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# Perfluorinated 6-isopropyl-2,4,5-trimethylbenzonitrile

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In the title compound, 3-fluoro-6-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]-2,4,5-tris(trifluoromethyl)benzonitrile, $C_{13}F_{17}N$, the benzene ring is puckered due to steric repulsion between substituents.

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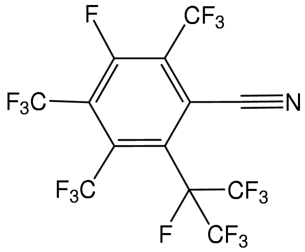
**Key indicators**

Single-crystal X-ray study
$T$ = 100 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.057
$wR$ factor = 0.134
Data-to-parameter ratio = 12.5

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

## Comment

The title compound, (I), was obtained as a by-product during our studies concerning fluoride-ion induced perfluoro-alkylation reactions of highly fluorinated aromatic systems (Chambers & Sargent, 1981; Brooke, 1997; Richmond, 2001).



(I)

The molecule of (I) (Fig. 1) is sterically overcrowded, resulting in some very short intramolecular non-bonded distances, *viz*. F1$\cdots$F2 = 2.467 (4) Å, F1$\cdots$F8 = 2.503 (4) Å, F6$\cdots$F10 = 2.503 (4) Å, F9$\cdots$F12 = 2.430 (4) Å, F11$\cdots$F12 = 2.547 (4) Å, C7$\cdots$F4 = 2.681 (4) Å, C7$\cdots$F15 = 2.606 (4) Å and C7$\cdots$F18 = 2.962 (4) Å, *cf.* the standard van der Waals contact distances (Rowland & Taylor, 1996) F$\cdots$F = 2.90 Å and C$\cdots$F = 3.22 Å. As a result, the substituents tilt out of the benzene plane and induce some puckering of the ring itself. Thus atoms C1, C2, C3 and C4 of the ring are coplanar within 0.012 (2) Å, but C5 and C6 deviate from the plane by −0.102 (6) and 0.052 (6) Å, respectively. The deviations of the substituent atoms are −0.196 (6) (C7), −0.467 (7) (N), −0.073 (7) (C8), −0.035 (6) (F1), 0.119 (7) (C9), −0.69 (1) (C10) and 0.35 (1) Å (C11).

The C—F bond distances in the trifluoromethyl groups range from 1.316 (4) to 1.342 (4) Å, with an average of 1.330 (7) Å.

## Experimental

A mixture containing tetrafluorophthalonitrile (5.0 g, 25 mmol) and dried KF (2.2 g, 100 mmol) in anhydrous DMF (25 ml) was heated to 323 K under dry $N_2$ with a cold-finger condenser attached, containing acetone/$CO_2$. $Me_3SiCF_3$ (14 g, 100 ml) in anhydrous DMF (5 ml) was added slowly to the reaction vessel *via* a syringe. The mixture was stirred at 323 K for 6 h. The deep red solution was transferred to an autoclave (160 ml) under dry $N_2$. Hexafluoropropene (15 g, 90 mmol)

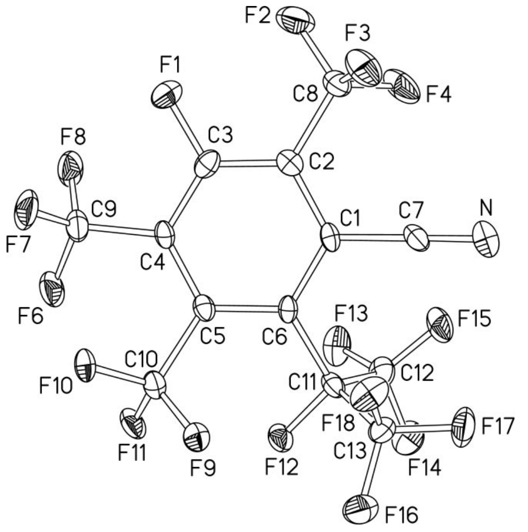


**Figure 1**
Molecular structure of (I). Atomic displacement ellipsoids are drawn at the 50% probability level.

was transferred into the autoclave under vacuum. The autoclave was sealed and heated to 358 K over a period of 48 h, and then opened in a vacuum. 7.0 g (41 mmol) of hexafluoropropene was recovered. Continuous extraction into perfluoromethylcyclohexane followed by evaporation of the solvent on a rotary evaporator gave a very small quantity of (I) as a white solid. Analysis found: C 31.6, N 2.8%; $C_{13}F_{17}N$ requires: C 31.4, N 2.8%. The $^{19}F$ NMR data (field strength 376 MHz) are listed in Table 2. The very broad peak at −55.0 p.p.m. can be interpreted as a poorly resolved multiplet of the $C10F_3$ group, resulting from unusual rotation of this group and the adjacent perfluoroisopropyl group.

### *Crystal data*

$C_{13}F_{17}N$
$M_r = 493.14$
Monoclinic, $P2_1/n$
$a = 9.521$ (1) Å
$b = 9.410$ (1) Å
$c = 17.426$ (2) Å
$\beta = 103.44$ (1)°
$V = 1518.5$ (3) Å$^3$
$Z = 4$

$D_x = 2.157$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 692 reflections
$\theta = 10.2$–21.4°
$\mu = 0.28$ mm$^{-1}$
$T = 100$ (2) K
Needle, colourless
0.55 × 0.08 × 0.02 mm

### *Data collection*

Bruker SMART 1K CCD area detector diffractometer
$\omega$ scans
Absorption correction: none
10 711 measured reflections
3497 independent reflections

1972 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.106$
$\theta_{\rm max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -22 \rightarrow 22$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.134$
$S = 1.02$
3497 reflections
280 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.8197P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.34$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.35$ e Å$^{-3}$

**Table 1**
Selected interatomic distances (Å).

| | | | |
|---|---|---|---|
| N−C7 | 1.148 (5) | C3−C4 | 1.386 (5) |
| F1−C3 | 1.336 (4) | C4−C5 | 1.400 (5) |
| F12−C11 | 1.377 (4) | C4−C9 | 1.532 (5) |
| C1−C2 | 1.403 (5) | C5−C6 | 1.405 (5) |
| C1−C6 | 1.407 (5) | C5−C10 | 1.542 (5) |
| C1−C7 | 1.443 (5) | C6−C11 | 1.539 (5) |
| C2−C3 | 1.384 (5) | C11−C12 | 1.559 (5) |
| C2−C8 | 1.520 (5) | C11−C13 | 1.578 (5) |

**Table 2**
$^{19}F$ NMR spectrum of (I).

| $\delta$ (p.p.m.) | Intensity | Multiplicity | Coupling (Hz) | Assignment |
|---|---|---|---|---|
| −55.0 | 3 | *br m* | – | $C10F_3$ |
| −56.5 | 3 | *dq* | $^4J_{FF}$ 28, $^5J_{FF}$ 4 | $C9F_3$ |
| −57.2 | 3 | *d* | $^4J_{FF}$ 28 | $C8F_3$ |
| −69.2 | 6 | *br s* | – | $C12F_3$, $C13F_3$ |
| −100.6 | 1 | *sept* | $^4J_{FF}$ 28 | F1 |
| −163.5 | 1 | *q* | $^3J_{FF}$ 54 | F12 |

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL*(Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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