Triclinic
$P\bar{1}$
$a = 7.2790 (10)$ Å
$b = 8.455 (2)$ Å
$c = 11.408 (2)$ Å
$\alpha = 67.730 (10)°$
$\beta = 85.150 (10)°$
$\gamma = 84.51 (2)°$
$V = 645.9 (2)$ Å^3
$Z = 2$
$D_x = 1.508$ Mg m^{-3}
$D_m = 1.52$ Mg m^{-3}
D_m measured by flotation in benzene/1-bromo-2-chlorobenzene

Cell parameters from 25 reflections
$\theta = 11.8–18.8°$
$\mu = 0.122$ mm^{-1}
$T = 293 (2)$ K
Block
0.56 × 0.31 × 0.28 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.89$, $T_{\max} = 0.97$
2399 measured reflections
2399 independent reflections

2002 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 26.96°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 240 min
intensity decay: −0.2%

Refinement

Refinement on F^2
$R(F) = 0.045$
$wR(F^2) = 0.124$
$S = 1.05$
2399 reflections
192 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.1525P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1A—C2A	1.344 (2)	O1B—C7B	1.283 (2)
N1A—C6A	1.358 (2)	O2B—C7B	1.227 (2)
C2A—N3A	1.313 (3)	C7B—C1B	1.495 (3)
N3A—C5A	1.347 (2)	C1B—C6B	1.387 (3)
C4A—N9A	1.379 (2)	C1B—C2B	1.389 (3)
C4A—C5A	1.384 (2)	C2B—F2B	1.354 (2)
C4A—C6A	1.400 (3)	C2B—C3B	1.367 (3)
C5A—N7A	1.362 (2)	C3B—C4B	1.376 (3)
C6A—N10A	1.320 (2)	C4B—C5B	1.379 (3)
N7A—C8A	1.349 (3)	C5B—C6B	1.367 (3)
C8A—N9A	1.314 (3)		
C2A—N1A—C6A	121.2 (2)	O2B—C7B—O1B	123.9 (2)
N3A—C2A—N1A	127.1 (2)	O2B—C7B—C1B	121.9 (2)
C2A—N3A—C5A	111.7 (2)	O1B—C7B—C1B	114.2 (2)
N9A—C4A—C5A	111.0 (2)	C6B—C1B—C2B	116.2 (2)
N9A—C4A—C6A	131.5 (2)	C6B—C1B—C7B	120.2 (2)
C5A—C4A—C6A	117.5 (2)	C2B—C1B—C7B	123.6 (2)
N3A—C5A—N7A	128.0 (2)	F2B—C2B—C3B	117.3 (2)
N3A—C5A—C4A	126.8 (2)	F2B—C2B—C1B	119.9 (2)
N7A—C5A—C4A	105.2 (2)	C3B—C2B—C1B	122.8 (2)
N10A—C6A—C4A	125.1 (2)	C2B—C3B—C4B	119.0 (2)
N1A—C6A—C4A	115.6 (2)	C3B—C4B—C5B	120.3 (2)
C8A—N7A—C5A	106.6 (2)	C6B—C5B—C4B	119.3 (2)
N9A—C8A—N7A	114.0 (2)	C5B—C6B—C1B	122.4 (2)
C8A—N9A—C4A	103.2 (2)		

Table 2. *Contact distances (Å)*

N1A···O1B^{i}	2.560 (2)	O1W···N3A^{iii}	2.895 (2)
N7A···O1W^{ii}	2.751 (2)	N10A···O2B^{i}	2.867 (2)
O1W···O2B^{iii}	2.805 (2)	N10A···N9A^{iv}	2.994 (2)

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, y-1, z$; (iii) $-x, 1-y, 1-z$; (iv) $1-x, -y, -z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XPMA* and *ZORTEP* (Zsolnai, 1995).

The authors would like to thank Edward Foos for preparing the crystal and measuring the data.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1179). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,*N*′-Bis(pentafluorophenyl)urea

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Abstract

In the crystal structure of the title compound, $C_{13}H_2F_{10}N_2O$, there is a close contact of a H atom and an F atom as a three-centered hydrogen-bonding interaction.

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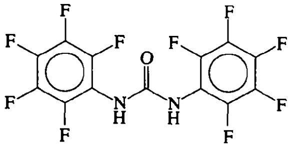
† Deceased, 1994.

Comment

The structure of the title compound, (I), was determined in order to shed light on the controversy about the ability of a covalently bound F atom to act as a hydrogen-bond acceptor.



(I)

The N,N'-bis(pentafluorophenyl)urea molecule has ten potential fluorine acceptors for the protons of the two secondary amines; nevertheless, the hydrogen-bond pattern is the $C(4)$ $[R^1_2(6)]$ pattern found for many other urea structures (*e.g.* N,N'-diphenylurea; Dannecker, Kopf & Rust, 1979). Both of the N—H groups in one molecule donate protons to the same carbonyl O atom, which is located in an adjacent molecule (at x, $\frac{1}{2}-y$, $\frac{1}{2}+z$). The N1···O and N2···O distances are 2.929 (3) and 2.798 (3) Å, the H1···O and H2···O distances are calculated to be 2.17 and 2.00 Å, respectively, and the corresponding N—H···O angles are calculated to be 147 and 154°. In contrast, the closest (calculated) intermolecular H···F contacts (also to a molecule at x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) are 2.87 (H1···F22) and 3.22 Å (H2···F16), and the closest intramolecular contacts are 2.81 (H1···F12) and 2.59 Å (H2···F26). The F atoms in this structure do not act as hydrogen-bond acceptors.

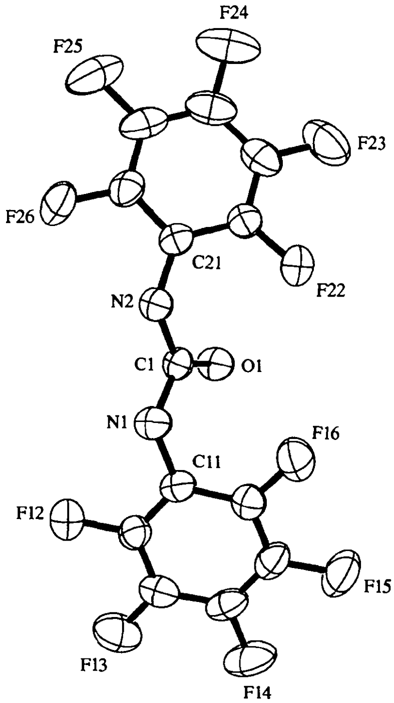


Fig. 1. The crystal structure of N,N'-bis(pentafluorophenyl)urea. Displacement ellipsoids are plotted at the 50% probability level. The numbers of the unlabeled atoms in the phenyl rings are the same as for the attached F atoms.

The pentafluorophenyl rings make torsion angles of about 50° with the urea moiety.

Experimental

The title compound was purchased from Lancaster Chemicals and recrystallized from 95% ethanol at room temperature.

Crystal data

$C_{13}H_2F_{10}N_2O$
$M_r = 392.17$
Orthorhombic
Pbca
$a = 11.799$ (2) Å
$b = 25.195$ (4) Å
$c = 9.009$ (2) Å
$V = 2678.2$ (9) Å^3
$Z = 8$
$D_x = 1.945$ Mg m^{-3}
$D_m = 1.98$ Mg m^{-3}
D_m measured by flotation in a 1:2 $CHBr_3$–$CHCl_3$ solution

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.7$–15.9°
$\mu = 0.220$ mm^{-1}
$T = 293$ (2) K
Block
0.5 × 0.2 × 0.2 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.92$, $T_{\max} = 0.96$
2603 measured reflections
2603 independent reflections

1696 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 28°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 32$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 240 min
intensity decay: 0.1%

Refinement

Refinement on F^2
$R(F) = 0.049$
$wR(F^2) = 0.107$
$S = 1.021$
2603 reflections
235 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 1.612P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.062$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Bond lengths are all normal. The average e.s.d. on a C—C bond length is 0.004 Å

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994).

The authors thank Professor John M. Hughes for advice on data collection and structure refinement.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Zsolnai, L. & Huttner, G. (1994). *ZORTEP. Interactive Graphics Program*. University of Heidelberg, Germany.

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(3*R*,2′*S*)-2′-*N*-Dibenzylamino-1,2:5,6-di-*O*-isopropylidène-α-D-allofuranose-3-spiro-3′-β-propiolactone

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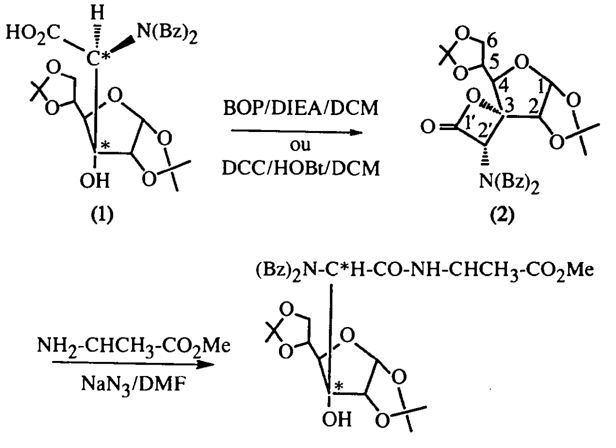
(*Reçu le 18 juin 1996, accepté le 21 novembre 1996*)

Abstract

The stereochemistry of (3*R*,2′*S*)-2′-*N*-dibenzylamino-1,2:5,6-di-*O*-isopropylidene-α-D-allofuranose-3-spiro-3′-propano-3-lactone, $C_{28}H_{33}NO_7$, has been established. The three five-membered rings adopt envelope conformations. The spiropropiolactone ring is practically planar.

Commentaire

Nous avons décrit la synthèse diastéréosélective du glycos-3-yl-α-amino ester (1) (Bouifraden, El Hadrami, Lavergne & Viallefont, 1993) dont la stéréochimie a été établie par la diffraction des rayons X (Chiaroni, Riche, Bouifraden, Ittobane, Lavergne & Viallefont, 1994). A partir de (1), nous avons pu préparer la spirolactone (2) dont l'ouverture par différents nucléophiles a été réalisée. En particulier l'action d'α-amino esters sur (2) conduit à une nouvelle série de glycopeptides dont par exemple le composé (3). La stéréochimie de la spirolactone (2) a été établie à partir de sa structure cristalline.



(1) (2) (3)

La molécule est représentée en perspective sur la Fig. 1. Comme le montrent les angles de torsion, les trois cycles à cinq chaînons sont dans des conformations de type enveloppe où les atomes O1, O4 et O6 sont en dehors du plan moyen des quatre autres atomes de ces cycles. Le cycle propiolactonique est approxima-

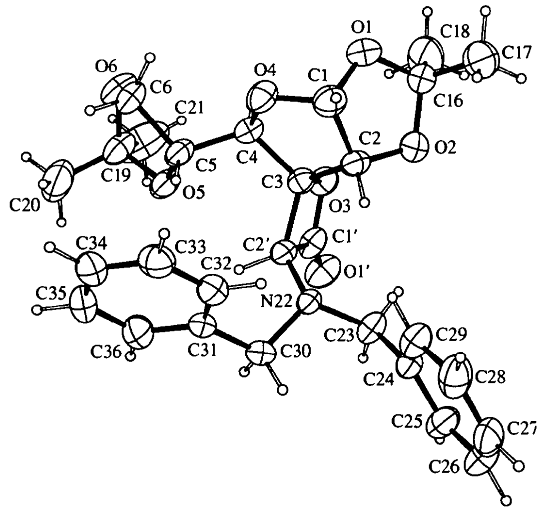


Fig. 1. Vue en perspective de (2). Ellipsoïdes de déplacement thermique tracés au niveau de probabilité de 30%.

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