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2,2′-Bis(trifluoromethyl)diphenyldiazene

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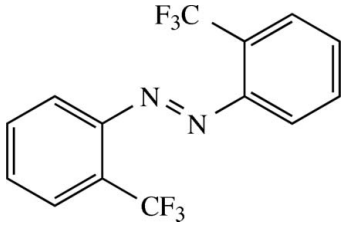
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.105; data-to-parameter ratio = 12.5.

The title compound, $C_{14}H_8F_6N_2$, a fluorinated azo dye, crystallizes with one half-molecule in the asymmetric unit. The mid-point of the N=N double bond coincides with a centre of symmetry; the azene unit is *trans*-configured. The molecular skeleton formed by the C and N atoms is essentially planar. The crystal structure is characterized by the π-stacking of azene functions and the aromatic residues, the mid-point of the azene group being 3.5626 (7) Å distant from the centroids of the aromatic rings above and below. Weak lateral intercolumn contacts are established *via* C—H···F interactions.

Related literature

For the synthesis of the compound (as a by-product), see: Bart (1922). For the crystal structures of related compounds, in which similar geometrical features are observed, see: Harada *et al.* (1997).



Experimental

Crystal data

$C_{14}H_8F_6N_2$
M_r = 318.22
Monoclinic, $P2_1/n$
a = 4.7573 (2) Å
b = 9.7204 (6) Å
c = 14.2018 (8) Å
β = 94.216 (3)°
V = 654.95 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 200 (2) K
0.25 × 0.04 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
4443 measured reflections
1271 independent reflections
952 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.105
S = 1.05
1271 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···F1^{i}	0.95	2.85	3.648 (2)	143
C4—H4···F2^{i}	0.95	2.83	3.722 (2)	157
C5—H5···F1^{ii}	0.95	2.77	3.598 (2)	147
C6—H6···F3^{ii}	0.95	2.73	3.511 (2)	140

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+\frac{5}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2362).

References

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supplementary materials

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2,2'-Bis(trifluoromethyl)diphenyldiazene

R. Betz and P. Klüfers

Comment

The title compound (I) was obtained as a byproduct in the synthesis of (2-trifluoromethyl)phenylarsonic acid.

In the planar, C_i-symmetric molecules, the trifluoromethyl groups are present in a conformation that has one F atom in coplanarity to the aromatic plane. Bond lengths and angles are in agreement with the values apparent in the literature for a similar compound (Harada *et al.*, 1997).

In the crystal structure, columns of π-stacked molecules are laterally connected *via* weak C–H···F interactions with a minimum C···F distance of about 3.5 Å (Fig. 2).

Experimental

The compound was obtained as a byproduct upon the attempted synthesis of (2-trifluoromethyl)phenylarsonic acid in analogy to a published procedure (Bart, 1922). Crystals suitable for X-ray analysis were obtained as sublimate upon evaporation of the crude reaction mixture to dryness on a rotavapor.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to U_{iso}(H) = 0.051 (3).

Figures

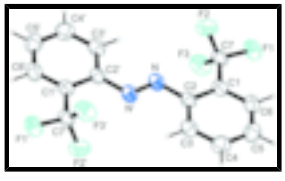

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms [symmetry code: (') 1 − *x*, 1 − *y*, −*z*].

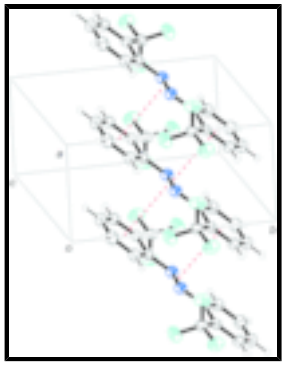

Fig. 2. A portion of the crystal packing showing the azene···aromatic π stacking interactions as dashed lines.

2,2'-Bis(trifluoromethyl)diphenyldiazene

Crystal data

$C_{14}H_8F_6N_2$	F_{000} = 320
M_r = 318.22	D_x = 1.614 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4994 reflections
a = 4.7573 (2) Å	θ = 3.1–26.0º
b = 9.7204 (6) Å	μ = 0.16 mm^{-1}
c = 14.2018 (8) Å	T = 200 (2) K
β = 94.216 (3)º	Rod, orange
V = 654.95 (6) Å^3	0.25 × 0.04 × 0.02 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	952 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.052
Monochromator: MONTEL, graded multilayered X-ray optics	θ_{max} = 26.0º
T = 200(2) K	θ_{min} = 3.6º
CCD; rotation images; thick slices scans	h = −5→5
Absorption correction: none	k = −11→11
4443 measured reflections	l = −17→17
1271 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.0735P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.105	$(\Delta/\sigma)_{max} < 0.001$
S = 1.05	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
1271 reflections	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
102 parameters	Extinction correction: SHELXL
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.031 (7)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}

F1	0.9746 (2)	0.40050 (12)	0.29989 (7)	0.0571 (4)
F2	0.5575 (2)	0.44043 (12)	0.23976 (7)	0.0533 (4)
F3	0.8962 (2)	0.57465 (11)	0.21007 (7)	0.0547 (4)
N	0.5569 (3)	0.49160 (14)	0.04058 (9)	0.0380 (4)
C1	0.8891 (3)	0.35540 (17)	0.13717 (11)	0.0352 (4)
C2	0.7549 (3)	0.38081 (16)	0.04783 (11)	0.0342 (4)
C3	0.8175 (4)	0.29934 (17)	−0.02873 (12)	0.0416 (4)
H3	0.7252	0.3158	−0.0893	0.051 (3)*
C4	1.0132 (4)	0.19490 (18)	−0.01662 (13)	0.0478 (5)
H4	1.0577	0.1405	−0.0691	0.051 (3)*
C5	1.1445 (4)	0.16919 (18)	0.07167 (14)	0.0485 (5)
H5	1.2786	0.0969	0.0797	0.051 (3)*
C6	1.0818 (4)	0.24820 (18)	0.14827 (12)	0.0425 (5)
H6	1.1710	0.2291	0.2089	0.051 (3)*
C7	0.8283 (3)	0.44241 (18)	0.22031 (11)	0.0396 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0606 (7)	0.0785 (8)	0.0302 (6)	0.0028 (6)	−0.0097 (5)	0.0024 (5)
F2	0.0415 (6)	0.0775 (8)	0.0415 (6)	−0.0055 (5)	0.0061 (5)	−0.0087 (5)
F3	0.0727 (8)	0.0489 (7)	0.0425 (7)	−0.0138 (5)	0.0046 (5)	−0.0077 (4)
N	0.0415 (8)	0.0418 (8)	0.0299 (7)	−0.0015 (6)	−0.0025 (6)	0.0013 (6)
C1	0.0338 (9)	0.0400 (9)	0.0314 (9)	−0.0072 (7)	−0.0004 (6)	0.0029 (7)
C2	0.0354 (9)	0.0344 (8)	0.0323 (9)	−0.0047 (7)	0.0004 (7)	0.0026 (7)
C3	0.0484 (10)	0.0422 (10)	0.0337 (9)	−0.0031 (8)	0.0007 (7)	−0.0001 (7)
C4	0.0547 (11)	0.0424 (10)	0.0471 (11)	−0.0001 (8)	0.0082 (8)	−0.0061 (8)
C5	0.0487 (11)	0.0398 (10)	0.0567 (12)	0.0032 (8)	0.0016 (9)	0.0026 (9)
C6	0.0398 (10)	0.0440 (10)	0.0424 (10)	−0.0033 (8)	−0.0051 (7)	0.0093 (8)
C7	0.0375 (9)	0.0503 (10)	0.0302 (9)	−0.0057 (8)	−0.0030 (7)	0.0042 (7)

Geometric parameters (Å, °)

F1—C7	1.3460 (19)	C2—C3	1.395 (2)
F2—C7	1.337 (2)	C3—C4	1.380 (3)
F3—C7	1.336 (2)	C3—H3	0.9500
N—N^{i}	1.248 (2)	C4—C5	1.382 (3)
N—C2	1.429 (2)	C4—H4	0.9500
C1—C6	1.389 (2)	C5—C6	1.382 (3)
C1—C2	1.400 (2)	C5—H5	0.9500
C1—C7	1.498 (2)	C6—H6	0.9500
N^{i}—N—C2	113.83 (16)	C4—C5—C6	120.28 (16)
C6—C1—C2	119.24 (15)	C4—C5—H5	119.9
C6—C1—C7	119.88 (14)	C6—C5—H5	119.9
C2—C1—C7	120.88 (15)	C5—C6—C1	120.42 (16)
C3—C2—C1	119.80 (15)	C5—C6—H6	119.8
C3—C2—N	123.30 (14)	C1—C6—H6	119.8
C1—C2—N	116.90 (14)	F3—C7—F2	106.29 (14)

C4—C3—C2	120.09 (16)	F3—C7—F1	105.58 (13)
C4—C3—H3	120.0	F2—C7—F1	105.57 (13)
C2—C3—H3	120.0	F3—C7—C1	113.29 (13)
C3—C4—C5	120.15 (17)	F2—C7—C1	113.54 (13)
C3—C4—H4	119.9	F1—C7—C1	111.92 (14)
C5—C4—H4	119.9		
C6—C1—C2—C3	−0.5 (2)	C4—C5—C6—C1	−0.9 (3)
C7—C1—C2—C3	179.25 (15)	C2—C1—C6—C5	1.3 (2)
C6—C1—C2—N	179.50 (14)	C7—C1—C6—C5	−178.50 (15)
C7—C1—C2—N	−0.7 (2)	C6—C1—C7—F3	118.04 (17)
N^{i}—N—C2—C3	0.5 (2)	C2—C1—C7—F3	−61.7 (2)
N^{i}—N—C2—C1	−179.51 (16)	C6—C1—C7—F2	−120.59 (16)
C1—C2—C3—C4	−0.6 (2)	C2—C1—C7—F2	59.7 (2)
N—C2—C3—C4	179.36 (15)	C6—C1—C7—F1	−1.2 (2)
C2—C3—C4—C5	1.0 (3)	C2—C1—C7—F1	179.06 (13)
C3—C4—C5—C6	−0.3 (3)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···F1^{ii}	0.95	2.85	3.648 (2)	143
C4—H4···F2^{ii}	0.95	2.83	3.722 (2)	157
C5—H5···F1^{iii}	0.95	2.77	3.598 (2)	147
C6—H6···F3^{iii}	0.95	2.73	3.511 (2)	140

Symmetry codes: (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) −*x*+5/2, *y*−1/2, −*z*+1/2.

Fig. 1

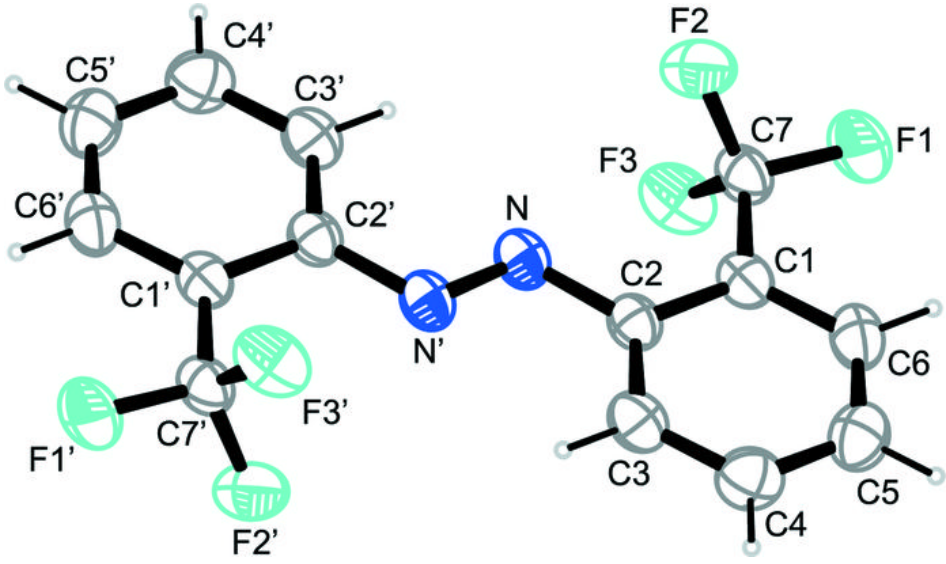

Fig. 2