Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$$B_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

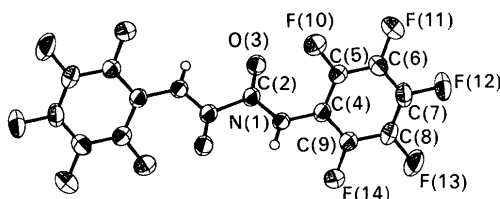|	x	y	z	B_{eq} ($Å^2$)
N(1)	0.9503 (1)	0.3442 (3)	0.3423 (1)	3.15 (4)
C(2)	0.9736 (1)	0.5469 (3)	0.4289 (1)	3.29 (5)
O(3)	0.9561 (1)	0.7815 (2)	0.4018 (1)	4.59 (4)
C(4)	0.9003 (1)	0.3854 (3)	0.2065 (1)	3.02 (5)
C(5)	0.9328 (1)	0.5723 (4)	0.1375 (1)	3.64 (5)
C(6)	0.8804 (1)	0.6168 (4)	0.0058 (1)	4.18 (6)
C(7)	0.7962 (1)	0.4688 (4)	−0.0612 (1)	4.07 (6)
C(8)	0.7652 (1)	0.2749 (4)	0.0035 (1)	3.81 (6)
C(9)	0.8157 (1)	0.2373 (3)	0.1362 (1)	3.28 (5)
F(10)	1.0184 (1)	0.7110 (2)	0.1966 (1)	5.45 (4)
F(11)	0.9131 (1)	0.8025 (3)	−0.0576 (1)	6.32 (5)
F(12)	0.7454 (1)	0.5122 (3)	−0.1887 (1)	6.16 (4)
F(13)	0.6848 (1)	0.1209 (3)	−0.0618 (1)	5.79 (4)
F(14)	0.7816 (1)	0.0533 (2)	0.1977 (1)	4.46 (3)

Table 2. *Bond lengths* ($Å$) *and angles* (°)

N(1)—C(2)	1.342 (2)	N(1)—C(4)	1.408 (2)
C(2)—O(3)	1.211 (2)	C(4)—C(5)	1.383 (3)
C(5)—F(10)	1.339 (2)	C(5)—C(6)	1.376 (2)
C(6)—F(11)	1.340 (2)	C(6)—C(7)	1.366 (2)
C(7)—F(12)	1.333 (2)	C(7)—C(8)	1.365 (3)
C(8)—F(13)	1.343 (2)	C(8)—C(9)	1.377 (2)
C(9)—F(14)	1.333 (2)	C(9)—C(4)	1.378 (2)
C(2)—C(2ⁱ)	1.533 (4)		

C(2)—N(1)—C(4)	122.3 (1)	O(3)—C(2)—N(1)	125.5 (1)
C(9)—C(4)—C(5)	117.0 (1)	C(9)—C(4)—N(1)	120.5 (1)
C(5)—C(4)—N(1)	122.5 (1)	F(10)—C(5)—C(6)	117.8 (1)
F(10)—C(5)—C(4)	120.7 (1)	C(6)—C(5)—C(4)	121.4 (1)
F(11)—C(6)—C(7)	119.8 (1)	F(11)—C(6)—C(5)	119.9 (1)
C(7)—C(6)—C(5)	120.3 (2)	F(12)—C(7)—C(8)	120.5 (1)
F(12)—C(7)—C(6)	120.2 (2)	C(8)—C(7)—C(6)	119.3 (1)
F(13)—C(8)—C(7)	120.2 (1)	F(13)—C(8)—C(9)	119.6 (2)
C(7)—C(8)—C(9)	120.2 (1)	F(14)—C(9)—C(8)	119.0 (1)
F(14)—C(9)—C(4)	119.4 (1)	C(8)—C(9)—C(4)	121.6 (1)
N(1)—C(2)—C(2ⁱ)	112.9 (2)	O(3)—C(2)—C(2ⁱ)	121.5 (2)

Symmetry code: (i) $2 - x, 1 - y, 1 - z$.

Fig. 1. *ORTEP*II drawing of (I). Ellipsoids are drawn at the 50% probability level while isotropic H thermal parameters are represented by spheres of arbitrary size.

Final atomic parameters are listed in Table 1.* Bond lengths and angles are listed in Table 2. Fig. 1 shows an *ORTEP*II drawing (Johnson, 1976) of the molecule with its atom labels.

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* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54496 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0521]

Related literature. The crystallographic studies of several urea cytokinins have been carried out in order to provide the basis for consideration of the stereochemical structure–activity relationships (Yamaguchi & Shudo, 1991).

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Structure of N-(2,3,5,6-Tetrafluoropyridyl)-N'-phenylurea

BY KENTARO YAMAGUCHI AND GO MATSUMURA

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AND NAOKI HAGA AND KOICHI SHUDO

Faculty of Pharmaceutical Sciences, University of Tokyo, 7-3-1 *Hongo, Bunkyo-ku, Tokyo* 113, *Japan*

Abstract. $C_{12}H_7F_4N_3O$, $M_r = 285.20$, monoclinic, $P2_1/n$, $a = 7.594$ (1), $b = 24.316$ (4), $c = 6.298$ (4) $Å$, $\beta = 96.47$ (1)°, $V = 1155.7$ (3) $Å^3$, $Z = 4$, $D_x = 1.639$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1.54050 $Å$, $\mu = 1.322$ mm^{-1}, $F(000) = 576$, $T = 295$ K, final $R = 0.055$ for 1194 reflections. The phenyl ring lies at

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

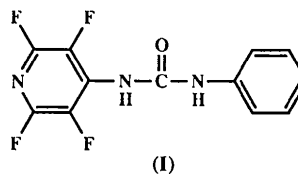|	x	y	z	$B_{eq}(Å^2)$
N(1)	0.9401 (4)	0.7895 (1)	0.6162 (6)	3.82 (11)
C(2)	1.0405 (5)	0.7426 (1)	0.5965 (7)	3.58 (12)
O(3)	1.1800 (3)	0.7338 (1)	0.7112 (4)	4.10 (9)
C(4)	1.0005 (5)	0.8334 (1)	0.7437 (6)	3.21 (12)
C(5)	1.0689 (5)	0.8281 (1)	0.9582 (7)	3.83 (13)
C(6)	1.1377 (6)	0.8742 (2)	1.0626 (7)	4.98 (17)
N(7)	1.1360 (5)	0.9237 (1)	0.9851 (6)	5.33 (14)
C(8)	1.0627 (6)	0.9290 (1)	0.7886 (8)	4.70 (16)
C(9)	0.9958 (5)	0.8860 (1)	0.6663 (6)	3.60 (13)
F(10)	1.0674 (3)	0.7799 (1)	1.0592 (4)	5.97 (9)
F(11)	1.2147 (3)	0.8674 (1)	1.2641 (4)	7.77 (12)
F(12)	1.0587 (4)	0.9794 (1)	0.7098 (5)	7.67 (11)
F(13)	0.9233 (3)	0.8954 (1)	0.4643 (4)	4.91 (8)
N(14)	0.9707 (4)	0.7084 (1)	0.4417 (5)	3.41 (10)
C(15)	1.0310 (5)	0.6555 (1)	0.3949 (6)	3.04 (12)
C(16)	1.1169 (5)	0.6212 (1)	0.5474 (7)	3.74 (13)
C(17)	1.1598 (6)	0.5685 (1)	0.4945 (8)	4.53 (15)
C(18)	1.1170 (6)	0.5483 (2)	0.2894 (9)	4.95 (17)
C(19)	1.0311 (7)	0.5831 (2)	0.1377 (9)	5.09 (17)
C(20)	0.9894 (6)	0.6360 (1)	0.1879 (7)	4.01 (14)

Table 2. *Selected bond lengths* ($Å$) *and angles* ($°$)

N(1)—C(4)	1.381 (5)	N(1)—C(2)	1.386 (5)
C(2)—O(3)	1.232 (4)	C(2)—N(14)	1.344 (5)
C(4)—C(9)	1.369 (6)	C(4)—C(5)	1.398 (5)
C(5)—F(10)	1.335 (5)	C(5)—C(6)	1.372 (7)
C(6)—N(7)	1.297 (7)	C(6)—F(11)	1.347 (5)
N(7)—C(8)	1.305 (6)	C(8)—F(12)	1.321 (5)
C(8)—C(9)	1.362 (6)	C(9)—F(13)	1.348 (4)
N(14)—C(15)	1.406 (5)		

C(4)—N(1)—C(2)	122.5 (3)	O(3)—C(2)—N(14)	124.2 (3)
O(3)—C(2)—N(1)	122.4 (3)	N(14)—C(2)—N(1)	113.5 (3)
C(9)—C(4)—N(1)	121.4 (3)	C(9)—C(4)—C(5)	115.0 (3)
N(1)—C(4)—C(5)	123.6 (3)	F(10)—C(5)—C(6)	120.8 (3)
F(10)—C(5)—C(4)	121.3 (3)	C(6)—C(5)—C(4)	117.9 (4)
N(7)—C(6)—F(11)	117.0 (4)	N(7)—C(6)—C(5)	126.1 (4)
F(11)—C(6)—C(5)	116.9 (4)	C(6)—N(7)—C(8)	115.8 (4)
N(7)—C(8)—F(12)	116.0 (4)	N(7)—C(8)—C(9)	123.4 (4)
F(12)—C(8)—C(9)	120.5 (4)	F(13)—C(9)—C(8)	119.4 (3)
F(13)—C(9)—C(4)	119.1 (3)	C(8)—C(9)—C(4)	121.5 (4)
C(2)—N(14)—C(15)	127.3 (3)		

32.1 (4)° to the urea moiety and the dihedral angle between the tetrafluoropyridyl and urea moieties is 46.9 (4)°. Two intermolecular hydrogen bonds, N—HO [N(1)—O(3) 3.097 (4) and N(14)—O(3) 2.869 (4) Å], were observed.

Experimental. (I) was prepared by the reaction of tetrafluoro-substituted aminopyridine with oxalyl chloride in C_6H_6, followed by elimination of CHOCl and condensation with aniline. Recrystallization from $CH_3(CH_2)_4CH_3/CH_3COOC_2H_5$ gave colorless prisms (0.15 × 0.04 × 0.50 mm) having m.p. 488.5–490.5 K. The combustion analysis of this compound was consistent with the structure. Rigaku AFC-5 four-circle diffractometer used with ω–2θ-scan method, ω-scan width (1.3 + 0.14tanθ)° and scan speed 16° min^{-1}. Lattice parameters obtained from least-squares analysis of 25 reflections with 2θ values ranging from 52 to 60°. Of 2006 reflections scanned

within index range h -8–8, k 0–27, l 0–7 up to $\sin\theta/\lambda < 0.56$ Å^{-1}, $R_{int} = 0.05$ for 175 reflections, 1725 unique reflections [$F > \sigma(F)$] classified as observed. Three standard reflections measured every 150 reflections, intensity variation < 3%. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985), a version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\Sigma w[||F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(|F_o|) + 0.005(F_c)]$; $\sigma(F_o)$ was determined from counting statistics. All H atoms located from the difference map and theoretical calculations were refined, initial thermal parameters set at equivalent isotropic thermal parameter of each bonded atom. Final discrepancy indices $R = 0.055$, $wR = 0.048$, $S = 1.553$ for 209 variables and 1195 reflections with $F > 3\sigma(F)$. Maximum $\Delta/\sigma = 0.28$ in final least-squares cycle. Final difference Fourier excursions 0.27 and -0.25 e Å^{-3}. All major computations performed on a PANAFACOM computer with the *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

(I)

Final atomic parameters are listed in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows an *ORTEP*II drawing (Johnson, 1976) of the molecule with its atom labels.
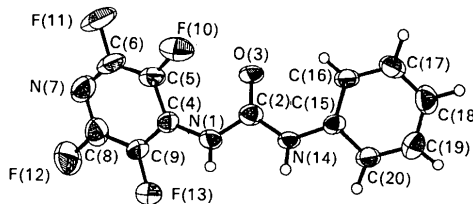
Fig. 1. *ORTEP*II drawing of (I). Ellipsoids are drawn at the 50% probability level while isotropic H thermal parameters are represented by spheres of arbitrary size.

Related literature. During the course of our studies on plant growth regulators, some heteroaromatic ureas, such as N-(2-chloro-4-pyridyl)-N'-phenylurea and N-(4-pyridyl)-N'-phenylurea showed a strong cytokinin activity (Takahashi, Shudo, Okamoto, Yamada & Isogai, 1978). Crystallographic studies on several urea cytokinins have been carried out (Yamaguchi & Shudo, 1991).

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Structures of (I) 5a,10b-*cis*- and (II) 5a,10b-*trans*-10-Methoxy-3,3,4-trimethyl-1,2a,3,4,5,5a,6,10b-octahydrobenzo[g][1,3]oxazolidino[2,3,4-*de*][2,4a]naphthyridine

BY KEVIN A. ANDERSEN, M. M. MILLER AND OREN P. ANDERSON*

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Abstract. (I) $C_{17}H_{24}N_2O_2$, $M_r = 288.39$, monoclinic, $P2_1/c$, $a = 13.033$ (2), $b = 17.763$ (4), $c = 6.761$ (1) Å, $\beta = 104.96$ (1)°, $V = 1512.0$ (5) Å^3, $Z = 4$, $D_x = 1.27$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 6.7$ cm^{-1}, $F(000) = 624$, $T = 115$ K, $R = 0.053$, $wR = 0.066$ for 1671 unique observed reflections. (II) $C_{17}H_{24}N_2O_2$, $M_r = 288.39$, monoclinic, $P2_1/n$, $a = 12.872$ (4), $b = 17.279$ (4), $c = 13.619$ (2) Å, $\beta = 96.59$ (2)°, $V = 3009$ (1) Å^3, $Z = 8$, $D_x = 1.27$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.8$ cm^{-1}, $F(000) = 1248$, $T = 148$ K, $R = 0.067$, $wR = 0.067$ for 3747 unique observed reflections. The stereoisomeric compounds are heterotricyclic about an N bridgehead atom.

Experimental. Crystals (colorless prisms) of (I) and (II) were obtained from hexane solutions by Dr Tomasz Glinka and Professor Robert M. Williams

(I) (II)

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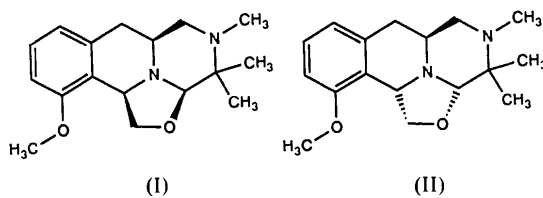
* To whom all correspondence should be addressed.

(Colorado State University). For (I), crystal size 0.48 × 0.47 × 0.26 mm; for (II), crystal size 0.24 × 0.21 × 0.50 mm. Nicolet *R3m* diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections [$2\theta_{av} = 53.97°$ for (I), 16.34° for (II)]. Data collected for (I) ($\theta/2\theta$ scans) to $(\sin\theta)/\lambda = 0.5313$ Å^{-1}, $-8 \leq h \leq 0$, $0 \leq k \leq 19$, $-14 \leq l \leq 14$. Data collected for (II) (Wyckoff ω scans) to $(\sin\theta)/\lambda = 0.5947$ Å^{-1}, $0 \leq h \leq 16$, $0 \leq k \leq 21$, $-17 \leq l \leq 17$. Three standard reflections [400, 020, 005 for (I); 400, 040, 004 for (II)] every 97 reflections, no trend in intensity observed; Lorentz and polarization corrections; no absorption correction applied for either crystal due to low absorption coefficients; 1887 unique reflections measured for (I), 5310 unique reflections measured for (II), 1671 reflections with $F_o > 2.5\sigma(F_o)$ observed for (I), 3747 for (II).

Both structures solved by direct methods (*SOLV*; Sheldrick, 1985); block-diagonal [max. 103 parameters/block; 202 parameters (I), 403 (II); data/parameters ratio = 8.3 (I), 9.3 (II)] weighted $\{w = [\sigma^2(F) + gF^2]^{-1}, g = 1.38 \times 10^{-3}$ for (I), 6.40×10^{-3} for (II)$\}$ least-squares refinement on *F*. H atoms in idealized positions [C—H = 0.96 Å, $U(H) = 1.2 \times U_{iso}(C)$]. Non-H atoms refined with anisotropic thermal parameters. At convergence $[(\Delta/\sigma)_{max} = 0.062$ (I), 0.209 (II), $(\Delta/\sigma)_{mean} = 0.017$ for last two cycles