lized from chloroform to afford X-ray quality single crystals of (IV) (m.p. 482–483 K).

Crystal data

$C_{27}H_{18}N_2O_2$
$M_r = 402.43$
Triclinic
$P\bar{1}$
$a = 10.1662\,(16)$ Å
$b = 12.404\,(3)$ Å
$c = 9.0923\,(14)$ Å
$\alpha = 100.677\,(15)°$
$\beta = 102.334\,(12)°$
$\gamma = 111.728\,(14)°$
$V = 995.4\,(3)$ Å^3
$Z = 2$
$D_x = 1.3428\,(4)$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.3–11.9°$
$\mu = 0.086$ mm^{-1}
$T = 296$ K
Irregular fragment
$0.41 \times 0.38 \times 0.30$ mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
ω scans (rate 8° min^{-1} in ω)
Absorption correction: none
3730 measured reflections
3511 independent reflections
2052 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.0158$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 12$
$k = -14 \rightarrow 13$
$l = -10 \rightarrow 10$
3 standard reflections every 150 reflections intensity decay: −0.5%

Refinement

Refinement on F^2
$R(F) = 0.0350$
$wR(F^2) = 0.0863$
$S = 1.091$
3510 reflections
281 parameters
H atoms riding (C—H 0.93–0.98 Å)
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.129P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.151$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.145$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0109 (18)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O2—C14	1.353 (2)	N1—N2	1.391 (2)
O2—C12	1.466 (2)	N1—C22	1.440 (2)
N1—C21	1.381 (2)	N2—C14	1.286 (2)
C14—O2—C12	117.98 (14)	O2—C12—C13	111.26 (15)
C21—N1—N2	124.9 (2)	N2—C14—O2	120.0 (2)
C21—N1—C22	122.7 (2)	N2—C14—C15	124.6 (2)
N2—N1—C22	112.38 (15)	O2—C14—C15	115.5 (2)
C14—N2—N1	118.0 (2)	N1—C21—C20	114.9 (2)
O2—C12—C11	107.28 (14)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *TEXSAN SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS* and *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN*, *SHELXL*93 and *PLATON* (Spek, 1990).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FG1251). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). **C53**, 519–521

(±)-*N*-[1,8(10)-Menthadien-3-yl]toluenesulfonamide

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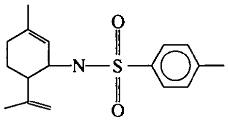
Abstract

In the cyclohexene ring of the title compound, $C_{17}H_{23}NO_2S$, four C atoms are planar and the other two atoms are on either side of the best plane. The toluenesulfonamide group is in an axial configuration. The molecules are linked in chains by N—H···O hydrogen bonds [N···O 2.948 (2) and H···O 2.14 (2) Å].

Comment

The cyclohexene ring of the title compound, (I), is in a half-chair conformation; the C9–C12 atoms are coplanar (within experimental error), with atom C7 deviating by 0.413 (2) Å from one side of the plane and C8 by 0.307 (2) Å from the other. The total puckering amplitude (Cremer & Pople, 1975) is 0.469 (2) Å and there is a pseudo-twofold axis running through the midpoints of the C7—C8 and C10—C11 bonds with an asymmetry parameter Δ_2 of 0.0152 (7) (Nardelli, 1983*b*).



(I)

The best planes calculated through the benzene and cyclohexene rings form a dihedral angle of 45.3 (1)° with one another. The toluenesulfonamide group is attached axially to the ring. The torsion angles N1—C7—C8—C9 and N1—C7—C12—C11 have values of −55.9 (2) and 74.0 (2)°, respectively. The isopropenyl group is attached equatorially to the ring. The angle between the cyclohexene plane and the isopropenyl group is 39.3 (1)°. The C101 atom is in the C9–C12 plane and atom C41 is in the plane of the phenyl ring, within experimental error. The molecules are linked into a chain by strong N—H···O hydrogen bonds [N1—H1 0.80 (3), N1···O2^{i} 2.948 (2), H1···O2^{i} 2.14 (3) Å and N1—H1···O2^{i} 176 (3)°; symmetry code: (i) $-x$, $-y+1$, $-z$].

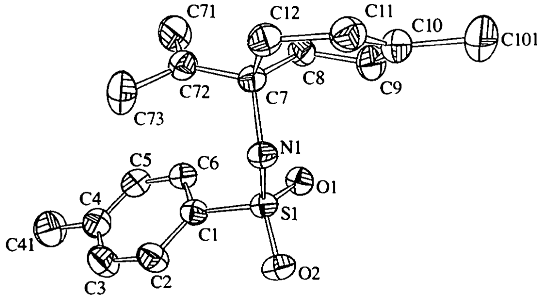


Fig. 1. *ORTEP*II (Johnson, 1976) drawing and the atomic numbering scheme of the title molecule. Ellipsoids are plotted at the 40% probability level.

Experimental

The title compound, which is of interest due to its expected biological activity, was obtained by the reaction of bis(*p*-toluenesulfonyl)selenodiimide with (*S*)-(−)-limonene (Uzarewicz, Wyżlic & Ścianowski, 1995). The colourless needle crystals were obtained by slow evaporation from chloroform or *n*-heptane–ether solution at room temperature.

Crystal data

$C_{17}H_{23}NO_2S$
$M_r = 305.42$
Monoclinic
$P2_1/c$
$a = 10.268\ (1)$ Å
$b = 13.845\ (2)$ Å
$c = 11.742\ (2)$ Å
$\beta = 101.73\ (1)°$
$V = 1634.4\ (4)$ Å^3
$Z = 4$
$D_x = 1.2411$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 34 reflections
$\theta = 4.4–25.8°$
$\mu = 1.785$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.2 × 0.2 × 0.1 mm
Colourless

Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6513 measured reflections
3515 independent reflections
3140 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$

$\theta_{\rm max} = 80.34°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 17$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R(F) = 0.0484$
$wR(F^2) = 0.1401$
$S = 1.059$
3512 reflections
282 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2 + 0.1294P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.098$
$\Delta\rho_{\rm max} = 0.643$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.513$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C7	1.497 (2)	C72—C73	1.500 (3)
C7—C72	1.527 (2)	C10—C11	1.326 (3)
C7—C8	1.533 (2)	C10—C9	1.487 (2)
C7—C12	1.538 (2)	C10—C101	1.502 (2)
C8—C9	1.512 (2)	C12—C11	1.500 (2)
C72—C71	1.317 (3)		
C7—N1—S1	128.41 (10)	C71—C72—C7	122.6 (2)
N1—C7—C72	110.95 (11)	C73—C72—C7	116.59 (15)
N1—C7—C8	110.92 (11)	C11—C10—C9	121.67 (15)
C72—C7—C8	113.49 (12)	C11—C10—C101	121.2 (2)
N1—C7—C12	104.57 (11)	C9—C10—C101	117.1 (2)
C72—C7—C12	108.62 (12)	C10—C9—C8	113.81 (14)
C8—C7—C12	107.82 (12)	C11—C12—C7	113.76 (13)
C9—C8—C7	113.27 (12)	C10—C11—C12	124.06 (15)
C71—C72—C73	120.7 (2)		

Data collection: *KM*-4 *Software* (Kuma Diffraction, 1995). Cell refinement: *KM*-4 *Software*. Data reduction: *KM*-4 *Software*. Program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *PARST* (Nardelli, 1983*a*).

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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.

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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

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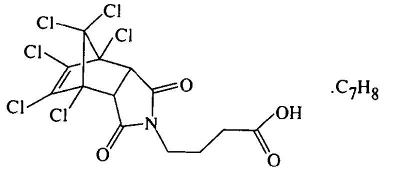
Abstract

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.

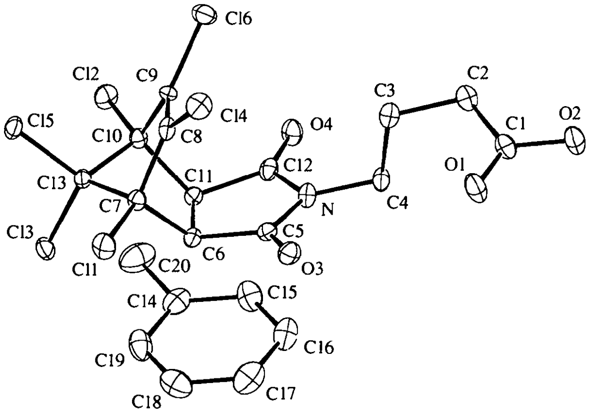


Fig. 1. View of the title compound showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.