c	0.6142(1)	0.14615(6)	0.11149(7)	0.0432(6)	0.0534(7)	0.0626(8)	0.0002(6)	−0.0092(6)	0.0042(7)
O(1)	8c	0.6524(3)	0.1965(2)	0.1669(2)	0.071(2)	0.068(2)	0.072(2)	−0.018(2)	−0.028(2)	−0.001(2)
C(1)	8c	0.5464(3)	0.1997(2)	0.0446(2)	0.046(3)	0.044(2)	0.048(3)	0.002(2)	−0.004(2)	0.002(2)
C(2)	8c	0.5039(4)	0.1557(2)	−0.0163(2)	0.046(2)	0.045(3)	0.038(3)	−0.004(2)	0.002(2)	0.004(2)
O(2)	8c	0.5511(2)	0.1007(2)	−0.0297(2)	0.059(2)	0.049(2)	0.065(2)	0.015(2)	0.002(2)	−0.005(2)
N(1)	8c	0.4171(3)	0.1772(2)	−0.0541(2)	0.044(2)	0.044(2)	0.041(2)	0.003(2)	−0.003(2)	−0.006(2)
C(3)	8c	0.3846(4)	0.1359(2)	−0.1178(2)	0.057(3)	0.052(3)	0.041(3)	−0.001(2)	0.001(2)	−0.013(2)
C(37)	8c	0.3972(4)	0.1769(3)	−0.1875(2)	0.083(4)	0.094(4)	0.042(3)	−0.026(3)	0.009(3)	−0.011(3)
C(4)	8c	0.3590(3)	0.2439(2)	−0.0419(2)	0.045(2)	0.045(3)	0.051(3)	0.004(2)	−0.004(2)	0.003(2)
C(47)	8c	0.4261(4)	0.3073(2)	−0.0628(3)	0.070(3)	0.048(3)	0.071(4)	−0.001(3)	0.002(3)	0.009(3)
C(11)	8c	0.4958(4)	0.1061(2)	0.1480(2)	0.049(3)	0.041(3)	0.046(3)	0.008(2)	−0.007(2)	0.002(2)
C(12)	8c	0.4470(4)	0.1342(2)	0.2079(3)	0.067(3)	0.051(3)	0.055(3)	0.001(3)	−0.005(3)	−0.013(3)
C(13)	8c	0.3536(4)	0.1046(3)	0.2366(3)	0.073(4)	0.069(3)	0.048(3)	0.007(3)	0.008(3)	−0.008(3)
C(14)	8c	0.3131(4)	0.0470(2)	0.2019(3)	0.057(3)	0.047(3)	0.051(3)	0.006(2)	0.011(3)	0.011(2)
C(15)	8c	0.3617(4)	0.0172(2)	0.1439(3)	0.072(3)	0.040(3)	0.060(3)	−0.008(2)	0.014(3)	−0.003(3)
C(16)	8c	0.4546(4)	0.0466(2)	0.1164(2)	0.063(3)	0.040(2)	0.047(3)	0.010(2)	0.007(3)	−0.003(2)
C(31)	8c	0.2730(4)	0.1041(2)	−0.1071(2)	0.060(3)	0.046(3)	0.046(3)	0.000(2)	−0.002(2)	−0.016(2)
C(32)	8c	0.2658(5)	0.0450(3)	−0.0640(3)	0.078(4)	0.050(3)	0.073(4)	−0.002(3)	−0.001(3)	−0.004(3)
C(33)	8c	0.1665(7)	0.0136(3)	−0.0543(4)	0.110(5)	0.063(4)	0.100(5)	−0.024(4)	0.016(4)	0.001(4)
C(34)	8c	0.0761(6)	0.0393(4)	−0.0853(4)	0.090(5)	0.100(6)	0.116(6)	−0.038(4)	0.016(5)	−0.015(5)
C(35)	8c	0.0828(4)	0.0971(4)	−0.1270(4)	0.055(4)	0.110(5)	0.103(5)	−0.005(4)	−0.013(3)	−0.003(4)
C(36)	8c	0.1815(4)	0.1300(3)	−0.1380(3)	0.063(3)	0.068(3)	0.068(3)	−0.001(3)	−0.010(3)	0.001(3)
C(41)	8c	0.3055(3)	0.2485(2)	0.0315(2)	0.037(2)	0.045(2)	0.042(3)	0.009(2)	−0.009(2)	0.000(2)
C(42)	8c	0.2526(4)	0.1912(2)	0.0617(3)	0.049(3)	0.049(3)	0.058(3)	0.007(2)	−0.004(3)	−0.004(2)
C(43)	8c	0.2000(4)	0.1955(3)	0.1260(3)	0.050(3)	0.068(3)	0.065(4)	0.009(3)	0.005(3)	0.009(3)
C(44)	8c	0.1981(4)	0.2561(3)	0.1646(3)	0.070(4)	0.093(4)	0.053(3)	0.023(4)	0.003(3)	−0.001(3)
C(45)	8c	0.2481(5)	0.3130(3)	0.1362(3)	0.092(4)	0.072(4)	0.052(3)	0.025(3)	−0.010(3)	−0.021(3)
C(46)	8c	0.3016(4)	0.3103(2)	0.0704(3)	0.077(4)	0.049(3)	0.056(3)	0.005(3)	−0.012(3)	−0.007(2)

References

1. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
2. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.