Lists of structure factors, anisotropic temperature factors, calculated coordinates of H atoms, mean-planes data and important torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55195 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1004]

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1,8-Dichloro-5-oxa-10-azatricyclo[5.3.1.$0^{3,8}$]-undecan-9-one: Structure of a Hydrolysed Diels–Alder Addition Product

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Abstract

The title molecule is bisected by a crystallographic mirror plane which contains the Cl, O and N atoms. Pairs of molecules are linked across an inversion centre by a strong hydrogen bond between the amine H and ketonic O atoms [NH···O 2.05(4) Å, N—H···O 167(3)°].

Comment

The structure determination reported herein forms part of an investigation into the Diels–Alder addition of diolefins to trichloro-1,2,4-triazine. In contrast to mono-olefins where a second addition occurs only to a very limited extent (Barlow, Haszeldine & Simpkin, 1982), the title molecule [(1), major product] results from a regioselective Diels–Alder addition to diallyl ether (see scheme below) followed by a second intramolecular addition. A minor isomer (2) is formed by competing [1,5] sigmatropic shift of hydrogen and intramolecular addition.

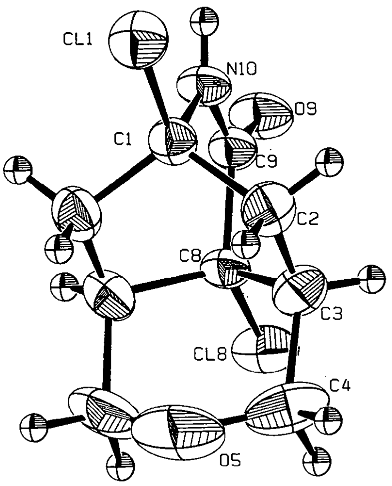
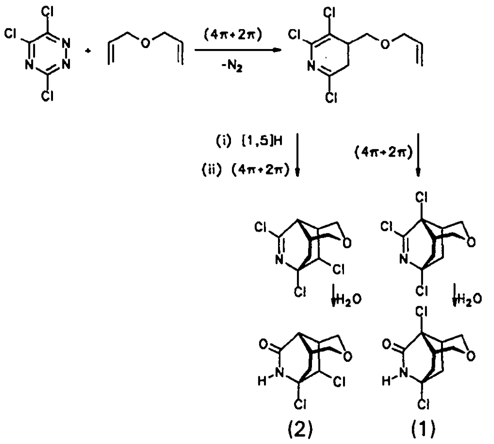


Fig. 1. *ORTEP*II drawing of the title molecule showing atom-numbering scheme.



Experimental

Crystal data

$C_9H_{11}Cl_2NO_2$
M_r = 236.1
Monoclinic
$C2/m$
a = 12.669 (5) Å
b = 8.760 (2) Å
c = 9.475 (5) Å
β = 107.38 (3)°
V = 1003.6 (7) Å^3
Z = 4
D_x = 1.562 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 7.1–22.2°
μ = 0.62 mm^{-1}
T = 293 K
Block
0.3 × 0.3 × 0.2 mm
Colourless

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Data collection

AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: azimuthal scans (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.79$, $T_{\max} = 1.0$
997 measured reflections
997 independent reflections
789 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.065$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 10$
$l = -11 \rightarrow 10$
3 standard reflections monitored every 150 reflections intensity variation: 20%

Refinement

Refinement on F
Final $R = 0.036$
$wR = 0.044$
$S = 1.95$
789 reflections
99 parameters
All H-atom parameters refined

Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: MSC/AFC diffractometer control. Cell refinement: MSC/AFC diffractometer control. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	0.86304 (8)	0	0.05118 (10)	0.0514 (5)
Cl(8)	0.66142 (7)	0	0.55819 (10)	0.0532 (5)
C(1)	0.7913 (3)	0	0.1873 (4)	0.036 (1)
C(2)	0.7201 (2)	0.1417 (3)	0.1745 (3)	0.046 (1)
C(3)	0.6658 (2)	0.1406 (3)	0.3007 (3)	0.046 (1)
C(4)	0.5399 (2)	0.1338 (5)	0.2432 (4)	0.075 (2)
O(5)	0.5029 (2)	0	0.1598 (4)	0.087 (2)
C(8)	0.7080 (2)	0	0.3978 (3)	0.035 (1)
C(9)	0.8352 (3)	0	0.4496 (4)	0.035 (1)
N(10)	0.8731 (2)	0	0.3329 (3)	0.038 (1)
O(9)	0.8921 (2)	0	0.5789 (2)	0.046 (1)

Table 2. *Geometric parameters* (Å, °)

Cl(1)—C(1)	1.786 (4)	C(3)—C(8)	1.535 (3)
Cl(8)—C(8)	1.787 (3)	C(4)—O(5)	1.413 (4)
C(1)—C(2)	1.518 (3)	C(8)—C(9)	1.539 (4)
C(1)—N(10)	1.457 (4)	C(9)—N(10)	1.330 (4)
C(2)—C(3)	1.548 (4)	C(9)—O(9)	1.222 (4)
C(3)—C(4)	1.525 (4)		
Cl(1)—C(1)—C(2)	111.1 (2)	Cl(8)—C(8)—C(3)	111.4 (2)
Cl(1)—C(1)—N(10)	108.2 (2)	Cl(8)—C(8)—C(9)	108.0 (2)
C(2)—C(1)—C(2)	109.7 (3)	C(3)—C(8)—C(3)	106.8 (3)
C(2)—C(1)—N(10)	108.3 (2)	C(3)—C(8)—C(9)	109.6 (2)

A mixture of diallyl ether (7.01 g, 71.5 mmol) and 3,5,6-trichloro-1,2,4-triazine (2.00 g, 10.8 mmol) sealed *in vacuo* in a Rotaflo tube (*ca* 50 ml) and treated at 343 K for 4 d gave nitrogen (0.19 g, 6.8 mmol, 63%), unchanged diallyl ether (6.24 g, 64.7 mmol, 89% recovered) and a higher-boiling residue (2.28 g). The residue was extracted with diethyl ether to give a brown solution and a light-brown moisture-sensitive solid (0.71 g) which had partially hydrolysed as shown by the presence of NH bands (IR and ^{1}H NMR). The solid was dissolved in dichloromethane (5 ml), water (10 ml) was added and the mixture stirred for 3 h. The organic layer was separated, dried using $MgSO_4$ and the solvent removed *in vacuo* to afford a solid (0.68 g), which was sublimed *in vacuo* at 393-413 K to give a mixture (0.67 g, 2.8 mmol, 26%; found C 45.4, H 4.4, N 5.7, Cl 30.0%, calculated for $C_9H_{11}NO_2Cl_2$ C 45.7, H 4.6, N 5.9, Cl 30.0%) of two amide isomers in the ratio 6:1 (^{1}H NMR), which showed only one spot on the thin-layer chromatogram using a variety of solvents as eluant. Recrystallization from chloroform and then acetone gave the major isomer, 1,8-dichloro-5-oxa-10-azatricyclo[5.3.1.0^{3,8}]undecan-9-one which was formed by hydrolysis of 1,8,9-trichloro-5-oxa-10-azatricyclo[5.3.-1.0^{3,8}]undec-9-ene (0.50 g, 2.1 mmol, 19.5%; found C 45.9, H 4.7, N 5.8, Cl 30.1%, $M^{+\cdot}$ 235/237/2; $C_9H_{11}NO_2Cl_2$ requires C 45.7, H 4.6, N 5.9, Cl 30.0%, M 235/237/239), m.p. 501 K.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55312 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1017]

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Oxonium (*R*)-*O*-Acetylmandelate

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Abstract

The three H atoms of the oxonium H_3O^+ cation are disordered equally over four orientations and are involved

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