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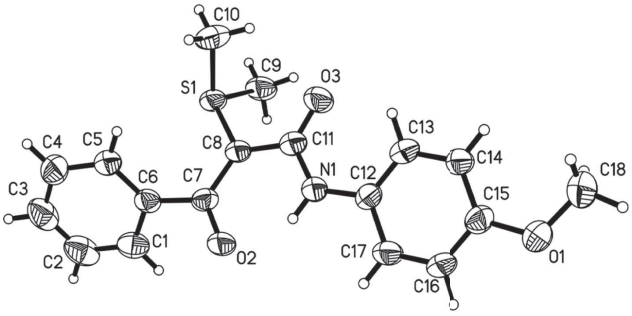
Crystal structure of 2-(dimethylsulfanylidene)-N-(4-methoxyphenyl)-3-oxo-3-phenylpropanamide



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Abstract

$C_{18}H_{19}NO_3S$, monoclinic, *Pc* (no. 7), $a = 7.710(2)$ Å, $b = 5.701(2)$ Å, $c = 19.053(6)$ Å, $\beta = 99.031(6)°$, $V = 827.1(4)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0361$, $wR_{ref}(F^2) = 0.0885$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

2-(Dimethylsulfuranylidene)acetophenone and 4-methoxy-*N*-(pivaloyloxy) benzamide were prepared according to the methods reported in the literatures [9, 10]. A solution of 4-methoxy-*N*-(pivaloyloxy) benzamide (5.026 g, 0.02 mol), 2-(dimethylsulfuranylidene) acetophenone (3.605 g, 0.02 mol), NaOH (0.800 g, 0.02 mol) in 1,2-dichloroethane (DCE) (70 mL) was stirred at 50 °C under Ar. After the reaction, the mixture was washed with water three times. The organic phase was dried with anhydrous Na_2SO_4, and concentrated. The crude products were purified by crystallization by ethyl acetate/hexane. The title compound was obtained as white solid in 92% yield.

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Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.17×0.18×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.10 cm^{-1}
Diffractometer, scan mode:	Bruker APEX2 CCD area-detector diffractometer, φ and ω scans
$2\theta_{max}$:	55.28°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5612, 2730
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2343
$N(param)_{refined}$:	208
Programs:	Bruker programs [11], SIR97 [12], SHELX [13], WinGX[14]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*a*	0.5367	0.7175	0.1457	0.056
H(13)	2*a*	0.3403	0.2550	0.2212	0.058
H(1A)	2*a*	0.5037	1.4066	0.0396	0.063
H(17)	2*a*	0.7836	0.6155	0.2241	0.067
H(14)	2*a*	0.4841	−0.0013	0.3037	0.059
H(5)	2*a*	0.1597	0.9511	−0.0644	0.058
H(9A)	2*a*	−0.1570	1.0885	0.1390	0.085
H(9B)	2*a*	0.0339	1.1610	0.1730	0.085
H(9C)	2*a*	−0.0306	0.9028	0.1806	0.085
H(16)	2*a*	0.9279	0.3596	0.3049	0.071
H(18A)	2*a*	0.8067	−0.2317	0.4345	0.099
H(18B)	2*a*	0.6663	−0.2558	0.3659	0.099
H(18C)	2*a*	0.6402	−0.0700	0.4240	0.099
H(3)	2*a*	0.2680	1.5501	−0.1570	0.086
H(10A)	2*a*	−0.2100	0.7530	0.0271	0.099
H(10B)	2*a*	−0.0803	0.5941	0.0780	0.099
H(10C)	2*a*	−0.0481	0.6381	−0.0001	0.099
H(4)	2*a*	0.1160	1.2009	−0.1612	0.075
H(2)	2*a*	0.4550	1.6569	−0.0562	0.080

Experimental details

All H atoms were added according to theoretical models with isotropic displacement parameters and allowed to ride on their parent atoms [C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C),N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N)].

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*a*	0.04081(6)	0.9589(1)	0.06923(4)	0.0265(3)	0.0535(3)	0.0540(3)	−0.0007(3)	0.0034(2)	0.0069(3)
O(2)	2*a*	0.5458(3)	0.9552(3)	0.0771(1)	0.0279(8)	0.077(1)	0.061(1)	−0.0008(9)	0.0069(8)	0.019(1)
N(1)	2*a*	0.4653(2)	0.6347(4)	0.1658(1)	0.032(1)	0.061(1)	0.047(1)	−0.0014(9)	0.0033(9)	0.013(1)
O(1)	2*a*	0.8166(3)	0.0203(4)	0.3657(1)	0.043(1)	0.075(1)	0.076(1)	0.0037(9)	−0.002(1)	0.031(1)
C(7)	2*a*	0.3846(3)	0.9868(4)	0.0589(1)	0.029(1)	0.050(1)	0.045(1)	0.000(1)	0.005(1)	−0.001(1)
C(6)	2*a*	0.3346(3)	1.1522(4)	−0.0018(1)	0.032(1)	0.046(1)	0.045(1)	0.004(1)	0.011(1)	0.002(1)
C(15)	2*a*	0.7196(3)	0.1585(4)	0.3153(1)	0.035(1)	0.055(1)	0.047(1)	0.008(1)	0.003(1)	0.004(1)
C(13)	2*a*	0.4583(3)	0.2792(4)	0.2389(1)	0.033(1)	0.056(1)	0.053(2)	−0.005(1)	−0.004(1)	0.002(1)
C(12)	2*a*	0.5438(3)	0.4674(4)	0.2151(1)	0.034(1)	0.048(1)	0.035(1)	0.002(1)	0.0039(9)	−0.002(1)
C(8)	2*a*	0.2561(3)	0.8687(4)	0.0922(1)	0.026(1)	0.050(1)	0.045(1)	0.000(1)	0.0043(9)	0.005(1)
C(1)	2*a*	0.4245(3)	1.3650(4)	−0.0005(2)	0.045(1)	0.048(1)	0.066(2)	−0.001(1)	0.015(1)	−0.007(1)
C(11)	2*a*	0.2926(3)	0.6833(4)	0.1455(1)	0.033(1)	0.049(1)	0.046(1)	−0.001(1)	0.004(1)	0.002(1)
C(17)	2*a*	0.7219(3)	0.4923(5)	0.2406(2)	0.032(1)	0.062(2)	0.071(2)	−0.005(1)	0.005(1)	0.020(1)
C(14)	2*a*	0.5444(3)	0.1253(4)	0.2884(2)	0.039(1)	0.048(1)	0.059(2)	−0.003(1)	0.004(1)	0.007(1)
C(5)	2*a*	0.2199(3)	1.0928(5)	−0.0626(2)	0.038(1)	0.056(1)	0.050(2)	−0.002(1)	0.004(1)	0.005(1)
C(9)	2*a*	−0.0373(3)	1.0369(5)	0.1498(2)	0.035(1)	0.069(2)	0.069(2)	−0.001(1)	0.015(1)	−0.002(1)
C(16)	2*a*	0.8083(3)	0.3403(5)	0.2892(2)	0.028(1)	0.070(2)	0.075(2)	−0.001(1)	−0.004(1)	0.018(2)
C(18)	2*a*	0.7251(4)	−0.1478(6)	0.4003(2)	0.065(2)	0.067(2)	0.064(2)	0.006(2)	0.005(2)	0.019(2)
C(3)	2*a*	0.2839(4)	1.4505(6)	−0.1179(2)	0.054(2)	0.078(2)	0.085(3)	0.015(2)	0.021(2)	0.041(2)
C(10)	2*a*	−0.0895(4)	0.7068(5)	0.0402(2)	0.041(1)	0.073(2)	0.078(2)	−0.011(1)	−0.005(1)	−0.004(2)
C(4)	2*a*	0.1938(4)	1.2417(6)	−0.1207(2)	0.045(1)	0.085(2)	0.057(2)	0.006(2)	0.003(1)	0.016(2)
C(2)	2*a*	0.3969(4)	1.5135(5)	−0.0580(2)	0.060(2)	0.043(1)	0.101(3)	0.003(1)	0.023(2)	0.017(2)
O(3)	2*a*	0.1751(2)	0.5801(3)	0.1698(1)	0.0362(9)	0.070(1)	0.074(1)	−0.0036(9)	0.0086(9)	0.024(1)

Discussion

Sulfur ylides has becoming more and more important in organic synthesis for the construction of organic molecules with diversified and complicated structures [1]. A sulfur ylide is a nucleophilic reagent with leaving group. It reacts easily with electron-deficient compounds, such as aldehydes, ketones and imines [2]. Recently, sulfur ylides are found to participate in domino cyclization reaction to synthesize derivatives of three-membered rings, five-memberd rings, six-membered rings and bridged ring systems [3–8]. Because of the general usability of ylids as synthetic intermediates, it has been desirable to investigate the synthesis of different ylids. The title compound, a new sulfur ylide, was synthesized for the first time by the Lossen rearrangement of 2-(dimethylsulfuranylidene)acetophenone with 4-methoxy-*N*-(pivaloyloxy)benzamide in the presence of a base. The crystal structure contains one molecues in the asymmetric unit. In the molecule, the bond lengths and bond angles in the phenyl rings are generally normal. The torsion angles of C(1)—C(6)—C(7)—C(8) and C(8)—C(11)—N(1)—C(12) are 52.8° and 18.1°, respectively. The crystal structure is stabilized by an intramolecular N—H$\cdots$O hydrogen bond and weak intermolecular C—H$\cdots$O hydrogen bonds.

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