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## Structure Reports

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2,2,2-Trichloro-*N*-(2,5-dimethylphenyl)-acetamideB. Thimme Gowda,<sup>a\*</sup> Sabine Foro<sup>b</sup> and Hartmut Fuess<sup>b</sup><sup>a</sup>Department of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and <sup>b</sup>Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

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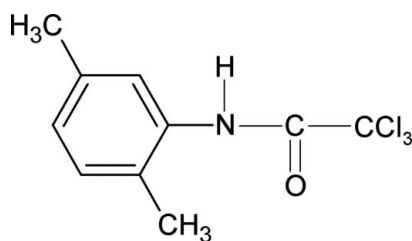
Received 3 April 2008; accepted 5 April 2008

Key indicators: single-crystal X-ray study;  $T = 299$  K; mean  $\sigma(\text{C}-\text{C}) = 0.014$  Å; disorder in main residue;  $R$  factor = 0.089;  $wR$  factor = 0.309; data-to-parameter ratio = 15.0.

The N—H bond in the title compound,  $\text{C}_{10}\text{H}_{10}\text{Cl}_3\text{NO}$ , is *syn* to the 2-methyl and *anti* to the 5-methyl substituent of the aromatic ring. Adjacent molecules are linked into chains through N—H $\cdots$ O hydrogen bonding. Two Cl atoms are each disordered equally over two sites.

## Related literature

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



## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_{10}\text{Cl}_3\text{NO}$  $M_r = 266.54$ Orthorhombic,  $P2_12_12_1$  $a = 4.9173$  (9) Å $b = 11.290$  (1) Å $c = 21.070$  (2) Å $V = 1169.7$  (3) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation  
 $\mu = 0.76$  mm<sup>-1</sup> $T = 299$  (2) K  
 $0.16 \times 0.12 \times 0.06$  mm

## Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector  
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)Diffraction, 2007)  
 $T_{\min} = 0.889$ ,  $T_{\max} = 0.956$   
6121 measured reflections  
2314 independent reflections  
703 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.071$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.088$  $wR(F^2) = 0.308$  $S = 0.86$ 

2314 reflections

154 parameters

37 restraints

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.27$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.76$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

887 Friedel pairs

Flack parameter:  $-0.4$  (4)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N7}-\text{H7N}\cdots\text{O6}^i$	0.86	2.12	2.984 (11)	178

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *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: NG2441).

## References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* **E63**, o2343–o2344.  
 Gowda, B. T., Kožíšek, J., Tokarčík, M. & Fuess, H. (2007). *Acta Cryst.* **E63**, o2571–o2572.  
 Oxford Diffraction (2004). *CrysAlis CCD*. Oxford Diffraction Ltd. Köln, Germany.  
 Oxford Diffraction (2007). *CrysAlis RED*. Oxford Diffraction Ltd. Köln, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Shilpa & Gowda, B. T. (2007). *Z. Naturforsch. Teil A*, **62**, 84–90.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

**supplementary materials**

*Acta Cryst.* (2008). E64, o828 [ doi:10.1107/S1600536808009264 ]

## 2,2,2-Trichloro-*N*-(2,5-dimethylphenyl)acetamide

B. T. Gowda, S. Foro and H. Fuess

### Comment

In the present work, the structure of 2,2,2-trichloro-*N*-(2,5-dimethylphenyl)acetamide (25DMPTCA) has been determined to study the effect of substituents on the structures of *N*-aromatic amides (Gowda, Foro *et al.*, 2007; Gowda, Kožišek *et al.*, 2007). The conformation of the N—H bond in 25DMPTCA is *syn* to the 2-methyl and *anti* to the 5-methyl substituents in the aromatic ring (Fig. 1), similar to the *syn* conformation observed with respect to the 2-methyl substituent in 2,2,2-trichloro-*N*-(2-methylphenyl)acetamide (2MPTCA) (Gowda, Kožišek *et al.*, 2007). The bond parameters in 25DMPTCA are similar to those in 2MPTCA, 2,2,2-trichloro-*N*-(2,6-dimethylphenyl)-acetamide and other acetanilides (Gowda, Foro *et al.*, 2007; Gowda, Kožišek *et al.*, 2007). The intermolecular N—H...O hydrogen bonds link the molecules into chains (Table 1 and Fig.2). The Cl atoms of CCl<sub>3</sub> group are disordered and Cl1 and Cl3 were refined using a split model with site-occupation factors 0.5:0.5. No reliable disorder model could be produced for Cl2.

### Experimental

The title compound was prepared according to the literature method (Shilpa and 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 and Gowda, 2007). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

### Refinement

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å, N—H = 0.86 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the  $U_{eq}$  of the parent atom).

The Cl atoms of CCl<sub>3</sub> group are disordered and Cl1 and Cl3 were refined using a split model with site-occupation factors 0.5:0.5. No reliable disorder model could be produced for Cl2. The C—Cl distances were restrained to 1.77 (2) Å and the distances in the disordered groups were restrained to be equal.

The compound is a weak anomalous scatterer with minor intensity at high  $\theta$  value. The low fraction of unique data is above the  $2\sigma$  level (30°).

### Figures

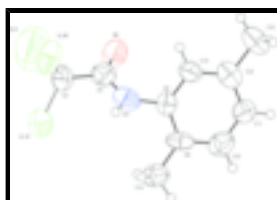


Fig. 1. Molecular structure of the title compound, showing the atom labeling scheme. The displacement ellipsoids drawn at the 50% probability level.

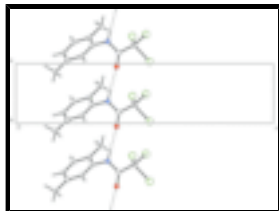


Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

## 2,2,2-Trichloro-*N*-(2,5-dimethylphenyl)acetamide

### Crystal data

$C_{10}H_{10}Cl_3NO$

$M_r = 266.54$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.9173$  (9) Å

$b = 11.290$  (1) Å

$c = 21.070$  (2) Å

$V = 1169.7$  (3) Å<sup>3</sup>

$Z = 4$

$F_{000} = 544$

$D_x = 1.514$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1061 reflections

$\theta = 2.6$ – $28.1^\circ$

$\mu = 0.76$  mm<sup>-1</sup>

$T = 299$  (2) K

Prism, colourless

$0.16 \times 0.12 \times 0.06$  mm

### Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

$T = 299$ (2) K

Rotation method data acquisition using  $\omega$  and  $\phi$  scans.

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)

$T_{\min} = 0.889$ ,  $T_{\max} = 0.956$

6121 measured reflections

2314 independent reflections

703 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.071$

$\theta_{\text{max}} = 26.4^\circ$

$\theta_{\text{min}} = 3.4^\circ$

$h = -6 \rightarrow 6$

$k = -14 \rightarrow 13$

$l = -26 \rightarrow 23$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.088$

$wR(F^2) = 0.309$

$S = 0.86$

2314 reflections

154 parameters

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1675P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.003$

$\Delta\rho_{\text{max}} = 0.27$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.76$  e Å<sup>-3</sup>

Extinction correction: none

37 restraints

Absolute structure: Flack (1983), 887 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: -0.4 (4)

Secondary atom site location: difference Fourier map

### Special details

**Experimental.** empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11A	0.4909 (19)	0.3895 (7)	0.5055 (3)	0.096 (2)	0.50
C11B	0.301 (3)	0.3888 (10)	0.5125 (4)	0.139 (4)	0.50
C12	0.0276 (14)	0.5253 (7)	0.5201 (3)	0.218 (3)	
C13A	0.459 (2)	0.6280 (6)	0.4526 (3)	0.102 (2)	0.50
C13B	0.226 (2)	0.6324 (7)	0.4737 (5)	0.141 (3)	0.50
O6	-0.0518 (17)	0.4448 (7)	0.3920 (4)	0.092 (3)	
N7	0.3670 (16)	0.4054 (7)	0.3571 (4)	0.069 (2)	
H7N	0.5356	0.4163	0.3662	0.083*	
C4	0.311 (2)	0.4931 (7)	0.4592 (4)	0.082 (3)	
C5	0.180 (3)	0.4444 (9)	0.4003 (6)	0.075 (3)	
C8	0.308 (2)	0.3488 (8)	0.2991 (4)	0.055 (3)	
C9	0.4334 (19)	0.2416 (9)	0.2866 (5)	0.062 (3)	
C10	0.358 (2)	0.1877 (10)	0.2267 (5)	0.079 (3)	
H10	0.4336	0.1151	0.2154	0.095*	
C11	0.178 (2)	0.2420 (10)	0.1863 (4)	0.067 (3)	
H11	0.1371	0.2054	0.1480	0.080*	
C12	0.055 (2)	0.3486 (10)	0.2002 (5)	0.069 (3)	
C13	0.125 (2)	0.4011 (9)	0.2572 (4)	0.062 (3)	
H13	0.0466	0.4735	0.2679	0.074*	
C14	0.624 (2)	0.1827 (8)	0.3309 (5)	0.075 (3)	
H14A	0.5312	0.1655	0.3700	0.090*	
H14B	0.7750	0.2341	0.3392	0.090*	
H14C	0.6878	0.1103	0.3123	0.090*	
C15	-0.138 (2)	0.4037 (11)	0.1548 (5)	0.091 (3)	
H15A	-0.2838	0.3497	0.1462	0.109*	
H15B	-0.0441	0.4218	0.1160	0.109*	
H15C	-0.2094	0.4753	0.1727	0.109*	

## supplementary materials

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11A	0.116 (6)	0.109 (5)	0.063 (3)	0.023 (5)	0.000 (4)	-0.003 (3)
C11B	0.172 (8)	0.151 (7)	0.093 (5)	-0.036 (7)	0.005 (6)	0.037 (5)
C12	0.213 (6)	0.278 (7)	0.163 (4)	0.011 (6)	0.007 (4)	-0.053 (4)
C13A	0.135 (6)	0.086 (4)	0.086 (4)	-0.038 (4)	0.016 (4)	-0.025 (3)
C13B	0.151 (7)	0.117 (6)	0.154 (6)	0.018 (6)	-0.027 (6)	-0.049 (5)
O6	0.052 (4)	0.127 (7)	0.099 (5)	0.011 (5)	-0.007 (4)	-0.040 (5)
N7	0.049 (5)	0.068 (5)	0.092 (6)	-0.004 (5)	-0.014 (5)	0.016 (5)
C4	0.091 (8)	0.070 (7)	0.084 (7)	0.002 (7)	-0.025 (7)	-0.019 (6)
C5	0.056 (7)	0.081 (8)	0.088 (7)	0.011 (7)	-0.018 (7)	-0.019 (6)
C8	0.055 (6)	0.061 (6)	0.048 (5)	-0.009 (6)	0.004 (6)	-0.005 (5)
C9	0.045 (5)	0.056 (6)	0.086 (7)	0.011 (6)	0.004 (6)	0.018 (6)
C10	0.079 (8)	0.064 (7)	0.095 (8)	-0.004 (7)	0.000 (7)	-0.012 (6)
C11	0.077 (8)	0.064 (7)	0.061 (6)	-0.004 (7)	0.005 (6)	0.005 (5)
C12	0.063 (7)	0.078 (8)	0.066 (6)	-0.016 (7)	-0.003 (6)	0.022 (6)
C13	0.062 (6)	0.062 (5)	0.062 (6)	0.006 (6)	-0.005 (6)	0.010 (5)
C14	0.073 (7)	0.050 (6)	0.102 (7)	0.001 (7)	-0.004 (7)	0.000 (6)
C15	0.080 (8)	0.119 (9)	0.074 (6)	-0.001 (9)	-0.017 (7)	0.019 (7)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C11A—C4	1.761 (11)	C10—C11	1.373 (14)
C11B—C4	1.628 (11)	C10—H10	0.9300
C12—C4	1.931 (10)	C11—C12	1.378 (13)
C13A—C4	1.693 (10)	C11—H11	0.9300
C13B—C4	1.656 (10)	C12—C13	1.382 (14)
O6—C5	1.151 (11)	C12—C15	1.484 (14)
N7—C5	1.367 (13)	C13—H13	0.9300
N7—C8	1.411 (11)	C14—H14A	0.9600
N7—H7N	0.8600	C14—H14B	0.9600
C4—C5	1.503 (13)	C14—H14C	0.9600
C8—C9	1.383 (12)	C15—H15A	0.9600
C8—C13	1.393 (13)	C15—H15B	0.9600
C9—C10	1.449 (14)	C15—H15C	0.9600
C9—C14	1.480 (12)		
C5—N7—C8	125.7 (8)	C10—C9—C14	121.5 (9)
C5—N7—H7N	117.1	C11—C10—C9	121.1 (10)
C8—N7—H7N	117.1	C11—C10—H10	119.5
C5—C4—C11B	106.9 (8)	C9—C10—H10	119.5
C5—C4—C13B	113.0 (8)	C10—C11—C12	122.8 (10)
C11B—C4—C13B	123.5 (8)	C10—C11—H11	118.6
C5—C4—C13A	116.5 (7)	C12—C11—H11	118.6
C11B—C4—C13A	136.1 (7)	C11—C12—C13	116.8 (9)
C13B—C4—C13A	43.0 (5)	C11—C12—C15	120.6 (10)
C5—C4—C11A	115.4 (7)	C13—C12—C15	122.6 (11)

C11B—C4—C11A	32.1 (4)	C12—C13—C8	121.9 (9)
C13B—C4—C11A	131.0 (7)	C12—C13—H13	119.1
C13A—C4—C11A	115.4 (7)	C8—C13—H13	119.1
C5—C4—C12	107.8 (8)	C9—C14—H14A	109.5
C11B—C4—C12	69.8 (7)	C9—C14—H14B	109.5
C13B—C4—C12	61.1 (6)	H14A—C14—H14B	109.5
C13A—C4—C12	101.2 (5)	C9—C14—H14C	109.5
C11A—C4—C12	96.8 (6)	H14A—C14—H14C	109.5
O6—C5—N7	124.5 (10)	H14B—C14—H14C	109.5
O6—C5—C4	123.3 (12)	C12—C15—H15A	109.5
N7—C5—C4	112.1 (10)	C12—C15—H15B	109.5
C9—C8—C13	122.6 (9)	H15A—C15—H15B	109.5
C9—C8—N7	118.0 (9)	C12—C15—H15C	109.5
C13—C8—N7	119.4 (9)	H15A—C15—H15C	109.5
C8—C9—C10	114.8 (9)	H15B—C15—H15C	109.5
C8—C9—C14	123.7 (9)		
C8—N7—C5—O6	-7.0 (17)	C13—C8—C9—C10	-0.2 (13)
C8—N7—C5—C4	176.0 (8)	N7—C8—C9—C10	178.7 (9)
C11B—C4—C5—O6	84.0 (14)	C13—C8—C9—C14	-178.4 (9)
C13B—C4—C5—O6	-55.0 (16)	N7—C8—C9—C14	0.6 (13)
C13A—C4—C5—O6	-102.5 (14)	C8—C9—C10—C11	0.6 (14)
C11A—C4—C5—O6	117.4 (13)	C14—C9—C10—C11	178.8 (9)
C12—C4—C5—O6	10.4 (14)	C9—C10—C11—C12	-1.1 (17)
C11B—C4—C5—N7	-99.0 (10)	C10—C11—C12—C13	1.0 (15)
C13B—C4—C5—N7	121.9 (10)	C10—C11—C12—C15	179.7 (10)
C13A—C4—C5—N7	74.5 (11)	C11—C12—C13—C8	-0.6 (14)
C11A—C4—C5—N7	-65.6 (11)	C15—C12—C13—C8	-179.3 (9)
C12—C4—C5—N7	-172.6 (7)	C9—C8—C13—C12	0.3 (14)
C5—N7—C8—C9	-127.3 (10)	N7—C8—C13—C12	-178.7 (9)
C5—N7—C8—C13	51.7 (12)		

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>
N7—H7N...O6 <sup>i</sup>	0.86	2.12	2.984 (11)	178

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

Fig. 1

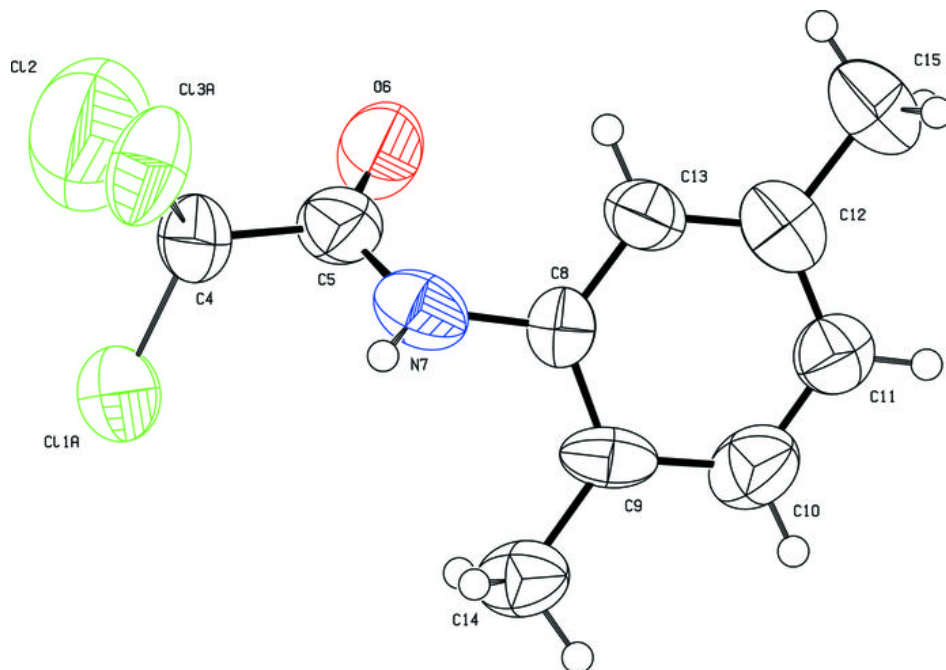


Fig. 2

