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2,2,2-Trichloro-*N*-(2-methylphenyl)acetamide

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Key indicators

Single-crystal X-ray study
T = 304 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.131
Data-to-parameter ratio = 10.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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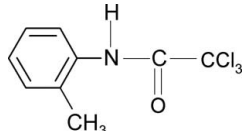
The conformation of the N—H bond in the structure of the title compound (2MPTCA), $C_9H_8Cl_3NO$, is *syn* to the *ortho*-methyl substituent, in contrast to the *anti* conformation observed for the side-chain-unsubstituted *N*-(2-methylphenyl)acetamide, with somewhat slightly different bond parameters. Molecules of 2MPTCA are linked into a chain through N—H···O hydrogen bonds.

Comment

As part of a study to systematize the effect of the substituents on the solid-state structures of *N*-aromatic amides (Gowda *et al.*, 2004, 2006; Gowda, Kozisek, Svoboda & Fuess, 2007; Gowda *et al.*, 2007*a*,*b*; Gowda, Paulus *et al.*, 2007), the structure of 2,2,2-trichloro-*N*-(2-methylphenyl)acetamide (2MPTCA), (I), has been determined. The conformation of the N—H bond in the structure is *syn* to the *ortho*-methyl substituent (Fig. 1), similar to that observed for 2,2,2-trimethyl-*N*-(2-methylphenyl)acetamide (2MPTMA) (Gowda, Kožíšek, Tokarčík & Fuess, 2007*b*), but in contrast to the *anti* conformation observed for the side-chain-unsubstituted *N*-(2-methylphenyl)acetamide (2MPA) (Gowda *et al.*, 2007*a*). The bond parameters of 2MPTCA are similar to those of 2MPA, 2MPTMA and the ring-unsubstituted 2,2,2-trichloro-*N*-phenylacetamide (Dou *et al.*, 1994). The molecules in 2MPTCA are linked into chains through N—H···O hydrogen bonding (Table 1).



(I)

The title compound exhibits positional disorder of the $-CCl_3$ group which was modelled using two sets of atomic sites with refined occupancies of 0.61 (1) and 0.39 (1). The disorder is such that the minor component of atom C4 is displaced 0.33 (2) Å from the major position, and minor atoms Cl1, Cl2 and Cl3 are rotated 6 (1), 28 (2) and 28 (2)°, respectively, with respect to the major orientations.

Experimental

The title compound was prepared according to a literature method (Gowda *et al.*, 2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2003). Single crystals of the title compound were obtained by slow evaporation of an ethanol solution and used for X-ray diffraction at room temperature.

Crystal data

$C_9H_8Cl_3NO$
M_r = 252.51
Orthorhombic, $P2_12_12_1$
a = 10.150 (2) Å
b = 10.216 (2) Å
c = 10.817 (2) Å
V = 1121.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.78 mm^{-1}
T = 304 (2) K
0.55 × 0.16 × 0.13 mm

Data collection

Stoe STADI4 four-circle diffractometer
Absorption correction: analytical (Clark & Reid, 1995)
T_{min} = 0.675, T_{max} = 0.907
1893 measured reflections
1683 independent reflections
1257 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
S = 1.04
1683 reflections
159 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: (Flack, 1983), 396 Friedel pairs
Flack parameter: −0.19 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7···O6^{i}	0.78 (5)	2.25 (5)	2.964 (4)	153 (5)

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

All H atoms attached to C atoms were positioned geometrically and treated as riding, with C—H = 0.93 (CH aromatic) or 0.96Å (CH_3) and U_{iso}(H) = 1.5U_{eq}(CH_3). The H atom attached to the N atom was found in a Fourier map and was refined freely, with U_{iso}(H) = 1.2U_{eq}(NH). In the refinement of the disordered –CCl_3 group, the C—Cl bond lengths were restrained to 1.75 (1) Å and the tetrahedral angle was maintained with restraints on the Cl···Cl distances of 2.88 (5) Å within a particular component (Cl1···Cl2, etc.). All disordered atoms were refined as anisotropic using displacement restraints and constraints; namely rigid-bond approximation for C—Cl bonds, and exact equality of displacement parameters for the pairs Cl2*A*/Cl2*B* and Cl3*A*/Cl3*B*.

Data collection: *STADI4* (Stoe & Cie, 1987); cell refinement: *STADI4*; 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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

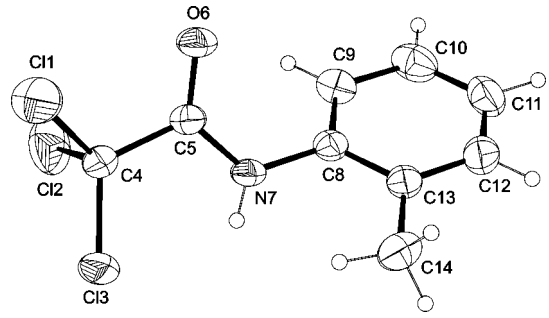


Figure 1
The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radius. Only one disorder component is shown.

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