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2,2,5,5,6-*exo*,8,8,9,10,10-Decachlorobornane

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.080
Data-to-parameter ratio = 21.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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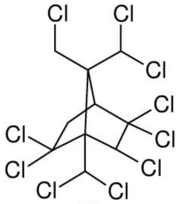
The title compound, $C_{10}H_8Cl_{10}$, is a highly chlorinated Toxaphene congener with the trivial name 'Parlar 69'. X-ray crystallographic analysis reveals a structure different from that reported previously on the basis of spectroscopic characterization.

Comment

The title compound (I) was first isolated in 1993 (Burhenne *et al.*, 1993) from a mixture of deca- and nonachloroterpenes obtained by further chlorination of technical Toxaphene, an insecticide that is one of the twelve priority persistent organic pollutants. Compound (I) was given the trivial name 'Parlar 69' since it corresponded to the 69th peak on a gas chromatogram of technical Toxaphene (Xu *et al.*, 1994). It was characterized by ^1H and ^{13}C NMR spectroscopy and assigned a structure that should be named 2,2,5,5,6-*exo*,8,9,9,10,10-decachlorobornane, according to IUPAC rules (Andrews & Vetter, 1995). For more than a decade, Parlar 69 was believed to have the structure assigned by Burhenne *et al.* (1993), although those authors named it incorrectly as 2,2,5,5,6-*exo*,8,8,9,10,10-decachlorobornane. We report here for the first time the crystal structure of (I).



(I)

The molecular structure of (I) (Fig. 1) is shown to be 2,2,5,5,6-*exo*,8,8,9,10,10-decachlorobornane. Although this is the name that has been used consistently to describe the compound, the structural assignment of Burhenne *et al.* (1993) includes an incorrect orientation for the chlorinated methyl groups bound to C7. The X-ray crystallographic analysis shows that the CH_2Cl group (C9) lies on the same side of the C1—C7—C4 bridge as the methylene group (C3). This is confirmed by NMR spectroscopy, where NOE cross-peaks are evident between protons at C9 and C3. The fact that our compound (I) is identical to that reported as Parlar 69 (Burhenne *et al.*, 1993) is established by comparison of ^1H and ^{13}C NMR spectra, which match in every detail.

Compound (I) is racemic. An attempt to prepare a single enantiomer by separating an enriched fraction of the compound from a mixture of polychlorinated terpenes obtained from an optically active α-pinene was not successful.

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A small amount of crystalline material was isolated, but found to be a racemic mixture rather than a single enantiomer.

Experimental

Compound (I) was obtained according to the literature method (Burhenne *et al.*, 1993), starting from camphene *via* stepwise chlorination. The resulting mixture of highly chlorinated bornanes was chromatographed on a silica column with hexane as eluent. Isolated compound (I) was recrystallized from hexane.

Crystal data

$C_{10}H_8Cl_{10}$
$M_r = 482.66$
Monoclinic, $P2_1/n$
$a = 8.2822$ (2) Å
$b = 12.8466$ (3) Å
$c = 14.9950$ (3) Å
$\beta = 91.2825$ (12)°
$V = 1595.04$ (6) Å^3

$Z = 4$
$D_x = 2.010$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.73$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.20 × 0.13 × 0.12 mm

Data collection

Bruker Kappa-APEX-II CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*MULABS* in *PLATON*; Spek, 2003)
$T_{\min} = 0.735$, $T_{\max} = 0.813$

25472 measured reflections
3941 independent reflections
3168 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.087$
$\theta_{\max} = 28.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.080$
$S = 1.05$
3941 reflections
181 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.006P)^2 + 4.1485P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

All H atoms were visible in electron density maps, but were placed in idealized positions and allowed to ride on their parent atoms at C—H distances of 0.99 (methylene) and 1.00 Å (methine), and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *COLLECT* (Bruker, 2004); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR2002* (Burla, *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

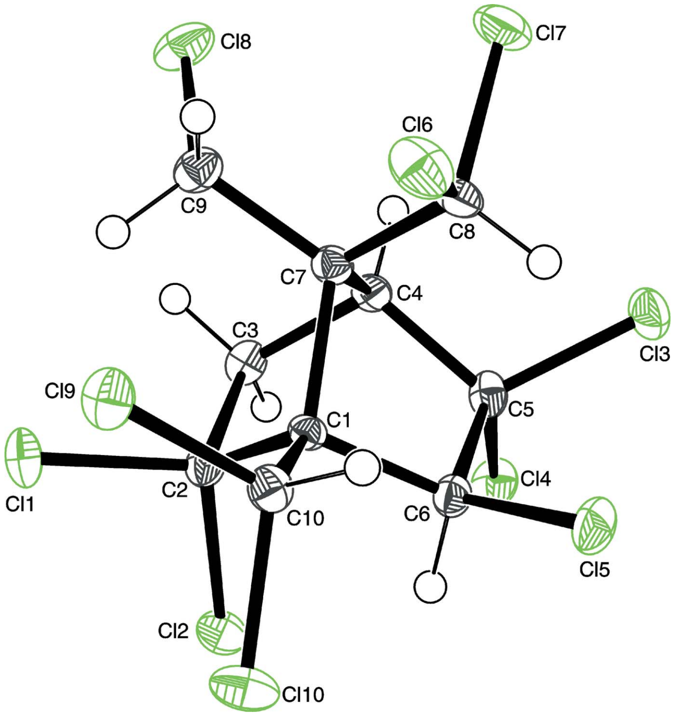

Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary size.

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