Copernicus University, 87-100 Toruń, Poland) for the preparation of crystals of the title compound.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: NA1220). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Johnson, C. K. (1976). *ORTEP*II. ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kuma Diffraction (1995). *KM-4 Software*. Version 8.0. Kuma Diffraction, Wrocław, Poland.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
Nardelli, M. (1983*a*). *Comput. Chem.* **7**, 95–98.
Nardelli, M. (1983*b*). *Acta Cryst.* C**39**, 1141–1142.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. University of Cambridge, England.
Sheldrick, G. M. (1993). *SHELXL93. Program for Refinement of Crystal Structures*. University of Göttingen, Germany.
Uzarewicz, A., Wyżlic, J. & Ścianowski, J. (1995). *Pol. J. Chem.* **69**, 681.

Acta Cryst. (1997). C**53**, 521-522

4-(1,7,8,9,10,10-Hexachloro-3,5-dioxo-4-azatricyclo[5.2.1.0^{2,6}]dec-8-en-4-yl)butyric Acid Toluene Solvate

BEATA BARTKOWSKA, FRANK M. BOHNEN, CARL KRÜGER AND WILHELM F. MAIER

Max-Planck-Institut für Kohlenforschung, Kaiser-Wilhelm-Platz 1, D-45470 Mülheim an der Ruhr, Germany. E-mail: bohnen@mpi-muelheim.mpg.de

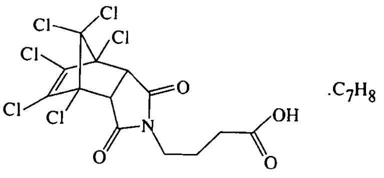
(*Received 31 July 1996; accepted 16 December 1996*)

Abstract

Two molecules of the title compound, $C_{13}H_9Cl_6NO_4.C_7H_8$, are aggregated pairwise by hydrogen bonding. The O···O hydrogen-bond distance is 2.647 (2) Å.

Comment

Pericyclic reactions are known to be very effective for the synthesis of complex molecules (Oppolzer, 1984). In recent years, the antibody catalysis of pericyclic reactions and other reaction types has been developed (Braisted & Schultz, 1990). The catalytic antibodies were obtained by biotechnological methods (Benkovic, 1992). The title compound, (I), belongs to the class of antigene molecules that are used as transition-state analogues (Houk, Gonzalez & Yi, 1995) which catalyze very effectively the reaction of tetrachlorothiophene dioxide and *N*-ethylmaleimide in a cascade of two pericyclic reactions (Hilvert, Hill, Nared & Auditor, 1989). It was therefore of interest to investigate the structural details of the title haptene.



(I)

The structure of (I) can be divided into three parts. The hexachloronorbornene unit is bound *endo* with respect to butanamide. The amide N atom is substituted by a butyric acid group. The C—Cl bond lengths range from 1.692 (2) to 1.763 (2) Å. The C8—Cl4 [1.692 (2) Å] and C9—Cl6 [1.703 (2) Å] bond lengths at the sp^2-hybridized C atoms are significantly longer than the C7—Cl1 [1.750 (2) Å], C10—Cl2 [1.751 (2) Å], C13—Cl5 [1.758 (2) Å] and C13—Cl3 [1.763 (2) Å] bonds at the sp^3-hybridized C atoms. The sum of the three C—N—C angles at the imide N atom is 359.7°. The N—C4 bond length [1.463 (2) Å] is longer than the N—C5 [1.391 (3) Å] and N—C12 [1.388 (2) Å] bond lengths. This indicates, as expected, a delocalized π-electron system along the imide part of the molecule (O3—C5—N—C12—O4) (Cambridge Structural Database, 1996). Two molecules of 4-(1,7,8,9,10,10-hexachloro-3,5-dioxo-4-azatricyclo[5.2.1.0^{2,6}]dec-8-en-4-yl)butyric acid are aggregated by hydrogen bonding. The O···O hydrogen-bond distance is 2.647 (2) Å, indicating a strong hydrogen bond (Cambridge Structural Database, 1996). The aggregation leads to a packing pattern containing pockets which are occupied by toluene molecules.

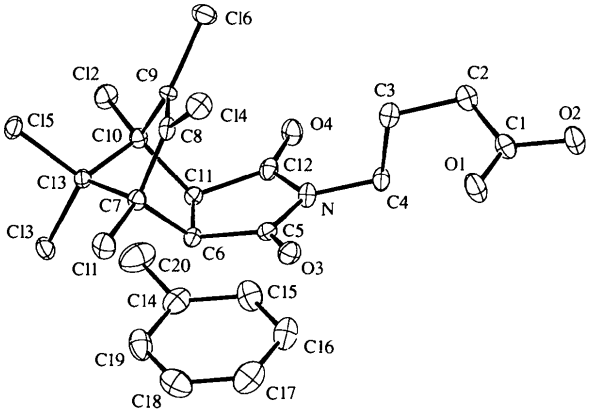


Fig. 1. View of the title compound showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

Experimental

The title compound was synthesized by reaction of 1,4,5,6,7,7-hexachloro-5-norbornene-2,3-dicarboxylic anhydride and 4-aminobutyric acid in toluene under reflux. Upon cooling, the title compound crystallized in 96.9% yield (Bohnen, 1996).

Crystal data

$C_{13}H_9Cl_6NO_4.C_7H_8$
$M_r = 547.88$
Triclinic
$P\bar{1}$
$a = 8.766\,(2)$ Å
$b = 10.759\,(2)$ Å
$c = 13.158\,(2)$ Å
$\alpha = 105.084\,(11)°$
$\beta = 104.568\,(12)°$
$\gamma = 97.278\,(10)°$
$V = 1134.9\,(3)$ Å^3
$Z = 2$
$D_x = 1.604$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 15.16–22.53°$
$\mu = 0.771$ mm^{-1}
$T = 298$ K
Rectangular
$0.60 \times 0.35 \times 0.21$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
5160 measured reflections
5160 independent reflections
4838 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 27.44°$
$h = -11 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 30 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0337$
$wR(F^2) = 0.1090$
$S = 1.159$
5160 reflections
348 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 1.132P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.603$ e Å^{-3}
$\Delta\rho_{\min} = -0.435$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1—O1	1.224 (3)	C7—Cl1	1.750 (2)
C1—O2	1.322 (3)	C8—C9	1.325 (3)
C1—C2	1.498 (3)	C8—Cl4	1.692 (2)
C2—C3	1.519 (3)	C9—C10	1.515 (3)
C3—C4	1.531 (3)	C9—Cl6	1.703 (2)
C4—N	1.463 (2)	C10—C11	1.555 (3)
C5—O3	1.203 (2)	C10—C13	1.561 (3)
C5—N	1.391 (3)	C10—Cl2	1.751 (2)
C5—C6	1.519 (3)	C11—C12	1.520 (3)
C6—C11	1.545 (3)	C12—O4	1.207 (3)
C6—C7	1.555 (3)	C12—N	1.388 (2)
C7—C8	1.536 (3)	C13—Cl5	1.758 (2)
C7—C13	1.555 (3)	C13—Cl3	1.763 (2)
N—C4—C3	111.3 (2)	O4—C12—C11	127.3 (2)
O3—C5—N	124.3 (2)	N—C12—C11	107.7 (2)
O3—C5—C6	127.8 (2)	C12—N—C5	114.2 (2)
N—C5—C6	107.8 (2)	C12—N—C4	123.8 (2)
C11—C6—C7	103.25 (14)	C5—N—C4	121.7 (2)
O4—C12—N	125.0 (2)		

The title compound was solved by direct methods (*SHELXS*86; Sheldrick, 1990) and refined by full-matrix least squares where the quantity minimized was $\Sigma(F_o^2 - F_c^2)$ (*SHELXL*93; Sheldrick, 1993). The positions of the H atoms were calculated and refined isotropically using a riding model.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *DATAP* (Coppens, Leiserowitz & Rabinovich, 1965). Program(s) used to solve structure: *SHELXS*86. Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP*II (Johnson, 1976).

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

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: NA1268). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Benkovic, S. J. (1992). *Annu. Rev. Biochem.* **61**, 29–54.
Bohnen, F. M. (1996). PhD thesis, University of Essen, Germany.
Braisted, A. C. & Schultz, P. G. (1990). *J. Am. Chem. Soc.* **112**, 7430–7431.
Cambridge Structural Database (1996). Version 5.11 of April 1996. Cambridge Crystallographic Data Centre, Cambridge, England.
Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 4656–4663.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Hilvert, D., Hill, K. W., Nared, K. D. & Auditor, M.-T. M. (1989). *J. Am. Chem. Soc.* **111**, 9261–9262.
Houk, K. N., Gonzalez, J. & Yi, L. (1995). *Acc. Chem. Res.* **28**, 81–90.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Oppolzer, W. (1984). *Angew. Chem.* **96**, 840–853.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.