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4-Azidobenzenesulfonyl Chloride

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Abstract

The title compound, *p*-$C_6H_4N_3SO_2Cl$, is used as raw material for the esterification of aliphatic and aromatic mono- and polyfunctional alcohols. In this way mono-, bis- and triazido compounds are synthezized, which, for example, are applied in the process of lithographic structuring of polymers.

Experimental

p-Azidobenzene sulfonate reacts with thionyl chloride to form the title compound, which recrystallized from diethyl ether. Further details about the preparation were described by Sauer, Bendig & Heutzenröder (1988). Important compound characteristics are m.p.: 59–61°C; decomposition: 150°C; IR(KBr): 1175(SO_2) 1380(SO_2), 1585(C—N), 2110(N—N) and 2130(N—N) cm^{-1}; UV(solvent dioxane): 284 nm, 19800 l $mol^{-1}cm^{-1}$.

Refinement

The extinction coefficient was within 1 e.s.d. meaningless and was left out of the refinement.

Computing details

Data collection: *IPDS2*.75 (Stoe, 1997); cell refinement: *IPDS2*.75 (Stoe, 1997); data reduction: *IPDS2*.75 (Stoe, 1997); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: XSTEP-2.04 (Stoe, (1996); software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993).

4-Azidobenzenesulfonyl chloride

Crystal data

$C_6H_4ClN_3O_2S$	V = 855.5 (3) Å^3
M_r = 217.63	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$
a = 8.1249 (15) Å	μ = 0.66 mm^{-1}
b = 10.900 (2) Å	T = 180 (2) K
c = 10.132 (2) Å	0.56 × 0.54 × 0.46 mm
β = 107.57 (2)°	

CIF access

Data collection

STOE Ipds diffractometer	1484 independent reflections
Absorption correction: none	1352 reflections with $I > 2\sigma(I)$
7124 measured reflections	$R_{int} = 0.099$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$	134 parameters
$wR(F^2) = 0.135$	All H-atom parameters refined
$S = 1.10$	$\Delta\rho_{max} = 0.39$ e Å^{-3}
1483 reflections	$\Delta\rho_{min} = -0.42$ e Å^{-3}

Acknowledgements

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References

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Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93* a Program for Crystal Structure Refinement. Universität Göttingen, Germany.
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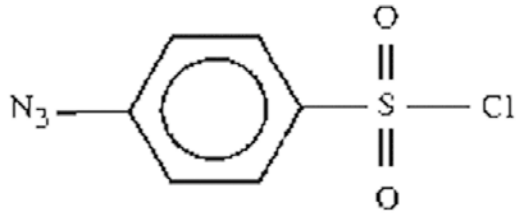
Scheme 1



supplementary materials

4-Azidobenzenesulfonyl chloride

Crystal data

$C_6H_4ClN_3O_2S$	F_{000} = 440
M_r = 217.63	D_x = 1.690 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 333(1) K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71071 Å
a = 8.1249 (15) Å	Cell parameters from 5000 reflections
b = 10.900 (2) Å	θ = 3.2–25.0º
c = 10.132 (2) Å	μ = 0.66 mm^{-1}
β = 107.57 (2)º	T = 180 (2) K
V = 855.5 (3) Å^3	Cube-like, colorless
Z = 4	0.56 × 0.54 × 0.46 mm

Data collection

STOE Ipds diffractometer	1484 independent reflections
Radiation source: fine-focus sealed X-ray tube	1352 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.099
Detector resolution: 6.667 pixels mm^{-1}	θ_{max} = 25.1º
T = 180(2) K	θ_{min} = 2.8º
φ–rotation,φ–total=220°,φ–incr.=1.4° scans	h = −10→9
Absorption correction: none	k = −13→13
7124 measured reflections	l = −12→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.032	All H-atom parameters refined
$wR(F^2)$ = 0.135	Calculated $w = 1/[\sigma^2(F_o^2) + (0.0305P)^2 + 0.1292P]$ where $P = (F_o^2 + 2F_c^2)/3$?
S = 1.10	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
1483 reflections	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
134 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. During data collection the crystal was in cold N2 gas of the Cryostream Cooler (Oxford Cryosystems, 1992) mounted on a φ-axis diffractometer supplied with an area detector.

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 on F^2 for ALL reflections except for 1 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors *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	0.1586 (2)	0.1554 (2)	−0.0185 (2)	0.0246 (4)
C2	0.1897 (3)	0.1278 (2)	−0.1432 (2)	0.0283 (4)
H2	0.164 (4)	0.185 (2)	−0.215 (3)	0.051 (7)*
C3	0.2634 (3)	0.0160 (2)	−0.1557 (2)	0.0288 (4)
H3	0.289 (3)	−0.003 (2)	−0.238 (2)	0.036 (5)*
C4	0.3041 (2)	−0.0662 (2)	−0.0455 (2)	0.0242 (4)
C5	0.2731 (2)	−0.0365 (2)	0.0783 (2)	0.0251 (4)
H5	0.296 (3)	−0.089 (2)	0.142 (3)	0.038 (6)*
C6	0.1991 (3)	0.0749 (2)	0.0918 (2)	0.0256 (4)
H6	0.172 (3)	0.0958 (18)	0.173 (2)	0.030 (5)*
Cl	0.27396 (8)	0.40907 (4)	0.06774 (6)	0.0462 (2)
N41	0.3815 (2)	−0.17792 (14)	−0.0672 (2)	0.0298 (4)
N42	0.4161 (2)	−0.25245 (15)	0.0307 (2)	0.0301 (4)
N43	0.4566 (3)	−0.3279 (2)	0.1101 (2)	0.0435 (5)
O1	−0.0271 (3)	0.3436 (2)	−0.1352 (2)	0.0655 (6)
O2	−0.0117 (2)	0.29354 (12)	0.1059 (2)	0.0428 (4)
S1	0.06459 (7)	0.29709 (4)	−0.00289 (5)	0.0309 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0232 (10)	0.0241 (9)	0.0229 (8)	−0.0011 (7)	0.0017 (7)	−0.0042 (7)
C2	0.0327 (11)	0.0311 (9)	0.0181 (8)	−0.0032 (8)	0.0029 (7)	0.0004 (8)
C3	0.0360 (12)	0.0334 (10)	0.0163 (8)	−0.0048 (8)	0.0070 (8)	−0.0051 (8)
C4	0.0232 (10)	0.0233 (8)	0.0253 (9)	−0.0054 (7)	0.0061 (7)	−0.0061 (7)
C5	0.0294 (11)	0.0251 (9)	0.0196 (8)	−0.0020 (7)	0.0058 (8)	0.0012 (7)
C6	0.0269 (10)	0.0294 (9)	0.0197 (9)	−0.0019 (7)	0.0061 (7)	−0.0036 (7)
Cl	0.0530 (4)	0.0288 (3)	0.0671 (4)	−0.0090 (2)	0.0335 (3)	−0.0133 (2)
N41	0.0366 (10)	0.0264 (8)	0.0282 (8)	0.0002 (7)	0.0124 (7)	−0.0034 (7)
N42	0.0318 (9)	0.0252 (8)	0.0374 (9)	−0.0023 (7)	0.0169 (7)	−0.0067 (8)
N43	0.0561 (13)	0.0303 (9)	0.0506 (11)	0.0061 (9)	0.0261 (10)	0.0062 (9)
O1	0.0787 (15)	0.0642 (11)	0.0363 (9)	0.0428 (11)	−0.0088 (9)	0.0027 (8)
O2	0.0396 (9)	0.0381 (8)	0.0584 (10)	0.0031 (6)	0.0264 (8)	−0.0046 (7)
S1	0.0295 (3)	0.0305 (3)	0.0293 (3)	0.0082 (2)	0.0037 (2)	−0.0003 (2)

Geometric parameters (Å, °)

C1—C6	1.381 (3)	C5—C6	1.380 (3)
C1—C2	1.394 (2)	C5—H5	0.84 (2)
C1—S1	1.752 (2)	C6—H6	0.94 (2)
C2—C3	1.380 (3)	Cl—S1	2.0398 (8)
C2—H2	0.94 (3)	N41—N42	1.247 (2)
C3—C4	1.391 (3)	N42—N43	1.128 (2)
C3—H3	0.94 (2)	O1—S1	1.416 (2)
C4—C5	1.390 (2)	O2—S1	1.419 (2)
C4—N41	1.418 (2)		
C6—C1—C2	122.2 (2)	C6—C5—H5	121.9 (15)
C6—C1—S1	119.43 (14)	C4—C5—H5	118.2 (15)
C2—C1—S1	118.41 (14)	C5—C6—C1	118.9 (2)
C3—C2—C1	118.4 (2)	C5—C6—H6	121.5 (13)
C3—C2—H2	121.2 (15)	C1—C6—H6	119.5 (13)
C1—C2—H2	120.3 (15)	N42—N41—C4	116.14 (15)
C2—C3—C4	120.0 (2)	N43—N42—N41	171.8 (2)
C2—C3—H3	119.6 (14)	O1—S1—O2	121.09 (12)
C4—C3—H3	120.4 (14)	O1—S1—C1	110.45 (9)
C5—C4—C3	120.7 (2)	O2—S1—C1	110.55 (8)
C5—C4—N41	123.3 (2)	O1—S1—Cl	105.22 (11)
C3—C4—N41	116.0 (2)	O2—S1—Cl	104.92 (8)
C6—C5—C4	119.8 (2)	C1—S1—Cl	102.67 (7)
C6—C1—C2—C3	0.1 (3)	C5—C4—N41—N42	2.4 (3)
S1—C1—C2—C3	−179.85 (15)	C3—C4—N41—N42	−179.1 (2)
C1—C2—C3—C4	0.2 (3)	C4—N41—N42—N43	−173.9 (14)
C2—C3—C4—C5	−0.7 (3)	C6—C1—S1—O1	−157.0 (2)
C2—C3—C4—N41	−179.2 (2)	C2—C1—S1—O1	23.0 (2)
C3—C4—C5—C6	0.8 (3)	C6—C1—S1—O2	−20.2 (2)
N41—C4—C5—C6	179.2 (2)	C2—C1—S1—O2	159.7 (2)
C4—C5—C6—C1	−0.5 (3)	C6—C1—S1—Cl	91.3 (2)
C2—C1—C6—C5	0.1 (3)	C2—C1—S1—Cl	−88.8 (2)
S1—C1—C6—C5	179.98 (14)		