2-Amino-5-(4-chlorophenyl)-1-methylimidazole

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The asymmetric unit of the title compound, $C_{10}H_{10}ClN_3$, contains four independent molecules, two of which are related by a pseudo-inversion centre. Intermolecular $N-H\cdots N$ and $N-H\cdots Cl$ interactions are present in the crystal.

Comment

According to the Cambridge Structural Database (Allen & Kennard, 1993) and to the best of our knowledge, the X-ray structure of the title compound, (I), has not been determined previously. The present analysis showed that four independent molecules are present in the asymmetric unit (molecules A, B, C and D). The planar phenyl ring (C6—C11) is twisted about the C5—C6 bond with respect to the planar imidazole moiety (N1—C5) by 22.8 (1), 29.8 (1), 28.9 (1) and 29.3 (1)° for molecules A–D, respectively. The main structural features of these four molecules are essentially identical, as shown in Table 1.
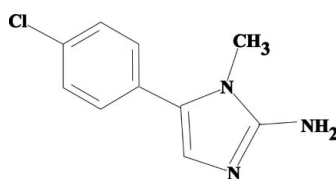

(I)

There are four endocyclic C—N bonds of the imidazole ring and one exocyclic C—N bond involving an amino group. As might be expected, the shortest C—N bond is the endocyclic C2=N3 double bond [mean 1.319 (3) Å], whereas the other C—N bond lengths follow the order C2—N1 [mean 1.359 (3)] < C2—N13 [mean 1.367 (3)] < C4—N3 [mean 1.377 (3)] < C5—N1 [mean 1.402 (3) Å]. A reason why the C2—N1 and C2—N13 single bonds are shorter than the other C—N bonds is the possibility of conjugation between the lone pairs of atoms N1 and N13 and the C2=N3 double bond.

Although amino–imino tautomerism is possible for α-aminoheterocycles, X-ray data confirm the amine form of the present structure.

A system of intermolecular $N-H\cdots N$ and $N-H\cdots Cl$ contacts is formed in the crystal (Table 2). The hydrogen bonds N13C—H10$C\cdots$N3C^{iii} (molecule C) and N13D—H10$D\cdots$N3D^{iv} (molecule D) [symmetry codes: (iii) $1-x, 1-y, 1-z$; (iv) $1-x, 2-y, 2-z$] link molecules C or D in the crystal into centrosymmetric dimers, whereas molecules A and B are linked by similar $N-H\cdots N$ bonds to form a pseudocentrosymmetric dimer (Fig. 1). Moreover, molecules C and D form endless chains, as shown in Fig. 2 and Table 2. Molecules

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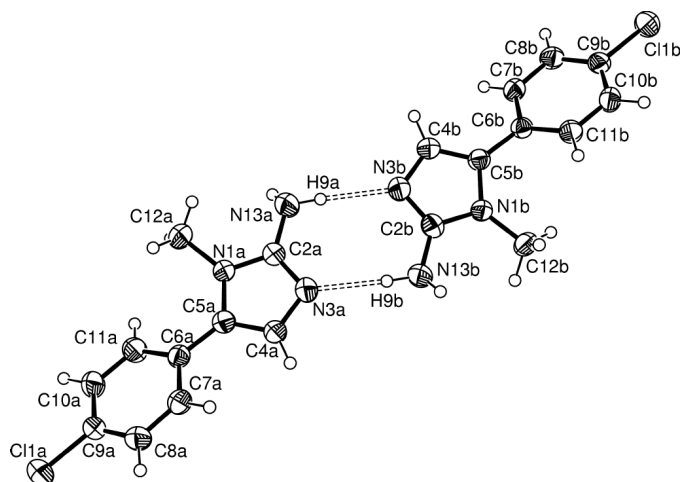
Figure 1
View of the pseudocentrosymmetric dimer of molecules *A* and *B* with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

A and *B* display similar packing, forming endless chains with long H···Cl contacts, as shown in Table 2.

Experimental

The title compound was prepared according to the procedure of Babaev & Belykh (2001). A single crystal of (I) was obtained while attempting to perform alkylation of the title compound with ethyl bromide. In a single experiment, 0.2 g of 2-amino-1-methyl-5-(4–chlorophenyl)imidazole was refluxed with excess EtBr in 10 ml MeCN for 3 h. After cooling the reaction mixture, the precipitated crystals were of the unchanged aminoimidazole.

Crystal data

$C_{10}H_{10}ClN_3$	$Z = 8$
$M_r = 207.66$	$D_x = 1.401$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.552$ (5) Å	Cell parameters from 25
$b = 12.833$ (5) Å	reflections
$c = 15.432$ (11) Å	$\theta = 16.0–17.0°$
$\alpha = 95.53$ (2)°	$\mu = 0.35$ mm^{-1}
$\beta = 106.57$ (2)°	$T = 293$ (2) K
$\gamma = 97.02$ (2)°	Prism, colourless
$V = 1969.0$ (18) Å^3	$0.3 \times 0.3 \times 0.3$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 27.0°$
ω scans	$h = -13 \rightarrow 12$
Absorption correction: none	$k = -16 \rightarrow 16$
8918 measured reflections	$l = 0 \rightarrow 19$
8591 independent reflections	2 standard reflections
5153 reflections with $I > 2\sigma(I)$	every 200 reflections
$R_{int} = 0.017$	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$+ 0.5308P]$
$wR(F^2) = 0.133$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.047$
8591 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
666 parameters	$\Delta\rho_{min} = -0.41$ e Å^{-3}
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

Cl1*A*—C9*A*	1.740 (3)	Cl1*C*—C9*C*	1.740 (2)
N1*A*—C2*A*	1.360 (3)	N1*C*—C2*C*	1.356 (3)
N1*A*—C5*A*	1.400 (3)	N1*C*—C5*C*	1.404 (3)
N1*A*—C12*A*	1.461 (3)	N1*C*—C12*C*	1.455 (3)
C2*A*—N3*A*	1.323 (3)	C2*C*—N3*C*	1.316 (3)
C2*A*—N13*A*	1.367 (3)	C2*C*—N13*C*	1.366 (3)
N3*A*—C4*A*	1.375 (3)	N3*C*—C4*C*	1.382 (3)
C4*A*—C5*A*	1.360 (3)	C4*C*—C5*C*	1.358 (3)
C5*A*—C6*A*	1.447 (3)	C5*C*—C6*C*	1.454 (3)
C6*A*—C11*A*	1.398 (3)	C6*C*—C11*C*	1.396 (3)
C6*A*—C7*A*	1.398 (3)	C6*C*—C7*C*	1.407 (3)
C7*A*—C8*A*	1.373 (4)	C7*C*—C8*C*	1.375 (4)
C8*A*—C9*A*	1.374 (4)	C8*C*—C9*C*	1.375 (4)
C9*A*—C10*A*	1.374 (4)	C9*C*—C10*C*	1.388 (3)
C10*A*—C11*A*	1.384 (4)	C10*C*—C11*C*	1.380 (3)
Cl1*B*—C9*B*	1.737 (3)	Cl1*D*—C9*D*	1.738 (2)
N1*B*—C2*B*	1.360 (3)	N1*D*—C2*D*	1.359 (3)
N1*B*—C5*B*	1.402 (3)	N1*D*—C5*D*	1.402 (3)
N1*B*—C12*B*	1.450 (3)	N1*D*—C12*D*	1.454 (3)
C2*B*—N3*B*	1.319 (3)	C2*D*—N3*D*	1.317 (3)
C2*B*—N13*B*	1.371 (3)	C2*D*—N13*D*	1.365 (3)
N3*B*—C4*B*	1.376 (3)	N3*D*—C4*D*	1.376 (3)
C4*B*—C5*B*	1.363 (3)	C4*D*—C5*D*	1.360 (3)
C5*B*—C6*B*	1.457 (3)	C5*D*—C6*D*	1.457 (3)
C6*B*—C11*B*	1.395 (3)	C6*D*—C11*D*	1.397 (3)
C6*B*—C7*B*	1.399 (3)	C6*D*—C7*D*	1.401 (3)
C7*B*—C8*B*	1.378 (4)	C7*D*—C8*D*	1.373 (3)
C8*B*—C9*B*	1.379 (4)	C8*D*—C9*D*	1.380 (4)
C9*B*—C10*B*	1.384 (4)	C9*D*—C10*D*	1.384 (3)
C10*B*—C11*B*	1.383 (4)	C10*D*—C11*D*	1.380 (4)
C2*A*—N1*A*—C5*A*	107.06 (19)	C2*C*—N1*C*—C5*C*	106.64 (19)
C2*A*—N1*A*—C12*A*	122.8 (2)	C2*C*—N1*C*—C12*C*	123.5 (2)
C5*A*—N1*A*—C12*A*	128.7 (2)	C5*C*—N1*C*—C12*C*	128.4 (2)
N3*A*—C2*A*—N1*A*	112.2 (2)	N3*C*—C2*C*—N1*C*	112.7 (2)
N3*A*—C2*A*—N13*A*	125.0 (2)	N3*C*—C2*C*—N13*C*	124.5 (2)
N1*A*—C2*A*—N13*A*	122.7 (2)	N1*C*—C2*C*—N13*C*	122.8 (2)
C2*A*—N3*A*—C4*A*	104.3 (2)	C2*C*—N3*C*—C4*C*	104.3 (2)
C5*A*—C4*A*—N3*A*	112.4 (2)	C5*C*—C4*C*—N3*C*	111.9 (2)
C4*A*—C5*A*—N1*A*	104.1 (2)	C4*C*—C5*C*—N1*C*	104.4 (2)
C4*A*—C5*A*—C6*A*	129.6 (2)	C4*C*—C5*C*—C6*C*	129.6 (2)
N1*A*—C5*A*—C6*A*	126.2 (2)	N1*C*—C5*C*—C6*C*	125.5 (2)
C11*A*—C6*A*—C7*A*	116.0 (2)	C11*C*—C6*C*—C7*C*	117.1 (2)
C11*A*—C6*A*—C5*A*	124.2 (2)	C11*C*—C6*C*—C5*C*	123.7 (2)
C7*A*—C6*A*—C5*A*	119.7 (2)	C7*C*—C6*C*—C5*C*	119.1 (2)
C8*A*—C7*A*—C6*A*	122.7 (2)	C8*C*—C7*C*—C6*C*	121.6 (2)
C7*A*—C8*A*—C9*A*	119.2 (2)	C7*C*—C8*C*—C9*C*	119.6 (2)
C10*A*—C9*A*—C8*A*	120.8 (2)	C8*C*—C9*C*—C10*C*	120.8 (2)
C10*A*—C9*A*—Cl1*A*	118.8 (2)	C8*C*—C9*C*—Cl1*C*	120.09 (19)
C8*A*—C9*A*—Cl1*A*	120.4 (2)	C10*C*—C9*C*—Cl1*C*	119.14 (19)
C9*A*—C10*A*—C11*A*	119.2 (3)	C11*C*—C10*C*—C9*C*	119.2 (2)
C10*A*—C11*A*—C6*A*	122.1 (2)	C10*C*—C11*C*—C6*C*	121.7 (2)
C2*B*—N1*B*—C5*B*	106.56 (19)	C2*D*—N1*D*—C5*D*	106.24 (19)
C2*B*—N1*B*—C12*B*	124.0 (2)	C2*D*—N1*D*—C12*D*	123.7 (2)
C5*B*—N1*B*—C12*B*	129.3 (2)	C5*D*—N1*D*—C12*D*	127.9 (2)
N3*B*—C2*B*—N1*B*	112.7 (2)	N3*D*—C2*D*—N1*D*	112.8 (2)
N3*B*—C2*B*—N13*B*	124.9 (2)	N3*D*—C2*D*—N13*D*	124.9 (2)
N1*B*—C2*B*—N13*B*	122.3 (2)	N1*D*—C2*D*—N13*D*	122.2 (2)
C2*B*—N3*B*—C4*B*	104.3 (2)	C2*D*—N3*D*—C4*D*	104.4 (2)
C5*B*—C4*B*—N3*B*	112.0 (2)	C5*D*—C4*D*—N3*D*	111.8 (2)
C4*B*—C5*B*—N1*B*	104.4 (2)	C4*D*—C5*D*—N1*D*	104.8 (2)
C4*B*—C5*B*—C6*B*	129.5 (2)	C4*D*—C5*D*—C6*D*	129.4 (2)
N1*B*—C5*B*—C6*B*	126.0 (2)	N1*D*—C5*D*—C6*D*	125.4 (2)
C11*B*—C6*B*—C7*B*	117.0 (2)	C11*D*—C6*D*—C7*D*	117.2 (2)
C11*B*—C6*B*—C5*B*	123.9 (2)	C11*D*—C6*D*—C5*D*	123.2 (2)
C7*B*—C6*B*—C5*B*	119.1 (2)	C7*D*—C6*D*—C5*D*	119.5 (2)
C8*B*—C7*B*—C6*B*	122.0 (2)	C8*D*—C7*D*—C6*D*	121.8 (2)
C7*B*—C8*B*—C9*B*	119.2 (2)	C7*D*—C8*D*—C9*D*	119.4 (2)
C8*B*—C9*B*—C10*B*	120.8 (2)	C8*D*—C9*D*—C10*D*	120.6 (2)
C8*B*—C9*B*—Cl1*B*	120.02 (19)	C8*D*—C9*D*—Cl1*D*	119.71 (19)
C10*B*—C9*B*—Cl1*B*	119.2 (2)	C10*D*—C9*D*—Cl1*D*	119.7 (2)
C11*B*—C10*B*—C9*B*	119.2 (2)	C11*D*—C10*D*—C9*D*	119.4 (2)
C10*B*—C11*B*—C6*B*	121.8 (2)	C10*D*—C11*D*—C6*D*	121.5 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N13C—H10C···N3C[i]	0.85 (3)	2.11 (3)	2.955 (3)	173 (3)
N13D—H10D···N3D[ii]	0.85 (3)	2.11 (3)	2.949 (3)	175 (3)
N13A—H9A···N3B[iii]	0.89 (3)	2.09 (3)	2.979 (4)	176 (2)
N13B—H9B···N3A[iv]	0.89 (3)	2.11 (3)	2.998 (3)	176 (3)
N13C—H9C···Cl1C[iii]	0.81 (4)	2.90 (4)	3.646 (3)	154 (3)
N13D—H9D···Cl1D[iii]	0.82 (3)	2.91 (4)	3.650 (3)	152 (3)
N13A—H10A···Cl1A[iii]	0.78 (3)	3.21 (3)	3.696 (3)	122 (3)
N13B—H10B···Cl1B[iv]	0.82 (3)	3.11 (3)	3.729 (3)	134 (3)

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

All H atoms were refined isotropically; the C—H distances were in the range 0.78 (3)–1.04 (3) Å.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *WinGX*98 (Farrugia, 1998) and *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1998) and *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97.
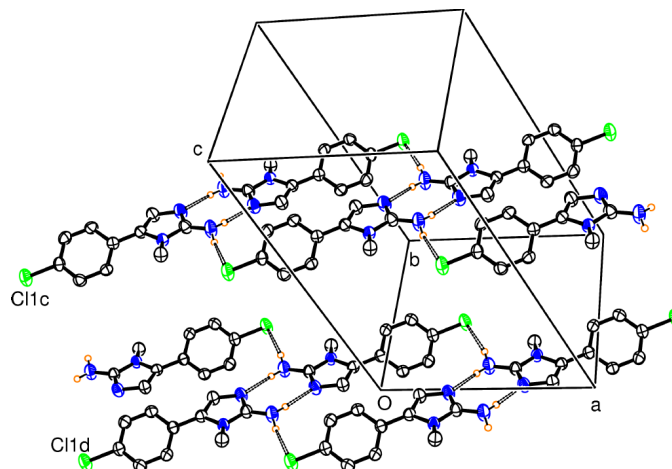
Figure 2
Part of the structure showing the formation of endless chains involving N—H···Cl hydrogen bonds. For the sake of clarity, H atoms not participating in the hydrogen bonding have been omitted, and no labels have been shown apart from those of the Cl atoms to differentiate the two chains formed by molecules *C* and *D*.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Babaev, E. V. & Belykh, E. N. (2001). Unpublished work.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1998). *ORTEP*-3 for Windows and *WinGX*98. University of Glasgow, Scotland.
Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.