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N,*N*,4-Trichlorobenzenesulfonamide

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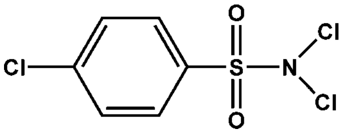
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.129; data-to-parameter ratio = 16.0.

The structure of the title compound (NNDC4CBSA), $C_6H_4Cl_3NO_2S$, resembles that of *N*,*N*-dichloro-4-methylbenzenesulfonamide (NNDC4MBSA). In particular, the structure is relatively simple, unlike those of the sodium salts of *N*-chloroarylsulfonamides. Furthermore, the S—N distance of 1.759 (5) Å is consistent with the value of 1.735 (5) Å observed with NNDC4MBSA, the value for an S—N single bond. The molecules are packed into zigzag chains in the direction of the *a* axis.

Related literature

For related structures, see: Gowda *et al.* (2002, 2005, 2007); Gowda, D'Souza & Kumar (2003); Gowda, Jyothi & Damodara (2003); Gowda, Jyothi, Kozisek & Fuess (2003); Gowda & Shetty (2004); Gowda & Kumar (2003); Minkwitz *et al.* (1997); Olmstead & Power (1986); George *et al.* (2000).



Experimental

Crystal data

$C_6H_4Cl_3NO_2S$
M_r = 260.51
Monoclinic, $P2_1/c$
a = 7.028 (2) Å
b = 16.059 (3) Å
c = 10.492 (3) Å
β = 123.542 (17)°
V = 987.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.10 mm^{-1}
T = 299 (2) K
0.50 × 0.30 × 0.16 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.609, $T_{\max}$ = 0.843
2909 measured reflections
1893 independent reflections
1463 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.028
3 standard reflections
frequency: 180 min
intensity decay: 18.2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.129
S = 1.06
1893 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.47 e Å^{-3}
$\Delta\rho_{\min}$ = −0.51 e Å^{-3}

The crystal structure was measured on a Stoe diffractometer in July 2000. There was a decrease of intensity as the compound in the X-ray beam was unstable. The software rescaled all the data in the correct way, hence the high intensity decay.

Data collection: *STADI4* (Stoe & Cie, 1996); cell refinement: *STADI4*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2172).

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supplementary materials

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***N*,*N*,4-Trichlorobenzenesulfonamide**

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Comment

The chemistry of N-halo compounds is of interest as they show distinct physical, chemical and biological properties. Many of these compounds exhibit pharmacological, fungicidal and herbicidal activities due to their oxidizing action in aqueous, partial aqueous and non-aqueous media. Thus N-halo arylsulfonamides are of interest in synthetic, mechanistic, analytical and biological chemistry (Gowda *et al.*, 2002, 2005, 2007; Gowda, D'Souza & Kumar, 2003; Gowda, Jyothi & Damodara, 2003; Gowda, Jyothi, Kozisek & Fuess, 2003; Gowda & Shetty, 2004; Gowda & Kumar, 2003). In the present work, the structure of *N*,*N*,4-trichlorobenzenesulfonamide (NNDC4MBSA) has been determined to explore the substituent effects on the solid state structures of sulfonamides and *N*-haloarylsulfonamides (Gowda *et al.*, 2003*c*; 2007). The structure of NNDC4CBSA (Fig. 1) resembles that of *N*,*N*-dichloro-4-methylbenzenesulfonamide (Minkwitz *et al.*, 1997). The structures of arylsulfonamides and *N*,*N*-dichloroarylsulfonamides are relatively simple (Minkwitz *et al.*, 1997; Gowda, Jyothi, Kozisek & Fuess, 2003; Gowda *et al.*, 2007), while those of the sodium salts of *N*-chloroarylsulfonamides are relatively complex (Olmstead & Power, 1986; George *et al.*, 2000). Thus comparison of the first two categories of compounds with the latter is not straight forward. Hence comparison is made between arylsulfonamides and *N*,*N*-dichloroarylsulfonamides (Minkwitz *et al.*, 1997; Gowda, Jyothi, Kozisek & Fuess, 2003). The C—*S* and *S*—O bond lengths slightly decrease on N-chlorination of arylsulfonamides to *N*,*N*-dichloroarylsulfonamides, while S—N bond lengths slightly increase on N-chlorination. The O—S—O, O1—S—C1 and O2—S—C1 bond angles increase on N-chlorination, while O1—S—N, O2—S—N and N—S—C1 bond angles generally decrease on N-chlorination.

Experimental

The *N*,*N*-dichloro-4-chlorobenzenesulfonamide(NNDC4CBSA) was prepared by further chlorination of the sodium salt of *N*-chloro-4-chlorobenzenesulfonamide (NaNC4CBSA) (Gowda *et al.*, 2003 *b*). NaNC4CBSA was in turn prepared by the N-chlorination of 4-chlorobenzenesulfonamide (4CBSA)(Gowda *et al.*, 2003 *a*). 4CBSA was prepared by the chlorosulphonation of chlorobenzene to 4-chlorobenzenesulfonylchloride and subsequent conversion of the latter to 4CBSA (Gowda *et al.*, 2002). Pure chlorine gas was passed through clear solution of 4CBSA in 4*M* NaOH at 70° C for about 1 hr. The precipitated NaNC4CBSA was filtered, washed, dried and recrystallized from water. Further, pure chlorine gas was bubbled through clear aqueous solution of NaNC4CBSA for about 1 hr. NNDC4CBSA precipitated was filtered, washed, dried and recrystallized from pure acetic acid. Purity of the compound was checked by determining its melting point and by estimating, iodometrically, the amount of active chlorine present in it. The compound was further characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2003 *a*). Single crystals of NNDC4CBSA were obtained by recrystallization from its water free chloroform or pure acetic acid solution and used for X-ray diffraction studies at room temperature.

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$

Figures

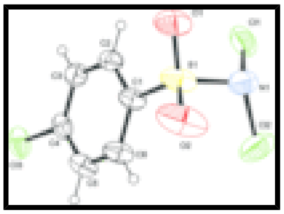

Fig. 1. *ORTEP* view of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

N,*N*,4-Trichlorobenzenesulfonamide

Crystal data

$C_6H_4Cl_3NO_2S$

$M_r = 260.51$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.028$ (2) Å

$b = 16.059$ (3) Å

$c = 10.492$ (3) Å

$\beta = 123.542$ (17)°

$V = 987.0$ (5) Å^3

$Z = 4$

$F_{000} = 520$

$D_x = 1.753$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 30 reflections

$\theta = 16.8–19.9°$

$\mu = 1.10$ mm^{-1}

$T = 299$ (2) K

Prism, colorless

0.50 × 0.30 × 0.16 mm

Data collection

Stoe Stadi-4 four-circle diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299(2)$ K

profile fitted scan $2\theta/\omega$ 1/1

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{min} = 0.609$, $T_{max} = 0.843$

2909 measured reflections

1893 independent reflections

1463 reflections with $I > 2\sigma(I)$

$R_{int} = 0.028$

$\theta_{max} = 26.1°$

$\theta_{min} = 5.3°$

$h = -1\rightarrow8$

$k = -19\rightarrow0$

$l = -12\rightarrow11$

3 standard reflections

every 180 min

intensity decay: 18.2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.129$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 1.1354P]$
where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.06$	$(\Delta/\sigma)_{\max} = 0.003$
1893 reflections	$\Delta\rho_{\max} = 0.47$ e Å^{-3}
118 parameters	$\Delta\rho_{\min} = -0.51$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.4426 (6)	0.10921 (19)	0.7590 (4)	0.0581 (8)
C2	0.3401 (6)	0.1415 (2)	0.6138 (4)	0.0586 (8)
H2	0.3309	0.1988	0.5986	0.07*
C3	0.2521 (6)	0.0882 (2)	0.4923 (4)	0.0624 (8)
H3	0.1827	0.1087	0.3933	0.075*
C4	0.2678 (5)	0.0029 (2)	0.5187 (4)	0.0587 (8)
C5	0.3736 (7)	−0.0292 (2)	0.6638 (5)	0.0681 (9)
H5	0.3859	−0.0865	0.6791	0.082*
C6	0.4603 (7)	0.0237 (2)	0.7850 (4)	0.0712 (10)
H6	0.5304	0.0029	0.8839	0.085*
N1	0.8345 (7)	0.1993 (2)	0.9797 (4)	0.0989 (13)
O1	0.4435 (7)	0.25683 (17)	0.8619 (4)	0.1012 (10)
O2	0.5656 (10)	0.1377 (2)	1.0389 (4)	0.1493 (19)
S1	0.5485 (2)	0.17781 (6)	0.91307 (12)	0.0862 (4)
Cl1	0.8487 (2)	0.24204 (7)	0.83545 (15)	0.1027 (5)
Cl2	0.9967 (3)	0.10945 (9)	1.04611 (15)	0.1527 (9)
Cl3	0.15203 (16)	−0.06413 (7)	0.36448 (12)	0.0834 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.084 (2)	0.0433 (15)	0.0655 (19)	0.0008 (15)	0.0527 (18)	−0.0033 (14)
C2	0.0661 (19)	0.0439 (16)	0.068 (2)	0.0034 (14)	0.0379 (17)	0.0018 (14)
C3	0.0604 (19)	0.0605 (19)	0.0627 (19)	0.0019 (15)	0.0317 (16)	−0.0036 (15)
C4	0.0518 (17)	0.0589 (18)	0.076 (2)	−0.0108 (14)	0.0421 (16)	−0.0190 (16)
C5	0.094 (3)	0.0426 (16)	0.090 (3)	−0.0070 (16)	0.065 (2)	−0.0049 (16)
C6	0.113 (3)	0.0488 (18)	0.071 (2)	0.0012 (18)	0.063 (2)	0.0061 (16)

N1	0.124 (3)	0.0581 (18)	0.0560 (18)	0.009 (2)	0.0132 (19)	−0.0120 (15)
O1	0.150 (3)	0.0591 (15)	0.110 (2)	0.0228 (17)	0.081 (2)	−0.0109 (15)
O2	0.319 (6)	0.086 (2)	0.108 (3)	−0.009 (3)	0.159 (4)	−0.0083 (19)
S1	0.1578 (11)	0.0511 (5)	0.0720 (6)	0.0065 (6)	0.0776 (7)	−0.0054 (4)
Cl1	0.1127 (9)	0.0792 (7)	0.0955 (8)	−0.0364 (6)	0.0445 (7)	−0.0213 (6)
Cl2	0.1630 (15)	0.0953 (9)	0.0810 (8)	0.0433 (9)	−0.0073 (9)	−0.0078 (7)
Cl3	0.0699 (6)	0.0842 (7)	0.0971 (7)	−0.0202 (5)	0.0468 (5)	−0.0418 (6)

Geometric parameters (Å, °)

C1—C2	1.377 (4)	C5—C6	1.361 (5)
C1—C6	1.393 (4)	C5—H5	0.9300
C1—S1	1.747 (3)	C6—H6	0.9300
C2—C3	1.367 (5)	N1—Cl1	1.714 (5)
C2—H2	0.9300	N1—Cl2	1.729 (4)
C3—C4	1.390 (5)	N1—S1	1.759 (5)
C3—H3	0.9300	O1—S1	1.416 (3)
C4—C5	1.373 (5)	O2—S1	1.413 (3)
C4—Cl3	1.727 (3)		

C2—C1—C6	121.6 (3)	C4—C5—H5	120.3
C2—C1—S1	118.8 (2)	C5—C6—C1	119.1 (3)
C6—C1—S1	119.6 (3)	C5—C6—H6	120.4
C3—C2—C1	119.1 (3)	C1—C6—H6	120.4
C3—C2—H2	120.4	Cl1—N1—Cl2	110.0 (3)
C1—C2—H2	120.4	Cl1—N1—S1	109.80 (18)
C2—C3—C4	119.1 (3)	Cl2—N1—S1	110.5 (2)
C2—C3—H3	120.5	O2—S1—O1	121.3 (2)
C4—C3—H3	120.5	O2—S1—C1	110.49 (19)
C5—C4—C3	121.7 (3)	O1—S1—C1	110.38 (18)
C5—C4—Cl3	119.4 (3)	O2—S1—N1	103.3 (3)
C3—C4—Cl3	118.9 (3)	O1—S1—N1	102.5 (2)
C6—C5—C4	119.4 (3)	C1—S1—N1	107.51 (17)
C6—C5—H5	120.3		

C6—C1—C2—C3	0.5 (5)	C6—C1—S1—O2	−21.4 (4)
S1—C1—C2—C3	−178.5 (3)	C2—C1—S1—O1	20.7 (4)
C1—C2—C3—C4	0.3 (5)	C6—C1—S1—O1	−158.4 (3)
C2—C3—C4—C5	−1.4 (5)	C2—C1—S1—N1	−90.4 (3)
C2—C3—C4—Cl3	178.9 (2)	C6—C1—S1—N1	90.6 (3)
C3—C4—C5—C6	1.8 (5)	Cl1—N1—S1—O2	175.7 (2)
Cl3—C4—C5—C6	−178.6 (3)	Cl2—N1—S1—O2	54.2 (3)
C4—C5—C6—C1	−1.0 (6)	Cl1—N1—S1—O1	−57.5 (2)
C2—C1—C6—C5	−0.2 (6)	Cl2—N1—S1—O1	−179.0 (2)
S1—C1—C6—C5	178.9 (3)	Cl1—N1—S1—C1	58.9 (2)
C2—C1—S1—O2	157.6 (4)	Cl2—N1—S1—C1	−62.7 (2)

Fig. 1