

Iminopyrrole derivatives containing electron-withdrawing substituents: the formation of dimers and supra-molecular arrangements

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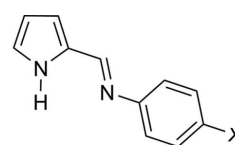
The crystal structures of two *p*-substituted phenylformimino-pyrrole derivatives, namely 2-[(4-fluorophenyl)iminomethyl]pyrrole, C₁₁H₉FN₂, (1), and 2-[(1*H*-pyrrol-2-ylmethylidene)amino]benzonitrile, C₁₂H₉N₃, (2), bear F and C≡N electron-withdrawing groups, respectively. Both structures feature two independent molecules in the asymmetric unit forming dimers *via* N—H···N hydrogen bonds. In the case of (1), each dimer interacts with two other dimers *via* C—H···F contacts, thus forming one-dimensional chains in the *b* direction, whereas in the case of (2), a weak C—H···N interaction connects the dimers in one-dimensional chains in the (110) direction.

Comment

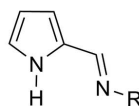
Pyrrole and its substituted derivatives are well known five-membered heterocyclic compounds which are involved in a wide range of applications, in areas such as natural products, bioactive molecules, pharmaceuticals, anion-binding systems, *etc.* (Rao & Jothilingam, 2001; Braun *et al.*, 2001; Hewton *et al.*, 2002; Sessler *et al.*, 2005). Moreover, 2-(*N*-aryl or *N*-alkyl)iminopyrroles, (I) (see Scheme), are usually employed as ligand precursors in the preparation of coordination compounds, generally after deprotonation of the pyrrole NH group (Mashima & Tsurugi, 2005). The synthetic strategy for the preparation of these iminopyrrole derivatives is very straightforward, consisting of a Vilsmeier–Haack acylation (Garrido *et al.*, 1984) followed by a condensation reaction with a suitable aliphatic or aromatic amine. The resulting complexes are mostly used as precatalysts in polymerization reactions (Mashima & Tsurugi, 2005; Matsugi & Fujita, 2008), but they can also have applications, for example, in luminescence (Wu *et al.*, 2003, 2004).

Recently, we reported the synthesis of new Ni (Bellabarba *et al.*, 2003), Co (Carabineiro *et al.*, 2007, 2008), Na (Gomes *et al.*, 2010) and Zn (Gomes *et al.*, 2009) complexes containing 2-(arylimino)pyrrolyl ligands, where the Ni derivatives were active in the oligomerization of ethylene and the Zn derivatives showed luminescent properties. In these publications, we also reported the molecular structure of some ligand precursors, namely 2-[1-(mesitylimino)ethyl]pyrrole, 2-[1-(2,6-diisopropylphenylimino)ethyl]pyrrole, two polymorphs of 2-[(phenylimino)methyl]pyrrole and 2-[(2,6-diisopropylphenylimino)methyl]pyrrole, which can be found in the Cambridge Structural Database (CSD, Version 5.32; Allen, 2002) with refcodes UMUKUI (Bellabarba *et al.*, 2003), LILYUB (Carabineiro *et al.*, 2007), CUKHUM (Gomes *et al.*, 2010), CUKHUM01 (Gomes *et al.*, 2010) and CUKJEY (Gomes *et al.*, 2010), respectively (see Scheme).

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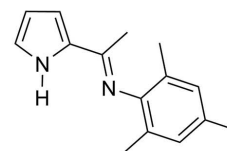


(1) X = F
(2) X = CN

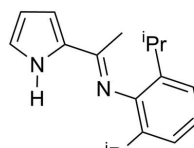


R = alkyl or aryl

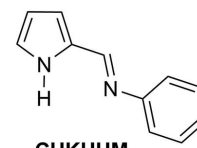
(I)



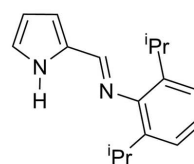
UMUKUI



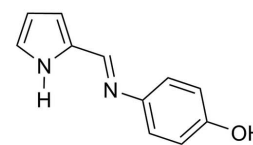
LILYUB



CUKHUM
CUKHUM01



CUKJEY



VIYWUW

In the present work, we report the crystal structures of 2-[(4-fluorophenyl)iminomethyl]pyrrole, (1), and 2-[(1*H*-pyrrol-2-ylmethylidene)amino]benzonitrile, (2), in which the *para* positions of the benzene rings are substituted by the electron-withdrawing F or C≡N (nitrile) groups. The corresponding molecular structures are depicted in Figs. 1 and 2, respectively. In both compounds, the asymmetric unit contains two independent molecules, where the pyrrole moieties show planar backbones with similar features. A brief analysis of the bond distances and angles in these derivatives (Table 1) reveals that

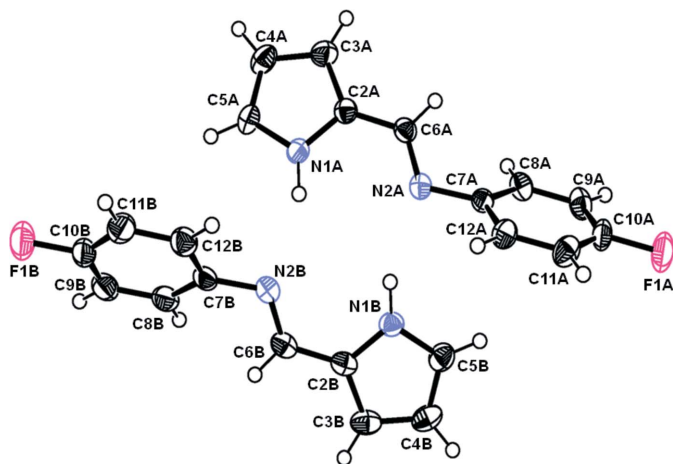


Figure 1
The two independent molecules in the asymmetric unit of (1). Displacement ellipsoids are drawn at the 50% probability level.

the longest bond in the pyrrole ring is C3–C4, with values ranging from 1.390 (3) to 1.397 (2) Å, and that the shortest bond is N1–C5. The imine distance varies between 1.2809 (19) and 1.290 (2) Å (Table 1). The torsion angles N2–C6–C2–N1 of -2.6 (3) and -2.4 (3) $^\circ$ [molecules *A* and *B* of (1)], and -4.1 (2) and -0.3 (3) $^\circ$ [molecules *A* and *B* of (2)] show that the pyrrole ring and the formimino group are nearly coplanar. The C2–C6 distances in the range 1.416 (2)–1.426 (2) Å are slightly shorter than the normal values for typical Csp^2 – Csp^2 single bonds (1.476 Å; Allen *et al.*, 1987), indicating an extension of the pyrrole ring π -electron delocalization towards the formimino substituent. For both (1) and (2), the benzene substituents of the iminic fragments adopt dihedral angles of around 45° relative to the pyrrole ring [49.08 (9), 45.33 (10), 52.05 (9) and 39.10 (10) $^\circ$ for molecules *A* and *B* of (1) and (2), respectively]. In fact, this is also true for CUKHUM [46.97 (7) and 45.31 (8) $^\circ$] and CUKHUM01 [41.96 (12), 8.93 (12), 47.96 (13) and 41.28 (12) $^\circ$] that do not have any substituents on the benzene ring, but is not the case for UMUKUI [85.66 (8) $^\circ$], LILYUB [86.54 (9) and 83.48 (9) $^\circ$] and CUKJEY [83.84 (10) and 86.17 (9) $^\circ$], in which the benzene rings are rotated about the $N_{\text{imine}}-C_{\text{ar}}$ bond, being nearly perpendicular to the formiminopyrrole plane defined by atoms N2–C6–C2–N1, owing to the high steric hindrance exerted by the alkyl groups in the 2- and 6-positions (Bellabarba *et al.*, 2003; Carabineiro *et al.*, 2007; Gomes *et al.*, 2010). The value of 8.93 (12) $^\circ$ found in one of the four molecules of polymorph CUKHUM01, which is substantially different from all the others, making the benzene ring almost coplanar with the iminopyrrole fragment, is due to the supramolecular arrangement.

It is known from the literature (Bellabarba *et al.*, 2003; Munro *et al.*, 2006; Carabineiro *et al.*, 2007; Gomes *et al.*, 2010) that these types of organic derivatives assemble as formiminopyrrole dimers through the formation of two complementary hydrogen bonds between a pyrrole NH group and the imine N atom belonging to the other molecule of the pair. In agreement with this, compounds (1) and (2) both show

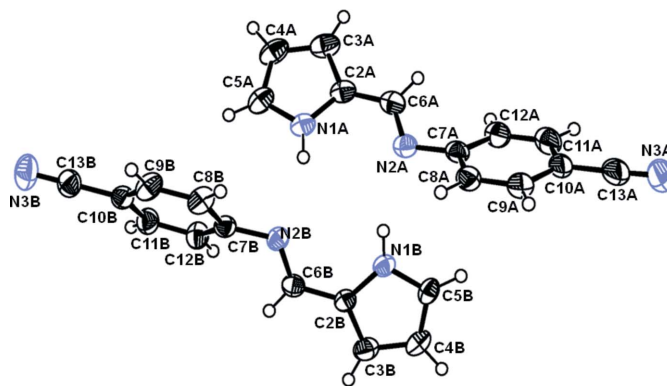


Figure 2
The two independent molecules in the asymmetric unit of (2). Displacement ellipsoids are drawn at the 50% probability level.

dimerization of the iminopyrrole molecules through an $R_2^2(10)$ motif, as can be seen in Figs. 3 and 4. However, in contrast to what is observed in the more hindered derivatives UMUKUI, LILYUB or CUKJEY, in which the iminopyrrole molecules of the dimer are coplanar, in (1) and (2), and also in CUKHUM, both molecules composing each dimer are not coplanar, the wings of the pyrrole moieties making angles of 150.55 (10), 138.77 (10) and 151.94 (8) $^\circ$ in the cases of (1), (2) and CUKHUM, respectively.

In compound (1), the most important observed intermolecular interactions are the two complementary hydrogen bonds N1A–H1A \cdots N2B and N1B–H1B \cdots N2A (Table 2). However, a C–H \cdots F short contact is found with H \cdots F distances and C–H \cdots F angles within the limits reported in the literature for this type of contact (Shimoni & Glusker, 1994; Howard *et al.*, 1996; Dunitz & Taylor, 1997). In fact, these interactions, always involving molecule *B*, occur between the F atom and the pyrrole NH group at position 3 of the neighbouring dimer, forming one-dimensional chains in the *b* direction (Fig. 3). Conversely, in derivative (2), in addition to the N1A–H1A \cdots N2B and N1B–H1B \cdots N2A

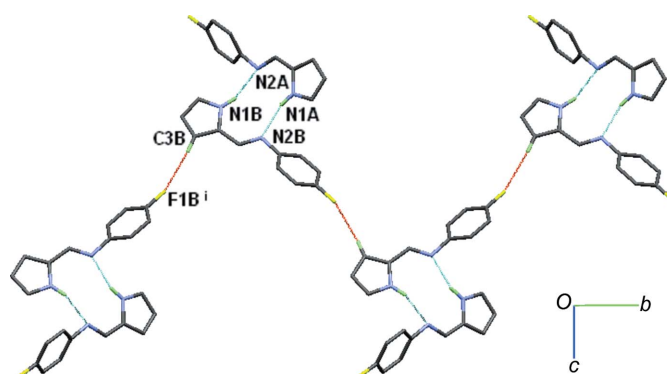


Figure 3
The packing of (1), showing the one-dimensional zigzag chains in the *b* direction and the $R_2^2(10)$ motifs forming the dimers. Donor and acceptor atoms are identified. Dashed lines represent N–H \cdots N and C–H \cdots F interactions (blue and red, respectively, in the electronic version of the paper). [Symmetry code: (i) $1 - x, -\frac{1}{2} + y, \frac{3}{2} - z$.]

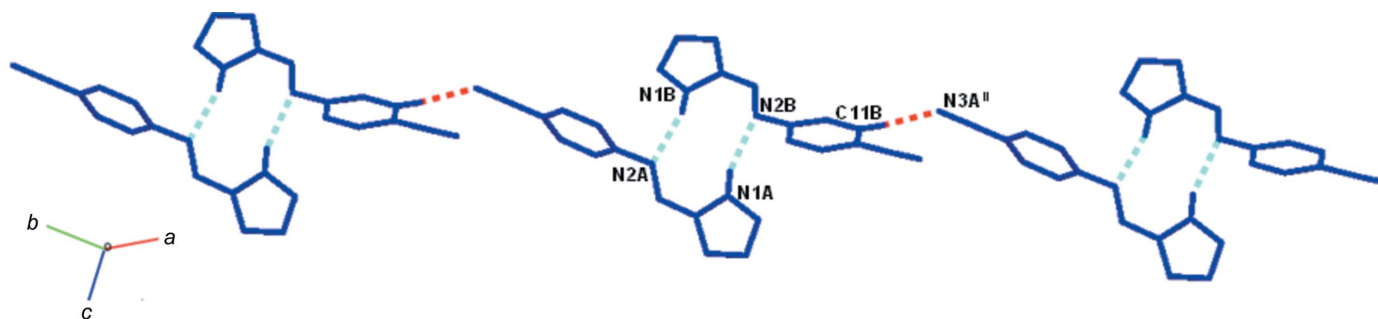


Figure 4

The packing of (2), showing a chain of dimers along the (110) direction and the $R_2^2(10)$ motifs forming the dimers. Donor and acceptor atoms are identified. Dashed lines represent N—H...N and C—H...N interactions (blue and red, respectively, in the electronic version of the paper). [Symmetry code: (ii) $x + 1, y - 1, z$]

hydrogen bonds, one can also notice the existence of a weak intermolecular C—H...N hydrogen bond, forming chains in the (110) direction, in which the nitrile group interacts weakly with an aromatic *meta*-H atom of the neighbouring dimer (Fig. 4), the latter having a more electropositive character than the corresponding H atom of derivative (1), owing to the proximity of the more powerful electron-withdrawing nitrile group. This is in agreement with the ^1H and ^{13}C NMR data for the corresponding *meta*-H and *meta*-C nuclei that are clearly more deshielded in compound (2) than in (1) [δ ^1H 7.67–7.63 versus 7.19–7.13 and δ ^{13}C 133.4 versus 116.8 p.p.m. for (2) and (1), respectively] (Figs. 3 and 4).

Moreover, comparing these two crystal structures with VIYWUW (Heinze *et al.*, 2008), an iminopyrrole derivative containing a hydroxy substituent in the *para* position of the benzene ring (see Scheme), it is possible to notice that the benzene substituent of the iminic fragment also lies around 45° relative to the pyrrole ring [dihedral angle = $49.59(5)^\circ$], and that all the distances within the molecule are in agreement with those observed for the derivatives discussed above. On the other hand, the formation of dimers through the establishment of complementary N—H...N hydrogen bonds is disabled in this compound, because of the presence of the *para*-hydroxy substituent, which is involved in three different interactions, *viz.* O—H...N, N—H...O and C—H...O. Similarly to derivative (2), this compound also forms an extended one-dimensional chain.

Experimental

n-Hexane, diethyl ether and absolute ethanol were predried over activated 4 Å molecular sieves and then distilled from sodium and kept under a nitrogen atmosphere. The synthetic procedure followed for the syntheses of (1) and (2) was that used previously by our group (Bellabarba *et al.*, 2003; Carabineiro *et al.*, 2007, 2008; Gomes *et al.*, 2010). 2-Formylpyrrole [10.5 mmol in the case of (1) and 14.4 mmol in the case of (2)], aniline (1 equivalent), a catalytic amount of *p*-toluenesulfonic acid and MgSO_4 (to remove the water formed in the reaction mixture) were suspended in absolute ethanol in a round-bottomed flask fitted with a condenser and a CaCl_2 guard tube. The mixture was refluxed overnight. After cooling to room temperature, CH_2Cl_2 was added and the suspension filtered through Celite and washed through with more CH_2Cl_2 . After removal of all volatiles, the

product was dissolved in refluxing *n*-hexane. In the case of (1), the resulting solution was stored at 253 K, yielding yellow prismatic crystals (yield 54%). However, in the case of (2), the product, which was an oil, was insoluble in *n*-hexane and partially soluble in diethyl ether. After evaporation of these solvents, the initial oil was transformed into a brown solid, which was purified by sublimation at a temperature of 353 K and a pressure of 10 Pa (yield 49%). Yellow crystals of (2) suitable for X-ray diffraction were obtained by recrystallization from diethyl ether at 253 K.

NMR data for (1): ^1H (300 MHz, CDCl_3): δ_{H} 9.98 (*br s*, 1H, NH), 8.25 (*s*, 1H, CH=N), 7.19–7.13 (*m*, 2H, *m*-phenyl), 7.10–7.02 (*m*, 2H, *o*-phenyl), 6.86 (*br s*, 1H, H5), 6.69 (*dd*, $J_{\text{HH}} = 1.38$ Hz, 1H, H3), 6.29 (*dd*, $J_{\text{HH}} = 2.49$ Hz, 1H, H4). $^{13}\text{C}\{^1\text{H}\}$ (75 MHz, CDCl_3): δ_{C} 160.9 (*d*, $^1J_{\text{CF}} = 242$ Hz, *p*-phenyl), 149.8 (CH=N), 147.8 (*ipso*-phenyl), 130.7 (C2), 123.3 (C3), 122.2 (*d*, $^3J_{\text{CF}} = 8$ Hz, *o*-phenyl), 116.8 (C4), 115.9 (*d*, $^2J_{\text{CF}} = 22$ Hz, *m*-phenyl), 110.5 (C5).

NMR data for (2): ^1H (300 MHz, CDCl_3): δ_{H} 9.53 (*br s*, 1H, NH), 8.21 (*s*, 1H, CH=N), 7.67–7.63 (*m*, 2H, *m*-phenyl), 7.22–7.18 (*m*, 2H, *o*-phenyl), 7.03 (*d*, $J_{\text{HH}} = 10.8$ Hz, 1H, H5), 6.76 (*dd*, $J_{\text{HH}} = 1.2$ Hz, 1H, H3), 6.34 (*m*, 1H, H4). $^{13}\text{C}\{^1\text{H}\}$ (75 MHz, CDCl_3): δ_{C} 155.7 (*ipso*-phenyl), 151.1 (CH=N), 133.4 (*m*-phenyl), 130.4 (C2), 124.2 (C3), 121.7 (*o*-phenyl), 119.2 (C4), 118.2 (C≡N), 111.1 (*p*-phenyl), 108.4 (C5).

Compound (1)

Crystal data

$\text{C}_{11}\text{H}_9\text{FN}_2$	$V = 3782.9(3) \text{ \AA}^3$
$M_r = 188.20$	$Z = 16$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 9.5542(5) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 18.7198(9) \text{ \AA}$	$T = 150 \text{ K}$
$c = 21.1509(12) \text{ \AA}$	$0.60 \times 0.60 \times 0.30 \text{ mm}$

Data collection

Bruker APEXII CCD diffractometer	21637 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3605 independent reflections
$T_{\text{min}} = 0.946, T_{\text{max}} = 0.972$	2479 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.100$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
$S = 1.03$	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
3605 reflections	
261 parameters	

Table 1

Selected bond distances and angles (Å, °) for compounds (1) and (2).

	(1A)	(1B)	(2A)	(2B)
N1—C2	1.371 (2)	1.370 (2)	1.364 (2)	1.365 (2)
C2—C3	1.381 (2)	1.382 (2)	1.382 (2)	1.388 (2)
C3—C4	1.395 (3)	1.393 (3)	1.397 (2)	1.390 (3)
C4—C5	1.363 (3)	1.365 (2)	1.367 (2)	1.366 (2)
N1—C5	1.357 (2)	1.356 (2)	1.356 (2)	1.362 (2)
C2—C6	1.423 (2)	1.426 (2)	1.422 (2)	1.416 (2)
N2—C6	1.284 (2)	1.281 (2)	1.2809 (19)	1.290 (2)
N2—C7	1.418 (2)	1.422 (2)	1.416 (2)	1.414 (2)
C5—N1—C2	108.94 (15)	108.98 (15)	109.27 (15)	109.20 (15)
N1—C2—C3	107.28 (15)	107.20 (16)	107.45 (14)	106.99 (16)
C2—C3—C4	107.63 (16)	107.80 (16)	107.48 (15)	107.91 (17)
C3—C4—C5	107.37 (15)	107.19 (16)	107.24 (15)	107.27 (16)
C4—C5—N1	108.77 (16)	108.82 (17)	108.55 (16)	108.63 (17)
N1—C2—C6	123.21 (15)	123.74 (15)	123.23 (15)	123.42 (15)
C2—C6—N2	123.62 (16)	124.17 (16)	123.42 (16)	124.13 (16)

Table 2

Hydrogen-bond geometry (Å, °) for compounds (1) and (2).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
(1) N1A—H1A...N2B	0.908 (19)	2.131 (19)	2.990 (2)	157.4 (17)
(1) N1B—H1B...N2A	0.925 (19)	2.108 (19)	2.9896 (19)	158.8 (17)
(1) C3B—H3B...F1B ⁱ	0.95	2.59	3.521 (2)	168
(2) N1A—H1A...N2B	0.873 (18)	2.147 (18)	2.978 (2)	159.0 (16)
(2) N1B—H1B...N2A	0.88 (2)	2.128 (19)	2.955 (2)	157.4 (18)
(2) C11B—H11B...N3A ⁱⁱ	0.95	2.42	3.357 (3)	170

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

Compound (2)

Crystal data

C₁₂H₉N₃ $V = 2072.6 (4) \text{ \AA}^3$
 $M_r = 195.22$ $Z = 8$
 Monoclinic, $P2_1/n$ $\text{Mo } K\alpha$ radiation
 $a = 12.5935 (13) \text{ \AA}$ $\mu = 0.08 \text{ mm}^{-1}$
 $b = 10.1843 (11) \text{ \AA}$ $T = 150 \text{ K}$
 $c = 16.901 (2) \text{ \AA}$ $0.30 \times 0.22 \times 0.20 \text{ mm}$
 $\beta = 107.035 (5)^\circ$

Data collection

Bruker APEXII CCD 18378 measured reflections
 diffractometer 3944 independent reflections
 Absorption correction: multi-scan 2443 reflections with $I > 2\sigma(I)$
 (SADABS; Sheldrick, 1996) $R_{\text{int}} = 0.050$
 $T_{\text{min}} = 0.977, T_{\text{max}} = 0.985$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$ H atoms treated by a mixture of independent and constrained refinement
 $wR(F^2) = 0.096$
 $S = 1.00$
 3944 reflections $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
 279 parameters $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

All H atoms, except for the pyrrole NH atoms, were placed in idealized positions and allowed to refine as riding on their parent C atoms, with C—H distances of 0.95 Å for aromatic H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms of the pyrrole NH groups were located in a difference Fourier map and allowed to refine freely, giving N—H distances of 0.874 (18)–0.92 (2) Å.

For both compounds, data collection: APEX2 (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SIR2004 (Burla et al., 2005);

program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae et al., 2006); software used to prepare material for publication: enCIFer (Allen et al., 2004), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LG3062). Services for accessing these data are described at the back of the journal.

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