0·009 (1). There were 72 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(\Delta/\sigma)_{max} = 0·01$, $R = 0·037$, $wR = 0·045$, $S = 2·23$, final difference Fourier map excursions 0·35 and $-0·37$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1–4, atom coordinates, bond distances and bond angles for molecules (I) and (II) respectively, follows that shown in Figs. 1 and 2.

Related literature. Compound (I) is the monoketo analog of the commercial energetic compound RDX (Choi & Prince, 1972). It can also be compared

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52363 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

to the structure of 1,3,5-tris(dimethylamino)hexa-hydro-1,3,5-triazine-2,4,6-trione (Zurn, Schwarz, Rozdzinski & Schmidt, 1982). A search of the current release of the Cambridge Structural Database did not produce any compounds similar to (II).

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Structures of Two Diastereomers of the Tetrahydrobenzothiophene Moiety of Breynolide

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Abstract. (1) ($\pm$)-3-Triisopropylsiloxy-2,3,3a,7a-tetrahydrobenzo[b]thiophen-5(4H)-one 1,1-dioxide, $C_{17}H_{30}O_4SSi$, $M_r = 358·57$, triclinic, $P\bar{1}$, $a = 7·4383 (14)$, $b = 8·1000 (15)$, $c = 16·895 (3)$ Å, $\alpha = 80·202 (14)$, $\beta = 88·800 (15)$, $\gamma = 76·231 (14)°$, $V = 974·0 (3)$ Å^3, $Z = 2$, $D_x = 1·22$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 2·339$ cm^{-1}, $F(000) = 388$, $T = 298$ K, $R = 0·0436$ for 3086 reflections $[F_o \geq 4\sigma(F_o)]$. (2) ($\pm$)-3-*tert*-Butyldiphenylsiloxy-2,3,3a,7a-tetrahydrobenzo[b]thiophen-5(4H)-one 1,1-dioxide, $C_{24}H_{28}O_4SSi$, $M_r = 440·63$, triclinic, $P\bar{1}$, $a = 9·113 (2)$, $b = 11·020 (2)$, $c = 12·551 (3)$ Å, $\alpha = 74·01 (2)$, $\beta = 70·83 (2)$, $\gamma = 88·70 (2)°$, $V = 1141·4 (4)$ Å^3, $Z = 2$, $D_x = 1·28$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 2·125$ cm^{-1}, $F(000) = 468$, $T = 298$ K, $R = 0·0424$ for 4262 reflections $[F_o \geq 4\sigma(F_o)]$. The two rings are *cis*-fused with the cyclohexenone ring in the sofa conformation and the tetrahydrothiophene ring in the half-chair conformation for both (1) and (2). However, the bulky siloxy groups assume different orientations in the two compounds. In (1),

the triisopropylsiloxy group is equatorial and close intramolecular contacts between H2A and H4B [2·29 (3) Å], O11 and C4 [2·934 (3) Å] and O9 and C7 [2·979 (3) Å] are observed. In (2), the *tert*-butyldiphenylsiloxy group is axial and close contacts arise between O11 and C4 [2·864 (3) Å], O11 and C5 [2·930 (3) Å] and O9 and C7 [2·892 (3) Å].

Experimental. Compounds (1) and (2) were prepared *via* an intermolecular Diels–Alder reaction of *trans*-1-methoxy-3-trimethylsilyloxy-1,3-butadiene (Danishefski & Kitahara, 1974) and the corresponding protected allylic alcohol obtained from 2,5-
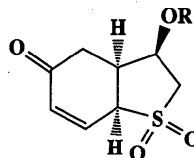
(1) R = triisopropylsilyl
(2) R = *tert*-butyldiphenylsilyl

Table 1. *Data-collection and structure-refinement details*

	(1)	(2)
Mode		ω scan
Scan range	Symmetrically over 1° about $K\alpha_{1,2}$ maximum	
Background	Measured at ends of scan	
Scan rate (° min^{-1})	3–6	4–8
Max. decay correction on I	< 3%	< 8%
2θ range (°)	4·0–55·0	4·0–50·0
Standard reflections	$\bar{2}18$, 141, $1\bar{2}9$, 133	$2\bar{3}3$, 261, $34\bar{1}$, $\bar{1}3\bar{3}$
Reflections measured	5479	5540
Unique reflections	4163	5243
R_{int}	0·0120	0·0130
Range of h,k,l	$-2\rightarrow9$ $-10\rightarrow10$ $-21\rightarrow21$	$0\rightarrow-11$ $-14\rightarrow14$ $-16\rightarrow16$
Reflections used, $F > 4\sigma(F_o)$	3086	4262
Reflections omitted	1077	981
No. of variables	328	383
R, wR	0·0436, 0·0473	0·0424, 0·0540
R_{all}, wR_{all}	0·0642, 0·0507	0·0531, 0·0556
Goodness of fit, S	1·445	1·865
Max. $|\Delta/\sigma|$	< 0·1	< 0·1
Min., max. peak in ΔF map (e Å^{-3})	$-0·34, 0·21$	$-0·37, 0·32$

The lattice parameters were obtained from the least-squares refinement of 49 reflections with $20·2 < 2\theta < 28·9°$ for (1) and of 50 reflections with $23·1 < 2\theta < 30·0°$ for (2).

Table 2. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for non-H atoms of* (1)

$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i{}^* a_j{}^* \mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

	x	y	z	U_{eq}
S1	0·79906 (9)	$-0·18444$ (7)	0·46833 (3)	0·0413 (2)
C2	0·7710 (4)	0·0175 (3)	0·40049 (14)	0·0402 (9)
C3	0·7909 (3)	$-0·0326$ (3)	0·31680 (12)	0·0320 (7)
C3a	0·6957 (3)	$-0·1810$ (3)	0·31841 (13)	0·0313 (7)
C4	0·4843 (3)	$-0·1271$ (3)	0·3228 (2)	0·0407 (8)
C5	0·3944 (3)	$-0·2766$ (3)	0·34314 (14)	0·0418 (8)
C6	0·5019 (4)	$-0·4338$ (3)	0·39332 (14)	0·0456 (9)
C7	0·6743 (4)	$-0·4490$ (3)	0·41811 (14)	0·0434 (9)
C7a	0·7802 (3)	$-0·3145$ (3)	0·39181 (13)	0·0340 (7)
O8	0·2368 (2)	$-0·2657$ (2)	0·32158 (12)	0·0648 (8)
O9	0·6460 (3)	$-0·1815$ (2)	0·52186 (10)	0·0613 (7)
O10	0·9835 (3)	$-0·2367$ (2)	0·50276 (11)	0·0667 (8)
O11	0·7130 (2)	0·1107 (2)	0·25649 (8)	0·0359 (5)
Si12	0·82792 (8)	0·18351 (7)	0·17783 (3)	0·0319 (2)
C13	0·8786 (4)	0·0267 (3)	0·10460 (15)	0·0450 (9)
C14	1·0211 (5)	$-0·1412$ (4)	0·1342 (2)	0·0636 (13)
C15	0·7037 (6)	$-0·0116$ (5)	0·0739 (3)	0·073 (2)
C16	0·6593 (3)	0·3839 (3)	0·12737 (14)	0·0412 (8)
C17	0·5652 (6)	0·5046 (4)	0·1837 (2)	0·0688 (13)
C18	0·7386 (6)	0·4797 (4)	0·0544 (2)	0·0682 (13)
C19	1·0520 (3)	0·2132 (3)	0·21587 (14)	0·0446 (9)
C20	1·1902 (5)	0·2483 (6)	0·1511 (3)	0·076 (2)
C21	1·0283 (6)	0·3451 (5)	0·2732 (2)	0·072 (2)

dihydrothiophene 1,1-dioxide. A detailed account of the syntheses of these compounds will be described elsewhere (Martin & Daniel, 1989). The data crystal for (1) was a colorless plate of approximate dimensions $0·12 \times 0·30 \times 0·32$ mm. The data crystal for (2) was a colorless block that was cut from a larger crystal and had approximate dimensions $0·49 \times 0·56 \times 0·70$ mm. Crystals of both were obtained by slow evaporation from ethyl acetate. The data for both were collected at room temperature on a Nicolet *P*3 diffractometer using a graphite monochromator. Instrument and crystal stability were monitored by the remeasurement of four check reflections throughout the course of data collection. The data were corrected for decay and Lp effects but not for absorption. Data reduction and decay correction were performed using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1987). Details of data collection and structure refinement are listed in Table 1. The structures were solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976) with anisotropic thermal parameters for the non-H atoms. The H-atom positions were obtained from a ΔF map. The function $\Sigma w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0·5kI^{-1/2}\{[\sigma(I)]^2 + (0·02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{peak} - I_{background}) \times$ (scan rate), where 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics: $\sigma(I) = [(I_{peak} + I_{background})^{1/2} \times$ (scan rate)]. The scattering

Table 3. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for non-H atoms of* (2)

$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i{}^* a_j{}^* \mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

	x	y	z	U_{eq}
S1	0·05317 (7)	0·22070 (5)	0·00138 (4)	0·0551 (2)
C2	$-0·0442$ (3)	0·2132 (3)	0·1527 (2)	0·0574 (10)
C3	0·0544 (2)	0·1389 (2)	0·22194 (15)	0·0387 (6)
C3a	0·1342 (2)	0·0431 (2)	0·15667 (15)	0·0425 (7)
C4	0·2602 (3)	$-0·0236$ (2)	0·2005 (2)	0·0550 (9)
C5	0·4127 (3)	0·0519 (2)	0·1570 (2)	0·0579 (9)
C6	0·4553 (3)	0·1463 (2)	0·0413 (2)	0·0562 (9)
C7	0·3580 (3)	0·1728 (2)	$-0·0183$ (2)	0·0553 (8)
C7a	0·1976 (3)	0·1107 (2)	0·0243 (2)	0·0469 (7)
O8	0·5037 (2)	0·0323 (2)	0·2115 (2)	0·0925 (10)
O9	0·1269 (3)	0·3436 (2)	$-0·0659$ (2)	0·0838 (9)
O10	$-0·0509$ (2)	0·1665 (2)	$-0·0404$ (2)	0·0775 (9)
O11	0·16820 (13)	0·21882 (11)	0·22938 (10)	0·0380 (4)
Si12	0·13921 (5)	0·31090 (4)	0·31845 (4)	0·0318 (2)
C13	$-0·0735$ (2)	0·3381 (2)	0·37314 (14)	0·0345 (6)
C14	$-0·1716$ (2)	0·2433 (2)	0·4680 (2)	0·0439 (7)
C15	$-0·3314$ (2)	0·2526 (2)	0·5110 (2)	0·0536 (8)
C16	$-0·3957$ (2)	0·3573 (2)	0·4605 (2)	0·0582 (10)
C17	$-0·3026$ (3)	0·4518 (2)	0·3681 (2)	0·0585 (10)
C18	$-0·1423$ (2)	0·4424 (2)	0·3245 (2)	0·0459 (7)
C19	0·1875 (2)	0·2284 (2)	0·45369 (15)	0·0359 (6)
C20	0·2734 (2)	0·1221 (2)	0·4642 (2)	0·0436 (7)
C21	0·3068 (2)	0·0677 (2)	0·5668 (2)	0·0512 (8)
C22	0·2566 (2)	0·1186 (2)	0·6596 (2)	0·0493 (8)
C23	0·1715 (2)	0·2237 (2)	0·6521 (2)	0·0485 (8)
C24	0·1371 (2)	0·2772 (2)	0·5503 (2)	0·0426 (7)
C25	0·2774 (2)	0·4548 (2)	0·2284 (2)	0·0458 (7)
C26	0·2637 (4)	0·5522 (3)	0·2966 (3)	0·0757 (13)
C27	0·4432 (3)	0·4093 (3)	0·2017 (2)	0·0650 (10)
C28	0·2506 (4)	0·5148 (3)	0·1112 (2)	0·0705 (11)

factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from Cromer & Liberman (1970). The scattering factors for the H atoms

Table 4. *Bond lengths (Å) and angles (°) for non-H atoms of (1)*

1	2	3	1—2	1—2—3
C2	S1	C7a	1·801 (2)	96·28 (11)
C2	S1	O9		111·16 (11)
C7a	S1	O9	1·828 (2)	109·77 (12)
C7a	S1	O10		109·51 (11)
O9	S1	O10	1·437 (2)	118·07 (11)
O10	S1	C2	1·440 (2)	109·86 (13)
C3	C2	S1	1·531 (3)	104·5 (2)
C3a	C3	O11	1·530 (3)	111·9 (2)
C3a	C3	C2		106·8 (2)
O11	C3	C2	1·422 (2)	111·2 (2)
C4	C3a	C7a	1·532 (3)	111·4 (2)
C4	C3a	C3		114·0 (2)
C7a	C3a	C3	1·535 (3)	105·2 (2)
C5	C4	C3a	1·505 (4)	113·8 (2)
C6	C5	O8	1·468 (3)	121·1 (2)
C6	C5	C4		117·4 (2)
O8	C5	C4	1·214 (3)	121·4 (2)
C7	C6	C5	1·329 (4)	122·4 (2)
C7a	C7	C6	1·494 (4)	123·0 (2)
S1	C7a	C3a		104·00 (14)
S1	C7a	C7		113·7 (2)
C3a	C7a	C7		114·4 (2)
Si12	O11	C3	1·665 (2)	124·21 (13)
C13	Si12	C16	1·891 (3)	107·80 (11)
C13	Si12	C19		109·44 (12)
C13	Si12	O11		111·26 (10)
C16	Si12	C19	1·881 (2)	116·50 (12)
C16	Si12	O11		103·66 (9)
C19	Si12	O11	1·881 (3)	108·06 (9)
C14	C13	C15	1·526 (4)	110·2 (3)
C14	C13	Si12		115·1 (2)
C15	C13	Si12	1·527 (5)	112·8 (2)
C17	C16	C18	1·521 (4)	112·0 (2)
C17	C16	Si12		114·8 (2)
C18	C16	Si12	1·528 (4)	113·3 (2)
C20	C19	C21	1·524 (5)	110·4 (3)
C20	C19	Si12		115·0 (2)
C21	C19	Si12	1·538 (5)	114·2 (2)

Table 5 (*cont.*)

1	2	3	1—2	1—2—3
C14	C13	C18	1·402 (2)	117·3 (2)
C14	C13	Si12		117·52 (13)
C18	C13	Si12	1·385 (3)	125·18 (11)
C15	C14	C13	1·389 (3)	121·6 (2)
C16	C15	C14	1·374 (3)	119·6 (2)
C17	C16	C15	1·370 (3)	120·1 (2)
C18	C17	C16	1·394 (3)	120·3 (2)
C13	C18	C17		121·1 (2)
C20	C19	C24	1·395 (3)	117·1 (2)
C20	C19	Si12		124·5 (2)
C24	C19	Si12	1·397 (3)	118·44 (14)
C21	C20	C19	1·390 (3)	121·0 (2)
C22	C21	C20	1·371 (3)	120·4 (2)
C23	C22	C21	1·378 (3)	120·1 (2)
C24	C23	C22	1·384 (3)	119·5 (2)
C19	C24	C23		121·9 (2)
C26	C25	C27	1·527 (4)	108·7 (2)
C26	C25	C28		110·3 (2)
C26	C25	Si12		111·29 (14)
C27	C25	C28	1·541 (3)	108·3 (2)
C27	C25	Si12		106·76 (14)
C28	C25	Si12	1·533 (4)	111·3 (2)

Table 5. *Bond lengths (Å) and angles (°) for non-H atoms of (2)*

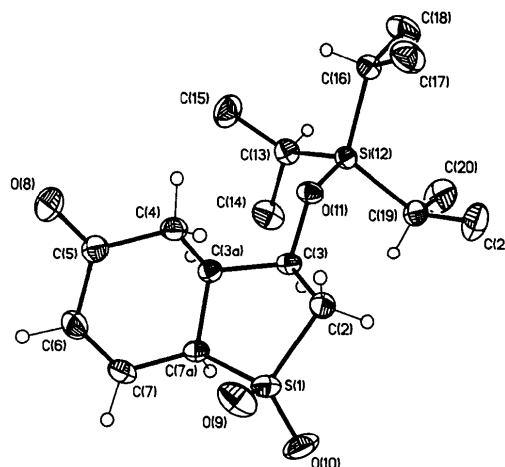| 1	2	3	1—2	1—2—3
C2	S1	C7a	1·792 (3)	97·15 (11)
C2	S1	O10		108·84 (12)
C7a	S1	O10	1·807 (2)	109·27 (12)
C7a	S1	O9		110·20 (12)
O10	S1	O9	1·434 (3)	117·70 (13)
O9	S1	C2	1·427 (2)	111·72 (13)
C3	C2	S1	1·523 (3)	106·9 (2)
C3a	C3	O11	1·538 (3)	110·0 (2)
C3a	C3	C2		106·6 (2)
O11	C3	C2	1·418 (3)	111·9 (2)
C4	C3a	C7a	1·522 (3)	111·6 (2)
C4	C3a	C3		113·9 (2)
C7a	C3a	C3	1·535 (2)	108·69 (14)
C5	C4	C3a	1·491 (3)	115·1 (2)
C6	C5	O8	1·469 (3)	120·3 (2)
C6	C5	C4		117·7 (2)
O8	C5	C4	1·220 (4)	121·9 (2)
C7	C6	C5	1·318 (4)	121·9 (2)
C7a	C7	C6	1·491 (3)	123·6 (2)
S1	C7a	C3a		103·84 (13)
S1	C7a	C7		113·18 (14)
C3a	C7a	C7		114·7 (2)
Si12	O11	C3	1·664 (2)	126·78 (10)
C13	Si12	C19	1·880 (2)	105·31 (8)
C13	Si12	C25		116·75 (8)
C13	Si12	O11		109·62 (8)
C19	Si12	C25	1·877 (2)	110·76 (9)
C19	Si12	O11		110·29 (8)
C25	Si12	O11	1·883 (2)	104·13 (8)

Fig. 1. View of (1) showing the atom-labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. H atoms are of arbitrary size.
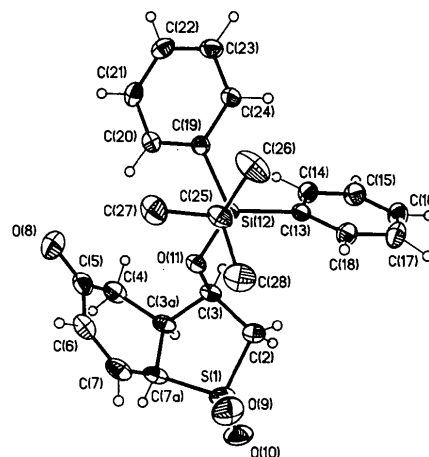

Fig. 2. View of (2) showing the atom-labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. H atoms are of arbitrary size.

were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).* Figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). The positional and equivalent isotropic thermal parameters for non-H atoms are listed in Tables 2 and 3 for (1) and (2), respectively. The bond lengths and angles for the non-H atoms are listed in Tables 4 and 5. The atom-labeling scheme for (1) is shown in Fig. 1 and that for (2) in Fig. 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The above compounds were prepared in the course of a study directed towards the synthesis of breynolide (Sakai, Ohkuma, Koshiyama, Naito & Kawaguchi, 1976; Nishiyama, Ikeda, Yoshida & Yamamura, 1989). Breynolide is the aglycon of Breynin A which has been shown to reduce cholesterol in the blood of mammals (Koshiyama, Hatori, Ohkuma, Sakai, Imanishi, Ohbayashi & Kawaguchi, 1976). In an attempt to control the

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* Tables of anisotropic thermal parameters, H-atom positional and thermal parameters, bond distances and angles involving the H atoms, torsion angles, structure-factor amplitudes, a unit-cell packing diagram and a view of each molecule highlighting the different orientations of the siloxy groups have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52372 (81 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

facial selectivity of a Diels–Alder intermolecular reaction, several different siloxy-protected adducts were prepared and structures investigated to determine any facial bias.

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Structure at 173 K of 1,2-Bis(1-naphthyl)cyclopentene

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Abstract. $C_{25}H_{20}$, $M_r = 320\cdot4$, monoclinic, $I2$, $a = 10\cdot566\,(6)$, $b = 7\cdot618\,(3)$, $c = 10\cdot795\,(7)$ Å, $\beta = 108\cdot00\,(4)°$, $V = 826\cdot3\,(8)$ Å³, $Z = 2$, $D_x = 1\cdot288$ g cm⁻³, $\mu = 0\cdot68$ cm⁻¹, $\lambda(Mo\,K\alpha) = 0\cdot71073$ Å, $F(000) = 340$, $T = 173$ K, $R = 0\cdot0373$ for 1134 reflections $[F_o \geq 6\sigma(F_o)]$. No single-crystal photochemical reaction analogous to those reported for 1,2-diphenylcyclopentene in solution and for 1,2-bis(1-naphthyl)ethylene in the crystal was observed. This is consistent with the considerable twist of the naphthyl groups [dihedral angles: naphthyl–cyclopentene $64\cdot9\,(2)$, naphthyl–naphthyl $76\cdot8\,(2)°$].

Experimental. The title compound (1) was synthesized, using a procedure published for the 2-naphthyl isomer (Rio & Bricout, 1971), by addition of two equivalents of the Grignard reagent from α-bromonaphthalene to cyclobutanecarboxylic acid methyl

(1)