organic compounds

Figure 1
View of (I) showing the atom-numbering scheme and ellipsoids at the 50% probability level.

4-Chloro-*N*-(4-cyano-2-nitrophenyl)-3-nitrobenzamide

Leroy Cronin,*† Dave A. Adams, David J. Nightingale and James H. Clark

Department of Chemistry, University of York, Heslington, York YO10 5DD, England
Correspondence e-mail: l.cronin@ed.ac.uk

The structure of the title compound, $C_{14}H_7ClN_4O_5$, comprises two nearly coplanar phenyl rings connected *via* an amido moiety.

Comment

In the course of an investigation of the effect of water on the fluorodenitration of substituted benzonitriles, it was observed that hydrolysis of the –CN moiety was possible. In an attempted fluorination of 4-chloro-3-nitrobenzonitrile, the title compound, (I), was observed as a previously uncharacterized product in such reactions (Adams *et al.*, 1999).
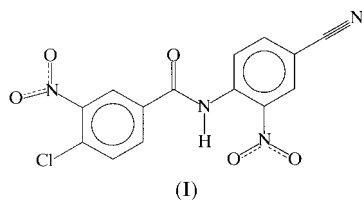
In (I) (Fig. 1), the amido unit is planar with an O3—C8—N3—H3 torsion angle of 171 (2)°. However, the C10—C9—C8—O3 and C4—C5—N3—C8 torsion angles of 22.3 (4) and −28.0 (4)°, respectively, indicate significant deviation from planarity with the amido group. In the chlorine-containing ring, the nitro group is twisted out of the plane of the phenyl ring [the O4—N4—C11—C10 torsion angle is 53.3 (4)°]. This is presumably due to the steric clash between the Cl atom and nitro group. In contrast, the nitro group *ortho* to the amido group on the other phenyl ring is more coplanar with the ring [the O2—N2—C6—C5 torsion angle is −14.5 (4)°]. This conformation may be stabilized, at least in part, by a hydrogen-bond interaction between the N—H of the amido group and an O atom of the nitro group [N3···O2 2.662 (5), N3—H3···O2 2.06 (3) Å and N3—H3···O2 125 (3)°]. The planes of the phenyl rings are inclined to each other at an angle of 3.2 (3)°. Examination of the packing of the molecules
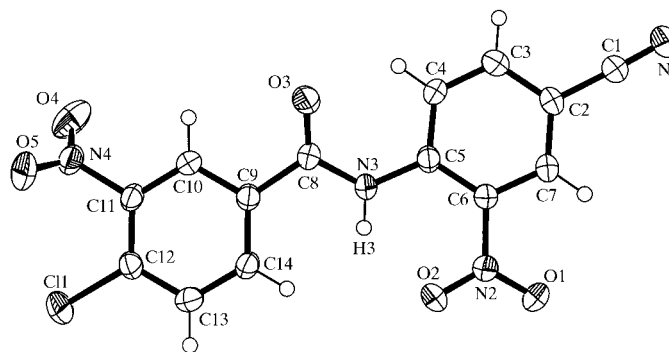
† Current address: Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 1NH, Scotland.

reveals a head-to-tail phenyl–phenyl interaction between adjacently stacked molecules of 3.77 (1) Å, indicating a weak intermolecular π-stacking interaction.

Experimental

4-Chloro-3-nitrobenzonitrile (0.182 g, 1 mmol) was placed in a round-bottomed flask. Dimethyl sulfoxide (10 ml, distilled and stored under argon) was added and the solution heated to 353 K under argon. Tetramethylammonium fluoride (0.20 g, 1.70 mmol; TMAF·$\frac{4}{3}$H₂O, prepared by drying the tetrahydrate under vacuum at 333 K for 2 d) was added to the solution. After 1 h, the reaction mixture was cooled in an ice bath. The organics were extracted into ether, washed well with water, dried (magnesium sulfate) and the ether removed on a rotary evaporator. The crude mixture was then recrystallized from acetonitrile to give yellow needles (Adams *et al.*, 1999).

Crystal data

$C_{14}H_7ClN_4O_5$	$Z = 2$
$M_r = 346.69$	$D_x = 1.656$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.804$ (13) Å	Cell parameters from 20
$b = 12.931$ (6) Å	reflections
$c = 7.49$ (4) Å	$\theta = 9{-}15°$
$\alpha = 91.59$ (16)°	$\mu = 0.312$ mm^{-1}
$\beta = 112.9$ (2)°	$T = 293$ (2) K
$\gamma = 88.12$ (8)°	Needle, yellow
$V = 695$ (4) Å^3	$0.55 \times 0.20 \times 0.20$ mm

Data collection

Rigaku four-circle AFC-6 diffract-ometer	1747 reflections with $I > 2\sigma(I)$
ω–2θ scans	$\theta_{max} = 25.03°$
Absorption correction: empirical *via* ψ scans (North *et al.*, 1968)	$h = 0 \to 9$
	$k = -15 \to 15$
$T_{min} = 0.782$, $T_{max} = 0.940$	$l = -8 \to 8$
2453 measured reflections	3 standard reflections
2453 independent reflections	every 150 reflections
	intensity variation: 0.5%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2 + 0.1533P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	
$wR(F^2) = 0.145$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.053$	$(\Delta/\sigma)_{max} = 0.001$
2452 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
220 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

Cl1—C12	1.724 (3)	O5—N4	1.219 (6)
O1—N2	1.225 (4)	N1—C1	1.147 (4)
O2—N2	1.234 (3)	N2—C6	1.465 (4)
N3—C8	1.377 (5)	O3—C8	1.210 (4)
N3—C5	1.409 (4)	N4—C11	1.472 (4)
O4—N4	1.213 (5)	C1—C2	1.445 (4)
C8—N3—C5	125.4 (3)	O5—N4—C11	117.7 (3)
O1—N2—O2	122.5 (2)	N1—C1—C2	177.8 (3)
O1—N2—C6	118.3 (2)	O3—C8—N3	123.9 (3)
O2—N2—C6	119.2 (2)	O3—C8—C9	121.1 (3)
O4—N4—O5	124.9 (3)	N3—C8—C9	114.9 (3)
O4—N4—C11	117.3 (3)		

The H atom on N3 was located in a difference Fourier synthesis. It was allowed to refine positionally with $U_{iso} = 1.2U_{eq}(N3)$. The phenyl H atoms were placed geometrically and thereafter refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1993); cell refinement: *TEXSAN*; data reduction: *TEXSAN*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *SHELXL*93.

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