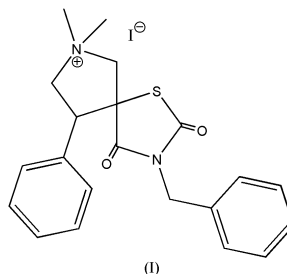


3-Benzyl-7,7-dimethyl-9-phenyl-1-thia-3-aza-7-azoniaspiro[4.4]nonane-2,4-dione iodide**Xiao-Fang Li, Ya-Qing Feng,*
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lxf7212@yahoo.com.cn**Key indicators**Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
 R factor = 0.039
 wR factor = 0.104
Data-to-parameter ratio = 17.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{21}\text{H}_{23}\text{IN}_2\text{O}_2\text{S}$, was synthesized by the intermolecular [3 + 2]-cycloaddition of azomethine ylide and 5-benzylidene-3-benzylthiazolidine-2,4-dione, followed by alkylation with methyl iodide. The spiro ring involves thiazolidine and pyrrolidine rings, the former planar and the latter in an envelope conformation.

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Spiro compounds represent an important class of naturally occurring substances, characterized by highly pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important processes for the construction of spiro compounds (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I), is reported. The molecular structure of (I) is illustrated in Fig. 1. All bond lengths and angles of (I) are within normal ranges (Table 1).



There is a spiro system in (I), involving pyrrolidine and thiazolidine rings. The pyrrolidine ring C6–C3–C4–N2–C5 has an envelope conformation. C6–C3–C4–N2 is almost planar, the mean deviation from this plane being 0.0304 (3) Å. Atom C5 is out of this plane by 0.6221 (2) Å, forming the flap of the envelope. The dihedral angle between the C6–C5–N2 and C6–C3–C4–N2 planes is 138.4 (4)°. The dihedral angle between the C6–C3–C4–N2 plane and phenyl plane C16–C17–C18–C19–C20–C21 is 111.7 (4)°. The thiazolidine ring S1–C2–N1–C1–C3 is planar, the mean deviation from this plane being 0.0330 (3) Å. Atoms O1 and O2 deviate by 0.0624 (3) and 0.1308 (3) Å, respectively, from this plane. The dihedral angle between the C6–C3–C4–N2 plane of the pyrrolidine ring and the thiazolidine ring is 94.7 (4)°.

Experimental

A 100 ml round-bottomed flask, equipped with a condenser and an air moisture-impermeable seal, was charged with sarcosine (4 mmol), paraformaldehyde (8 mmol) and 5-benzylidene-3-benzylthiazolidine-2,4-dione (2 mmol). The reaction mixture was refluxed in dry toluene (60 ml) until the starting 5-benzylidene-3-benzylthiazolidine-2,4-dione was consumed. After evaporation of the solvent, the

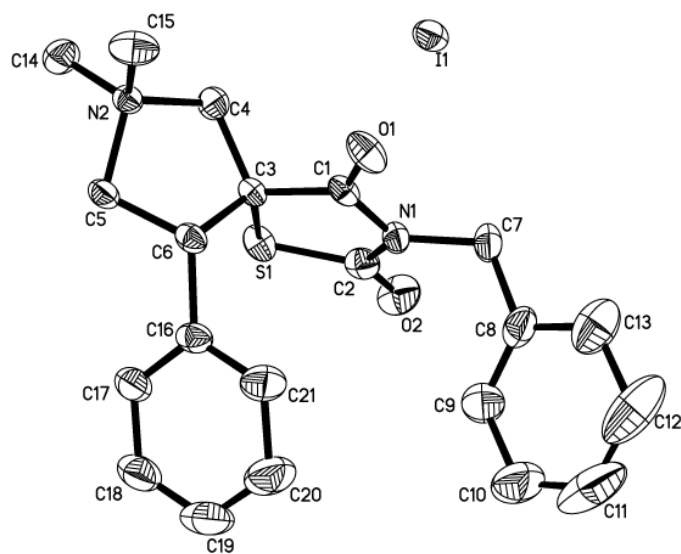


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for clarity.

residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 2:1); the product was obtained by column chromatography then stirred with methyl iodide in acetone at room temperature for 1 h. The title compound, (I), was obtained by filtration. M.p. 535–536 K; IR (KBr): 2920 (CH₂CH₃), 1753, 1676 (C=O) cm⁻¹; ¹H NMR (CD₃CN, p.p.m.): 3.52 (6H, s), 4.04–4.11 (1H, m), 4.34–4.42 (2H, m), 4.50–4.59 (2H, m), 4.68–4.79 (2H, m), 6.94–7.44 (10H, m); 20 mg of (I) was dissolved in 15 ml CH₃CN, and the solution was kept at room temperature for 20 d. Natural evaporation gave colorless single crystals of (I), suitable for X-ray analysis.

Crystal data

C₂₁H₂₃IN₂O₂S⁺·I⁻
M_r = 494.37
Monoclinic, P2₁/c
a = 20.791 (7) Å
b = 7.606 (3) Å
c = 13.484 (5) Å
β = 90.685 (5)°
V = 2132.1 (12) Å³
Z = 4

D_x = 1.540 Mg m⁻³
Mo Kα radiation
Cell parameters from 953 reflections
θ = 2.9–25.6°
μ = 1.62 mm⁻¹
T = 293 (2) K
Plate, colorless
0.30 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1997)
T_{min} = 0.675, T_{max} = 0.851
11395 measured reflections

4347 independent reflections
2790 reflections with I > 2σ(I)
R_{int} = 0.040
θ_{max} = 26.4°
h = -25 → 25
k = -9 → 5
l = -16 → 16

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.039
wR(F²) = 0.104
S = 1.02
4347 reflections
247 parameters
H-atoms parameter constrained

w = 1/[σ²(F_o²) + (0.084P)²]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.74 e Å⁻³
Δρ_{min} = -0.47 e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0077 (4)

Table 1

Selected geometric parameters (Å, °).

S1–C2	1.759 (5)	O1–C1	1.215 (5)
S1–C3	1.828 (4)	O2–C2	1.214 (5)
C2–S1–C3	92.8 (2)	O2–C2–S1	124.1 (4)
C1–N1–C2	116.4 (4)	N1–C2–S1	111.5 (3)
C5–N2–C4	104.7 (3)		
C6–C3–C4–N2	-6.2 (4)	C1–C3–C6–C5	151.1 (3)

The H atoms were included in the riding-model approximation with displacement parameters related to the atoms to which they were bonded.

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

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