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2,4,6-Triisopropylbenzenesulfonamide from single-crystal data

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Key indicators

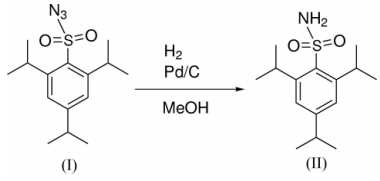
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.136
Data-to-parameter ratio = 19.4

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

Refinement of the title structure, $C_{15}H_{25}NO_2S$, using low-temperature single-crystal X-ray data, confirms the results from powder diffraction data [Tremayne, MacLean, Tang & Glidewell (1999). *Acta Cryst*. B**55**, 1068–1074]. The S═O distances are 1.4252 (18) and 1.4301 (16) Å, and the S—N distance is 1.601 (2) Å.

Comment

The structure of the title compound, (II), has been reported recently by Tremayne *et al.* (1999), who were not able to obtain single crystals and resorted to Monte Carlo structure solution from low-temperature powder diffraction data obtained at 120 K using synchrotron radiation. Their method involved trial structures that were randomly moved within the unit cell and evaluated by a full-profile fitting technique. They employed rotation about the four bonds to the phenyl group as conformational variables, and refined the final model by Rietveld techniques. We have prepared the compound *via* a hydrogenation reaction from the corresponding azide (see scheme), and we were fortunate to obtain single crystals. Determination of the structure afforded the opportunity to evaluate the success of the method of Tremayne *et al.* (1999).



The molecular structure is shown in Fig. 1. Agreement with the previous results is very good. Coordinates of the non-H atoms obtained from single-crystal and powder data differ by small displacements of 0.023 (13)–0.143 (16) Å, the mean value being only 0.081 Å. Some of the differences between the two determinations may be attributed to the 20 K difference in temperature. The torsion angles about the benzene-substituent bonds were correctly predicted by the method of Tremayne *et al.* (1999), showing maximum deviation from the single-crystal results of only 6 (1)°, for the angle about C2—C7. As pointed out by Tremayne *et al.* (1999), the powder data yield longer S—N and S—C distances but shorter S—O distances than the single-crystal data. In addition, the single-crystal data have yielded benzene—C(iPr) distances that are slightly shorter [1.515 (3)–1.529 (3) Å] than those from powder data [1.56 (1)–1.57 (2) Å], as well as C—C(methyl) distances that are slightly longer [1.502 (4)–1.533 (3) *versus* 1.484 (7)–1.502 (7) Å]. The precision of the single-crystal results is one-half to one order of magnitude higher.

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An advantage of the single-crystal data is that H atoms could be located, including those of the NH_2 group, which are involved in hydrogen bonding. Our results confirm that the postulated H-atom positions reported by Tremayne *et al.* (1999), based on intermolecular contacts, are essentially correct. The hydrogen bonding, which those authors have fully discussed, involves centrosymmetric $R_2^2(8)$ rings and spiral chains; geometric details are given in Table 2.

Experimental

2,4,6-Triisopropylbenzenesulfonyl azide, (I), (1.17 g, 3.78 mmol, 3.00 equivalents) was placed in a hydrogenation flask and dissolved in methanol (10 ml). The flask was placed in a Parr hydrogenator for 3 d at 50 psi (1 psi $\simeq$ 6893 Pa) along with 0.30 g Pd/C. The product was purified by column chromatography with 75% hexane/25% ethyl acetate. The compound was dissolved in 90% hexane/10% dichloromethane (10 ml) and allowed to stand undisturbed for 12 h, after which colorless rhombic plates of (II) were collected.

Crystal data

$C_{15}H_{25}NO_2S$
M_r = 283.42
Monoclinic, $P2_1/c$
a = 16.963 (5) Å
b = 8.108 (2) Å
c = 11.752 (3) Å
β = 104.836 (11)°
V = 1562.4 (7) Å^3
Z = 4
D_x = 1.205 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3779 reflections
θ = 2.5–27.5°
μ = 0.21 mm^{-1}
T = 100 K
Rhombic plate, colorless
0.20 × 0.20 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer (with Oxford Cryostream)
ω scans with κ offsets
Absorption correction: multi-scan (*HKL SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.931, T_{max} = 0.990
26 071 measured reflections
3590 independent reflections
2729 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -22 \rightarrow 21$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.136
S = 1.06
3590 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 1.3564P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0046 (12)

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.4252 (18)	S1—N1	1.601 (2)
S1—O1	1.4301 (16)	S1—C1	1.798 (2)
O2—S1—O1	118.63 (13)	O2—S1—C1	109.66 (10)
O2—S1—N1	106.09 (14)	O1—S1—C1	107.32 (9)
O1—S1—N1	107.30 (10)	N1—S1—C1	107.33 (11)
O1—S1—C1—C2	60.87 (17)	C5—C4—C10—C12	−55.6 (3)
C1—C2—C7—C8	115.4 (2)	C5—C6—C13—C15	62.7 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.84 (3)	2.12 (3)	2.947 (3)	170 (3)
N1—H2N···O2^{ii}	0.84 (3)	2.39 (3)	2.844 (3)	114 (2)

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

H atoms attached to C atoms were placed in idealized positions, with C—H distances of 0.95–1.00 Å, and thereafter treated as riding. A torsional parameter was refined for each methyl group. Displacement parameters for H atoms were assigned as $U_{iso} = 1.2U_{eq}$ of the attached atom (1.5 for methyl). The coordinates of H atoms attached to N atoms were freely refined.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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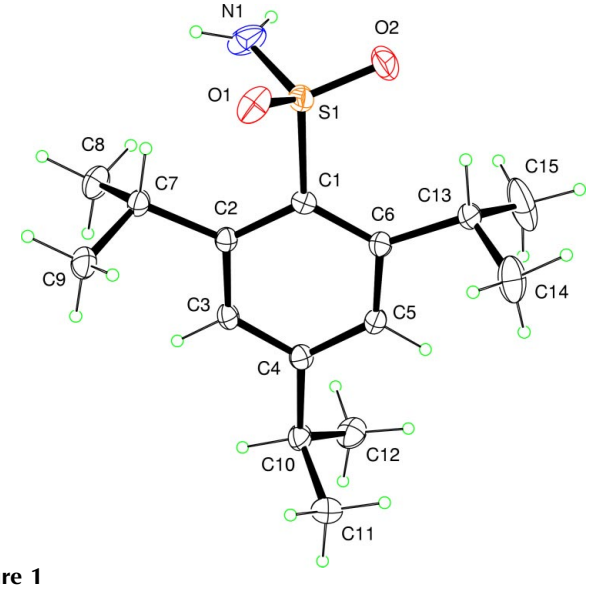

Figure 1
The molecular structure of (II), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level.

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