

## Refinement

Refinement on  $F^2$  $R = 0.041$  $wR = 0.042$  $S = 1.225$ 

1613 reflections

188 parameters

All H-atom parameters refined

 $w = 1.155/[\sigma^2(F) + 0.0006(F)^2]$  $(\Delta/\sigma)_{\max} = 0.02$  $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$ 

Extinction correction: none

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 3. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) for compound (II)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
N1	0.8810 (1)	0.0794 (2)	1.00709 (9)	0.0295 (8)
O1	0.8616 (1)	-0.0704 (1)	1.01849 (8)	0.0372 (8)
O2	0.8365 (1)	0.0300 (1)	0.67821 (7)	0.0396 (8)
O3	0.8495 (1)	-0.2073 (1)	0.64794 (8)	0.0366 (8)
C1	0.8279 (1)	-0.0985 (2)	0.6960 (1)	0.029 (1)
C2	0.7895 (2)	-0.1507 (2)	0.7730 (1)	0.036 (1)
C3	0.7941 (1)	-0.0359 (2)	0.8393 (1)	0.032 (1)
C4	0.8985 (1)	-0.0021 (2)	0.8696 (1)	0.032 (1)
C5	0.8997 (1)	0.1110 (2)	0.9348 (1)	0.0273 (9)
C6	0.9185 (1)	0.2682 (2)	0.9185 (1)	0.0276 (9)
C7	0.8671 (1)	0.3782 (2)	0.9557 (1)	0.032 (1)
C8	0.8871 (2)	0.5244 (2)	0.9417 (1)	0.041 (1)
C9	0.9592 (2)	0.5621 (2)	0.8910 (1)	0.044 (1)
C10	1.0103 (2)	0.4548 (2)	0.8534 (1)	0.042 (1)
C11	0.9897 (2)	0.3082 (2)	0.8660 (1)	0.036 (1)
HO3	0.868 (2)	-0.168 (3)	0.598 (2)	0.072 (8)
HO1	0.848 (2)	-0.070 (2)	1.070 (2)	0.057 (7)

Table 4. Geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for compound (II)

O1—N1	1.405 (2)	C5—C6	1.484 (2)
O2—C1	1.215 (2)	C6—C7	1.391 (3)
O3—C1	1.321 (2)	C7—C8	1.382 (3)
C1—C2	1.498 (3)	C8—C9	1.380 (3)
C2—C3	1.525 (3)	C9—C10	1.375 (3)
C3—C4	1.525 (3)	C6—C11	1.396 (3)
N1—C5	1.285 (2)	C10—C11	1.383 (3)
C4—C5	1.503 (3)		
O1—N1—C5	113.2 (1)	C4—C5—C6	121.5 (1)
O2—C1—O3	123.1 (2)	C5—C6—C7	121.1 (2)
O2—C1—C2	124.1 (2)	C5—C6—C11	120.2 (2)
O3—C1—C2	112.8 (2)	C7—C6—C11	118.8 (2)
C1—C2—C3	114.1 (2)	C6—C7—C8	120.6 (2)
C2—C3—C4	112.6 (2)	C7—C8—C9	120.0 (2)
C3—C4—C5	110.9 (2)	C8—C9—C10	120.2 (2)
N1—C5—C4	122.6 (2)	C9—C10—C11	120.3 (2)
N1—C5—C6	115.9 (2)	C6—C11—C10	120.2 (2)

(I) was obtained by the reaction of 3-benzoylpropionic acid with hydroxylamine hydrochloride in alkaline solution and purified by recrystallization. (II) was obtained by the reaction of 4-benzoylbutyric acid with hydroxylamine hydrochloride in alkaline solution and also purified by recrystallization. Crystals of both (I) and (II) were grown from ethanol/water solution by slow evaporation. Melting points were found to be 400.5–401.5 and 379–380.2 K for (I) and (II), respectively.

Both structures were solved by use of *SHELXS86* (Sheldrick, 1990) and refined by application of *SHELXL76* (Sheldrick, 1976). Most of the H atoms were positioned according to geometric criteria but the two hydroxyl H atoms were located from a difference Fourier map.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71394 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1067]

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*Acta Cryst.* (1994). **C50**, 81–83

1-Chloro-1,2,2-triphenylethene,  $\text{C}_{20}\text{H}_{15}\text{Cl}$ 

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(Received 24 March 1993; accepted 5 July 1993)

## Abstract

All phenyl rings are twisted out of the plane of the ethene framework. The molecular geometry shows no significant difference to that expected.

## Comment

The title compound was synthesized by a published method (Crenshaw & Zimmer, 1983) as part of our efforts to obtain vinyl-chlorosilanes (Ziche, Auner & Behm, 1992). It has also found interest as a nonsteroidal anti-

estrogene (Jellinck & Garland, 1963; Murphy & Sutherland, 1981; Watts, Murphy & Sutherland, 1984).

The introduction of an additional phenyl group and of the Cl atom produces no change in the central C=C double bond [C1=C2 1.323 (3) Å] of the *trans*-stilbene skeleton [e.g. 1.338 Å (Hoekstra, Meertens & Vos, 1975), 1.326 (2) Å (Bouwstra, Schouten & Kroon, 1984)]. Strain is avoided by twisting the phenyl rings out of the plane of the ethene moiety (C1, C11, C11, C2, C21, C31); the corresponding interplanar angles are 45.80 (8)° (C11–C16, ring 1), 123.45 (7)° (C21–C26, ring 2) and 129.99 (5)° (C31–C36, ring 3). The interplanar angles between the rings are 123.39 (7)° for ring 1/ring 2, 84.27 (7)° for ring 1/ring 3 and 82.29 (7)° for ring 2/ring 3. A similar structure has been determined for 1-anisyl-1-bromo-2,2-diphenylethane (Kaftori, Apeloig & Rappoport, 1985). The bond length C1=C2 for this compound is 1.314 Å; the only marked difference is found for the torsion angle C1–C2–C31–C36, which is –68.6° compared to –48.7 (3)° in the present structure.

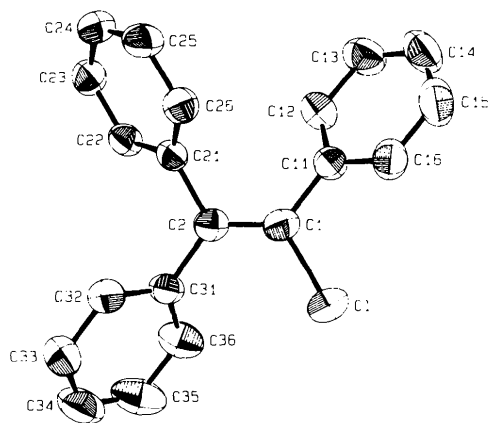


Fig. 1. An ORTEP drawing (Johnson, 1965) of the title compound with atom numbering (50% probability ellipsoids).

## Experimental

### Crystal data

C<sub>20</sub>H<sub>15</sub>Cl  
*M<sub>r</sub>* = 290.8  
 Orthorhombic  
*Pbca*  
*a* = 9.063 (2) Å  
*b* = 18.205 (2) Å  
*c* = 18.681 (1) Å  
*V* = 3082.2 (7) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.253 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 17.5–24.7°  
 $\mu$  = 2.36 cm<sup>-1</sup>  
*T* = 293 K  
 Prism  
 0.50 × 0.50 × 0.50 mm  
 Colourless

### Data collection

Enraf-Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 3084 measured reflections

$\theta_{\max}$  = 25.0°  
*h* = 0 → 10  
*k* = 0 → 21  
*l* = 0 → 22

2283 independent reflections  
 2283 observed reflections  
 $|I| > 0.0$

3 standard reflections  
 frequency: 60 min  
 intensity variation: 1.3%

### Refinement

Refinement on *F*

*R* = 0.055

*wR* = 0.035

*S* = 3.154

2283 reflections

250 parameters

All H-atom parameters re-  
 fined

*w* = 1/ $\sigma^2(F_o)$

( $\Delta/\sigma$ )<sub>max</sub> = 0.001

$\Delta\rho_{\max}$  = 0.20 e Å<sup>-3</sup>

$\Delta\rho_{\min}$  = –0.29 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallogra-*  
*phy* (1974, Vol. IV, Table  
 2.2B)

Data collection: CAD-4 software (Enraf-Nonius, 1988); the  $\omega$  scan width was (1.40 + 0.30tan $\theta$ )° ± 25% before and after each reflection for background counts. Cell refinement: CAD-4 software; *SET4-CELDIM* (de Boer & Duisenberg, 1984). Data reduction: *STRUXIII* (Schmidt, Birkhahn, Massa, Kiprof & Herdtweck, 1987); *SDP-Plus* (Frenz, 1988). Program(s) used to solve structure: *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program used to refine structure: *SDP-Plus*. Software used to prepare material for publication: *PLATON92* (Spek, 1990).

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \cdot a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Cl	0.30323 (9)	0.17018 (3)	0.29441 (3)	0.0820 (2)
C1	0.2460 (2)	0.19681 (11)	0.20818 (10)	0.0484 (7)
C2	0.2098 (2)	0.26600 (10)	0.19560 (10)	0.0436 (7)
C11	0.2457 (2)	0.13379 (10)	0.15750 (9)	0.0460 (7)
C12	0.1279 (2)	0.12224 (12)	0.11184 (11)	0.0558 (8)
C13	0.1273 (3)	0.06261 (13)	0.06562 (12)	0.0700 (9)
C14	0.2446 (3)	0.01493 (11)	0.06514 (12)	0.0779 (9)
C15	0.3623 (3)	0.02603 (12)	0.10975 (13)	0.0744 (10)
C16	0.3629 (3)	0.08465 (11)	0.15617 (11)	0.0581 (8)
C21	0.1777 (2)	0.29012 (10)	0.12015 (10)	0.0395 (6)
C22	0.0462 (2)	0.32543 (11)	0.10462 (10)	0.0453 (7)
C23	0.0160 (2)	0.34782 (11)	0.03536 (10)	0.0534 (8)
C24	0.1158 (3)	0.33569 (12)	–0.01866 (10)	0.0569 (8)
C25	0.2466 (3)	0.30113 (12)	–0.00350 (10)	0.0565 (8)
C26	0.2783 (2)	0.27859 (11)	0.06558 (10)	0.0479 (7)
C31	0.2035 (2)	0.32546 (10)	0.25102 (10)	0.0457 (6)
C32	0.2713 (3)	0.39178 (11)	0.23685 (10)	0.0567 (8)
C33	0.2672 (3)	0.44823 (12)	0.28678 (12)	0.0737 (9)
C34	0.1948 (3)	0.43775 (13)	0.35084 (12)	0.0824 (9)
C35	0.1275 (3)	0.37306 (15)	0.36495 (12)	0.0853 (10)
C36	0.1307 (3)	0.31659 (13)	0.31569 (11)	0.0664 (9)

Table 2. Bond lengths (Å) and angles (°)

Cl–C1	1.760 (2)	C2–C21	1.505 (3)
C1–C2	1.323 (3)	C2–C31	1.499 (3)
C1–C11	1.487 (3)		
Cl–C1–C2	119.86 (15)	C1–C2–C21	119.49 (17)
C1–C1–C11	111.75 (14)	C1–C2–C31	125.06 (17)
C2–C1–C11	128.39 (17)		

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71469 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1043]

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*Acta Cryst.* (1994). **C50**, 83–85

## Structure of Isopropylidene Sedoheptulosan

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(Received 24 March 1993; accepted 7 July 1993)

## Abstract

The title compound, 2,7-anhydro-4,5-*O*-isopropylidene- $\beta$ -D-*altro*-2-heptulopyranose, C<sub>10</sub>H<sub>16</sub>O<sub>6</sub>, has a pyranoid conformation which is a distorted *E*<sub>0</sub> conformation with  $Q = 0.609$  (1) Å,  $\theta = 147.4$  (1)° and  $\Phi = 188.1$  (3)° [Cremer & Pople (1975). *J. Am. Chem. Soc.* **97**, 1354–1358].

The structure is stabilized by hydrogen bonds. The absolute structure, determined through the Flack parameter,  $x = -0.08$  (14) [Flack (1983). *Acta Cryst.* **A39**, 876–881], corresponds to the known chirality of the title compound (Friedel opposites collected and not merged).

## Comment

The title compound was synthesized according to known procedures (Haskins, Hann & Hudson, 1952) and crystals melting at 500 K were obtained from methanol by slow evaporation. The positions of all H atoms were refined with no distance restraints. Both hydroxyl groups O1—H10 and O3—H20 form intermolecular hydrogen bonds. Details of the hydrogen-bonding scheme are given in Table 3. Of the above hydrogen bonds, the latter results in an infinite one-dimensional chain along [0 0 1]. Other distances and angles are generally as expected.

It should be mentioned that this structure is of the 1,6-anhydro- $\beta$ -D-glycopyranose type and therefore the puckering parameters,  $Q = 0.609$  (1) Å,  $\theta = 147.4$  (1)° and  $\Phi = 188.1$  (3)° (Cremer & Pople, 1975), can be compared with these compounds. A similar conformation of the pyranoid ring, as found in the title compound, is reported for 1,6-anhydro-3,4-*O*-isopropylidene- $\beta$ -D-talopyranose (Panagiotopoulos, 1974) and 1,6-anhydro-3,4-*O*-isopropylidene- $\beta$ -D-galactopyranose (Cano, Foces-Foces, Jimenez-Barbero & Martin-Lomas, 1984), and some selected benzylidene derivatives (Cano, Foces-Foces, Jimenez-Barbero, Bernabe & Martin-Lomas, 1986).

The conformation of the 2,7-anhydro five-membered ring is  $Q = 0.396$  (1) Å and  $\Phi = 263.3$  (2)°. For the other

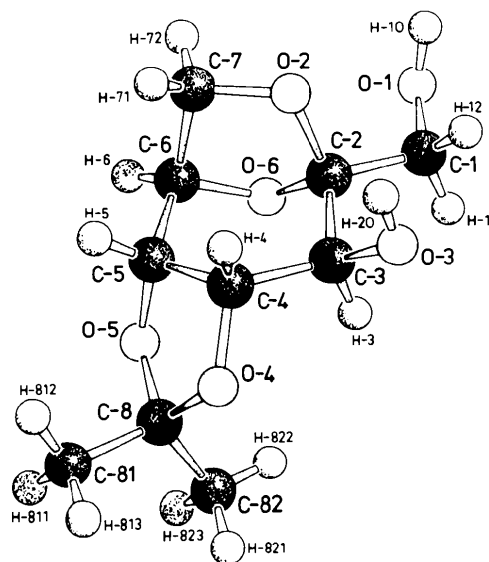


Fig. 1. *SCHAKAL88* drawing (Keller, 1986) of the molecular conformation and atomic numbering scheme.