Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methoxy-5-nitrobenzene-1,3-diyl bis(benzenesulfonate)

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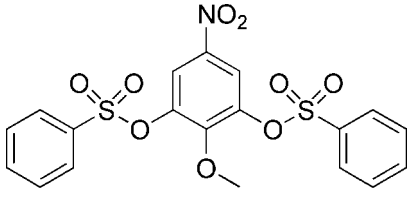
Received 25 August 2007; accepted 7 September 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.131; data-to-parameter ratio = 13.7.

In the title molecule, $C_{19}H_{15}NO_9S_2$, the dihedral angle between the nitro group and the benzene ring to which it is attached is 11.68 (8)°. The N—C bond length of 1.466 (3) Å indicates that no conjugation exists between the nitro group and the benzene ring.

Related literature

For related literature, see: Desai *et al.* (2001); Hoogenraad *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{19}H_{15}NO_9S_2$
M_r = 465.44
Monoclinic, $P2_1/c$
a = 10.787 (2) Å
b = 10.895 (2) Å
c = 16.876 (3) Å
β = 93.91 (3)°
V = 1978.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.909, T_{max} = 0.938
4084 measured reflections
3872 independent reflections
2898 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
S = 1.05
3872 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The work was financed by grants (project 30672516) from the National Natural Science Foundation of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2490).

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  doi:10.1107/S160053680704398X

supplementary materials

Acta Cryst. (2007). E**63**, o4050 [**doi:10.1107/S160053680704398X**]

2-Methoxy-5-nitrobenzene-1,3-diyl bis(benzenesulfonate)

Z.-P. Xiao, R.-Q. Fang, H.-Q. Li and H.-L. Zhu

Comment

Aromatic nitro compounds are potential intermediates for a variety of fine chemicals. They are easily reduced to the corresponding aromatic amines (Desai *et al.*, 2001). Therefore, aromatic nitro compounds are frequently used in the synthesis of pharmaceuticals (Hoogenraad *et al.*, 2004). We herein reported a crystal structure of 2-methoxy-5-nitrobenzene-1,3-diyl dibenzenesulfonate. As shown in Fig. 1, the nitro group (N1/O1/O2) makes a dihedral angle with the benzene ring (C1—C6) of 11.68 (8) °. In spite of the fact that the nitro group is almost coplanar with the benzene ring, no conjugation is observed as evidenced by the N1—C1 bond length of 1.466 (3) Å. This is an agreement with the standard values of single N—C bond (1.47–1.50 Å) and all other bonds distances are in the the normal ranges (Allen *et al.*, 1987).

Experimental

K_2CO_3 was charged into a solution of 2-hydroxy-5-nitrobenzene-1,3-diyl dibenzenesulfonate (4.66 g, 10 mmol) in anhydrous acetone (30 ml) with stirring. CH_3I (0.66 ml, 13 mmol) was added dropwise to the reaction mixture at room temperature. The reaction mixture was then refluxed for 8 h. The residue was filtered and the cake washed with acetone. A shallow yellow solid was given in a yield of 85% after removal of the solvent. Recrystallization from acetone furnished volorless blocks for single-crystal structure determination.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) and C—H = 0.93 Å for the aromatic H atoms or U_{iso}(H) = 1.5U_{eq}(C) and C—H = 0.96 Å for methyl H atoms.

Figures

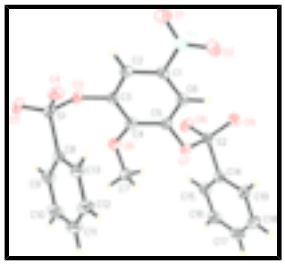


Fig. 1. Molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

2-Methoxy-5-nitrobenzene-1,3-diyl bis(benzenesulfonate)

Crystal data

$C_{19}H_{15}NO_9S_2$ F_{000} = 960

M_r = 465.44	D_x = 1.562 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2407 reflections
a = 10.787 (2) Å	θ = 1.3–25.4º
b = 10.895 (2) Å	μ = 0.32 mm^{-1}
c = 16.876 (3) Å	T = 298 (2) K
β = 93.91 (3)º	Block, colorless
V = 1978.7 (6) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	3872 independent reflections
Radiation source: fine-focus sealed tube	2898 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.023
T = 298(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = 0→13
T_{min} = 0.909, T_{max} = 0.938	k = 0→13
4084 measured reflections	l = −20→20

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.2038P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.131	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.05	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
3872 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
282 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.029 (2)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S2	−0.23217 (6)	0.74187 (5)	0.01047 (4)	0.03603 (19)
S1	0.37812 (6)	0.76657 (6)	−0.11612 (4)	0.0407 (2)
O3	0.23769 (16)	0.81284 (14)	−0.13794 (10)	0.0410 (4)
O7	−0.09188 (15)	0.68927 (15)	0.02396 (9)	0.0404 (4)
O6	0.10742 (17)	0.85730 (15)	−0.01565 (10)	0.0461 (5)
O9	−0.30660 (17)	0.64741 (17)	−0.02568 (10)	0.0515 (5)
O4	0.39210 (18)	0.64822 (17)	−0.15073 (11)	0.0524 (5)
O8	−0.22762 (19)	0.85748 (16)	−0.02797 (11)	0.0559 (5)
O2	−0.0900 (2)	0.38064 (19)	−0.19229 (12)	0.0659 (6)
O5	0.44911 (18)	0.86750 (18)	−0.14039 (12)	0.0596 (5)
C8	0.3875 (2)	0.7536 (2)	−0.01236 (14)	0.0377 (5)
C3	0.1401 (2)	0.7322 (2)	−0.12367 (14)	0.0354 (5)
C4	0.0728 (2)	0.7565 (2)	−0.05757 (13)	0.0347 (5)
C5	−0.0243 (2)	0.6747 (2)	−0.04417 (13)	0.0355 (5)
C6	−0.0520 (2)	0.5758 (2)	−0.09344 (14)	0.0379 (5)
H6	−0.1144	0.5208	−0.0823	0.046*
N1	−0.0167 (2)	0.4585 (2)	−0.21397 (14)	0.0485 (5)
C14	−0.2601 (2)	0.7581 (2)	0.11059 (14)	0.0367 (5)
C2	0.1108 (2)	0.6383 (2)	−0.17603 (14)	0.0400 (6)
H2	0.1544	0.6276	−0.2212	0.048*
C1	0.0152 (2)	0.5605 (2)	−0.15972 (14)	0.0383 (5)
C13	0.3827 (3)	0.6383 (2)	0.02110 (16)	0.0499 (7)
H13	0.3757	0.5687	−0.0108	0.060*
O1	0.0290 (2)	0.4572 (2)	−0.27821 (13)	0.0733 (7)
C9	0.3998 (3)	0.8590 (3)	0.03325 (17)	0.0517 (7)
H9	0.4035	0.9358	0.0096	0.062*
C15	−0.2564 (3)	0.8751 (2)	0.14369 (16)	0.0486 (6)
H15	−0.2377	0.9433	0.1136	0.058*
C19	−0.2863 (3)	0.6544 (3)	0.15400 (16)	0.0506 (7)
H19	−0.2893	0.5772	0.1305	0.061*
C10	0.4065 (3)	0.8469 (3)	0.11472 (19)	0.0673 (9)
H10	0.4151	0.9161	0.1469	0.081*
C7	0.0787 (3)	0.8778 (3)	0.06494 (15)	0.0518 (7)
H7A	−0.0060	0.9044	0.0661	0.078*
H7B	0.1329	0.9399	0.0882	0.078*
H7C	0.0900	0.8029	0.0945	0.078*
C11	0.4003 (3)	0.7318 (4)	0.14827 (19)	0.0696 (10)
H11	0.4042	0.7244	0.2033	0.084*
C17	−0.3050 (3)	0.7856 (4)	0.26666 (18)	0.0701 (10)
H17	−0.3195	0.7948	0.3200	0.084*

C16	−0.2811 (3)	0.8871 (3)	0.22228 (18)	0.0628 (8)
H16	−0.2816	0.9645	0.2454	0.075*
C18	−0.3080 (3)	0.6690 (3)	0.23317 (17)	0.0653 (9)
H18	−0.3246	0.6009	0.2640	0.078*
C12	0.3885 (3)	0.6280 (3)	0.10238 (18)	0.0689 (9)
H12	0.3844	0.5513	0.1261	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S2	0.0391 (3)	0.0318 (3)	0.0376 (3)	−0.0018 (2)	0.0057 (2)	0.0007 (2)
S1	0.0459 (4)	0.0375 (4)	0.0401 (4)	−0.0039 (3)	0.0134 (3)	0.0019 (3)
O3	0.0488 (10)	0.0323 (9)	0.0428 (9)	−0.0041 (8)	0.0098 (8)	0.0080 (7)
O7	0.0427 (9)	0.0417 (10)	0.0376 (9)	0.0011 (8)	0.0080 (7)	0.0017 (8)
O6	0.0589 (11)	0.0347 (9)	0.0461 (10)	−0.0088 (8)	0.0137 (8)	−0.0080 (8)
O9	0.0499 (11)	0.0551 (12)	0.0494 (10)	−0.0125 (9)	0.0030 (8)	−0.0120 (9)
O4	0.0617 (12)	0.0487 (11)	0.0481 (10)	0.0066 (9)	0.0134 (9)	−0.0091 (9)
O8	0.0721 (13)	0.0399 (10)	0.0565 (11)	0.0052 (9)	0.0100 (10)	0.0157 (9)
O2	0.0823 (15)	0.0460 (12)	0.0691 (14)	−0.0170 (11)	0.0038 (11)	−0.0126 (10)
O5	0.0583 (12)	0.0552 (12)	0.0678 (12)	−0.0132 (10)	0.0235 (10)	0.0135 (10)
C8	0.0370 (12)	0.0383 (13)	0.0381 (13)	−0.0048 (10)	0.0049 (10)	−0.0025 (10)
C3	0.0409 (13)	0.0303 (12)	0.0351 (12)	−0.0017 (10)	0.0041 (10)	0.0057 (10)
C4	0.0413 (13)	0.0277 (12)	0.0353 (12)	0.0032 (10)	0.0032 (10)	0.0016 (9)
C5	0.0390 (13)	0.0327 (12)	0.0352 (12)	0.0018 (10)	0.0047 (10)	0.0028 (10)
C6	0.0424 (13)	0.0290 (12)	0.0421 (13)	−0.0011 (10)	0.0007 (10)	0.0053 (10)
N1	0.0511 (13)	0.0431 (13)	0.0506 (13)	−0.0002 (11)	−0.0025 (10)	−0.0088 (11)
C14	0.0365 (12)	0.0347 (13)	0.0395 (13)	−0.0006 (10)	0.0063 (10)	−0.0025 (10)
C2	0.0463 (14)	0.0392 (14)	0.0351 (12)	0.0051 (11)	0.0060 (10)	−0.0007 (10)
C1	0.0444 (13)	0.0315 (12)	0.0383 (13)	0.0013 (10)	−0.0024 (10)	−0.0031 (10)
C13	0.0646 (18)	0.0391 (14)	0.0456 (15)	−0.0090 (13)	0.0021 (13)	0.0040 (12)
O1	0.0871 (16)	0.0800 (16)	0.0545 (12)	−0.0138 (13)	0.0181 (11)	−0.0297 (12)
C9	0.0532 (16)	0.0452 (16)	0.0569 (16)	−0.0068 (13)	0.0041 (13)	−0.0136 (13)
C15	0.0517 (16)	0.0411 (15)	0.0532 (15)	−0.0022 (12)	0.0057 (12)	−0.0095 (12)
C19	0.0578 (17)	0.0437 (15)	0.0515 (16)	−0.0002 (13)	0.0131 (13)	0.0071 (12)
C10	0.0559 (18)	0.083 (2)	0.0621 (19)	−0.0016 (17)	−0.0033 (15)	−0.0365 (18)
C7	0.0597 (17)	0.0515 (16)	0.0451 (14)	−0.0122 (14)	0.0096 (12)	−0.0128 (13)
C11	0.0580 (19)	0.109 (3)	0.0413 (16)	−0.0024 (19)	0.0005 (14)	−0.0044 (18)
C17	0.0575 (19)	0.115 (3)	0.0375 (15)	0.0043 (19)	0.0043 (13)	−0.0124 (18)
C16	0.0611 (19)	0.069 (2)	0.0582 (18)	−0.0009 (16)	0.0040 (15)	−0.0286 (16)
C18	0.069 (2)	0.078 (2)	0.0500 (17)	0.0014 (18)	0.0142 (15)	0.0166 (16)
C12	0.083 (2)	0.075 (2)	0.0475 (17)	−0.0125 (19)	0.0011 (16)	0.0149 (16)

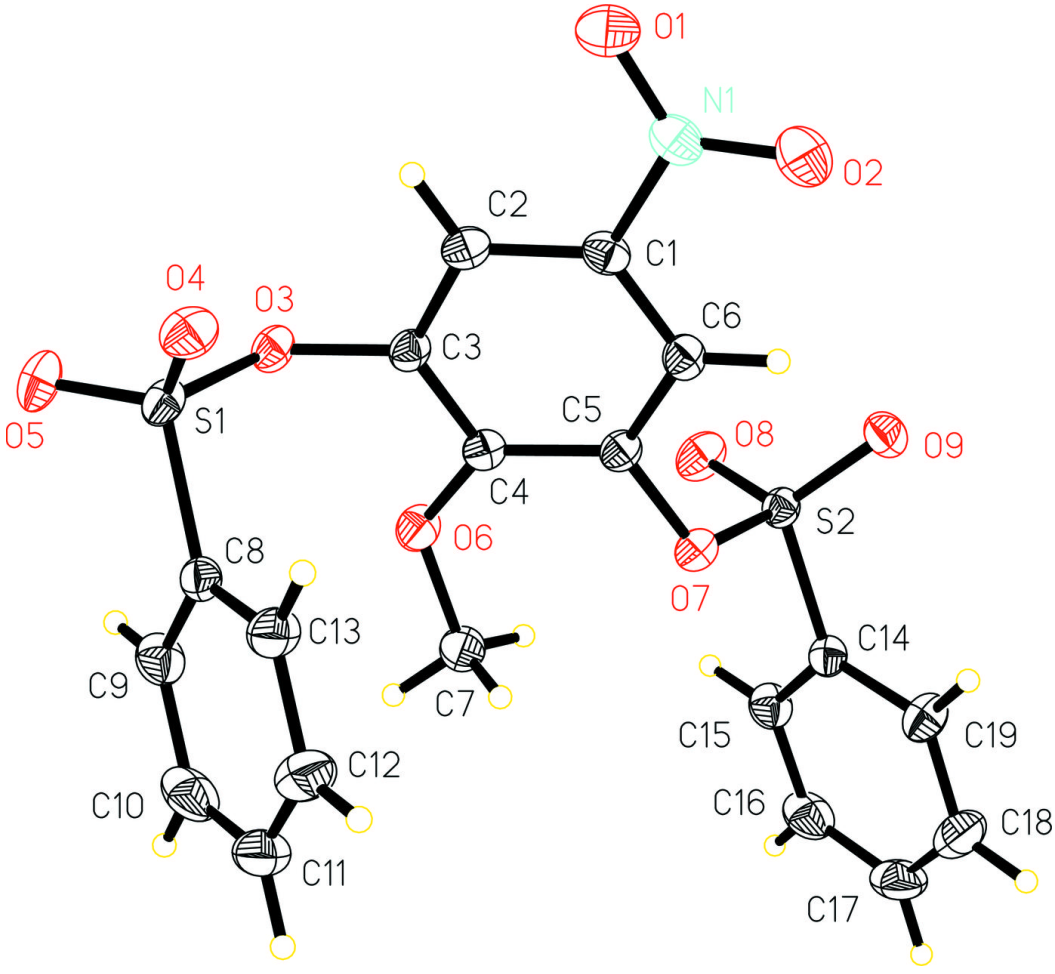
Geometric parameters (Å, °)

S2—O9	1.4175 (18)	C14—C15	1.391 (3)
S2—O8	1.4192 (18)	C2—C1	1.377 (3)
S2—O7	1.6197 (18)	C2—H2	0.9300
S2—C14	1.745 (2)	C13—C12	1.374 (4)
S1—O5	1.4164 (19)	C13—H13	0.9300

S1—O4	1.4278 (19)	C9—C10	1.378 (4)
S1—O3	1.6150 (19)	C9—H9	0.9300
S1—C8	1.753 (2)	C15—C16	1.377 (4)
O3—C3	1.405 (3)	C15—H15	0.9300
O7—C5	1.411 (3)	C19—C18	1.381 (4)
O6—C4	1.345 (3)	C19—H19	0.9300
O6—C7	1.433 (3)	C10—C11	1.380 (5)
O2—N1	1.232 (3)	C10—H10	0.9300
C8—C13	1.379 (3)	C7—H7A	0.9600
C8—C9	1.384 (3)	C7—H7B	0.9600
C3—C2	1.375 (3)	C7—H7C	0.9600
C3—C4	1.397 (3)	C11—C12	1.371 (5)
C4—C5	1.406 (3)	C11—H11	0.9300
C5—C6	1.381 (3)	C17—C16	1.370 (5)
C6—C1	1.384 (3)	C17—C18	1.390 (5)
C6—H6	0.9300	C17—H17	0.9300
N1—O1	1.221 (3)	C16—H16	0.9300
N1—C1	1.466 (3)	C18—H18	0.9300
C14—C19	1.387 (3)	C12—H12	0.9300
O9—S2—O8	118.93 (12)	C2—C1—C6	121.9 (2)
O9—S2—O7	107.45 (11)	C2—C1—N1	119.3 (2)
O8—S2—O7	108.49 (11)	C6—C1—N1	118.9 (2)
O9—S2—C14	111.12 (11)	C12—C13—C8	118.8 (3)
O8—S2—C14	111.47 (12)	C12—C13—H13	120.6
O7—S2—C14	96.97 (10)	C8—C13—H13	120.6
O5—S1—O4	120.56 (12)	C10—C9—C8	118.2 (3)
O5—S1—O3	102.05 (11)	C10—C9—H9	120.9
O4—S1—O3	108.19 (11)	C8—C9—H9	120.9
O5—S1—C8	110.87 (12)	C16—C15—C14	118.1 (3)
O4—S1—C8	109.59 (11)	C16—C15—H15	120.9
O3—S1—C8	104.02 (10)	C14—C15—H15	120.9
C3—O3—S1	117.91 (14)	C18—C19—C14	118.1 (3)
C5—O7—S2	117.04 (14)	C18—C19—H19	120.9
C4—O6—C7	123.80 (19)	C14—C19—H19	120.9
C13—C8—C9	122.2 (2)	C9—C10—C11	119.7 (3)
C13—C8—S1	118.77 (19)	C9—C10—H10	120.2
C9—C8—S1	119.0 (2)	C11—C10—H10	120.2
C2—C3—C4	123.0 (2)	O6—C7—H7A	109.5
C2—C3—O3	119.8 (2)	O6—C7—H7B	109.5
C4—C3—O3	117.1 (2)	H7A—C7—H7B	109.5
O6—C4—C3	115.7 (2)	O6—C7—H7C	109.5
O6—C4—C5	128.0 (2)	H7A—C7—H7C	109.5
C3—C4—C5	116.2 (2)	H7B—C7—H7C	109.5
C6—C5—C4	122.0 (2)	C12—C11—C10	121.5 (3)
C6—C5—O7	118.4 (2)	C12—C11—H11	119.2
C4—C5—O7	119.4 (2)	C10—C11—H11	119.2
C5—C6—C1	118.5 (2)	C16—C17—C18	121.0 (3)
C5—C6—H6	120.7	C16—C17—H17	119.5
C1—C6—H6	120.7	C18—C17—H17	119.5

O1—N1—O2	124.2 (2)	C17—C16—C15	120.4 (3)
O1—N1—C1	118.1 (2)	C17—C16—H16	119.8
O2—N1—C1	117.7 (2)	C15—C16—H16	119.8
C19—C14—C15	122.4 (2)	C19—C18—C17	119.8 (3)
C19—C14—S2	118.91 (19)	C19—C18—H18	120.1
C15—C14—S2	118.67 (19)	C17—C18—H18	120.1
C3—C2—C1	118.2 (2)	C11—C12—C13	119.5 (3)
C3—C2—H2	120.9	C11—C12—H12	120.2
C1—C2—H2	120.9	C13—C12—H12	120.2

Fig. 1


O1
N1
O2
C2
C1
O4
O3
C6
C3
O5
S1
C5
O8
O9
C4
S2
O6
C8
O7
C13
C14
C9
C7
C15
C19
C12
C16
C18
C10
C11
C17