

C3—C2—C12	120.4 (2)	121.2 (4)
O2—C3—C2	60.3 (2)	60.0 (3)
O2—C3—C4	115.8 (2)	116.3 (4)
C2—C3—C4	120.4 (3)	120.7 (4)
C3—C4—C5	114.3 (2)	113.9 (4)
C4—C5—C6	109.7 (2)	109.2 (4)
C4—C5—C11	107.1 (2)	108.2 (4)
C6—C5—C11	118.2 (2)	116.8 (4)
C1—C6—C5	106.5 (2)	106.3 (3)
C1—C6—C7	112.7 (2)	112.3 (3)
C5—C6—C7	115.9 (2)	116.9 (3)
C6—C7—C8	110.3 (2)	109.9 (4)
C6—C7—C13	109.4 (2)	109.7 (4)
C6—C7—C14	112.0 (2)	112.9 (4)
C8—C7—C13	104.4 (2)	104.3 (4)
C8—C7—C14	112.4 (2)	113.1 (4)
C13—C7—C14	108.0 (2)	106.4 (4)
C7—C8—C9	117.8 (3)	118.5 (4)
C8—C9—C10	115.4 (3)	116.1 (4)
C9—C10—C11	111.4 (3)	111.1 (4)
O1—C11—C5	—	112.6 (4)
O1—C11—C10	—	114.3 (4)
O1—C11—C15	—	59.1 (3)
C5—C11—C10	118.3 (2)	118.7 (4)
C5—C11—C15	120.6 (3)	119.7 (4)
O1—C15—C11	—	60.1 (3)
C10—C11—C15	121.2 (3)	117.4 (5)
C6—C1—C2—C3	21.1 (1)	20.8 (2)
C1—C2—C3—C4	2.4 (1)	2.4 (2)
C2—C3—C4—C5	10.0 (1)	10.8 (2)
C3—C4—C5—C6	-45.1 (2)	-46.4 (3)
C4—C5—C6—C1	67.0 (2)	68.0 (3)
C5—C6—C1—C2	-55.0 (2)	-55.0 (3)

H atoms were introduced at idealized positions in the calculations before the last refinement cycle but not refined. Refinement was by full-matrix least-squares methods including anisotropic displacement parameters for all non-H atoms.

Program used for data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Program used throughout the analysis: *CAD-4 SDP-Plus* (Frenz, 1985). Program used to solve the structures: *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) (completed by Fourier synthesis). Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1169). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Benharref, A., Bernardini, A., Fkih-Tetouani, S., Jacquier, R. & Viallefont, P. (1981). *J. Chem. Res. Synop.* **12**, 372–373.
- Benharref, A., Chekroun, A. & Lavergne, J. P. (1991). *Bull. Soc. Chim. Fr.* **128**, 738–741.
- Chiaroni, A., Pais, M., Riche, C., Benharref, A., Chekroun, A. & Lavergne, J. P. (1991). *Acta Cryst.* **C47**, 1945–1948.
- Chiaroni, A., Riche, C., Benharref, A., Chekroun, A. & Lavergne, J. P. (1992). *Acta Cryst.* **C48**, 1720–1722.
- Challand, B. D., Hikino, H., Kornis, G., Lange, G. & de Mayo, P. (1969). *J. Org. Chem.* **34**, 794–806.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Frenz, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 1.1. Enraf-Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

- Joseph, T. C. & Dev, S. (1968a). *Tetrahedron*, **24**, 3809–3827.
- Joseph, T. C. & Dev, S. (1968b). *Tetrahedron*, **24**, 3841–3852.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Nambudiry, M. E. N. & Rao, G. S. (1974). *Ind. J. Chem.* **12**, 889–890.
- Narula, A. P. S. & Dev, S. (1977). *Tetrahedron*, **33**, 813–816.

Acta Cryst. (1995). **C51**, 2661–2665

Six Substituted Thiazolidine-2-thiones

A. LAKNIFLI

Departement de Chimie, Faculte des Sciences d'Agadir, Universite Ibnou Zohr, BP 285 Agadir, Morocco

M. PIERROT

LBS-URA 1409, Centre Scientifique Saint Jerome, 13397 Marseille, France

F. CHANON AND M. CHANON

AM3 URA 1410, Centre Scientifique Saint Jerome, 13397 Marseille, France

(Received 10 February 1995; accepted 16 June 1995)

Abstract

The X-ray structures of six substituted thiazolidine-2-thiones are presented: *trans*-4,5-dimethylthiazolidine-2-thione, C₅H₉NS₂, (1), *cis*-4,5-dimethylthiazolidine-2-thione, C₅H₉NS₂, (2), *cis*-3,4,5-trimethylthiazolidine-2-thione, C₆H₁₁NS₂, (3), 4-methylthiazolidine-2-thione, C₄H₇NS₂, (4), 4-*tert*-butylthiazolidine-2-thione, C₇H₁₃NS₂, (5), 3-isopropylthiazolidine-2-thione, C₆H₁₁NS₂, (6). In this series the observed torsion angle S1—C5—C4—N3 (τ) varies from 22.5 (6) to 34.4 (8)° for compounds (1)–(5) and is equal to 9.7 (7)° for the 3-isopropyl-substituted compound (6). In compound (6), the C4—C5 distance is equal to 1.454 (5) Å, while it is in the range 1.513 (6)–1.54 (2) Å in the other five structures.

Comment

The aim of this work is to complete a series of conformational studies of the substituted thiazolidine-2-thione family, hereafter TH-2 TH (Nuzzo, Pierrot, Baldy, Chanon & Chanon, 1984; Lakanfli, Pierrot, Chanon & Chanon, 1995, and references therein). The present

report is concerned with a series of six derivatives with alkyl substituents at position 3, 4 and 5 of the heterocycle: *trans*-4,5-dimethyl-TH-2 TH, (1), *cis*-4,5-dimethyl-TH-2 TH, (2), *cis*-3,4,5-trimethyl-TH-2 TH, (3), 4-methyl-TH-2 TH, (4), 4-*tert*-butyl-TH-2 TH, (5), and 3-isopropyl-TH-2 TH, (6).

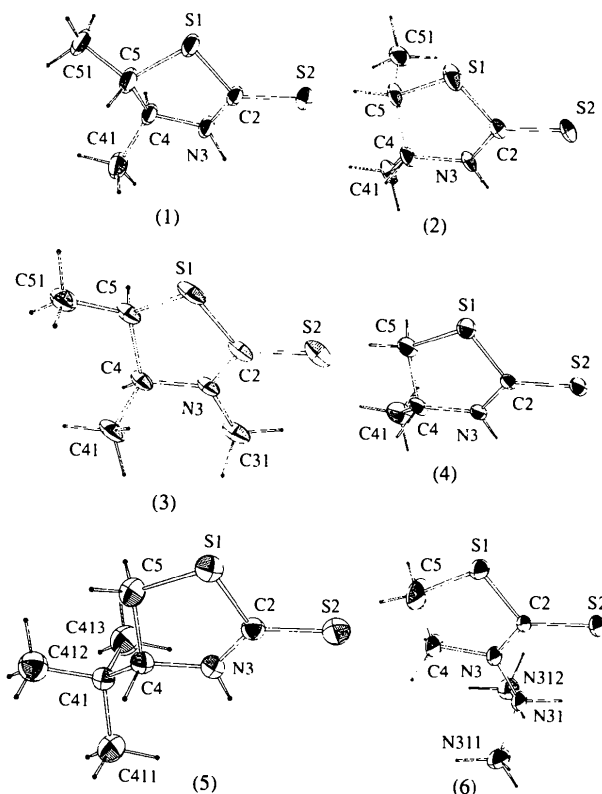
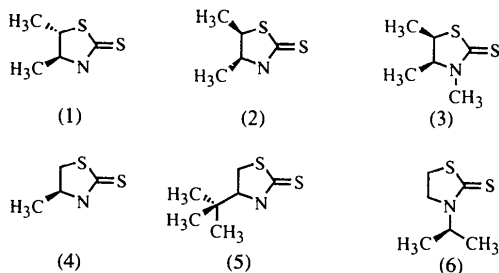


Fig. 1. ORTEP (Johnson, 1976) views of compounds (1)–(6) with ellipsoids at 50% probability and the atomic numbering schemes.

ORTEP (Johnson, 1976) views of the molecules and the atom numbering are shown in Fig. 1. As can be seen from Table 2, bond distances and angles are similar in the six compounds, except for those involving C4 and C5. For instance, the S1—C5 distance varies for five structures between 1.774 (6) and 1.819 (3) Å, but is equal to 1.846 (10) Å in (2), a value which is close to the largest values found in the TH-2 TH series [1.847 (5) (Xiaojie, Zhan-He & Chong-Xi, 1982) and 1.855 (5) Å (Nagao, Ikeda, Inoue, Yagi, Shiro & Fujita, 1985)]. It is worthwhile noting that the largest differences in distances and angles occur in two cases: in the crystal structures of (2) and (6). In the latter structure, the magnitudes of two angles (N3—C4—C5 and C4—C5—S1) are significantly different from those found in the other structures. However, the largest discrepancy is observed for the C4—C5 distance, which for structures (1) to (5) varies from 1.513 (6) [in (1)] to 1.54 (2) Å [in (2)] but is equal to 1.454 (5) Å in (6).

An interesting feature is shown in Fig. 2. For the first five molecules the observed conformation corresponds to a half chair with the C4 and C5 atoms above and below the planar S1—C2(=S2)—N3 fragment, the corresponding distances being given in Table 3. In these structures, the torsion angle S1—C5—C4—N3 (τ) varies from 22.5 (6) [in (4)] to 34.4 (8)° [in (2)]. On the other hand, the observed conformation in (6) is envelope-like, the C4 atom being almost in the mean plane of S1, C2, S2 and N3 [deviating by -0.042 (6) Å] and the C5 atom being above it [by 0.109 (7) Å]. Such a situation has been encountered frequently for N-substituted TH-2 TH derivatives. Thus, for donor substituents like alkyls, τ varies within 0–12° and the C4—C5 distance is less than 1.46 Å (Table 4). Alternatively, for attractor substituents [phenyl or O=C...R'; 22 structures found in the Cambridge Structural Database (Allen *et al.*, 1987)] τ is larger than 25° and the C4—C5 distance takes a normal value of 1.50–1.54 Å.

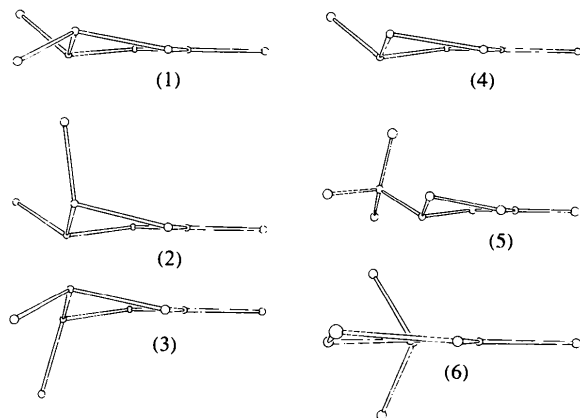


Fig. 2. View of the six molecules in the plane of the fragment S1—C2(=S2)—N3 showing the variation in conformations.

Experimental

The compounds were prepared following the procedures described by Bafford, Chanon & Metzger (1973) and crystallized by evaporation at room temperature of 50:50 benzene–cyclohexane solutions.

Compound (1)*Crystal data*

C₅H₉NS₂
M_r = 147.26
 Triclinic
P $\bar{1}$
a = 6.576 (2) Å
b = 7.517 (2) Å
c = 8.227 (3) Å
 α = 89.88 (5)°
 β = 70.69 (5)°
 γ = 74.00 (5)°
V = 367.1 (3) Å³
Z = 2
D_x = 1.332 Mg m⁻³

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 1528 measured reflections
 1312 independent reflections
 966 observed reflections
 $[I > 3\sigma(I)]$

Refinement

Refinement on *F*
R = 0.047
wR = 0.045
S = 0.539
 966 reflections
 73 parameters
 H-atom parameters not
 refined
 $w = 1/[\sigma^2(F)/4F^2 + 0.0016F^2]$

Compound (2)*Crystal data*

C₅H₉NS₂
M_r = 147.26
 Monoclinic
*P*2₁/*c*
a = 6.312 (2) Å
b = 10.036 (3) Å
c = 11.887 (4) Å
 β = 105.39 (5)°
V = 726.0 (6) Å³
Z = 4
D_x = 1.347 Mg m⁻³

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25
 reflections
 θ = 14–16°
 μ = 0.600 mm⁻¹
T = 293 K
 Prismatic
 0.5 × 0.4 × 0.3 mm
 Colourless

*R*_{int} = 0.032
 θ_{\max} = 22°
h = 0 → 7
k = -8 → 8
l = -9 → 9
 3 standard reflections
 frequency: 60 min
 intensity decay: 0.023%

$(\Delta/\sigma)_{\max}$ = 0.08
 $\Delta\rho_{\max}$ = 0.43 e Å⁻³
 $\Delta\rho_{\min}$ = -0.41 e Å⁻³
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25
 reflections
 θ = 14–16°
 μ = 0.607 mm⁻¹
T = 293 K
 Prismatic
 0.5 × 0.3 × 0.2 mm
 Colourless

*R*_{int} = 0.022
 θ_{\max} = 26°
h = -7 → 7
k = 0 → 12
l = 0 → 14

1598 measured reflections
 1428 independent reflections
 789 observed reflections
 $[I > 3\sigma(I)]$

Refinement

Refinement on *F*
R = 0.063
wR = 0.072
S = 1.59
 789 reflections
 73 parameters
 H-atom parameters not
 refined
 $w = 1/[\sigma^2(F)/4F^2 + 0.0016F^2]$

Compound (3)*Crystal data*

C₆H₁₁NS₂
M_r = 161.29
 Monoclinic
*P*2₁/*c*
a = 9.465 (3) Å
b = 7.036 (2) Å
c = 12.236 (4) Å
 β = 93.15 (5)°
V = 813.6 (7) Å³
Z = 4
D_x = 1.317 Mg m⁻³

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 none
 1637 measured reflections
 1453 independent reflections
 972 observed reflections
 $[I > 3\sigma(I)]$

Refinement

Refinement on *F*
R = 0.062
wR = 0.074
S = 1.42
 972 reflections
 163 parameters
 H-atom parameters not
 refined
 $w = 1/[\sigma^2(F)/4F^2 + 0.0016F^2]$

Compound (4)*Crystal data*

C₄H₇NS₂
M_r = 133.24

3 standard reflections
 frequency: 60 min
 intensity decay: 0.018%

$(\Delta/\sigma)_{\max}$ = 0.03
 $\Delta\rho_{\max}$ = 0.63 e Å⁻³
 $\Delta\rho_{\min}$ = -0.62 e Å⁻³
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25
 reflections
 θ = 14–16°
 μ = 0.548 mm⁻¹
T = 293 K
 Cubic
 0.6 × 0.6 × 0.5 mm
 Colourless

*R*_{int} = 0.035
 θ_{\max} = 25°
h = -11 → 11
k = 0 → 8
l = 0 → 14
 3 standard reflections
 frequency: 60 min
 intensity decay: 0.018%

$(\Delta/\sigma)_{\max}$ = 0.05
 $\Delta\rho_{\max}$ = 0.56 e Å⁻³
 $\Delta\rho_{\min}$ = -0.58 e Å⁻³
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Mo *K*α radiation
 λ = 0.71073 Å

Orthorhombic <i>P</i> 2 ₁ 2 ₁ <i>a</i> = 8.583 (3) Å <i>b</i> = 8.710 (3) Å <i>c</i> = 17.030 (6) Å <i>V</i> = 1273 (1) Å ³ <i>Z</i> = 8 <i>D_x</i> = 1.390 Mg m ⁻³	Cell parameters from 25 reflections <i>θ</i> = 14–16° <i>μ</i> = 0.685 mm ⁻¹ <i>T</i> = 293 K Prismatic 0.4 × 0.4 × 0.3 mm Colourless	<i>S</i> = 0.701 1810 reflections 91 parameters H-atom parameters not refined <i>w</i> = 1/[<i>σ</i> ² (<i>F</i>)/4 <i>F</i> ² + 0.0016 <i>F</i> ²]	Extinction correction: none Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
--	--	--	---

Data collection

Enraf–Nonius CAD-4 diffractometer <i>ω</i> /2 <i>θ</i> scans Absorption correction: none 1346 measured reflections 1221 independent reflections 1027 observed reflections [<i>I</i> > 3 <i>σ</i> (<i>I</i>)]	<i>R</i> _{int} = 0.021 <i>θ</i> _{max} = 25° <i>h</i> = 0 → 9 <i>k</i> = 0 → 11 <i>l</i> = 0 → 26 3 standard reflections frequency: 60 min intensity decay: 0.025%
--	--

Refinement

Refinement on <i>F</i> <i>R</i> = 0.045 <i>wR</i> = 0.040 <i>S</i> = 1.209 1057 reflections 127 parameters H-atom parameters not refined <i>w</i> = 1/[<i>σ</i> ² (<i>F</i>)/4 <i>F</i> ² + 0.0016 <i>F</i> ²]	(<i>Δ</i> / <i>σ</i>) _{max} = 0.04 <i>Δρ</i> _{max} = 0.53 e Å ⁻³ <i>Δρ</i> _{min} = -0.61 e Å ⁻³ Extinction correction: none Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
--	---

Compound (5)*Crystal data*

C ₇ H ₁₃ NS ₂ <i>M_r</i> = 175.32 Monoclinic <i>P</i> 2 ₁ / <i>c</i> <i>a</i> = 9.351 (3) Å <i>b</i> = 9.098 (3) Å <i>c</i> = 10.838 (3) Å <i>β</i> = 81.54 (5)° <i>V</i> = 912.0 (9) Å ³ <i>Z</i> = 4 <i>D_x</i> = 1.277 Mg m ⁻³	Mo <i>Kα</i> radiation <i>λ</i> = 0.71073 Å Cell parameters from 25 reflections <i>θ</i> = 14–16° <i>μ</i> = 0.494 mm ⁻¹ <i>T</i> = 293 K Prismatic 0.4 × 0.3 × 0.2 mm Colourless
---	--

Data collection

Enraf–Nonius CAD-4 diffractometer <i>ω</i> /2 <i>θ</i> scans Absorption correction: none 2357 measured reflections 2121 independent reflections 1810 observed reflections [<i>I</i> > 3 <i>σ</i> (<i>I</i>)]	<i>R</i> _{int} = 0.027 <i>θ</i> _{max} = 24° <i>h</i> = -10 → 10 <i>k</i> = 0 → 10 <i>l</i> = 0 → 12 3 standard reflections frequency: 60 min intensity decay: 0.018%
--	---

Refinement

Refinement on <i>F</i> <i>R</i> = 0.0352 <i>wR</i> = 0.0345	(<i>Δ</i> / <i>σ</i>) _{max} = 0.038 <i>Δρ</i> _{max} = 0.41 e Å ⁻³ <i>Δρ</i> _{min} = -0.42 e Å ⁻³
---	---

Compound (6)*Crystal data*

C ₆ H ₁₁ NS ₂ <i>M_r</i> = 161.29 Orthorhombic <i>P</i> na2 ₁ <i>a</i> = 13.494 (4) Å <i>b</i> = 8.205 (3) Å <i>c</i> = 7.491 (2) Å <i>V</i> = 829.4 (7) Å ³ <i>Z</i> = 4 <i>D_x</i> = 1.292 Mg m ⁻³	Mo <i>Kα</i> radiation <i>λ</i> = 0.71073 Å Cell parameters from 25 reflections <i>θ</i> = 14–16° <i>μ</i> = 0.537 mm ⁻¹ <i>T</i> = 293 K Cubic 0.6 × 0.6 × 0.6 mm Colourless
---	--

Data collection

Enraf–Nonius CAD-4 diffractometer <i>ω</i> /2 <i>θ</i> scans Absorption correction: none 2426 measured reflections 2311 independent reflections 1085 observed reflections [<i>I</i> > 3 <i>σ</i> (<i>I</i>)]	<i>R</i> _{int} = 0.025 <i>θ</i> _{max} = 26° <i>h</i> = 0 → 17 <i>k</i> = 0 → 10 <i>l</i> = 0 → 19 3 standard reflections frequency: 60 min intensity decay: 0.017%
--	---

Refinement

Refinement on <i>F</i> <i>R</i> = 0.0398 <i>wR</i> = 0.0383 <i>S</i> = 0.556 1085 reflections 82 parameters H-atom parameters not refined	<i>w</i> = 1/[<i>σ</i> ² (<i>F</i>)/4 <i>F</i> ² + 0.0016 <i>F</i> ²] (<i>Δ</i> / <i>σ</i>) _{max} = 0.04 <i>Δρ</i> _{max} = 0.47 e Å ⁻³ <i>Δρ</i> _{min} = -0.45 e Å ⁻³ Extinction correction: none Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
---	---

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	<i>B</i> _{eq} = (4/3)∑ _{<i>i</i>} ∑ _{<i>j</i>} β _{<i>ij</i>} <i>a_i</i> · <i>a_j</i> .			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Compound (1)				
S2	0.0958 (2)	0.2394 (2)	0.8514 (2)	4.91 (3)
S1	0.5893 (2)	0.0642 (2)	0.6846 (2)	4.44 (3)
N3	0.3260 (5)	-0.1134 (4)	0.8485 (5)	3.90 (8)
C2	0.3203 (6)	0.0546 (5)	0.8043 (5)	3.30 (9)
C4	0.5444 (6)	-0.2536 (5)	0.8084 (6)	3.7 (1)
C5	0.7063 (7)	-0.1852 (6)	0.6628 (7)	5.1 (1)
C51	0.9466 (7)	-0.2513 (7)	0.6477 (7)	5.8 (1)
C41	0.5306 (8)	-0.4427 (6)	0.7647 (8)	6.6 (2)
Compound (2)				
S1	0.0457 (5)	0.2832 (3)	0.7621 (3)	5.15 (7)
S2	0.2691 (4)	0.1216 (3)	0.9694 (3)	4.93 (7)
N3	-0.150 (1)	0.1006 (8)	0.8431 (7)	3.7 (2)

C2	0.046 (2)	0.157 (1)	0.8638 (8)	3.7 (2)
C4	-0.334 (2)	0.164 (1)	0.7525 (9)	3.9 (2)
C5	-0.221 (2)	0.229 (1)	0.6664 (9)	4.5 (3)
C41	-0.504 (2)	0.058 (1)	0.701 (1)	4.9 (3)
C51	-0.181 (2)	0.140 (1)	0.571 (1)	5.6 (3)

Compound (3)

S2	0.8258 (2)	0.1307 (3)	0.6622 (2)	4.38 (2)
S1	0.6836 (3)	0.0973 (2)	0.4402 (2)	5.69 (3)
C5	0.7361 (3)	-0.0875 (4)	0.3467 (3)	3.67 (8)
N3	0.8436 (1)	-0.1612 (3)	0.5230 (2)	3.33 (7)
C2	0.7940 (2)	0.0082 (2)	0.5472 (3)	3.69 (8)
C4	0.7817 (3)	-0.2511 (2)	0.4217 (3)	3.40 (8)
C31	0.9394 (4)	-0.2662 (3)	0.5971 (5)	4.70 (1)
C51	0.6205 (4)	-0.1295 (3)	0.2599 (4)	5.10 (2)
C41	0.6650 (5)	-0.3855 (4)	0.4490 (4)	4.70 (2)

Compound (4)

S2	0.4718 (2)	0.0298 (3)	0.9319 (1)	3.91 (4)
S2'	0.2363 (3)	-0.2678 (2)	1.2495 (1)	4.49 (5)
S1	0.7085 (3)	0.0869 (3)	1.0546 (1)	4.76 (5)
S1'	0.3321 (3)	-0.0434 (3)	1.1287 (1)	5.02 (5)
N3	0.7692 (7)	-0.0636 (7)	0.9316 (3)	3.6 (1)
N3'	0.1818 (8)	0.0329 (7)	1.2493 (4)	3.8 (1)
C2	0.6524 (9)	0.0084 (9)	0.9648 (4)	3.0 (2)
C2'	0.243 (1)	-0.0878 (9)	1.2167 (4)	3.2 (2)
C4	0.9149 (9)	-0.089 (1)	0.9765 (5)	4.3 (2)
C4'	0.189 (1)	0.1803 (9)	1.2073 (5)	3.8 (2)
C5	0.910 (1)	0.040 (1)	1.0392 (5)	4.8 (2)
C5'	0.315 (1)	0.160 (1)	1.1453 (6)	5.5 (2)
C41	1.053 (1)	-0.082 (1)	0.9253 (7)	7.0 (3)
C41'	0.220 (1)	0.309 (1)	1.2619 (6)	6.6 (3)

Compound (5)

S2	0.81342 (7)	0.41333 (8)	0.11502 (6)	4.01 (1)
S1	0.89914 (7)	0.34950 (8)	0.36219 (6)	3.79 (1)
N3	1.0741 (2)	0.4540 (2)	0.1834 (2)	3.14 (4)
C2	0.9386 (2)	0.4111 (3)	0.2095 (2)	2.86 (4)
C4	1.1611 (3)	0.4621 (3)	0.2858 (2)	3.01 (5)
C5	1.0880 (3)	0.3526 (3)	0.3806 (2)	3.83 (6)
C41	1.3233 (2)	0.4356 (3)	0.2432 (2)	3.15 (5)
C411	1.3841 (3)	0.5641 (3)	0.1628 (3)	4.20 (6)
C412	1.3983 (2)	0.4336 (3)	0.3578 (3)	4.86 (7)
C413	1.3509 (3)	0.2940 (3)	0.1692 (3)	5.30 (7)

Compound (6)

S2	0.37454 (9)	0.1536 (2)	0.6582 (4)	4.79 (2)
S1	0.30645 (9)	-0.0716 (1)	0.9412 (2)	4.87 (2)
N3	0.4032 (3)	0.1873 (4)	1.0118 (4)	3.95 (7)
C2	0.3673 (3)	0.1058 (5)	0.8744 (5)	3.29 (7)
C4	0.3831 (4)	0.1187 (6)	1.1871 (5)	5.7 (1)
C5	0.3375 (5)	-0.0411 (7)	1.1690 (8)	8.9 (1)
C31	0.4560 (3)	0.3425 (5)	0.9937 (6)	4.40 (9)
C311	0.5582 (4)	0.3296 (7)	1.0765 (9)	6.3 (1)
C312	0.3976 (4)	0.4813 (6)	1.0736 (9)	6.3 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

	(1)	(2)	(3)	(4)*	(5)	(6)
S1—C2	1.739 (4)	1.751 (11)	1.746 (3)	1.735 (6)	1.735 (2)	1.745 (4)
S1—C5	1.804 (4)	1.846 (10)	1.819 (3)	1.797 (6)	1.806 (3)	1.774 (6)
S2—C2	1.659 (3)	1.657 (9)	1.664 (3)	1.662 (6)	1.665 (2)	1.670 (4)
N3—C2	1.307 (5)	1.323 (12)	1.321 (4)	1.304 (7)	1.316 (3)	1.320 (5)
N3—C4	1.462 (4)	1.500 (11)	1.483 (2)	1.476 (8)	1.471 (3)	1.454 (5)
C4—C5	1.513 (6)	1.54 (2)	1.520 (2)	1.537 (10)	1.520 (3)	1.454 (5)
S1—C2—S2	122.3 (2)	121.5 (6)	121.43 (5)	120.8 (3)	121.4 (1)	120.1 (2)
S2—C2—N3	127.2 (3)	127.9 (8)	127.36 (5)	127.9 (4)	127.3 (2)	128.1 (3)
C5—S1—C2	92.8 (2)	92.6 (5)	92.39 (6)	92.8 (2)	91.5 (1)	92.7 (2)
S1—C2—N3	110.5 (2)	110.6 (6)	111.20 (5)	111.1 (4)	111.3 (2)	111.8 (3)
C2—N3—C4	118.9 (3)	117.4 (8)	116.31 (4)	119.3 (4)	118.3 (2)	116.1 (4)
N3—C4—C5	105.6 (3)	104.5 (8)	105.48 (5)	104.1 (5)	103.5 (2)	110.1 (4)
C4—C5—S1	105.6 (2)	102.9 (7)	103.89 (4)	106.6 (4)	106.6 (2)	108.5 (4)

* Averaged values for the two independent molecules.

Table 3. Distance (\AA) to the S1—C2—S2—N3 fragment and value of the torsion angle τ ($^\circ$)

	(1)	(2)	(3)	(4)*	(5)	(6)
C4	-0.130 (4)	-0.197 (10)	-0.205 (4)	-0.205 (5)	-0.186 (2)	-0.042 (6)
C5	0.309 (5)	0.398 (11)	0.345 (4)	0.239(5)	0.311 (3)	0.109 (7)
τ	26.1 (4)	34.4 (8)	-32.9 (4)	26.2(5)	-29.8 (3)	-9.7 (7)
				0.280(6)	22.5(6)	

* Two independent molecules.

Table 4. Some examples of planar conformations

N substituent	τ angle ($^\circ$)	C4—C5 (\AA)	References
Cyclohexyl	7.5 (5)	1.463 (4)	Nuzzo, Pierrot, Baldy, Chanon & Chanon (1984)
Isopropyl	9.7 (7)	1.454 (5)	This work
<i>tert</i> -Butyl (molecule 1)	0 (1)	1.40 (1)	Jaud <i>et al.</i> (1995)
(molecule 2)	12.7 (9)	1.45 (1)	

Data were collected using *CAD-4 Software* (Enraf-Nonius, 1989). *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) was used to solve the structures, the solutions being completed by difference Fourier syntheses. H atoms were introduced at idealized positions in the calculations before the last refinement cycles but not refined. Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms. The *SDP* (Frenz, 1985) programs were used throughout the analyses.

Lists of structure factors for compounds (1) and (4) only, and anisotropic displacement parameters, H-atom coordinates and complete geometry for all six compounds have been deposited with the IUCr (Reference: AB1269). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* pp. S1—S19.
- Bafford, K. A., Chanon, F. & Metzger, J. (1973). *Bull. Soc. Chim. Fr.* pp. 973—977.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Frenz, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 1.1. Enraf-Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Jaud, J., Raynaud, B., Baldy, A., Chanon, F., Lknifli, A. & Chanon, M. (1995). *Z. Kristallogr.* **210**, 311.
- Lknifli, A., Pierrot, M., Chanon, F. & Chanon, M. (1995). *Acta Cryst.* **C51**, 2113—2116.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Nagao, Y., Ikeda, T., Inoue, T., Yagi, M., Shiro, M. & Fujita, E. (1985). *J. Org. Chem.* **50**, 4072—4080.
- Nuzzo, O., Pierrot, M., Baldy, A., Chanon, F. & Chanon, M. (1984). *Can. J. Chem.* **62**, 1410—1413.
- Xiaojie, X., Zhan-He, C. & Chong-Xi, L. (1982). *Acta Chim. Sinica*, **40**, 752—754. (In Chinese.)