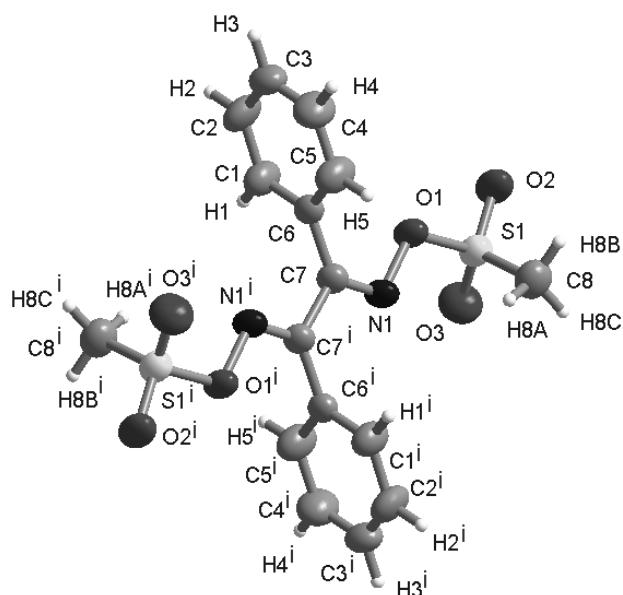


Crystal structure of *N*-[(methylsulfonyl)oxy]-*N*-((*E*)-2-(methylsulfonyl)oxy)imino-1,2-diphenylethylidene)amine, $C_{16}H_{16}N_2O_6S_2$

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Abstract

$C_{16}H_{16}N_2O_6S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 5.392(2)$ Å, $b = 9.300(4)$ Å, $c = 9.420(4)$ Å, $\alpha = 93.933(9)$ °, $\beta = 90.20(1)$ °, $\gamma = 106.375(9)$ °, $V = 452.0$ Å³, $Z = 1$, $R_{gt}(F) = 0.047$, $wR_{ref}(F^2) = 0.157$, $T = 293$ K.

Source of material

All reagents and solvents used in this work were commercially available and used as purchased. Methanesulfonyl chloride (0.4 g, 3.5×10^{-3} 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^{-3} 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 B₁₂ 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—C7ⁱ is clearly characterized by the distance of 1.439 Å. The double bond of C7=N1 and C7ⁱ=N1ⁱ is characterized by the distance of 1.281 Å. The conjugation of the π bonding electrons in ON1=C7—C7ⁱ=N1ⁱO system reduced the length of the C7—C7ⁱ bond. The molecules packing in the crystal structure (figure, bottom) is stabilized by three weak intermolecular interactions, of which oxygen O3 of the sulphonyl functions in both methylsulphonyl 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 $K\alpha$ radiation (0.71075 Å)
μ :	3.30 cm ⁻¹
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{\max}$:	54.94°
$N(hkl)$ measured, $N(hkl)$ unique:	2059, 2059
Criterion for I_{obs} , $N(hkl)$ gt:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1741
$N(\text{param})$ refined:	119
Programs:	SHELXS-97, SHELXL-97 [6]

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

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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
S(1)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)	<i>2i</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)

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