bond between sp^2-hybridized carbon atoms, in agreement with previous results, irrespective of whether the 5-phenyl ring is substituted or not. In this context it may be noted that the value of the N(4)—C(5)—C(1′)—C(6′) torsion angle, −62°, would preclude significant electron delocalization across C(5)—C(1′).

An intermolecular hydrogen bond occurs between the carbonyl O atom and the amide N atom of the centrosymmetrically related molecule at $(-1-x, -1-y, -2-z)$. Pertinent parameters are O(2)···H(1), 2·04 (3), O(2)···N(1) 2·916 (3) Å and angle O(2)···N(1)—H(1), 7 (2)°. Other intermolecular contacts correspond to normal van der Waals interactions.

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References

Braestrup, C. & Squires, R. F. (1978). *Eur. J. Pharmacol.* **48**, 263–270.

Chananont, P., Hamor, T. A. & Martin, I. L. (1981). *Acta Cryst.* B**37**, 1371–1375.

Fryer, R. I., Leimgruber, W. & Trybulski, E. W. (1982). *J. Med. Chem.* **25**, 1050–1055.

Hamor, T. A. & Martin, I. L. (1983). *Progress in Medicinal Chemistry*, Vol. 20, edited by G. P. Ellis & G. B. West, pp. 157–223. Amsterdam: Elsevier.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Sheldrick, G. M. (1978). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

Squires, R. F. & Braestrup, C. (1977). *Nature (London)*, **266**, 732–734.

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4,4′-Thiodi(α-toluenethiol)

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Abstract. $C_{14}H_{14}S_3$, $M_r = 278\cdot4$, monoclinic, $C2/c$, $a = 14\cdot135$ (5), $b = 5\cdot949$ (2), $c = 17\cdot301$ (6) Å, $\beta = 114\cdot11$ (3)°, $V = 1327\cdot9$ (8) Å^3, $Z = 4$, $D_x = 1\cdot40$ (1) g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu(\text{Mo } K\alpha) = 4\cdot73$ cm^{-1}, $F(000) = 584$, $T = 292$ K, $R = 0\cdot057$ for 529 unique reflections. Atom S(2) lies on a crystallographic twofold axis which relates two toluenethiol moieties of the molecule. The phenyl ring is at 33·4 (5)° to the plane defined by C(4)—S(2)—C(4′) and the valency angle at S(2) is 105·8 (3)°.

Introduction. The X-ray study of 4,4′-thiodi(α-toluenethiol), hereafter referred to as (I), is a continuation of our previous investigation on aryl–sulfuric and alkyl–aryl–sulfuric compounds which are basic structures in the chemistry of polythioesters (Ratajczak-Sitarz, Katrusiak, Kałuski & Garbarczyk, 1987; Garbarczyk, 1984; Andreetti, Garbarczyk & Królikowska, 1981). In the structure of (I) we were mainly interested in the conformation of the molecule – *i.e.* in the torsion of the phenyl ring around bond S(2)—C(4) – and in the geometry of S(2), which bridges the phenyl rings in the molecule.

Experimental. Single crystals of (I) suitable for X-ray investigation were obtained by slow evaporation of its xylene solution at room temperature. They were very small colourless parallelepipeds. The crystal selected for data collection had dimensions of 0·10 × 0·06 × 0·05 mm. The measurement was carried out on a Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\alpha$ radiation. The unit-cell parameters were determined by a least-squares fit to 15 automatically centred reflections ($8\cdot39 \leq 2\theta \leq 14\cdot14°$). Two control reflections were monitored after each 100 intensity measurements. θ–2θ scan method with a variable scan speed (from 2·1 to 29·3° min^{-1}) was used. 1241 reflections were collected up to $2\theta = 45\cdot0°$, with indices: h 0/18, k 0/10, l −20/20. The background and integrated intensity for each reflection were calculated according to the profile-analysis method of Lehmann & Larsen (1974). Of 906 unique reflections ($R_{\text{int}} = 0\cdot033$) 529 reflections had $I \geq 1\cdot96\sigma(I)$ and were used in the structure refinement. Only Lp corrections were applied. The structure was solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). After the refinement of the non-hydrogen atoms with anisotropic temperature factors all H atoms, except that at S(1) which could not be found, were located from a ΔF map and were included in the refinement with isotropic temperature factors. The function minimized was

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$\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F)$. The final cycles of the full-matrix least-squares refinement gave $R = 0{\cdot}057$, $wR = 0{\cdot}047$ and $S = 1{\cdot}34$. For all the refined parameters the final Δ/σ was smaller than 0·07, the highest peak on the final ΔF map was 0·20, the minimum −0·21 e Å^{-3}. The final atomic parameters are listed in Table 1.* Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) were applied. Most of the calculations were performed with *SHELX*76 (Sheldrick, 1976) on a RIAD 32 computer.

Discussion. A perspective view of the molecule of (I), its atom numbering and its selected torsion angles are presented in Fig. 1. S(2) is positioned on a twofold axis in the crystal lattice; thus two moieties of the molecule are symmetry related. The torsion of the phenyl ring about S(2)—C(4) can be conveniently described by torsion angle C(4′)—S(2)—C(4)—C(3) (prime refers to the symmetry-related atoms of the same molecule) – see Fig. 1. In the similar structure of diphenyl sulfide this value was 55·6° (Rozsondai, Moore, Gregory & Hargittai, 1977) while the two independent torsion angles about the S—C bond of 1,4-bis(phenylthio)-benzene had values of 14·9 and 59·2° (Andreetti, Garbarczyk & Królikowska, 1981). In the crystalline phase of poly-*p*-phenylene sulfide this angle was 45° (Tabor, Magré & Boon, 1971). Comparison of the conformation of these structures suggests that the 'soft' torsion about the S—C bond depends both on the intramolecular forces and on the steric hindrance within the molecules. In (I) the intramolecular distances from H(3) to C(3′), 2·71 (6), and to C(4), 2·82 (6) Å, indicate close contacts of these atoms commensurate with the sum of their van der Waals radii (Kitaigorodski, 1976).

The bond lengths and valency angles of (I) are listed in Table 2. Valency angle C(4)—S(2)—C(4′) is very similar to the corresponding value of 105·4 (2)° found in 1,4-bis(phenylthio)benzene (Andreetti, Garbarczyk & Królikowska, 1981) and slightly wider than 103·7° reported for diphenyl sulfide (Rozsondai, Moore, Gregory & Hargittai, 1977). The S(2)—C(4) bond length agrees very well with the corresponding values in diphenyl sulfide (1·771 Å) and in 1,4-bis(phenylthio)benzene [1·777 (4) and 1·783 (4) Å].

The phenyl ring of (I) is almost perfectly planar – the χ^2 value of the least-squares plane fitted to the atoms of this ring is 3·6. S(2) is −0·074 (1) Å off this plane, C(7) 0·049 (11) and S(1) 1·487 (1) Å. The most significant deformations of the phenyl ring (when compared with the benzene ring) are observed in the vicinity of atom C(1): valency angle C(2)—C(1)—C(6) is slightly sharpened while the valency angles at the adjacent C(2) and C(6) atoms are widened.

A stereoscopic view of the unit-cell contents of (I) is presented in Fig. 2. There are only van der Waals interactions between the molecules in the crystal lattice.

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Table 1. *Fractional coordinates of the atoms, their temperature factors* (U_{eq} *for the non-*H *atoms and* U_{iso} *for the* H *atoms*) *and* C—H *distances*

$$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	U_{eq}(Å^2)
S(1)	−0·4167 (2)	0·7848 (5)	−0·0303 (1)	0·067 (1)
C(1)	−0·2924 (6)	0·584 (1)	0·1232 (4)	0·040 (3)
C(2)	−0·2087 (7)	0·663 (1)	0·1954 (5)	0·040 (4)
C(3)	−0·1185 (6)	0·543 (1)	0·2357 (5)	0·039 (3)
C(4)	−0·1094 (5)	0·335 (1)	0·2024 (4)	0·035 (3)
C(5)	−0·1926 (6)	0·248 (1)	0·1306 (4)	0·040 (3)
C(6)	−0·2808 (6)	0·375 (1)	0·0931 (5)	0·036 (3)
C(7)	−0·3876 (7)	0·722 (2)	0·0802 (6)	0·050 (4)
S(2)	0·0 (0)	0·1536 (5)	0·2500 (0)	0·050 (1)

	x	y	z	U_{iso}(Å^2)	d_{C-H}(Å)
H(2)	−0·211 (4)	0·787 (10)	0·218 (3)	0·03 (2)	0·85 (5)
H(3)	−0·060 (5)	0·607 (12)	0·289 (4)	0·09 (3)	1·03 (7)
H(5)	−0·190 (5)	0·099 (13)	0·095 (4)	0·10 (3)	1·08 (8)
H(6)	−0·341 (5)	0·312 (11)	0·037 (4)	0·06 (2)	1·05 (6)
H(71)	−0·388 (6)	0·857 (14)	0·102 (5)	0·09 (4)	0·89 (8)
H(72)	−0·458 (5)	0·645 (13)	0·072 (4)	0·09 (3)	1·05 (7)

Table 2. *Bond lengths* (Å) *and valency angles* (°)

S(1)—C(7)	1·814 (9)	C(4)—S(2)	1·785 (7)
C(7)—C(1)	1·493 (11)	C(4)—C(5)	1·419 (9)
C(1)—C(2)	1·407 (10)	C(5)—C(6)	1·381 (10)
C(2)—C(3)	1·389 (10)	C(6)—C(1)	1·384 (10)
C(3)—C(4)	1·395 (9)		

S(1)—C(7)—C(1)	113·8 (6)	S(2)—C(4)—C(5)	115·8 (6)
C(7)—C(1)—C(2)	120·8 (8)	C(4)—C(5)—C(6)	118·5 (8)
C(1)—C(2)—C(3)	123·0 (8)	C(5)—C(6)—C(1)	122·8 (8)
C(2)—C(3)—C(4)	117·9 (8)	C(6)—C(1)—C(2)	117·0 (8)
C(3)—C(4)—S(2)	123·2 (6)	C(6)—C(1)—C(7)	122·2 (8)
C(3)—C(4)—C(5)	120·7 (7)		

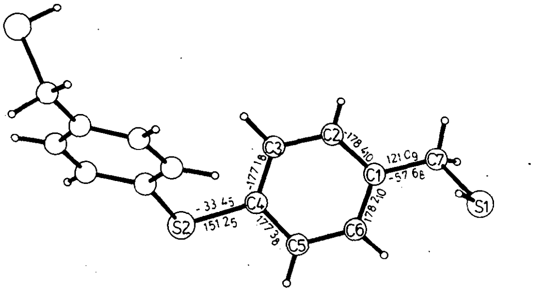

Fig. 1. A perspective drawing of a molecule of (I) as viewed along the direction perpendicular to one of the phenyl rings (Motherwell & Clegg, 1978). Selected torsion angles are given for non-hydrogen atoms.

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* Lists of structure factors, anisotropic thermal parameters of non-hydrogen atoms and H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44380 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

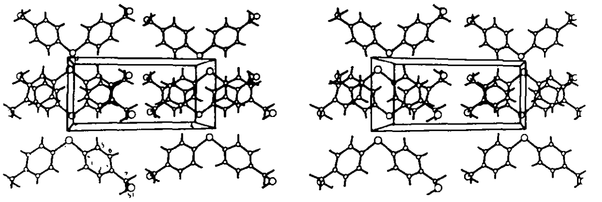

Fig. 2. A stereodiagram of the unit-cell contents (Motherwell & Clegg, 1978).

References

ANDREETTI, G. D., GARBARCZYK, J. & KRÓLIKOWSKA, M. (1981). *Cryst. Struct. Commun.* **10**, 789–792.

GARBARCZYK, J. (1984). *Makromol. Chem.* **185**, 2033–2041.

International Tables for X-ray Crystallography. (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KITAIGORODSKI, A. I. (1976). *Kryształy Molekularne.* Warszawa: PWN.

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

RATAJCZAK-SITARZ, M., KATRUSIAK, A., KALUSKI, Z. & GARBARCZYK, J. (1987). *Acta Cryst.* C**43**, 2389–2391.

ROZSONDAI, B., MOORE, J. H., GREGORY, D. C. & HARGITTAI, I. (1977). *Acta Chim. Acad. Sci. Hung.* **94**, 321–331.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

TABOR, B. J., MAGRÉ, E. P. & BOON, J. (1971). *Eur. Polym. J.* **7**, 1127.

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Structure of 1,5-Diazabicyclo[5.4.0]undecan-6-one

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Abstract. $C_9H_{16}N_2O$, $M_r = 168{\cdot}24$, monoclinic, $P2_1/c$, $a = 12{\cdot}287\,(2)$, $b = 5{\cdot}167\,(1)$, $c = 15{\cdot}186\,(2)$ Å, $\beta = 107{\cdot}97\,(1)°$, $V = 917{\cdot}09\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}216$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 6{\cdot}08$ cm^{-1}, $F(000) = 368$, room temperature, $R = 0{\cdot}067$ for 1546 reflexions with $I > 3\sigma(I)$. The seven-membered and six-membered rings are in chair conformations. Molecules related by the centre of inversion form hydrogen-bonded dimers, H···O 2·14 (5) Å, N—H···O 132 (2)°. The atoms participating in the hydrogen bond form an eight-membered ring which is almost flat (except for the H atoms).

Introduction. The title compound was obtained first by Paquett & Scott (1968). Recently a more simple and efficient synthesis of this heterocyclic system has been reported (Guryn, 1987). This compound is now used as a substrate in the synthesis of hypotensive drugs which act on the central nervous system (Guryn, Szadowska, Czarnecka & Wejman, 1987).

The present structural work has been undertaken to obtain more detailed information about the bond system and conformation of the 1,5-diazabicycloundecanone rings.

Experimental. Colourless, needle-shaped crystals obtained from acetone, dimensions 0·25 × 0·25 × 0·4 mm; cell parameters and intensity data measured on an Enraf–Nonius CAD-4 diffractometer. Orientation matrix from 25 carefully centred reflexions with $\theta_{\max} = 51°$, graphite-monochromatized Cu $K\alpha$, radiation range of h, k and l −15→15, 0→6, 0→8 respectively; total of 2162 independent reflexions measured to $(\sin\theta)/\lambda = 0{\cdot}61$ Å^{-1}; data not corrected for absorption (controls: orientation matrix and intensity $1\bar{1}5$ with respect to $P2_1/a$), $\mu r = 0{\cdot}15$; 1546 reflexions with $I > 3\sigma(I)$ used in refinement. Solution by direct methods using *SHELX76* (Sheldrick, 1976); refinement on F in space group $P2_1/c$ by full-matrix least-squares procedure; after initial anisotropic refine-

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