Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,4,6-Trichloroaniline

B. Thimme Gowda,[a]* Ingrid Svoboda[b] and Hartmut Fuess[b]

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

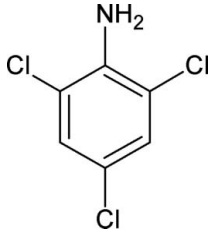
Received 23 June 2007; accepted 25 June 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.022; wR factor = 0.052; data-to-parameter ratio = 12.9.

The cell parameters of the title compound, $C_6H_4Cl_3N$, have already been determined [Schlemper & Konnert (1967). *Acta Cryst.* **22**, 918; Andrianov *et al.* (1971). *Zh. Strukt. Khim.* **12**, 736–737], but no coordinates were available. There are two molecules in the asymmetric unit. The molecules are linked through N—H···N and N—H···Cl hydrogen bonds.

Related literature

For related literature, see: Andrianov *et al.* (1971); Gowda *et al.* (2000, 2004, 2006); Gowda, Foro & Fuess (2007); Gowda, Paulus, Svoboda & Fuess (2007); Schlemper & Konnert (1967).



Experimental

Crystal data

$C_6H_4Cl_3N$
M_r = 196.45
Monoclinic, $P2_1$
a = 13.1933 (9) Å
b = 3.7913 (3) Å
c = 15.774 (1) Å
β = 111.546 (8)°
V = 733.88 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 100 (2) K
0.40 × 0.06 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur single-crystal X-ray diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.654, T_{max} = 0.955
6014 measured reflections
2640 independent reflections
2394 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.052
S = 1.06
2640 reflections
205 parameters
1 restraint
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 909 Friedel pairs
Flack parameter: 0.05 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N1^{i}	0.81 (3)	2.48 (3)	3.229 (3)	155 (2)
N1—H1A···Cl1	0.81 (3)	2.61 (3)	2.983 (2)	110 (2)
N1—H1B···Cl3	0.84 (3)	2.60 (3)	2.991 (2)	110 (2)
N2—H2A···Cl1	0.85 (3)	2.78 (3)	3.542 (2)	150 (2)
N2—H2B···Cl6^{ii}	0.83 (3)	2.85 (3)	3.626 (2)	156 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3* (Farrugia, 1997); 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: BT2410).

References

Andrianov, V. G., Korotkevich, A. F. & Struchkov, Yu. T. (1971). *Zh. Strukt. Khim.* **12**, 736–737.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gowda, B. T., Foro, S. & Fuess, H. (2007). *Acta Cryst.* E**63**, o1981–o1982.
Gowda, B. T., Kozisek, J. & Fuess, H. (2006). *Z. Naturforsch. Teil A*, **61**, 588–594.
Gowda, B. T., Paulus, H., Svoboda, I. & Fuess, H. (2007). *Z. Naturforsch. Teil A*, **62**, 331–337.
Gowda, B. T., Svoboda, I. & Fuess, H. (2000). *Z. Naturforsch. Teil A*, **55**, 779–790.
Gowda, B. T., Svoboda, I. & Fuess, H. (2004). *Z. Naturforsch. Teil A*, **59**, 845–852.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Schlemper, E. O. & Konnert, J. (1967). *Acta Cryst.* **22**, 918.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 doi:10.1107/S1600536807030929

supplementary materials

Acta Cryst. (2007). E**63**, o3347 [**doi:10.1107/S1600536807030929**]

2,4,6-Trichloroaniline

B. T. Gowda, I. Svoboda and H. Fuess

Comment

In the present work, the structure of trichloroaniline has been determined at 100 K, as part of our study of the effect of ring and side chain substitutions on the solid state structures of chemically and biologically important compounds (Gowda *et al.*, 2000; Gowda *et al.*, 2004; Gowda *et al.*, 2006; Gowda, Foro, Fuess, 2007; Gowda, Paulus, Svoboda & Fuess, 2007). The cell pararameters of the title compound have already been determined (Schlemper & Konnert, 1967; Andrianov *et al.*, 1971). There are two molecules in the asymmetric unit. The molecules are linked through N—H···N and N—H···Cl hydrogen bonds (Table 1 & Fig. 2).

Experimental

Single crystals of the title compound were obtained from a slow evaporation of an ethanolic solution of the analytical grade commercial sample.

Refinement

The H atoms were located in difference map and their positions refined, with U_{iso}(H) = 1.2 U_{eq} (parent atom).

Figures

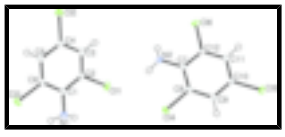


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

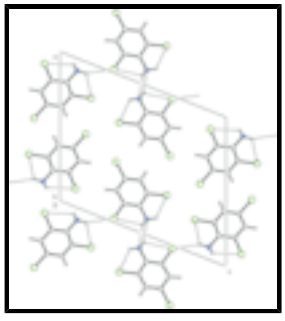


Fig. 2. The crystal packing of the title compound showing the hydrogen bonds as dashed lines.

2,4,6-Trichloroaniline

Crystal data

$C_6H_4Cl_3N$ F_{000} = 392

M_r = 196.45	D_x = 1.778 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2998 reflections
a = 13.1933 (9) Å	θ = 2.8–26.5º
b = 3.7913 (3) Å	μ = 1.16 mm^{-1}
c = 15.774 (1) Å	T = 100 (2) K
β = 111.546 (8)º	Needle, colourless
V = 733.88 (9) Å^3	0.40 × 0.06 × 0.04 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur single-crystal X-ray diffractometer with a Sapphire CCD detector	2640 independent reflections
Radiation source: Enhance (Mo) X-ray Source	2394 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
Detector resolution: 8.4012 pixels mm^{-1}	θ_{max} = 26.4º
T = 100(2) K	θ_{min} = 2.5º
Rotation method data acquisition using ω and φ scans	h = −16→16
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	k = −3→4
T_{min} = 0.654, T_{max} = 0.955	l = −19→19
6014 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	Only H-atom coordinates refined
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 0.0195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.052	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
2640 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
205 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 909 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.05 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.17763 (17)	0.1364 (8)	−0.48017 (15)	0.0136 (5)
C2	−0.15064 (17)	0.1814 (7)	−0.38618 (15)	0.0136 (5)
C3	−0.21934 (19)	0.0893 (7)	−0.34146 (16)	0.0161 (5)
H3	−0.1964 (19)	0.127 (8)	−0.2782 (16)	0.019*
C4	−0.32004 (18)	−0.0566 (7)	−0.39203 (16)	0.0145 (5)
C5	−0.35067 (17)	−0.1116 (8)	−0.48459 (15)	0.0139 (5)
H5	−0.4162 (19)	−0.201 (7)	−0.5161 (16)	0.017*
C6	−0.27979 (18)	−0.0155 (7)	−0.52667 (14)	0.0145 (5)
C7	0.18568 (16)	−0.1233 (8)	−0.04221 (14)	0.0123 (5)
C8	0.26254 (17)	−0.2849 (7)	−0.07128 (14)	0.0137 (5)
C9	0.36317 (17)	−0.4004 (8)	−0.01229 (14)	0.0130 (5)
H9	0.4118 (18)	−0.505 (7)	−0.0361 (15)	0.016*
C10	0.38899 (16)	−0.3535 (7)	0.08034 (14)	0.0129 (5)
C11	0.31648 (17)	−0.1988 (7)	0.11387 (15)	0.0124 (5)
H11	0.3328 (18)	−0.175 (7)	0.1782 (16)	0.015*
C12	0.21624 (17)	−0.0889 (7)	0.05230 (15)	0.0127 (5)
N1	−0.10582 (16)	0.2177 (6)	−0.52298 (15)	0.0183 (5)
H1A	−0.058 (2)	0.357 (9)	−0.4968 (17)	0.022*
H1B	−0.138 (2)	0.262 (7)	−0.5786 (17)	0.022*
N2	0.08434 (16)	−0.0177 (6)	−0.10248 (14)	0.0167 (5)
H2A	0.077 (2)	0.014 (8)	−0.1578 (18)	0.020*
H2B	0.0519 (19)	0.132 (8)	−0.0836 (16)	0.020*
Cl1	−0.02316 (4)	0.36022 (18)	−0.32306 (4)	0.01969 (14)
Cl2	−0.40873 (4)	−0.17199 (18)	−0.33787 (4)	0.01946 (15)
Cl3	−0.31863 (5)	−0.09196 (19)	−0.64317 (4)	0.02145 (16)
Cl4	0.22936 (4)	−0.34462 (19)	−0.18807 (3)	0.01808 (15)
Cl5	0.51505 (4)	−0.49705 (17)	0.15626 (4)	0.01731 (15)
Cl6	0.12272 (4)	0.09515 (17)	0.09410 (4)	0.01712 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0135 (10)	0.0103 (14)	0.0163 (11)	0.0037 (11)	0.0047 (9)	0.0008 (10)
C2	0.0108 (10)	0.0084 (14)	0.0167 (11)	−0.0006 (11)	−0.0007 (9)	0.0011 (10)
C3	0.0191 (11)	0.0136 (15)	0.0128 (11)	0.0024 (11)	0.0027 (9)	0.0003 (11)
C4	0.0155 (11)	0.0084 (15)	0.0228 (12)	0.0008 (11)	0.0105 (10)	0.0019 (10)
C5	0.0109 (10)	0.0118 (15)	0.0165 (11)	−0.0002 (12)	0.0020 (9)	0.0004 (11)
C6	0.0173 (11)	0.0131 (15)	0.0100 (10)	0.0032 (11)	0.0014 (9)	−0.0015 (10)
C7	0.0127 (10)	0.0080 (13)	0.0165 (11)	−0.0026 (11)	0.0059 (9)	0.0025 (10)

C8	0.0162 (11)	0.0142 (15)	0.0117 (10)	−0.0056 (11)	0.0061 (9)	−0.0018 (10)
C9	0.0132 (10)	0.0106 (14)	0.0178 (11)	−0.0026 (11)	0.0087 (9)	−0.0017 (10)
C10	0.0091 (10)	0.0115 (14)	0.0162 (11)	0.0002 (11)	0.0024 (9)	0.0027 (11)
C11	0.0144 (11)	0.0091 (15)	0.0132 (11)	−0.0030 (10)	0.0047 (9)	−0.0001 (10)
C12	0.0135 (10)	0.0093 (15)	0.0194 (11)	−0.0017 (11)	0.0109 (9)	−0.0009 (10)
N1	0.0165 (10)	0.0214 (15)	0.0166 (10)	−0.0027 (10)	0.0056 (9)	−0.0021 (10)
N2	0.0134 (10)	0.0205 (14)	0.0165 (10)	0.0031 (10)	0.0060 (8)	0.0023 (10)
Cl1	0.0148 (3)	0.0204 (4)	0.0189 (3)	−0.0034 (3)	0.0004 (2)	0.0003 (3)
Cl2	0.0201 (3)	0.0214 (4)	0.0196 (3)	−0.0022 (3)	0.0105 (2)	0.0010 (3)
Cl3	0.0260 (3)	0.0255 (4)	0.0116 (3)	−0.0037 (3)	0.0056 (2)	−0.0032 (3)
Cl4	0.0162 (3)	0.0254 (4)	0.0120 (3)	−0.0030 (3)	0.0044 (2)	−0.0027 (3)
Cl5	0.0130 (3)	0.0201 (4)	0.0163 (3)	0.0027 (3)	0.0025 (2)	0.0003 (2)
Cl6	0.0166 (3)	0.0168 (4)	0.0220 (3)	0.0013 (3)	0.0119 (2)	−0.0020 (3)

Geometric parameters (Å, °)

C1—N1	1.385 (3)	C7—C12	1.400 (3)
C1—C6	1.400 (3)	C8—C9	1.383 (3)
C1—C2	1.402 (3)	C8—Cl4	1.745 (2)
C2—C3	1.382 (3)	C9—C10	1.385 (3)
C2—Cl1	1.746 (2)	C9—H9	0.94 (2)
C3—C4	1.389 (3)	C10—C11	1.382 (3)
C3—H3	0.94 (2)	C10—Cl5	1.742 (2)
C4—C5	1.380 (3)	C11—C12	1.386 (3)
C4—Cl2	1.739 (2)	C11—H11	0.96 (2)
C5—C6	1.380 (3)	C12—Cl6	1.744 (2)
C5—H5	0.89 (2)	N1—H1A	0.81 (3)
C6—Cl3	1.741 (2)	N1—H1B	0.84 (2)
C7—N2	1.385 (3)	N2—H2A	0.85 (3)
C7—C8	1.398 (3)	N2—H2B	0.83 (3)
N1—C1—C6	122.4 (2)	C9—C8—C7	123.41 (19)
N1—C1—C2	122.3 (2)	C9—C8—Cl4	118.41 (17)
C6—C1—C2	115.2 (2)	C7—C8—Cl4	118.17 (16)
C3—C2—C1	123.3 (2)	C8—C9—C10	118.2 (2)
C3—C2—Cl1	118.81 (17)	C8—C9—H9	119.4 (14)
C1—C2—Cl1	117.87 (18)	C10—C9—H9	122.4 (14)
C2—C3—C4	118.4 (2)	C11—C10—C9	121.4 (2)
C2—C3—H3	119.1 (15)	C11—C10—Cl5	119.34 (16)
C4—C3—H3	122.4 (15)	C9—C10—Cl5	119.23 (17)
C5—C4—C3	121.0 (2)	C10—C11—C12	118.41 (19)
C5—C4—Cl2	119.55 (18)	C10—C11—H11	121.7 (14)
C3—C4—Cl2	119.49 (17)	C12—C11—H11	119.8 (14)
C6—C5—C4	118.8 (2)	C11—C12—C7	123.1 (2)
C6—C5—H5	121.5 (15)	C11—C12—Cl6	118.73 (17)
C4—C5—H5	119.6 (15)	C7—C12—Cl6	118.21 (16)
C5—C6—C1	123.3 (2)	C1—N1—H1A	116.0 (18)
C5—C6—Cl3	118.36 (17)	C1—N1—H1B	112.1 (17)
C1—C6—Cl3	118.35 (17)	H1A—N1—H1B	114 (3)
N2—C7—C8	122.3 (2)	C7—N2—H2A	117.7 (17)

N2—C7—C12	122.1 (2)	C7—N2—H2B	116.6 (17)
C8—C7—C12	115.46 (19)	H2A—N2—H2B	113 (3)
N1—C1—C2—C3	−177.4 (3)	N2—C7—C8—C9	−178.4 (3)
C6—C1—C2—C3	−0.9 (4)	C12—C7—C8—C9	−1.0 (4)
N1—C1—C2—Cl1	2.1 (4)	N2—C7—C8—Cl4	1.5 (4)
C6—C1—C2—Cl1	178.57 (19)	C12—C7—C8—Cl4	178.84 (19)
C1—C2—C3—C4	0.3 (4)	C7—C8—C9—C10	0.0 (4)
Cl1—C2—C3—C4	−179.3 (2)	Cl4—C8—C9—C10	−179.8 (2)
C2—C3—C4—C5	0.6 (4)	C8—C9—C10—C11	0.6 (4)
C2—C3—C4—Cl2	−179.5 (2)	C8—C9—C10—Cl5	179.9 (2)
C3—C4—C5—C6	−0.7 (4)	C9—C10—C11—C12	−0.2 (4)
Cl2—C4—C5—C6	179.4 (2)	Cl5—C10—C11—C12	−179.51 (19)
C4—C5—C6—C1	−0.1 (4)	C10—C11—C12—C7	−0.8 (4)
C4—C5—C6—Cl3	179.1 (2)	C10—C11—C12—Cl6	178.2 (2)
N1—C1—C6—C5	177.3 (3)	N2—C7—C12—C11	178.8 (2)
C2—C1—C6—C5	0.8 (4)	C8—C7—C12—C11	1.4 (4)
N1—C1—C6—Cl3	−1.9 (4)	N2—C7—C12—Cl6	−0.3 (4)
C2—C1—C6—Cl3	−178.4 (2)	C8—C7—C12—Cl6	−177.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N1^{i}	0.81 (3)	2.48 (3)	3.229 (3)	155 (2)
N1—H1A···Cl1	0.81 (3)	2.61 (3)	2.983 (2)	110 (2)
N1—H1B···Cl3	0.84 (3)	2.60 (3)	2.991 (2)	110 (2)
N2—H2A···Cl1	0.85 (3)	2.78 (3)	3.542 (2)	150 (2)
N2—H2B···Cl6^{ii}	0.83 (3)	2.85 (3)	3.626 (2)	156 (2)

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

Fig. 1

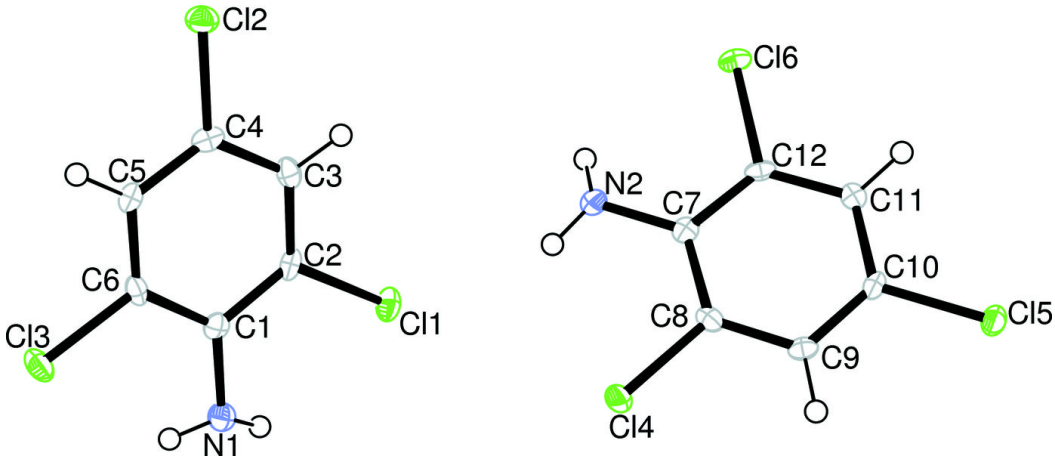


Fig. 2