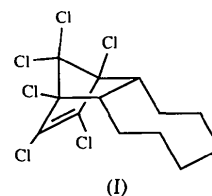


- Eisenstein, O., Procter, G. & Dunitz, J. D. (1978). *Helv. Chim. Acta*, **61**, 2538–2541.
- Elliot, R. J. & Richards, W. G. (1982). *J. Mol. Struct.* **87**, 247–254.
- Evans, K. L., Horn, G. W., Fronczek, F. R. & Gandour, R. D. (1990). *Acta Cryst.* **C46**, 502–504.
- Evans, K. L., Oliver, M. A., Rosas-García & Gandour, R. D. (1994). Unpublished work.
- Evans, K. L., Prince, P., Huang, E. T., Boss, K. R. & Gandour, R. D. (1990). *Tetrahedron Lett.* **31**, 6753–6756.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Houk, K. N., Rondan, N. G., Schleyer, P. v. R., Kaufmann, E. & Clark, T. (1985). *J. Am. Chem. Soc.* **107**, 2821–2823.
- Huang, E. T., Evans, K. L., Fronczek, F. R. & Gandour, R. D. (1992). *Acta Cryst.* **C48**, 765–767.
- Ivanchikova, I. D., Usabaliyeva, G. É., Schastnev, P. V., Moroz, A. A. & Shvartsberg, M. S. (1992). *Bull. Acad. Sci. USSR Div. Chem. Sci. (Engl. Transl.)*, **41**, 1672–1679.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Prince, P., Miller, J. A., Fronczek, F. R. & Gandour, R. D. (1989). *Acta Cryst.* **C45**, 1086–1087.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sinnott, M. L. (1988). *Adv. Phys. Org. Chem.* **24**, 113–204.
- Strozier, R. W., Caramella, P. & Houk, K. N. (1979). *J. Am. Chem. Soc.* **101**, 1340–1343.
- Wang, Y., Cheng, M. C., Lin, L. C., Koh, V. C. & Yang, M. (1985). *Acta Cryst.* **C41**, 924–926.

the sides of the twist-chair for the major conformer are 81.1(4) and $-51.9(4)^\circ$. The norbornene C=C bond length is 1.331(4) Å.

Comment

The crystal structures of a number of Diels–Alder substituted cycloocta(e)ne and cyclohexa(e)ne adducts have been determined in our laboratories in the past three years (García & McLaughlin, 1991; García, Fronczek & McLaughlin, 1991*a,b,c*, 1992*a,b*; García, McLaughlin & Fronczek, 1991*a,b*; Li, Fronczek & McLaughlin, 1992; García, Morales, Fronczek & McLaughlin, 1994; García & Fronczek, 1994, 1995) on account of a study of conformation-dependent π – σ – π electronic interactions (García, Fronczek & McLaughlin, 1991*a*; García & McLaughlin, 1991; García & Fronczek, 1994). The crystal structure determination of the title compound, (I), is part of an ongoing program of structure analysis of some new derivatives bearing the cyclooctane moiety.



Structural data for *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (García, McLaughlin & Fronczek, 1991*a*), *endo-endo-anti*-17,17,18,18-tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (García, McLaughlin & Fronczek, 1991*a*), *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloro-17,17-dimethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (García, Fronczek & McLaughlin, 1992*b*), *endo*-1,10,11,12-tetrachloro-13,13-dimethoxytricyclo[8.2.1.0^{2,9}]trideca-5,11-diene (Li, Fronczek & McLaughlin, 1992), *endo-endo-syn*-16,16-dimethoxy-1,5,6,7,8,12,13,14,15,15-decachloropentacyclo[10.2.1.1^{5,8}.0^{2,11}.0^{4,9}]hexadeca-6,13-diene (García & Fronczek, 1994), *endo-endo-syn*-1,5,6,7,8,12,13,14-octachloro-15,15,16,16-tetramethoxypentacyclo[10.2.1.1^{5,8}.0^{2,11}.0^{4,9}]hexadeca-6,13-diene (García & Fronczek, 1994) and *endo-endo-syn*-4,13-dioxo-1,7,8,9,10,16,17,18-octachloro-19,19,20,20-tetramethoxypentacyclo[14.2.1.1^{7,10}.0^{2,15}.0^{6,11}]jicosa-8,17-diene (García & Fronczek, 1995) are in agreement with those of the title compound.

The cyclooctane ring has a twist-chair conformation (Hendrickson, 1967*b*) with bond angles θ_1 (C2—C1—C8) = 119.5(2), θ_2 (C1—C8—C7) = 115.6(3), θ_3 (C8—C7—C6) = 116.1(3), θ_4 (C7—C6—C5) = 113.4(4), θ_5 (C6—C5—C4) = 114.6(4), θ_6 (C5—C4—C3) = 114.6(3), θ_7 (C4—C3—C2) = 113.8(2) and θ_8 (C3—C2—C1) = 118.7(2) $^\circ$ that are distorted from the theoretical values (Hendrickson, 1967*a*) of the twist-

Acta Cryst. (1995). **C51**, 987–989

endo-1,10,11,12,13,13-Hexachloro-tricyclo[8.2.1.0^{2,9}]trideca-11-ene

J. GABRIEL GARCÍA*

Lawrence Berkeley Laboratory, UC Berkeley,
MS 55-121, Berkeley, CA 94720, USA

FRANK R. FRONCZEK

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

(Received 5 April 1994; accepted 11 October 1994)

Abstract

In *endo*-1,10,11,12,13,13-hexachlorotricyclo[8.2.1.0^{2,9}]trideca-11-ene, C₁₃H₁₄Cl₆, the cyclooctane ring adopts a twist-chair conformation. There is disorder involving the two outermost C atoms of the eight-membered ring in which a conformer having the opposite twist handedness has 15% population. The near-zero torsion angle of the twist-chair at the ring-fusion bond has a magnitude of 2.3(4) $^\circ$. The torsion angles about the bonds comprising

chair and chair conformations (of cyclooctane) having bond angles $\theta_1 = \theta_4 = \theta_5 = \theta_8 = 117^\circ$, $\theta_2 = \theta_3 = \theta_6 = \theta_7 = 115^\circ$ and $\theta_1 = \theta_5 = 114^\circ$, $\theta_2 = \theta_3 = \theta_4 = \theta_6 = \theta_7 = \theta_8 = 116^\circ$, respectively. The torsion angles ω_1 (C3—C2—C1—C8) = 2.3 (4), ω_2 (C2—C1—C8—C7) = -83.9 (3), ω_3 (C1—C8—C7—C6) = 81.1 (4), ω_4 (C8—C7—C6—C5) = -73.9 (5), ω_5 (C7—C6—C5—C4) = 106.2 (4), ω_6 (C6—C5—C4—C3) = -59.5 (5), ω_7 (C5—C4—C3—C2) = -51.9 (4) and ω_8 (C4—C3—C2—C1) = 89.3 (3)° are distorted from the twist-chair and chair conformations (of cyclooctane) having torsion angles of $\omega_1 = 37.3$, $\omega_2 = -109.3$, $\omega_3 = 109.3$, $\omega_4 = -37.3$, $\omega_5 = -37.3$, $\omega_6 = 109.3$, $\omega_7 = -109.3$ and $\omega_8 = 37.3^\circ$, and $\omega_1 = 0$, $\omega_2 = -76.2$, $\omega_3 = 119.9$, $\omega_4 = -76.2$, $\omega_5 = 0$, $\omega_6 = 76.2$, $\omega_7 = -119.9$ and $\omega_8 = 76.2^\circ$, respectively (Hendrickson, 1967a). The conformation of the minor conformer, including atoms C5A and C6A, is also twist-chair with the opposite sense of twist about the C5—C6 bond.

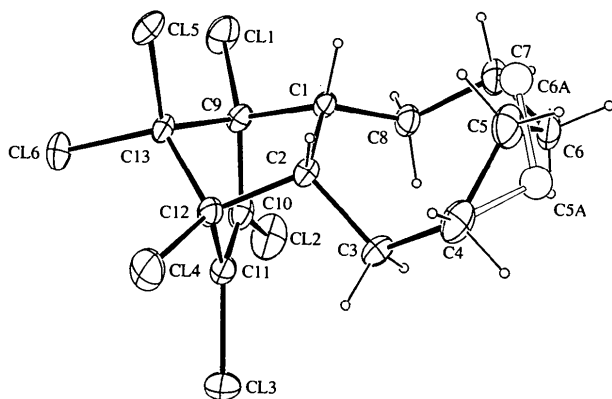


Fig. 1. ORTEP drawing (Johnson, 1965) of the title molecule representing heavy atoms at the 30% probability level. The 15% populated sites are unshaded.

Experimental

Crystals (m.p. 557–559 K) suitable for analysis were prepared by reacting cyclooctene with hexachlorocyclopentadiene using a modification of the method of Akhtar, Fray & Yarrow (1968) and grown by evaporation of a 2,2,5-trimethylpentane solution.

Crystal data

C₁₃H₁₄Cl₆
M_r = 383.0
 Monoclinic
*P*2₁/*n*
a = 9.4029 (6) Å
b = 13.6474 (10) Å
c = 13.1142 (10) Å
 β = 107.806 (6)°
V = 1602.2 (4) Å³
Z = 4
D_x = 1.589 Mg m⁻³

Cu K α radiation
 λ = 1.5418 Å
 Cell parameters from 25 reflections
 θ = 25–34°
 μ = 9.9 mm⁻¹
T = 295 K
 Fragment
 0.50 × 0.42 × 0.27 mm
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: empirical
 $T_{\min} = 0.563$, $T_{\max} = 0.998$
 6727 measured reflections
 3250 independent reflections
 2681 observed reflections
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.040$
 $\theta_{\text{max}} = 75^\circ$
 $h = 0 \rightarrow 11$
 $k = -17 \rightarrow 17$
 $l = -16 \rightarrow 15$
 3 standard reflections
 frequency: 120 min
 intensity decay: 6.9%
 (linear correction)

Refinement

Refinement on *F*
R = 0.056
wR = 0.080
S = 2.640
 2681 reflections
 181 parameters
 H-atom parameters not refined
 $w = 4F^2/[\sigma^2(I) + (0.02F\sigma)^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.03$

$\Delta\rho_{\text{max}} = 0.62 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
 Extinction correction:
 $(1 + gI_c)^{-1}$ applied to *F_c*
 Extinction coefficient:
 $g = 7.0 (2) \times 10^{-6}$
 Atomic scattering factors
 from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B, 2.3.1)

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

*B*_{iso} for C5A and C6A; *B*_{eq} = $(8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$ for others.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{iso} / <i>B</i> _{eq}
C11	0.5074 (1)	0.46502 (8)	0.87498 (6)	5.34 (2)
C12	0.4683 (1)	0.57456 (7)	0.64219 (9)	6.61 (2)
C13	0.1807 (1)	0.4637 (1)	0.45303 (7)	6.89 (3)
C14	0.05287 (9)	0.2835 (1)	0.56717 (8)	6.26 (3)
C15	0.25475 (9)	0.27339 (8)	0.83631 (5)	4.96 (2)
C16	0.13089 (8)	0.46002 (8)	0.76239 (7)	5.48 (2)
C1	0.4928 (3)	0.3145 (2)	0.7289 (2)	2.58 (4)
C2	0.3614 (3)	0.2625 (2)	0.6409 (2)	2.83 (5)
C3	0.3826 (3)	0.2385 (2)	0.5325 (2)	3.67 (6)
C4	0.4514 (4)	0.1384 (3)	0.5285 (3)	5.25 (8)
C5	0.5980 (5)	0.1186 (3)	0.6202 (4)	5.41 (9)
C5A	0.613 (2)	0.145 (2)	0.558 (2)	4.1 (4)
C6	0.7239 (4)	0.1906 (4)	0.6252 (3)	4.65 (8)
C6A	0.679 (2)	0.147 (2)	0.678 (2)	4.1 (4)
C7	0.7512 (4)	0.2621 (3)	0.7156 (3)	4.92 (8)
C8	0.6359 (3)	0.3431 (3)	0.7023 (2)	3.61 (6)
C9	0.4130 (3)	0.4072 (2)	0.7541 (2)	3.04 (5)
C10	0.3735 (3)	0.4723 (2)	0.6550 (2)	3.72 (6)
C11	0.2621 (3)	0.4285 (3)	0.5812 (2)	3.93 (6)
C12	0.2293 (3)	0.3337 (2)	0.6306 (2)	3.28 (6)
C13	0.2551 (3)	0.3687 (2)	0.7456 (2)	3.10 (5)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.577 (3)	C6—C7	1.497 (6)
C1—C8	1.540 (4)	C1—C9	1.557 (4)
C2—C3	1.529 (4)	C6A—C7	1.73 (2)
C2—C12	1.550 (4)	C3—C4	1.520 (5)
C7—C8	1.521 (5)	C4—C5	1.551 (5)
C4—C5A	1.45 (2)	C9—C11	1.750 (3)
C9—C10	1.524 (4)	C5—C6	1.524 (6)
C9—C13	1.546 (4)	C10—C12	1.692 (3)
C10—C11	1.331 (4)	C11—C13	1.690 (3)
C5A—C6A	1.51 (3)	C11—C12	1.520 (5)
C12—C14	1.752 (3)	C13—C16	1.767 (3)
C12—C13	1.530 (4)	C13—C15	1.764 (3)
C2—C1—C8	119.5 (2)	C2—C1—C9	101.6 (2)
C8—C1—C9	110.7 (2)	C5—C6—C7	113.4 (4)
C1—C2—C3	118.7 (2)	C1—C2—C12	102.1 (2)

C3—C2—C12	112.4 (2)	C2—C3—C4	113.8 (2)
C3—C4—C5	114.6 (3)	C3—C4—C5A	111 (1)
C5A—C6A—C7	108 (2)	C4—C5—C6	114.6 (4)
C6—C7—C8	116.1 (3)	C6A—C7—C8	115.2 (7)
C1—C8—C7	115.6 (3)	C11—C9—C1	114.5 (2)
C11—C9—C10	115.7 (2)	C4—C5A—C6A	110 (2)
C11—C9—C13	115.3 (2)	C1—C9—C10	107.5 (2)
C1—C9—C13	102.9 (2)	C10—C9—C13	99.1 (2)
C12—C10—C9	124.6 (2)	C2—C12—C11	108.0 (2)
C12—C10—C11	128.3 (3)	C2—C12—C13	103.2 (2)
C9—C10—C11	106.9 (3)	C11—C12—C13	99.4 (2)
C13—C11—C10	127.7 (3)	C15—C13—C16	107.4 (2)
C13—C11—C12	125.2 (2)	C15—C13—C9	113.7 (2)
C10—C11—C12	106.8 (2)	C15—C13—C12	113.7 (2)
C14—C12—C2	114.4 (2)	C16—C13—C9	114.1 (2)
C14—C12—C11	114.6 (2)	C16—C13—C12	115.6 (2)
C14—C12—C13	115.6 (2)	C9—C13—C12	92.0 (2)
C8—C1—C2—C3	2.3 (4)	C2—C1—C8—C7	-83.9 (3)
C6—C7—C8—C1	81.1 (4)	C5—C6—C7—C8	-73.9 (5)
C4—C5—C6—C7	106.2 (4)	C3—C4—C5—C6	-59.5 (5)
C2—C3—C4—C5	-51.9 (4)	C1—C2—C3—C4	89.3 (3)
C4—C5A—C6A—C7	-110.5 (16)	C3—C4—C5A—C6A	81.4 (17)

Atoms C5 and C6 are disordered over two sites with unequal occupancies. The major sites were assigned occupancies of 85%, based on earlier population-parameter refinement, and were refined anisotropically. The minor sites C5A and C6A were assigned 15% occupancy and were refined isotropically. H atoms were placed in calculated positions with C—H 0.95 Å and $B_{\text{iso}}(\text{H}) = 1.3B_{\text{eq}}(\text{C})$, except for those of the minor disorder sites which were not included.

Programs used were *MolEN* (Fair, 1990), *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *ORTEP* (Johnson, 1965).

We thank DOE for support of this research through grant No. DE-AC03-76SF00098.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Akhtar, I. A., Fray, G. I. & Yarrow, J. M. (1968). *J. Chem. Soc. C*, pp. 812–815.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Garcia, J. G. & Fronczek, F. R. (1994). Unpublished results.
- Garcia, J. G. & Fronczek, F. R. (1995). *Acta Cryst.* **C50**, 468–471.
- Garcia, J. G., Fronczek, F. R. & McLaughlin, M. L. (1991a). *Tetrahedron Lett.* **32**, 3289–3292.
- Garcia, J. G., Fronczek, F. R. & McLaughlin, M. L. (1991b). *Acta Cryst.* **C47**, 1998–2000.
- Garcia, J. G., Fronczek, F. R. & McLaughlin, M. L. (1991c). *Acta Cryst.* **C47**, 2009–2011.
- Garcia, J. G., Fronczek, F. R. & McLaughlin, M. L. (1992a). *Acta Cryst.* **C48**, 195–197.
- Garcia, J. G., Fronczek, F. R. & McLaughlin, M. L. (1992b). *Acta Cryst.* **C48**, 197–199.
- Garcia, J. G. & McLaughlin, M. L. (1991). *Tetrahedron Lett.* **32**, 3292–3296.
- Garcia, J. G., McLaughlin, M. L. & Fronczek, F. R. (1991a). *Acta Cryst.* **C47**, 206–209.
- Garcia, J. G., McLaughlin, M. L. & Fronczek, F. R. (1991b). *Acta Cryst.* **C47**, 451–453.

- Garcia, J. G., Morales, G. A., Fronczek, F. R. & McLaughlin, M. L. (1994). *Acta Cryst.* **C50**, 317–324.
- Hendrickson, J. B. (1967a). *J. Am. Chem. Soc.* **89**, 7036–7043.
- Hendrickson, J. B. (1967b). *J. Am. Chem. Soc.* **89**, 7047–7061.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Li, J., Fronczek, F. R. & McLaughlin, M. L. (1992). *Acta Cryst.* **C48**, 200–201.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1995). **C51**, 989–991

3-(4-Methylphenyl)-1-(3-thienyl)-2-propen-1-one

HE YOUPIING, SU GENBO AND SHI JIANQIU

Fujian Institute of Research on the Structure of Matter, Academia Sinica, Fuzhou, Fujian 350002, People's Republic of China

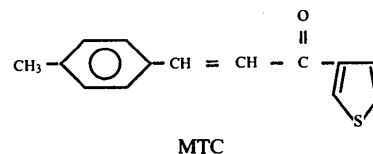
(Received 22 November 1993; accepted 15 July 1994)

Abstract

The title compound, $\text{C}_{14}\text{H}_{12}\text{OS}$, is roughly planar with a dihedral angle of $4.67(8)^\circ$ between the planes of the phenyl and thienyl rings. The O—C—C torsion angle in the central $\text{C}_2\text{H}_2\text{CO}$ group is $1.9(5)^\circ$.

Comment

Chalcone crystals have recently been developed as new and highly efficient organic non-linear optical materials. We have synthesized a series of substituted thiophene chalcone derivatives of which the title compound, 3-(4-methylphenyl)-1-(3-thienyl)-2-propen-1-one (MTC), is an example.



The crystal structure belongs to the centrosymmetric space group $P2_1/n$. The O(1)—C(10)—C(9)—C(8) torsion angle in the central $\text{C}_2\text{H}_2\text{CO}$ group is $1.9(5)^\circ$ and the dihedral angle between the planes of the phenyl and thienyl rings is $4.67(8)^\circ$. Thus, the molecule is roughly planar. These structural data may be compared with those of its isomer (Li & Su, 1993) which