Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# (1*S*,4*R*)-1,2,3,4,7,7-Hexachloro-5,6-bis(chloromethyl)bicyclo[2.2.1]hepta-2,5-diene

**Zakiya S. Wilson, Alfonso Davila, Mark L. McLaughlin,‡ Steven F. Watkins and Frank R. Fronczek***

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

‡ Current addresses: Department of Chemistry, University of South Florida, 4202 E. Fowler Avenue, CHE 205A, Tampa, FL 33620-5250, USA, and Drug Discovery Program, H. Lee Moffitt Cancer Center and Research Institute, 12902 Magnolia Drive, MRC 4East, Tampa, FL 33612, USA.

Correspondence e-mail: ffroncz@lsu.edu

**Key indicators**

Single-crystal X-ray study
*T* = 100 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.025
*wR* factor = 0.045
Data-to-parameter ratio = 36.8

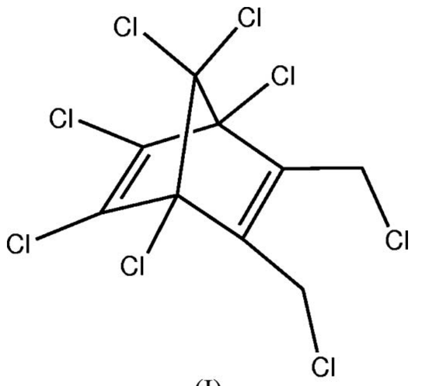
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 November 2006
Accepted 5 December 2006

In the title compound, $C_9H_4Cl_8$, the molecules have approximate mirror symmetry, with the two C=C—CH$_2$—Cl torsion angles differing by only 6.1 (2)°.

## Comment

The title compound, (I), was reported by Hoch & Clegg (1959), synthesized by a Diels–Alder reaction between chlorinated reactants. It has been patented as a bactericide, with potential use in antiseptic cleansing agents (Mark, 1965; 1966). Our primary goal in studying this compound was to develop a new synthetic pathway to cyclopentadienylation. The Diels–Alder adducts of 1,4-but-2-yne with different cyclopentadienes could be subjected to double elimination with a strong base to make new conjugated dienes. These dienes would be used to make new Diels–Alder adducts which, by further transformations, would form novel cyclopentadienes. We synthesized (I) by the Diels–Alder reaction of 1,4-dichlorobut-2-yne and 1,2,3,4,5,5-hexachlorocyclopenta-1,3-diene.



(I)

In compound (I) (Fig. 1), the molecules have approximate mirror symmetry, with the largest deviation across the local mirror being the conformations of the two chloromethyl groups. The two C=C—CH$_2$—Cl torsion angle magnitudes differ by only 6.1 (2)°. The crystal structure of the analogous compound having the C1—C2 bond saturated, the insecticide Alodan, has been reported previously (Kennard *et al.*, 1981). The conformation of Alodan differs substantially from local mirror symmetry, with analogous torsion angles of 161.6 (6) and −74.1 (5)°. The C—Cl distances in (I) are in the range 1.6945 (14)–1.8137 (14) Å (Table 1).

Figs. 2 and 3 illustrate the molecular packing within the unit cell of (I), showing how the molecules assemble around the $4_1$ axis, leading to a rather long *c* axis.

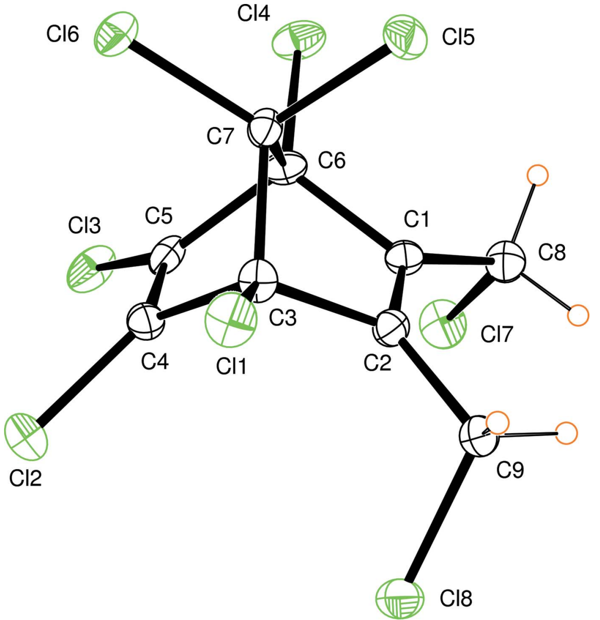


**Figure 1**
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as spheres of arbitrary radii.

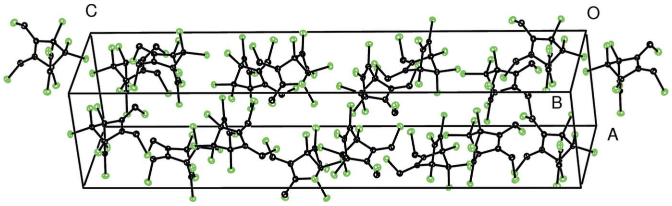


**Figure 2**
The unit-cell contents of (I), with H atoms omitted.

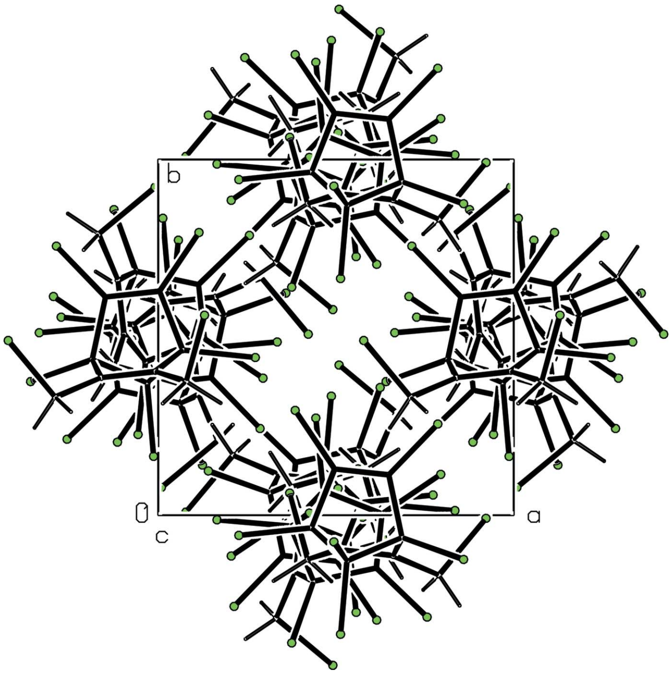


**Figure 3**
The molecular packing of (I), viewed down the fourfold screw axis.

## Experimental

1,2,3,4,5,5-Hexachlorocyclopentadiene (50 ml) and 1,4-dichloro-2-butyne (7.38 g) were reacted at 573 K for 21 h in a Parr bomb, producing 23.55 g of the title compound in 99% yield (m.p. 373–374 K). Crystals of (I) were grown by slow evaporation of a solution in ethanol.

### *Crystal data*

$C_9H_4Cl_8$
$M_r = 395.72$
Tetragonal, $P4_12_12$
$a = 8.5180$ (10) Å
$c = 38.236$ (5) Å
$V = 2774.3$ (6) Å$^3$
$Z = 8$
$D_x = 1.895$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 1.60$ mm$^{-1}$
$T = 100$ K
Fragment, light brown
0.25 × 0.25 × 0.20 mm

### *Data collection*

Nonius KappaCCD area-detector diffractometer (with Oxford Cryosystems Cryostream cooler)
$\omega$ scans with $\kappa$ offsets
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\rm min} = 0.670$, $T_{\rm max} = 0.726$
17778 measured reflections
5705 independent reflections
4162 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\rm max} = 34.9°$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.045$
$S = 1.01$
5705 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0061P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.33$ e Å$^{-3}$
Absolute structure: Flack (1983), with 2143 Friedel pairs
Flack parameter: −0.08 (5)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C3 | 1.7543 (13) | Cl5—C7 | 1.7715 (14) |
| Cl2—C4 | 1.6945 (14) | Cl6—C7 | 1.7667 (15) |
| Cl3—C5 | 1.7071 (13) | Cl7—C8 | 1.8004 (13) |
| Cl4—C6 | 1.7538 (14) | Cl8—C9 | 1.8137 (14) |
| C2—C1—C8—Cl7 | 111.76 (15) | C1—C2—C9—Cl8 | −105.69 (15) |

All H atoms were placed in idealized positions, with C—H distances of 0.99 Å, and thereafter treated as riding, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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*.

The purchase of the diffractometer was made possible by grant No. LEQSF(1999–2000)-ESH-TR-13, administered by the Louisiana Board of Regents.

**o304** Wilson *et al.* • $C_9H_4Cl_8$ *Acta Cryst.* (2007). E**63**, o303–o305

## References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Hoch, P. E. & Clegg, J. M. (1959). *J. Am. Chem. Soc.* **81**, 5413–5417.

Kennard, C. H. L., Smith, G. & Palm, T.-B. (1981). *Acta Cryst.* B**37**, 1953–1955.

Mark, V. (1965). US Patent No. 3 205 274.

Mark, V. (1966). US Patent No. 3 294 691.

Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

*Acta Cryst.* (2007). E**63**, o303–o305 Wilson *et al.* • $C_9H_4Cl_8$ **o305**