

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: none
 1812 measured reflections
 1812 independent reflections
 1634 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 27.39^\circ$
 $h = -6 \rightarrow 6$
 $k = 0 \rightarrow 18$
 $l = 0 \rightarrow 13$
 3 standard reflections
 frequency: 30 min
 intensity decay: 10%

Refinement

Refinement on F^2
 $R(F) = 0.0498$
 $wR(F^2) = 0.1355$
 $S = 1.101$
 1812 reflections
 113 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.1049P)^2 + 0.1396P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.766 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.733 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

References

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Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C11	0.5820 (4)	0.83649 (14)	0.9424 (2)	0.0377 (4)
C12	0.5490 (5)	0.8573 (2)	1.0738 (2)	0.0440 (5)
C13	0.3679 (7)	0.9086 (2)	1.1091 (3)	0.0602 (7)
C21	0.1651 (4)	0.6497 (2)	1.0875 (2)	0.0377 (4)
C22	0.3244 (4)	0.56510 (14)	1.1197 (2)	0.0412 (5)
C23	0.5286 (5)	0.5670 (2)	1.1986 (2)	0.0486 (5)
S1	0.53204 (8)	0.71000 (3)	0.90186 (4)	0.0336 (2)
S2	0.16519 (8)	0.68598 (4)	0.92458 (4)	0.0367 (2)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

C11—C12	1.483 (3)	C21—S2	1.837 (2)
C11—S1	1.830 (2)	C22—C23	1.313 (3)
C12—C13	1.295 (4)	S1—S2	2.0303 (8)
C21—C22	1.475 (3)		
C12—C11—S1	112.97 (14)	C23—C22—C21	123.8 (2)
C13—C12—C11	124.2 (2)	C11—S1—S2	104.33 (7)
C22—C21—S2	112.84 (14)	C21—S2—S1	105.17 (7)

An absorption correction was not applied because μ was small for both compounds. A correction was applied to both compounds for 10% decay. The structures were solved by direct methods (*SHELXS86*; Sheldrick, 1990) and refined by full-matrix least squares where the quantity minimized was $[\sum(F_o - F_c)^2]$ (*SHELXL93*; Sheldrick, 1993). The H atoms were refined using a riding model.

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovich, 1965); molecular graphics: *ORTEPII* (Johnson, 1976).

BB thanks the Alfried Krupp von Bohlen und Halbach–Stiftung for financial support

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1003). Services for accessing these data are described at the back of the journal.

Acta Cryst. (1997). **C53**, 1066–1068

Tetravinylsilane

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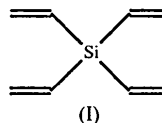
(Received 28 August 1996; accepted 19 March 1997)

Abstract

A single crystal of tetravinylsilane, C₈H₁₂Si, was grown in a capillary at low temperature using zone-melting techniques. Intensity data were collected at 95 K. The title compound crystallizes in space group *Pbca* with one molecule in the asymmetric unit.

Comment

The structure of the title compound is of interest for the synthesis of new silicon-containing materials, for example, dendrimers and polymers (Seyferth, Son, Rheingold & Ostrander, 1994). We have investigated the structure of tetravinylsilane, (I), in order to determine whether it is a suitable candidate for solid-state photochemistry (Schmidt, 1976).



The Si atom is bonded to four C atoms which form an almost ideal tetrahedron. The C—Si—C angles are between 107.85 (6) and 111.34 (6)°. The average Si—C_{sp²} distance of 1.860 (1) Å, as well as the C=C double-bond lengths of 1.318 (1) Å, are characteristic for unsaturated silicon-containing compounds. The molecular point group of tetravinylsilane is C₁. However, disregarding C8, it shows a pseudo-C₃ rotation axis when viewed along the C7—Si bond. The shortest intermolecular distances found are 3.679 Å. The shortest intermolecular distances between almost parallel double bonds are 3.880 and 3.996 Å. Planes passing through these bonds form an angle of 24° with one another. The possibility of a photochemical reaction in the solid state cannot therefore be ruled out (Fig. 2).

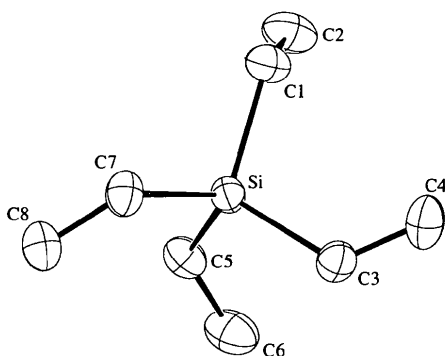


Fig. 1. View of the title molecule showing the labeling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

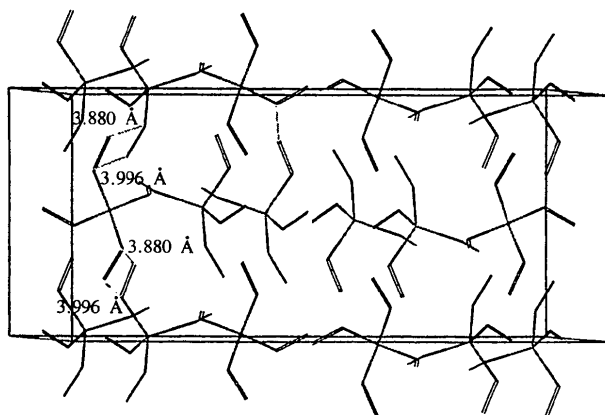


Fig. 2. Packing of tetravinylsilane.

Experimental

Tetravinylsilane obtained from Aldrich was purified by preparative GC. The crystal used for analysis was grown by a zone-melting technique in a capillary of outer diameter 0.3 mm with a focused laser beam as heat source (Boese & Nussbaumer, 1994).

Crystal data

C₈H₁₂Si
M_r = 136.27
 Orthorhombic
Pbca
a = 8.9952 (13) Å
b = 20.460 (2) Å
c = 9.864 (2) Å
V = 1815.3 (5) Å³
Z = 8
D_x = 0.997 Mg m⁻³
D_m not measured

Mo *K*α radiation
λ = 0.71069 Å
 Cell parameters from 25 reflections
θ = 9.5–24.2°
μ = 0.180 mm⁻¹
T = 98 K
 Cylinder
 0.8 × 0.3 × 0.3 mm
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2*θ* scans
 Absorption correction: none
 2103 measured reflections
 2103 independent reflections
 1787 reflections with *I* > 2*σ*(*I*)

*θ*_{max} = 27.60°
h = 0 → 11
k = 0 → 12
l = 0 → 26
 3 standard reflections
 frequency: 30 min
 intensity decay: 10%

Refinement

Refinement on *F*²
R(*F*) = 0.0399
wR(*F*²) = 0.1222
S = 1.079
 2103 reflections
 130 parameters
 All H atoms refined
w = 1/[*σ*²(*F*_o²) + (0.1*P*)²]
 where *P* = (*F*_o² + 2*F*_c²)/3

(*Δ*/*σ*)_{max} = –0.001
*Δρ*_{max} = 0.396 e Å⁻³
*Δρ*_{min} = –0.342 e Å⁻³
 Extinction correction: none
 Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3) \sum_i \sum_j U^{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C1	0.0851 (2)	0.71623 (6)	0.38072 (12)	0.0334 (3)
C2	0.1195 (2)	0.72815 (7)	0.25321 (14)	0.0423 (4)
C3	0.18292 (14)	0.59506 (6)	0.53142 (13)	0.0321 (3)
C4	0.3163 (2)	0.62131 (9)	0.54367 (15)	0.0418 (4)
C5	–0.04221 (15)	0.58508 (6)	0.29817 (13)	0.0336 (3)
C6	0.0251 (2)	0.53302 (7)	0.2494 (2)	0.0446 (4)
C7	–0.13010 (15)	0.64391 (7)	0.56888 (13)	0.0327 (3)
C8	–0.2542 (2)	0.60975 (8)	0.5686 (2)	0.0389 (3)
Si	0.02355 (4)	0.63491 (2)	0.44370 (3)	0.0244 (2)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.318 (2)	C5—C6	1.316 (2)
C1—Si	1.8604 (12)	C5—Si	1.8574 (13)
C3—C4	1.320 (2)	C7—C8	1.317 (2)
C3—Si	1.8624 (13)	C7—Si	1.8624 (13)
C2—C1—Si	123.64 (10)	C1—Si—C7	110.73 (6)
C4—C3—Si	124.33 (11)	C3—Si—C7	107.85 (6)
C6—C5—Si	125.46 (12)	C1—Si—C5	109.13 (6)
C8—C7—Si	125.12 (11)	C3—Si—C5	111.34 (6)
C1—Si—C3	108.50 (6)	C7—Si—C5	109.29 (6)

An absorption correction was not applied because *μ* was small. A correction was applied for 10% decay. The structure was solved by direct methods (*SHELXS86*; Sheldrick, 1990) and refined by full-matrix least squares where the quantity minimized was [*Σ*(*F*_o–*F*_c)²] (*SHELXL93*; Sheldrick, 1993). The H atoms were refined using a riding model.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovich, 1965). Molecular graphics: *ORTEPII* (Johnson, 1976).

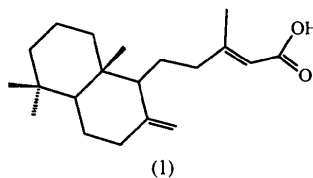
BB thanks the Alfried Krupp von Bohlen und Halbach–Stiftung for financial support

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1004). Services for accessing these data are described at the back of the journal.

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precursors of vegetal origin, in view of the diminishing availability of whales. The (+)-isomer of the title compound, (1), has proved to be a powerful synthetic tool for the preparation of *trans*-decalin derivatives with a strong ambergris odor (Gaitán, 1996). The title compound can be isolated by chemical resolution of the racemic mixture, which is obtained by cross-sectioning the trunk of *Eperua purpurea* (De Santis & Medina, 1981). The (–)-enantiomeric constituent and the (+)-isomer are expected to have the same structural characteristics, except for their configuration. This paper reports the results of an X-ray crystal structure study of (1), carried out in order to determine the conformation and crystal packing of the molecule.



A view of the molecular structure is shown in Fig. 1. The (–)-enantiomer was chosen in accord with the high negative value for the specific optical rotation of the ethanolic solution of the crystals obtained after resolution. The C—C bond distances of the *trans*-decalinic fragment of (1) are similar to those found (single-bond range 1.497–1.575 Å) in two related *trans*-decalinic molecular fragments (Singh, Gray, Skelton, Waterman & White, 1991; Grant, Hanton & Wong, 1995), while the bond lengths C8—C17 [1.324 (5) Å]

Acta Cryst. (1997). **C53**, 1068–1070

(13E)-(–)-Labda-8(17),13-dien-15-oic Acid

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(Received 23 October 1996; accepted 19 February 1997)

Abstract

In the title compound, C₂₀H₃₂O₂, the two six-membered rings have a ¹C₄ chair conformation and the rings are *trans*-fused. The crystal structure is stabilized by approximately symmetric R₂²(8)-type hydrogen bonds.

Comment

As part of our efforts towards the introduction of new starting materials for the synthesis of ambergris odorants, we have directed our attention to synthetic

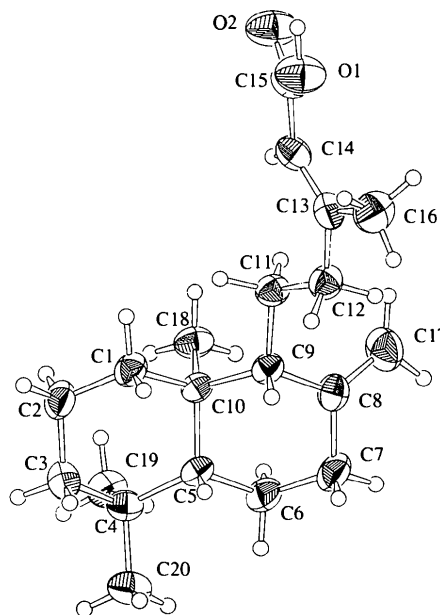


Fig. 1. Molecular diagram of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level.