

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω - θ scans
 Absorption correction: empirical via ψ scans (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.528$, $T_{\max} = 0.603$
 2624 measured reflections
 2624 independent reflections

2240 reflections with $I > \sigma(I)$
 $\theta_{\max} = 64.94^\circ$
 $h = 0 \rightarrow 11$
 $k = 0 \rightarrow 18$
 $l = 0 \rightarrow 20$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F
 $R = 0.040$
 $wR = 0.059$
 $S = 2.203$
 2240 reflections
 245 parameters
 H atoms refined isotropically
 $w = 4F_o^2 / [\sigma^2(F_o^2) + 0.0016F_o^4]$
 $(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
 Extinction correction: isotropic (Zachariasen, 1963)
 Extinction coefficient: 0.14×10^{-5}
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Si—C15	1.879 (1)	C6—C7	1.390 (2)
Si—C16	1.874 (1)	C7—C8	1.374 (2)
Si—C17	1.853 (2)	C8—C14	1.401 (2)
Si—C18	1.854 (2)	C9—C10	1.358 (2)
C1—C2	1.372 (2)	C9—C14	1.446 (2)
C1—C11	1.407 (2)	C9—C15	1.516 (2)
C2—C3	1.388 (2)	C10—C11	1.449 (2)
C3—C4	1.365 (2)	C10—C16	1.512 (2)
C4—C12	1.413 (2)	C11—C12	1.420 (2)
C5—C6	1.360 (2)	C12—C13	1.450 (2)
C5—C13	1.410 (2)	C13—C14	1.418 (2)
C15—Si—C16	95.20 (6)	C16—Si—C18	114.3 (1)
C15—Si—C17	111.28 (8)	C17—Si—C18	111.16 (9)
C15—Si—C18	113.06 (7)	Si—C15—C9	104.17 (8)
C16—Si—C17	110.95 (8)	Si—C16—C10	104.23 (8)

Data collection: *CAD-4 Operations Manual* (Enraf-Nonius, 1977). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *BEGIN* in *SDP-Plus* (Frenz, 1990). Program(s) used to solve structure: *FOUR* in *SDP-Plus*. Program(s) used to refine structure: *LSFM* in *SDP-Plus*. Molecular graphics: *ICRVIEW* (Erlebacher & Carrell, 1992). Software used to prepare material for publication: *CIFVAX* in *MolEN* (Fair, 1990).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: MU1274). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,3-Bis(*p*-fluorophenyl)triazene

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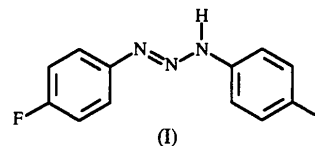
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Abstract

Molecules of the title compound, $\text{C}_{12}\text{H}_9\text{F}_2\text{N}_3$, contain a triazene group ($-\text{N}=\text{N}-\text{NH}$), having an extended conformation, and are linked by $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds to form chains.

Comment

p-Substituted derivatives of formamidine having the amidine group ($\text{N}=\text{CH}-\text{NH}$) form cyclic dimers in crystals as a result of two $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds (Anulewicz, Krygowski & Pniewska, 1987, 1990; Anulewicz, Krygowski, Jaroszewska-Manaj & Pniewska, 1991; Anulewicz, Wawer, Krygowski, Männle & Limbach, 1997). The structure of 1,3-bis(*p*-fluorophenyl)triazene, (I) (Fig. 1), has been determined in order to check whether the title molecule, containing a triazene group ($-\text{N}=\text{N}-\text{NH}$), forms similar dimers. The triazene group adopts an extended conformation with the 1,3-positioned phenyl rings arranged *cis* with respect to one another. The same arrangement is observed in other 1,3-diaryltriazene derivatives (Walton, Jenkins & Neidle, 1991). The molecule is nearly planar and the angles between the triazene group and the phenyl rings are 1.1 (3) and 10.1 (3)°.



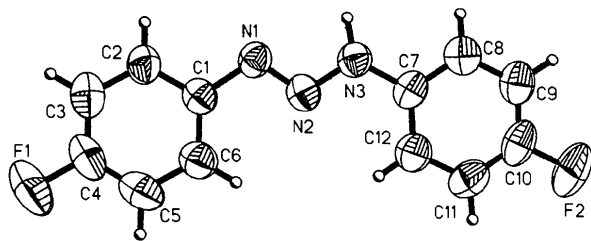


Fig. 1. A view of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Some very significant differences [*ca* 9.1 (3)°] in the values of the exocyclic angles at the C1 atom are observed. The corresponding angle differences at the C7 atom are much smaller [*ca* 2.4 (3)°]. This effect could be due to the different nature of the N atoms to which both C atoms are bonded or may be caused by steric interaction between the ring *ortho*-H atoms and the substituent as was found for Ph—OCH₃ (Krygowski *et al.*, 1994) and Ph—N=N—R (Krygowski, Anulewicz & Hiberty, 1996).

Experimental

The title compound was synthesized according to the method of Hartmann & Dickey (1943) as modified by Männle (1995).

Crystal data

C₁₂H₉F₂N₃
M_r = 233.22
 Orthorhombic
*P*2₁2₁2₁
a = 4.6830 (10) Å
b = 10.482 (2) Å
c = 22.529 (5) Å
V = 1105.9 (4) Å³
Z = 4
D_x = 1.401 Mg m⁻³
D_m not measured

Cu *K*α radiation
 λ = 1.54178 Å
 Cell parameters from 25 reflections
 θ = 13–24°
 μ = 0.94 mm⁻¹
T = 293 (2) K
 Prism
 0.3 × 0.2 × 0.1 mm
 Colourless

Data collection

Kuma KM-4 diffractometer
 ω/θ scans
 Absorption correction: none
 1089 measured reflections
 1089 independent reflections
 834 reflections with
 $I > 2\sigma(I)$

θ_{\max} = 65.04°
 $h = 0 \rightarrow 5$
 $k = 0 \rightarrow 12$
 $l = 0 \rightarrow 23$
 3 standard reflections
 every 100 reflections
 intensity decay: 1.5%

Refinement

Refinement on *F*²
R(*F*) = 0.0466
 $wR(F^2)$ = 0.1376
S = 1.013

$(\Delta/\sigma)_{\max}$ = -0.001
 $\Delta\rho_{\max}$ = 0.216 e Å⁻³
 $\Delta\rho_{\min}$ = -0.190 e Å⁻³
 Extinction correction: none

1086 reflections
 166 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.0899P)^2 + 0.0933P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from
International Tables for Crystallography (Vol. C)
 Absolute configuration:
 Flack (1983)
 Flack parameter = 0.2 (5)

Table 1. Selected geometric parameters (Å, °)

N1—N2	1.272 (3)	N2—N3	1.334 (4)
N1—C1	1.431 (4)	N3—C7	1.401 (4)
N2—N1—C1	111.9 (3)	C2—C1—N1	115.9 (3)
N1—N2—N3	112.9 (2)	C8—C7—N3	118.6 (3)
N2—N3—C7	119.0 (3)	C12—C7—N3	121.1 (3)
C6—C1—N1	125.0 (3)		

The H atom attached to N3 was located in a difference map and freely refined with *U*_{iso}, while the other H atoms were constrained with a riding model. There was no indication of any disorder in the triazene unit.

Data collection: *KM-4 Software* (Kuma Diffraction, 1991). Cell refinement: *KM-4 Software*. Data reduction: *KM-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL93*.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: CF1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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