1,3,5-Tris(4-fluorobenzenesulfonyl)-1,3,5-triazacyclohexane

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The title compound, $C_{21}H_{18}F_3N_3O_6S_3$, has a chair conformation that is similar to the majority of the 1,3,5-triazacyclohexane rings reported in the literature. It is the product of a condensation reaction between fluorobenzenesulfonamide and formaldehyde, and its empirical and structural formula were determined by X-ray analysis. The molecules are located on mirror planes in space group $Pnma$.

Comment

The title compound, (I) (Fig. 1), contains a 1,3,5-triazacyclohexane ring with three 4-fluorobenzenesulfonyl substituents. It is a product of a condensation reaction between fluorobenzenesulfonamide and formaldehyde; in such reactions, various numbers of reactant equivalents may combine into one polyamine product. In this case, X-ray analysis proved the product to be a cyclic trimer containing three equivalents of each reactant. The 1,3,5-triazacyclohexane ring takes on the chair conformation which is typical of these rings. The structure of a very similar compound (lacking only the F atoms), *viz*. 1,3,5-tris(benzenesulfonyl)-1,3,5-triazacyclohexane, (II), has been reported (Rivero *et al.*, 1978). Both (I) and (II) have a chair conformation, with two sulfonyl substituents situated in equatorial positions, and a third axial, on the plane of the ring, and in both crystal structures the rings are situated on mirror planes.

(I)
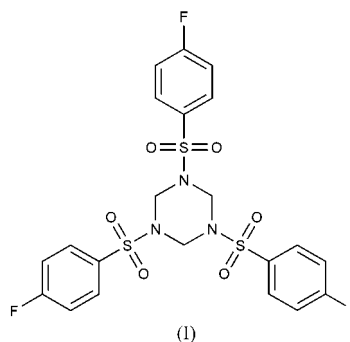
There are 33 similar compounds in the Cambridge Structural Database (CSD; Version 5.24; Allen, 2002), all containing the 1,3,5-triazacyclohexane ring with different substituents on the N atoms. 32 of these compounds have the chair conformation, the exception being 1,3,5-tris(*p*-nitrophenyl)-1,3,5-triazacyclohexane (Adam *et al.*, 1993), which has a twist–boat conformation. Table 1 gives the average torsion angles associated with chair-shaped 1,3,5-triazacyclohexane rings in previously reported structures. The torsion angles in (I) are slightly larger than these averages, but fall within the range of angles reported in the CSD.
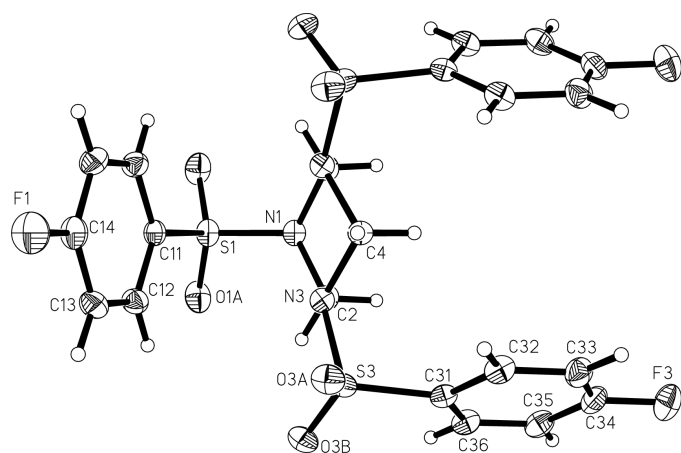
Figure 1
View of the title compound with 25% probability ellipsoids. There is a mirror plane passing through the middle of the compound, making half of the molecule independent.
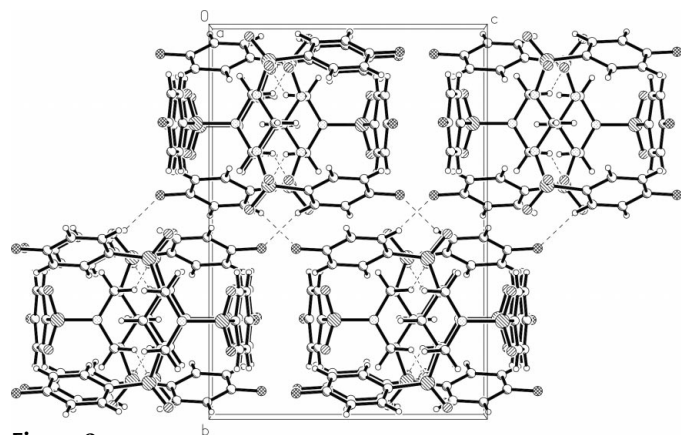

Figure 2
Packing diagram of the title compound, viewed down the *a* axis.

Fig. 2 illustrates the packing of the title molecule, which is simple close packing, marked by no particularly strong interactions. There is only one intermolecular contact, F3···H36($\frac{3}{2} - x$, $-y$, $\frac{1}{2} + z$) of 2.518 Å, that is slightly less than the van der Waals distance of 2.54 Å (Rowland & Taylor, 1996).

Experimental

A mixture of *para*-formaldehyde (0.085 g, 2.85 mmol), *p*-fluoro-benzenesulfonamide (1.0 g, 5.75 mmol) and acetic acid (0.068 g, 1.14 mmol) was heated at 413 K for 16 h. The resulting solution was evaporated on a rotary evaporator to dryness and the residue was fractional-crystallized from methanol–CH_2Cl_2 to yield the product. The product has a melting point of 512–514 K.

Crystal data

$C_{21}H_{18}F_3N_3O_6S_3$	Mo $K\alpha$ radiation
$M_r = 561.56$	Cell parameters from 66
Orthorhombic, $Pnma$	reflections
$a = 12.2603 (12)$ Å	$\theta = 10.0–13.5°$
$b = 16.4739 (13)$ Å	$\mu = 0.39$ mm^{-1}
$c = 11.6085 (6)$ Å	$T = 293 (2)$ K
$V = 2344.6 (3)$ Å^3	Block, colorless
$Z = 4$	$0.45 \times 0.43 \times 0.34$ mm
$D_x = 1.591$ Mg m^{-3}	

Data collection

Bruker $P4$ diffractometer	$R_{int} = 0.015$
$2\theta/\omega$ scans	$\theta_{max} = 26.0°$
Absorption correction: by integra-	$h = -1 \rightarrow 15$
tion (Wuensch & Prewitt, 1965)	$k = -20 \rightarrow 8$
$T_{min} = 0.855$, $T_{max} = 0.893$	$l = -1 \rightarrow 14$
2707 measured reflections	3 standard reflections
2390 independent reflections	every 97 reflections
1942 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$+ 1.0368P]$
$wR(F^2) = 0.096$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2390 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.0043 (6)

Table 1
Comparison of torsion angles (°) in the 1,3,5-triazacyclohexane ring in the CSD with those in (I).

Torsion	Average[a]	Minimum	Maximum	(I)
N1—C2—N3—C4	−57.193	−62.495	−42.857	−58.0 (2)
C2—N3—C4—N5	57.499	45.484	64.052	59.3 (3)
N3—C4—N5—C6	−56.289	−62.231	−46.254	−59.3 (3)
C4—N5—C6—N1	55.063	44.809	61.292	58.0 (2)
N5—C6—N1—C2	−55.192	−61.488	−42.518	−59.3 (3)
C6—N1—C2—N3	55.871	41.295	60.660	59.3 (3)

Note: (*a*) CSD (Version 5.24 of November 2002; Allen, 2002).

H atoms (4) in methylene groups were placed at ideal (Sheldrick, 1997) tetrahedral positions with a C—H distance of 0.97 Å. H atoms in phenyl groups (6) were placed in ideal trigonal positions with a C—H distance of 0.93 Å. Both types rode on their bonded neighbors during the refinement, with periodic re-idealization, and their atom displacement factors were set to be isotropic, with a value equal to $1.2U_{eq}$ of the neighboring C atom.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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