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3-[3,5-Dimethoxy-2-(trimethylsilyl)phenyl]-4-trimethylsilylsydnone

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Key indicators

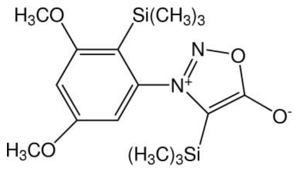
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.066
wR factor = 0.152
Data-to-parameter ratio = 26.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{16}H_{26}N_2O_4Si_2$, is a mesoionic compound of the sydnone class. Its molecular structure reveals the two planar ring fragments mutually twisted by 74.88 (10)°. The crystal packing is characterized by a one-dimensional molecular array generated by C—H···O interactions.

Comment

The title compound, (I), was synthesized as part of a series of sydnones to determine the effect of substitution on the sydnone ring. The focus of the synthesis was to design substituents on the sydnone ring that would allow a release of NO *via* ring opening. Generally, for stabilization of the system the substitution at the N4 position by an aromatic fragment is necessary. In previous work (Turnbull & Krein, 1997) we have shown that the sydnone ring controls *ortho*-lithiation upon treatment of an arylsydnone with an alkyllithium, followed by a suitable electrophile. Disubstitution (at the *ortho*-aryl and sydnone 4-positions) using reactive electrophiles occurs, whereas *ortho*-substitution alone can be effected with less reactive electrophiles such as Weinreb amides (Turnbull *et al.*, 1998). Given this powerful avenue to aryl ring substitution, (I) was synthesized by a lithiation protocol from 3-(3,5-dimethoxyphenyl)sydnone as part of a study aimed at determining the relative directing effect for *ortho*-lithiation of the methoxy groups and the sydnone ring. The structure reported here (Fig. 1) confirms the planned substitution reaction.



(I)

In this compound, the benzene and sydnone rings are almost perpendicular owing to the bulky trimethylsilyl substituents on each ring. The angle between the mean planes is 74.88 (10)°. The molecules are connected by two inversion-related C—H···O hydrogen bonds involving methoxy O atoms and methyl H atoms of the trimethylsilyl group (Table 1, Fig. 2), generating a dimer. The dimers formed are interconnected into a chain along *a* through C15—H15*C*···O5^{i} and C14—H14···O5^{i} hydrogen bonds (symmetry code in Table 1).

Experimental

The title compound (1) was prepared by the treatment of 3-(3,5-dimethoxyphenyl)sydnone with butyllithium followed by chlorotrimethylsilane and recrystallization from dichloromethane and hexane (3:1) (Sun, 2005).

Crystal data

$C_{16}H_{26}N_2O_4Si_2$
$M_r = 366.57$
Monoclinic, $P2_1/c$
$a = 12.5402$ (12) Å
$b = 8.1958$ (8) Å
$c = 19.3421$ (19) Å
$\beta = 98.786$ (2)°
$V = 1964.6$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.20$ mm^{-1}
$T = 100$ (2) K
0.53 × 0.41 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{\min} = 0.933$, $T_{\max} = 0.968$
22233 measured reflections
5968 independent reflections
5726 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.152$
$S = 1.22$
5968 reflections
225 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O5^{i}	0.95	2.63	3.219 (3)	120
C15—H15C···O5^{i}	0.98	2.67	3.594 (3)	158
C18—H18C···O13^{ii}	0.98	2.62	3.549 (4)	159

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x, -y+2, -z+1$.

All H atoms were placed in calculated positions with C—H$_{\rm methyl}$ = 0.98 Å and C—H$_{\rm aromatic}$ = 0.95 Å. They were included in the refinement in the riding-model approximation with $U_{\rm iso}$(H) values of 1.2 or 1.5 times $U_{\rm eq}$ of the carrier atom. Residual electron density is found in the vicinity of the Si atoms, most likely due to inadequacy in the displacement parameters.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (McArdle, 1995), *PLATON* (Spek, 2003) and *Mercury* (Macrae *et al.*, 2006).

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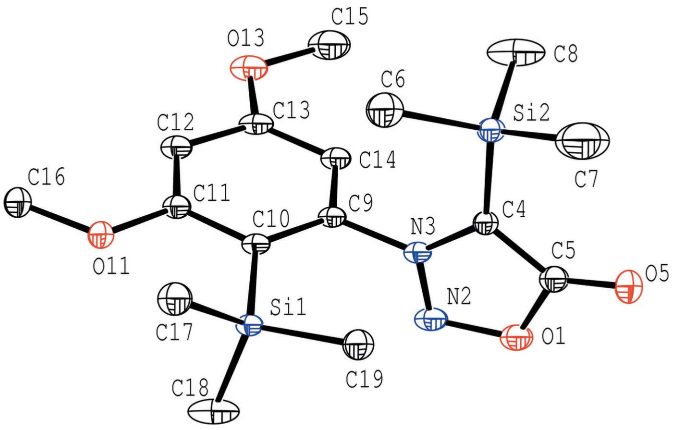

Figure 1
The molecular structure of (I) showing 40% probability displacement ellipsoids. H atoms have been omited for clarity.

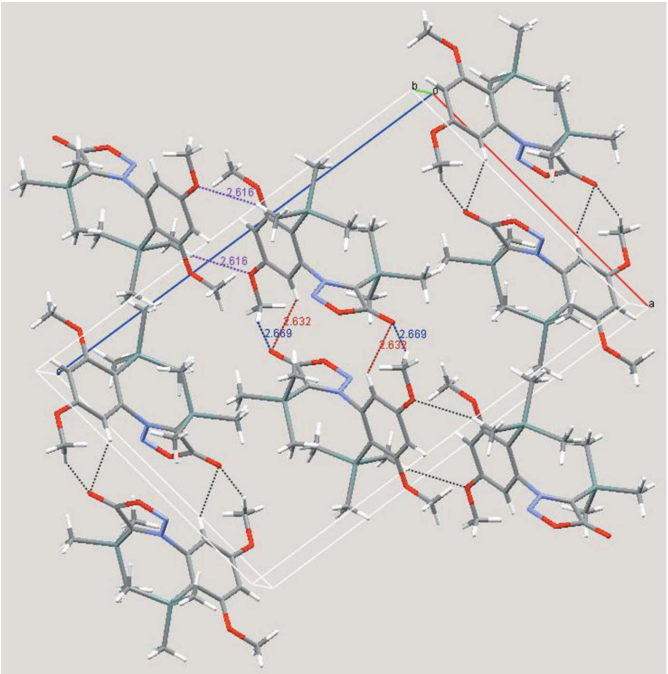

Figure 2
Packing diagram for (I), showing the closest intermolecular contacts (dashed lines).

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