

O15	0.6413 (2)	0.93930 (10)	0.0747 (4)	0.0331 (6)
O19	0.3091 (2)	0.99390 (10)	0.5199 (5)	0.0382 (6)
N8	0.6712 (2)	0.79920 (10)	-0.5981 (5)	0.0298 (6)
C2	0.7835 (3)	0.79050 (10)	-0.7568 (6)	0.0325 (7)
C4	0.6137 (3)	0.73810 (10)	-0.8353 (6)	0.0360 (8)
C5	0.6217 (4)	0.69050 (10)	-0.7765 (7)	0.0380 (8)
C7	0.5628 (3)	0.76570 (10)	-0.6090 (7)	0.0396 (9)
C9	0.6640 (3)	0.83420 (10)	-0.4253 (6)	0.0280 (7)
C10	0.7622 (3)	0.86850 (10)	-0.4344 (6)	0.0312 (7)
C11	0.7506 (3)	0.90240 (10)	-0.2632 (6)	0.0306 (7)
C12	0.6425 (3)	0.90340 (10)	-0.0802 (6)	0.0290 (7)
C13	0.5454 (3)	0.86980 (10)	-0.0710 (6)	0.0325 (8)
C14	0.5564 (3)	0.83560 (10)	-0.2424 (6)	0.0319 (8)
C16	0.5271 (3)	0.94480 (10)	0.2533 (6)	0.0293 (7)
C17	0.5473 (3)	0.98980 (10)	0.3631 (6)	0.0317 (8)
C18	0.4551 (3)	1.00020 (10)	0.5934 (6)	0.0304 (7)
C20	0.4781 (4)	1.04590 (10)	0.6872 (7)	0.0425 (10)
C21	0.7069 (5)	0.64030 (10)	-0.4822 (10)	0.0580 (14)

Table 2. Selected geometric parameters (Å, °)

O1—C2	1.202 (4)	C4—C5	1.509 (4)
O3—C2	1.362 (4)	C4—C7	1.531 (5)
O3—C4	1.460 (4)	C9—C10	1.408 (4)
O6—C5	1.407 (4)	C9—C14	1.387 (4)
O6—C21	1.412 (5)	C10—C11	1.381 (4)
O15—C12	1.374 (4)	C11—C12	1.391 (4)
O15—C16	1.428 (4)	C12—C13	1.385 (4)
O19—C18	1.436 (3)	C13—C14	1.389 (4)
N8—C2	1.365 (4)	C16—C17	1.519 (4)
N8—C7	1.455 (4)	C17—C18	1.511 (4)
N8—C9	1.410 (4)	C18—C20	1.514 (4)
C2—O3—C4	110.0 (2)	N8—C9—C14	119.7 (3)
C5—O6—C21	111.0 (3)	C10—C9—C14	118.4 (3)
C12—O15—C16	118.9 (2)	C9—C10—C11	120.1 (3)
C2—N8—C7	112.1 (3)	C10—C11—C12	121.0 (3)
C2—N8—C9	125.0 (2)	O15—C12—C11	115.1 (3)
C7—N8—C9	122.7 (2)	O15—C12—C13	125.7 (3)
O1—C2—O3	120.8 (3)	C11—C12—C13	119.1 (3)
O1—C2—N8	129.5 (3)	C12—C13—C14	120.2 (3)
O3—C2—N8	109.7 (2)	C9—C14—C13	121.1 (3)
O3—C4—C5	109.3 (3)	O15—C16—C17	105.1 (2)
O3—C4—C7	105.1 (2)	C16—C17—C18	114.9 (2)
C5—C4—C7	114.0 (3)	O19—C18—C17	107.9 (2)
O6—C5—C4	109.6 (3)	O19—C18—C20	110.4 (3)
N8—C7—C4	102.2 (2)	C17—C18—C20	111.9 (3)
N8—C9—C10	121.9 (3)		
C4—O3—C2—O1	-178.4 (3)	C5—C4—C7—N8	129.6 (3)
C4—O3—C2—N8	2.7 (3)	O3—C4—C5—O6	60.1 (3)
C2—O3—C4—C5	-130.9 (3)	C7—C4—C5—O6	-57.1 (3)
C2—O3—C4—C7	-8.2 (3)	O3—C4—C7—N8	10.1 (3)
C21—O6—C5—C4	172.1 (3)	N8—C9—C10—C11	179.4 (3)
C16—O15—C12—C11	174.8 (3)	C14—C9—C10—C11	0.3 (5)
C16—O15—C12—C13	-3.8 (4)	N8—C9—C14—C13	-179.4 (3)
C12—O15—C16—C17	-172.2 (2)	C10—C9—C14—C13	-0.3 (5)
C7—N8—C2—O1	-174.3 (3)	C9—C10—C11—C12	-0.1 (6)
C9—N8—C2—O1	0.4 (5)	C10—C11—C12—O15	-178.7 (3)
C7—N8—C2—O3	4.5 (4)	C10—C11—C12—C13	-0.1 (4)
C9—N8—C2—O3	179.2 (3)	O15—C12—C13—C14	178.6 (3)
C7—N8—C9—C10	-173.7 (3)	C11—C12—C13—C14	0.1 (5)
C2—N8—C9—C14	-168.7 (3)	C12—C13—C14—C9	0.1 (6)
C7—N8—C9—C14	5.4 (4)	O15—C16—C17—C18	-169.2 (2)
C2—N8—C9—C10	12.2 (5)	C16—C17—C18—O19	-56.7 (3)
C2—N8—C7—C4	-9.2 (3)	C16—C17—C18—C20	-178.4 (3)
C9—N8—C7—C4	176.0 (3)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	H...A	D—H...A
O19—H19...O19 ⁱ	2.850 (4)	174.0 (50)

Symmetry code: (i) $\frac{1}{2} - x, 2 - y, \frac{1}{2} + z$.

Most calculations were performed with *SHELX76* (Sheldrick, 1976), the structure was solved using *SHELXS86* (Sheldrick, 1985) and *PLATON* (Spek, 1990) was used for the generation of the CIF.

JW thanks the National Foundation for Scientific Research (FNRS, Belgium) for his Research Assistant position. The authors acknowledge Delalande Research (Groupe Synthelabo) for providing the sample and thank Dr J. J. Koenig for helpful discussions. They also thank IBM Belgium, the FNRS and the Faculté Notre-Dame de la Paix for the use of the Namur Scientific Computing Facilities.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Koenig, J. J., Moureau, F., Vercauteren, D. P., Durant, F., Ducrey, F. & Jarreau, F. X. (1992). Presented at the XVIIIth CIMP Congress.
- Moureau, F., Wouters, J., Vercauteren, D. P., Evrard, G., Durant, F., Ducrey, F., Koenig, J. J. & Jarreau, F. X. (1992). *Eur. J. Med. Chem.* **27**, 939–948.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Wouters, J., Moureau, F., Dory, M., Evrard, G., Durant, F., Ducrey, F., Koenig, J. J. & Jarreau, F. X. (1993). *Trends in QSAR and Molecular Modelling 92*, edited by C. Wermuth, pp. 291–293. Leiden: ESCOM.
- Wouters, J., Moureau, F., Vercauteren, D. P., Evrard, G., Durant, F., Ducrey, F., Koenig, J. J. & Jarreau, F. X. (1994). *J. Neural Transm. Suppl.* **41**. In the press.
- Wouters, J., Perpete, P., Norberg, B., Evrard, G. & Durant, F. (1994). *Acta Cryst.* **C50**, 97–100.

Acta Cryst. (1994). **C50**, 1492–1494

(Z,Z)-5-Anilino-1,1,1,6,6,6-hexafluoro-3,4-diazahepta-2,4-diene-2-amine

MOHAMAD ABDUL-GHANI, BRIAN BEAGLEY,
ROBIN G. PRITCHARD AND ANTHONY E. TIPPING

*Department of Chemistry, University of Manchester
Institute of Science and Technology, PO Box 88,
Manchester M60 1QD, England*

(Received 3 December 1993; accepted 6 April 1994)

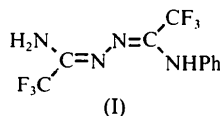
Abstract

The title molecule, C₁₀H₈F₆N₄, has been characterized crystallographically, thus establishing its isomeric form. This form has a C=N—N=C substructure with a torsion angle of 145 (1)° about the N—N bond. There is an extensive hydrogen-bonding system, such that each

molecule is doubly bonded to each of two neighbours, related to the first by different twofold axes.

Comment

The structure of (*Z,Z*)-5-chloro-1,1,1,6,6,6-hexafluoro-3,4-diazahexa-2,4-diene-2-amine has recently been characterized crystallographically (Abdul-Ghani, Pritchard & Tipping, 1994). It reacts with aniline under anhydrous conditions to give the title molecule, (*Z,Z*)-5-anilino-1,1,1,6,6,6-hexafluoro-3,4-diazahexa-2,4-diene-2-amine (I) and aniline hydrochloride. After purification by flash chromatography, the title molecule was recrystallized from *n*-pentane/dichloromethane (2:1 *v/v*).



A literature search (CSSR, 1984) revealed only one other molecule (Tinant, Declercq, van Meerssche, Bouvy, Janousek & Viehe, 1985), besides the chloro precursor of the title molecule (Abdul-Ghani, Pritchard & Tipping, 1994), possessing a similar CC(N)=N—N=C substructure to that of the title molecule. All three molecules have comparable bond lengths for this substructure. The title molecule and the chloro precursor are twisted about the N—N bond by similar amounts with torsion angles of 145 (1) and 2×154 (1)°, respectively (the chloro precursor has two molecules in the asymmetric unit), but the molecule of Tinant, Declercq, van Meerssche, Bouvy, Janousek & Viehe (1985) has two approximately planar C=N—N=C systems [177 and 180 (1)°]. Hydrogen bonds observed in the

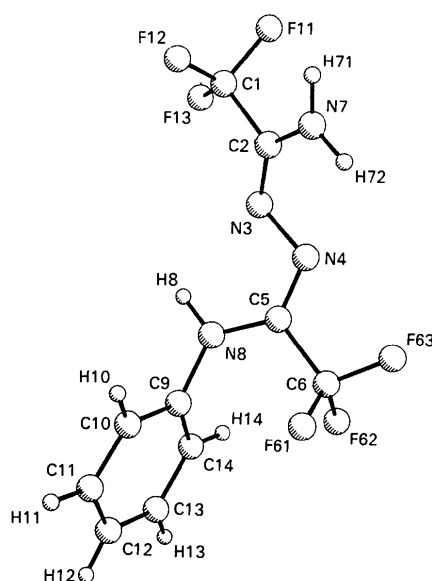


Fig. 1. The title molecule showing atom labelling.

title molecule are N3···H8—N8 (at $-x, y-x, \frac{2}{3}-z$) and N4···H72—N7 (at $y, x, 1-z$) with N···N lengths of 3.00 (1) and 2.99 (1) Å, respectively. These hydrogen bonds generate two more by different twofold symmetry operations, such that the molecule at x, y, z also has N8—H8···N3 and N7—H72···N4 hydrogen bonds and is doubly linked to each of the stated neighbours.

Experimental

Crystal data

C₁₀H₈F₆N₄
 $M_r = 298.19$
 Trigonal
 $P3_221$
 $a = 9.480$ (2) Å
 $c = 24.792$ (2) Å
 $V = 1929$ (1) Å³
 $Z = 6$
 $D_x = 1.540$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 6.5-11.9^\circ$
 $\mu = 0.151$ mm⁻¹
 $T = 296$ K
 Block
 $0.4 \times 0.3 \times 0.2$ mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 3142 measured reflections
 2075 independent reflections
 773 observed reflections
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 24^\circ$
 $h = 0 \rightarrow 10$
 $k = -10 \rightarrow 8$
 $l = 0 \rightarrow 26$
 3 standard reflections
 frequency: 120 min
 intensity variation: none

Refinement

Refinement on F
 $R = 0.065$
 $wR = 0.030$
 $S = 2.37$
 773 reflections
 188 parameters
 H-atom parameters not refined
 Weights: default scheme in TEXSAN (Molecular Structure Corporation, 1985)

$(\Delta/\sigma)_{\text{max}} = 0.07$
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$					
	Occupancy	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
F11	0.85 (1)	-0.228 (02)	-0.4393 (14)	0.4512 (06)	9.5 (6)
F12	0.85 (1)	-0.031 (02)	-0.417 (02)	0.4080 (09)	11 (1)
F13	0.85 (1)	-0.198 (02)	-0.3597 (12)	0.3710 (05)	8.4 (7)
F61	1.00	0.4166 (09)	0.4532 (08)	0.4152 (03)	8.8 (4)
F62	1.00	0.2015 (11)	0.4654 (09)	0.3945 (03)	9.3 (5)
F63	1.00	0.2151 (09)	0.3708 (08)	0.4708 (02)	9.0 (4)
N3	1.00	0.0008 (12)	-0.0634 (11)	0.4038 (04)	4.3 (5)
N4	1.00	0.0813 (12)	0.0890 (11)	0.4290 (04)	4.6 (5)
N7	1.00	0.0383 (12)	-0.1668 (11)	0.4872 (03)	5.8 (6)
N8	1.00	0.2050 (10)	0.1862 (11)	0.3452 (03)	5.2 (5)
C1	1.00	-0.113 (02)	-0.348 (02)	0.4132 (06)	6.0 (8)

C2	1.00	-0.014 (02)	-0.175 (02)	0.4367 (05)	4.1 (7)
C5	1.00	0.172 (02)	0.202 (02)	0.3969 (05)	4.4 (7)
C6	1.00	0.257 (02)	0.379 (02)	0.4185 (06)	7.1 (9)
C9	1.00	0.3135 (14)	0.3121 (14)	0.3082 (05)	4.6 (6)
C10	1.00	0.461 (02)	0.326 (02)	0.2956 (05)	7.6 (8)
C11	1.00	0.5649 (14)	0.435 (02)	0.2581 (05)	8.1 (9)
C12	1.00	0.522 (02)	0.5394 (14)	0.2344 (05)	6.8 (9)
C13	1.00	0.376 (02)	0.529 (02)	0.2443 (06)	8 (1)
C14	1.00	0.2692 (14)	0.414 (02)	0.2818 (05)	6.7 (7)
F14	0.15 (1)	-0.092 (10)	-0.362 (08)	0.361 (02)	9.0
F15	0.15 (1)	-0.069 (10)	-0.458 (12)	0.436 (03)	9.0
F16	0.15 (1)	-0.266 (07)	-0.414 (11)	0.424 (04)	9.0

Table 2. Selected geometric parameters (Å, °)

F11—C1	1.37 (2)	N8—C5	1.35 (2)
F12—C1	1.25 (3)	N8—C9	1.45 (1)
F13—C1	1.29 (2)	C1—C2	1.54 (2)
F61—C6	1.31 (2)	C5—C6	1.55 (2)
F62—C6	1.32 (2)	C9—C10	1.37 (2)
F63—C6	1.35 (2)	C9—C14	1.40 (2)
N3—N4	1.40 (1)	C10—C11	1.38 (2)
N3—C2	1.29 (2)	C11—C12	1.38 (2)
N4—C5	1.27 (1)	C12—C13	1.36 (3)
N7—C2	1.33 (1)	C13—C14	1.40 (2)
N4—N3—C2	110.1 (9)	N4—C5—N8	127 (1)
N3—N4—C5	113 (1)	N4—C5—C6	118 (1)
C5—N8—C9	128.3 (9)	N8—C5—C6	115 (1)
F11—C1—F12	104 (2)	F61—C6—F62	110 (1)
F11—C1—F13	104 (1)	F61—C6—F63	109 (1)
F11—C1—C2	106 (1)	F61—C6—C5	113 (2)
F12—C1—F13	113 (2)	F62—C6—F63	106 (2)
F12—C1—C2	113 (1)	F62—C6—C5	111 (1)
F13—C1—C2	115 (1)	F63—C6—C5	107 (1)
C2—C1—F14	116 (3)	N8—C9—C10	119 (1)
C2—C1—F15	112 (3)	N8—C9—C14	122 (1)
C2—C1—F16	112 (5)	C10—C9—C14	118 (1)
F14—C1—F15	102 (6)	C9—C10—C11	122 (2)
F14—C1—F16	110 (6)	C10—C11—C12	118 (2)
F15—C1—F16	104 (6)	C11—C12—C13	122 (1)
N3—C2—N7	132 (1)	C12—C13—C14	119 (2)
N3—C2—C1	113 (1)	C9—C14—C13	120 (1)
N7—C2—C1	115 (1)		

The structure was solved using direct methods (MITHRIL; Gilmore, 1984) in the space group $P3_2$ (No. 145) which requires two molecules in the asymmetric unit; the two molecules found were clearly related by a twofold axis at $(\frac{2}{3}, y, \frac{1}{2})$. Space group $P3_2$ has equivalent 3_2 axes at $(\frac{2}{3}, y, \frac{1}{2}, z)$ and $(0, 0, z)$. A change to space group $P3_221$ (No. 154) was implied, but its 3_2 axes were no longer equivalent; the added twofold axes pass through $(0, 0, z)$. The solution of the present structure in $P3_221$ was achieved in two steps. First, x and y coordinates were reduced by $\frac{2}{3}$ and $\frac{1}{3}$, respectively, moving the known twofold axis to $(0, y - \frac{1}{3}, \frac{1}{2})$, placing the molecules around the 3_2 axis at $(0, 0, z)$, but requiring a z translation of $-\frac{1}{6}$ to bring the molecules to valid equivalent positions in $P3_221$. The known twofold axis was now at $(0, y - \frac{1}{3}, \frac{1}{2})$, which may be redesignated as $(0, x, \frac{1}{2})$ which is acceptable in $P3_221$ and requires only one molecule per asymmetric unit. Data collection and cell refinement: CAD-4 software. Data reduction: SHELX76 (Sheldrick, 1976). Program(s) used to refine structure: TEXSAN (Molecular Structure Corporation, 1985). Molecular graphics: PLUTO (Motherwell & Clegg, 1978). Software used to prepare material for publication: CIF (Hall, Allen & Brown, 1991).

The CAD-4 diffractometer was funded by the SERC which is also thanked for funding the Chemical Databank Services used for crystallographic literature searches.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Abdul-Ghani, M., Pritchard, R. G. & Tipping, A. E. (1994). *Acta Cryst. C50*, 722–724.
- CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual*. SERC Daresbury Laboratory, Warrington, England.
- Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
- Hall, S. R., Allen, F. H. & Brown, I. D. (1991). *Acta Cryst. A47*, 655–685.
- Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Tinant, B., Declercq, J.-P., van Meerssche, M., Bouvy, A., Janousek, Z. & Viehe, H. G. (1985). *J. Chem. Soc. Perkin Trans. 2*, pp. 1419–1421.

Acta Cryst. (1994). **C50**, 1494–1497

Methyl- α -D-galacturonic Acid Methyl Ester

DORIANO LAMBA

Istituto di Strutturistica Chimica Giordano Giacomello, CNR, Area della Ricerca di Roma, PO Box 10, I-00016 Monterotondo Stazione (Roma), Italy

GIANCARLO FABRIZI

Dipartimento di Chimica, Ingegneria Chimica e Materiali, Università dell'Aquila, I-67100 l'Aquila, Italy

BETTY MATSUHIRO

Departamento de Química, Facultad de Ciencia, Universidad de Santiago de Chile, Casilla 307, Santiago 2, Chile

(Received 5 July 1993; accepted 17 December 1993)

Abstract

The structure of methyl methyl- α -D-galacturonate monohydrate, C₈H₁₄O₇·H₂O, has been redetermined. The sugar ring has the expected ⁴C₁ conformation. The orientation of the methoxy group is *gauche-trans* and