# *threo*-1,2-Dibromo-1-triphenylsilyl-2-phenylethane

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**Abstract.** $C_{26}H_{22}Br_2Si$, $M_r = 522 \cdot 4$, triclinic, $A\bar{1}$,* $a = 15 \cdot 828$ (2), $b = 28 \cdot 237$ (3), $c = 10 \cdot 508$ (2) Å, $\alpha = 89 \cdot 86$ (2), $\beta = 84 \cdot 09$ (2), $\gamma = 81 \cdot 20$ (2)°, $U = 4616 \cdot 0$ Å$^3$, $Z = 8$, $D_c = 1 \cdot 50$ g cm$^{-3}$, $F(000) = 2096$. Cu $K\alpha$ radiation, $\mu = 54 \cdot 8$ cm$^{-1}$. $R = 0 \cdot 062$ for 2210 counter reflections. The two independent molecules have enantiomeric *threo* configurations with the Br atoms adopting a *gauche* conformation.

**Introduction.** The title compound is formed by the addition of bromine to *trans*-triphenylsilylstyrene. The structure was determined to deduce the stereo-

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* The reduced primitive cell has $a = 10 \cdot 508$, $b = 15 \cdot 052$, $c = 15 \cdot 828$ Å, $\alpha = 96 \cdot 18$, $\beta = 95 \cdot 91$, $\gamma = 110 \cdot 29°$ and can be obtained by the matrix transformation $(0,0,1/0,0 \cdot 5,-0 \cdot 5/-1,0,0)$.

chemistry of the addition. A preliminary account has been published (Brook, Duff, Hitchcock & Mason, 1976).

A crystal $0 \cdot 5 \times 0 \cdot 2 \times 0 \cdot 1$ mm was used for data collection on a Hilger & Watts Y290 four-circle diffractometer. Cell dimensions were calculated from the setting angles for 12 reflections $[\lambda(\text{Cu } K\alpha_1) = 1 \cdot 5405$ Å]. Intensities for unique reflections with $2 < \theta < 40°$ were collected by an $\omega/2\theta$ step scan with Cu $K\alpha$ radiation and a graphite-crystal monochromator. Three standard reflections remeasured after every 100 reflections showed no significant changes. The data were corrected for Lorentz and polarization effects and also for absorption with *ABSCOR* in the XRAY system (Stewart, 1972). 2210 reflections with $I > 3\sigma(I)$ were used in the analysis.

Table 1. *Atomic fractional coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

| | $x$ | $y$ | $z$ | | $x$ | $y$ | $z$ |
|---|---|---|---|---|---|---|---|
| Br(1) | 1849·4 (13) | 4490·3 (7) | 4223·4 (15) | C(24) | 1953 (10) | 5394 (6) | 11426 (15) |
| Br(2) | 0656·3 (11) | 4917·5 (6) | 7093·5 (18) | C(25) | 2509 (10) | 5581 (5) | 10614 (15) |
| Br(3) | 3253·6 (12) | 3149·1 (6) | 3756·7 (15) | C(26) | 2697 (9) | 5386 (5) | 9373 (14) |
| Br(4) | 4541·1 (10) | 2748·2 (6) | 0870·3 (15) | C(27) | 3458 (8) | 3177 (4) | 1017 (11) |
| Si(1) | 2639 (2) | 4744 (1) | 7320 (3) | C(28) | 3334 (8) | 3520 (5) | 2132 (13) |
| Si(2) | 2515 (3) | 2860 (1) | 0663 (3) | C(29) | 3969 (8) | 3862 (4) | 2218 (12) |
| C(1) | 1675 (8) | 4448 (4) | 6955 (11) | C(30) | 4764 (9) | 3739 (5) | 2657 (13) |
| C(2) | 1760 (9) | 4114 (5) | 5809 (13) | C(31) | 5298 (10) | 4087 (6) | 2731 (14) |
| C(3) | 1097 (8) | 3784 (5) | 5769 (12) | C(32) | 5060 (10) | 4533 (6) | 2278 (14) |
| C(4) | 1317 (9) | 3328 (5) | 6052 (14) | C(33) | 4286 (10) | 4656 (5) | 1821 (14) |
| C(5) | 0720 (13) | 2990 (7) | 6050 (18) | C(34) | 3733 (8) | 4313 (5) | 1818 (12) |
| C(6) | −0070 (14) | 3176 (8) | 5812 (20) | C(35) | 1547 (8) | 3315 (5) | 0676 (12) |
| C(7) | −0330 (13) | 3630 (8) | 5514 (19) | C(36) | 1157 (9) | 3448 (5) | −0467 (13) |
| C(8) | 0258 (11) | 3962 (6) | 5508 (15) | C(37) | 0411 (9) | 3777 (5) | −0484 (13) |
| C(9) | 3601 (8) | 4277 (4) | 7309 (12) | C(38) | 0045 (9) | 4011 (5) | 0634 (14) |
| C(10) | 4029 (9) | 4194 (5) | 8444 (13) | C(39) | 0399 (9) | 3888 (5) | 1754 (13) |
| C(11) | 4742 (9) | 3843 (5) | 8429 (14) | C(40) | 1130 (8) | 3553 (4) | 1761 (12) |
| C(12) | 5055 (9) | 3567 (5) | 7395 (15) | C(41) | 2796 (8) | 2600 (4) | −1015 (12) |
| C(13) | 4665 (9) | 3637 (5) | 6293 (13) | C(42) | 2373 (9) | 2238 (5) | −1387 (14) |
| C(14) | 3949 (8) | 3987 (5) | 6248 (12) | C(43) | 2503 (10) | 2069 (5) | −2658 (15) |
| C(15) | 2863 (8) | 5235 (4) | 6196 (12) | C(44) | 3055 (9) | 2243 (5) | −3474 (14) |
| C(16) | 3534 (9) | 5163 (5) | 5258 (13) | C(45) | 3488 (9) | 2593 (5) | −3150 (14) |
| C(17) | 3711 (9) | 5555 (5) | 4430 (13) | C(46) | 3352 (9) | 2777 (5) | −1894 (13) |
| C(18) | 3168 (9) | 5983 (5) | 4587 (13) | C(47) | 2269 (8) | 2385 (5) | 1837 (12) |
| C(19) | 2519 (9) | 6052 (5) | 5510 (14) | C(48) | 1429 (10) | 2352 (5) | 2161 (14) |
| C(20) | 2341 (8) | 5682 (5) | 6336 (12) | C(49) | 1188 (11) | 1993 (6) | 3092 (16) |
| C(21) | 2334 (8) | 5009 (5) | 8970 (12) | C(50) | 1819 (10) | 1707 (5) | 3608 (14) |
| C(22) | 1780 (9) | 4815 (5) | 9870 (13) | C(51) | 2650 (9) | 1743 (5) | 3293 (13) |
| C(23) | 1587 (9) | 5018 (5) | 11106 (14) | C(52) | 2888 (9) | 2081 (5) | 2383 (13) |

The Br atoms were located by direct methods and the Si and C atoms on successive difference maps. During full-matrix least-squares refinement (Br and Si anisotropic) the two independent molecules were refined in alternate cycles to reduce any effects of the pseudo symmetry centre relating them. H atoms were included at idealized positions (C—H = 1·08 Å) with $U = 0.063$ Å$^2$ and not refined. The weighting scheme was $w = 1/\{1 + [(F_o - 40)/96)]^2\}$. The refinement converged at $R = 0.062$, $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.078$ with a maximum shift to error of 0·07. Scattering factors for neutral atoms were from Cromer & Waber (1965), except those for H which were from Stewart, Davidson & Simpson (1965). Dispersion corrections for Br and Si were from Cromer (1965).

The final atom positions are listed in Table 1.* The structure refinement was done with the XRAY system (Stewart, 1972).

**Discussion.** The two independent molecules are shown in Fig. 1 with relevant molecular parameters listed in Table 2. The two molecules have enantiomeric configurations and both are derived by the unexpected *syn* addition of bromine to *trans*-triphenylsilylstyrene. This has been rationalized by Brook, Duff & Reynolds (1976) in terms of steric effects on the thermodynamics of *syn* and *anti* addition.

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Table 2. *Bond lengths and angles with e.s.d.'s in parentheses and selected torsion angles for the two independent molecules*

(a) Bonds (Å)

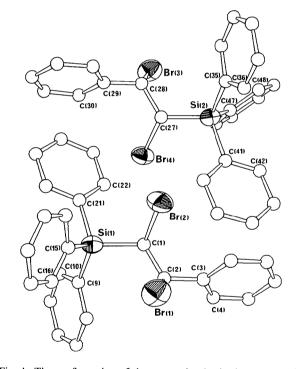| Molecule A | | Molecule B | |
|---|---|---|---|
| Br(1)—C(2) | 1·978 (14) | Br(3)—C(28) | 2·004 (13) |
| Br(2)—C(1) | 1·918 (11) | Br(4)—C(27) | 1·934 (11) |
| Si(1)—C(1) | 1·920 (14) | Si(2)—C(27) | 1·922 (14) |
| Si(1)—C(9) | 1·853 (12) | Si(2)—C(35) | 1·841 (12) |
| Si(1)—C(15) | 1·870 (13) | Si(2)—C(47) | 1·878 (13) |
| Si(1)—C(21) | 1·877 (13) | Si(2)—C(41) | 1·896 (13) |
| C(1)—C(2) | 1·514 (18) | C(27)—C(28) | 1·500 (18) |
| C(2)—C(3) | 1·509 (21) | C(28)—C(29) | 1·507 (20) |
| Phenyl rings | | Phenyl rings | |
| C—C max. | 1·450 (20) | C—C max. | 1·471 (23) |
| C—C min. | 1·325 (20) | C—C min. | 1·310 (21) |
| C—C average | 1·380 | C—C average | 1·378 |

(b) Angles (°)

| Molecule A | | Molecule B | |
|---|---|---|---|
| Br(1)—C(2)—C(1) | 109·2 (9) | Br(3)—C(28)—C(27) | 108·9 (9) |
| Br(1)—C(2)—C(3) | 111·1 (9) | Br(3)—C(28)—C(29) | 109·1 (9) |
| Br(2)—C(1)—C(2) | 114·1 (9) | Br(4)—C(27)—C(28) | 114·4 (9) |
| Br(2)—C(1)—Si(1) | 109·6 (6) | Br(4)—C(27)—Si(2) | 112·4 (6) |
| C(1)—Si(1)—C(9) | 109·0 (6) | C(27)—Si(2)—C(35) | 108·2 (6) |
| C(1)—Si(1)—C(15) | 113·6 (6) | C(27)—Si(2)—C(47) | 114·7 (6) |
| C(1)—Si(1)—C(21) | 104·9 (6) | C(27)—Si(2)—C(41) | 106·2 (6) |
| C(9)—Si(1)—C(15) | 110·3 (5) | C(35)—Si(2)—C(47) | 107·9 (6) |
| C(9)—Si(1)—C(21) | 110·3 (6) | C(35)—Si(2)—C(41) | 108·6 (6) |
| C(15)—Si(1)—C(21) | 108·5 (6) | C(47)—Si(2)—C(41) | 111·0 (6) |
| Phenyl rings | | Phenyl rings | |
| C—C—C max. | 126·0 (2) | C—C—C max. | 123·3 (2) |
| C—C—C min. | 115·0 (2) | C—C—C min. | 114·1 (2) |
| C—C—C average | 120·0 | C—C—C average | 120·0 |



Fig. 1. The configuration of the two molecules in the asymmetric unit. Carbon atoms are labelled consecutively around the phenyl rings.

Table 2 (*cont.*)

(c) Torsion angles (°)

| | | | |
|---|---|---|---|
| Br(1)—C(2)—C(1)—Br(2) | 63 | Br(3)—C(28)—C(27)—Br(4) | −66 |
| C(3)—C(2)—C(1)—Br(2) | −65 | C(29)—C(28)—C(27)—Br(4) | 59 |
| Br(2)—C(1)—Si(1)—C(9) | 176 | Br(4)—C(27)—Si(2)—C(35) | −174 |
| Br(2)—C(1)—Si(1)—C(15) | −61 | Br(4)—C(27)—Si(2)—C(47) | 65 |
| Br(2)—C(1)—Si(1)—C(21) | 58 | Br(4)—C(27)—Si(2)—C(41) | −58 |
| C(1)—Si(1)—C(9)—C(10) | −119 | C(27)—Si(2)—C(35)—C(36) | 111 |
| C(1)—Si(1)—C(15)—C(16) | −104 | C(27)—Si(2)—C(47)—C(48) | 141 |
| C(1)—Si(1)—C(21)—C(22) | 29 | C(27)—Si(2)—C(41)—C(46) | −24 |

The two independent molecules are related by a pseudo inversion centre which exactly relates the Br and Si atoms and nearly relates the remainder of the molecules. The main exception is the orientations of the phenyl rings C(15) → (20) and C(47) → (52) which differ by 37°. This is obvious from the torsion angles C(1)–Si(1)–C(15)–C(16) and C(27)–Si(2)–C(47)–C(48) which are −104 and 141° respectively. The equivalent bond lengths and angles in the two molecules are equal within experimental error. Br(2)–C(1) and Br(4)–C(27), average 1·93 (1) Å, are systematically shorter than Br(1)–C(2) and Br(3)–C(28), average 1·99 (1) Å. For comparison, the C–Br lengths in hexabromoethane average 1·944 (5) Å (Mandel & Donohue, 1972), but quite a wide variation in C–Br lengths is found in other structures. A value of 1·909 (12) Å occurs in (4-bromo-1,2,3,4-tetraphenyl-*cis*-1,3-butadienyl)dimethylphenyltin (Boer, van Remoortere, North & Reeke, 1971), and 2·00 (3) Å in (+)-1,2-dibromo-2-phenethyl *p*-tolyl sulphoxide (Iwasaki, Mitamura & Tsuchihashi, 1975). In 3-bromo-2,2-diphenyl-2-sila-*Δ*3-1-tetralone (Vidal, Galigné & Falgueirettes, 1972) where a C atom is bonded to both Br

and Si, the C–Br length is 1·911 (5) Å, although in this case the C atom is trigonally hybridized.

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# Bis(triphenylmethyl) Ether

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**Abstract.** $C_{38}H_{30}O$, monoclinic, $P2_1/c$, $a = 14\cdot791$ (2), $b = 10\cdot598$ (2), $c = 17\cdot521$ (3) Å, $\beta = 96\cdot528$ (12)°; $M_r = 502\cdot62$; $D_c = 1\cdot223$, $D_m = 1\cdot220$ g cm$^{-3}$; $Z = 4$; $\mu$(Mo $K\alpha$) $= 0\cdot37$ cm$^{-1}$. The final residual from 3020 observed reflections was 0·041. The COC angle is 127·9 (1)°, and the C–O bond distances are 1·454 (2) and 1·465 (2) Å.

**Introduction.** The compound was prepared by reaction of chlorotriphenylmethane with mercury(II) oxide (Gomberg, 1913) and crystals were grown from toluene solution. The space group was assigned uniquely from the systematic absences: $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$. A crystal in the shape of a thick plate of approximate dimensions 0·5 × 0·32 × 0·13 mm was used for data collection. Data were collected on a Philips PW 1100 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions were

derived from least-squares fits to the reflecting positions of 25 reflections each measured 15 times during data collection. The intensities of 5279 reflections within the quadrant $\pm h$, $+k$, $+l$, with $3 \le \theta \le 25°$ were measured using the $\omega$–$2\theta$ scan technique with a scan speed of 0·05° s$^{-1}$ in $\omega$ and a scan width of $(0\cdot9 + 0\cdot1 \tan \theta)°$ in $\omega$. Backgrounds were measured at both ends of each scan. Three reference reflections were measured every 6 h during data collection and showed only small random deviations about their mean intensities. Lorentz and polarization corrections were applied to the data, but no corrections for absorption were made.

The structure was solved with the fast, centrosymmetric direct-methods program in the *SHELX* system (Sheldrick, 1976) and refined by blocked-full-matrix least squares with complex neutral-atom scattering factors (Cromer & Mann, 1968) and a weighting scheme $w = 1/\sigma^2(F)$, with 3020 unique reflections