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2,2-Dichloro-1-(3,3,6-trimethyl-9-oxo-1,5-diazabicyclo[4.3.0]nonan-5-yl)-ethanone

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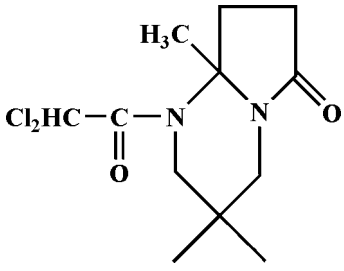
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.129; data-to-parameter ratio = 21.0.

In the title molecule, $C_{12}H_{18}Cl_2N_2O_2$, the six-membered ring is in a chair conformation and the five-membered ring is in an envelope conformation. In the crystal, weak intermolecular bifurcated (C—H)$_2$···O hydrogen bonds connect molecules into chains along [010].

Related literature

For synthetic applications of 1,5-diazabicyclo compounds, see: Hutton & Bartlett (2007); Koptelov *et al.* (2011); Taylor *et al.* (2010). For the bioactivity of *N*-dichloroacety diazabicyclo derivatives, see: Burton *et al.* (1994); Hatzios (2004); Loniovereror (1993). For the synthetic procedure, see: Sun & Ye (2010).



Experimental

Crystal data

$C_{12}H_{18}Cl_2N_2O_2$
M_r = 293.18
Monoclinic, $P2_1/c$
a = 9.4442 (18) Å
b = 14.116 (3) Å
c = 11.7555 (16) Å
β = 115.067 (11)°
V = 1419.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 298 K
0.42 × 0.40 × 0.28 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.832, T_{max} = 0.884
10829 measured reflections
3492 independent reflections
2556 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.04
3492 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.98	2.23	3.200 (3)	170
C3—H3B···O2^{i}	0.97	2.50	3.390 (2)	153

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5271).

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supporting information

Acta Cryst. (2011). E**67**, o2020 [doi:10.1107/S1600536811027176]

2,2-Dichloro-1-(3,3,6-trimethyl-9-oxo-1,5-diazabicyclo[4.3.0]nonan-5-yl)ethanone

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S1. Comment

1,5-Diazabicyclo compounds are important synthetic targets due to their biological activity (Hutton & Bartlett, 2007, Koptelov *et al.*, 2011) and catalytic activity (Taylor *et al.*, 2010). It was discovered that *N*-dichloroacetyl-1,5-diazabicyclo compounds act as herbicide safeners and these compounds have drawn widespread attention in agricultural biochemistry (Burton *et al.*, 1994, Hatzios, 2004, Loniovereror, 1993). As a part of our ongoing investigation on the bioactivities of safeners we have determined the crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. In the crystal, weak interolecular bifurcated (C—H)$_2$···O hydrogen bonds connect molecules into one-dimensional chains along [010]. (Fig. 2).

S2. Experimental

The title compound was prepared according to the literature procedure (Sun *et al.*, 2010). The single-crystal suitable for X-ray structural analysis was obtained by slow evaporation of a solution of the title compound in petroleum ether and ethyl acetate at room temperature.

S3. Refinement

All H atoms were initially located in a difference Fourier map. The C—H atoms were then constrained to an ideal geometry, with C—H distances of 0.96-98 Å, and U_{iso}(H) = 1.2-1.5U_{eq}(C). There is a relatively short H···H contact ca. 1.87Å. This appears to be influenced by the hydrogen bonding.

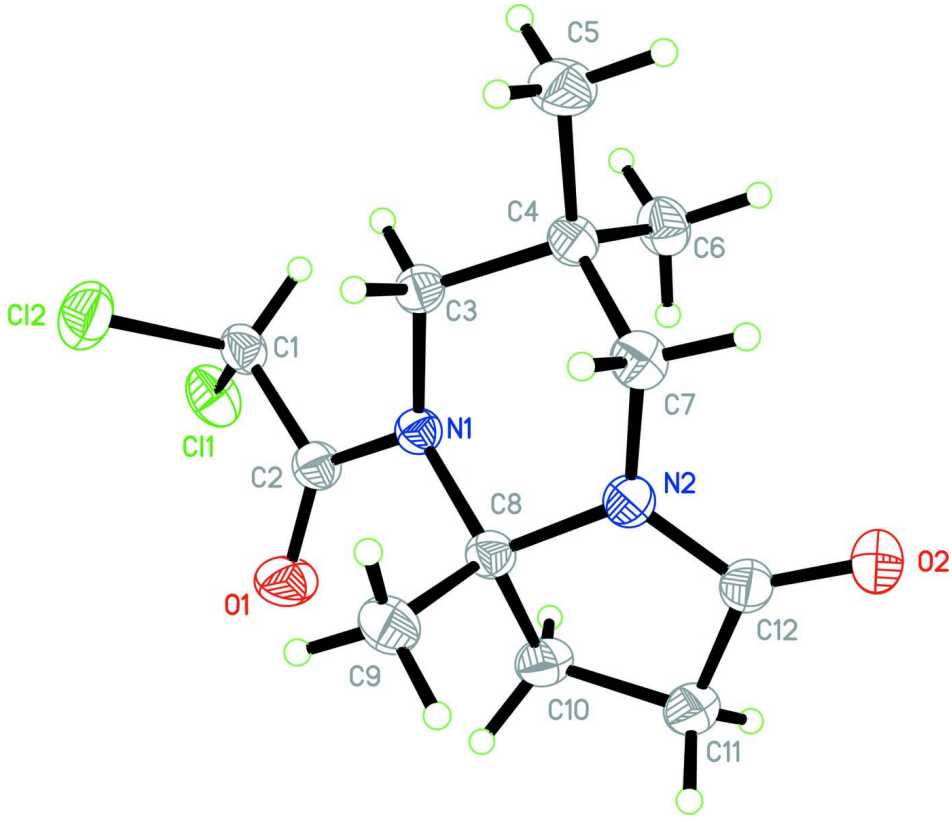

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

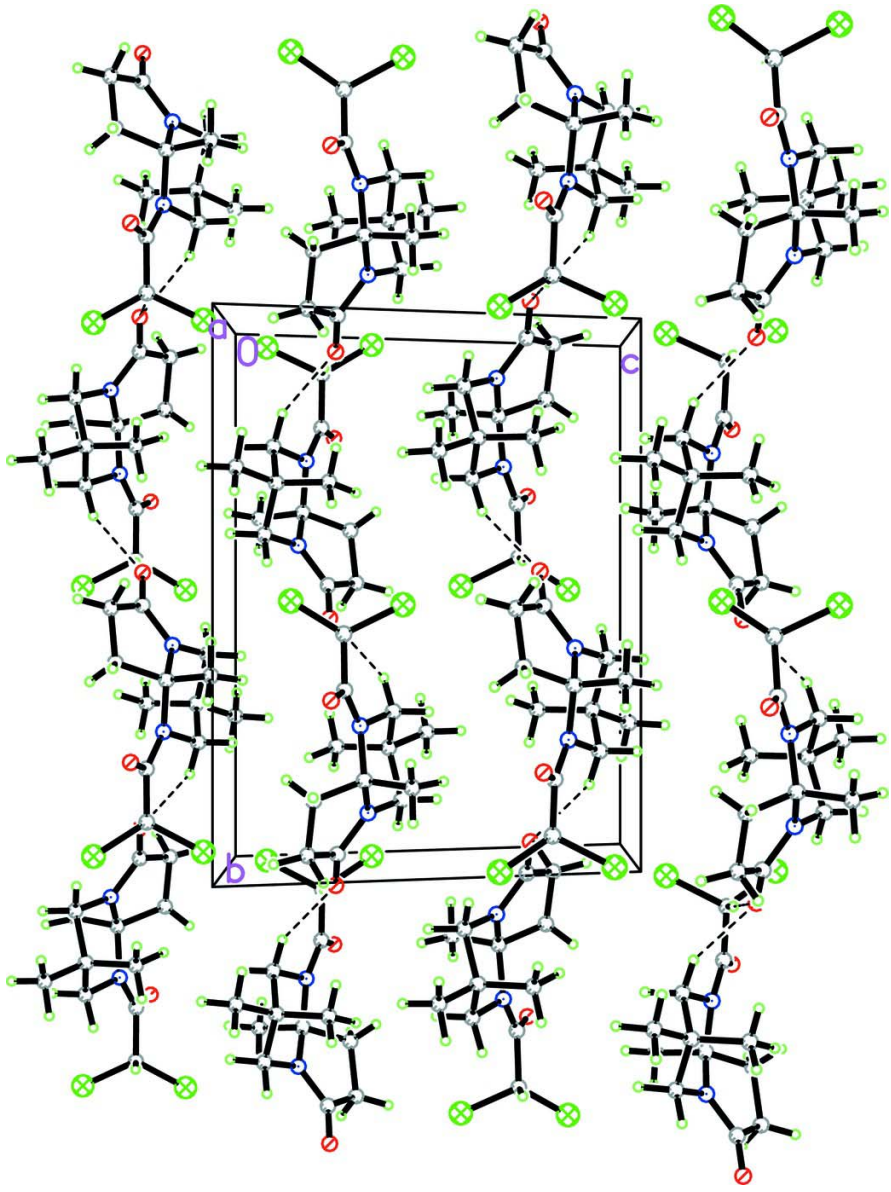

Figure 2

Part of the crystal structure showing the weak intermolecular C—H···O hydrogen bonds as dashed lines.

2,2-Dichloro-1-(3,3,6-trimethyl-9-oxo-1,5-diazabicyclo[4.3.0]nonan-5-yl)ethanone

Crystal data

$C_{12}H_{18}Cl_2N_2O_2$
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$a = 9.4442\ (18)$ Å
$b = 14.116\ (3)$ Å
$c = 11.7555\ (16)$ Å
$\beta = 115.067\ (11)°$
$V = 1419.6\ (4)$ Å^3
$Z = 4$
$F(000) = 616$
$D_x = 1.372$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3515 reflections
$\theta = 2.4–27.1°$
$\mu = 0.45$ mm^{-1}
$T = 298$ K
Block, colourless
0.42 × 0.40 × 0.28 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.832, T_{max} = 0.884
10829 measured reflections
3492 independent reflections
2556 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.3°, θ_{min} = 2.4°
h = −12→12
k = −18→18
l = −9→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.04
3492 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 0.2384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.38732 (6)	−0.01048 (4)	0.34216 (5)	0.05901 (18)
Cl2	0.21200 (7)	−0.01735 (4)	0.07141 (6)	0.0696 (2)
O1	0.43042 (15)	−0.18995 (10)	0.24212 (16)	0.0618 (4)
O2	0.06902 (18)	−0.54370 (10)	0.24058 (16)	0.0638 (4)
N1	0.17907 (15)	−0.24099 (9)	0.17670 (13)	0.0342 (3)
N2	0.10521 (16)	−0.40443 (10)	0.15860 (14)	0.0388 (3)
C1	0.2408 (2)	−0.07096 (12)	0.21585 (17)	0.0419 (4)
H1	0.1428	−0.0703	0.2257	0.050*
C2	0.29300 (19)	−0.17366 (12)	0.21273 (17)	0.0399 (4)
C3	0.01328 (18)	−0.21728 (12)	0.09737 (15)	0.0364 (4)
H3A	−0.0029	−0.2147	0.0102	0.044*
H3B	−0.0089	−0.1549	0.1205	0.044*
C4	−0.10098 (18)	−0.28847 (12)	0.10984 (15)	0.0366 (4)
C5	−0.2656 (2)	−0.26170 (16)	0.0151 (2)	0.0581 (5)
H5A	−0.2699	−0.2615	−0.0679	0.087*
H5B	−0.2913	−0.1998	0.0346	0.087*

H5C	−0.3392	−0.3070	0.0193	0.087*
C6	−0.0909 (2)	−0.28828 (14)	0.24271 (18)	0.0485 (4)
H6A	−0.1645	−0.3329	0.2480	0.073*
H6B	−0.1148	−0.2261	0.2627	0.073*
H6C	0.0128	−0.3057	0.3011	0.073*
C7	−0.0579 (2)	−0.38544 (12)	0.07744 (17)	0.0419 (4)
H7A	−0.1235	−0.4337	0.0893	0.050*
H7B	−0.0742	−0.3866	−0.0098	0.050*
C8	0.22976 (19)	−0.33990 (11)	0.16558 (16)	0.0367 (4)
C9	0.2611 (2)	−0.34929 (15)	0.04896 (19)	0.0516 (5)
H9A	0.2991	−0.4119	0.0457	0.077*
H9B	0.3380	−0.3035	0.0525	0.077*
H9C	0.1660	−0.3384	−0.0247	0.077*
C10	0.3679 (2)	−0.37342 (13)	0.28688 (18)	0.0477 (4)
H10A	0.4655	−0.3695	0.2784	0.057*
H10B	0.3763	−0.3353	0.3582	0.057*
C11	0.3290 (2)	−0.47623 (14)	0.3029 (2)	0.0531 (5)
H11A	0.3784	−0.5195	0.2666	0.064*
H11B	0.3628	−0.4917	0.3909	0.064*
C12	0.1545 (2)	−0.48091 (12)	0.23385 (19)	0.0454 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0585 (3)	0.0609 (3)	0.0632 (3)	−0.0227 (2)	0.0313 (3)	−0.0199 (2)
Cl2	0.0747 (4)	0.0676 (4)	0.0629 (4)	0.0028 (3)	0.0257 (3)	0.0219 (3)
O1	0.0345 (7)	0.0509 (8)	0.0934 (12)	−0.0021 (6)	0.0206 (7)	−0.0014 (7)
O2	0.0683 (9)	0.0460 (7)	0.0921 (12)	0.0003 (7)	0.0483 (9)	0.0128 (8)
N1	0.0325 (7)	0.0336 (6)	0.0362 (7)	−0.0005 (5)	0.0141 (6)	−0.0005 (5)
N2	0.0405 (8)	0.0346 (7)	0.0426 (8)	−0.0015 (6)	0.0189 (6)	−0.0034 (6)
C1	0.0399 (9)	0.0389 (9)	0.0505 (10)	−0.0074 (7)	0.0226 (8)	−0.0032 (8)
C2	0.0360 (9)	0.0392 (8)	0.0431 (9)	−0.0025 (7)	0.0156 (8)	−0.0012 (7)
C3	0.0345 (8)	0.0391 (8)	0.0329 (8)	0.0000 (6)	0.0116 (7)	0.0019 (7)
C4	0.0336 (8)	0.0387 (8)	0.0359 (8)	−0.0028 (6)	0.0133 (7)	−0.0029 (7)
C5	0.0363 (10)	0.0621 (12)	0.0633 (13)	−0.0026 (9)	0.0089 (9)	0.0008 (10)
C6	0.0567 (11)	0.0495 (10)	0.0487 (11)	−0.0041 (8)	0.0313 (9)	−0.0047 (8)
C7	0.0418 (9)	0.0404 (9)	0.0412 (9)	−0.0073 (7)	0.0153 (8)	−0.0097 (7)
C8	0.0367 (8)	0.0344 (8)	0.0414 (9)	−0.0001 (6)	0.0189 (7)	−0.0026 (7)
C9	0.0573 (11)	0.0563 (11)	0.0524 (11)	0.0039 (9)	0.0342 (10)	−0.0030 (9)
C10	0.0419 (10)	0.0461 (10)	0.0507 (11)	0.0072 (8)	0.0153 (9)	0.0024 (8)
C11	0.0530 (11)	0.0509 (11)	0.0594 (12)	0.0123 (9)	0.0278 (10)	0.0129 (9)
C12	0.0565 (11)	0.0381 (9)	0.0533 (11)	0.0051 (8)	0.0345 (10)	0.0008 (8)

Geometric parameters (Å, º)

Cl1—C1	1.7627 (18)	C5—H5B	0.9600
Cl2—C1	1.7715 (19)	C5—H5C	0.9600
O1—C2	1.215 (2)	C6—H6A	0.9600

O2—C12	1.224 (2)	C6—H6B	0.9600
N1—C2	1.362 (2)	C6—H6C	0.9600
N1—C3	1.482 (2)	C7—H7A	0.9700
N1—C8	1.499 (2)	C7—H7B	0.9700
N2—C12	1.348 (2)	C8—C9	1.526 (2)
N2—C7	1.452 (2)	C8—C10	1.544 (2)
N2—C8	1.462 (2)	C9—H9A	0.9600
C1—C2	1.537 (2)	C9—H9B	0.9600
C1—H1	0.9800	C9—H9C	0.9600
C3—C4	1.527 (2)	C10—C11	1.528 (3)
C3—H3A	0.9700	C10—H10A	0.9700
C3—H3B	0.9700	C10—H10B	0.9700
C4—C7	1.522 (2)	C11—C12	1.499 (3)
C4—C6	1.524 (2)	C11—H11A	0.9700
C4—C5	1.528 (2)	C11—H11B	0.9700
C5—H5A	0.9600		

C2—N1—C3	121.54 (13)	C4—C6—H6C	109.5
C2—N1—C8	115.95 (13)	H6A—C6—H6C	109.5
C3—N1—C8	116.53 (12)	H6B—C6—H6C	109.5
C12—N2—C7	123.66 (15)	N2—C7—C4	108.86 (13)
C12—N2—C8	114.61 (15)	N2—C7—H7A	109.9
C7—N2—C8	121.72 (14)	C4—C7—H7A	109.9
C2—C1—Cl1	109.38 (12)	N2—C7—H7B	109.9
C2—C1—Cl2	107.50 (13)	C4—C7—H7B	109.9
Cl1—C1—Cl2	110.34 (9)	H7A—C7—H7B	108.3
C2—C1—H1	109.9	N2—C8—N1	107.82 (13)
Cl1—C1—H1	109.9	N2—C8—C9	110.68 (14)
Cl2—C1—H1	109.9	N1—C8—C9	110.48 (14)
O1—C2—N1	124.36 (16)	N2—C8—C10	101.88 (14)
O1—C2—C1	119.10 (15)	N1—C8—C10	112.40 (13)
N1—C2—C1	116.54 (14)	C9—C8—C10	113.16 (15)
N1—C3—C4	113.10 (13)	C8—C9—H9A	109.5
N1—C3—H3A	109.0	C8—C9—H9B	109.5
C4—C3—H3A	109.0	H9A—C9—H9B	109.5
N1—C3—H3B	109.0	C8—C9—H9C	109.5
C4—C3—H3B	109.0	H9A—C9—H9C	109.5
H3A—C3—H3B	107.8	H9B—C9—H9C	109.5
C7—C4—C6	110.55 (14)	C11—C10—C8	104.57 (15)
C7—C4—C3	107.05 (14)	C11—C10—H10A	110.8
C6—C4—C3	110.96 (14)	C8—C10—H10A	110.8
C7—C4—C5	109.74 (15)	C11—C10—H10B	110.8
C6—C4—C5	110.41 (16)	C8—C10—H10B	110.8
C3—C4—C5	108.05 (15)	H10A—C10—H10B	108.9
C4—C5—H5A	109.5	C12—C11—C10	104.01 (15)
C4—C5—H5B	109.5	C12—C11—H11A	111.0
H5A—C5—H5B	109.5	C10—C11—H11A	111.0
C4—C5—H5C	109.5	C12—C11—H11B	111.0

H5A—C5—H5C	109.5	C10—C11—H11B	111.0
H5B—C5—H5C	109.5	H11A—C11—H11B	109.0
C4—C6—H6A	109.5	O2—C12—N2	124.68 (19)
C4—C6—H6B	109.5	O2—C12—C11	126.95 (18)
H6A—C6—H6B	109.5	N2—C12—C11	108.34 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.98	2.23	3.200 (3)	170
C3—H3*B*···O2^{i}	0.97	2.50	3.390 (2)	153

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1/2.