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ap-8-Chloro-1,4-dimethyl-9-(2-methylbenzyl)triptycene and *sc**(9*S**)-8-Chloro-1,4-dimethyl-9-(2-methylbenzyl)triptycene

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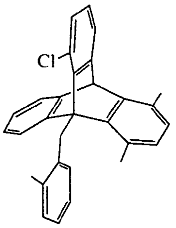
Abstract

Two atropisomers, *ap* and *sc**(9*S**), of the title compound [8-chloro-1,4-dimethyl-9-(2-methylbenzyl)-9,10-dihydro-9,10-*o*-benzenoanthracene, $C_{30}H_{25}Cl$] have been isolated and their structures determined by X-ray diffraction. The structures of the two isomers are essentially the same as those of the analogous bromo derivatives. The crystal of the *sc**(9*S**) isomer decomposed when it was heated to 493 K, owing to the *sc*–*ap* transition.

Comment

Atropisomerism is defined as stereoisomerism due to restricted rotation about a single bond where the isomers can actually be isolated (Eliel, 1962). Kinetic studies of the isomerization of 9-(2-methylbenzyl)triptycene have been reported (Yamamoto, Nemoto & Ohashi, 1992). The dynamic ^{13}C NMR study suggested that the molecule has an energy barrier of 49.6 kJ mol^{-1} for gear rotation of the 2-methylbenzyl group. The introduction of a substituent at the *peri* position was expected to raise the energy barrier enough for atropisomerism to occur. When Cl and Br atoms were introduced at the *peri* position, two stable *ap* and *sc**(9*S**) isomers were isolated for both derivatives. Classical kinetic studies in toluene-d_8 solutions revealed that the isomerization barriers at 350 K are 110.5 and 113.8 kJ mol^{-1} for the chloro and bromo derivatives, respectively. In both derivatives, the *sc**(9*S**) isomer was found to be thermally isomerized to the *ap* isomer in the solid state. The structures of the two isomers of the bromo derivative were determined by X-ray diffraction (Nemoto, Ono, Uchida, Ohashi & Yamamoto, 1994) and confirmed those assumed from the NMR study. However, the crystal of the *sc**(9*S**) isomer decomposed during the transition to the *ap* form, contrary to the assumption made from the appearance of the crystal. In order to confirm the above results, the crystal structures of the two isomers of the chloro derivative, (I), have been determined by X-ray analysis.



(I)

The crystal structure of the *ap* isomer (Fig. 1) has two crystallographically independent molecules (*A* and *B*) and is isomorphous with that of the *ap* isomer of the bromo derivative. On the other hand, the crystal of the *sc**(9*S**) isomer (Fig. 2) has a different structure from that of the bromo derivative and also from that of the *ap* isomer (Fig. 1). As observed in the bromo derivative, the crystal of the *sc**(9*S**) isomer decomposed when it was transformed to the *ap* isomer upon heating. This may suggest that the transformation will proceed with retention of the single-crystal form only if the crystals of the *sc**(9*S**) and *ap* isomers have approximately the same structure.

The molecular structures of both the *ap* and *sc**(9*S**) isomers are shown in Fig. 3; only molecule *A* of the *ap* isomer (*ap-A*) is shown, since the two crystallographically independent molecules have approximately the same conformation. The *sc**(9*S**) molecule has a disordered structure. The Cl atom bonded to C13 appeared in the refinement and is denoted as Cl2. This is a mirror image of the original Cl atom, Cl1. The ratio of Cl1 to Cl2 is 74:26. The 2-methylbenzyl group is

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disordered in two positions, C24–C30 and C242–C302, the ratio of which is 74:26. Since the Cl2 atom comes too close to the major part of the 2-methylbenzyl group, the minor part should contact with the Cl2 atom. The ratio of the disordered structure of the 2-methylbenzyl group is consistent with that of the Cl atom, and *vice versa*. The C9—C23—C24(or C242) angles are different in the two disordered structures. The fairly high *R* value for the structure determination may be due to the disorder.

The 2-methylbenzyl groups of the *ap* and *sc**(9*S**) isomers are *trans* and *gauche*, respectively, to the

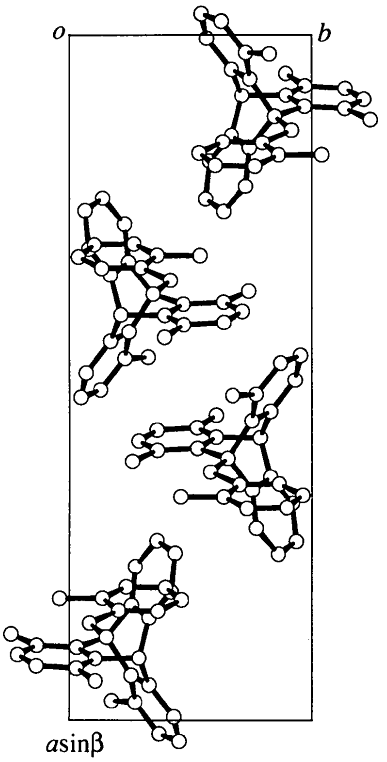

Fig. 1. The crystal structure of the *ap* isomer viewed along the *c* axis.

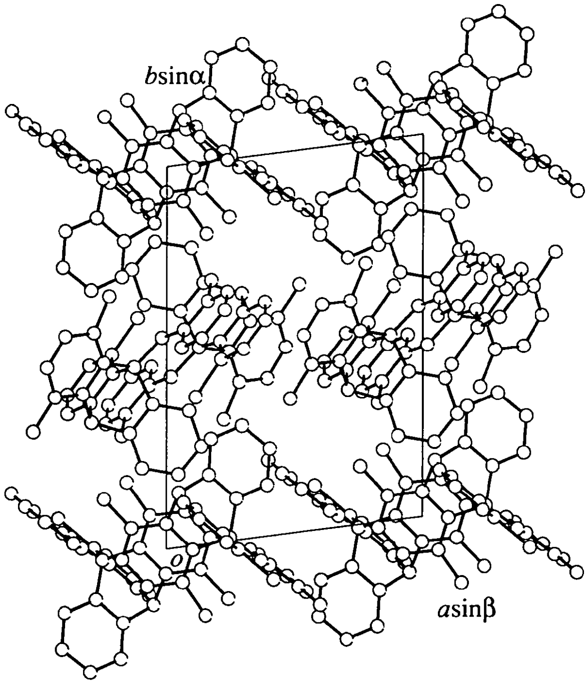

Fig. 2. The crystal structure of the *sc**(9*S**) isomer viewed along the *c* axis. The major part of the disordered structure is shown.

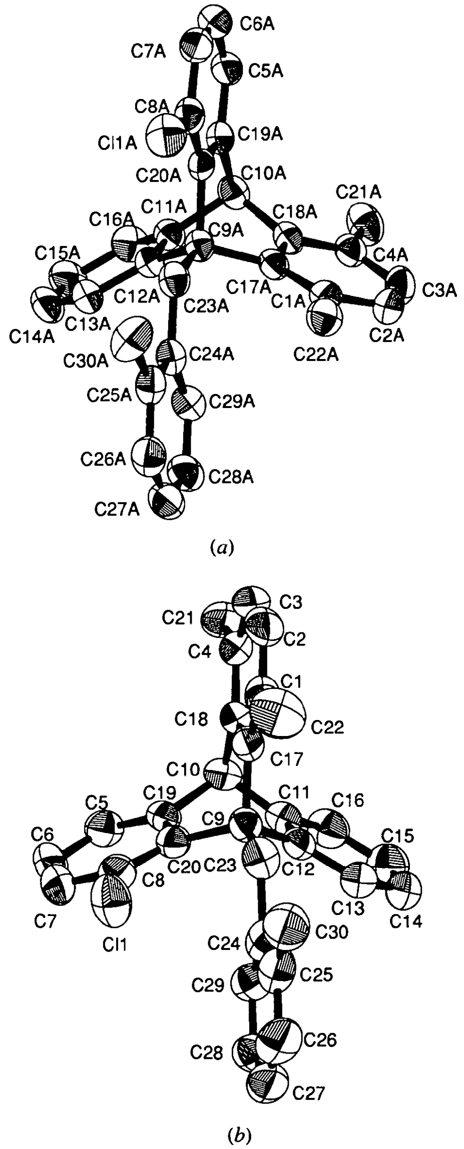

Fig. 3. (*a*) A perspective view of molecule *A* of the *ap* isomer (30% probability) with the numbering scheme. (*b*) A perspective view of a molecule of the *sc**(9*S**) isomer (30% probability) with the numbering scheme. Only the major part of the disordered structure is shown.

chlorophenyl moieties. The C20—C9—C23—C24 torsion angles are −179.3 (2), 179.2 (2) and 63.9 (7)° for the *ap-A*, *ap-B* and *sc**(9*S**) isomers, respectively. These values are similar to the corresponding values for the bromo derivative, *i.e.* 179.9 (6), 178.2 (6) and 59.4 (11)°, respectively. Bond distances and angles are in good agreement with the corresponding values for the bromo derivative.

Experimental

ap isomer

Crystal data

$C_{30}H_{25}Cl$
$M_r = 420.95$
Triclinic
$P\bar{1}$
$a = 10.0284\,(10)$ Å
$b = 15.7731\,(13)$ Å
$c = 15.6012\,(15)$ Å
$\alpha = 114.715\,(8)°$
$\beta = 102.418\,(14)°$
$\gamma = 78.189\,(9)°$
$V = 2170.9\,(3)$ Å^3
$Z = 4$
$D_x = 1.288$ Mg m^{-3}
$D_m = 1.293$ Mg m^{-3}
D_m measured by flotation in CCl_4/C_6H_{12}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10$–$15°$
$\mu = 1.650$ mm^{-1}
$T = 296\,(2)$ K
Plate-like
0.3 × 0.2 × 0.15 mm
Colorless

Data collection

Rigaku AFC-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6214 measured reflections
6214 independent reflections
5878 observed reflections $[I > 2\sigma(I)]$

$\theta_{\max} = 62.55°$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 16$
$l = 0 \rightarrow 17$
3 standard reflections monitored every 50 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0608$
$wR(F^2) = 0.2107$
$S = 1.075$
6214 reflections
565 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1553P)^2 + 0.7348P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.396$ e Å^{-3}
$\Delta\rho_{\min} = -0.549$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for the ap isomer*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl1*A*	0.11317 (10)	0.38640 (6)	0.94645 (7)	0.0644 (3)
C1*A*	0.2829 (3)	0.4117 (2)	0.6805 (2)	0.0431 (7)
C2*A*	0.3934 (3)	0.4117 (3)	0.6383 (2)	0.0551 (8)
C3*A*	0.4642 (3)	0.4875 (3)	0.6702 (3)	0.0568 (9)
C4*A*	0.4335 (3)	0.5672 (2)	0.7490 (2)	0.0450 (7)
C5*A*	0.3836 (3)	0.6141 (2)	1.0374 (2)	0.0495 (8)
C6*A*	0.3851 (4)	0.5648 (3)	1.0927 (3)	0.0599 (9)
C7*A*	0.3005 (4)	0.4969 (3)	1.0612 (2)	0.0583 (9)
C8*A*	0.2128 (3)	0.4760 (2)	0.9743 (2)	0.0481 (8)
C9*A*	0.1254 (3)	0.5159 (2)	0.8165 (2)	0.0379 (6)
C10*A*	0.2917 (3)	0.6445 (2)	0.8863 (2)	0.0430 (7)
C11*A*	0.1430 (3)	0.6858 (2)	0.8705 (2)	0.0399 (6)
C12*A*	0.0534 (3)	0.6181 (2)	0.8356 (2)	0.0375 (6)
C13*A*	−0.0874 (3)	0.6474 (2)	0.8271 (2)	0.0458 (7)
C14*A*	−0.1353 (3)	0.7423 (2)	0.8506 (2)	0.0553 (8)
C15*A*	−0.0454 (4)	0.8081 (2)	0.8821 (3)	0.0596 (9)
C16*A*	0.0955 (4)	0.7800 (2)	0.8929 (2)	0.0508 (8)
C17*A*	0.2436 (3)	0.4943 (2)	0.7558 (2)	0.0385 (6)
C18*A*	0.3253 (3)	0.5681 (2)	0.7927 (2)	0.0407 (7)
C19*A*	0.2974 (3)	0.5946 (2)	0.9517 (2)	0.0411 (7)
C20*A*	0.2088 (3)	0.5245 (2)	0.9162 (2)	0.0393 (6)
C21*A*	0.5171 (4)	0.6484 (3)	0.7870 (3)	0.0609 (9)
C22*A*	0.2236 (4)	0.3189 (2)	0.6392 (2)	0.0535 (8)
C23*A*	0.0178 (3)	0.4472 (2)	0.7757 (2)	0.0426 (7)
C24*A*	−0.0677 (3)	0.4324 (2)	0.6794 (2)	0.0435 (7)
C25*A*	−0.1596 (3)	0.3646 (2)	0.6433 (2)	0.0489 (8)
C26*A*	−0.2366 (4)	0.3470 (3)	0.5539 (3)	0.0583 (9)
C27*A*	−0.2264 (4)	0.3939 (3)	0.4983 (3)	0.0627 (10)
C28*A*	−0.1370 (4)	0.4611 (2)	0.5341 (3)	0.0558 (8)
C29*A*	−0.0585 (3)	0.4793 (2)	0.6221 (2)	0.0473 (7)
C30*A*	−0.1737 (5)	0.3111 (3)	0.7008 (3)	0.0672 (11)
Cl1*B*	−0.41997 (9)	0.11638 (6)	0.54445 (7)	0.0635 (3)
C1*B*	−0.0643 (3)	0.0938 (2)	0.8166 (2)	0.0426 (7)
C2*B*	0.0741 (3)	0.0925 (2)	0.8591 (2)	0.0519 (8)
C3*B*	0.1709 (3)	0.0146 (3)	0.8282 (2)	0.0513 (8)
C4*B*	0.1360 (3)	−0.0666 (2)	0.7520 (2)	0.0438 (7)
C5*B*	−0.0666 (4)	−0.1163 (2)	0.4617 (2)	0.0504 (8)
C6*B*	−0.1286 (4)	−0.0679 (3)	0.4047 (2)	0.0589 (9)
C7*B*	−0.2353 (4)	0.0019 (3)	0.4333 (2)	0.0594 (10)
C8*B*	−0.2822 (3)	0.0258 (2)	0.5198 (2)	0.0464 (7)
C9*B*	−0.2478 (3)	−0.0087 (2)	0.6797 (2)	0.0366 (6)
C10*B*	−0.0485 (3)	−0.1429 (2)	0.6139 (2)	0.0412 (7)
C11*B*	−0.1654 (3)	−0.1796 (2)	0.6298 (2)	0.0383 (6)
C12*B*	−0.2728 (3)	−0.1094 (2)	0.6620 (2)	0.0364 (6)
C13*B*	−0.3934 (3)	−0.1344 (2)	0.6701 (2)	0.0410 (7)
C14*B*	−0.4028 (3)	−0.2285 (2)	0.6466 (2)	0.0465 (7)
C15*B*	−0.2936 (3)	−0.2967 (2)	0.6180 (2)	0.0492 (7)
C16*B*	−0.1738 (3)	−0.2731 (2)	0.6079 (2)	0.0479 (7)
C17*B*	−0.1032 (3)	0.0107 (2)	0.7419 (2)	0.0359 (6)
C18*B*	−0.0005 (3)	−0.0657 (2)	0.7071 (2)	0.0385 (6)
C19*B*	−0.1125 (3)	−0.0935 (2)	0.5469 (2)	0.0412 (7)
C20*B*	−0.2208 (3)	−0.0216 (2)	0.5797 (2)	0.0390 (6)
C21*B*	0.2427 (3)	−0.1512 (3)	0.7178 (3)	0.0627 (9)
C22*B*	−0.1539 (4)	0.1880 (2)	0.8568 (2)	0.0504 (8)
C23*B*	−0.3712 (3)	0.0625 (2)	0.7180 (2)	0.0406 (7)
C24*B*	−0.4041 (3)	0.0772 (2)	0.8143 (2)	0.0422 (7)
C25*B*	−0.5166 (3)	0.1430 (2)	0.8494 (2)	0.0492 (8)
C26*B*	−0.5480 (4)	0.1592 (3)	0.9376 (3)	0.0649 (10)
C27*B*	−0.4713 (4)	0.1115 (3)	0.9921 (3)	0.0664 (11)
C28*B*	−0.3605 (4)	0.0463 (3)	0.9577 (3)	0.0603 (9)
C29*B*	−0.3280 (3)	0.0296 (2)	0.8700 (2)	0.0501 (8)
C30*B*	−0.6024 (4)	0.1977 (3)	0.7915 (3)	0.0718 (12)

Table 2. *Selected geometric parameters (Å, °) for the ap isomer*

C9*A*—C12*A*	1.557 (4)	C9*B*—C12*B*	1.559 (4)
C9*A*—C17*A*	1.570 (4)	C9*B*—C17*B*	1.570 (4)
C9*A*—C20*A*	1.566 (4)	C9*B*—C20*B*	1.563 (4)
C9*A*—C23*A*	1.533 (4)	C9*B*—C23*B*	1.530 (4)
C10*A*—C11*A*	1.513 (4)	C10*B*—C11*B*	1.515 (4)
C10*A*—C18*A*	1.507 (4)	C10*B*—C18*B*	1.512 (4)
C10*A*—C19*A*	1.513 (4)	C10*B*—C19*B*	1.516 (4)
C11*A*—C12*A*	1.392 (4)	C11*B*—C12*B*	1.384 (4)
C17*A*—C18*A*	1.409 (4)	C17*B*—C18*B*	1.411 (4)
C19*A*—C20*A*	1.415 (4)	C19*B*—C20*B*	1.407 (4)
C23*A*—C9*A*—C12*A*	110.1 (2)	C23*B*—C9*B*—C12*B*	109.8 (2)
C23*A*—C9*A*—C20*A*	115.6 (2)	C23*B*—C9*B*—C20*B*	116.0 (2)
C12*A*—C9*A*—C20*A*	102.9 (2)	C12*B*—C9*B*—C20*B*	102.4 (2)
C23*A*—C9*A*—C17*A*	116.5 (2)	C23*B*—C9*B*—C17*B*	116.6 (2)

C12*A*—C9*A*—C17*A*	108.8 (2)	C12*B*—C9*B*—C17*B*	108.7 (2)
C20*A*—C9*A*—C17*A*	101.9 (2)	C20*B*—C9*B*—C17*B*	102.1 (2)
C18*A*—C10*A*—C11*A*	107.7 (2)	C18*B*—C10*B*—C11*B*	107.5 (2)
C18*A*—C10*A*—C19*A*	105.6 (2)	C18*B*—C10*B*—C19*B*	105.0 (2)
C11*A*—C10*A*—C19*A*	106.0 (2)	C11*B*—C10*B*—C19*B*	106.0 (2)
C12*A*—C11*A*—C10*A*	112.4 (2)	C12*B*—C11*B*—C10*B*	112.4 (2)
C11*A*—C12*A*—C9*A*	114.5 (2)	C11*B*—C12*B*—C9*B*	114.9 (2)
C18*A*—C17*A*—C9*A*	111.7 (2)	C18*B*—C17*B*—C9*B*	111.6 (2)
C17*A*—C18*A*—C10*A*	114.3 (2)	C17*B*—C18*B*—C10*B*	114.4 (2)
C20*A*—C19*A*—C10*A*	114.2 (3)	C20*B*—C19*B*—C10*B*	114.4 (2)
C19*A*—C20*A*—C9*A*	111.7 (2)	C19*B*—C20*B*—C9*B*	112.0 (2)

*sc**(9*S**) isomer

Crystal data

$C_{30}H_{25}Cl$
$M_r = 420.95$
Monoclinic
$P2_1/a$
$a = 23.803$ (3) Å
$b = 8.4646$ (10) Å
$c = 11.083$ (2) Å
$\beta = 98.13$ (4)°
$V = 2210.5$ (5) $Å^3$
$Z = 4$
$D_x = 1.265$ Mg m^{-3}
$D_m = 1.264$ Mg m^{-3}
D_m measured by flotation in CCl_4/C_6H_{12}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 20 reflections
$\theta = 10$–15°
$\mu = 1.620$ mm^{-1}
$T = 296$ (2) K
Cubic
0.30 × 0.30 × 0.25 mm
Colorless

Data collection

Rigaku AFC-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2842 measured reflections
2772 independent reflections
2415 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.0209$
$\theta_{max} = 62.46°$
$h = 0 \rightarrow 27$
$k = 0 \rightarrow 9$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 50 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0890$
$wR(F^2) = 0.2291$
$S = 1.109$
2771 reflections
282 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0983P)^2 + 3.5398P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.005$

$\Delta\rho_{max} = 0.447$ e $Å^{-3}$
$\Delta\rho_{min} = -0.645$ e $Å^{-3}$
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0040 (7)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for the sc*(9S*) isomer*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl1†	0.97204 (9)	0.1739 (3)	0.0246 (2)	0.0807 (9)
Cl2†	0.7388 (3)	0.1555 (8)	−0.2260 (6)	0.084 (3)
C1	0.8924 (2)	−0.1366 (7)	−0.3442 (6)	0.064 (2)
C2	0.9058 (3)	−0.2143 (8)	−0.4473 (7)	0.074 (2)
C3	0.9219 (2)	−0.1382 (8)	−0.5469 (6)	0.070 (2)
C4	0.9254 (2)	0.0277 (7)	−0.5490 (5)	0.0576 (15)
C5	0.9949 (2)	0.4319 (7)	−0.3202 (5)	0.0577 (14)
C6	1.0307 (2)	0.4537 (8)	−0.2111 (5)	0.066 (2)
C7	1.0197 (2)	0.3716 (7)	−0.1093 (5)	0.064 (2)
C8	0.9739 (2)	0.2707 (7)	−0.1141 (5)	0.0554 (14)
C9	0.8804 (2)	0.1530 (6)	−0.2478 (5)	0.0487 (13)
C10	0.9107 (2)	0.2872 (6)	−0.4398 (5)	0.0499 (13)
C11	0.85233 (11)	0.3300 (4)	−0.4241 (3)	0.0477 (12)
C12	0.83391 (12)	0.2559 (4)	−0.3248 (3)	0.0461 (12)
C13	0.77788 (13)	0.2721 (4)	−0.3046 (3)	0.0608 (15)
C14	0.74025 (11)	0.3623 (5)	−0.3837 (4)	0.073 (2)
C15	0.75866 (15)	0.4364 (4)	−0.4830 (3)	0.074 (2)
C16	0.8147 (2)	0.4203 (4)	−0.5032 (3)	0.063 (2)
C17	0.8952 (2)	0.0294 (6)	−0.3455 (5)	0.0508 (13)
C18	0.9114 (2)	0.1056 (6)	−0.4479 (5)	0.0489 (13)
C19	0.9492 (2)	0.3300 (6)	−0.3246 (4)	0.0481 (13)
C20	0.9355 (2)	0.2539 (6)	−0.2202 (4)	0.0467 (12)
C21	0.9435 (3)	0.1064 (9)	−0.6579 (5)	0.077 (2)
C22	0.8760 (4)	−0.2410 (8)	−0.2438 (7)	0.100 (3)
C23	0.8603 (2)	0.0826 (7)	−0.1339 (5)	0.0596 (15)
C24†	0.8416 (4)	0.2028 (9)	−0.0373 (6)	0.059 (3)
C25†	0.8238 (4)	0.1367 (9)	0.0658 (7)	0.070 (2)
C26†	0.8072 (4)	0.2339 (11)	0.1555 (6)	0.081 (3)
C27†	0.8084 (4)	0.3971 (11)	0.1422 (6)	0.083 (3)
C28†	0.8262 (4)	0.4631 (8)	0.0392 (7)	0.080 (3)
C29†	0.8428 (5)	0.3660 (9)	−0.0505 (6)	0.067 (3)
C30†	0.8241 (5)	−0.0404 (11)	0.0884 (10)	0.088 (3)
C242†	0.8503 (14)	0.1657 (26)	−0.0347 (22)	0.047 (7)
C252†	0.8298 (14)	0.0797 (17)	0.0566 (25)	0.061 (8)
C262†	0.8074 (12)	0.1577 (22)	0.1495 (22)	0.085 (12)
C272†	0.8056 (13)	0.3218 (23)	0.1510 (22)	0.072 (11)
C282†	0.8260 (14)	0.4079 (17)	0.0596 (25)	0.065 (9)
C292†	0.8484 (13)	0.3298 (25)	−0.0332 (22)	0.048 (7)
C302†	0.8343 (10)	−0.1014 (19)	0.0620 (22)	0.056 (6)

† Disordered atom site (see *Comment*).

Table 4. *Selected geometric parameters (Å, °) for the sc*(9S*) isomer*

C9—C23	1.532 (7)	C10—C19	1.507 (7)
C9—C20	1.559 (7)	C10—C18	1.540 (7)
C9—C12	1.563 (6)	C11—C12	1.39
C9—C17	1.581 (7)	C17—C18	1.407 (7)
C10—C11	1.470 (6)	C19—C20	1.402 (7)
C23—C9—C20	113.8 (4)	C19—C10—C18	106.2 (4)
C23—C9—C12	112.8 (4)	C12—C11—C10	113.0 (3)
C20—C9—C12	108.2 (4)	C11—C12—C9	113.9 (3)
C23—C9—C17	115.6 (4)	C18—C17—C9	111.3 (5)
C20—C9—C17	103.5 (4)	C17—C18—C10	113.8 (5)
C12—C9—C17	101.7 (4)	C20—C19—C10	113.8 (4)
C11—C10—C19	108.2 (4)	C19—C20—C9	112.2 (4)
C11—C10—C18	105.8 (4)		

For both compounds, data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structures: *MITHRIL* (Gilmore, 1984) for the *ap* isomer; *SHELXS*86 (Sheldrick, 1990) for the *sc**(9*S**) isomer. For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1189). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Redetermination of 4,5-Di(benzoylthio)-4,5-didehydro-1,3-dithiolane-2-thione at 120 K

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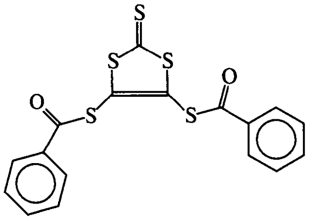
Abstract

The disorder present in the crystal structure of the title thiocarboxylic acid ester [2-oxo-1,3-dithiol-4,5-diyl bis(thiobenzoate), $C_{17}H_{10}O_2S_5$] at 288 K is absent at 120 K.

Comment

Disorder may occur in solvent molecules, methyl groups and spherical anions such as BF_4^-, PF_6^- or SO_4^{2-} by a process of free rotation (dynamic disorder) or by the occurrence of more than one position of similar energy (static disorder). Even where disorder persists on cooling, confirming the presence of static disorder, the reduction in thermal motion, possibly containing elements of dynamic disorder, can make it much easier to model the static disorder.

The 4,5-dimercapto-1,3-dithiole-2-thione (dmit) ligand has been widely used in the synthesis of metal complexes, including molecular inorganic superconductors derived from transition metal dmit complexes (Cassoux & Valade, 1992). The rather unstable dmit ligand can be stabilized by preparation of the title compound, (dmit[COPh]$_2$; Steimecke, 1979), (I). Any variation in molecular conformation of this compound with temperature would be particularly relevant to conductivity studies. At 288 K, a single oxygen (O7) in the compound is reported to be disordered equally over three positions (Solans, Font-Bardía, Font-Altaba, Vicente & Seguí, 1987). The current low-temperature study shows a single position for this atom but confirms it is the most thermally active atom in the molecule; the chemically equivalent O15 atom is the second most thermally active site. At 120 K, the C7—O7 bond length of 1.202 (6) Å is normal, while at 288 K three bond lengths of 1.241 (20), 1.268 (52) and 1.299 (30) Å correspond to the three disordered O-atom positions. There are no significant differences between other bond lengths at 120 and 288 K, and the different values for some chemically equivalent bonds and angles are reproduced in this study.



(I)

At 120 K, valence angles about C7 are similar to those about C15. As expected, there is a small decrease in cell volume [from 1796 (1) to 1738 (2) Å^3] on lowering the temperature and a corresponding increase in

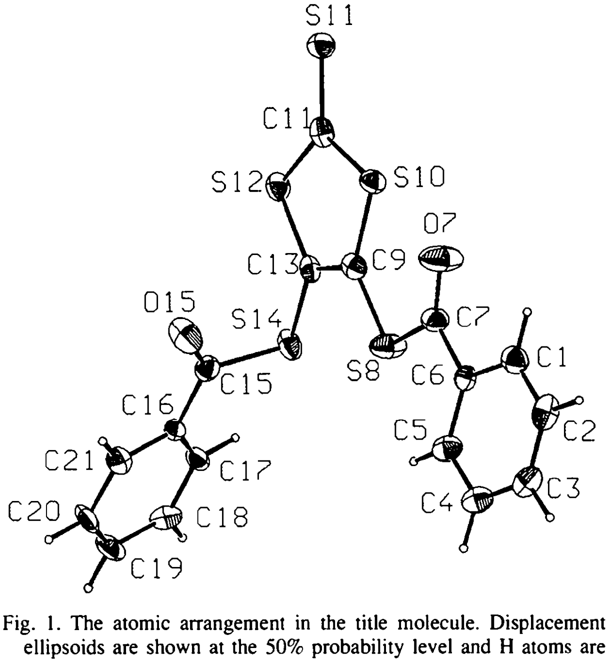


Fig. 1. The atomic arrangement in the title molecule. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary radii.