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4,4′-Bipyridyl–2-chloro-4-nitrobenzoic acid (1/2)

Kazuma Gotoh and Hiroyuki Ishida*

Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan
Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

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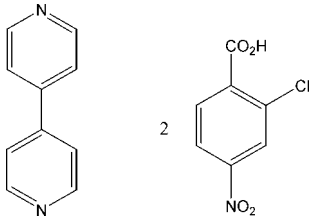
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 15.3.

In the crystal structure of the title 1:2 cocrystal, $C_{10}H_8N_2\cdot 2C_7H_4ClNO_4$, the two components are held together by short O—H···N hydrogen bonds. The pyridine rings of the bipyridyl molecule are twisted by 27.90 (5)° with respect to each other. The 1:2 units are connected by C—H···O hydrogen bonds, forming a centrosymmetric supramolecular ring.

Related literature

For related compounds, see: Ishida & Fukunaga (2004); Ishida *et al.* (2004).



Experimental

Crystal data

$C_{10}H_8N_2\cdot 2C_7H_4ClNO_4$
M_r = 559.32
Monoclinic, $P2_1/n$
a = 12.0875 (11) Å
b = 8.0695 (9) Å
c = 24.610 (2) Å
β = 101.143 (2)°
V = 2355.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 173 (1) K
0.48 × 0.45 × 0.38 mm

Data collection

Rigaku R-AXIS RAPID-II diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.780, T_{max} = 0.880
19103 measured reflections
5379 independent reflections
4838 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.08
5379 reflections
351 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*O*···N3	0.93 (3)	1.70 (3)	2.6330 (14)	176 (2)
O6—H6*O*···N4	1.05 (3)	1.55 (3)	2.5984 (14)	177 (3)
C10—H10···O4^{i}	0.95	2.41	3.2271 (16)	144
C13—H13···O3^{ii}	0.95	2.34	3.2926 (17)	175
C15—H15···O3^{iii}	0.95	2.59	3.4360 (17)	149
C24—H24···O8^{iv}	0.95	2.52	3.2552 (19)	134

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2205).

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  doi:10.1107/S1600536807052865

supplementary materials

Acta Cryst. (2007). E**63**, o4500 [**doi:10.1107/S1600536807052865**]

4,4'-Bipyridyl-2-chloro-4-nitrobenzoic acid (1/2)

K. Gotoh and H. Ishida

Comment

The title compound was investigated as part of a study on *D*—H···*A* hydrogen bonding (*D* = N, O or C; *A* = N, O or Cl) in chloro- and nitro-substituted benzoic acid–amine systems (Ishida & Fukunaga, 2004; Ishida *et al.*, 2004).

The asymmetric unit comprises one 4,4'-bipyridyl and two 2-chloro-4-nitorobenzoic acid molecules (Fig. 1), which are held together by short O—H···N hydrogen bonds (Table 1) to form a 1:2 unit. The pyridine rings of the bipyridyl molecule are twisted by 27.90 (5)° to each other. The pyridine N3/C15—C18 ring makes a dihedral angle of 16.82 (5)° with the benzene C1—C6 ring of the adjacent benzoic acid, while the N4/C20—C24 ring forms an angle of 64.52 (5)° with the C8—C13 ring. The benzene C1—C6 ring makes dihedral angles of 31.17 (3) and 9.85 (3)° with the carboxyl C7/O1/O2 plane and the nitro N1/O3/O4 plane, respectively, and the benzene C8—C13 ring forms angles of 39.25 (3) and 15.99 (3)° with the C14/O5/O6 and N2/O7/O8 planes, respectively.

The two 1:2 units related by an inversion center are connected by C—H···O hydrgoen bonds (Table 1) to form a supramolecular ring (Fig. 2). The rings are further connected by C—H···O hydrogen bonds, leading to a three-dimensional hydrogen-bonded network.

Experimental

Single crystals of the title compound were obtained by slow evaporation from an acetonitrile solution of 4,4'-bipyridyl (20 mg) and 2-chloro-4-nitrobenzoic acid (51 mg).

Refinement

H atoms were located in a difference Fourier map. Then O-bound H atoms were refined freely (refined distances are given in Table 1). The C-bound H atoms were included in the riding model approximation with C—H = 0.95 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

Fig. 1. The molecular structure of the title compound, with the atom labeling. Displacement ellipsoids for non-H atoms are drawn at 50% probability level. The dashed lines indicate hydrogen bonds.

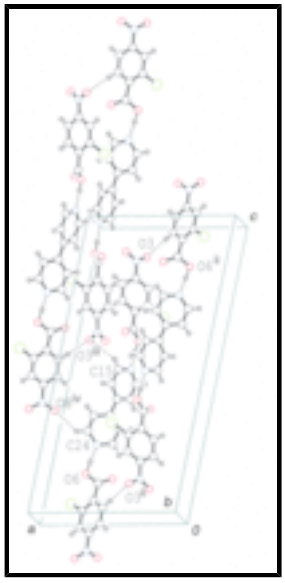

Fig. 2. A partial packing diagram of the title compound, viewed approximately down the *b* axis. The dashed lines indicate hydrogen bonds. symmetry operation are given in Table 1.

4,4'-Bipyridyl–2-chloro-4-nitrobenzoic acid (1/2)

Crystal data

$C_{10}H_8N_2{\cdot}2C_7H_4ClNO_4$	F_{000} = 1144.00
M_r = 559.32	D_x = 1.577 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: -P 2yn	Cell parameters from 3516 reflections
a = 12.0875 (11) Å	θ = 6.1–29.9º
b = 8.0695 (9) Å	μ = 0.34 mm^{-1}
c = 24.610 (2) Å	T = 173 (1) K
β = 101.143 (2)º	Block, colorless
V = 2355.2 (4) Å^3	0.48 × 0.45 × 0.38 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID-II diffractometer	4838 reflections with $I > 2\sigma(I)$
Detector resolution: 10.00 pixels mm^{-1}	R_{int} = 0.023
T = 173(1) K	θ_{max} = 27.5º
ω scans	θ_{min} = 3.0º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −15→15
T_{min} = 0.780, T_{max} = 0.880	k = −10→10
19103 measured reflections	l = −30→31
5379 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.091$	$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.6629P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.002$
5379 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
351 parameters	$\Delta\rho_{min} = -0.44$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.43587 (3)	0.07017 (5)	0.694192 (13)	0.03123 (10)
Cl2	0.77647 (3)	0.41014 (4)	0.053835 (13)	0.03098 (9)
O1	0.64197 (9)	0.44155 (12)	0.63346 (4)	0.0305 (2)
O2	0.56764 (9)	0.19023 (13)	0.61217 (4)	0.0325 (2)
O3	0.63818 (8)	0.07543 (12)	0.89943 (4)	0.0294 (2)
O4	0.76390 (8)	0.27037 (13)	0.91028 (4)	0.0323 (2)
O5	0.51496 (8)	0.60528 (13)	0.12896 (4)	0.0293 (2)
O6	0.70335 (8)	0.59355 (14)	0.14746 (4)	0.0316 (2)
O7	0.65421 (9)	0.68033 (15)	−0.14127 (4)	0.0372 (2)
O8	0.49099 (9)	0.79252 (13)	−0.14216 (4)	0.0357 (2)
N1	0.69168 (9)	0.18509 (13)	0.88134 (4)	0.0239 (2)
N2	0.57867 (9)	0.72654 (14)	−0.11814 (4)	0.0267 (2)
N3	0.57221 (9)	0.28775 (14)	0.51053 (4)	0.0264 (2)
N4	0.67380 (9)	0.53108 (15)	0.24725 (4)	0.0275 (2)
C1	0.62647 (10)	0.26355 (15)	0.70768 (5)	0.0219 (2)
C2	0.55600 (10)	0.16182 (15)	0.73223 (5)	0.0205 (2)
C3	0.57687 (10)	0.13632 (15)	0.78929 (5)	0.0214 (2)

H3	0.5292	0.0669	0.8059	0.026*
C4	0.66862 (10)	0.21453 (15)	0.82118 (5)	0.0214 (2)
C5	0.73956 (11)	0.31852 (17)	0.79904 (5)	0.0279 (3)
H5	0.8012	0.3726	0.8220	0.033*
C6	0.71721 (11)	0.34070 (18)	0.74214 (5)	0.0286 (3)
H6	0.7652	0.4107	0.7260	0.034*
C7	0.61162 (10)	0.30665 (16)	0.64683 (5)	0.0240 (2)
C8	0.60901 (10)	0.63901 (15)	0.05405 (5)	0.0212 (2)
C9	0.68021 (10)	0.55902 (15)	0.02378 (5)	0.0210 (2)
C10	0.67249 (10)	0.58905 (15)	−0.03255 (5)	0.0226 (2)
H10	0.7211	0.5347	−0.0529	0.027*
C11	0.59194 (10)	0.70030 (15)	−0.05791 (5)	0.0223 (2)
C12	0.51996 (11)	0.78311 (17)	−0.02985 (5)	0.0269 (3)
H12	0.4657	0.8595	−0.0484	0.032*
C13	0.52944 (11)	0.75120 (17)	0.02624 (5)	0.0265 (3)
H13	0.4808	0.8068	0.0463	0.032*
C14	0.60568 (10)	0.60941 (15)	0.11444 (5)	0.0224 (2)
C15	0.64224 (12)	0.41360 (17)	0.50570 (5)	0.0283 (3)
H15	0.6778	0.4700	0.5383	0.034*
C16	0.66500 (11)	0.46500 (17)	0.45522 (5)	0.0264 (3)
H16	0.7151	0.5548	0.4537	0.032*
C17	0.61383 (10)	0.38400 (16)	0.40671 (5)	0.0222 (2)
C18	0.54069 (11)	0.25415 (17)	0.41208 (5)	0.0270 (3)
H18	0.5037	0.1956	0.3802	0.032*
C19	0.52215 (11)	0.21083 (18)	0.46407 (5)	0.0280 (3)
H19	0.4716	0.1225	0.4669	0.034*
C20	0.57619 (11)	0.46059 (17)	0.25273 (5)	0.0270 (3)
H20	0.5199	0.4440	0.2205	0.032*
C21	0.55386 (11)	0.41087 (16)	0.30343 (5)	0.0254 (3)
H21	0.4837	0.3607	0.3055	0.031*
C22	0.63533 (11)	0.43498 (15)	0.35154 (5)	0.0225 (2)
C23	0.73605 (11)	0.50991 (19)	0.34539 (5)	0.0295 (3)
H23	0.7935	0.5299	0.3769	0.035*
C24	0.75228 (12)	0.55521 (19)	0.29311 (6)	0.0318 (3)
H24	0.8217	0.6053	0.2897	0.038*
H2O	0.568 (2)	0.230 (3)	0.5766 (11)	0.081 (8)*
H6O	0.692 (2)	0.573 (4)	0.1880 (13)	0.103 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02687 (16)	0.0445 (2)	0.02162 (15)	−0.01317 (13)	0.00296 (12)	−0.00208 (13)
Cl2	0.03437 (18)	0.03004 (17)	0.02701 (17)	0.01092 (12)	0.00216 (13)	0.00226 (12)
O1	0.0397 (5)	0.0305 (5)	0.0223 (4)	−0.0026 (4)	0.0084 (4)	0.0046 (4)
O2	0.0406 (5)	0.0384 (5)	0.0186 (4)	−0.0114 (4)	0.0056 (4)	0.0007 (4)
O3	0.0352 (5)	0.0320 (5)	0.0223 (4)	0.0032 (4)	0.0092 (4)	0.0053 (4)
O4	0.0369 (5)	0.0349 (5)	0.0218 (4)	0.0021 (4)	−0.0021 (4)	−0.0056 (4)
O5	0.0260 (5)	0.0390 (5)	0.0243 (4)	0.0020 (4)	0.0081 (4)	0.0045 (4)

O6	0.0253 (5)	0.0506 (6)	0.0184 (4)	−0.0021 (4)	0.0033 (4)	0.0038 (4)
O7	0.0367 (5)	0.0524 (6)	0.0250 (5)	−0.0062 (5)	0.0124 (4)	0.0023 (5)
O8	0.0395 (6)	0.0390 (6)	0.0246 (5)	0.0009 (4)	−0.0036 (4)	0.0064 (4)
N1	0.0272 (5)	0.0262 (5)	0.0178 (5)	0.0066 (4)	0.0033 (4)	−0.0009 (4)
N2	0.0309 (6)	0.0288 (5)	0.0198 (5)	−0.0073 (4)	0.0034 (4)	0.0021 (4)
N3	0.0274 (5)	0.0335 (6)	0.0180 (5)	0.0016 (4)	0.0034 (4)	0.0029 (4)
N4	0.0290 (5)	0.0355 (6)	0.0185 (5)	0.0016 (5)	0.0060 (4)	0.0015 (4)
C1	0.0217 (5)	0.0252 (6)	0.0190 (5)	0.0006 (4)	0.0046 (4)	0.0021 (5)
C2	0.0194 (5)	0.0227 (6)	0.0192 (5)	−0.0002 (4)	0.0033 (4)	−0.0012 (4)
C3	0.0232 (5)	0.0233 (6)	0.0189 (5)	0.0001 (4)	0.0068 (4)	0.0005 (5)
C4	0.0244 (6)	0.0237 (6)	0.0163 (5)	0.0040 (4)	0.0041 (4)	0.0001 (4)
C5	0.0259 (6)	0.0320 (7)	0.0238 (6)	−0.0057 (5)	−0.0001 (5)	0.0009 (5)
C6	0.0271 (6)	0.0338 (7)	0.0248 (6)	−0.0081 (5)	0.0045 (5)	0.0049 (5)
C7	0.0216 (6)	0.0313 (6)	0.0195 (6)	0.0008 (5)	0.0051 (4)	0.0024 (5)
C8	0.0225 (5)	0.0221 (5)	0.0193 (5)	−0.0028 (4)	0.0042 (4)	0.0014 (5)
C9	0.0216 (5)	0.0193 (5)	0.0212 (6)	−0.0011 (4)	0.0018 (4)	0.0011 (4)
C10	0.0239 (6)	0.0232 (6)	0.0211 (6)	−0.0021 (4)	0.0053 (4)	−0.0025 (5)
C11	0.0244 (6)	0.0242 (6)	0.0177 (5)	−0.0050 (5)	0.0026 (4)	0.0019 (4)
C12	0.0264 (6)	0.0287 (6)	0.0255 (6)	0.0037 (5)	0.0046 (5)	0.0061 (5)
C13	0.0270 (6)	0.0295 (6)	0.0243 (6)	0.0050 (5)	0.0086 (5)	0.0027 (5)
C14	0.0266 (6)	0.0207 (5)	0.0202 (6)	−0.0011 (4)	0.0056 (5)	0.0001 (4)
C15	0.0329 (7)	0.0326 (7)	0.0185 (6)	0.0003 (5)	0.0027 (5)	−0.0017 (5)
C16	0.0307 (6)	0.0264 (6)	0.0216 (6)	−0.0026 (5)	0.0040 (5)	−0.0007 (5)
C17	0.0232 (6)	0.0257 (6)	0.0173 (5)	0.0049 (5)	0.0028 (4)	0.0015 (5)
C18	0.0300 (6)	0.0307 (6)	0.0187 (5)	−0.0020 (5)	0.0006 (5)	0.0007 (5)
C19	0.0281 (6)	0.0329 (7)	0.0222 (6)	−0.0028 (5)	0.0028 (5)	0.0040 (5)
C20	0.0290 (6)	0.0330 (7)	0.0180 (6)	0.0011 (5)	0.0019 (5)	−0.0005 (5)
C21	0.0256 (6)	0.0301 (6)	0.0202 (6)	−0.0003 (5)	0.0033 (5)	0.0010 (5)
C22	0.0261 (6)	0.0238 (6)	0.0174 (6)	0.0037 (5)	0.0038 (5)	0.0003 (4)
C23	0.0265 (6)	0.0409 (8)	0.0196 (6)	−0.0025 (5)	0.0009 (5)	0.0008 (5)
C24	0.0274 (6)	0.0445 (8)	0.0236 (6)	−0.0040 (6)	0.0053 (5)	0.0016 (6)

Geometric parameters (Å, °)

Cl1—C2	1.7338 (12)	C8—C13	1.3994 (17)
Cl2—C9	1.7354 (12)	C8—C9	1.4001 (17)
O1—C7	1.2142 (16)	C8—C14	1.5136 (16)
O2—C7	1.3105 (16)	C9—C10	1.3926 (17)
O2—H2O	0.93 (3)	C10—C11	1.3814 (17)
O3—N1	1.2292 (15)	C10—H10	0.9500
O4—N1	1.2257 (15)	C11—C12	1.3830 (18)
O5—C14	1.2176 (15)	C12—C13	1.3869 (17)
O6—C14	1.3037 (16)	C12—H12	0.9500
O6—H6O	1.05 (3)	C13—H13	0.9500
O7—N2	1.2239 (15)	C15—C16	1.3868 (18)
O8—N2	1.2304 (15)	C15—H15	0.9500
N1—C4	1.4720 (15)	C16—C17	1.3963 (17)
N2—C11	1.4749 (15)	C16—H16	0.9500
N3—C19	1.3382 (17)	C17—C18	1.3936 (18)

N3—C15	1.3420 (18)	C17—C22	1.4889 (17)
N4—C20	1.3399 (18)	C18—C19	1.3859 (18)
N4—C24	1.3408 (17)	C18—H18	0.9500
C1—C6	1.3965 (18)	C19—H19	0.9500
C1—C2	1.4012 (17)	C20—C21	1.3858 (18)
C1—C7	1.5140 (16)	C20—H20	0.9500
C2—C3	1.3934 (16)	C21—C22	1.3996 (17)
C3—C4	1.3812 (17)	C21—H21	0.9500
C3—H3	0.9500	C22—C23	1.3935 (18)
C4—C5	1.3845 (18)	C23—C24	1.3874 (19)
C5—C6	1.3854 (18)	C23—H23	0.9500
C5—H5	0.9500	C24—H24	0.9500
C6—H6	0.9500		
C7—O2—H2O	106.9 (16)	C10—C11—N2	118.53 (11)
C14—O6—H6O	109.7 (16)	C12—C11—N2	118.40 (11)
O4—N1—O3	124.00 (10)	C11—C12—C13	117.92 (12)
O4—N1—C4	117.97 (11)	C11—C12—H12	121.0
O3—N1—C4	118.02 (10)	C13—C12—H12	121.0
O7—N2—O8	123.99 (11)	C12—C13—C8	121.58 (12)
O7—N2—C11	118.53 (11)	C12—C13—H13	119.2
O8—N2—C11	117.47 (11)	C8—C13—H13	119.2
C19—N3—C15	117.66 (11)	O5—C14—O6	124.96 (11)
C20—N4—C24	118.06 (11)	O5—C14—C8	119.25 (11)
C6—C1—C2	118.10 (11)	O6—C14—C8	115.77 (10)
C6—C1—C7	115.31 (11)	N3—C15—C16	122.91 (12)
C2—C1—C7	126.53 (11)	N3—C15—H15	118.5
C3—C2—C1	120.95 (11)	C16—C15—H15	118.5
C3—C2—Cl1	116.79 (9)	C15—C16—C17	119.61 (12)
C1—C2—Cl1	122.19 (9)	C15—C16—H16	120.2
C4—C3—C2	118.31 (11)	C17—C16—H16	120.2
C4—C3—H3	120.8	C18—C17—C16	117.08 (11)
C2—C3—H3	120.8	C18—C17—C22	121.33 (11)
C3—C4—C5	122.94 (11)	C16—C17—C22	121.59 (12)
C3—C4—N1	118.22 (11)	C19—C18—C17	119.73 (12)
C5—C4—N1	118.84 (11)	C19—C18—H18	120.1
C4—C5—C6	117.48 (11)	C17—C18—H18	120.1
C4—C5—H5	121.3	N3—C19—C18	123.00 (13)
C6—C5—H5	121.3	N3—C19—H19	118.5
C5—C6—C1	122.21 (12)	C18—C19—H19	118.5
C5—C6—H6	118.9	N4—C20—C21	122.82 (12)
C1—C6—H6	118.9	N4—C20—H20	118.6
O1—C7—O2	124.90 (11)	C21—C20—H20	118.6
O1—C7—C1	119.31 (11)	C20—C21—C22	119.59 (12)
O2—C7—C1	115.77 (11)	C20—C21—H21	120.2
C13—C8—C9	118.23 (11)	C22—C21—H21	120.2
C13—C8—C14	115.87 (11)	C23—C22—C21	117.07 (11)
C9—C8—C14	125.83 (11)	C23—C22—C17	121.94 (11)
C10—C9—C8	121.31 (11)	C21—C22—C17	120.99 (12)
C10—C9—Cl2	117.08 (9)	C24—C23—C22	119.88 (12)

C8—C9—Cl2	121.52 (9)	C24—C23—H23	120.1
C11—C10—C9	117.94 (11)	C22—C23—H23	120.1
C11—C10—H10	121.0	N4—C24—C23	122.58 (13)
C9—C10—H10	121.0	N4—C24—H24	118.7
C10—C11—C12	123.02 (11)	C23—C24—H24	118.7
C6—C1—C2—C3	−0.97 (18)	O7—N2—C11—C12	166.12 (12)
C7—C1—C2—C3	−177.76 (12)	O8—N2—C11—C12	−14.64 (17)
C6—C1—C2—Cl1	175.86 (10)	C10—C11—C12—C13	−0.40 (19)
C7—C1—C2—Cl1	−0.92 (18)	N2—C11—C12—C13	176.83 (11)
C1—C2—C3—C4	0.29 (18)	C11—C12—C13—C8	0.0 (2)
Cl1—C2—C3—C4	−176.71 (9)	C9—C8—C13—C12	0.28 (19)
C2—C3—C4—C5	0.90 (19)	C14—C8—C13—C12	−176.81 (12)
C2—C3—C4—N1	−178.94 (10)	C13—C8—C14—O5	37.27 (17)
O4—N1—C4—C3	−170.86 (11)	C9—C8—C14—O5	−139.58 (13)
O3—N1—C4—C3	9.85 (16)	C13—C8—C14—O6	−141.32 (12)
O4—N1—C4—C5	9.29 (17)	C9—C8—C14—O6	41.84 (17)
O3—N1—C4—C5	−169.99 (11)	C19—N3—C15—C16	−0.6 (2)
C3—C4—C5—C6	−1.3 (2)	N3—C15—C16—C17	0.0 (2)
N1—C4—C5—C6	178.52 (12)	C15—C16—C17—C18	0.44 (19)
C4—C5—C6—C1	0.6 (2)	C15—C16—C17—C22	179.80 (12)
C2—C1—C6—C5	0.5 (2)	C16—C17—C18—C19	−0.22 (19)
C7—C1—C6—C5	177.68 (13)	C22—C17—C18—C19	−179.57 (12)
C6—C1—C7—O1	−29.31 (17)	C15—N3—C19—C18	0.8 (2)
C2—C1—C7—O1	147.56 (13)	C17—C18—C19—N3	−0.4 (2)
C6—C1—C7—O2	149.46 (12)	C24—N4—C20—C21	−0.6 (2)
C2—C1—C7—O2	−33.68 (18)	N4—C20—C21—C22	0.4 (2)
C13—C8—C9—C10	−0.20 (18)	C20—C21—C22—C23	0.27 (19)
C14—C8—C9—C10	176.58 (11)	C20—C21—C22—C17	179.61 (12)
C13—C8—C9—Cl2	−176.64 (10)	C18—C17—C22—C23	−152.85 (13)
C14—C8—C9—Cl2	0.14 (17)	C16—C17—C22—C23	27.82 (19)
C8—C9—C10—C11	−0.17 (18)	C18—C17—C22—C21	27.84 (18)
Cl2—C9—C10—C11	176.42 (9)	C16—C17—C22—C21	−151.49 (13)
C9—C10—C11—C12	0.48 (19)	C21—C22—C23—C24	−0.7 (2)
C9—C10—C11—N2	−176.75 (10)	C17—C22—C23—C24	179.96 (13)
O7—N2—C11—C10	−16.52 (17)	C20—N4—C24—C23	0.1 (2)
O8—N2—C11—C10	162.71 (11)	C22—C23—C24—N4	0.5 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···N3	0.93 (3)	1.70 (3)	2.6330 (14)	176 (2)
O6—H6O···N4	1.05 (3)	1.55 (3)	2.5984 (14)	177 (3)
C10—H10···O4^{i}	0.95	2.41	3.2271 (16)	144
C13—H13···O3^{ii}	0.95	2.34	3.2926 (17)	175
C15—H15···O3^{iii}	0.95	2.59	3.4360 (17)	149
C24—H24···O8^{iv}	0.95	2.52	3.2552 (19)	134

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+3/2; (iv) *x*+1/2, −*y*+3/2, *z*+1/2.

Fig. 1

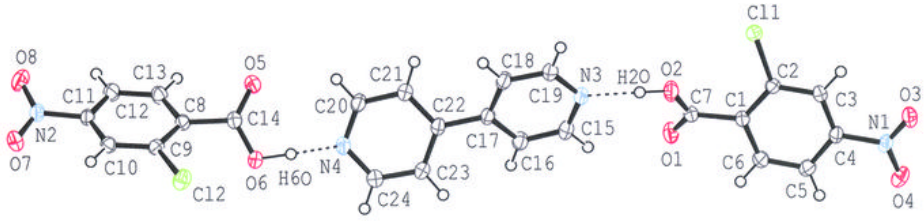
O8
C13
O5
C11
C12
C8
C14
C21
C18
N3
H2O
O2
C11
C20
C22
C19
C7
C1
C2
C3
O3
N2
C10
C9
C17
C15
N1
O7
C12
O6
H6O
N4
C16
O1
C6
C4
C24
C23
C5
O4

Fig. 2