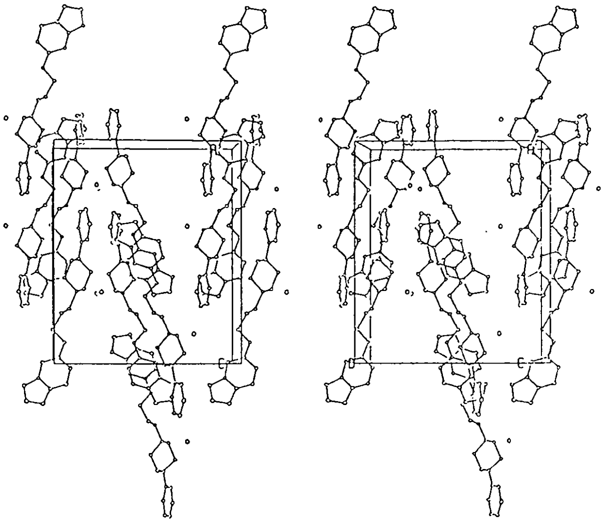

Fig. 2. The crystal packing viewed along the *c* axis.

Related literature. Pharmacological study demonstrates that this substance is a selective 5-HT_{1A} (serotonin$_{1A}$) receptor agonist (Matsuda, Seong, Aono, Kanda, Baba, Saito, Tobe & Iwata, 1989).

The authors would like to thank Mr Mitsuo Egawa and Mr Kunimiki Ohtsu of Mitsubishi Kasei Co. Ltd for supplying the crystals and Professors Heitaroh Iwata and Akemichi Baba of the Pharmaceutical Department, Osaka University, for their kind assistance. KO would also like to thank Mr Osamu Kamei, the President of the Computer, Electronics, and Medical College in Osaka.

References

Ashida, T. (1979). *HBLS-V. The Universal Crystallographic Computing System – Osaka*, pp. 53–59. The Computation Center, Osaka Univ., Japan.

Fujii, S. (1979). *MOLCON. The Universal Crystallographic Computing System – Osaka*, pp. 66–77. The Computation Center, Osaka Univ., Japan.

Johnson, C. K. (1965). *ORTEP*. ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Matsuda, T., Seong, Y. H., Aono, H., Kanda, T., Baba, A., Saito, K., Tobe, A. & Iwata, H. (1989). *Eur. J. Pharmacol.* **170**, 75–82.

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

Sheldrick, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Acta Cryst. (1993). C**49**, 1127–1128

Structure of 2,2-Bis(3,5-di-*tert*-butylphenyl)propane-2′,2″-dithiol

By Wolfgang Hiller

Anorganisch-chemisches Institut der Technischen Universität München, Lichtenbergstr. 4, *D*-8046 *Garching, Germany*

and Wolfgang Rundel

Institut für Organische Chemie der Universität Tübingen, Auf der Morgenstelle 18, *D*-7400 *Tübingen, Germany*

(*Received* 6 *May* 1992; *accepted* 28 *January* 1993)

Abstract. $C_{31}H_{48}S_2$, $M_r = 484.86$, monoclinic, $C2/c$, $a = 18.479$ (2), $b = 9.294$ (1), $c = 18.126$ (2) Å, $\beta = 99.41$ (1)°, $V = 3071.3$ Å^3, $Z = 4$, $D_x = 1.049$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.627$ mm^{-1}, $F(000) = 1064$, $T = 293$ K. The final R value was 0.055 for 2413 significant [$I > 3\sigma(I)$] reflections. The crystal structure confirms the close relationship of the thiol groups (S···S′ = 4.29 Å) which explains the observed elimination of hydrogen sulfide leading to the formation of the corresponding thioxanthene.

Experimental. The compound was synthesized by a Newman–Kwart rearrangement (Newman & Karnes, 1966; Kwart & Evans, 1966). In contrast to the very stable 2,4,6-tri-*tert*-butylbenzenethiol (Rundel, 1968), the title compound proved to be rather sensitive, in that it readily eliminates hydrogen sulfide, for instance when heated to its melting point (440 K). Crystals were grown by slowly cooling a warm saturated ethyl acetate solution of the pure (crystallized twice from ethyl acetate) compound. A yellow single crystal of approximate dimensions 0.15 × 0.15 ×

0108-2701/93/061127-02$06.00

Table 1. *Data collection and structure refinement parameters*

Crystal shape	Prism
Diffractometer used	Enraf–Nonius CAD-4
Method of intensity measurement	ω/θ
Number and θ range of reflections for lattice parameters	25; 27–42°
Method used for absorption correction	*DIFABS* (Walker & Stuart, 1983)
Minimum absorption correction	0.539
Maximum absorption correction	1.376
Average absorption correction	0.957
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement	0.588 Å^{-1}
Range of h, k and l	0 to 22, 0 to 11, −21 to 21
Standard reflections	10,0,$\bar{8}$; 247; 060
Interval, intensity variation	2 h, none
Reflections measured; θ range	2034; 69° (274 unobserved)
Observed reflections	2413 with $I > 3\sigma(I)$
Method used to solve structure	Direct methods
Use of F or F^2 in LS refinement	F
Method of locating H atoms	Difference electron density map
Weighting scheme	$1/\sigma_F^2$
Parameters refined	247
Value of R	0.055
Value of wR	0.048
Ratio of max. LS shift to e.s.d. (Δ/σ)	0.001
Max. height in final ΔF map	0.255 e Å^{-3}
S	1.334
Secondary-extinction coefficient	1.76 (2) × 10^{-6} (Zachariasen, 1963)
Sources of atomic scattering factors	*International Tables* (1974, Vol. IV)
Computer used	DEC MicroVAX 3500
Programs used	*MolEN* (Enraf–Nonius, 1990)

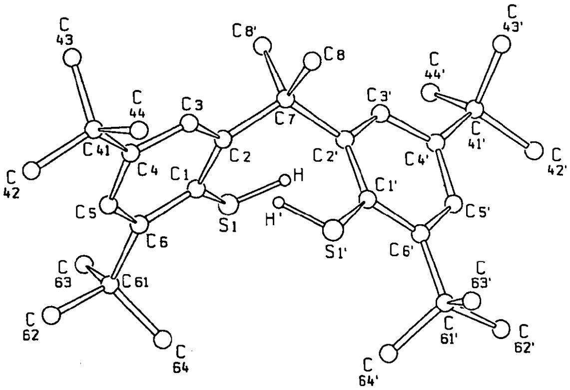

Fig. 1. Plot of the title molecule showing the atom-numbering scheme.

0.25 mm was mounted on a glass fiber. The systematic absences pointed to space group $C2/c$ or Cc, of which the centrosymmetric space group was later confirmed. In the final full-matrix least-squares refinement, all non-H atoms were assigned anisotropic atomic displacement parameters. A summary of data collection and structure refinement parameters is given in Table 1. Final atomic coordinates are listed in Table 2,* distances and angles in Table 3. A graphic representation (Keller, 1988) of the molecule is shown in Fig. 1.

Table 2. *Atomic positional and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S	0.57577 (4)	0.5861 (1)	0.35337 (5)	0.0803 (6)
C1	0.4817 (1)	0.5352 (3)	0.3430 (1)	0.049 (1)
C2	0.4547 (1)	0.4150 (3)	0.2987 (1)	0.050 (1)
C3	0.3844 (2)	0.3687 (3)	0.3028 (2)	0.055 (2)
C4	0.3386 (1)	0.4336 (3)	0.3461 (1)	0.052 (2)
C5	0.3653 (2)	0.5572 (3)	0.3843 (1)	0.055 (2)
C6	0.4363 (2)	0.6117 (3)	0.3837 (1)	0.049 (1)
C7	0.5	0.3258 (4)	0.25	0.056 (1)
C8	0.4514 (2)	0.2262 (4)	0.1942 (2)	0.085 (2)
C41	0.2636 (2)	0.3703 (4)	0.3512 (2)	0.066 (2)
C42	0.2211 (2)	0.4611 (6)	0.4009 (3)	0.135 (4)
C43	0.2756 (2)	0.2199 (5)	0.3869 (3)	0.113 (3)
C44	0.2186 (2)	0.3533 (6)	0.2737 (2)	0.121 (4)
C61	0.4571 (2)	0.7530 (3)	0.4274 (2)	0.061 (2)
C62	0.3933 (2)	0.8146 (4)	0.4615 (2)	0.095 (3)
C63	0.5203 (2)	0.7283 (4)	0.4925 (2)	0.076 (3)
C64	0.4776 (2)	0.8682 (4)	0.3733 (2)	0.088 (3)

Table 3. *Distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

S—C1	1.781 (3)	C5—C6	1.391 (4)
S—H	1.250 (1)	C6—C61	1.552 (4)
C1—C2	1.419 (4)	C7—C8	1.547 (5)
C1—C6	1.412 (4)	C41—C42	1.540 (6)
C2—C3	1.383 (4)	C41—C43	1.542 (5)
C2—C7	1.553 (3)	C41—C44	1.519 (5)
C3—C4	1.383 (5)	C61—C62	1.528 (5)
C4—C5	1.389 (4)	C61—C63	1.536 (4)
C4—C41	1.522 (5)	C61—C64	1.542 (5)

C1—S—H	107.1 (2)	C2—C7—C8	112.5 (2)
S—C1—C2	121.1 (3)	C2—C7—C8′	104.9 (2)
S—C1—C6	118.5 (2)	C8—C7—C8′	106.4 (3)
C2—C1—C6	120.4 (2)	C4—C41—C42	112.4 (3)
C1—C2—C3	117.5 (2)	C4—C41—C43	107.8 (2)
C1—C2—C7	124.8 (2)	C4—C41—C44	110.5 (3)
C3—C2—C7	117.7 (2)	C42—C41—C43	107.7 (3)
C2—C3—C4	124.2 (3)	C42—C41—C44	110.1 (3)
C3—C4—C5	116.3 (3)	C43—C41—C44	108.2 (3)
C3—C4—C41	120.9 (2)	C6—C61—C62	111.7 (3)
C4—C4—C41	122.8 (3)	C6—C61—C63	111.5 (2)
C4—C5—C6	123.6 (3)	C6—C61—C64	109.5 (2)
C1—C6—C5	117.8 (2)	C62—C61—C63	106.6 (3)
C1—C6—C61	124.3 (2)	C62—C61—C64	106.4 (3)
C5—C6—C61	118.0 (3)	C63—C61—C64	111.0 (3)
C2—C7—C2′	115.5 (3)		

Related literature. A similar compound, 2′,2″-dithio-2,2-bis(3,5-di-*tert*-butylphenyl)propane, $C_{31}H_{46}S_2$, has been published by Hiller & Rundel (1990).

References

Enraf–Nonius (1990). *MolEN. Molecular Structure Solution Package*. Enraf–Nonius, Delft, The Netherlands.

Hiller, W. & Rundel, W. (1990). *Z. Kristallogr.* **193**, 313–314.

Keller, E. (1988). *SCHAKAL*. A Fortran program for the graphic representation of molecules and crystallogrpahic models. Univ. of Freiburg, Germany.

Kwart, H. & Evans, E. R. (1966). *J. Org. Chem.* **31**, 410–412.

Newman, M. S. & Karnes, H. A. (1966). *J. Org. Chem.* **31**, 3980–3984.

Rundel, W. (1968). *Chem. Ber.* **101**, 2956–2962.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55889 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1011]