Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	$U_{\rm eq}$
N1	0.3049 (3)	0.6303 (1)	0.3089 (1)	0.0493 (5)
N2	0.1935 (3)	0.5713 (1)	0.3400 (1)	0.0515 (5)
O1	0.5595 (3)	0.5895 (1)	0.4994 (1)	0.0694 (5)
O2	0.5984 (3)	0.7635 (1)	0.2608 (1)	0.0707 (5)
C1	0.6393 (4)	0.7302 (1)	0.3333 (1)	0.0485 (6)
C2	0.5031 (4)	0.6630 (1)	0.3622 (1)	0.0457 (6)
C3	0.6204 (4)	0.6436 (1)	0.4556 (1)	0.0490 (6)
C4	0.8215 (4)	0.7014 (1)	0.4852 (1)	0.0457 (6)
C5	0.9816 (5)	0.7109 (1)	0.5690 (1)	0.0593 (7)
C6	1.1559 (5)	0.7706 (1)	0.5777 (2)	0.0665 (8)
C7	1.1722 (5)	0.8194 (1)	0.5054 (2)	0.0641 (8)
C8	1.0124 (4)	0.8110 (1)	0.4219 (2)	0.0547 (7)
C9	0.8380 (4)	0.7515 (1)	0.4129 (1)	0.0452 (6)
C10	−0.0163 (4)	0.5336 (1)	0.2845 (1)	0.0487 (6)
C11	−0.1370 (5)	0.4728 (1)	0.3227 (2)	0.0604 (7)
C12	−0.3391 (5)	0.4341 (1)	0.2706 (2)	0.0687 (8)
C13	−0.4220 (5)	0.4554 (1)	0.1816 (2)	0.0691 (8)
C14	−0.3017 (5)	0.5161 (1)	0.1441 (2)	0.0713 (9)
C15	−0.0988 (4)	0.5562 (1)	0.1958 (2)	0.0600 (7)

Table 2. *Selected geometric parameters* (Å, °)

N1—N2	1.303 (3)	C5—C6	1.379 (3)
N1—C2	1.316 (2)	C6—C7	1.383 (4)
N2—C10	1.414 (2)	C7—C8	1.386 (4)
O1—C3	1.230 (3)	C8—C9	1.377 (3)
O2—C1	1.217 (2)	C10—C11	1.386 (3)
C1—C2	1.472 (3)	C10—C15	1.375 (3)
C1—C9	1.486 (2)	C11—C12	1.375 (3)
C2—C3	1.466 (2)	C12—C13	1.372 (4)
C3—C4	1.476 (3)	C13—C14	1.380 (3)
C4—C5	1.393 (2)	C14—C15	1.388 (3)
C4—C9	1.399 (2)		
N2—N1—C2	118.0 (2)	C5—C6—C7	121.1 (2)
N1—N2—C10	120.1 (2)	C6—C7—C8	121.7 (2)
O2—C1—C2	128.6 (2)	C7—C8—C9	117.5 (2)
O2—C1—C9	125.7 (2)	C1—C9—C8	128.9 (2)
C2—C1—C9	105.7 (1)	C4—C9—C8	121.5 (2)
N1—C2—C1	121.9 (1)	C1—C9—C4	109.6 (2)
N1—C2—C3	129.4 (2)	N2—C10—C15	121.5 (2)
C1—C2—C3	108.8 (1)	N2—C10—C11	117.6 (2)
O1—C3—C2	125.8 (2)	C11—C10—C15	120.9 (2)
O1—C3—C4	127.8 (2)	C10—C11—C12	119.5 (2)
C2—C3—C4	106.5 (2)	C11—C12—C13	120.5 (2)
C3—C4—C5	130.2 (2)	C12—C13—C14	119.8 (2)
C3—C4—C9	109.4 (1)	C13—C14—C15	120.7 (2)
C5—C4—C9	120.4 (2)	C10—C15—C14	118.7 (2)
C4—C5—C6	117.9 (2)		

All H atoms were located unambiguously in $\Delta\rho$ maps and refined with isotropic displacement parameters. Programs used for data collection, cell refinement and data reduction: Enraf–Nonius CAD-4 software. Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983). Program used for molecular graphics: *ORTEP*II (Johnson, 1976).

References

Bigelow, R. W. (1976). *J. Phys. Chem.* **80**, 2694–2700.

Hocaoğlu, N., Uyar, T. & Türker, L. (1990). *Dyes Pigment.* **12**, 187–195.

İde, S., Kendi, E. & Ertan, N. (1994). In preparation.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Özbey, S., Kendi, E., Hocaoğlu, N., Uyer, T. & Mak, T. C. W. (1993). *Acta Cryst.* C**49**, 2165–2167.

Özbey, S., Kendi, E., Hocaoğlu, N., Uyar, T., Mak, T. C. W. & Cascarano, G. L. (1994). *Acta Cryst.* C**50**, 629–631.

Pendergrass, D. P., Paul, J. C. & Curtin, D. Y. (1972). *J. Am. Chem. Soc.* **94**, 8730–8737.

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.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1109). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1994). C**50**, 1507–1511

Structures of (1) *N*-(2-Pyridyl)-, (2) *N*-(3-Pyridyl)- and (3) *N*-(4-Pyridyl)-*N*'-(4-chlorophenyl)urea

PIERRE LE MAGUERES AND LAHCÈNE OUAHAB*

Laboratoire de Chimie du Solide et
Inorganique Moléculaire, URA 1495 CNRS,
Université de Rennes I, 35042 Rennes CEDEX, France

ALEXANDRE HOCQUET

ENS Cachan, 94235 Cachan CEDEX, France

JOSETTE FOURNIER*

CREPA, 8 rue Becquerel, 49040 Beaucouzé, France

(*Received* 9 *June* 1993; *accepted* 10 *December* 1993)

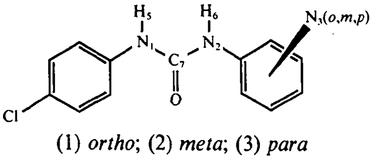
Abstract

The three compounds ($C_{12}H_{10}ClN_3O$) have similar molecular structures but differ in the position of the N atom in the pyridyl ring. The effects are reflected in the existence of some intra- or intermolecular hydrogen bonds between the urea N atoms and the pyridyl N atom {1.89 (4) [(1) intra], 2.21 (3) and 2.14 (3) [(2) inter], 2.17 (2) [(3) inter]} and between the carbonyl O and the pyridyl N atom {2.07 (4) Å [(1) inter]}. From the intermolecular distances the crystal structure of (1) can be viewed as a succession of 'dimers' in three dimensions, and that of (2) or (3) as a one-dimensional molecular chain.

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

Comment

As plant growth regulators, several pyridyl ureas show high cytokinin activity (Vassilev, Izorwska, Yonova & Dimcheva, 1987). Structural studies have been carried out recently on *N*-(2,3,5,6-tetrafluoropyridyl)-*N*′-phenylurea (Yamaguchi, Matsumura, Haga & Shudo, 1992), *N*-(4-pyridyl)-*N*′-phenylurea, *N*-(3-pyridyl)-*N*′-phenylurea, *N*-(3-chloro-4-pyridyl)-*N*′-phenylurea, *N*-(2-chloro-4-pyridyl)-*N*′-phenylurea and *N*-(4-pyridyl)-*N*′-phenyl-*N*′-methylurea (Yamaguchi & Shudo, 1991). Intramolecular hydrogen bonds in (1) have been studied in solution using ^{1}H and ^{13}C NMR (Sudha & Sathyanarayana, 1985).



(1) *ortho*; (2) *meta*; (3) *para*

The N3 atom in the pyridyl ring is located at a different position in each of the title compounds, namely, *ortho* in (1), *meta* in (2) and *para* in (3). The position of the N3 atom influences the molecular and crystal structures of the compounds because hydrogen bonding involving N3 can occur in order to obtain the most stable structure. In compound (1) (Fig. 1) the N3 atom is in the *ortho* position and the molecular structure shows a *ZEZ* configuration for the two rings with respect to the urea core so as to create an intramolecular hydrogen bond N3—H5 [1.89 (4) Å]. The other two compounds keep the *ZZZ* or *ZZ* configuration with respect to the central unit and do not possess an intramolecular hydrogen bridge.

The crystal structures have revealed different intermolecular hydrogen bonds for the three compounds. Thus, in compound (1) (Fig. 1), both the O atoms and the H6 atom of the central unit interact with H6 and O, respectively, of a neighbouring molecule [O···H6 2.07 (4) Å]. This structure can be viewed as a succession of dimers in three dimensions. In compound (2) (Fig. 2), the *meta* position of N3 in the pyridyl ring leads to two intermolecular hydrogen bonds between N3 and two H atoms of another molecule [N3—H5 2.21 (3) and N3—H6 2.14 (3) Å]. In this case, a one-dimensional molecular chain is formed along the $b + c$ direction. Finally, in compound (3) (Fig. 3), the N3 atom in the *para* position forms only one intermolecular hydrogen bridge [N3—H5 2.17 (2) Å] and creates a one-dimensional molecular chain along the *b* axis.

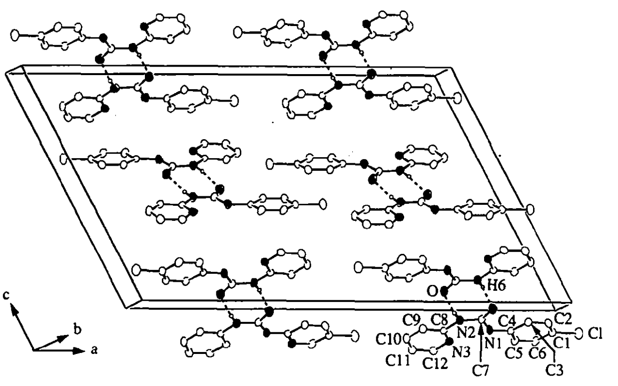


Fig. 1. View of the crystal structure of (1).

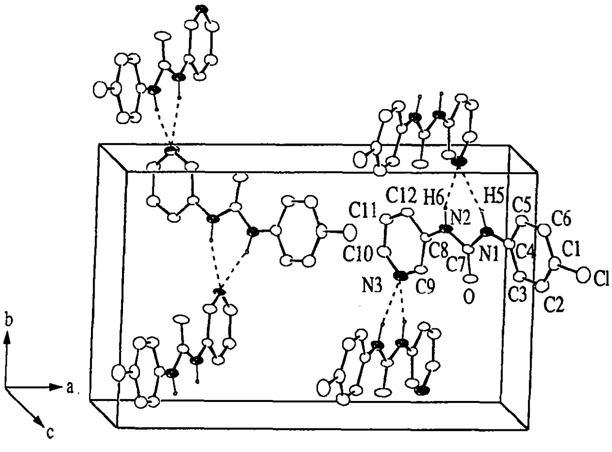


Fig. 2. View of the crystal structure of (2).

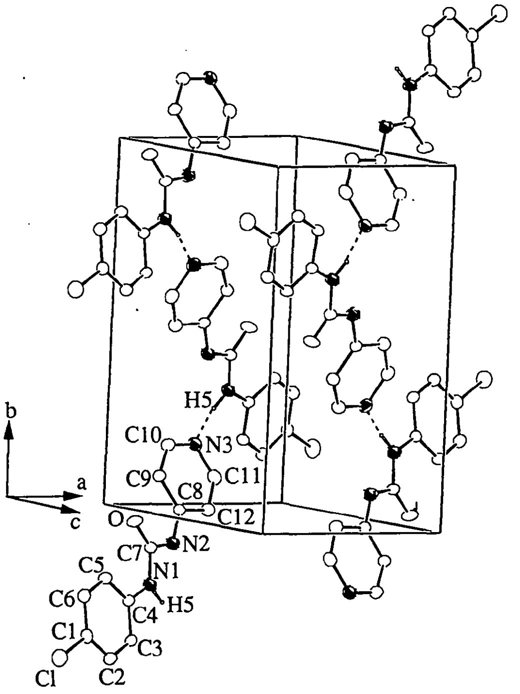


Fig. 3. View of the crystal structure of (3).

Moreover, for the three compounds, the C7—O, N1—C4 and N2—C8 bond lengths are very close (see Tables 4, 5 and 6). The dihedral angles between the pyridyl and chlorophenyl rings are 6, 3 and 11°, respectively, for the three compounds.

Experimental

Compounds (1), (2) and (3) were prepared by the reaction in 1,4-dioxane of 4-chlorophenylisocyanate with 2-aminopyridine, 3-aminopyridine and 4-aminopyridine, respectively. Recrystallization from ethanol yielded white needles of (1) and colourless prisms of (2) and (3), which have melting points of 487, 497 and 504 K, respectively.

Compound (1)

Crystal data

$C_{12}H_{10}ClN_3O$
$M_r = 247.69$
Monoclinic
$C2/c$
$a = 27.099\ (8)$ Å
$b = 5.855\ (5)$ Å
$c = 16.636\ (8)$ Å
$\beta = 116.69\ (4)°$
$V = 2359\ (3)$ Å^3
$Z = 8$
$D_x = 1.395$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4$–$9.5°$
$\mu = 0.307$ mm^{-1}
$T = 293$ K
Needle
1.0 × 0.2 × 0.2 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: none
2106 measured reflections
2014 independent reflections
843 observed reflections $[I \geq 3\sigma(I)]$

$R_{\rm int} = 0.024$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 32$
$k = 0 \rightarrow 6$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity variation: <4%

Refinement

Refinement on F
$R = 0.049$
$wR = 0.059$
$S = 1.383$
843 reflections
195 parameters
$w = 4F_o^2/[\sigma^2(I) + (0.07F_o^2)^2]$
$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.268$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.020$ e Å^{-3}

Extinction correction: $|F_c|(1 + gI_c)^{-1}$
Extinction coefficient: $g = 5.694 \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (2)

Crystal data

$C_{12}H_{10}ClN_3O$
$M_r = 247.69$
Orthorhombic
$Pna2_1$
$a = 16.628\ (5)$ Å
$b = 10.573\ (2)$ Å
$c = 6.559\ (6)$ Å
$V = 1153\ (1)$ Å^3
$Z = 4$
$D_x = 1.427$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–$14°$
$\mu = 0.314$ mm^{-1}
$T = 293$ K
Prisms
0.7 × 0.3 × 0.1 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: none
1112 measured reflections
1112 independent reflections
844 observed reflections $[I \geq 3\sigma(I)]$

$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity variation: <2%

Refinement

Refinement on F
$R = 0.033$
$wR = 0.037$
$S = 1.464$
844 reflections
194 parameters
$w = 4F_o^2/[\sigma^2(I) + (0.04F_o^2)^2]$
$(\Delta/\sigma)_{\rm max} = 0.32$
$\Delta\rho_{\rm max} = 0.284$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.027$ e Å^{-3}

Extinction correction: $|F_c|(1 + gI_c)^{-1}$
Extinction coefficient: $g = 1.869 \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (3)

Crystal data

$C_{12}H_{10}ClN_3O$
$M_r = 247.69$
Monoclinic
$P2_1/n$
$a = 7.000\ (10)$ Å
$b = 12.813\ (2)$ Å
$c = 12.524\ (2)$ Å
$\beta = 94.30\ (5)°$
$V = 1120\ (2)$ Å^3
$Z = 4$
$D_x = 1.469$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7$–$12°$
$\mu = 0.323$ mm^{-1}
$T = 293$ K
Prism
1.5 × 0.3 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: none
2376 measured reflections
2006 independent reflections
1546 observed reflections $[I \geq 3\sigma(I)]$

$R_{\rm int} = 0.015$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 15$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity variation: <1%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.063$
$S = 1.546$

Extinction correction: $|F_c|(1 + gI_c)^{-1}$
Extinction coefficient: 1.083×10^{-7}

1546 reflections
195 parameters
$w = 4F_o^2/[\sigma^2(I) + (0.07F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.279$ e Å^{-3}
$\Delta\rho_{min} = -0.012$ e Å^{-3}

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (1)

B_{iso} for H atoms; $B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	B_{iso}/B_{eq}
Cl	0.50347 (6)	0.1326 (3)	0.1148 (1)	6.88 (4)
O	0.7003 (1)	0.6718 (5)	0.0349 (2)	4.67 (8)
N1	0.7160 (1)	0.3325 (6)	0.1108 (2)	3.70 (9)
N2	0.7822 (1)	0.4972 (6)	0.0764 (2)	3.89 (9)
N3	0.8154 (1)	0.1522 (6)	0.1525 (2)	3.7 (1)
C1	0.5659 (2)	0.1916 (8)	0.1139 (3)	3.9 (1)
C2	0.5739 (2)	0.4015 (9)	0.0827 (3)	4.5 (1)
C3	0.6223 (2)	0.4521 (8)	0.0802 (3)	4.0 (1)
C4	0.6654 (2)	0.2971 (7)	0.1112 (3)	3.2 (1)
C5	0.6574 (2)	0.0865 (8)	0.1450 (3)	4.1 (1)
C6	0.6083 (2)	0.0398 (8)	0.1457 (3)	4.5 (1)
C7	0.7305 (2)	0.5074 (8)	0.0723 (3)	3.7 (1)
C8	0.8242 (2)	0.3356 (7)	0.1135 (3)	3.3 (1)
C9	0.8729 (2)	0.3756 (8)	0.1090 (3)	4.2 (1)
C10	0.9146 (2)	0.215 (1)	0.1445 (3)	5.0 (1)
C11	0.9061 (2)	0.0232 (9)	0.1859 (3)	4.9 (1)
C12	0.8567 (2)	−0.0021 (9)	0.1878 (3)	4.3 (1)
H1	0.549 (1)	0.491 (5)	0.064 (2)	1.5 (7)
H2	0.629 (2)	0.578 (8)	0.059 (3)	5 (1)
H3	0.692 (1)	−0.005 (7)	0.172 (2)	3.4 (9)
H4	0.603 (1)	−0.085 (6)	0.167 (2)	2.7 (9)
H5	0.745 (2)	0.247 (7)	0.138 (2)	4 (1)
H6	0.791 (1)	0.588 (7)	0.049 (2)	4 (1)
H7	0.874 (2)	0.493 (7)	0.077 (2)	4 (1)
H8	0.952 (1)	0.244 (7)	0.144 (2)	4 (1)
H9	0.936 (2)	−0.075 (7)	0.209 (3)	5 (1)
H10	0.848 (2)	−0.119 (7)	0.215 (3)	7 (1)

Table 2. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (2)

B_{iso} for H atoms; $B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	B_{iso}/B_{eq}
Cl	0.45665 (5)	0.2235 (1)	0.579†	6.06 (2)
O	0.6664 (2)	0.1102 (2)	−0.3099 (5)	5.46 (5)
N1	0.6408 (2)	0.3070 (2)	−0.1821 (5)	4.01 (5)
N2	0.7189 (2)	0.2865 (2)	−0.4617 (5)	4.14 (6)
N3	0.7981 (2)	0.0616 (2)	−0.8433 (5)	5.47 (7)
C1	0.5112 (2)	0.2445 (3)	0.3548 (6)	4.73 (7)
C2	0.5307 (2)	0.1431 (3)	0.2343 (7)	5.19 (8)
C3	0.5737 (2)	0.1595 (3)	0.0574 (6)	4.64 (7)
C4	0.5976 (2)	0.2808 (3)	−0.0040 (5)	3.68 (6)
C5	0.5775 (2)	0.3818 (3)	0.1213 (6)	4.47 (7)
C6	0.5351 (2)	0.3644 (3)	0.2994 (7)	5.14 (8)
C7	0.6748 (2)	0.2245 (2)	−0.3171 (5)	3.69 (6)
C8	0.7563 (2)	0.2351 (2)	−0.6345 (5)	3.35 (6)
C9	0.7609 (2)	0.1065 (3)	−0.6773 (6)	4.43 (7)
C10	0.8320 (2)	0.1425 (3)	−0.9737 (6)	5.04 (8)
C11	0.8311 (2)	0.2715 (3)	−0.9415 (7)	4.63 (7)
C12	0.7930 (2)	0.3170 (2)	−0.7710 (6)	4.00 (7)
H1	0.511 (2)	0.060 (3)	0.287 (7)	7 (1)
H2	0.585 (2)	0.086 (3)	−0.043 (6)	6.9 (9)
H3	0.592 (2)	0.460 (3)	0.079 (7)	6.0 (7)
H4	0.518 (2)	0.432 (3)	0.375 (5)	4.7 (7)
H5	0.652 (2)	0.386 (3)	−0.211 (6)	5.6 (8)
H6	0.721 (2)	0.374 (2)	−0.457 (5)	3.6 (6)
H7	0.731 (2)	0.049 (3)	−0.576 (5)	5.9 (8)
H8	0.866 (2)	0.110 (3)	−1.105 (5)	5.3 (8)
H9	0.864 (2)	0.336 (3)	−1.045 (8)	8 (1)
H10	0.793 (1)	0.405 (2)	−0.747 (5)	4.1 (6)

† Coordinate fixed to define origin.

Table 3. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (3)

B_{iso} for H atoms; $B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	B_{iso}/B_{eq}
Cl	−0.27462 (9)	0.86304 (7)	1.12818 (6)	6.24 (2)
O	0.3152 (2)	0.5139 (1)	0.9015 (1)	4.74 (4)
N1	0.3933 (2)	0.6855 (1)	0.9271 (2)	3.71 (4)
N2	0.5917 (2)	0.5796 (1)	0.8410 (2)	3.80 (4)
N3	0.8294 (3)	0.3229 (2)	0.6885 (2)	4.69 (5)
C1	−0.0764 (3)	0.8084 (2)	1.0717 (2)	3.91 (5)
C2	0.0762 (3)	0.8706 (2)	1.0516 (2)	3.95 (5)
C3	0.2303 (3)	0.8276 (2)	1.0044 (2)	3.58 (4)
C4	0.2322 (3)	0.7223 (2)	0.9769 (2)	3.25 (4)
C5	0.0762 (3)	0.6606 (2)	0.9995 (2)	3.68 (5)
C6	−0.0778 (3)	0.7036 (2)	1.0464 (2)	3.96 (5)
C7	0.4228 (3)	0.5869 (2)	0.8910 (2)	3.40 (4)
C8	0.6638 (3)	0.4920 (2)	0.7929 (2)	3.32 (4)
C9	0.5625 (3)	0.4007 (2)	0.7720 (2)	4.10 (5)
C10	0.6496 (4)	0.3211 (2)	0.7196 (2)	4.78 (6)
C11	0.9250 (3)	0.4112 (2)	0.7118 (2)	4.70 (5)
C12	0.8514 (3)	0.4954 (2)	0.7621 (2)	4.22 (5)
H1	0.078 (3)	0.939 (2)	1.074 (2)	5.0 (6)
H2	0.328 (3)	0.869 (2)	0.987 (2)	3.7 (5)
H3	0.070 (3)	0.591 (2)	0.984 (2)	4.1 (5)
H4	−0.179 (3)	0.658 (2)	1.065 (2)	5.4 (6)
H5	0.487 (3)	0.733 (2)	0.909 (2)	5.8 (6)
H6	0.660 (3)	0.636 (2)	0.841 (2)	3.8 (5)
H7	0.439 (3)	0.389 (2)	0.794 (2)	4.5 (5)
H8	0.574 (3)	0.259 (2)	0.699 (2)	6.1 (7)
H9	1.060 (3)	0.412 (2)	0.691 (2)	5.6 (6)
H10	0.927 (4)	0.557 (2)	0.774 (2)	7.1 (7)

Table 4. *Selected geometric parameters* (Å, °) *for* (1)

Cl—C1	1.733 (6)	C3—C4	1.383 (6)
O—C7	1.235 (5)	C3—H2	0.88 (5)
N1—C4	1.389 (6)	C4—C5	1.412 (7)
N1—C7	1.356 (7)	C5—C6	1.364 (8)
N1—H5	0.87 (4)	C5—H3	0.99 (4)
N2—C7	1.375 (7)	C6—H4	0.86 (4)
N2—C8	1.392 (5)	C8—C9	1.376 (8)
N2—H6	0.80 (4)	C9—C10	1.383 (7)
N3—C8	1.330 (6)	C9—H7	0.88 (4)
N3—C12	1.351 (6)	C10—C11	1.388 (8)
C1—C2	1.389 (7)	C10—H8	1.02 (4)
C1—C6	1.358 (6)	C11—C12	1.361 (8)
C2—C3	1.362 (8)	C11—H9	0.92 (5)
C2—H1	0.80 (3)	C12—H10	0.90 (5)
C4—N1—C7	127.6 (3)	C1—C6—C5	121.6 (5)
C4—N1—H5	125 (3)	C1—C6—H4	116 (2)
C7—N1—H5	107 (3)	C5—C6—H4	122 (2)
C7—N2—C8	131.5 (4)	O—C7—N1	124.0 (5)
C7—N2—H6	119.4 (4)	O—C7—N2	119.3 (5)
C8—N2—H6	108.6 (4)	N1—C7—N2	116.7 (4)
C8—N3—C12	117.2 (4)	N2—C8—N3	118.4 (4)
Cl—C1—C2	119.8 (4)	N2—C8—C9	118.2 (4)
Cl—C1—C6	121.6 (4)	N3—C8—C9	123.4 (4)
C2—C1—C6	118.5 (5)	C8—C9—C10	118.7 (5)
C1—C2—C3	121.2 (4)	C8—C9—H7	118.2 (4)
C1—C2—H1	118 (3)	C10—C9—H7	122.5 (6)
C3—C2—H1	120 (3)	C9—C10—C11	118.5 (5)
C2—C3—C4	120.6 (5)	C9—C10—H8	120.0 (5)
C2—C3—H2	126 (3)	C11—C10—H8	121.4 (5)
C4—C3—H2	114 (3)	C10—C11—C12	118.9 (4)
N1—C4—C3	124.7 (4)	C10—C11—H9	114.2 (6)
N1—C4—C5	117.4 (4)	C12—C11—H9	126.9 (6)
C3—C4—C5	117.9 (5)	N3—C12—C11	123.3 (5)
C4—C5—C6	120.1 (4)	N3—C12—H10	111.9 (5)
C4—C5—H3	112 (2)	C11—C12—H10	124.7 (5)
C6—C5—H3	128 (2)		

Table 5. *Selected geometric parameters* (Å, °) *for* (2)

Cl—C1	1.740 (4)	C3—C4	1.401 (4)
O—C7	1.217 (3)	C3—H2	1.03 (4)
N1—C4	1.399 (4)	C4—C5	1.389 (4)

N1—C7	1.365 (4)	C5—C6	1.376 (5)
N1—H5	0.88 (3)	C5—H3	0.91 (3)
N2—C7	1.367 (4)	C6—H4	0.91 (3)
N2—C8	1.402 (4)	C8—C9	1.391 (4)
N2—H6	0.93 (2)	C8—C12	1.387 (4)
N3—C9	1.340 (5)	C9—H7	1.03 (3)
N3—C10	1.334 (5)	C10—C11	1.380 (4)
C1—C2	1.370 (5)	C10—H8	1.09 (3)
C1—C6	1.378 (5)	C11—C12	1.373 (5)
C2—C3	1.374 (6)	C11—H9	1.11 (4)
C2—H1	1.00 (3)	C12—H10	0.95 (3)
C4—N1—C7	128.8 (2)	C1—C6—C5	119.7 (3)
C4—N1—H5	118 (2)	C1—C6—H4	119 (2)
C7—N1—H5	113 (2)	C5—C6—H4	121 (2)
C7—N2—C8	127.8 (2)	O—C7—N1	124.2 (3)
C7—N2—H6	118 (2)	O—C7—N2	124.3 (3)
C8—N2—H6	114 (2)	N1—C7—N2	111.5 (2)
C9—N3—C10	119.2 (3)	N2—C8—C9	124.5 (3)
Cl—C1—C2	120.7 (3)	N2—C8—C12	118.4 (2)
Cl—C1—C6	119.4 (3)	C9—C8—C12	117.1 (3)
C2—C1—C6	119.9 (3)	N3—C9—C8	122.5 (3)
C1—C2—C3	120.8 (3)	N3—C9—H7	122 (2)
C1—C2—H1	114 (2)	C8—C9—H7	115 (2)
C3—C2—H1	125 (2)	N3—C10—C11	122.1 (3)
C2—C3—C4	120.4 (3)	N3—C10—H8	122 (2)
C2—C3—H2	122 (2)	C11—C10—H8	116 (2)
C4—C3—H2	117 (2)	C10—C11—C12	118.4 (3)
N1—C4—C3	124.5 (3)	C10—C11—H9	120 (2)
N1—C4—C5	117.7 (3)	C12—C11—H9	121 (2)
C3—C4—C5	117.7 (3)	C8—C12—C11	120.7 (3)
C4—C5—C6	121.5 (3)	C8—C12—H10	120 (2)
C4—C5—H3	117 (3)	C11—C12—H10	119 (2)
C6—C5—H3	121 (3)		

Table 6. *Selected geometric parameters* (Å, °) *for* (3)

Cl—C1	1.751 (2)	C3—C4	1.392 (3)
O—C7	1.214 (3)	C3—H2	0.91 (2)
N1—C4	1.410 (3)	C4—C5	1.394 (3)
N1—C7	1.363 (3)	C5—C6	1.380 (3)
N1—H5	0.94 (2)	C5—H3	0.92 (2)
N2—C7	1.382 (3)	C6—H4	0.96 (2)
N2—C8	1.386 (3)	C8—C9	1.382 (3)
N2—H6	0.87 (2)	C8—C12	1.397 (3)
N3—C10	1.345 (3)	C9—C10	1.380 (4)
N3—C11	1.336 (3)	C9—H7	0.94 (2)
C1—C2	1.372 (3)	C10—H8	0.97 (2)
C1—C6	1.379 (4)	C11—C12	1.368 (4)
C2—C3	1.383 (3)	C11—H9	1.00 (2)
C2—H1	0.92 (2)	C12—H10	0.96 (3)
C4—N1—C7	127.1 (2)	C1—C6—C5	119.6 (2)
C4—N1—H5	119 (2)	C1—C6—H4	122 (2)
C7—N1—H5	113 (2)	C5—C6—H4	119 (1)
C7—N2—C8	127.1 (2)	O—C7—N1	124.6 (2)
C7—N2—H6	116 (1)	O—C7—N2	123.7 (2)
C8—N2—H6	117 (1)	N1—C7—N2	111.6 (2)
C10—N3—C11	114.5 (2)	N2—C8—C9	124.6 (2)
Cl—C1—C2	119.6 (2)	N2—C8—C12	118.4 (2)
Cl—C1—C6	119.4 (2)	C9—C8—C12	117.0 (2)
C2—C1—C6	121.1 (6)	C8—C9—C10	118.5 (2)
C1—C2—c3	119.3 (2)	C8—C9—H7	124 (1)
C1—C2—H1	119 (1)	C10—C9—H7	118 (1)
C3—C2—H1	121 (1)	N3—C10—C9	125.6 (2)
C2—C3—C4	120.9 (2)	N3—C10—H8	116 (1)
C2—C3—H2	120 (1)	C9—C10—H8	118 (1)
C4—C3—H2	119 (1)	N3—C11—C12	124.6 (2)
N1—C4—C3	117.1 (2)	N3—C11—H9	115 (1)
N1—C4—C5	124.4 (2)	C12—C11—H9	121 (1)
C3—C4—C5	118.5 (2)	C8—C12—C11	119.8 (2)
C4—C5—C6	120.6 (2)	C8—C12—H10	120 (2)
C4—C5—H3	122 (1)	C11—C12—H10	120 (2)
C6—C5—H3	117 (1)		

The structure was solved by direct methods and successive difference Fourier syntheses. All of the H atoms were located by difference Fourier synthesis. Full-matrix least-squares anisotropic (β_{ij}) refinement was carried out for all atoms except the H atoms, which were refined isotropically. All calculations were performed on a MicroVAX 3100 using the *MolEN* programs (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf-Nonius, Delft, The Netherlands.

Sudha, L. & Sathyanarayana, N. (1985). *J. Mol. Struct.* **131**, 141-146.

Vassilev, G., Izorwska, P., Yonova, P. & Dimcheva, Z. (1987). *Dokl. Bolg. Akad. Nauk*, **40**, 109-112.

Yamaguchi, K., Matsumura, G., Haga, N. & Shudo, K. (1992). *Acta Cryst.* C**48**, 559-561.

Yamaguchi, F. & Shudo, K. (1991). *J. Agric. Food Chem.* **39**, 793-796.

Acta Cryst. (1994). C**50**, 1511-1514

1-Carboxymethyl-3-hydroxy-2-methyl-4(1*H*)-pyridinone (Monoclinic Form 2)

CHRIS ORVIG, STEVEN J. RETTIG AND ZAIHUI ZHANG

Department of Chemistry,
University of British Columbia, 2036 Main Mall,
Vancouver, BC, Canada V6T 1Z1

(*Received* 7 *May* 1993; *accepted* 17 *January* 1994)

Abstract

The structure of a second monoclinic polymorph of 1-carboxymethyl-3-hydroxy-2-methyl-4-pyridinone (3-hydroxy-2-methyl-4-oxopyridine-1-acetic acid, $C_8H_9NO_4$) is reported. The molecule has a delocalized zwitterionic structure much like that reported for form 1 [Zhang, Rettig & Orvig (1992). *Can. J. Chem.* **70**, 763–770]. The structure contains a legitimate intermolecular C—H···O hydrogen bond [H···O = 2.18 (2), C···O = 3.155 (3) Å, C—H···O = 165 (2)°]. The two forms differ primarily in the details of the hydrogen bonding.

Comment

Tautomerism is one of the most important features of heterocyclic compounds. For 4-pyridinones there