Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Chloro-2-propyl mesityl sulfone

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Crystalline 2-chloro-2-propyl mesityl sulfone (α-chloroisopropyl mesityl sulfone), $C_{12}H_{17}ClO_2S$, exhibits much less rotational disorder of its α-chloroisopropyl group than that exhibited by the α-bromoisopropyl group in the related crystalline 2-bromo-2-propyl mesityl sulfone.

Received 17 April 2001
Accepted 8 May 2001
Online 15 June 2001

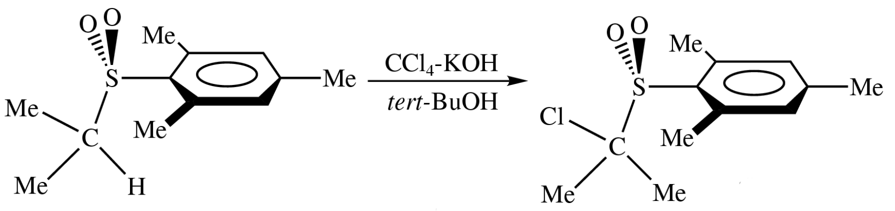
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.040
wR factor = 0.121
Data-to-parameter ratio = 14.3

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

Comment

Our recent observations that the 2-bromo-2-propyl (α-bromoisopropyl) group in crystalline 2-bromo-2-propyl mesityl sulfone exhibits significant rotational disorder (Chan-Yu-King *et al.*, 2001), while the same group in crystalline 2-bromo-2-propyl phenyl sulfone is completely ordered (Robinson *et al.*, 2001) prompted us to examine the X-ray structure of 2-chloro-2-propyl mesityl sulfone, (I).



(I)

The structure of (I) (Fig. 1) shows the two sites of the α-chloroisopropyl group that are somewhat rotationally disordered. Restrained site-occupancy refinement of these sites produced the following values: Cl2 = 0.800 (4), C12 = 0.199 (10), Cl3 = 0.198 (4), C13 = 0.801 (4). Thus, *ca* 80% of the rotamers have their Cl atom near O1 of the sulfonyl group and *ca* 20% have their Cl atom near O2; the third rotamer does not exist in this structure. This small rotational disorder may be associated with the small intra- and intermolecular non-bonding distances. For example, the intramolecular distances O2···O7 and O1···C8 are, respectively, 0.41 and 0.45 Å less than the sum of their van der Waals radii, while C2···C12, C6···C13, C7···C13 and C8···C12 exceed this sum by, respectively, only 0.07, 0.18, 0.10 and 0.13 Å. Intermolecular distances O2···O7 and O2···C9 are larger than the sum of their van der Waals radii by, respectively, only 0.12 and 0.15 Å. The contact distances for the above interactions are given in Table 2. Free rotation of the α-chloroisopropyl group of (I) in solution is apparent from its ^{1}H NMR ($CDCl_3$)

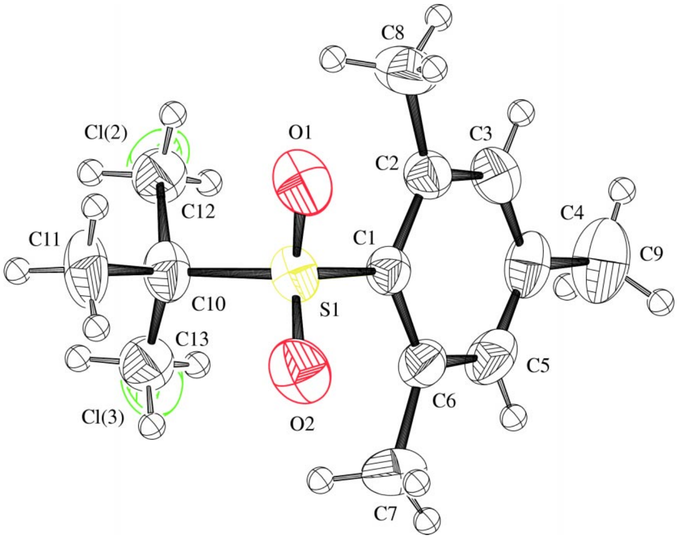


Figure 1
The molecular structure and atom-numbering scheme for (I) with displacement ellipsoids at the 50% probablilty level. The illusion of concurrent positioning of Cl2/C12 Me and Cl3/C13 Me, respectively, reflects the rotational disorder of the α-chloisopropyl group.

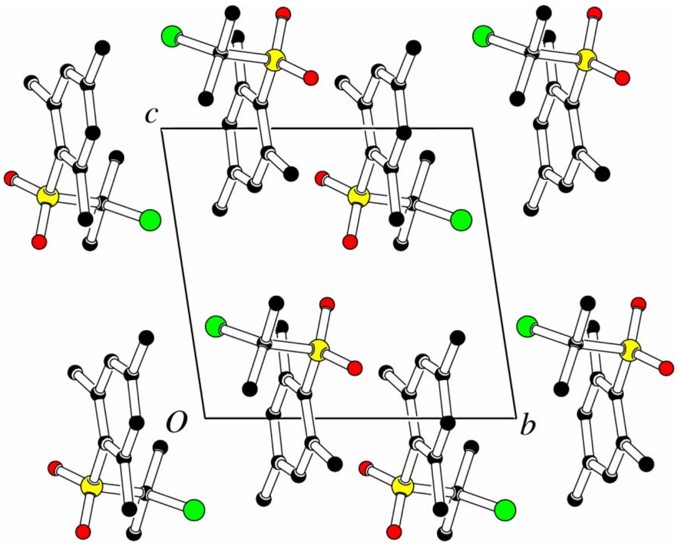


Figure 2
The molecular packing in (I). The disorder is not shown and the H atoms are omitted to improve clarity. Color code: green = Cl, red = O, yellow = S.

spectrum, which exhibits one sharp singlet (6H) for the isopropyl methyl groups.

Experimental

2–Chloro-2-propyl mesityl sulfone, (I), was prepared from mesityl 2-propyl sulfone by treatment with CCl_4-powdered KOH-*tert*-BuOH (Hua, 1979). Column chromatography (silica gel, benzene/choroform) afforded colorless crystals (m.p. 360–362 K). Recrystallization from hexanes provided crystals with the same melting point, and were used in this study.

Crystal data

$C_{12}H_{17}ClO_2S$
$M_r = 260.70$
Triclinic, $P\overline{1}$
$a = 9.237\ (2)$ Å
$b = 9.4592\ (13)$ Å
$c = 8.2079\ (6)$ Å
$\alpha = 99.333\ (9)^\circ$
$\beta = 93.671\ (11)^\circ$
$\gamma = 66.342\ (14)^\circ$
$V = 648.23\ (18)$ Å^3
$Z = 2$
$D_x = 1.336$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 12.5–12.9^\circ$
$\mu = 0.44$ mm^{-1}
$T = 296$ K
Fragment, colorless
0.41 × 0.36 × 0.20 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.853$, $T_{max} = 0.916$
2442 measured reflections
2286 independent reflections
1805 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 25.0^\circ$
$h = 0 \rightarrow 10$
$k = -10 \rightarrow 11$
$l = -9 \rightarrow 9$
3 standard reflections every 100 reflections intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.121$
$S = 1.06$
2286 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.2754P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl2—C10	1.763 (3)	C13—C10	1.517 (5)
Cl3—C10	1.712 (5)	C11—C10	1.566 (3)
C12—C10	1.556 (8)	S1—C10	1.839 (3)
C13—C10—C11	110.0 (3)	C11—C10—Cl2	111.3 (2)
C11—C10—C12	113.0 (13)	C13—C10—S1	113.4 (3)
C11—C10—Cl3	111.8 (4)	C11—C10—S1	106.16 (17)
C12—C10—Cl3	106.5 (9)	C12—C10—S1	109.6 (15)
C13—C10—Cl2	107.3 (3)		

Table 2
Contact geometry (Å).

O2···C7	2.805 (4)	C7···C13	3.503 (7)
O1···C8	2.765 (4)	C8···C12	3.53 (4)
C2···C12	3.47 (4)	O2···C7^{i}	3.341 (4)
C6···C13	3.577 (8)	O2···C9^{ii}	3.371 (4)

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $-x, 1 - y, -z$.

The sum of the site occupancy values for C12 and C13 as well as Cl3 and Cl2 were restrained to 1.0. and the site-occupancy sum for each of the two disordered Cl/C sites was also restrained to 1.0. The C10—C and C10—Cl distances of the disordered C and Cl atoms were restrained to chemically reasonable distances of 1.54 and 1.76 Å, respectively, and the C10—C12—Cl2 and C10—C13—Cl3 angles were restrained so that the C10—C12 and C10—Cl2 as well as

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the C10—C13 and C10—Cl3 bond vectors would be collinear. Without this restraint, the disordered Cl and C atoms refined to decidedly non-tetrahedral geometry with unacceptable displacement parameters. The restraint used to accomplish the bond vector collinearity was the *SHELXL*97 command: *DANG* 0.22 0.001 C12 Cl2 C13 Cl3. The 0.22 value is the difference between the restrained C10—C and C10—Cl distances given above. The Cl atoms were refined anisotropically. A satisfactory anisotropic refinement of the two disordered C atoms could not be attained so they were refined isotropically with a restraint to force both of their thermal displacements to be equal to that of C13. The rotational orientation of the C7, C8, and C9 methyl groups were refined by the circular Fourier method available in *SHELXL*97 (Sheldrick, 1997). All H atoms are riding. Although tetrahedral geometry of the α-chloroisopropyl group was not forced during the refinement, Table 1 shows that the group is relatively well behaved.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation,1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR*92 (Burla *et al.*, 1989); program(s) used to refine structure: *LS* in *TEXSAN* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965) in *TEXSAN*; software used to prepare material for publication: *TEXSAN, SHELXL*97, and *PLATON* (Spek, 2000).

Partial support of this research from the University Research Foundation – La Jolla is gratefully acknowledged.

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