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2,2-Dichloro-*N*-(2,3-dimethylphenyl)-acetamideB. Thimme Gowda,^{a*} Ingrid Svoboda,^b Sabine Foro,^b Helmut Paulus^b and Hartmut Fuess^b^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

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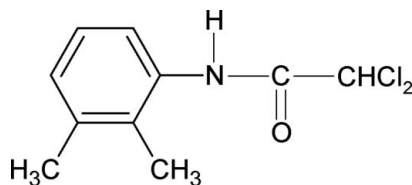
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Key indicators: single-crystal X-ray study; $T = 297$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 14.8.

The conformation of the N—H bond in the title compound, $\text{C}_{10}\text{H}_{11}\text{Cl}_2\text{NO}$, is *syn* to both the 2- and 3-methyl substituents in the aromatic ring, similar to that of the 2-chloro and 3-chloro substituents in 2,2-dichloro-*N*-(2,3-dichlorophenyl)-acetamide and the 2-methyl substituent in 2,2-dichloro-*N*-(2-methylphenyl)acetamide, but in contrast to the *anti* conformation observed with respect to the 3-methyl substituent in 2,2-dichloro-*N*-(3-methylphenyl)acetamide. The bond parameters in the title compound are similar to those in 2,2-dichloro-*N*-phenylacetamide and other acetanilides. The molecules of the title compound are linked into chains through N—H \cdots O and C—H \cdots O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2006); Gowda, Foro & Fuess (2007); Gowda, Kozisek *et al.* (2007); Shilpa & Gowda (2007).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_{11}\text{Cl}_2\text{NO}$
 $M_r = 232.10$

 Monoclinic, $C2/c$
 $a = 21.516$ (8) Å

 $b = 4.678$ (2) Å
 $c = 22.179$ (9) Å
 $\beta = 91.54$ (2)°
 $V = 2231.6$ (16) Å³
 $Z = 8$

 Mo $K\alpha$ radiation
 $\mu = 0.55$ mm⁻¹
 $T = 297$ (2) K
 $0.60 \times 0.16 \times 0.12$ mm

Data collection

 Stoe STAD14 4-circle diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.918$, $T_{\max} = 0.942$
 3934 measured reflections

 1969 independent reflections
 1595 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$
 3 standard reflections
 frequency: 180 min
 intensity decay: 6%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.102$
 $S = 1.07$
 1969 reflections
 133 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N6}-\text{H6N}\cdots\text{O5}^i$	0.78 (3)	2.07 (3)	2.830 (2)	165 (2)
$\text{C3}-\text{H3}\cdots\text{O5}^i$	0.98	2.31	3.100 (3)	137

Symmetry code: (i) $x, y + 1, z$.

Data collection: *STAD14* (Stoe & Cie, 1987); cell refinement: *STAD14*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

BTG thanks the Alexander von Humboldt Foundation, Bonn, Germany, for extensions of his research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2288).

References

- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP3*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* **E63**, o4708.
- Gowda, B. T., Kozisek, J., Svoboda, I. & Fuess, H. (2007). *Z. Naturforsch. Teil A*, **62**, 91–100.
- Gowda, B. T., Paulus, H., Kozisek, J., Tokarcik, M. T. & Fuess, H. (2006). *Z. Naturforsch. Teil A*, **61**, 675–682.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Shilpa & Gowda, B. T. (2007). *Z. Naturforsch. Teil A*, **62**, 84–90.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (1987). *STAD14* and *REDU4*. Stoe & Cie GmbH, Darmstadt, Germany.

supporting information

Acta Cryst. (2008). E64, o234 [https://doi.org/10.1107/S1600536807064550]

2,2-Dichloro-*N*-(2,3-dimethylphenyl)acetamide

B. Thimme Gowda, Ingrid Svoboda, Sabine Foro, Helmut Paulus and Hartmut Fuess

S1. Comment

In the present work, the structure of 2,2-Dichloro-*N*-(2,3-dimethylphenyl)acetamide (23DMPDCA) has been determined as part of a study of the substituent effects on the structures of *N*-aromatic amides (Gowda *et al.*, 2006; Gowda, Foro & Fuess, 2007; Gowda, Kozisek *et al.*, 2007). The conformation of the N—H bond in 23DMPDCA is *syn* to both the 2- and 3-methyl substituents in the aromatic ring (Fig. 1), similar to that of 2-chloro and 3-chloro substituents in the 2,2-dichloro-*N*-(2,3-dichlorophenyl)acetamide (23DCPDCA) (Gowda, Foro & Fuess, 2007) and 2-methyl substituent in 2,2-Dichloro-*N*-(2-methylphenyl)acetamide (2MPDCA) (Gowda *et al.*, 2006), but in contrast to the *anti* conformation observed with respect to the 3-methyl substituent in the 2,2-Dichloro-*N*-(3-methylphenyl)acetamide (3MPDCA) (Gowda *et al.*, 2006). The bond parameters in 23DMPDCA are similar to those in 2,2-dichloro-*N*-(phenyl)acetamide, 2MPDCA, 3MPDCA (Gowda *et al.*, 2006), 23DCPDCA (Gowda, Foro & Fuess, 2007) and other acetanilides. The molecules in 23DMPDCA are linked into infinite chains through simultaneous N—H···O and C—H···O hydrogen bonding (Table 1 and Fig. 2).

S2. Experimental

The title compound was prepared according to the literature method (Shilpa & Gowda, 2007). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Shilpa & Gowda, 2007). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

S3. Refinement

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å (CH aromatic) or 0.96 Å (CH₃) or 0.98 Å (CHCl₂) and N—H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{CH or NH})$ and $U_{\text{iso}}(\text{H}) = 1.4 U_{\text{eq}}(\text{CH}_3)$.

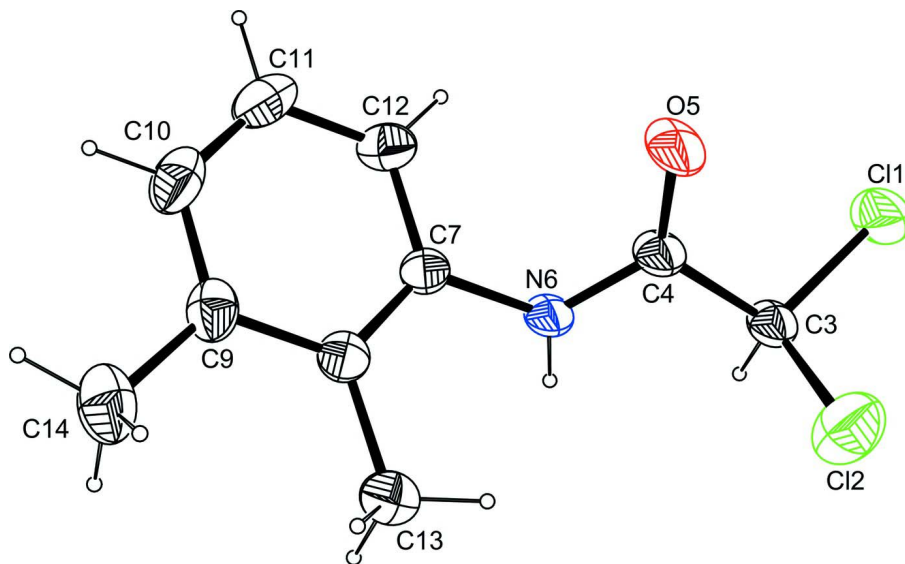


Figure 1

Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

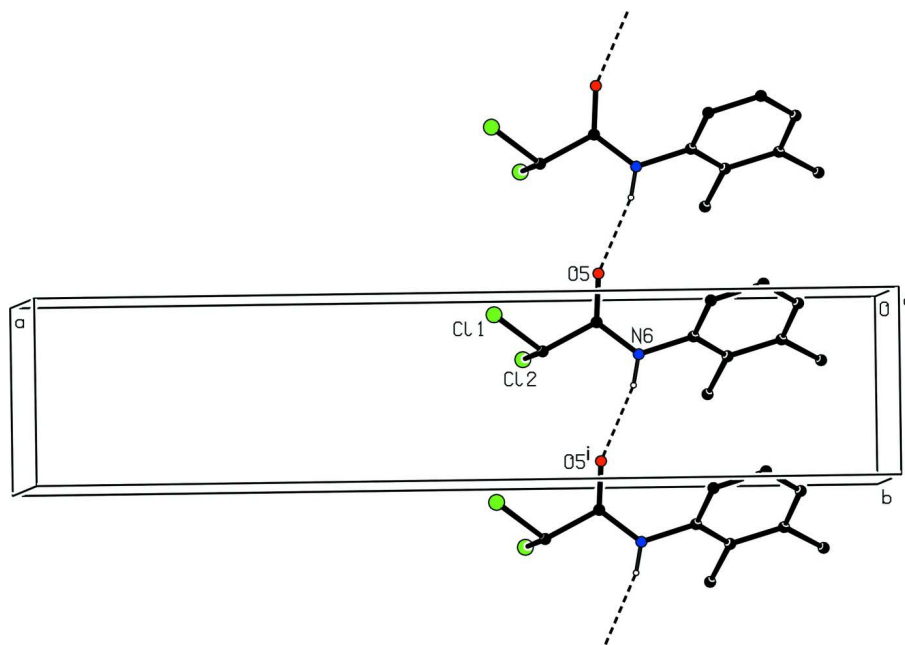


Figure 2

Partial packing view of the title compound with hydrogen bonding shown as dashed lines. [Symmetry code: (i) $x, 1 + x, y$]

2,2-Dichloro-N-(2,3-dimethylphenyl)acetamide

Crystal data

$C_{10}H_{11}Cl_2NO$

$M_r = 232.10$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 21.516 (8) \text{ \AA}$

$b = 4.678 (2) \text{ \AA}$

$c = 22.179 (9) \text{ \AA}$

$\beta = 91.54 (2)^\circ$

$V = 2231.6 (16) \text{ \AA}^3$
 $Z = 8$
 $F(000) = 960$
 $D_x = 1.382 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$
 Cell parameters from 44 reflections

$\theta = 18.0\text{--}20.6^\circ$
 $\mu = 0.55 \text{ mm}^{-1}$
 $T = 297 \text{ K}$
 Needle, light yellow
 $0.60 \times 0.16 \times 0.12 \text{ mm}$

Data collection

Stoe STADI4 4-circle diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Profile fitted scans $2\theta/\omega=1/1$
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.918$, $T_{\max} = 0.942$
 3934 measured reflections

1969 independent reflections
 1595 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -25 \rightarrow 25$
 $k = 0 \rightarrow 5$
 $l = 0 \rightarrow 26$
 3 standard reflections every 180 min
 intensity decay: 6%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.102$
 $S = 1.07$
 1969 reflections
 133 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 atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 2.0597P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e \AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.45285 (3)	0.09175 (16)	0.44140 (4)	0.0836 (3)
C12	0.41708 (4)	0.3272 (2)	0.32575 (3)	0.0945 (3)
C3	0.39612 (9)	0.2882 (4)	0.40110 (10)	0.0504 (5)
H3	0.3923	0.4778	0.4194	0.061*
C4	0.33333 (10)	0.1369 (4)	0.40248 (11)	0.0510 (5)
O5	0.33048 (8)	-0.1222 (3)	0.40441 (12)	0.0912 (7)
N6	0.28502 (8)	0.3103 (4)	0.39976 (9)	0.0462 (4)
H6N	0.2915 (11)	0.475 (6)	0.3986 (10)	0.057 (7)*
C7	0.22132 (9)	0.2200 (4)	0.39827 (9)	0.0435 (5)

C8	0.18107 (9)	0.3255 (4)	0.35353 (9)	0.0451 (5)
C9	0.11839 (10)	0.2396 (5)	0.35446 (10)	0.0541 (5)
C10	0.09995 (11)	0.0506 (6)	0.39835 (12)	0.0671 (7)
H10	0.0586	-0.0069	0.3987	0.081*
C11	0.14076 (12)	-0.0548 (5)	0.44137 (12)	0.0689 (7)
H11	0.1272	-0.1841	0.4700	0.083*
C12	0.20191 (11)	0.0313 (5)	0.44203 (10)	0.0563 (6)
H12	0.2298	-0.0362	0.4714	0.068*
C13	0.20344 (11)	0.5237 (5)	0.30556 (11)	0.0597 (6)
H13A	0.1936	0.7173	0.3161	0.084*
H13B	0.1834	0.4764	0.2677	0.084*
H13C	0.2476	0.5044	0.3022	0.084*
C14	0.07173 (12)	0.3510 (7)	0.30821 (13)	0.0790 (8)
H14A	0.0839	0.2942	0.2686	0.111*
H14B	0.0702	0.5558	0.3104	0.111*
H14C	0.0314	0.2737	0.3161	0.111*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0547 (4)	0.0786 (5)	0.1163 (6)	0.0004 (3)	-0.0202 (3)	0.0313 (4)
C12	0.0850 (5)	0.1212 (7)	0.0773 (5)	-0.0172 (5)	0.0046 (4)	0.0159 (4)
C3	0.0465 (11)	0.0325 (10)	0.0719 (14)	-0.0007 (9)	-0.0041 (10)	0.0007 (9)
C4	0.0486 (12)	0.0282 (10)	0.0759 (15)	-0.0035 (9)	-0.0056 (10)	0.0001 (9)
O5	0.0567 (10)	0.0243 (8)	0.192 (2)	-0.0026 (7)	-0.0065 (12)	0.0013 (10)
N6	0.0436 (9)	0.0232 (8)	0.0715 (12)	-0.0041 (7)	-0.0038 (8)	0.0022 (8)
C7	0.0442 (11)	0.0290 (9)	0.0572 (12)	-0.0043 (8)	0.0015 (9)	-0.0034 (8)
C8	0.0485 (11)	0.0329 (9)	0.0539 (11)	-0.0001 (8)	0.0032 (9)	-0.0075 (9)
C9	0.0443 (11)	0.0513 (12)	0.0665 (14)	-0.0024 (10)	-0.0004 (10)	-0.0148 (11)
C10	0.0506 (13)	0.0647 (15)	0.0865 (18)	-0.0170 (12)	0.0117 (12)	-0.0138 (14)
C11	0.0752 (17)	0.0575 (14)	0.0751 (16)	-0.0195 (13)	0.0219 (13)	0.0050 (13)
C12	0.0655 (14)	0.0433 (11)	0.0601 (13)	-0.0075 (11)	0.0004 (11)	0.0050 (10)
C13	0.0618 (14)	0.0518 (13)	0.0656 (14)	0.0024 (11)	-0.0004 (11)	0.0077 (11)
C14	0.0542 (14)	0.090 (2)	0.0917 (19)	0.0009 (14)	-0.0145 (13)	-0.0119 (16)

Geometric parameters (Å, °)

C11—C3	1.753 (2)	C9—C14	1.509 (3)
C12—C3	1.752 (2)	C10—C11	1.371 (4)
C3—C4	1.526 (3)	C10—H10	0.9300
C3—H3	0.9800	C11—C12	1.376 (3)
C4—O5	1.215 (2)	C11—H11	0.9300
C4—N6	1.319 (3)	C12—H12	0.9300
N6—C7	1.434 (3)	C13—H13A	0.9600
N6—H6N	0.78 (3)	C13—H13B	0.9600
C7—C12	1.385 (3)	C13—H13C	0.9600
C7—C8	1.390 (3)	C14—H14A	0.9600
C8—C9	1.408 (3)	C14—H14B	0.9600

C8—C13	1.500 (3)	C14—H14C	0.9600
C9—C10	1.381 (4)		
C4—C3—C12	108.55 (15)	C11—C10—C9	122.0 (2)
C4—C3—C11	110.56 (15)	C11—C10—H10	119.0
C12—C3—C11	110.33 (12)	C9—C10—H10	119.0
C4—C3—H3	109.1	C10—C11—C12	119.8 (2)
C12—C3—H3	109.1	C10—C11—H11	120.1
C11—C3—H3	109.1	C12—C11—H11	120.1
O5—C4—N6	125.1 (2)	C11—C12—C7	119.1 (2)
O5—C4—C3	120.6 (2)	C11—C12—H12	120.5
N6—C4—C3	114.27 (17)	C7—C12—H12	120.5
C4—N6—C7	124.88 (17)	C8—C13—H13A	109.5
C4—N6—H6N	117.8 (17)	C8—C13—H13B	109.5
C7—N6—H6N	117.3 (17)	H13A—C13—H13B	109.5
C12—C7—C8	122.20 (19)	C8—C13—H13C	109.5
C12—C7—N6	118.53 (18)	H13A—C13—H13C	109.5
C8—C7—N6	119.27 (18)	H13B—C13—H13C	109.5
C7—C8—C9	117.84 (19)	C9—C14—H14A	109.5
C7—C8—C13	121.34 (19)	C9—C14—H14B	109.5
C9—C8—C13	120.8 (2)	H14A—C14—H14B	109.5
C10—C9—C8	119.1 (2)	C9—C14—H14C	109.5
C10—C9—C14	120.1 (2)	H14A—C14—H14C	109.5
C8—C9—C14	120.8 (2)	H14B—C14—H14C	109.5
C12—C3—C4—O5	89.3 (3)	N6—C7—C8—C13	2.1 (3)
C11—C3—C4—O5	-31.8 (3)	C7—C8—C9—C10	-1.6 (3)
C12—C3—C4—N6	-89.0 (2)	C13—C8—C9—C10	178.2 (2)
C11—C3—C4—N6	149.90 (18)	C7—C8—C9—C14	178.5 (2)
O5—C4—N6—C7	-0.4 (4)	C13—C8—C9—C14	-1.6 (3)
C3—C4—N6—C7	177.77 (19)	C8—C9—C10—C11	0.5 (4)
C4—N6—C7—C12	51.9 (3)	C14—C9—C10—C11	-179.7 (2)
C4—N6—C7—C8	-128.6 (2)	C9—C10—C11—C12	1.0 (4)
C12—C7—C8—C9	1.5 (3)	C10—C11—C12—C7	-1.2 (4)
N6—C7—C8—C9	-178.04 (18)	C8—C7—C12—C11	-0.1 (3)
C12—C7—C8—C13	-178.4 (2)	N6—C7—C12—C11	179.4 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N6—H6N \cdots O5 ⁱ	0.78 (3)	2.07 (3)	2.830 (2)	165 (2)
C3—H3 \cdots O5 ⁱ	0.98	2.31	3.100 (3)	137

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