

Acta Crystallographica Section C Crystal Structure Communications

ISSN 0108-2701

A new polymorph of *N*-(6-methylpyridin-2-yl)mesitylenesulfonamide

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Received 10 May 2013 Accepted 19 September 2013

A new polymorph is reported of the pharmaceutically active sulfapyridine derivative, N-(6-methylpyridin-2-yl)mesitylenesulfonamide, in the zwitterionic form 2-methyl-6-{[(2,4,6-trimethylbenzene)sulfonyl]azanidyl]pyridin-1-ium, C₁₅H₁₈N₂O₂S. The observed dimorphism is solvent dependent. The phase described previously [Beloso, Castro, García-Vázquez, Pérez-Lourido, Romero & Sousa (2003). Z. Anorg. Allg. Chem. 629, 275-284] crystallizes from ethanol and several other organic solvents, whereas the new form described here is obtained as a phase-pure product from methanol. The molecules in both dimorphic phases are very similar and adopt the conformation which is also predicted for an individual molecule by force field calculations. However, the two forms differ in their packing and hydrogen bonding. Results from solvent-assisted grinding indicate that the new form is less stable than the previously published phase.

Keywords: crystal structure; pharmaceutically active compounds; sulfapyridine derivatives; zwitterions; polymorphism; packing polymorphs.

1. Introduction

Polymorphism is a common phenomenon in many classes of compounds (McCrone, 1965), among them many nitrogencontaining drugs. As far as the pharmaceutically active sulfonamides are concerned, polymorphism is well documented for 4-amino-N-pyridinylbenzenesulfonamide (Bernstein, 1984; Burger et al., 1980; Gouda et al., 1977; Kuhnert-Brandstätter & Wunsch, 1969; Mesley & Houghton, 1967; Pratt et al., 2011; Yang & Guillory, 1972). The title compound, N-(6-methylpyridin-2-yl)mesitylenesulfonamide, (1), is a sulfapyridine derivative; Beloso et al. (2003) described a form to which we will refer as phase I. The original authors crystallized phase I from ethanol, but our experiments revealed that the same phase is found from a variety of other organic solvents, such as dichloromethane, acetone, tetrahydrofuran and propan-2-ol. We report here a new polymorph of N-(6-methylpyridin-2-yl)mesitylenesulfonamide, denoted polymorph II,

which can be reproducibly obtained from methanol as a phasepure solid (Fig. 1).



2. Experimental

2.1. Synthesis and crystallization

The title compound was prepared according to the method of Beloso *et al.* (2003). 2-Amino-6-methylpyridine (2.000 g, 18.49 μ mol) and 2,4,6-methylphenylsulfonyl chloride (4.045 g, 18.49 μ mol) were dissolved in dichloromethane (20 ml). Sodium carbonate (2.003 g, 18.49 μ mol) in water (20 ml) was added dropwise. The reaction mixture was stirred overnight and water (100 ml) was added. The organic layer was separated and dried with anhydrous magnesium sulfate. The solvent was evaporated and the resulting oil was suspended in ethanol, and polymorph I was obtained as a white solid. Colourless block-shaped crystals of polymorph I were grown by slow evaporation of a solution of polymorph I in methanol at room temperature.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were treated as riding, with C-H = 0.95 Å for aryl or 0.98 Å for methyl groups, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups. The N-bound H atom was



Figure 1

Experimental powder pattern of the new polymorph, II (top profile, black), and calculated pattern based on the single-crystal diffraction result (bottom profile, red in the electronic version of the journal).

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Table 1

Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{18}N_2O_2S$
$M_{ m r}$	290.37
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	7.9753 (11), 8.1446 (11), 11.7776 (16)
α, β, γ (°)	86.829 (3), 81.600 (3), 67.124 (3)
$V(\dot{A}^3)$	697.29 (16)
Ζ	2
Radiation type	Μο Κα
$\mu \text{ (mm}^{-1})$	0.24
Crystal size (mm)	$0.19 \times 0.10 \times 0.09$
Data collection	
Diffractometer	Bruker D8 diffractometer with an APEX CCD detector
Absorption correction	Multi-scan (SADABS; Bruker, 1998)
T_{\min}, \bar{T}_{\max}	0.957, 0.979
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	9103, 3195, 2527
R _{int}	0.043
$(\sin \theta / \lambda)_{max} (\text{\AA}^{-1})$	0.657
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.046, 0.115, 1.03
No. of reflections	3195
No. of parameters	189
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta \rho_{\rm max}$, $\Delta \rho_{\rm min}$ (e Å ⁻³)	0.45, -0.39

Computer programs: SMART (Bruker, 2002), SAINT (Bruker, 2009), SHELXS97 (Sheldrick, 2008), SHELXL2012 (Sheldrick, 2008) and PLATON (Spek, 2009).

located in a difference Fourier map, and both the coordinates and the atomic displacement parameter were freely refined. The final N1-H1N bond length is 0.86 (2) Å, with $U_{\rm iso}({\rm H1N}) = 0.020$ (6) Å².

3. Results and discussion

Fig. 2 shows a displacement ellipsoid plot of the molecule of II, and selected interatomic distances and angles have been compiled in Table 2. In order to discuss the structural relationship between the two polymorphs, it is necessary to describe briefly the previously reported phase I (Beloso *et al.*, 2003). Its asymmetric unit contains two molecules of very



Figure 2

The molecular structure of polymorph II, with displacement ellipsoids drawn at the 50% probability level.

Table 2 Selected geometric parameters (Å, °).

S1-O1	1.4504 (15)	N1-C2	1.365 (3)
S1-O2	1.4434 (14)	N1-C1	1.370 (3)
S1-N2	1.5940 (17)	N2-C1	1.336 (3)
S1-C7	1.793 (2)		
O1-S1-O2	115.15 (9)	C1-N2-S1	122.42 (14)
O1-S1-N2	105.04 (9)	N2-C1-N1	113.68 (17)
O2-S1-N2	113.34 (9)	N2-C1-C5	130.48 (19)
O1-S1-C7	106.20 (9)	N1-C1-C5	115.83 (18)
O2-S1-C7	109.16 (9)	C3-C2-N1	118.79 (19)
N2-S1-C7	107.44 (9)	N1-C2-C6	116.93 (18)
C2-N1-C1	124.98 (18)		

similar conformation as a head-to-tail dimer linked by pico-line–imide $N-H\cdots N$ hydrogen bonds, with graph-set nota-



Figure 3

The hydrogen-bonded (dashed lines) dimer in (a) phase I and (b) phase II. H atoms bonded to C atoms have been omitted for clarity. (c) An overlay of the molecules of phases I and II underlines their conformational similarity. The molecule associated with atom S1 in phase I is shown with dashed bonds and the molecule of II with solid bonds.

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Table 3

Hydrogen-bond geometry (Å, °).

$D - H \cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - H \cdots$
$N1 - H1N \cdots O1^i$	0.86 (2)	2.05 (2)	2.879 (2)	161.9
Symmetry code: (i) -	-x + 2, -v + 1, -v	-z + 1.		



Figure 4

Minimum energy conformation of an isolated zwitterionic molecule of N-(6-methylpyridin-2-yl)mesitylenesulfonamide according to force field calculations.

tion $R_2^2(8)$ (Etter *et al.*, 1990) (Fig. 3*a*). The two aminopicoline rings involved in this hydrogen-bonded pair subtend an angle of $46.86(8)^{\circ}$, presumably a compromise of steric repulsion between the trimethylphenyl groups and attraction due to weak intermolecular $C-H \cdots O$ contacts. In the new phase, II, the asymmetric unit consists of a single molecule, and a different arrangement between hydrogen-bonded neighbours is found: an O atom of the sulfone group rather than the amide N atom acts as hydrogen-bond acceptor (Table 3), generating a 12-membered ring with graph-set notation $R_2^2(12)$ (Fig. 3b); for reasons of symmetry, the aminopicoline rings are coplanar.

Chemical intuition suggests, and structural parameters of reported sulfapyridine derivatives (Koo & Lee, 1979; Basak et al., 1984; Bernstein, 1988; Bar & Bernstein, 1985; Gelbrich et al., 2007; Pratt et al., 2011; Coles et al., 1999, 2000; Threlfall et al., 1998; Ghorab et al., 2012) confirm, that the tautomer with a



Table 4

Comparison of the experimentally observed molecular conformation in form I (Beloso et al., 2003; average over two independent molecules), in form II (this work) and for the minimum-energy conformation (force field calculations).

Torsion angle (°)	X-ray, polymorph I	X-ray, polymorph II	Force field minimum
N1-C1-N2-S1	-177.3	-176.39 (13)	-177.5
C1-N2-S1-C7	62.1	63.45 (17)	61.1
N2-S1-C7-C8	54.9	58.08 (17)	41.8

deprotonated imide N atom features strong delocalization of the electrons. Consequently, for both polymorphs, the imidepicoline N–C bonds are short [1.336 (3) Å in polymorph II,and 1.347 (3) and 1.342 (2) Å in the two residues of polymorph I] and the $N_{picoline} - C_{picoline} - N_{imide} - S$ torsion angles are small $[-176.39 (13)^{\circ}$ in polymorph II, and -175.96 (14)and $-178.70 (13)^{\circ}$ in the two residues of polymorph I].

Despite the clear differences in the intermolecular hydrogen bonds, the conformations of the individual molecules of polymorphs I and II are very similar, as shown in Fig. 3(c) and Table 4. Table 4 also describes the minimum energy conformation of a molecule in the gas phase according to simple force field calculations; an augmented MM2 force field was used (Fujitsu, 2003). The resulting minimum energy conformation is shown in Fig. 4. Fig. 5 underlines that the arrangement of the hydrogen-bonded pairs is different in both forms: I and II are genuine packing polymorphs. Form II is associated with an overall packing coefficient (Spek, 2009) of 70.0%, lower than that for polymorph I (71.6%). This slightly inferior space filling of the new polymorph, its lower melting point (433.8-435.1 versus 437.9-439.4 K for polymorph I) and its lower density indicate that the new phase might be thermodynamically less stable than polymorph I. More conclusive evidence stems from solvent-assisted grinding: we recall that the title compound, (1), is soluble in many common organic solvents but scarcely soluble in water. The new phase, II, is partially transformed into phase I when ground for 3 min with



Figure 5

Comparison of the packing in (left) polymorph I and (right) polymorph II as projections on the bc plane.



Figure 6

Experimental powder patterns of polymorph I (bottom profile, blue in the electronic version of the journal), polymorph II (middle profile, magenta) and polymorph II after solvent-assisted grinding (top profile, black); the arrows drawn downwards from the last (marked in green) indicate partial conversion of form II into form I.

a drop of water; the corresponding powder results are compiled in Fig. 6. No conversion between the polymorphs is observed upon grinding without any solvent.

4. Conclusion

The individual molecules in both forms do not differ significantly but rather adopt a conformation similar to that predicted for an isolated zwitterionic molecule. Global packing arguments, such as space filling or density, favour form I. If we follow the general expectation (Bernstein, 1987) and assume that the alternative polymorphs of the title compound are associated with comparable lattice energies, we may conclude that a contribution stabilizing the new form II is required. We may speculate that more efficient hydrogen bonds can be responsible for its existence. Financial support from the China Scholarship Council for FP is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FM3002). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2013). C69, 1217-1220 [doi:10.1107/S0108270113025936]

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Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART* (Bruker, 2001); data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2012* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2012* (Sheldrick, 2008).

2-Methyl-6-{[(2,4,6-trimethylbenzene)sulfonyl]azanidyl}pyridin-1-ium

Crystal data	
$C_{15}H_{18}N_{2}O_{2}S$ $M_{r} = 290.37$ Triclinic, $P\overline{1}$ $a = 7.9753 (11) \text{ Å}$ $b = 8.1446 (11) \text{ Å}$ $c = 11.7776 (16) \text{ Å}$ $a = 86.829 (3)^{\circ}$ $\beta = 81.600 (3)^{\circ}$ $\gamma = 67.124 (3)^{\circ}$ $V = 697.29 (16) \text{ Å}^{3}$	Z = 2 F(000) = 308 $D_x = 1.383 \text{ Mg m}^{-3}$ Mo K\alpha radiation, \lambda = 0.71073 \mathbf{A} Cell parameters from 1269 reflections $\theta = 2.7-25.4^{\circ}$ $\mu = 0.24 \text{ mm}^{-1}$ T = 100 K Block, colourless $0.19 \times 0.10 \times 0.09 \text{ mm}$
Data collection	
Bruker D8 diffractometer with APEX CCD detector Radiation source: Incoatec microsource Multilayer optics monochromator ω scans Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2003) $T_{\min} = 0.957, T_{\max} = 0.979$	9103 measured reflections 3195 independent reflections 2527 reflections with $I > 2\sigma(I)$ $R_{int} = 0.043$ $\theta_{max} = 27.8^{\circ}, \ \theta_{min} = 2.7^{\circ}$ $h = -10 \rightarrow 10$ $k = -10 \rightarrow 10$ $l = -15 \rightarrow 14$
Refinement Refinement on F^2 Least-squares matrix: full $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.115$ S = 1.03 3195 reflections 189 parameters 0 restraints Primary atom site location: structure-invariant direct methods	Secondary atom site location: difference Fourier map Hydrogen site location: inferred from neighbouring sites H atoms treated by a mixture of independent and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.4133P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{max} < 0.001$ $\Delta\rho_{max} = 0.45$ e Å ⁻³ $\Delta \alpha_{max} = -0.20$ a Å ⁻³

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 $F^2 > \sigma(F^2)$ 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.

 $U_{\rm iso}*/U_{\rm eq}$ Ζ х v 0.01535 (14) **S**1 0.82705 (7) 0.55137 (6) 0.30180 (4) 01 0.92771 (19) 0.36086 (18) 0.31141 (12) 0.0192(3)O2 0.63729(18) 0.60458 (19) 0.28521 (12) 0.0205(3)N1 0.8318(2)0.8557(2) 0.53085 (14) 0.0159 (4) H1N 0.902 (3) 0.570(2)0.020 (6)* 0.772 (3) N2 0.8525(2)0.6346(2)0.41429 (14) 0.0168 (4) C1 0.7867(3)0.8102 (3) 0.43271 (16) 0.0159 (4) C2 0.7785(3)1.0257(3)0.56965 (17) 0.0173(4)C3 0.6797(3)0.0192(4)1.1637 (3) 0.50479 (17) H3 0.6436 1.2834 0.5287 0.023* C4 0.40230 (18) 0.0200(4)0.6325 (3) 1.1258 (3) H4 0.024* 0.5645 1.2213 0.3564 C5 0.6820(3)0.9540(3) 0.36666 (17) 0.0186 (4) 0.022* H5 0.6462 0.9314 0.2976 C6 0.8320(3)1.0474(3)0.68275 (18) 0.0235(5)0.035* H6A 0.7837 1.1742 0.7024 0.9980 0.6778 0.035* H6B 0.9662 H6C 0.7811 0.9840 0.7422 0.035* C7 0.9465(3)0.6237(3)0.18139 (16) 0.0147(4)C8 1.1358 (3) 0.5800(3)0.18110 (17) 0.0164(4)C9 1.2314(3)0.6356(3)0.08962 (17) 0.0171(4)H9 0.6089 0.0904 0.021* 1.3583 C10 1.1491 (3) 0.7289(3) -0.00333(17)0.0179 (4) C11 -0.00193(17)0.9640(3)0.7663(3)0.0188(4)H11 0.9062 0.8285 -0.06520.023* C12 0.8585 (3) 0.7172 (3) 0.08756 (17) 0.0170 (4) C13 1.2450 (3) 0.4724(3)0.27382 (18) 0.0198 (4) 0.030* H13A 1.3601 0.4910 0.2691 H13B 1.2725 0.3456 0.2630 0.030* H13C 0.5110 0.3493 0.030* 1.1731 0.0236 (5) C14 1.2579 (3) 0.7826(3)-0.10316(18)H14A 1.1801 0.8970 -0.13360.035* 0.035* H14B 1.3002 0.6915 -0.1633H14C 0.7943 -0.07750.035* 1.3643 C15 0.6587(3)0.7662(3)0.07493 (18) 0.0213(5)0.8291 0.032* H15A 0.6326 0.0022 0.032* H15B 0.5807 0.8435 0.1388

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

supplementary materials

H15C	0.6334	0.6	5576	0.0756	0.032*	
Atomic	displacement par	ameters (Ų)				
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U ²³
S1	0.0155 (3)	0.0159 (3)	0.0150 (2)	-0.0066 (2)	-0.00188 (18)	0.00050 (18)
01	0.0232 (8)	0.0151 (7)	0.0206 (8)	-0.0089 (6)	-0.0042 (6)	0.0029 (6)
02	0.0148 (7)	0.0257 (8)	0.0215 (8)	-0.0088 (6)	-0.0014 (6)	-0.0003 (6)
N1	0.0155 (8)	0.0153 (9)	0.0164 (9)	-0.0057 (7)	-0.0022 (7)	0.0026 (7)
N2	0.0199 (9)	0.0156 (9)	0.0142 (8)	-0.0060 (7)	-0.0031 (7)	0.0005 (7)
C1	0.0132 (9)	0.0208 (10)	0.0136 (9)	-0.0077 (8)	0.0013 (7)	0.0004 (8)
C2	0.0142 (9)	0.0183 (10)	0.0182 (10)	-0.0059 (8)	0.0014 (8)	-0.0020 (8)
C3	0.0187 (10)	0.0166 (10)	0.0201 (10)	-0.0053 (8)	0.0009 (8)	-0.0011 (8)
C4	0.0170 (10)	0.0180 (11)	0.0207 (11)	-0.0032 (8)	-0.0012 (8)	0.0049 (8)
C5	0.0164 (10)	0.0217 (11)	0.0155 (10)	-0.0056 (8)	-0.0016 (8)	0.0013 (8)
C6	0.0269 (12)	0.0210 (11)	0.0228 (11)	-0.0084 (9)	-0.0054 (9)	-0.0016 (9)
C7	0.0171 (10)	0.0134 (10)	0.0139 (9)	-0.0065 (8)	-0.0010 (8)	-0.0008 (7)
C8	0.0183 (10)	0.0128 (10)	0.0179 (10)	-0.0054 (8)	-0.0022 (8)	-0.0026 (8)
C9	0.0137 (9)	0.0188 (10)	0.0195 (10)	-0.0069 (8)	-0.0008(8)	-0.0028 (8)
C10	0.0231 (11)	0.0156 (10)	0.0157 (10)	-0.0087 (8)	-0.0004 (8)	-0.0021 (8)
C11	0.0227 (11)	0.0171 (10)	0.0153 (10)	-0.0056 (9)	-0.0051 (8)	0.0015 (8)
C12	0.0177 (10)	0.0154 (10)	0.0173 (10)	-0.0052 (8)	-0.0036 (8)	-0.0018 (8)
C13	0.0168 (10)	0.0198 (11)	0.0220 (11)	-0.0060 (9)	-0.0047 (8)	0.0037 (8)
C14	0.0267 (11)	0.0254 (12)	0.0207 (11)	-0.0139 (10)	0.0013 (9)	0.0004 (9)
C15	0.0206 (11)	0.0251 (12)	0.0178 (10)	-0.0074(9)	-0.0064(8)	0.0022 (9)

Geometric parameters (Å, °)

S1—01	1.4504 (15)	C7—C12	1.418 (3)
S1—O2	1.4434 (14)	C8—C9	1.386 (3)
S1—N2	1.5940 (17)	C8—C13	1.516 (3)
S1—C7	1.793 (2)	C9—C10	1.392 (3)
N1—C2	1.365 (3)	С9—Н9	0.9500
N1-C1	1.370 (3)	C10—C11	1.384 (3)
N1—H1N	0.86 (2)	C10—C14	1.506 (3)
N2—C1	1.336 (3)	C11—C12	1.391 (3)
C1—C5	1.415 (3)	C11—H11	0.9500
С2—С3	1.364 (3)	C12—C15	1.513 (3)
С2—С6	1.496 (3)	C13—H13A	0.9800
C3—C4	1.400 (3)	C13—H13B	0.9800
С3—Н3	0.9500	C13—H13C	0.9800
C4—C5	1.370 (3)	C14—H14A	0.9800
C4—H4	0.9500	C14—H14B	0.9800
С5—Н5	0.9500	C14—H14C	0.9800
С6—Н6А	0.9800	C15—H15A	0.9800
C6—H6B	0.9800	C15—H15B	0.9800
С6—Н6С	0.9800	C15—H15C	0.9800
С7—С8	1.410 (3)		
O1—S1—O2	115.15 (9)	C9—C8—C7	118.65 (18)

O1—S1—N2	105.04 (9)	C9—C8—C13	116.94 (17)
O2—S1—N2	113.34 (9)	C7—C8—C13	124.40 (18)
O1—S1—C7	106.20 (9)	C8—C9—C10	122.68 (18)
O2—S1—C7	109.16 (9)	С8—С9—Н9	118.7
N2—S1—C7	107.44 (9)	С10—С9—Н9	118.7
C2—N1—C1	124.98 (18)	C11—C10—C9	117.36 (18)
C2—N1—H1N	117.0 (15)	C11—C10—C14	121.18 (18)
C1—N1—H1N	118.0 (15)	C9—C10—C14	121.43 (18)
C1—N2—S1	122.42 (14)	C10-C11-C12	123.24 (19)
N2—C1—N1	113.68 (17)	C10-C11-H11	118.4
N2—C1—C5	130.48 (19)	C12—C11—H11	118.4
N1—C1—C5	115.83 (18)	C11—C12—C7	117.89 (18)
C3—C2—N1	118.79 (19)	C11—C12—C15	116.56 (18)
C3—C2—C6	124.28 (19)	C7—C12—C15	125.54 (18)
N1—C2—C6	116.93 (18)	C8—C13—H13A	109.5
C2—C3—C4	118.86 (19)	C8—C13—H13B	109.5
С2—С3—Н3	120.6	H13A—C13—H13B	109.5
С4—С3—Н3	120.6	C8—C13—H13C	109.5
C5—C4—C3	121.48 (19)	H13A—C13—H13C	109.5
C5—C4—H4	119.3	H13B—C13—H13C	109.5
C3—C4—H4	119.3	C10—C14—H14A	109.5
C4—C5—C1	119.99 (19)	C10-C14-H14B	109.5
C4—C5—H5	120.0	H14A—C14—H14B	109.5
C1—C5—H5	120.0	C10—C14—H14C	109.5
С2—С6—Н6А	109.5	H14A—C14—H14C	109.5
С2—С6—Н6В	109.5	H14B—C14—H14C	109.5
H6A—C6—H6B	109.5	C12—C15—H15A	109.5
С2—С6—Н6С	109.5	C12—C15—H15B	109.5
H6A—C6—H6C	109.5	H15A—C15—H15B	109.5
H6B—C6—H6C	109.5	C12—C15—H15C	109.5
C8—C7—C12	120.14 (18)	H15A—C15—H15C	109.5
C8—C7—S1	117.37 (14)	H15B—C15—H15C	109.5
C12—C7—S1	122.46 (15)		
O1—S1—N2—C1	176.23 (15)	O1—S1—C7—C12	124.46 (16)
O2—S1—N2—C1	-57.23 (18)	N2—S1—C7—C12	-123.54 (17)
C7—S1—N2—C1	63.45 (17)	C12—C7—C8—C9	2.2 (3)
S1—N2—C1—N1	-176.39 (13)	S1—C7—C8—C9	-179.41 (14)
S1—N2—C1—C5	2.4 (3)	C12—C7—C8—C13	-176.71 (18)
C2—N1—C1—N2	-178.80 (17)	S1—C7—C8—C13	1.7 (3)
C2—N1—C1—C5	2.2 (3)	C7—C8—C9—C10	-1.7 (3)
C1—N1—C2—C3	-3.1 (3)	C13—C8—C9—C10	177.32 (18)
C1—N1—C2—C6	176.22 (18)	C8—C9—C10—C11	0.2 (3)
N1—C2—C3—C4	1.6 (3)	C8—C9—C10—C14	-178.12 (18)
C6—C2—C3—C4	-177.67 (19)	C9—C10—C11—C12	0.8 (3)
C2—C3—C4—C5	0.5 (3)	C14—C10—C11—C12	179.10 (19)
C3—C4—C5—C1	-1.3 (3)	C10—C11—C12—C7	-0.3 (3)
N2-C1-C5-C4	-178.71 (19)	C10—C11—C12—C15	-179.20 (19)
N1—C1—C5—C4	0.0 (3)	C8—C7—C12—C11	-1.3 (3)

O1—S1—C7—C8	-53.92 (17)	S1—C7—C12—C11	-179.59 (14)	
O2—S1—C7—C8	-178.64 (14)	C8—C7—C12—C15	177.58 (18)	
N2—S1—C7—C8	58.08 (17)	S1—C7—C12—C15	-0.8 (3)	
O2—S1—C7—C12	-0.26 (19)			

Hydrogen-bond geometry (Å, °)

	D—H	Н…А	D···A	<i>D</i> —H··· <i>A</i>
N1—H1N····O1 ⁱ	0.86 (2)	2.05 (2)	2.879 (2)	161.9

Symmetry code: (i) -x+2, -y+1, -z+1.

Comparison of the experimentally observed molecular conformation in form (I) (Beloso et al., 2003; average over two independent molecules), in form (II) (this work) and for the minimum-energy conformation (force field calculations).

Torsion angle (°)	X-ray (I)	X-ray (II)	Force field minimum
N1—C1—N2—S1	-177.3	-176.39 (13)	-177.5
C1—N2—S1—C7	62.1	63.45 (17)	61.1
N2—S1—C7—C8	54.9	58.08 (17)	41.8