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## Structure Reports

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## 2-Chloro-*N*-chloromethyl-*N*-(2-ethyl-6-methylphenyl)acetamide

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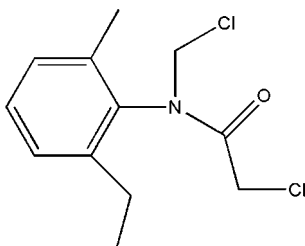
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 Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.075; data-to-parameter ratio = 16.6.

The title compound,  $\text{C}_{12}\text{H}_{15}\text{Cl}_2\text{NO}$ , was synthesized as an intermediate for the synthesis of the herbicide Acetochlor. The crystal structure exhibits weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, which link the molecules into zigzag chains along the  $b$  axis.

### Related literature

For details of the biological activities of Acetochlor, see: Breaux (1986). For bond-length data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

 $\text{C}_{12}\text{H}_{15}\text{Cl}_2\text{NO}$ 
 $M_r = 260.15$ 

 Orthorhombic,  $P2_12_12_1$ 
 $a = 8.3012$  (17) Å

 $b = 9.3787$  (19) Å

 $c = 16.575$  (3) Å

 $V = 1290.4$  (5) Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.48$  mm<sup>-1</sup>
 $T = 296$  (2) K

 $0.33 \times 0.27 \times 0.17$  mm

#### Data collection

Rigaku R-Axis RAPID IP area-detector diffractometer

Absorption correction: multi-scan (ABSCOR; Higashi, 1995)

 $T_{\min} = 0.857$ ,  $T_{\max} = 0.922$ 

20457 measured reflections

2403 independent reflections

 1506 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.051$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$ 
 $wR(F^2) = 0.075$ 
 $S = 0.77$ 

2403 reflections

145 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>
 $\Delta\rho_{\min} = -0.14$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

691 Friedel pairs

Flack parameter: 0.00 (9)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{Cl12}-\text{H12B}\cdots\text{O1}^i$	0.97	2.43	3.375 (4)	164

 Symmetry code: (i)  $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$ 

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2400).

### References

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**supplementary materials**

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## 2-Chloro-*N*-chloromethyl-*N*-(2-ethyl-6-methylphenyl)acetamide

Z.-W. Song

### Comment

Acetochlor is an herbicide developed by Monsanto and Zeneca. It is a member of the class of herbicides known as chloroacetanilides. Its mode of action is elongase inhibition, and inhibition of geranylgeranyl pyrophosphate (GGPP) cyclization enzymes, part of the gibberellin pathway (Breux, 1986). It is used to control weeds in corn, and is particularly useful as a replacement for atrazine in the case of some important weeds. The title compound, (I), was synthesized as an intermediate for the synthesis of Acetochlor. We report here the crystal structure of (I).

In (I) (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987). The mean plane N1/O1/C6/C10/C11 (with largest deviation of 0.036 (2) Å) and benzene ring C1-C6 form a dihedral angle of 78.0 (3)°. The crystal packing exhibits weak intermolecular C—H...O hydrogen bonds (Table 1), which link the molecules into zigzag chains along *b* axis.

### Experimental

The xylene solution containing *N*-methylene-2'-methyl-6'-ethyl-aniline was introduced into a mixture of 1.2 g (0.01 mol) of chloroacetyl chloride and 2 g xylene at 293 K to 313 K under continuous stirring. After about 15 minutes of stirring, 2.5 g of dry ethanol were introduced into mixture at 293 K to 313 K. The reaction mixture was stirred for 5 h, whereupon accoholysis proceeded. At the end of the reaction, 6 g of water were introduced into the mixture, and the phases were separated. The upper organic phase was washed acid-free with about 10 g of water, and the xylene solution, containing about 2.5 g of the desired end product, was separated. Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol and dichloromethane at room temperature.

### Refinement

H atoms were positioned geometrically (C—H = 0.93–0.97 Å), and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2$  (1.5 for methyl groups) times  $U_{\text{eq}}(\text{C})$ .

### Figures

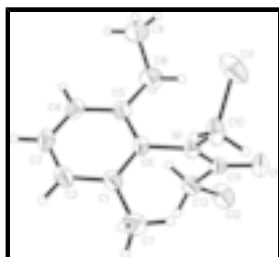


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

## 2-Chloro-*N*-chloromethyl-*N*-(2-ethyl-6-methylphenyl)acetamide

### Crystal data

$C_{12}H_{15}Cl_2NO$	$F_{000} = 544$
$M_r = 260.15$	$D_x = 1.339 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
Hall symbol: P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 8.3012 (17) \text{ \AA}$	Cell parameters from 987 reflections
$b = 9.3787 (19) \text{ \AA}$	$\theta = 2.2\text{--}27.5^\circ$
$c = 16.575 (3) \text{ \AA}$	$\mu = 0.48 \text{ mm}^{-1}$
$V = 1290.4 (5) \text{ \AA}^3$	$T = 296 (2) \text{ K}$
$Z = 4$	Plate, colorless
	$0.33 \times 0.27 \times 0.17 \text{ mm}$

### Data collection

Rigaku R-Axis RAPID IP area-detector diffractometer	2403 independent reflections
Radiation source: rotating anode	1506 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.051$
$T = 296(2) \text{ K}$	$\theta_{\text{max}} = 25.5^\circ$
$\omega$ scans at fixed $\chi = 45^\circ$	$\theta_{\text{min}} = 2.5^\circ$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.857$ , $T_{\text{max}} = 0.923$	$k = -11 \rightarrow 11$
20457 measured reflections	$l = -19 \rightarrow 20$

### Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0172P)^2]$
$wR(F^2) = 0.075$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.77$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2403 reflections	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
145 parameters	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 691 Friedel pairs
	Flack parameter: 0.00 (9)

### 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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.86248 (17)	1.10317 (8)	0.59963 (5)	0.0986 (4)
C12	0.61827 (15)	0.78116 (9)	0.88510 (5)	0.0859 (3)
O1	0.6020 (3)	0.9889 (2)	0.75349 (11)	0.0616 (6)
N1	0.7273 (3)	0.8602 (2)	0.65690 (13)	0.0444 (6)
C1	0.7314 (4)	0.6328 (3)	0.58464 (17)	0.0495 (7)
C2	0.8149 (4)	0.5122 (3)	0.56042 (17)	0.0605 (9)
H2A	0.7634	0.4437	0.5291	0.073*
C3	0.9741 (5)	0.4929 (3)	0.58248 (18)	0.0611 (9)
H3A	1.0287	0.4116	0.5656	0.073*
C4	1.0523 (4)	0.5913 (3)	0.62872 (17)	0.0558 (8)
H4A	1.1592	0.5761	0.6432	0.067*
C5	0.9737 (4)	0.7147 (3)	0.65459 (16)	0.0468 (7)
C6	0.8129 (3)	0.7325 (3)	0.63222 (14)	0.0417 (7)
C7	0.5558 (4)	0.6500 (4)	0.56248 (19)	0.0717 (10)
H7A	0.5165	0.7390	0.5832	0.108*
H7B	0.4947	0.5730	0.5853	0.108*
H7C	0.5447	0.6488	0.5048	0.108*
C8	1.0590 (4)	0.8198 (4)	0.7098 (2)	0.0742 (11)
H8A	0.9981	0.9080	0.7100	0.089*
H8B	1.0563	0.7819	0.7643	0.089*
C10	0.7080 (4)	0.9714 (3)	0.59945 (18)	0.0631 (9)
H10A	0.6057	1.0181	0.6094	0.076*
H10B	0.7029	0.9292	0.5461	0.076*
C11	0.6641 (3)	0.8780 (3)	0.73246 (16)	0.0456 (7)
C12	0.6724 (4)	0.7457 (3)	0.78494 (15)	0.0586 (8)
H12A	0.7812	0.7080	0.7837	0.070*
H12B	0.6009	0.6736	0.7631	0.070*
C9	1.2274 (5)	0.8537 (5)	0.6897 (3)	0.145 (2)
H9A	1.2684	0.9222	0.7275	0.217*
H9B	1.2325	0.8926	0.6362	0.217*
H9C	1.2911	0.7684	0.6923	0.217*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.1649 (10)	0.0562 (5)	0.0746 (6)	-0.0264 (7)	0.0055 (7)	0.0077 (4)
C12	0.1260 (8)	0.0752 (5)	0.0565 (5)	-0.0120 (6)	0.0273 (6)	-0.0026 (4)

## supplementary materials

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O1	0.0704 (15)	0.0524 (12)	0.0621 (12)	0.0181 (13)	0.0044 (12)	-0.0097 (10)
N1	0.0505 (15)	0.0416 (13)	0.0412 (13)	0.0070 (12)	-0.0038 (11)	-0.0019 (11)
C1	0.060 (2)	0.0421 (16)	0.0461 (16)	-0.0048 (16)	0.0026 (15)	-0.0013 (14)
C2	0.086 (3)	0.0459 (18)	0.0501 (17)	-0.0116 (19)	0.0115 (18)	-0.0087 (14)
C3	0.078 (3)	0.0427 (17)	0.063 (2)	0.0139 (19)	0.019 (2)	0.0035 (16)
C4	0.051 (2)	0.0562 (18)	0.0601 (19)	0.0094 (17)	0.0075 (16)	0.0121 (16)
C5	0.052 (2)	0.0452 (16)	0.0431 (15)	-0.0001 (15)	0.0030 (14)	0.0026 (14)
C6	0.0493 (19)	0.0379 (15)	0.0380 (14)	0.0062 (14)	0.0014 (13)	0.0006 (13)
C7	0.065 (2)	0.078 (2)	0.072 (2)	-0.0091 (19)	-0.0055 (19)	-0.0107 (18)
C8	0.063 (3)	0.077 (2)	0.083 (3)	0.0028 (19)	-0.023 (2)	-0.002 (2)
C10	0.084 (2)	0.0505 (17)	0.0543 (18)	0.0211 (17)	-0.0101 (18)	-0.0019 (15)
C11	0.0418 (18)	0.0460 (17)	0.0491 (16)	-0.0023 (15)	-0.0055 (14)	-0.0043 (14)
C12	0.069 (2)	0.0537 (17)	0.0531 (17)	-0.0057 (17)	0.0113 (16)	-0.0039 (15)
C9	0.058 (3)	0.104 (3)	0.272 (7)	-0.018 (3)	0.017 (4)	-0.060 (4)

### *Geometric parameters (Å, °)*

C11—C10	1.781 (3)	C5—C8	1.521 (4)
C12—C12	1.752 (3)	C7—H7A	0.9600
O1—C11	1.212 (3)	C7—H7B	0.9600
N1—C11	1.368 (3)	C7—H7C	0.9600
N1—C10	1.421 (3)	C8—C9	1.472 (5)
N1—C6	1.451 (3)	C8—H8A	0.9700
C1—C2	1.387 (4)	C8—H8B	0.9700
C1—C6	1.398 (4)	C10—H10A	0.9700
C1—C7	1.511 (4)	C10—H10B	0.9700
C2—C3	1.384 (4)	C11—C12	1.517 (4)
C2—H2A	0.9300	C12—H12A	0.9700
C3—C4	1.364 (4)	C12—H12B	0.9700
C3—H3A	0.9300	C9—H9A	0.9600
C4—C5	1.396 (4)	C9—H9B	0.9600
C4—H4A	0.9300	C9—H9C	0.9600
C5—C6	1.395 (4)		
C11—N1—C10	118.7 (2)	C9—C8—C5	116.5 (3)
C11—N1—C6	123.1 (2)	C9—C8—H8A	108.2
C10—N1—C6	118.2 (2)	C5—C8—H8A	108.2
C2—C1—C6	117.8 (3)	C9—C8—H8B	108.2
C2—C1—C7	119.9 (3)	C5—C8—H8B	108.2
C6—C1—C7	122.2 (3)	H8A—C8—H8B	107.3
C3—C2—C1	120.5 (3)	N1—C10—C11	115.3 (2)
C3—C2—H2A	119.7	N1—C10—H10A	108.5
C1—C2—H2A	119.7	C11—C10—H10A	108.5
C4—C3—C2	121.0 (3)	N1—C10—H10B	108.5
C4—C3—H3A	119.5	C11—C10—H10B	108.5
C2—C3—H3A	119.5	H10A—C10—H10B	107.5
C3—C4—C5	120.7 (3)	O1—C11—N1	122.1 (2)
C3—C4—H4A	119.6	O1—C11—C12	123.8 (3)
C5—C4—H4A	119.6	N1—C11—C12	114.1 (2)
C6—C5—C4	117.7 (3)	C11—C12—C12	112.13 (19)

C6—C5—C8	121.9 (3)	C11—C12—H12A	109.2
C4—C5—C8	120.3 (3)	C12—C12—H12A	109.2
C5—C6—C1	122.2 (3)	C11—C12—H12B	109.2
C5—C6—N1	119.5 (3)	C12—C12—H12B	109.2
C1—C6—N1	118.3 (2)	H12A—C12—H12B	107.9
C1—C7—H7A	109.5	C8—C9—H9A	109.5
C1—C7—H7B	109.5	C8—C9—H9B	109.5
H7A—C7—H7B	109.5	H9A—C9—H9B	109.5
C1—C7—H7C	109.5	C8—C9—H9C	109.5
H7A—C7—H7C	109.5	H9A—C9—H9C	109.5
H7B—C7—H7C	109.5	H9B—C9—H9C	109.5
C6—C1—C2—C3	-0.5 (4)	C11—N1—C6—C5	-79.7 (3)
C7—C1—C2—C3	-177.9 (3)	C10—N1—C6—C5	100.6 (3)
C1—C2—C3—C4	0.3 (5)	C11—N1—C6—C1	102.0 (3)
C2—C3—C4—C5	-0.4 (4)	C10—N1—C6—C1	-77.7 (3)
C3—C4—C5—C6	0.6 (4)	C6—C5—C8—C9	-140.5 (4)
C3—C4—C5—C8	176.7 (3)	C4—C5—C8—C9	43.6 (5)
C4—C5—C6—C1	-0.9 (4)	C11—N1—C10—C11	88.9 (3)
C8—C5—C6—C1	-176.9 (3)	C6—N1—C10—C11	-91.4 (3)
C4—C5—C6—N1	-179.1 (2)	C10—N1—C11—O1	-5.1 (4)
C8—C5—C6—N1	4.9 (4)	C6—N1—C11—O1	175.2 (3)
C2—C1—C6—C5	0.8 (4)	C10—N1—C11—C12	172.3 (3)
C7—C1—C6—C5	178.1 (3)	C6—N1—C11—C12	-7.4 (4)
C2—C1—C6—N1	179.1 (2)	O1—C11—C12—C12	-11.6 (4)
C7—C1—C6—N1	-3.6 (4)	N1—C11—C12—C12	171.1 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C12—H12B...O1 <sup>i</sup>	0.97	2.43	3.375 (4)	164

Symmetry codes: (i)  $-x+1, y-1/2, -z+3/2$ .

Fig. 1

