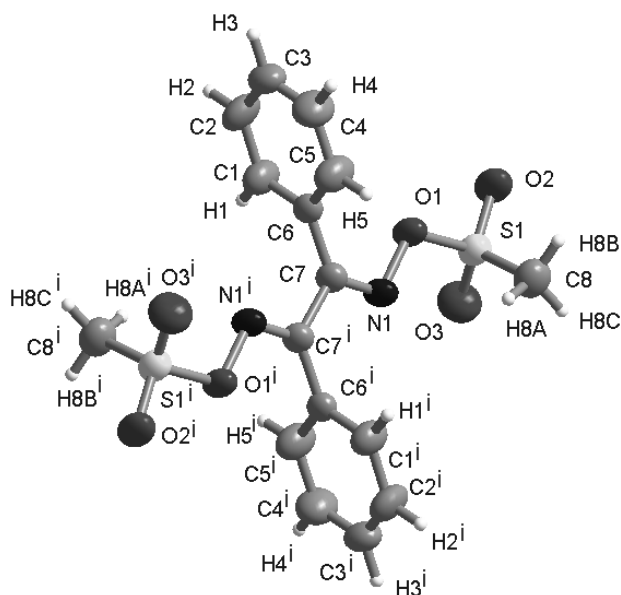


# Crystal structure of *N*-[(methylsulfonyl)oxy]-*N*-((*E*)-2-(methylsulfonyl)oxy]imino-1,2-diphenylethylidene)amine, C<sub>16</sub>H<sub>16</sub>N<sub>2</sub>O<sub>6</sub>S<sub>2</sub>

Ismail Warad\*, Mohammed Ghazzali, Saud Al-Resayes, Khalid Al-Farhan and Zied Al-Othman

King Saud University, Department of Chemistry, P.O. Box 2455, 11451 Riyadh, Saudi Arabia

Received May 22, 2010; accepted and available on-line August 4, 2010; CCDC no. 1267/3082



## Abstract

C<sub>16</sub>H<sub>16</sub>N<sub>2</sub>O<sub>6</sub>S<sub>2</sub>, triclinic, *P* $\bar{1}$  (no. 2), *a* = 5.392(2) Å, *b* = 9.300(4) Å, *c* = 9.420(4) Å,  $\alpha$  = 93.933(9)°,  $\beta$  = 90.20(1)°,  $\gamma$  = 106.375(9)°, *V* = 452.0 Å<sup>3</sup>, *Z* = 1, *R*<sub>g</sub>(*F*) = 0.047, *wR*<sub>ref</sub>(*F*<sup>2</sup>) = 0.157, *T* = 293 K.

## Source of material

All reagents and solvents use in this work were commercially available and used as purchased. Methanesulfonyl chloride (0.4 g, 3.5 × 10<sup>−3</sup> mole) was dissolved in 20 ml of dry THF and 4 ml of redistilled pyridine. The solution was cooled to −5 °C, then treated drop by drop with diphenylglyoxime solution (0.42 g, 1.75 × 10<sup>−3</sup> mole) dissolved in 20 ml of dry THF, during the addition the reaction temperature was kept constant for around one hour, the reaction then left to stir at room temperature for 12 h. 40 ml of distilled water and dichloromethane each was added to the reaction mixture, which was transferred to 200 ml separator funnel. The organic layer was separated and after evaporation by dichloromethane. White powder of 1.12 g (80 % yield) was collected. The crystals were grown by slow evaporation of solution of the compound in methanol.

## Discussion

Oximes/dioxime and their metal complexes are of current interest for their rich physicochemical properties, reactivity patterns and potential applications in many important chemical processes in

the areas of medicine [1], bioorganic systems [2], catalysis, electrochemical and electrooptical sensors [3,4]. The high stability of the complexes with vic-dioxime ligands has been extensively used for various purposes including model compounds for vitamin B<sub>12</sub> or trace metal analysis [5].

The *E* isomer of the title compound as a kinetically favored was detected over the *Z* isomer due to the steric factor. The single bond C7—C7<sup>i</sup> is clearly characterized by the distance of 1.439 Å. The double bond of C7=N1 and C7<sup>i</sup>=N1<sup>i</sup> is characterized by the distance of 1.281 Å. The conjugation of the  $\pi$  bonding electrons in ON1=C7—C7<sup>i</sup>=N1<sup>i</sup>O system reduced the length of the C7—C7<sup>i</sup> bond. The molecules packing in the crystal structure (figure, bottom) is stabilized by three weak intermolecular interactions, of which oxygen O3 of the sulfonyl functions in both methylsulfonyl moiety works as acceptor. There are two type of donors which interact with O3. The distance H8A...O3 of the methyl group is 2.611 Å and the angle C8—H8A...O3 is 153.85°. The distance of the interaction C1—H1...O3 is 2.702 Å and the relevant angle is 137.73°. There is no intramolecular H-bond in the molecule.

**Table 1.** Data collection and handling.

Crystal:	orange block, size 0.20 × 0.20 × 0.50 mm
Wavelength:	Mo <i>K</i> $\alpha$ radiation (0.71075 Å)
$\mu$ :	3.30 cm <sup>−1</sup>
Diffractionmeter, scan mode:	Rigaku RAXIS-RAPID, $\omega$
2 $\theta$ <sub>max</sub> :	54.94°
<i>N</i> ( <i>hkl</i> ) <sub>measured</sub> , <i>N</i> ( <i>hkl</i> ) <sub>unique</sub> :	2059, 2059
Criterion for <i>I</i> <sub>obs</sub> , <i>N</i> ( <i>hkl</i> ) <sub>gt</sub> :	<i>I</i> <sub>obs</sub> > 2 $\sigma$ ( <i>I</i> <sub>obs</sub> ), 1741
<i>N</i> ( <i>param</i> ) <sub>refined</sub> :	119
Programs:	SHELXS-97, SHELXL-97 [6]

**Table 2.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub>
H(1)	2i	0.0714	−0.1934	0.1591	0.072
H(2)	2i	0.0457	−0.3284	0.3578	0.082
H(3)	2i	0.3920	−0.2880	0.5095	0.073
H(4)	2i	0.7694	−0.1121	0.4660	0.086
H(5)	2i	0.8001	0.0284	0.2702	0.078
H(8B)	2i	0.6361	0.5016	0.2908	0.079
H(8C)	2i	0.5513	0.5568	0.1498	0.079
H(8A)	2i	0.6682	0.4202	0.1436	0.079

\* Correspondence author (e-mail: warad@ksu.edu.sa)

**Table 3.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>
S(1)	2 <i>i</i>	0.2528(1)	0.34796(6)	0.20961(6)	0.0526(3)	0.0428(3)	0.0491(3)	0.0190(2)	0.0153(2)	0.0069(2)
O(1)	2 <i>i</i>	0.3099(3)	0.1861(2)	0.2063(2)	0.081(1)	0.0468(8)	0.0437(8)	0.0287(8)	0.0256(8)	0.0103(6)
O(2)	2 <i>i</i>	0.1569(4)	0.3603(2)	0.3479(2)	0.092(1)	0.056(1)	0.060(1)	0.0302(9)	0.038(1)	0.0083(8)
O(3)	2 <i>i</i>	0.0999(4)	0.3546(2)	0.0894(2)	0.068(1)	0.083(1)	0.071(1)	0.038(1)	−0.0050(9)	0.005(1)
N(1)	2 <i>i</i>	0.3964(4)	0.1485(2)	0.0681(2)	0.069(1)	0.050(1)	0.0383(9)	0.0260(9)	0.0192(8)	0.0065(7)
C(1)	2 <i>i</i>	0.2136(5)	−0.1752(3)	0.2208(2)	0.055(1)	0.057(1)	0.060(1)	0.000(1)	−0.005(1)	0.016(1)
C(2)	2 <i>i</i>	0.1990(5)	−0.2564(3)	0.3397(3)	0.065(2)	0.057(2)	0.074(2)	−0.006(1)	0.005(1)	0.026(1)
C(3)	2 <i>i</i>	0.4043(6)	−0.2326(3)	0.4300(2)	0.086(2)	0.056(1)	0.046(1)	0.026(1)	0.007(1)	0.015(1)
C(4)	2 <i>i</i>	0.6277(6)	−0.1279(3)	0.4043(3)	0.073(2)	0.079(2)	0.061(2)	0.015(2)	−0.014(1)	0.015(1)
C(5)	2 <i>i</i>	0.6465(5)	−0.0440(3)	0.2867(3)	0.053(1)	0.067(2)	0.068(2)	0.000(1)	−0.006(1)	0.020(1)
C(6)	2 <i>i</i>	0.4382(4)	−0.0678(2)	0.1942(2)	0.048(1)	0.039(1)	0.0380(9)	0.0152(8)	0.0095(8)	0.0015(7)
C(7)	2 <i>i</i>	0.4541(4)	0.0241(2)	0.0699(2)	0.047(1)	0.044(1)	0.040(1)	0.0148(8)	0.0114(9)	0.0036(8)
C(8)	2 <i>i</i>	0.5618(5)	0.4704(3)	0.1971(3)	0.063(2)	0.059(2)	0.071(2)	0.009(1)	0.015(1)	0.007(1)

*Acknowledgment.* The authors gratefully acknowledges to the Sabic company for their supporting and technical assistance.

## References

1. Melson, G. A.: Coordination Chemistry of Macrocyclic Compounds. Plenum Press, New York, 1979.
2. Blower, J. P.: Small coordination complexes as radiopharmaceuticals for cancer targeting. *Transit. Metal Chem.* **23** (1998) 109–112.
3. Wolkert, A. W.; Hoffman, J. T.: Therapeutic Radiopharmaceuticals. *Chem. Rev.* **99** (1999) 2269–2292.
4. Mitchell, P.: Keilin's respiratory chain concept and its chemiosmotic consequences. *Science* **206** (1979) 1148–1159.
5. Laranleira, C. M.; Marusak, A. R.; Lappin, G. A.: Driving force effects in proton coupled electron transfer. *Inorg. Chim. Acta* **300** (2000) 186–190.
6. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.