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Synthesis of 5-(arylmethylideneamino)-4-(1*H*-benzo[*d*]imidazol-1-yl)pyrimidine hybrids: synthetic sequence and the molecular and supramolecular structures of two intermediates and three final products

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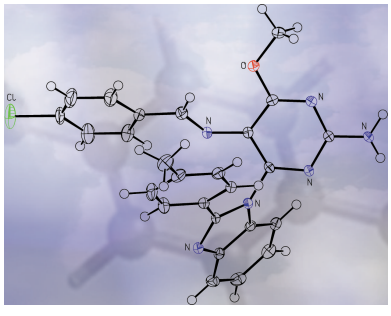
A concise and versatile synthesis of 5-(arylmethylideneamino)-4-(1*H*-benzo[*d*]-imidazol-1-yl)pyrimidines has been developed, starting from 4-(1*H*-benzo[*d*]-imidazol-1-yl)pyrimidines, and we report here the synthesis and spectroscopic and structural characterization of three such products, along with those of two intermediates in the reaction pathway. The intermediates 4-[2-(4-chlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl]-6-methoxypyrimidine-2,5-diamine, (II), and 4-[2-(4-bromophenyl)-1*H*-benzo[*d*]imidazol-1-yl]-6-methoxypyrimidine-2,5-diamine, (III), crystallize as the isostructural monohydrates $C_{18}H_{15}ClN_5O\cdot H_2O$ and $C_{18}H_{15}BrN_5O\cdot H_2O$, respectively, in which the components are linked into complex sheets by O—H···N and N—H···O hydrogen bonds. In the product (*E*)-4-methoxy-5-[(4-nitrobenzylidene)amino]-6-[2-(4-nitrophenyl)-1*H*-benzo[*d*]-imidazol-1-yl]pyrimidin-2-amine, which crystallizes as a 1:1 solvate with dimethyl sulfoxide, $C_{25}H_{18}N_8O_5\cdot C_2H_6OS$, (IV), inversion-related pairs of the pyrimidine component are linked by N—H···N hydrogen bonds to form cyclic centrosymmetric $R_2^2(8)$ dimers to which pairs of solvent molecules are linked by N—H···O hydrogen bonds. (*E*)-4-Methoxy-5-[(4-methylbenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine, $C_{27}H_{24}N_6O$, (V), crystallizes with Z' = 2 and the molecules are linked into a three-dimensional framework structure by a combination of N—H···N, C—H···N and C—H···π(arene) hydrogen bonds. The analogous product (*E*)-4-methoxy-5-[(4-chlorobenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine, $C_{26}H_{21}ClN_6O$, (VI), crystallizes from dimethyl sulfoxide in two forms: one, denoted (VI*a*), is isostructural with (V), and the other, denoted (VI*b*), crystallizes with Z' = 1, but as an unknown solvate in which the pyrimidine molecules are linked by N—H···N hydrogen bonds to form a ribbon containing two types of centrosymmetric ring.



1. Introduction

The benzimidazole unit has been shown to be an important heterocyclic fragment present in a large number of compounds with broad biological activity, including antimicrobial and antitumour activity (El-Gohary & Shaaban, 2017). In addition, aminopyrimidines are important building blocks for the synthesis of new heterocyclic systems (Abdul-Rida *et al.*, 2017), and they are also considered to constitute an important pharmacophoric fragment (Loving *et al.*, 2009), because of the wide biological activities that compounds containing this unit have shown, including anti-HIV activity (Al-Masoudi *et al.*,

2016) and cyclin-dependent kinase 2 (CDK2) inhibitory activity (Cortese *et al.*, 2016).

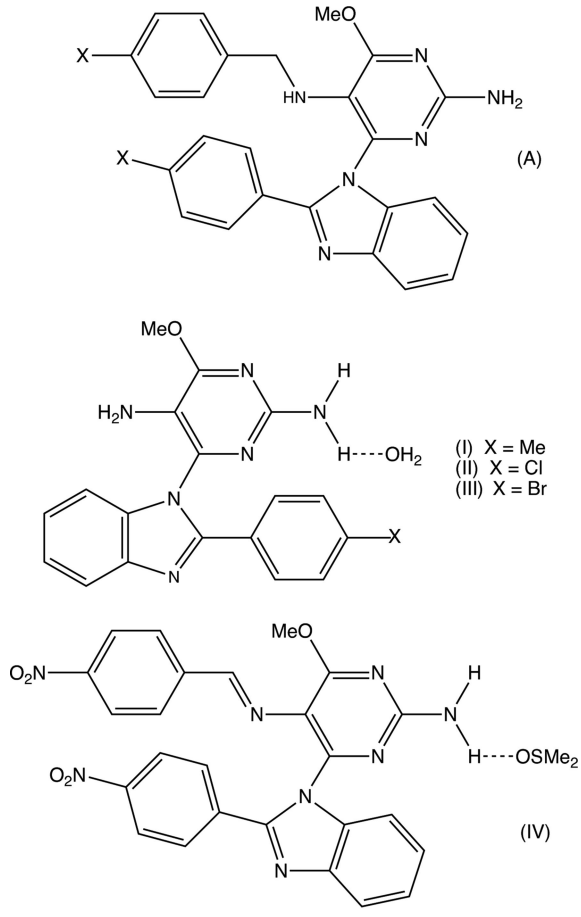
Molecules which include both benzimidazole and aminopyrimidine nuclei have been studied against some cancer cell lines, yielding interesting results that motivate the synthesis of this type of hybrid structures. This is the case for a series of novel fused pyrimido–benzimidazole systems reported recently, where one of the structures showed an IC_{50} value less than 2 μ*M* against the neuroblastoma SK-N-BE(2)-C and Kelly cell lines (Gadde *et al.*, 2023).

Non-fused pyrimidine–benzimidazole hybrids have also exhibited promising results for antitumour activity in human cancer cell lines (Sana *et al.*, 2021). A recent report has attributed the cytotoxicity of pyrimidine–benzimidazole hybrids to the presence of methoxy groups on the arene rings linked to the pyrimidine core, while the presence of electron-withdrawing groups seems to eliminate anticancer activity (Ismail *et al.*, 2022). However, any attempt to predict, prior to experimental evaluation, the effects of substituent variation in the products reported here would, perforce, be largely speculative and thus will not be pursued in this article.

We have recently reported the synthesis and the molecular and supramolecular structures of a set N^5-arylmethyl-6-methoxy-4-(2-aryl-1*H*-benzo[*d*]imidazol-1-yl)pyrimidine-2,5-diamines, where the two pendent aryl residues are identical, as they are both introduced in the reaction of N^4-(2-aminophenyl)-6-methoxypyrimidine-2,4,5-triamine with an aryl aldehyde in a 1:2 molar ratio (Vicentes *et al.*, 2019). Because of the biological importance of both the 2-aminopyrimidine residue (Koroleva *et al.*, 2010; Jadhav *et al.*, 2021) and the benzimidazole unit (Singh *et al.*, 2013; Wu *et al.*, 2022), whether alone or in combination, in the search for new biological targets (Sana *et al.*, 2021), we have now explored the combination of different aryl residues linked to the 5-amino group.

We report here an extension of the pyrimidine–benzimidazole hybrid systems reported previously (Vicentes *et al.*, 2019), in which the N^5-methylaryl-6-methoxy-4-(2-aryl-1*H*-benzo[*d*]imidazol-1-yl)pyrimidine-2,5-diamine precusors (A) (see Scheme 1) are subjected to debenzylation effected by ammonium hexanitratocerate(IV) (CAN) to produce the 6-methoxy-4-(2-aryl-1*H*-benzo[*d*]imidazol-1-yl)pyrimidine-2,5-diamines (I)–(III) (Scheme 1) for use as intermediates in the derivatization at the 5-amino group. When the corresponding reaction was attempted using the type (A) precursor having $X = Y = NO_2$, no debenzylation was observed, but instead the reaction produced a complex mixture from which only (*E*)-4-methoxy-5-[(4-nitrobenzylidene)amino]-6-[2-(4-nitrophenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine, (IV), could be isolated in pure form as a 1:1 solvate with dimethyl sulfoxide, in a yield of only 15%.

When compound (I) was condensed with 4-methylbenzaldehyde, the product (*E*)-4-methoxy-5-[(4-methylbenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine, (V), was formed in 74% yield and straightforwardly crystallized in the solvent-free form from a mixture of ethyl acetate and hexane. However, the corresponding reaction with 4-chlorobenzaldehyde gave, after crystallization, a mixture of two crystalline forms of (*E*)-4-chloro-5-[(4-methylbenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]-imidazol-1-yl]pyrimidin-2-amine, one denoted (VI*a*), which is isostructural with (V), together with a second form, denoted (VI*b*), which is a solvate of unknown constitution. The structure of compound (I) has already been reported (Vicentes *et al.*, 2019) and we report here the molecular and supramolecular structures of compounds (II)–(VI).



Scheme 1

2. Experimental

2.1. Synthesis and crystallization

The atom labelling is based throughout on the IUPAC chemical names, with the immediate substituents on the pyrimidine ring labelled according to their location; thus, N21, N41, N51 and O61, with appropriate modifications when $Z' = 2$, and with the rest of the substituent labels following the IUPAC name.

All of the signals in the ^{1}H and ^{13}C NMR spectra listed below were assigned using one-dimensional DEPT-135 ^{13}C spectra and two-dimensional COSY, HSQC and HMBC spectra.

The precursors of type (A) and the intermediate (I) (see Scheme 1) were prepared using previously described methods (Vicentes *et al.*, 2019). In the NMR listings given below, the

atom labelling for compounds (II)–(IV) and (VI) follows that used in Figs. 1–3 and 6, and the labelling for compound (V) follows that for (VI).

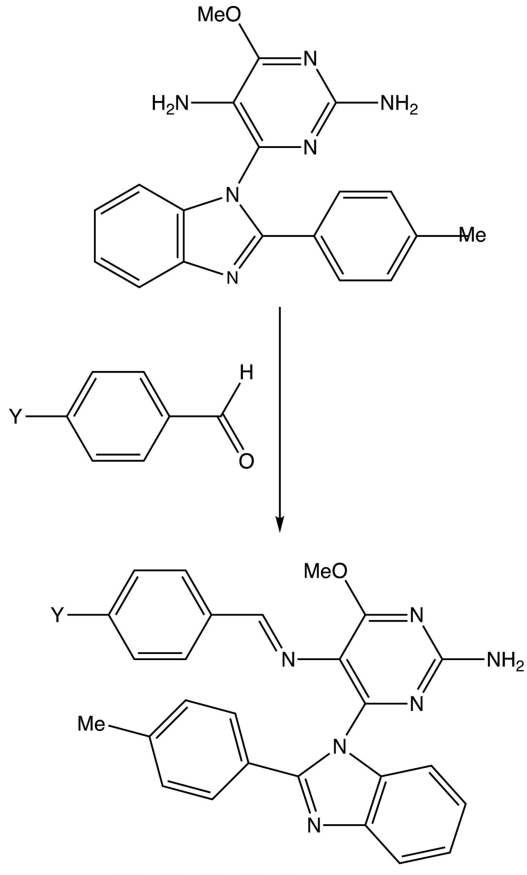
For the synthesis of compounds (II)–(IV), a solution of ammonium hexanitratocerate(IV) (0.69 g, 1.5 mmol) in a mixture of acetonitrile and water (3:1 *v*/*v*, 50 ml) was added to a solution of the appropriate precursor (A) [0.5 mmol; 0.22 g for (II) and 0.26 g for each of (III) and (IV)] in acetonitrile (10 ml); the resulting mixtures were then stirred for 2 h at 273 K. A saturated solution of sodium carbonate (15 ml) was then added and the acetonitrile was removed under reduced pressure. The residue was exhaustively extracted with ethyl acetate and the combined organic extracts were washed with water and then dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the crude solid products purified by column chromatography on silica gel (0.040–0.063 mm) using a mixture of ethyl acetate and hexane (3:2 *v*/*v*) as the eluent.

Compound (II): colourless solid, yield 54%, m.p. 510 K (decomposition). IR (ATR, cm^{-1}): 3494, 3399, 3303, 3188, 2922, 1606, 1562, 1467, 1450, 1403, 1261, 1241, 1092, 1011, 798, 739. NMR (DMSO-d_6): δ(^{1}H, 400 MHz) 7.79 (*ddd*, *J* = 8.0, 1.3, 0.7 Hz, 1H, H44), 7.62 (*d*, *J* = 8.8 Hz, 2H, H72, H76), 7.28 (*d*, *J* = 8.8 Hz, 2H, H73, H75), 7.25 (*dd*, *J* = 4.0, 1.5 Hz, 1H, H45), 7.21 (*dd*, *J* = 7.2, 1.3 Hz, 1H, H46), 7.15 (*ddd*, *J* = 7.8, 1.4, 0.7 Hz, 1H, H47), 4.56 (*s*, 2H,NH_2), 3.97 (*s*, 3H, OCH_3), 3.06 (*s*, 2H, NH_2); δ(^{13}C, 101 MHz) 162.33 (C6), 155.03 (C2), 150.94 (C42), 143.39 (C43*A*), 140.29 (C4), 136.25 (C74), 135.24 (C47*A*), 129.95 (C72, C76), 129.04 (C73, C75), 128.57 (C71), 124.12 (C46), 123.65 (C45), 120.30 (C44), 117.03 (C5), 110.86 (C47), 54.75 (OCH_3). HRMS (ESI–QTOF) *m*/*z* found 367.1069, $[M + H]^+$ requires for $C_{18}H_{15}ClN_6O$, 367.1069.

Compound (III): colourless solid, yield 48%, m.p. 508 K (decomposition). IR (ATR, cm^{-1}): 3494, 3398, 3302, 3185, 1338, 1609, 1562, 1466, 1450, 1401, 1241, 1053, 1008, 832, 741. NMR (DMSO-d_6): δ(^{1}H, 400 MHz) 7.75 (*d*, *J* = 7.3 Hz, 1H, H44), 7.63 (*m*, 4H, H72, H73, H75, H76), 7.32–7.21 (*m*, 1H, H45, H46), 7.12 (*d*, *J* = 7.4 Hz, 1H, H47), 5.99 (*s*, 2H, NH_2), 4.17 (*s*, 2H, NH_2), 3.96 (*s*, 3H, OCH_3); δ(^{13}C, 101 MHz) 161.49 (C6), 154.72 (C2), 150.65 (C42), 142.74 (C43*A*), 138.91 (C4), 135.55 (C74A), 131.54 (C73, C75), 130.16 (C72, C76), 129.46 (C71), 123.33 (C46), 123.25 (C45), 122.67 (C74), 119.34 (C44), 116.92 (C5), 111.32 (C47), 54.03 (OCH_3). HRMS (ESI–QTOF) *m*/*z* found 413.0541, $[M + H]^+$ requires for $C_{18}H_{15}BrN_6O$, 413.0545.

Compound (IV): yellow solid, yield 15%, m.p. 517 K (decomposition). IR (ATR, cm^{-1}): 3449, 3338, 3230, 1638, 1559, 1527, 1448, 1339, 853, 743. NMR (DMSO-d_6): δ(^{1}H, 400 MHz) 8.66 (*s*, 1H, H57), 8.18 (*d*, *J* = 8.8 Hz, 2H, H53, H55), 8.13 (*d*, *J* = 9.0 Hz, 2H, H73, H75), 7.88–7.84 (*m*, 1H, H44), 7.82 (*d*, *J* = 9.1 Hz, 2H, H72, H76), 7.59–7.51 (*m*, 3H, H47, H52, H56), 7.49 (*s*, 2H, NH_2), 7.41–7.27 (*m*, 2H, H45, H46), 4.01 (*s*, 3H, OCH_3); δ(^{13}C, 101 MHz) 164.07 (C6), 160.63 (C2), 156.79 (C57), 154.28 (C4), 150.82 (C74), 148.31 (C54), 147.57 (C42), 142.59 (C43*A*), 142.26 (C51), 136.55 (C71), 136.34 (C47*A*), 128.83 (C72, C73), 128.34 (C53, C56), 124.25 (46), 123.84 (C53, C55), 123.71 (C73, C75), 123.42 (C45), 119.81 (C44), 114.09 (C5), 112.24 (C47), 54.46 (OCH_3). HRMS (ESI–QTOF) *m*/*z* found 511.1471, $[M + H]^+$ requires for $C_{25}H_{18}N_8O_5$, 511.1471.

For the synthesis of compounds (V) and (VI) (Scheme 2), a mixture of compound (I) (0.17 g, 0.05 mmol) and the appropriate benzaldehyde (0.7 mmol) [84 mg of 4-methylbenzaldehyde for (V) or 92 mg of 4-chlorobenzaldehyde for (VI)] in acetic acid (3 ml) was stirred at ambient temperature for 1 h. The resulting precipitates were collected by filtration and washed first with an aqueous solution of sodium hydrogen carbonate (10% *w*/*v*) and then with water. The crude solid products were then purified by column chromatography on silica gel (0.040–0.063 mm) using a mixture of ethyl acetate and hexane (3:2 *v*/*v*) as eluent.



(V) Y = Me, Z' = 2
(VIa) Y = Cl, Z' = 2
(VIb) Y = Cl, Z' = 1, unknown solvate

Scheme 2

Compound (V): yellow solid, yield 74%, m.p. 491 K (decomposition). IR (ATR, cm^{-1}): 3303, 3157, 1652, 1606, 1576, 1522, 1449, 1345, 1248, 1078, 1041, 817, 738. NMR (DMSO-d_6): δ(^{1}H, 400 MHz) 8.33 (*s*, 1H, H57), 7.73 (*d*, *J* = 6.9 Hz, 1H, H44), 7.45 (*d*, *J* = 8.2 Hz, 2H, H72, H76), 7.37 (*d*, *J* = 6.7 Hz, 1H, H47), 7.29–7.19 (*m*, 4H, H45, H46, H52, H56), 7.16–7.07 (*m*, 6H, H73, H75, H53, H55, NH_2), 3.94 (*s*, 3H, OCH_3), 2.26 (*s*, 3H, C77), 2.25 (*s*, 3H, C58); δ(^{13}C, 101 MHz) 163.79 (C6), 160.29 (C2), 159.89 (C57), 152.97 (C42), 152.89 (C4), 142.67 (C43*A*), 140.92 (C34), 139.15 (C74), 136.35

Table 1
Experimental details.

For all structures: triclinic, $P\overline{1}$. Experiments were carried out at 100 K with Mo $K\alpha$ radiation using a Bruker D8 Venture diffractometer. Absorption was corrected for by multi-scan methods (*SADABS*; Bruker, 2016). H atoms were treated by a mixture of independent and constrained refinement.

	(II)	(III)	(IV)
Crystal data			
Chemical formula	$C_{18}H_{15}ClN_6O{\cdot}H_2O$	$C_{18}H_{15}BrN_6O{\cdot}H_2O$	$C_{25}H_{18}N_8O_5{\cdot}C_2H_6OS$
M_r	384.83	429.29	588.60
a, b, c (Å)	8.2156 (6), 11.0343 (7), 11.3968 (8)	8.1975 (7), 11.1963 (8), 11.3644 (10)	9.8192 (8), 10.2765 (7), 14.4096 (11)
α, β, γ (°)	107.980 (2), 109.725 (2), 98.541 (2)	107.938 (2), 109.866 (3), 98.683 (3)	71.718 (2), 74.872 (3), 88.786 (3)
V (Å^3)	887.43 (11)	894.39 (13)	1329.87 (18)
Z	2	2	2
μ (mm^{-1})	0.24	2.33	0.18
Crystal size (mm)	0.22 × 0.20 × 0.14	0.15 × 0.11 × 0.08	0.19 × 0.15 × 0.10
Data collection			
T_{min}, T_{max}	0.907, 0.967	0.719, 0.830	0.905, 0.982
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	35335, 4071, 3487	50505, 4457, 3696	66738, 6115, 4641
R_{int}	0.053	0.073	0.112
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.667	0.649
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.088, 1.06	0.030, 0.072, 1.09	0.050, 0.108, 1.05
No. of reflections	4071	4457	6115
No. of parameters	263	263	388
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.32	0.49, −0.71	0.32, −0.35

	(V)	(VI*a*)	(VI*b*)
Crystal data			
Chemical formula	$C_{27}H_{24}N_6O$	$C_{26}H_{21}ClN_6O$	$C_{26}H_{21}ClN_6O$
M_r	448.52	468.94	468.94
a, b, c (Å)	10.2203 (15), 14.821 (2), 16.594 (2)	10.2298 (7), 14.8344 (9), 16.5321 (10)	9.6520 (8), 9.7408 (10), 14.1445 (12)
α, β, γ (°)	99.616 (5), 92.153 (6), 106.083 (5)	99.672 (2), 92.038 (2), 106.704 (2)	98.183 (4), 104.638 (3), 90.059 (4)
V (Å^3)	2371.9 (6)	2359.4 (3)	1272.6 (2)
Z	4	4	2
μ (mm^{-1})	0.08	0.19	0.18
Crystal size (mm)	0.25 × 0.22 × 0.12	0.18 × 0.13 × 0.11	0.12 × 0.09 × 0.08
Data collection			
T_{min}, T_{max}	0.948, 0.990	0.926, 0.979	0.916, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	116861, 10869, 8351	126128, 10821, 8240	60695, 5849, 4653
R_{int}	0.074	0.079	0.074
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.650	0.651
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.127, 1.06	0.058, 0.164, 1.04	0.050, 0.115, 1.03
No. of reflections	10869	10821	5849
No. of parameters	631	629	315
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.50, −0.23	0.74, −0.61	0.34, −0.53

Computer programs: *APEX3* (Bruker, 2018), *SAINT* (Bruker, 2017), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

(C47*A*), 134.01 (C28), 129.06 (C73, C75), 128.88 (C53, C55), 127.87 (C71), 127.73 (C52, C56), 127.66 (C72, C76), 122.92 (C46), 122.55 (C45), 119.03 (C44), 116.05 (C5), 111.45 (C47), 54.18 (OCH_3), 21.02 (C77), 20.83 (C58). HRMS (ESI–QTOF) m/z found 449.2084, $[M + H]^+$ requires for $C_{27}H_{24}N_6O$, 449.2084.

Compound (VI): yellow solid, yield 95%, m.p. 493 K (decomposition). IR (ATR, cm^{-1}): 3308, 3138, 1652, 1573, 1520, 1450, 1362, 1080, 1042, 821, 735. NMR (DMSO-d_6): δ(^{1}H, 400 MHz) 8.43 (*s*, 1H, H57), 7.76 (*d*, J = 6.8 Hz, 1H, H44), 7.45 (*d*, J = 8.2 Hz, 2H, H72, H76), 7.44–7.36 (*m*, 3H, H47, H53, H55), 7.34 (*d*, J = 8.6 Hz, 2H, H52, H56), 7.31–7.19 (*m*, 4H, H45, H26, NH_2), 7.09 (*d*, J = 8.0 Hz, 2H, H73, H75), 3.97 (*s*, 3H, OCH_3), 2.25 (*s*, 3H, CCH_3); δ(^{13}C, 101 MHz) 163.85 (C6), 160.18 (C2), 158.37 (C57), 153.78 (C4), 153.01 (C42), 142.70 (C43*A*), 139.21 (C74), 136.34 (C47*A*), 135.50 (C54), 135.42 (C51), 129.21 (C52, C56), 128.92 (C73, C75), 128.62 (C53, C55), 127,86 (C71), 127.64 (C72, C76), 123.01 (C46), 122.66 (C45), 119.10 (C44), 115.24 (C5), 111.54 (C47), 54.28 (OCH_3), 20.85 (CH_3). HRMS (ESI–QTOF) m/z found 469.1538, $[M + H]^+$ requires for $C_{26}H_{21}ClN_6O$, 469.1538.

Crystals of compounds (II)–(V) suitable for single-crystal X-ray diffraction were grown by slow evaporation at ambient temperature and in the presence of air from a solution in

dimethyl sulfoxide for (II), (IV) and (V) or from a solution in methanol for (III), providing (II) and (III) as monohydrates, (IV) as a dimethyl sulfoxide (DMSO) solvate and (V) in the solvent-free form. A similar crystallization of (VI) from a solution in DMSO yielded two types of crystal, *i.e.* the more block-like solvent-free form (VI*a*) and the more plate-like solvate (VI*b*); no attempt was made to determine the relative quantities of the two crystalline forms.

2.2. Refinement

Crystal data, data collection and refinement details for compounds (II)–(VI) are summarized in Table 1. For (VI*b*), one reflection (010), which had been attenuated by the beam stop, and one bad outlier reflection ($\bar{1}03$) were omitted from the data set. All H atoms were located in difference maps. The H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (alkenic and aromatic) or 0.98 Å (CH_3), and with $U_{iso}(H) = kU_{eq}(C)$, where k = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. For the H atoms bonded to N or O atoms, the atomic coordinates were refined with $U_{iso}(H) = 1.2U_{eq}(N)$ or $1.5U_{eq}(O)$, giving the N—H and O—H distances shown in Table 3. For (VI*b*), conventional refinement converged only to R_1 = 0.146 and wR_2 = 0.3473. Examination of the structure of (VI*b*) at this point using *PLATON* (Spek, 2020) showed that the structure formed by the molecules of (VI) enclosed a void centred at $(0,0,\frac{1}{2})$, whose volume was *ca* 166 Å^3 in a unit cell of total volume 1272.6 (2) Å^3. The void thus occupies *ca* 13.0% of the total unit-cell volume, and there are a number of significant peaks in the difference map clustered within this void. The largest peak had a magnitude of 4.64 e Å^{-3} and further examination of this structure using the SQUEEZE procedure (Spek, 2015) indicated that the void contained around 43 electrons not hitherto accounted for. This number is consistent with the presence of one molecule of dimethyl sulfoxide, but no convincing solvent model could be developed from the difference peaks within the void and hence the reflection data were subjected to the SQUEEZE procedure (Spek, 2015), and the resultant modified reflection file was used for the refinement reported here. The CIF describing the structure obtained before the SQUEEZE procedure was applied has been included in the supporting information.

3. Results and discussion

Oxidation of the type (A) precursors having X = Cl or Br gave the products (II) and (III) (see Scheme 1) in exactly the same way as reported previously for the formation of (I) (Vicentes *et al.*, 2019). The formation of (I)–(III) presumably proceeds *via* the oxidation of the precursors to form the corresponding Schiff bases, which are hydrolysed to (I)–(III) during the subsequent work-up procedures. Accordingly, the formation of (IV), albeit in low yield, when $X = NO_2$, was unexpected, as it might be expected that this Schiff base would be more susceptible to hydrolysis than those having X = Me, Cl or Br. Condensation of (I) with two representative substituted benzaldehydes gave the required hybrid products (V) and (VI) in yields of 74 and 95%, respectively.

The new compounds (II)–(VI) reported here were all fully characterized by high-resolution mass spectrometry, by IR and ^{1}H and ^{13}C NMR spectroscopy, where the NMR spectra

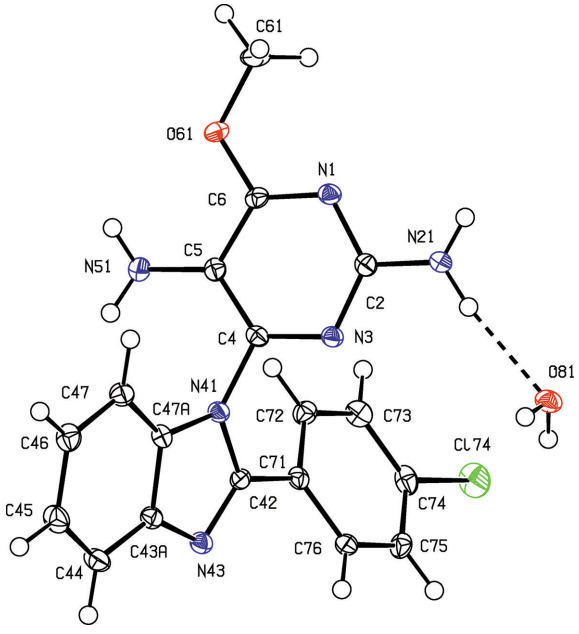


Figure 1
The two independent components in the structure of compound (II), showing the atom-labelling scheme and the hydrogen bond (drawn as a dashed line) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

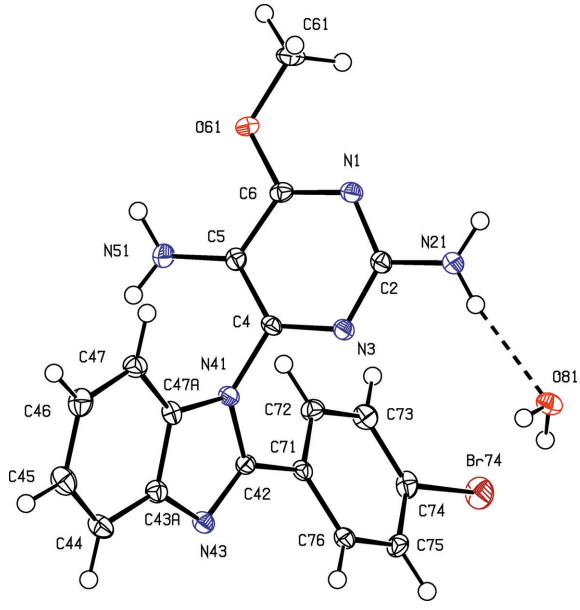


Figure 2
The two independent components in the structure of compound (III), showing the atom-labelling scheme and the hydrogen bond (drawn as a dashed line) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

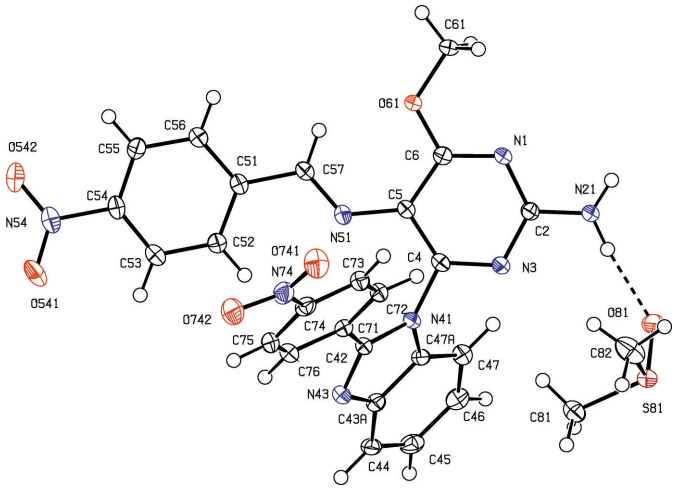


Figure 3
The two independent components in the structure of compound (IV), showing the atom-labelling scheme and the hydrogen bond (drawn as a dashed line) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

exhibited all of the expected signals, and by single-crystal X-ray diffraction. The crystallographic study confirmed fully the constitutions deduced from the spectra and, in addition, demonstrated the *E* configuration at the exocyclic C═N double bonds in (IV)–(VI), as well as providing information about the molecular conformations in the solid state and about the supramolecular assembly.

In the synthesis of the type (A) precursors (Vicentes *et al.*, 2019), the benzimidazole unit was constructed during the synthesis by condensation of an aldehyde with a pyrimidine-substituted benzene-1,2-diamine. The ability to incorporate a variety of substituents into both of these components, as well as into the aldehydes used in the formation of the products (V) and (VI), thus offers the possibility of forming a large library of variants containing multiple and varied substituents.

Table 2
Selected torsion angles (°) for intermediates (II) and (III).

Angle	(II)	(III)
N3—C4—N41—C42	−59.42 (19)	−59.7 (3)
N3—C4—N41—C47*A*	104.71 (15)	103.3 (2)
N41—C42—C71—C72	−34.0 (2)	−33.9 (3)
C5—C6—O61—C61	177.09 (17)	177.67 (17)

The intermediates (II) and (III) are isostructural (Table 1) with the methyl analogue (I) (Vicentes *et al.*, 2019) and they crystallize as monohydrates (Figs. 1 and 2). The product (IV) crystallizes as a stoichiometric solvate with dimethyl sulfoxide (Fig. 3), but the products (V) and (VI*a*), which are isostructural, crystallize in the solvent-free form with $Z' = 2$ (Figs. 4 and 5). The second crystalline form of compound (VI), denoted (VI*b*) (Fig. 6), also crystallizes as a solvate, but no coherent model for the disordered solvent could be developed from the peaks in the difference map; accordingly, the SQUEEZE procedure (Spek, 2015) was applied to the data set for this compound before the final refinements (see Section 2.2). For each of (V) and (VI*a*), a search for possible additional crystallographic symmetry revealed none; however, the two independent molecules in each of these compounds are related by an approximate, but noncrystallographic, twofold rotation axis (Figs. 4 and 5).

None of the pyrimidine components in compounds (II)–(VI) exhibits any internal symmetry, as indicated by the key torsion angles (Tables 2 and 3), and hence all are conformationally chiral (Moss, 1996; Flack & Bernardinelli, 1999), but the space groups (Table 1) confirm that, in every case, equal numbers of the two conformational enantiomers are present. For each of the products (IV)–(VI), the reference molecules were selected to have a positive sign for the torsion angles N*x*3—C*x*4—N*x*41—C*x*42, where x = 1 or 2 for (V) and (VI*a*), and x = nil for (IV) and (VI*b*) (Table 3). On this basis, each product has a negative sign for the torsion angle

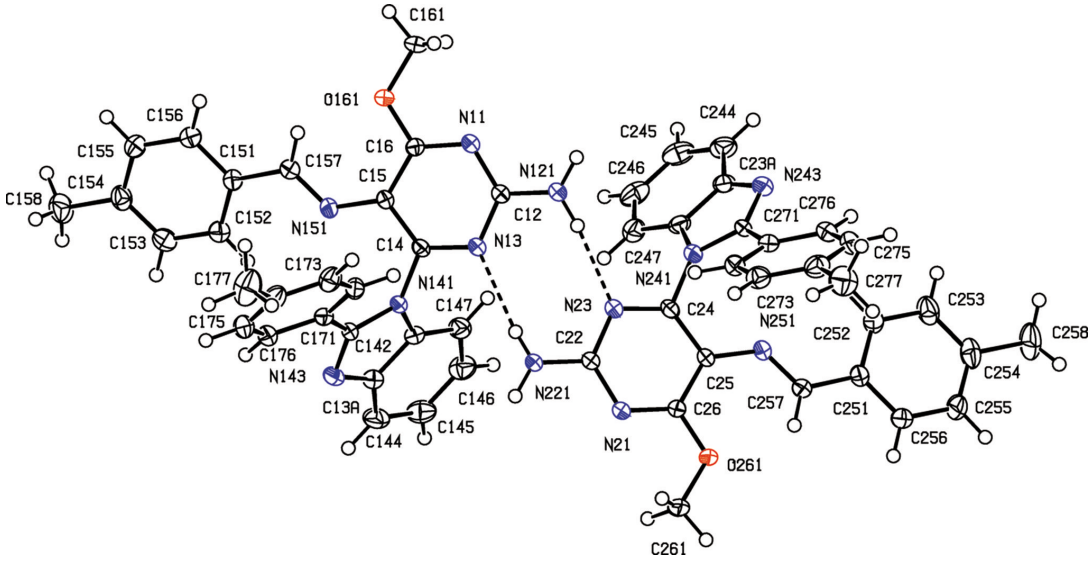


Figure 4
The two independent components in the structure of compound (V), showing the atom-labelling scheme and the hydrogen bonds (drawn as dashed lines) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

Table 3
Selected torsion angles (°) for products (IV)–(VI).

Angle	(IV) x = nil	(V) x = 1	(V) x = 2	(VI*a*) x = 1	(VI*a*) x = 2	(VI*b*) x = nil
φ_1	136.6 (2)	129.54 (18)	130.72 (18)	129.0 (2)	130.7 (2)	121.91 (18)
φ_2	−58.1 (3)	−68.6 (2)	−70.2 (2)	−69.3 (3)	−72.1 (3)	−62.7 (2)
φ_3	−15.6 (3)	−21.6 (3)	−25.7 (2)	−21.4 (3)	−26.0 (3)	−29.4 (3)
φ_4	177.0 (2)	171.48 (17)	173.80 (18)	171.8 (2)	175.7 (2)	145.43 (18)
φ_5	−4.3 (3)	−2.9 (3)	3.8 (3)	−4.9 (4)	0.4 (4)	−3.5 (3)
φ_6	−176.38 (18)	−174.4 (2)	177.12 (17)	179.8 (3)	174.8 (2)	−177.71 (17)

Notes: φ_1 represents the torsion angle N*x*3—C*x*4—N*x*41—C*x*42; φ_2 represents the torsion angles N3—C4—N41—C47*A* in (IV) and (VI*b*), and C*x*4—N*x*41—N*x*41—C*x*7A in (V) and (VI*a*) (see Figs. 3–6); φ_3 represents the torsion angle N*x*41—C*x*42—C*x*71—C*x*72; φ_4 represents the torsion angle C*x*4—C*x*5—N*x*51—C*x*57; φ_5 represents the torsion angle N*x*51—C*x*57—C*x*51—C*x*52; φ_6 represents the torsion angle C*x*5—C*x*6—O*x*61—C*x*61.

N3—C4—N41—C47*A* in (IV) and (VI*b*) or N*x*3—C*x*4—N*x*41—C*x*47 in (V) and (VI*a*) (see Figs. 3–6). All of the products have a negative sign for the torsion angle N*x*41—C*x*42—C*x*71—C*x*72 and, in each product, the magnitudes of the corresponding torsion angles are very similar (Table 3). Overall, the products (IV)–(VI) all have very similar molecular structures but their crystallization characteristics are different as noted above and, as discussed below, their supramolecular arrangements are also very different.

In the intermediates (II) and (III), the signs and magnitudes of the torsion angles N3—C4—N41—C42 and N3—C4—N41—C47*A* are effectively interchanged compared with the corresponding angles in the products (IV)–(VI) (Tables 2 and 3, and Figs. 1–6). In effect, the orientation of the benzimidazole unit in (II) and (III) relative to the pyrimidine differs from that in the products (IV)–(VI) by a rotation of *ca* 180° about the C—N bond linking these two ring systems. Since the imino atom N43/N*x*43 is involved in intermolecular hydrogen bonding in every compound apart from (IV) (Table 4), it is not easy to understand these orientational differences. In each of compounds (II)–(VI), the methoxy C atom is effectively coplanar with the adjacent pyrimidine ring, as indicated by the torsion angles involving these C atoms (Tables 2 and 3).

The intermediates (II) and (III) are isostructural with (I) (Vicentes *et al.*, 2019), and thus exhibit the same pattern of supramolecular assembly, forming complex sheets built from a

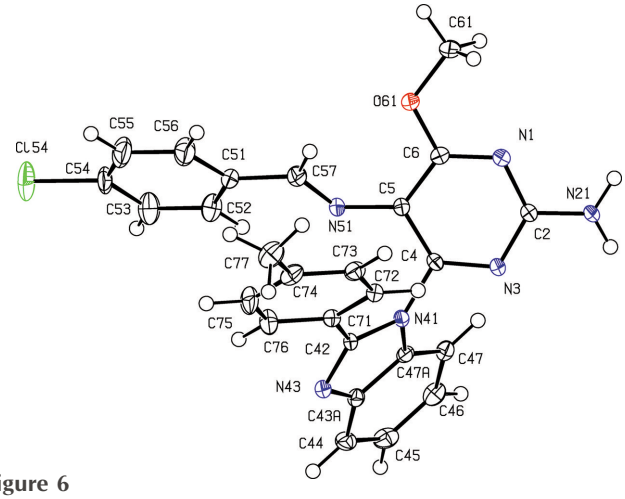


Figure 6
The molecular structure of the Z' = 1 form of compound (VI), denoted (VI*b*), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

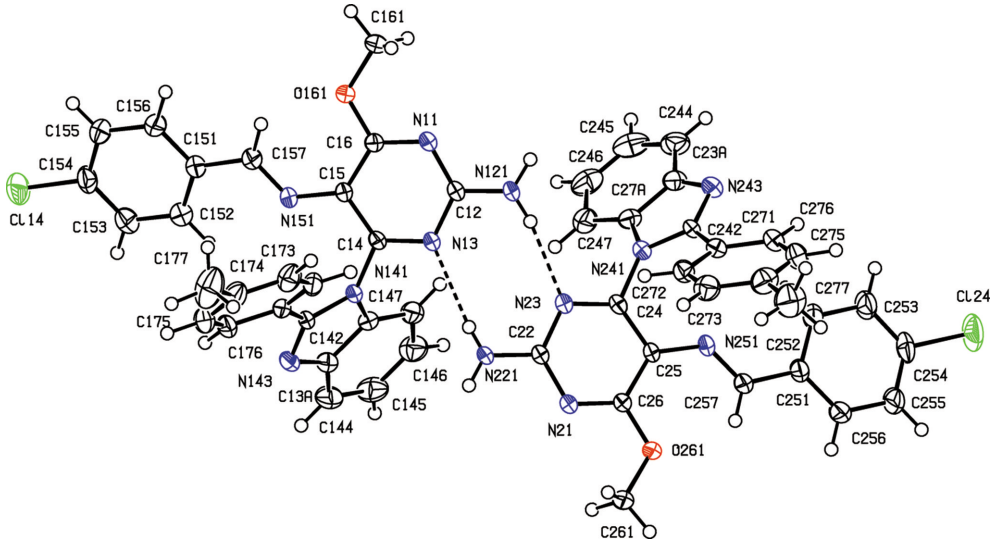


Figure 5
The two independent components in the structure of the Z' = 2 form of compound (VI), denoted (VI*a*), showing the atom-labelling scheme and the hydrogen bonds (drawn as dashed lines) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

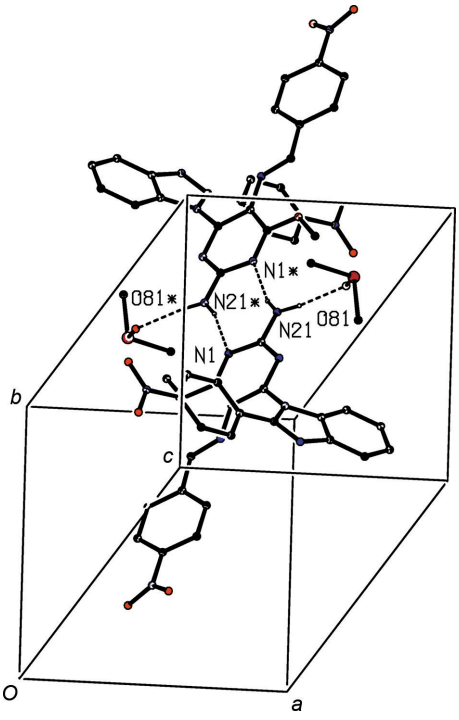


Figure 7
Part of the crystal structure of compound (IV), showing the formation of a centrosymmetric four-molecule aggregate built from N—H···O and N—H···N hydrogen bonds, which are drawn as dashed lines. For the sake of clarity, H atoms which are not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(-x+1, -y+2, -z+1)$.

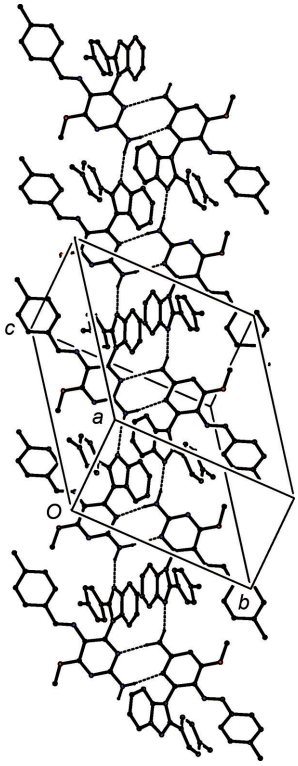


Figure 8
Part of the crystal structure of compound (V), showing the formation of a ribbon of alternating $R_2^2(8)$ and $R_4^4(16)$ rings running parallel to the [101] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms bonded to C atoms have all been omitted.

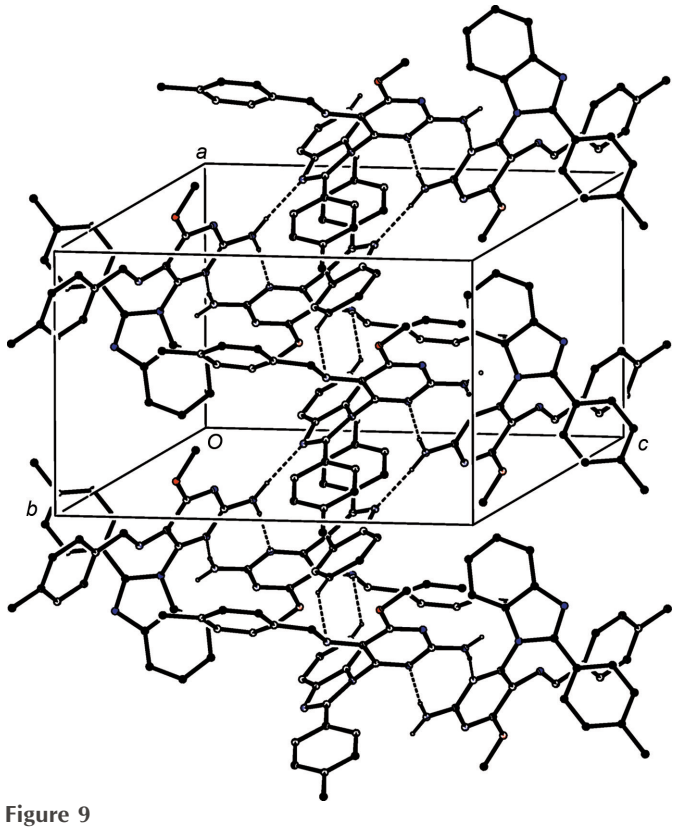


Figure 9
Part of the crystal structure of compound (V), showing the formation of a ribbon of alternating $R_2^2(8)$ and $R_2^2(16)$ rings running parallel to the [100] direction. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms bonded to C atoms but not involved in the motif shown have been omitted.

combination of O—H···N and N—H···O hydrogen bonds. No additional comment is required except to note that the structure of compound (III) contains a fairly short intermolecular Br···O contact whose dimensions are Br74···O61^{i} = 3.0972 (16) Å and C74—Br74···O61^{i} = 173.70 (9)° [symmetry code: (i) $x, y-1, z$], so that the Br···O distance is shorter than the sum of the conventional van der Waals radii of 3.41 Å (Rowland & Taylor, 1996). However, the conventional radii are derived assuming no directional variation in the effective van der Waals radius, but detailed database analysis (Nyburg & Faerman, 1985) for nonbonded contacts involving halogen atoms bonded to C atoms indicates significant angular variation, with the effective radii diminishing as the contact angle approaches 180°, as here. On this basis, the sum of the effective van der Waals radii, 3.08 Å, differs little from the distance observed here, so that this contact in compound (III) should not be regarded as structurally significant.

For the product (IV), the supramolecular assembly is very simple: inversion-related pyrimidine components are linked by N—H···N hydrogen bonds to form a cyclic centrosymmtric $R_2^2(8)$ dimer, to which inversion-related solvent molecules are linked by N—H···O hydrogen bonds (Fig. 7). There are no direction-specific interactions between the four-molecule aggregates of this type.

In the isostructural products (V) and (VI*a*), there are eight independent hydrogen bonds, four of the N—H···N type and

Table 4
Hydrogen bonds and short intramolecular contacts (Å, °) for compounds (II)–(VI).

*Cg*1 and *Cg*2 represent the centroids of the C171–C176 and C271–C276 rings, respectively.

	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(II)	N21—H21*A*···O81	0.90 (2)	2.10 (2)	2.989 (2)	171.1 (18)
	N21—H21*B*···O81^{i}	0.86 (2)	2.094 (19)	2.8840 (18)	153.0 (18)
	N51—H51*A*···O61	0.85 (2)	2.41 (2)	2.7059 (17)	101.1 (17)
	N51—H51*B*···N41	0.90 (2)	2.571 (19)	2.888 (2)	101.5 (14)
	O81—H81*A*···N43^{ii}	0.87 (2)	1.98 (2)	2.8506 (19)	178 (2)
	O81—H81*B*···N51^{iii}	0.84 (2)	2.11 (2)	2.927 (2)	164 (2)
(III)	N21—H21*A*···O81	0.83 (3)	2.17 (3)	2.992 (3)	172 (3)
	N21—H21*B*···O81^{i}	0.87 (3)	2.07 (3)	2.880 (3)	154 (2)
	N51—H51*A*···O61	0.91 (3)	2.41 (3)	2.706 (2)	98 (2)
	N51—H51*B*···N41	0.88 (3)	2.56 (3)	2.885 (3)	103 (2)
	O81—H81*A*···N43^{ii}	0.83 (3)	2.03 (3)	2.862 (3)	178 (4)
	O81—H81*B*···N51^{iii}	0.86 (3)	2.11 (3)	2.932 (3)	159 (3)
(IV)	N21—H21*A*···O81	0.89 (3)	1.94 (3)	2.826 (3)	173 (2)
	N21—H21*B*···N1^{iv}	0.86 (3)	2.32 (3)	3.161 (3)	166 (3)
(V)	N121—H12*A*···N23	0.90 (2)	2.13 (2)	3.022 (2)	171.5 (19)
	N121—H12*B*···N243^{v}	0.92 (2)	2.18 (2)	3.062 (2)	160 (2)
	N221—H22*A*···N13	0.84 (2)	2.19 (2)	3.023 (2)	173.4 (19)
	N221—H22*B*···N143^{ii}	0.90 (2)	2.11 (2)	2.994 (2)	169 (2)
	C146—H146···N151^{ii}	0.95	2.57	3.390 (3)	145
	C176—H176···N21^{vi}	0.95	2.58	3.464 (2)	154
	C155—H155···*Cg*1^{iv}	0.95	2.60	3.465 (2)	151
	C255—H255···*Cg*2^{vii}	0.95	2.87	3.784 (2)	163
(VI*a*)	N121—H12*A*···N23	0.86 (3)	2.18 (3)	3.016 (3)	168 (3)
	N121—H12*B*···N243^{v}	0.89 (3)	2.21 (3)	3.043 (3)	158 (3)
	N221—H22*A*···N13	0.78 (3)	2.23 (3)	3.012 (3)	173 (3)
	N221—H22*B*···N143^{ii}	0.82 (3)	2.18 (3)	2.988 (3)	168 (3)
	C146—H146···N151^{ii}	0.95	2.58	3.404 (4)	146
	C176—H176···N21^{vi}	0.95	2.57	3.449 (3)	154
	C155—H155···*Cg*1^{iv}	0.95	2.55	3.391 (3)	147
	C255—H255···*Cg*2^{vii}	0.95	2.85	3.754 (3)	160
(VI*b*)	N21—H21*A*···N3^{vi}	0.85 (2)	2.35 (2)	3.196 (2)	175.9 (19)
	N21—H21*B*···N43^{viii}	0.90 (2)	2.08 (2)	2.946 (2)	163.6 (19)

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

two each of the C—H···N and C—H···π(arene) types (Table 4), which together link the molecules into three-dimensional framework structures. The differences in the details of the C—H···N and C—H···π(arene) hydrogen bonds involving the two independent molecules confirms the lack of additional crystallographic symmetry. In the selected asymmetric units (Figs. 4 and 5), the two molecules are linked by N—H···N hydrogen bonds, and these dimeric units can be regarded as the basic building block for the three-dimensional assembly, which is readily analysed in terms of simple one-dimensional substructures (Ferguson *et al.*, 1998*a*,*b*; Gregson *et al.*, 2000). Two N—H···N hydrogen bonds, having atoms N143 and N243 as the acceptors (Table 4), link the basic dimers into a ribbon of alternating $R_2^2(8)$ and $R_4^4(16)$ rings running parallel to the [101] direction (Fig. 8). In the second substructure, the linking of the basic dimeric units by the two C—H···N hydrogen bonds generates a ribbon of alternating $R_2^2(8)$ and $R_2^2(16)$ rings running parallel to the [100] direction (Fig. 9). In the final substructure, the linkage of the dimers by two C—H···π(arene) hydrogen bonds generates a chain of rings running parallel to the [12$\bar{1}$] direction (Fig. 10). The combination of the chain motifs along [100], [101] and [12$\bar{1}$] suffices to generate a three-dimensional framework structure.

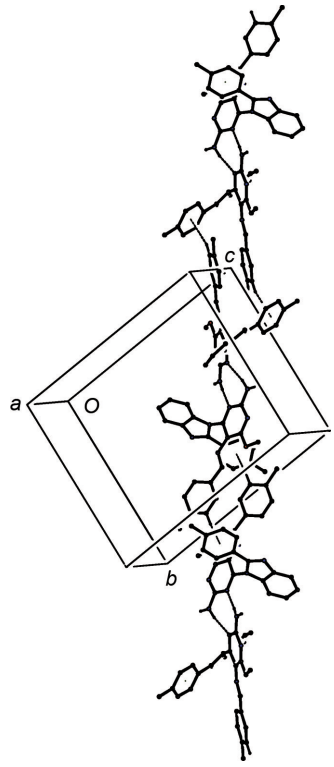


Figure 10
Part of the crystal structure of compound (V), showing the formation of a chain of rings along [12$\bar{1}$] built from N—H···N and C—H···π(arene) hydrogen bonds, which are drawn as dashed lines. For the sake of clarity, H atoms bonded to C atoms but not involved in the motif shown have been omitted.

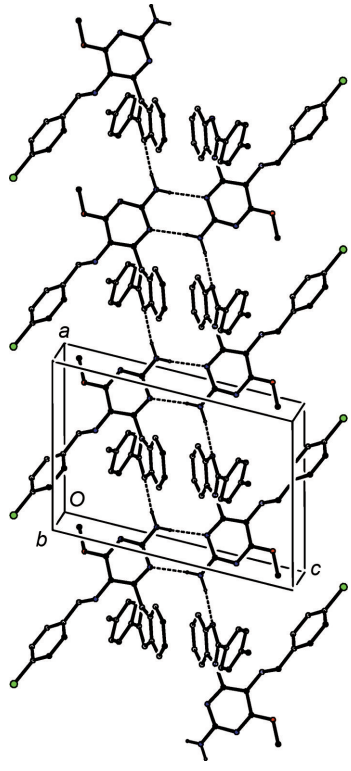


Figure 11
Part of the crystal structure of form (VI*b*), showing the formation of a ribbon of centrosymmetric $R_2^2(8)$ and $R_4^4(16)$ rings running parallel to [100]. Hydrogen bonds are drawn as dashed lines and, for the sake of clarity, H atoms bonded to C atoms have all been omitted.

Two N—H···N hydrogen bonds link the molecules of (VI*b*) into a ribbon of edge-fused centrosymmetric rings running parallel to [100], in which $R_2^2(8)$ rings (Etter, 1990; Etter *et al.*, 1990; Bernstein *et al.*, 1995) centred at (n, $\frac{1}{2}$, $\frac{1}{2}$) alternate with $R_4^4(16)$ rings centred at ($n + \frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) (Fig. 11), where n represents an integer in each case.

We have previously reported the structures of a wide range of multiply-substituted pyrimidines, but many of these carry either C-nitroso (Quesada *et al.*, 2002, 2004; Melguizo *et al.*, 2003) or C-formyl substituents (Cobo *et al.*, 2008), whose presence is associated with highly polarized electronic structures.

4. Summary

We have developed a versatile and efficient synthesis of 5-(arylmethylideneamino)-4-(1*H*-benzo[*d*]imidazol-1-yl)pyrimidine hybrids based on simple starting materials and we have characterized three products and two intermediates spectroscopically (IR, ^{1}H and ^{13}C NMR, and HRMS) and have determined their molecular and supramolecular structures.

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supporting information

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Synthesis of 5-(arylmethylideneamino)-4-(1*H*-benzo[*d*]imidazol-1-yl)pyrimidine hybrids: synthetic sequence and the molecular and supramolecular structures of two intermediates and three final products

Daniel E. Vicentes, Ricaurte Rodríguez, Justo Cobo and Christopher Glidewell

Computing details

For all structures, data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b) and *PLATON* (Spek, 2020).

4-[2-(4-Chlorophenyl)-1*H*-benzo[*d*]imidazol-1-yl]-6-methoxypyrimidine-2,5-diamine monohydrate (II)

Crystal data

$C_{18}H_{15}ClN_6O\cdot H_2O$
$M_r = 384.83$
Triclinic, $P\overline{1}$
$a = 8.2156$ (6) Å
$b = 11.0343$ (7) Å
$c = 11.3968$ (8) Å
$\alpha = 107.980$ (2)°
$\beta = 109.725$ (2)°
$\gamma = 98.541$ (2)°
$V = 887.43$ (11) Å^3
$Z = 2$
$F(000) = 400$
$D_x = 1.440$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4071 reflections
$\theta = 2.3$–27.5°
$\mu = 0.24$ mm^{-1}
$T = 100$ K
Block, colourless
0.22 × 0.20 × 0.14 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.907$, $T_{max} = 0.967$
35335 measured reflections
4071 independent reflections
3487 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -10\rightarrow10$
$k = -14\rightarrow14$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.088$
$S = 1.06$
4071 reflections
263 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0313P)^2 + 0.5991P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.09847 (16)	0.66440 (11)	0.07593 (11)	0.0146 (2)
C2	0.24508 (19)	0.63347 (13)	0.14631 (14)	0.0144 (3)
N3	0.29687 (16)	0.64478 (11)	0.27522 (11)	0.0139 (2)
C4	0.18934 (19)	0.68801 (13)	0.33495 (13)	0.0131 (3)
C5	0.03441 (19)	0.71774 (13)	0.27416 (14)	0.0142 (3)
C6	−0.00310 (18)	0.70323 (13)	0.13920 (14)	0.0138 (3)
N21	0.34981 (18)	0.59262 (13)	0.08236 (13)	0.0179 (3)
H21A	0.439 (3)	0.5615 (18)	0.1212 (18)	0.021*
H21B	0.315 (2)	0.5773 (18)	−0.002 (2)	0.021*
N41	0.25059 (16)	0.71042 (11)	0.47560 (11)	0.0137 (2)
C42	0.28648 (18)	0.62172 (13)	0.53726 (14)	0.0131 (3)
N43	0.37479 (16)	0.68083 (12)	0.66922 (12)	0.0148 (2)
C43A	0.40235 (19)	0.81626 (14)	0.69785 (14)	0.0151 (3)
C44	0.4893 (2)	0.92560 (15)	0.82095 (15)	0.0194 (3)
H44	0.5398	0.9142	0.9037	0.023*
C45	0.4993 (2)	1.05087 (15)	0.81834 (15)	0.0213 (3)
H45	0.5582	1.1265	0.9008	0.026*
C46	0.4244 (2)	1.06910 (14)	0.69675 (15)	0.0193 (3)
H46	0.4344	1.1567	0.6988	0.023*
C47	0.3363 (2)	0.96213 (14)	0.57387 (15)	0.0171 (3)
H47	0.2854	0.9738	0.4913	0.021*
C47A	0.32643 (18)	0.83684 (13)	0.57813 (14)	0.0142 (3)
C71	0.22506 (19)	0.47687 (13)	0.46302 (14)	0.0137 (3)
C72	0.06089 (19)	0.41557 (14)	0.35028 (14)	0.0165 (3)
H72	−0.0115	0.4681	0.3191	0.020*
C73	0.0028 (2)	0.27831 (15)	0.28340 (15)	0.0191 (3)
H73	−0.1093	0.2363	0.2072	0.023*
C74	0.1116 (2)	0.20376 (14)	0.32998 (15)	0.0191 (3)
Cl74	0.03913 (6)	0.03196 (4)	0.24436 (4)	0.02995 (12)
C75	0.2750 (2)	0.26232 (14)	0.44161 (15)	0.0176 (3)
H75	0.3476	0.2095	0.4717	0.021*
C76	0.33098 (19)	0.39939 (14)	0.50867 (14)	0.0147 (3)
H76	0.4420	0.4407	0.5861	0.018*
N51	−0.08849 (18)	0.74965 (13)	0.33228 (13)	0.0181 (3)
H51A	−0.147 (3)	0.7990 (19)	0.3019 (19)	0.022*
H51B	−0.038 (2)	0.7859 (18)	0.423 (2)	0.022*

O61	−0.15313 (14)	0.73322 (10)	0.07799 (10)	0.0176 (2)
C61	−0.1926 (2)	0.72615 (16)	−0.05776 (15)	0.0230 (3)
H61A	−0.1868	0.6402	−0.1128	0.035*
H61B	−0.1040	0.7978	−0.0556	0.035*
H61C	−0.3137	0.7356	−0.0971	0.035*
O81	0.65646 (15)	0.48395 (12)	0.18539 (11)	0.0191 (2)
H81A	0.647 (3)	0.432 (2)	0.228 (2)	0.029*
H81B	0.726 (3)	0.559 (2)	0.241 (2)	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0177 (6)	0.0123 (5)	0.0124 (5)	0.0041 (5)	0.0044 (5)	0.0049 (5)
C2	0.0175 (7)	0.0109 (6)	0.0141 (6)	0.0032 (5)	0.0064 (5)	0.0046 (5)
N3	0.0165 (6)	0.0129 (5)	0.0122 (5)	0.0045 (4)	0.0053 (5)	0.0052 (4)
C4	0.0172 (6)	0.0102 (6)	0.0117 (6)	0.0022 (5)	0.0057 (5)	0.0049 (5)
C5	0.0167 (7)	0.0119 (6)	0.0155 (7)	0.0041 (5)	0.0078 (5)	0.0059 (5)
C6	0.0152 (6)	0.0096 (6)	0.0148 (6)	0.0032 (5)	0.0036 (5)	0.0052 (5)
N21	0.0215 (6)	0.0230 (6)	0.0129 (6)	0.0111 (5)	0.0080 (5)	0.0083 (5)
N41	0.0175 (6)	0.0121 (5)	0.0122 (5)	0.0047 (4)	0.0059 (5)	0.0054 (4)
C42	0.0137 (6)	0.0153 (6)	0.0146 (6)	0.0063 (5)	0.0076 (5)	0.0081 (5)
N43	0.0175 (6)	0.0147 (6)	0.0135 (5)	0.0050 (5)	0.0067 (5)	0.0063 (5)
C43A	0.0172 (7)	0.0158 (7)	0.0145 (7)	0.0055 (5)	0.0078 (5)	0.0070 (5)
C44	0.0234 (7)	0.0192 (7)	0.0125 (7)	0.0046 (6)	0.0053 (6)	0.0051 (6)
C45	0.0240 (8)	0.0162 (7)	0.0167 (7)	0.0026 (6)	0.0059 (6)	0.0013 (6)
C46	0.0230 (7)	0.0137 (7)	0.0218 (7)	0.0046 (6)	0.0103 (6)	0.0064 (6)
C47	0.0211 (7)	0.0159 (7)	0.0170 (7)	0.0061 (6)	0.0086 (6)	0.0083 (6)
C47A	0.0156 (7)	0.0137 (6)	0.0128 (6)	0.0037 (5)	0.0068 (5)	0.0037 (5)
C71	0.0171 (7)	0.0144 (6)	0.0141 (6)	0.0049 (5)	0.0096 (5)	0.0071 (5)
C72	0.0173 (7)	0.0159 (7)	0.0184 (7)	0.0057 (5)	0.0078 (6)	0.0082 (6)
C73	0.0184 (7)	0.0177 (7)	0.0184 (7)	0.0014 (6)	0.0066 (6)	0.0058 (6)
C74	0.0277 (8)	0.0118 (6)	0.0204 (7)	0.0039 (6)	0.0145 (6)	0.0053 (6)
Cl74	0.0432 (3)	0.01167 (17)	0.0302 (2)	0.00458 (16)	0.01356 (19)	0.00493 (15)
C75	0.0238 (7)	0.0167 (7)	0.0217 (7)	0.0105 (6)	0.0143 (6)	0.0116 (6)
C76	0.0169 (7)	0.0172 (7)	0.0142 (6)	0.0060 (5)	0.0083 (5)	0.0087 (5)
N51	0.0200 (6)	0.0212 (6)	0.0176 (6)	0.0103 (5)	0.0098 (5)	0.0089 (5)
O61	0.0177 (5)	0.0193 (5)	0.0156 (5)	0.0088 (4)	0.0038 (4)	0.0079 (4)
C61	0.0255 (8)	0.0255 (8)	0.0163 (7)	0.0108 (6)	0.0027 (6)	0.0105 (6)
O81	0.0238 (6)	0.0207 (5)	0.0156 (5)	0.0065 (4)	0.0085 (4)	0.0101 (4)

Geometric parameters (Å, º)

N1—C6	1.3120 (18)	C46—C47	1.386 (2)
N1—C2	1.3601 (18)	C46—H46	0.9500
C2—N3	1.3432 (18)	C47—C47A	1.3896 (19)
C2—N21	1.3456 (19)	C47—H47	0.9500
N3—C4	1.3423 (18)	C71—C76	1.3960 (19)
C4—C5	1.370 (2)	C71—C72	1.397 (2)

C4—N41	1.4345 (17)	C72—C73	1.389 (2)
C5—N51	1.4125 (18)	C72—H72	0.9500
C5—C6	1.4141 (19)	C73—C74	1.387 (2)
C6—O61	1.3412 (17)	C73—H73	0.9500
N21—H21A	0.90 (2)	C74—C75	1.386 (2)
N21—H21B	0.858 (19)	C74—Cl74	1.7410 (15)
N41—C42	1.3818 (17)	C75—C76	1.388 (2)
N41—C47A	1.3941 (17)	C75—H75	0.9500
C42—N43	1.3156 (18)	C76—H76	0.9500
C42—C71	1.4705 (19)	N51—H51A	0.86 (2)
N43—C43A	1.3926 (18)	N51—H51B	0.897 (19)
C43A—C44	1.399 (2)	O61—C61	1.4437 (18)
C43A—C47A	1.4004 (19)	C61—H61A	0.9800
C44—C45	1.383 (2)	C61—H61B	0.9800
C44—H44	0.9500	C61—H61C	0.9800
C45—C46	1.404 (2)	O81—H81A	0.87 (2)
C45—H45	0.9500	O81—H81B	0.84 (2)

C6—N1—C2	116.25 (12)	C46—C47—C47A	116.30 (13)
N3—C2—N21	117.62 (13)	C46—C47—H47	121.8
N3—C2—N1	125.43 (13)	C47A—C47—H47	121.8
N21—C2—N1	116.90 (13)	C47—C47A—N41	131.28 (13)
C4—N3—C2	115.20 (12)	C47—C47A—C43A	123.28 (13)
N3—C4—C5	125.26 (13)	N41—C47A—C43A	105.43 (12)
N3—C4—N41	115.72 (12)	C76—C71—C72	119.63 (13)
C5—C4—N41	118.93 (12)	C76—C71—C42	119.16 (13)
C4—C5—N51	125.16 (13)	C72—C71—C42	121.19 (13)
C4—C5—C6	113.70 (12)	C73—C72—C71	120.42 (14)
N51—C5—C6	120.95 (13)	C73—C72—H72	119.8
N1—C6—O61	121.10 (12)	C71—C72—H72	119.8
N1—C6—C5	124.09 (13)	C74—C73—C72	118.78 (14)
O61—C6—C5	114.82 (12)	C74—C73—H73	120.6
C2—N21—H21A	119.5 (12)	C72—C73—H73	120.6
C2—N21—H21B	119.7 (12)	C75—C74—C73	121.86 (13)
H21A—N21—H21B	118.9 (17)	C75—C74—Cl74	119.65 (12)
C42—N41—C47A	106.42 (11)	C73—C74—Cl74	118.49 (12)
C42—N41—C4	128.98 (11)	C74—C75—C76	118.95 (13)
C47A—N41—C4	123.01 (11)	C74—C75—H75	120.5
N43—C42—N41	112.66 (12)	C76—C75—H75	120.5
N43—C42—C71	124.09 (12)	C75—C76—C71	120.35 (13)
N41—C42—C71	123.20 (12)	C75—C76—H76	119.8
C42—N43—C43A	105.59 (11)	C71—C76—H76	119.8
N43—C43A—C44	130.64 (13)	C5—N51—H51A	113.1 (12)
N43—C43A—C47A	109.88 (12)	C5—N51—H51B	113.7 (12)
C44—C43A—C47A	119.47 (13)	H51A—N51—H51B	109.7 (17)
C45—C44—C43A	117.87 (14)	C6—O61—C61	116.89 (11)
C45—C44—H44	121.1	O61—C61—H61A	109.5
C43A—C44—H44	121.1	O61—C61—H61B	109.5

C44—C45—C46	121.62 (14)	H61A—C61—H61B	109.5
C44—C45—H45	119.2	O61—C61—H61C	109.5
C46—C45—H45	119.2	H61A—C61—H61C	109.5
C47—C46—C45	121.44 (14)	H61B—C61—H61C	109.5
C47—C46—H46	119.3	H81A—O81—H81B	109.3 (19)
C45—C46—H46	119.3		

C6—N1—C2—N3	−2.9 (2)	C43A—C44—C45—C46	0.3 (2)
C6—N1—C2—N21	179.56 (12)	C44—C45—C46—C47	0.3 (2)
N21—C2—N3—C4	178.92 (12)	C45—C46—C47—C47A	0.0 (2)
N1—C2—N3—C4	1.4 (2)	C46—C47—C47A—N41	−179.37 (14)
C2—N3—C4—C5	1.2 (2)	C46—C47—C47A—C43A	−1.0 (2)
C2—N3—C4—N41	−175.32 (11)	C42—N41—C47A—C47	177.63 (15)
N3—C4—C5—N51	172.92 (13)	C4—N41—C47A—C47	10.8 (2)
N41—C4—C5—N51	−10.6 (2)	C42—N41—C47A—C43A	−0.93 (15)
N3—C4—C5—C6	−2.1 (2)	C4—N41—C47A—C43A	−167.73 (12)
N41—C4—C5—C6	174.41 (12)	N43—C43A—C47A—C47	−178.30 (13)
C2—N1—C6—O61	−178.48 (12)	C44—C43A—C47A—C47	1.7 (2)
C2—N1—C6—C5	1.9 (2)	N43—C43A—C47A—N41	0.40 (15)
C4—C5—C6—N1	0.4 (2)	C44—C43A—C47A—N41	−179.65 (13)
N51—C5—C6—N1	−174.85 (13)	N43—C42—C71—C76	−35.0 (2)
C4—C5—C6—O61	−179.29 (12)	N41—C42—C71—C76	147.80 (13)
N51—C5—C6—O61	5.50 (19)	N43—C42—C71—C72	143.28 (14)
N3—C4—N41—C42	−59.42 (19)	N41—C42—C71—C72	−34.0 (2)
C5—C4—N41—C42	123.78 (15)	C76—C71—C72—C73	−0.1 (2)
N3—C4—N41—C47A	104.21 (15)	C42—C71—C72—C73	−178.36 (13)
C5—C4—N41—C47A	−72.59 (17)	C71—C72—C73—C74	−0.6 (2)
C47A—N41—C42—N43	1.21 (16)	C72—C73—C74—C75	0.6 (2)
C4—N41—C42—N43	166.95 (13)	C72—C73—C74—Cl74	−179.23 (11)
C47A—N41—C42—C71	178.73 (12)	C73—C74—C75—C76	0.1 (2)
C4—N41—C42—C71	−15.5 (2)	Cl74—C74—C75—C76	179.93 (11)
N41—C42—N43—C43A	−0.94 (16)	C74—C75—C76—C71	−0.8 (2)
C71—C42—N43—C43A	−178.43 (13)	C72—C71—C76—C75	0.8 (2)
C42—N43—C43A—C44	−179.63 (15)	C42—C71—C76—C75	179.11 (12)
C42—N43—C43A—C47A	0.31 (16)	N1—C6—O61—C61	−2.57 (19)
N43—C43A—C44—C45	178.73 (15)	C5—C6—O61—C61	177.09 (12)
C47A—C43A—C44—C45	−1.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*A*···O81	0.90 (2)	2.10 (2)	2.989 (2)	171.1 (18)
N21—H21*B*···O81^{i}	0.86 (2)	2.094 (19)	2.8840 (18)	153.0 (18)
N51—H51*A*···O61	0.85 (2)	2.41 (2)	2.7059 (17)	101.1 (17)
N51—H51*B*···N41	0.90 (2)	2.571 (19)	2.888 (2)	101.5 (14)

O81—H81*A*···N43^{ii}	0.87 (2)	1.98 (2)	2.8506 (19)	178 (2)
O81—H81*B*···N51^{iii}	0.84 (2)	2.11 (2)	2.927 (2)	164 (2)

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

4-[2-(4-Bromophenyl)-1*H*-benzo[*d*]imidazol-1-yl]-6-methoxypyrimidine-2,5-diamine monohydrate (III)

Crystal data

$C_{18}H_{15}BrN_6O·H_2O$
M_r = 429.29
Triclinic, $P\overline{1}$
a = 8.1975 (7) Å
b = 11.1963 (8) Å
c = 11.3644 (10) Å
α = 107.938 (2)°
β = 109.866 (3)°
γ = 98.683 (3)°
V = 894.39 (13) Å^3
Z = 2
F(000) = 436
D_x = 1.594 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4457 reflections
θ = 2.3–28.3°
μ = 2.33 mm^{-1}
T = 100 K
Block, colourless
0.15 × 0.11 × 0.08 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.719, T_{max} = 0.830
50505 measured reflections
4457 independent reflections
3696 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 28.3°, θ_{min} = 2.3°
h = −10→10
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.072
S = 1.09
4457 reflections
263 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0122P)^2 + 1.2915P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.0997 (2)	0.66298 (16)	0.07575 (17)	0.0148 (3)
C2	0.2476 (3)	0.63435 (18)	0.1472 (2)	0.0143 (4)
N3	0.3002 (2)	0.64670 (16)	0.27695 (17)	0.0143 (3)
C4	0.1922 (3)	0.68865 (18)	0.33633 (19)	0.0130 (4)

C5	0.0354 (3)	0.71608 (18)	0.2743 (2)	0.0140 (4)
C6	−0.0022 (3)	0.70080 (18)	0.1386 (2)	0.0140 (4)
N21	0.3529 (3)	0.59482 (19)	0.08384 (19)	0.0188 (4)
H21A	0.434 (4)	0.565 (3)	0.119 (3)	0.023*
H21B	0.322 (3)	0.583 (2)	−0.001 (3)	0.023*
N41	0.2535 (2)	0.71161 (16)	0.47749 (16)	0.0133 (3)
C42	0.2896 (3)	0.62455 (19)	0.5397 (2)	0.0130 (4)
N43	0.3796 (2)	0.68330 (16)	0.67223 (17)	0.0149 (3)
C43A	0.4081 (3)	0.81718 (19)	0.7007 (2)	0.0151 (4)
C44	0.4971 (3)	0.9252 (2)	0.8241 (2)	0.0196 (4)
H44	0.5486	0.9144	0.9073	0.024*
C45	0.5073 (3)	1.0486 (2)	0.8206 (2)	0.0217 (4)
H45	0.5678	1.1235	0.9030	0.026*
C46	0.4307 (3)	1.0656 (2)	0.6987 (2)	0.0204 (4)
H46	0.4405	1.1519	0.7006	0.024*
C47	0.3410 (3)	0.9602 (2)	0.5754 (2)	0.0170 (4)
H47	0.2894	0.9714	0.4924	0.020*
C47A	0.3309 (3)	0.83645 (19)	0.5803 (2)	0.0142 (4)
C71	0.2262 (3)	0.48164 (18)	0.46518 (19)	0.0127 (4)
C72	0.0605 (3)	0.4223 (2)	0.3526 (2)	0.0162 (4)
H72	−0.0113	0.4748	0.3223	0.019*
C73	−0.0002 (3)	0.2870 (2)	0.2846 (2)	0.0182 (4)
H73	−0.1129	0.2461	0.2079	0.022*
C74	0.1074 (3)	0.21272 (19)	0.3312 (2)	0.0174 (4)
Br74	0.02424 (4)	0.02793 (2)	0.23620 (3)	0.02717 (7)
C75	0.2721 (3)	0.2692 (2)	0.4426 (2)	0.0165 (4)
H75	0.3437	0.2164	0.4724	0.020*
C76	0.3309 (3)	0.40459 (19)	0.5102 (2)	0.0146 (4)
H76	0.4431	0.4448	0.5875	0.017*
N51	−0.0886 (3)	0.74609 (18)	0.3316 (2)	0.0186 (4)
H51A	−0.148 (3)	0.800 (3)	0.301 (3)	0.022*
H51B	−0.039 (3)	0.780 (2)	0.421 (3)	0.022*
O61	−0.15381 (19)	0.72932 (14)	0.07624 (14)	0.0168 (3)
C61	−0.1941 (3)	0.7200 (2)	−0.0610 (2)	0.0221 (5)
H61A	−0.1875	0.6352	−0.1149	0.033*
H61B	−0.1060	0.7906	−0.0598	0.033*
H61C	−0.3162	0.7280	−0.1014	0.033*
O81	0.6558 (2)	0.48275 (16)	0.18500 (16)	0.0193 (3)
H81A	0.644 (4)	0.435 (3)	0.227 (3)	0.029*
H81B	0.728 (4)	0.557 (3)	0.246 (3)	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0179 (9)	0.0126 (8)	0.0123 (8)	0.0040 (6)	0.0046 (7)	0.0049 (6)
C2	0.0178 (10)	0.0104 (9)	0.0131 (9)	0.0029 (7)	0.0058 (8)	0.0035 (7)
N3	0.0169 (8)	0.0134 (8)	0.0126 (8)	0.0046 (6)	0.0060 (7)	0.0052 (6)
C4	0.0178 (10)	0.0108 (9)	0.0101 (9)	0.0031 (7)	0.0055 (8)	0.0044 (7)

C5	0.0172 (10)	0.0104 (9)	0.0151 (9)	0.0035 (7)	0.0075 (8)	0.0050 (7)
C6	0.0142 (9)	0.0113 (9)	0.0144 (9)	0.0027 (7)	0.0034 (8)	0.0056 (7)
N21	0.0217 (10)	0.0249 (9)	0.0148 (9)	0.0122 (8)	0.0095 (8)	0.0092 (7)
N41	0.0170 (8)	0.0116 (8)	0.0109 (8)	0.0040 (6)	0.0050 (7)	0.0044 (6)
C42	0.0141 (9)	0.0151 (9)	0.0139 (9)	0.0061 (7)	0.0077 (8)	0.0080 (7)
N43	0.0169 (9)	0.0145 (8)	0.0133 (8)	0.0038 (7)	0.0065 (7)	0.0055 (6)
C43A	0.0164 (10)	0.0148 (9)	0.0154 (10)	0.0042 (8)	0.0075 (8)	0.0063 (8)
C44	0.0224 (11)	0.0198 (10)	0.0129 (10)	0.0036 (8)	0.0054 (8)	0.0047 (8)
C45	0.0237 (11)	0.0161 (10)	0.0180 (10)	0.0007 (8)	0.0067 (9)	0.0016 (8)
C46	0.0233 (11)	0.0132 (9)	0.0234 (11)	0.0040 (8)	0.0101 (9)	0.0056 (8)
C47	0.0202 (10)	0.0163 (10)	0.0161 (10)	0.0054 (8)	0.0076 (8)	0.0080 (8)
C47A	0.0154 (10)	0.0131 (9)	0.0131 (9)	0.0028 (7)	0.0069 (8)	0.0032 (7)
C71	0.0159 (9)	0.0128 (9)	0.0119 (9)	0.0040 (7)	0.0075 (8)	0.0059 (7)
C72	0.0175 (10)	0.0159 (9)	0.0170 (10)	0.0058 (8)	0.0070 (8)	0.0081 (8)
C73	0.0165 (10)	0.0165 (10)	0.0172 (10)	0.0018 (8)	0.0051 (8)	0.0044 (8)
C74	0.0243 (11)	0.0105 (9)	0.0210 (10)	0.0041 (8)	0.0136 (9)	0.0062 (8)
Br74	0.03690 (14)	0.01234 (10)	0.02847 (13)	0.00458 (9)	0.01274 (10)	0.00493 (8)
C75	0.0207 (10)	0.0160 (10)	0.0203 (10)	0.0093 (8)	0.0120 (9)	0.0106 (8)
C76	0.0162 (10)	0.0171 (9)	0.0131 (9)	0.0052 (8)	0.0078 (8)	0.0072 (8)
N51	0.0194 (9)	0.0233 (9)	0.0188 (9)	0.0106 (8)	0.0110 (8)	0.0099 (8)
O61	0.0163 (7)	0.0198 (7)	0.0144 (7)	0.0084 (6)	0.0040 (6)	0.0077 (6)
C61	0.0256 (12)	0.0240 (11)	0.0144 (10)	0.0092 (9)	0.0027 (9)	0.0096 (9)
O81	0.0223 (8)	0.0224 (8)	0.0159 (7)	0.0065 (6)	0.0083 (6)	0.0104 (6)

Geometric parameters (Å, º)

N1—C6	1.309 (3)	C46—C47	1.384 (3)
N1—C2	1.357 (3)	C46—H46	0.9500
C2—N21	1.342 (3)	C47—C47A	1.395 (3)
C2—N3	1.343 (2)	C47—H47	0.9500
N3—C4	1.340 (3)	C71—C76	1.393 (3)
C4—C5	1.372 (3)	C71—C72	1.395 (3)
C4—N41	1.432 (2)	C72—C73	1.390 (3)
C5—N51	1.411 (3)	C72—H72	0.9500
C5—C6	1.414 (3)	C73—C74	1.387 (3)
C6—O61	1.345 (2)	C73—H73	0.9500
N21—H21A	0.82 (3)	C74—C75	1.383 (3)
N21—H21B	0.87 (3)	C74—Br74	1.900 (2)
N41—C42	1.381 (2)	C75—C76	1.389 (3)
N41—C47A	1.394 (2)	C75—H75	0.9500
C42—N43	1.316 (3)	C76—H76	0.9500
C42—C71	1.471 (3)	N51—H51A	0.90 (3)
N43—C43A	1.396 (3)	N51—H51B	0.88 (3)
C43A—C47A	1.398 (3)	O61—C61	1.447 (2)
C43A—C44	1.398 (3)	C61—H61A	0.9800
C44—C45	1.385 (3)	C61—H61B	0.9800
C44—H44	0.9500	C61—H61C	0.9800
C45—C46	1.398 (3)	O81—H81A	0.84 (3)

C45—H45	0.9500	O81—H81B	0.86 (3)
C6—N1—C2	116.25 (17)	C46—C47—C47A	115.99 (19)
N21—C2—N3	117.62 (19)	C46—C47—H47	122.0
N21—C2—N1	116.90 (18)	C47A—C47—H47	122.0
N3—C2—N1	125.43 (18)	N41—C47A—C47	131.07 (19)
C4—N3—C2	115.27 (17)	N41—C47A—C43A	105.73 (17)
N3—C4—C5	125.21 (18)	C47—C47A—C43A	123.17 (18)
N3—C4—N41	115.91 (17)	C76—C71—C72	119.76 (18)
C5—C4—N41	118.81 (17)	C76—C71—C42	119.38 (18)
C4—C5—N51	125.19 (18)	C72—C71—C42	120.84 (18)
C4—C5—C6	113.49 (18)	C73—C72—C71	120.43 (19)
N51—C5—C6	121.11 (18)	C73—C72—H72	119.8
N1—C6—O61	121.09 (18)	C71—C72—H72	119.8
N1—C6—C5	124.27 (18)	C74—C73—C72	118.57 (19)
O61—C6—C5	114.64 (18)	C74—C73—H73	120.7
C2—N21—H21A	119.5 (18)	C72—C73—H73	120.7
C2—N21—H21B	120.6 (17)	C75—C74—C73	122.08 (19)
H21A—N21—H21B	118 (2)	C75—C74—Br74	119.74 (15)
C42—N41—C47A	106.32 (16)	C73—C74—Br74	118.17 (16)
C42—N41—C4	128.77 (16)	C74—C75—C76	118.84 (19)
C47A—N41—C4	123.21 (16)	C74—C75—H75	120.6
N43—C42—N41	112.68 (17)	C76—C75—H75	120.6
N43—C42—C71	124.25 (18)	C75—C76—C71	120.32 (19)
N41—C42—C71	123.01 (17)	C75—C76—H76	119.8
C42—N43—C43A	105.60 (16)	C71—C76—H76	119.8
N43—C43A—C47A	109.65 (17)	C5—N51—H51A	112.7 (16)
N43—C43A—C44	130.60 (19)	C5—N51—H51B	113.7 (17)
C47A—C43A—C44	119.74 (19)	H51A—N51—H51B	109 (2)
C45—C44—C43A	117.6 (2)	C6—O61—C61	116.63 (16)
C45—C44—H44	121.2	O61—C61—H61A	109.5
C43A—C44—H44	121.2	O61—C61—H61B	109.5
C44—C45—C46	121.7 (2)	H61A—C61—H61B	109.5
C44—C45—H45	119.2	O61—C61—H61C	109.5
C46—C45—H45	119.2	H61A—C61—H61C	109.5
C47—C46—C45	121.8 (2)	H61B—C61—H61C	109.5
C47—C46—H46	119.1	H81A—O81—H81B	106 (3)
C45—C46—H46	119.1		
C6—N1—C2—N21	179.68 (18)	C43A—C44—C45—C46	0.5 (3)
C6—N1—C2—N3	−2.8 (3)	C44—C45—C46—C47	−0.1 (4)
N21—C2—N3—C4	178.87 (18)	C45—C46—C47—C47A	0.4 (3)
N1—C2—N3—C4	1.3 (3)	C42—N41—C47A—C47	177.7 (2)
C2—N3—C4—C5	1.4 (3)	C4—N41—C47A—C47	11.4 (3)
C2—N3—C4—N41	−175.54 (16)	C42—N41—C47A—C43A	−0.9 (2)
N3—C4—C5—N51	172.53 (19)	C4—N41—C47A—C43A	−167.15 (18)
N41—C4—C5—N51	−10.6 (3)	C46—C47—C47A—N41	−179.4 (2)
N3—C4—C5—C6	−2.3 (3)	C46—C47—C47A—C43A	−1.1 (3)

N41—C4—C5—C6	174.54 (16)	N43—C43A—C47A—N41	0.3 (2)
C2—N1—C6—O61	−178.81 (17)	C44—C43A—C47A—N41	−179.81 (18)
C2—N1—C6—C5	1.6 (3)	N43—C43A—C47A—C47	−178.35 (19)
C4—C5—C6—N1	0.7 (3)	C44—C43A—C47A—C47	1.5 (3)
N51—C5—C6—N1	−174.39 (19)	N43—C42—C71—C76	−35.0 (3)
C4—C5—C6—O61	−178.90 (17)	N41—C42—C71—C76	147.94 (19)
N51—C5—C6—O61	6.0 (3)	N43—C42—C71—C72	143.2 (2)
N3—C4—N41—C42	−59.7 (3)	N41—C42—C71—C72	−33.9 (3)
C5—C4—N41—C42	123.2 (2)	C76—C71—C72—C73	−0.4 (3)
N3—C4—N41—C47A	103.3 (2)	C42—C71—C72—C73	−178.57 (18)
C5—C4—N41—C47A	−73.8 (3)	C71—C72—C73—C74	0.0 (3)
C47A—N41—C42—N43	1.2 (2)	C72—C73—C74—C75	0.1 (3)
C4—N41—C42—N43	166.42 (19)	C72—C73—C74—Br74	−179.32 (15)
C47A—N41—C42—C71	178.53 (18)	C73—C74—C75—C76	0.3 (3)
C4—N41—C42—C71	−16.2 (3)	Br74—C74—C75—C76	179.64 (15)
N41—C42—N43—C43A	−0.9 (2)	C74—C75—C76—C71	−0.7 (3)
C71—C42—N43—C43A	−178.26 (18)	C72—C71—C76—C75	0.8 (3)
C42—N43—C43A—C47A	0.3 (2)	C42—C71—C76—C75	178.94 (18)
C42—N43—C43A—C44	−179.5 (2)	N1—C6—O61—C61	−1.9 (3)
N43—C43A—C44—C45	178.7 (2)	C5—C6—O61—C61	177.67 (17)
C47A—C43A—C44—C45	−1.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*A*···O81	0.83 (3)	2.17 (3)	2.992 (3)	172 (3)
N21—H21*B*···O81^{i}	0.87 (3)	2.07 (3)	2.880 (3)	154 (2)
N51—H51*A*···O61	0.91 (3)	2.41 (3)	2.706 (2)	98 (2)
N51—H51*B*···N41	0.88 (3)	2.56 (3)	2.885 (3)	103 (2)
O81—H81*A*···N43^{ii}	0.83 (3)	2.03 (3)	2.862 (3)	178 (4)
O81—H81*B*···N51^{iii}	0.86 (3)	2.11 (3)	2.932 (3)	159 (3)

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

(*E*)-4-Methoxy-5-[(4-nitrobenzylidene)amino]-6-[2-(4-nitrophenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine dimethyl sulfoxide monosolvate (IV)

Crystal data

$C_{25}H_{18}N_8O_5·C_2H_6OS$
M_r = 588.60
Triclinic, $P\overline{1}$
a = 9.8192 (8) Å
b = 10.2765 (7) Å
c = 14.4096 (11) Å
α = 71.718 (2)°
β = 74.872 (3)°
γ = 88.786 (3)°
V = 1329.87 (18) Å^3
Z = 2
F(000) = 612
D_x = 1.470 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6115 reflections
θ = 2.2–27.5°
μ = 0.18 mm^{-1}
T = 100 K
Block, yellow
0.19 × 0.15 × 0.10 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.905, T_{max} = 0.982
66738 measured reflections
6115 independent reflections
4641 reflections with $I > 2\sigma(I)$
R_{int} = 0.112
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −12→12
k = −13→13
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.108
S = 1.05
6115 reflections
388 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 1.3248P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.43253 (18)	0.79071 (17)	0.54652 (13)	0.0170 (4)
C2	0.5173 (2)	0.7901 (2)	0.60757 (15)	0.0171 (4)
N3	0.54460 (18)	0.67795 (17)	0.67807 (13)	0.0176 (4)
C4	0.4885 (2)	0.5594 (2)	0.68216 (15)	0.0168 (4)
C5	0.4052 (2)	0.5425 (2)	0.62107 (15)	0.0171 (4)
C6	0.3791 (2)	0.6694 (2)	0.55354 (15)	0.0167 (4)
N21	0.5784 (2)	0.90925 (18)	0.59978 (15)	0.0201 (4)
H21A	0.639 (3)	0.907 (2)	0.6365 (19)	0.024*
H21B	0.567 (3)	0.984 (3)	0.5548 (19)	0.024*
N41	0.52857 (18)	0.44323 (17)	0.75244 (13)	0.0172 (4)
C42	0.4490 (2)	0.3276 (2)	0.82333 (16)	0.0186 (4)
N43	0.52775 (19)	0.22943 (17)	0.85929 (14)	0.0205 (4)
C43A	0.6665 (2)	0.2806 (2)	0.81085 (16)	0.0197 (4)
C44	0.7926 (2)	0.2186 (2)	0.81965 (16)	0.0214 (5)
H44	0.7920	0.1275	0.8635	0.026*
C45	0.9176 (2)	0.2939 (2)	0.76256 (17)	0.0243 (5)
H45	1.0046	0.2534	0.7668	0.029*
C46	0.9196 (2)	0.4295 (2)	0.69810 (17)	0.0244 (5)
H46	1.0078	0.4791	0.6608	0.029*
C47	0.7960 (2)	0.4917 (2)	0.68806 (17)	0.0215 (4)

H47	0.7970	0.5829	0.6444	0.026*
C47A	0.6703 (2)	0.4149 (2)	0.74473 (16)	0.0178 (4)
C71	0.2950 (2)	0.3171 (2)	0.86662 (16)	0.0187 (4)
C72	0.2132 (2)	0.4310 (2)	0.85996 (16)	0.0197 (4)
H72	0.2563	0.5202	0.8213	0.024*
C73	0.0705 (2)	0.4157 (2)	0.90880 (16)	0.0212 (4)
H73	0.0147	0.4929	0.9029	0.025*
C74	0.0107 (2)	0.2847 (2)	0.96674 (16)	0.0208 (4)
C75	0.0889 (2)	0.1703 (2)	0.97656 (17)	0.0224 (5)
H75	0.0459	0.0820	1.0177	0.027*
C76	0.2306 (2)	0.1865 (2)	0.92565 (17)	0.0220 (5)
H76	0.2851	0.1084	0.9307	0.026*
N74	−0.1415 (2)	0.26614 (19)	1.01637 (14)	0.0245 (4)
O741	−0.20964 (18)	0.36831 (17)	1.01215 (14)	0.0336 (4)
O742	−0.19425 (18)	0.14756 (17)	1.05937 (13)	0.0315 (4)
N51	0.36085 (19)	0.40863 (17)	0.63256 (13)	0.0193 (4)
C57	0.2796 (2)	0.3757 (2)	0.58567 (16)	0.0186 (4)
H57	0.2474	0.4448	0.5362	0.022*
C51	0.2365 (2)	0.2300 (2)	0.60895 (16)	0.0177 (4)
C52	0.2897 (2)	0.1258 (2)	0.67645 (16)	0.0198 (4)
H52	0.3590	0.1482	0.7053	0.024*
C53	0.2422 (2)	−0.0098 (2)	0.70147 (17)	0.0214 (4)
H53	0.2779	−0.0810	0.7474	0.026*
C54	0.1414 (2)	−0.0392 (2)	0.65808 (16)	0.0199 (4)
C55	0.0860 (2)	0.0609 (2)	0.59078 (17)	0.0215 (4)
H55	0.0171	0.0376	0.5620	0.026*
C56	0.1345 (2)	0.1960 (2)	0.56703 (16)	0.0204 (4)
H56	0.0978	0.2668	0.5215	0.024*
N54	0.0912 (2)	−0.18335 (18)	0.68409 (15)	0.0256 (4)
O541	0.1070 (2)	−0.26502 (17)	0.76353 (15)	0.0404 (5)
O542	0.03663 (18)	−0.21507 (16)	0.62613 (13)	0.0311 (4)
O61	0.29795 (16)	0.66244 (14)	0.49389 (11)	0.0193 (3)
C61	0.2767 (2)	0.7888 (2)	0.42043 (16)	0.0203 (4)
H61A	0.2349	0.8537	0.4555	0.030*
H61B	0.2130	0.7696	0.3834	0.030*
H61C	0.3678	0.8288	0.3726	0.030*
S81	0.72769 (6)	0.86689 (6)	0.82975 (4)	0.02445 (14)
O81	0.76558 (17)	0.92433 (16)	0.71601 (12)	0.0258 (4)
C81	0.7307 (3)	0.6842 (2)	0.86096 (19)	0.0298 (5)
H81A	0.6692	0.6513	0.8281	0.045*
H81B	0.6969	0.6416	0.9347	0.045*
H81C	0.8276	0.6597	0.8372	0.045*
C82	0.5417 (3)	0.8747 (3)	0.8733 (2)	0.0361 (6)
H82A	0.5179	0.9709	0.8564	0.054*
H82B	0.5110	0.8308	0.9471	0.054*
H82C	0.4937	0.8266	0.8406	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0197 (9)	0.0135 (8)	0.0175 (9)	0.0018 (7)	−0.0077 (7)	−0.0026 (7)
C2	0.0173 (10)	0.0158 (9)	0.0176 (10)	0.0012 (8)	−0.0056 (8)	−0.0038 (8)
N3	0.0193 (9)	0.0146 (8)	0.0182 (9)	0.0000 (7)	−0.0071 (7)	−0.0023 (7)
C4	0.0173 (10)	0.0143 (9)	0.0169 (10)	0.0024 (8)	−0.0041 (8)	−0.0027 (8)
C5	0.0190 (10)	0.0143 (9)	0.0167 (10)	0.0003 (8)	−0.0052 (8)	−0.0029 (8)
C6	0.0165 (10)	0.0172 (10)	0.0160 (10)	0.0016 (8)	−0.0037 (8)	−0.0049 (8)
N21	0.0245 (10)	0.0142 (8)	0.0227 (10)	−0.0008 (7)	−0.0133 (8)	−0.0015 (7)
N41	0.0191 (9)	0.0135 (8)	0.0182 (9)	0.0002 (7)	−0.0084 (7)	−0.0013 (7)
C42	0.0255 (11)	0.0134 (9)	0.0178 (10)	−0.0005 (8)	−0.0096 (9)	−0.0028 (8)
N43	0.0258 (10)	0.0147 (8)	0.0223 (9)	0.0030 (7)	−0.0109 (8)	−0.0043 (7)
C43A	0.0249 (11)	0.0191 (10)	0.0179 (10)	0.0022 (8)	−0.0089 (9)	−0.0074 (8)
C44	0.0292 (12)	0.0186 (10)	0.0217 (11)	0.0081 (9)	−0.0145 (9)	−0.0079 (9)
C45	0.0248 (12)	0.0295 (12)	0.0259 (12)	0.0112 (9)	−0.0128 (10)	−0.0146 (10)
C46	0.0217 (11)	0.0290 (12)	0.0241 (12)	0.0018 (9)	−0.0068 (9)	−0.0103 (10)
C47	0.0228 (11)	0.0196 (10)	0.0214 (11)	0.0003 (8)	−0.0064 (9)	−0.0051 (9)
C47A	0.0220 (11)	0.0167 (10)	0.0174 (10)	0.0036 (8)	−0.0097 (8)	−0.0057 (8)
C71	0.0231 (11)	0.0175 (10)	0.0172 (10)	−0.0003 (8)	−0.0091 (9)	−0.0048 (8)
C72	0.0246 (11)	0.0157 (10)	0.0182 (10)	−0.0011 (8)	−0.0071 (9)	−0.0034 (8)
C73	0.0266 (12)	0.0177 (10)	0.0210 (11)	0.0024 (8)	−0.0090 (9)	−0.0064 (9)
C74	0.0212 (11)	0.0248 (11)	0.0160 (10)	−0.0031 (9)	−0.0055 (8)	−0.0053 (9)
C75	0.0266 (12)	0.0168 (10)	0.0223 (11)	−0.0048 (9)	−0.0088 (9)	−0.0019 (9)
C76	0.0279 (12)	0.0155 (10)	0.0227 (11)	0.0016 (9)	−0.0108 (9)	−0.0029 (8)
N74	0.0271 (10)	0.0243 (10)	0.0211 (10)	−0.0044 (8)	−0.0056 (8)	−0.0062 (8)
O741	0.0274 (9)	0.0307 (9)	0.0418 (11)	0.0026 (7)	−0.0044 (8)	−0.0142 (8)
O742	0.0306 (9)	0.0275 (9)	0.0296 (9)	−0.0085 (7)	−0.0037 (7)	−0.0026 (7)
N51	0.0242 (10)	0.0139 (8)	0.0202 (9)	0.0001 (7)	−0.0072 (8)	−0.0047 (7)
C57	0.0209 (11)	0.0144 (9)	0.0192 (10)	0.0023 (8)	−0.0062 (9)	−0.0032 (8)
C51	0.0176 (10)	0.0151 (9)	0.0185 (10)	0.0006 (8)	−0.0021 (8)	−0.0047 (8)
C52	0.0204 (11)	0.0180 (10)	0.0208 (11)	0.0004 (8)	−0.0062 (9)	−0.0053 (8)
C53	0.0227 (11)	0.0161 (10)	0.0217 (11)	0.0020 (8)	−0.0043 (9)	−0.0025 (8)
C54	0.0197 (11)	0.0148 (10)	0.0222 (11)	−0.0018 (8)	−0.0005 (9)	−0.0060 (8)
C55	0.0222 (11)	0.0202 (10)	0.0240 (11)	−0.0011 (8)	−0.0072 (9)	−0.0086 (9)
C56	0.0235 (11)	0.0164 (10)	0.0211 (11)	0.0015 (8)	−0.0073 (9)	−0.0047 (8)
N54	0.0253 (10)	0.0171 (9)	0.0317 (11)	0.0001 (7)	−0.0028 (8)	−0.0078 (8)
O541	0.0519 (12)	0.0171 (8)	0.0451 (11)	−0.0041 (8)	−0.0189 (9)	0.0051 (8)
O542	0.0340 (10)	0.0223 (8)	0.0384 (10)	−0.0052 (7)	−0.0059 (8)	−0.0143 (8)
O61	0.0255 (8)	0.0135 (7)	0.0210 (8)	0.0011 (6)	−0.0137 (6)	−0.0026 (6)
C61	0.0254 (11)	0.0166 (10)	0.0199 (11)	0.0018 (8)	−0.0127 (9)	−0.0017 (8)
S81	0.0335 (3)	0.0192 (3)	0.0239 (3)	0.0015 (2)	−0.0158 (2)	−0.0045 (2)
O81	0.0261 (9)	0.0257 (8)	0.0239 (8)	−0.0022 (7)	−0.0107 (7)	−0.0018 (7)
C81	0.0421 (15)	0.0194 (11)	0.0292 (13)	0.0045 (10)	−0.0156 (11)	−0.0049 (10)
C82	0.0415 (15)	0.0309 (13)	0.0273 (13)	0.0106 (11)	−0.0014 (11)	−0.0043 (11)

Geometric parameters (Å, º)

N1—C6	1.327 (3)	C75—C76	1.380 (3)
N1—C2	1.358 (3)	C75—H75	0.9500
C2—N21	1.335 (3)	C76—H76	0.9500
C2—N3	1.352 (3)	N74—O741	1.227 (2)
N3—C4	1.325 (3)	N74—O742	1.234 (2)
C4—C5	1.398 (3)	N51—C57	1.282 (3)
C4—N41	1.427 (2)	C57—C51	1.473 (3)
C5—N51	1.397 (2)	C57—H57	0.9500
C5—C6	1.426 (3)	C51—C56	1.397 (3)
C6—O61	1.333 (2)	C51—C52	1.398 (3)
N21—H21A	0.89 (3)	C52—C53	1.383 (3)
N21—H21B	0.86 (3)	C52—H52	0.9500
N41—C42	1.388 (3)	C53—C54	1.384 (3)
N41—C47A	1.398 (3)	C53—H53	0.9500
C42—N43	1.316 (3)	C54—C55	1.386 (3)
C42—C71	1.470 (3)	C54—N54	1.471 (3)
N43—C43A	1.387 (3)	C55—C56	1.385 (3)
C43A—C44	1.398 (3)	C55—H55	0.9500
C43A—C47A	1.404 (3)	C56—H56	0.9500
C44—C45	1.377 (3)	N54—O542	1.226 (2)
C44—H44	0.9500	N54—O541	1.234 (3)
C45—C46	1.409 (3)	O61—C61	1.446 (2)
C45—H45	0.9500	C61—H61A	0.9800
C46—C47	1.380 (3)	C61—H61B	0.9800
C46—H46	0.9500	C61—H61C	0.9800
C47—C47A	1.388 (3)	S81—O81	1.5025 (16)
C47—H47	0.9500	S81—C82	1.779 (3)
C71—C72	1.398 (3)	S81—C81	1.790 (2)
C71—C76	1.401 (3)	C81—H81A	0.9800
C72—C73	1.381 (3)	C81—H81B	0.9800
C72—H72	0.9500	C81—H81C	0.9800
C73—C74	1.388 (3)	C82—H82A	0.9800
C73—H73	0.9500	C82—H82B	0.9800
C74—C75	1.380 (3)	C82—H82C	0.9800
C74—N74	1.467 (3)		
C6—N1—C2	116.51 (17)	C76—C75—H75	120.6
N21—C2—N3	115.97 (18)	C74—C75—H75	120.6
N21—C2—N1	118.54 (18)	C75—C76—C71	120.7 (2)
N3—C2—N1	125.48 (18)	C75—C76—H76	119.7
C4—N3—C2	115.50 (17)	C71—C76—H76	119.7
N3—C4—C5	125.72 (18)	O741—N74—O742	123.6 (2)
N3—C4—N41	113.45 (17)	O741—N74—C74	118.69 (18)
C5—C4—N41	120.70 (17)	O742—N74—C74	117.68 (19)
N51—C5—C4	117.80 (18)	C57—N51—C5	125.60 (18)
N51—C5—C6	129.27 (19)	N51—C57—C51	119.58 (19)

C4—C5—C6	112.93 (18)	N51—C57—H57	120.2
N1—C6—O61	119.53 (18)	C51—C57—H57	120.2
N1—C6—C5	123.71 (19)	C56—C51—C52	119.34 (19)
O61—C6—C5	116.75 (17)	C56—C51—C57	119.24 (18)
C2—N21—H21A	117.7 (16)	C52—C51—C57	121.32 (19)
C2—N21—H21B	119.8 (16)	C53—C52—C51	120.4 (2)
H21A—N21—H21B	122 (2)	C53—C52—H52	119.8
C42—N41—C47A	106.25 (16)	C51—C52—H52	119.8
C42—N41—C4	130.80 (17)	C52—C53—C54	118.4 (2)
C47A—N41—C4	121.77 (17)	C52—C53—H53	120.8
N43—C42—N41	112.70 (19)	C54—C53—H53	120.8
N43—C42—C71	121.14 (19)	C53—C54—C55	123.04 (19)
N41—C42—C71	125.67 (18)	C53—C54—N54	118.58 (19)
C42—N43—C43A	105.57 (17)	C55—C54—N54	118.37 (19)
N43—C43A—C44	129.7 (2)	C56—C55—C54	117.6 (2)
N43—C43A—C47A	110.35 (18)	C56—C55—H55	121.2
C44—C43A—C47A	120.0 (2)	C54—C55—H55	121.2
C45—C44—C43A	117.7 (2)	C55—C56—C51	121.1 (2)
C45—C44—H44	121.1	C55—C56—H56	119.4
C43A—C44—H44	121.1	C51—C56—H56	119.4
C44—C45—C46	121.6 (2)	O542—N54—O541	124.11 (19)
C44—C45—H45	119.2	O542—N54—C54	118.45 (19)
C46—C45—H45	119.2	O541—N54—C54	117.44 (19)
C47—C46—C45	121.3 (2)	C6—O61—C61	117.90 (15)
C47—C46—H46	119.4	O61—C61—H61A	109.5
C45—C46—H46	119.4	O61—C61—H61B	109.5
C46—C47—C47A	116.9 (2)	H61A—C61—H61B	109.5
C46—C47—H47	121.5	O61—C61—H61C	109.5
C47A—C47—H47	121.5	H61A—C61—H61C	109.5
C47—C47A—N41	132.44 (19)	H61B—C61—H61C	109.5
C47—C47A—C43A	122.4 (2)	O81—S81—C82	106.67 (11)
N41—C47A—C43A	105.11 (18)	O81—S81—C81	106.62 (10)
C72—C71—C76	118.9 (2)	C82—S81—C81	97.53 (12)
C72—C71—C42	123.44 (19)	S81—C81—H81A	109.5
C76—C71—C42	117.34 (19)	S81—C81—H81B	109.5
C73—C72—C71	120.92 (19)	H81A—C81—H81B	109.5
C73—C72—H72	119.5	S81—C81—H81C	109.5
C71—C72—H72	119.5	H81A—C81—H81C	109.5
C72—C73—C74	118.4 (2)	H81B—C81—H81C	109.5
C72—C73—H73	120.8	S81—C82—H82A	109.5
C74—C73—H73	120.8	S81—C82—H82B	109.5
C75—C74—C73	122.2 (2)	H82A—C82—H82B	109.5
C75—C74—N74	118.73 (19)	S81—C82—H82C	109.5
C73—C74—N74	119.1 (2)	H82A—C82—H82C	109.5
C76—C75—C74	118.9 (2)	H82B—C82—H82C	109.5
C6—N1—C2—N21	177.04 (19)	N43—C43A—C47A—N41	−1.4 (2)
C6—N1—C2—N3	−4.0 (3)	C44—C43A—C47A—N41	178.52 (18)

N21—C2—N3—C4	−177.16 (19)	N43—C42—C71—C72	155.8 (2)
N1—C2—N3—C4	3.8 (3)	N41—C42—C71—C72	−15.6 (3)
C2—N3—C4—C5	−0.3 (3)	N43—C42—C71—C76	−17.9 (3)
C2—N3—C4—N41	175.71 (18)	N41—C42—C71—C76	170.8 (2)
N3—C4—C5—N51	176.8 (2)	C76—C71—C72—C73	−1.4 (3)
N41—C4—C5—N51	1.1 (3)	C42—C71—C72—C73	−174.91 (19)
N3—C4—C5—C6	−2.5 (3)	C71—C72—C73—C74	1.5 (3)
N41—C4—C5—C6	−178.27 (18)	C72—C73—C74—C75	−0.2 (3)
C2—N1—C6—O61	−178.33 (18)	C72—C73—C74—N74	−178.02 (18)
C2—N1—C6—C5	0.6 (3)	C73—C74—C75—C76	−1.2 (3)
N51—C5—C6—N1	−176.9 (2)	N74—C74—C75—C76	176.64 (19)
C4—C5—C6—N1	2.4 (3)	C74—C75—C76—C71	1.3 (3)
N51—C5—C6—O61	2.0 (3)	C72—C71—C76—C75	−0.1 (3)
C4—C5—C6—O61	−178.70 (18)	C42—C71—C76—C75	173.87 (19)
N3—C4—N41—C42	136.2 (2)	C75—C74—N74—O741	176.2 (2)
C5—C4—N41—C42	−47.6 (3)	C73—C74—N74—O741	−5.9 (3)
N3—C4—N41—C47A	−58.1 (3)	C75—C74—N74—O742	−4.6 (3)
C5—C4—N41—C47A	118.1 (2)	C73—C74—N74—O742	173.31 (19)
C47A—N41—C42—N43	−0.9 (2)	C4—C5—N51—C57	177.0 (2)
C4—N41—C42—N43	166.5 (2)	C6—C5—N51—C57	−3.8 (4)
C47A—N41—C42—C71	171.04 (19)	C5—N51—C57—C51	−177.24 (19)
C4—N41—C42—C71	−21.6 (3)	N51—C57—C51—C56	172.2 (2)
N41—C42—N43—C43A	0.1 (2)	N51—C57—C51—C52	−4.3 (3)
C71—C42—N43—C43A	−172.31 (18)	C56—C51—C52—C53	0.1 (3)
C42—N43—C43A—C44	−179.1 (2)	C57—C51—C52—C53	176.5 (2)
C42—N43—C43A—C47A	0.8 (2)	C51—C52—C53—C54	0.1 (3)
N43—C43A—C44—C45	−179.5 (2)	C52—C53—C54—C55	0.0 (3)
C47A—C43A—C44—C45	0.6 (3)	C52—C53—C54—N54	179.67 (19)
C43A—C44—C45—C46	0.7 (3)	C53—C54—C55—C56	−0.2 (3)
C44—C45—C46—C47	−1.2 (3)	N54—C54—C55—C56	−179.93 (19)
C45—C46—C47—C47A	0.4 (3)	C54—C55—C56—C51	0.4 (3)
C46—C47—C47A—N41	−179.0 (2)	C52—C51—C56—C55	−0.3 (3)
C46—C47—C47A—C43A	0.9 (3)	C57—C51—C56—C55	−176.9 (2)
C42—N41—C47A—C47	−178.7 (2)	C53—C54—N54—O542	−158.7 (2)
C4—N41—C47A—C47	12.5 (3)	C55—C54—N54—O542	21.0 (3)
C42—N41—C47A—C43A	1.4 (2)	C53—C54—N54—O541	21.2 (3)
C4—N41—C47A—C43A	−167.45 (18)	C55—C54—N54—O541	−159.1 (2)
N43—C43A—C47A—C47	178.65 (19)	N1—C6—O61—C61	2.6 (3)
C44—C43A—C47A—C47	−1.4 (3)	C5—C6—O61—C61	−176.38 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*A*···O81	0.89 (3)	1.94 (3)	2.826 (3)	173 (2)
N21—H21*B*···N1^{i}	0.86 (3)	2.32 (3)	3.161 (3)	166 (3)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.

(*E*)-4-Methoxy-5-[(4-methylbenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine (V)

Crystal data

$C_{27}H_{24}N_6O$
$M_r = 448.52$
Triclinic, $P\overline{1}$
$a = 10.2203$ (15) Å
$b = 14.821$ (2) Å
$c = 16.594$ (2) Å
$\alpha = 99.616$ (5)°
$\beta = 92.153$ (6)°
$\gamma = 106.083$ (5)°
$V = 2371.9$ (6) Å^3
$Z = 4$
$F(000) = 944$
$D_x = 1.256$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10869 reflections
$\theta = 2.1$–$27.5°$
$\mu = 0.08$ mm^{-1}
$T = 100$ K
Block, yellow
0.25 × 0.22 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.948$, $T_{max} = 0.990$
116861 measured reflections
10869 independent reflections
8351 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -13 \rightarrow 13$
$k = -19 \rightarrow 19$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.127$
$S = 1.06$
10869 reflections
631 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 1.7991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.50690 (15)	0.73334 (10)	0.78308 (9)	0.0235 (3)
C12	0.41049 (17)	0.64788 (12)	0.77283 (10)	0.0220 (3)
N13	0.32676 (14)	0.60663 (10)	0.70368 (9)	0.0221 (3)
C14	0.34780 (16)	0.65291 (11)	0.64107 (10)	0.0194 (3)
C15	0.44595 (17)	0.73959 (11)	0.64190 (10)	0.0196 (3)
C16	0.52296 (17)	0.77636 (12)	0.71918 (10)	0.0216 (3)
N121	0.39417 (17)	0.60051 (12)	0.83545 (10)	0.0292 (4)

H12A	0.336 (2)	0.5409 (16)	0.8253 (13)	0.035*
H12B	0.447 (2)	0.6247 (16)	0.8846 (14)	0.035*
N141	0.26366 (14)	0.60399 (10)	0.56760 (8)	0.0201 (3)
C142	0.18371 (17)	0.63770 (12)	0.51732 (10)	0.0218 (3)
N143	0.14892 (16)	0.58272 (10)	0.44472 (9)	0.0264 (3)
C13A	0.20799 (18)	0.50864 (12)	0.44582 (11)	0.0255 (4)
C144	0.2046 (2)	0.43054 (14)	0.38490 (12)	0.0339 (4)
H144	0.1585	0.4219	0.3322	0.041*
C145	0.2707 (2)	0.36639 (14)	0.40390 (13)	0.0366 (5)
H145	0.2689	0.3124	0.3635	0.044*
C146	0.3402 (2)	0.37834 (14)	0.48092 (13)	0.0346 (4)
H146	0.3840	0.3322	0.4916	0.041*
C147	0.34655 (19)	0.45565 (13)	0.54167 (12)	0.0283 (4)
H147	0.3945	0.4646	0.5939	0.034*
C17A	0.27898 (17)	0.51984 (12)	0.52247 (10)	0.0224 (3)
C171	0.12992 (17)	0.71857 (12)	0.54521 (11)	0.0229 (3)
C172	0.11523 (17)	0.74861 (12)	0.62793 (11)	0.0245 (4)
H172	0.1476	0.7197	0.6685	0.029*
C173	0.05367 (19)	0.82034 (14)	0.65129 (12)	0.0304 (4)
H173	0.0452	0.8402	0.7078	0.036*
C174	0.0039 (2)	0.86388 (14)	0.59347 (13)	0.0331 (4)
C175	0.0179 (2)	0.83314 (13)	0.51137 (13)	0.0338 (4)
H175	−0.0161	0.8613	0.4709	0.041*
C176	0.08036 (19)	0.76234 (13)	0.48710 (12)	0.0293 (4)
H176	0.0896	0.7433	0.4305	0.035*
C177	−0.0609 (3)	0.94266 (17)	0.61941 (17)	0.0509 (6)
H17A	−0.0911	0.9634	0.5707	0.076*
H17B	0.0060	0.9967	0.6546	0.076*
H17C	−0.1397	0.9191	0.6499	0.076*
N151	0.46591 (14)	0.77190 (10)	0.56732 (8)	0.0213 (3)
C157	0.54002 (18)	0.85420 (12)	0.56066 (11)	0.0254 (4)
H157	0.5814	0.8991	0.6089	0.031*
C151	0.56309 (18)	0.88143 (12)	0.48010 (11)	0.0244 (4)
C152	0.51068 (19)	0.81802 (13)	0.40670 (11)	0.0272 (4)
H152	0.4597	0.7540	0.4076	0.033*
C153	0.5329 (2)	0.84816 (14)	0.33271 (11)	0.0303 (4)
H153	0.4983	0.8039	0.2831	0.036*
C154	0.60487 (19)	0.94190 (14)	0.32911 (11)	0.0291 (4)
C155	0.65782 (19)	1.00461 (14)	0.40212 (12)	0.0307 (4)
H155	0.7077	1.0688	0.4011	0.037*
C156	0.63848 (19)	0.97428 (14)	0.47662 (12)	0.0304 (4)
H156	0.6774	1.0177	0.5261	0.037*
C158	0.6233 (2)	0.97494 (18)	0.24782 (13)	0.0430 (5)
H18A	0.5579	1.0105	0.2389	0.064*
H18B	0.6074	0.9193	0.2035	0.064*
H18C	0.7167	1.0164	0.2484	0.064*
O161	0.62184 (13)	0.85958 (9)	0.72573 (8)	0.0297 (3)
C161	0.7080 (3)	0.89220 (17)	0.80149 (15)	0.0593 (8)

H16A	0.6519	0.9012	0.8471	0.089*
H16B	0.7757	0.9530	0.7993	0.089*
H16C	0.7549	0.8446	0.8097	0.089*
N21	−0.01055 (14)	0.29156 (10)	0.72415 (8)	0.0215 (3)
C22	0.09026 (17)	0.37465 (12)	0.73092 (10)	0.0221 (3)
N23	0.20532 (15)	0.40024 (10)	0.78293 (9)	0.0233 (3)
C24	0.22094 (17)	0.33451 (12)	0.82525 (10)	0.0214 (3)
C25	0.12972 (17)	0.24398 (12)	0.82054 (10)	0.0215 (3)
C26	0.00910 (17)	0.22931 (12)	0.76795 (10)	0.0207 (3)
N221	0.07508 (17)	0.43676 (11)	0.68423 (10)	0.0269 (3)
H22A	0.142 (2)	0.4857 (16)	0.6866 (13)	0.032*
H22B	0.005 (2)	0.4226 (15)	0.6455 (13)	0.032*
N241	0.34341 (14)	0.36271 (10)	0.87881 (9)	0.0217 (3)
C242	0.35539 (17)	0.35562 (12)	0.96090 (10)	0.0203 (3)
N243	0.48056 (15)	0.36006 (11)	0.98711 (9)	0.0258 (3)
C23A	0.55648 (18)	0.37021 (12)	0.91968 (11)	0.0247 (4)
C244	0.69400 (19)	0.37809 (14)	0.91154 (13)	0.0341 (4)
H244	0.7523	0.3751	0.9564	0.041*
C245	0.7435 (2)	0.39028 (15)	0.83693 (14)	0.0390 (5)
H245	0.8374	0.3961	0.8308	0.047*
C246	0.6604 (2)	0.39430 (14)	0.77043 (14)	0.0384 (5)
H246	0.6986	0.4025	0.7199	0.046*
C247	0.5228 (2)	0.38660 (14)	0.77602 (12)	0.0313 (4)
H247	0.4655	0.3897	0.7308	0.038*
C27A	0.47344 (17)	0.37415 (12)	0.85153 (11)	0.0239 (4)
C271	0.24296 (17)	0.35167 (12)	1.01368 (10)	0.0234 (4)
C272	0.13865 (18)	0.39101 (12)	0.99731 (11)	0.0249 (4)
H272	0.1381	0.4188	0.9498	0.030*
C273	0.03565 (19)	0.39044 (13)	1.04892 (12)	0.0296 (4)
H273	−0.0357	0.4166	1.0359	0.036*
C274	0.0355 (2)	0.35166 (14)	1.12032 (12)	0.0318 (4)
C275	0.1422 (2)	0.31406 (14)	1.13722 (11)	0.0317 (4)
H275	0.1449	0.2884	1.1858	0.038*
C276	0.24413 (19)	0.31326 (13)	1.08489 (11)	0.0279 (4)
H276	0.3151	0.2865	1.0974	0.033*
C277	−0.0781 (2)	0.34854 (18)	1.17540 (13)	0.0440 (5)
H27A	−0.0398	0.3804	1.2313	0.066*
H27B	−0.1285	0.2819	1.1756	0.066*
H27C	−0.1403	0.3813	1.1553	0.066*
N251	0.16943 (15)	0.18153 (10)	0.86418 (9)	0.0232 (3)
C257	0.09527 (19)	0.10021 (13)	0.87222 (12)	0.0301 (4)
H257	0.0035	0.0778	0.8484	0.036*
C251	0.1503 (2)	0.04022 (13)	0.91830 (11)	0.0279 (4)
C252	0.28094 (19)	0.07247 (13)	0.95805 (11)	0.0280 (4)
H252	0.3371	0.1348	0.9558	0.034*
C253	0.3303 (2)	0.01453 (16)	1.00106 (13)	0.0363 (5)
H253	0.4204	0.0376	1.0275	0.044*
C254	0.2505 (2)	−0.07688 (15)	1.00643 (12)	0.0360 (5)

C255	0.1206 (3)	−0.10839 (14)	0.96754 (14)	0.0449 (6)
H255	0.0641	−0.1704	0.9705	0.054*
C256	0.0706 (2)	−0.05067 (15)	0.92392 (14)	0.0465 (6)
H256	−0.0196	−0.0739	0.8976	0.056*
C258	0.3051 (3)	−0.14011 (18)	1.05342 (16)	0.0529 (6)
H28A	0.3455	−0.1810	1.0158	0.079*
H28B	0.2301	−0.1800	1.0782	0.079*
H28C	0.3750	−0.1001	1.0966	0.079*
O261	−0.08904 (12)	0.14627 (8)	0.76290 (8)	0.0260 (3)
C261	−0.21247 (19)	0.13513 (14)	0.71211 (13)	0.0360 (5)
H26A	−0.1912	0.1357	0.6551	0.054*
H26B	−0.2508	0.1878	0.7314	0.054*
H26C	−0.2792	0.0743	0.7154	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0238 (7)	0.0226 (7)	0.0221 (7)	0.0029 (6)	−0.0059 (6)	0.0071 (6)
C12	0.0211 (8)	0.0220 (8)	0.0214 (8)	0.0032 (6)	−0.0034 (6)	0.0065 (7)
N13	0.0223 (7)	0.0214 (7)	0.0207 (7)	0.0023 (6)	−0.0053 (5)	0.0070 (6)
C14	0.0197 (8)	0.0199 (8)	0.0183 (8)	0.0057 (6)	−0.0037 (6)	0.0038 (6)
C15	0.0202 (8)	0.0193 (8)	0.0195 (8)	0.0054 (6)	−0.0020 (6)	0.0057 (6)
C16	0.0198 (8)	0.0182 (8)	0.0252 (8)	0.0022 (6)	−0.0039 (6)	0.0061 (6)
N121	0.0322 (9)	0.0265 (8)	0.0228 (8)	−0.0031 (7)	−0.0098 (6)	0.0099 (6)
N141	0.0223 (7)	0.0172 (7)	0.0192 (7)	0.0033 (5)	−0.0052 (5)	0.0044 (5)
C142	0.0229 (8)	0.0197 (8)	0.0204 (8)	0.0012 (6)	−0.0058 (6)	0.0076 (6)
N143	0.0294 (8)	0.0236 (7)	0.0230 (7)	0.0047 (6)	−0.0076 (6)	0.0025 (6)
C13A	0.0262 (9)	0.0223 (8)	0.0247 (9)	0.0030 (7)	−0.0035 (7)	0.0038 (7)
C144	0.0368 (11)	0.0293 (10)	0.0288 (10)	0.0050 (8)	−0.0067 (8)	−0.0035 (8)
C145	0.0371 (11)	0.0287 (10)	0.0383 (11)	0.0079 (8)	0.0001 (9)	−0.0059 (8)
C146	0.0326 (10)	0.0253 (9)	0.0458 (12)	0.0113 (8)	−0.0003 (9)	0.0020 (8)
C147	0.0268 (9)	0.0251 (9)	0.0321 (10)	0.0074 (7)	−0.0033 (7)	0.0047 (7)
C17A	0.0215 (8)	0.0189 (8)	0.0238 (8)	0.0020 (6)	−0.0014 (7)	0.0035 (6)
C171	0.0208 (8)	0.0196 (8)	0.0258 (9)	0.0015 (6)	−0.0051 (7)	0.0061 (7)
C172	0.0217 (8)	0.0259 (9)	0.0273 (9)	0.0051 (7)	0.0011 (7)	0.0115 (7)
C173	0.0277 (9)	0.0301 (10)	0.0348 (10)	0.0071 (8)	0.0087 (8)	0.0107 (8)
C174	0.0275 (10)	0.0260 (9)	0.0490 (12)	0.0087 (8)	0.0056 (8)	0.0130 (9)
C175	0.0338 (10)	0.0263 (9)	0.0424 (11)	0.0081 (8)	−0.0100 (9)	0.0131 (8)
C176	0.0332 (10)	0.0240 (9)	0.0283 (9)	0.0042 (7)	−0.0083 (8)	0.0079 (7)
C177	0.0528 (14)	0.0456 (13)	0.0697 (17)	0.0306 (12)	0.0206 (12)	0.0219 (12)
N151	0.0212 (7)	0.0225 (7)	0.0209 (7)	0.0060 (6)	−0.0006 (5)	0.0075 (6)
C157	0.0281 (9)	0.0235 (9)	0.0217 (8)	0.0029 (7)	−0.0029 (7)	0.0048 (7)
C151	0.0246 (9)	0.0251 (9)	0.0233 (8)	0.0049 (7)	0.0006 (7)	0.0080 (7)
C152	0.0296 (9)	0.0243 (9)	0.0265 (9)	0.0061 (7)	−0.0014 (7)	0.0053 (7)
C153	0.0326 (10)	0.0326 (10)	0.0241 (9)	0.0091 (8)	0.0003 (7)	0.0017 (7)
C154	0.0259 (9)	0.0379 (10)	0.0272 (9)	0.0103 (8)	0.0057 (7)	0.0132 (8)
C155	0.0280 (9)	0.0309 (10)	0.0312 (10)	0.0014 (8)	0.0023 (8)	0.0118 (8)
C156	0.0310 (10)	0.0284 (9)	0.0271 (9)	−0.0002 (8)	−0.0025 (8)	0.0076 (8)

C158	0.0463 (13)	0.0550 (14)	0.0287 (10)	0.0108 (11)	0.0076 (9)	0.0161 (10)
O161	0.0294 (7)	0.0245 (6)	0.0271 (7)	−0.0062 (5)	−0.0116 (5)	0.0091 (5)
C161	0.0632 (16)	0.0437 (13)	0.0466 (13)	−0.0270 (12)	−0.0380 (12)	0.0216 (11)
N21	0.0217 (7)	0.0210 (7)	0.0193 (7)	0.0018 (6)	−0.0041 (5)	0.0053 (5)
C22	0.0230 (8)	0.0209 (8)	0.0197 (8)	0.0019 (7)	−0.0046 (6)	0.0055 (6)
N23	0.0239 (7)	0.0231 (7)	0.0207 (7)	0.0021 (6)	−0.0042 (6)	0.0073 (6)
C24	0.0210 (8)	0.0256 (8)	0.0168 (8)	0.0052 (7)	−0.0025 (6)	0.0051 (6)
C25	0.0235 (8)	0.0213 (8)	0.0178 (8)	0.0028 (7)	−0.0041 (6)	0.0058 (6)
C26	0.0221 (8)	0.0190 (8)	0.0179 (8)	0.0008 (6)	−0.0025 (6)	0.0043 (6)
N221	0.0269 (8)	0.0225 (7)	0.0268 (8)	−0.0018 (6)	−0.0113 (6)	0.0106 (6)
N241	0.0187 (7)	0.0240 (7)	0.0213 (7)	0.0029 (6)	−0.0024 (5)	0.0073 (6)
C242	0.0201 (8)	0.0201 (8)	0.0188 (8)	0.0025 (6)	−0.0053 (6)	0.0057 (6)
N243	0.0228 (7)	0.0250 (7)	0.0281 (8)	0.0044 (6)	−0.0051 (6)	0.0065 (6)
C23A	0.0218 (8)	0.0204 (8)	0.0279 (9)	0.0018 (7)	−0.0026 (7)	0.0017 (7)
C244	0.0213 (9)	0.0304 (10)	0.0484 (12)	0.0054 (8)	−0.0038 (8)	0.0061 (9)
C245	0.0241 (10)	0.0332 (11)	0.0536 (13)	0.0030 (8)	0.0074 (9)	−0.0007 (9)
C246	0.0332 (11)	0.0313 (10)	0.0442 (12)	−0.0005 (8)	0.0131 (9)	0.0032 (9)
C247	0.0322 (10)	0.0298 (10)	0.0295 (10)	0.0033 (8)	0.0045 (8)	0.0080 (8)
C27A	0.0185 (8)	0.0201 (8)	0.0296 (9)	0.0006 (6)	−0.0002 (7)	0.0039 (7)
C271	0.0234 (8)	0.0211 (8)	0.0218 (8)	0.0014 (7)	−0.0035 (7)	0.0030 (7)
C272	0.0277 (9)	0.0237 (8)	0.0222 (8)	0.0057 (7)	−0.0001 (7)	0.0044 (7)
C273	0.0264 (9)	0.0276 (9)	0.0344 (10)	0.0085 (7)	0.0003 (8)	0.0037 (8)
C274	0.0322 (10)	0.0298 (10)	0.0288 (10)	0.0035 (8)	0.0055 (8)	0.0011 (8)
C275	0.0348 (10)	0.0360 (10)	0.0217 (9)	0.0037 (8)	−0.0011 (7)	0.0100 (8)
C276	0.0253 (9)	0.0310 (9)	0.0243 (9)	0.0029 (7)	−0.0047 (7)	0.0069 (7)
C277	0.0442 (13)	0.0528 (14)	0.0354 (11)	0.0134 (11)	0.0122 (10)	0.0078 (10)
N251	0.0266 (8)	0.0216 (7)	0.0204 (7)	0.0047 (6)	−0.0033 (6)	0.0063 (6)
C257	0.0289 (9)	0.0251 (9)	0.0322 (10)	−0.0004 (7)	−0.0110 (8)	0.0106 (8)
C251	0.0354 (10)	0.0228 (9)	0.0242 (9)	0.0056 (7)	−0.0053 (7)	0.0070 (7)
C252	0.0283 (9)	0.0295 (9)	0.0289 (9)	0.0090 (8)	0.0035 (7)	0.0113 (8)
C253	0.0295 (10)	0.0488 (12)	0.0385 (11)	0.0172 (9)	0.0038 (8)	0.0202 (10)
C254	0.0515 (13)	0.0351 (11)	0.0309 (10)	0.0235 (10)	0.0054 (9)	0.0129 (8)
C255	0.0671 (15)	0.0212 (9)	0.0412 (12)	0.0039 (10)	−0.0149 (11)	0.0104 (9)
C256	0.0533 (14)	0.0268 (10)	0.0488 (13)	−0.0061 (9)	−0.0275 (11)	0.0146 (9)
C258	0.0633 (16)	0.0527 (14)	0.0596 (15)	0.0304 (13)	0.0072 (12)	0.0332 (12)
O261	0.0216 (6)	0.0222 (6)	0.0295 (7)	−0.0028 (5)	−0.0095 (5)	0.0099 (5)
C261	0.0261 (10)	0.0295 (10)	0.0451 (12)	−0.0059 (8)	−0.0173 (8)	0.0148 (9)

Geometric parameters (Å, º)

N11—C16	1.319 (2)	N21—C26	1.317 (2)
N11—C12	1.352 (2)	N21—C22	1.354 (2)
C12—N121	1.339 (2)	C22—N221	1.334 (2)
C12—N13	1.351 (2)	C22—N23	1.356 (2)
N13—C14	1.330 (2)	N23—C24	1.332 (2)
C14—C15	1.391 (2)	C24—C25	1.394 (2)
C14—N141	1.425 (2)	C24—N241	1.426 (2)
C15—N151	1.402 (2)	C25—N251	1.399 (2)

C15—C16	1.419 (2)	C25—C26	1.427 (2)
C16—O161	1.346 (2)	C26—O261	1.3427 (19)
N121—H12A	0.90 (2)	N221—H22A	0.84 (2)
N121—H12B	0.92 (2)	N221—H22B	0.90 (2)
N141—C142	1.389 (2)	N241—C242	1.387 (2)
N141—C17A	1.395 (2)	N241—C27A	1.395 (2)
C142—N143	1.312 (2)	C242—N243	1.316 (2)
C142—C171	1.467 (2)	C242—C271	1.465 (2)
N143—C13A	1.394 (2)	N243—C23A	1.391 (2)
C13A—C144	1.396 (3)	C23A—C244	1.391 (3)
C13A—C17A	1.402 (2)	C23A—C27A	1.407 (2)
C144—C145	1.378 (3)	C244—C245	1.376 (3)
C144—H144	0.9500	C244—H244	0.9500
C145—C146	1.399 (3)	C245—C246	1.386 (3)
C145—H145	0.9500	C245—H245	0.9500
C146—C147	1.379 (3)	C246—C247	1.387 (3)
C146—H146	0.9500	C246—H246	0.9500
C147—C17A	1.390 (2)	C247—C27A	1.390 (3)
C147—H147	0.9500	C247—H247	0.9500
C171—C172	1.398 (2)	C271—C272	1.388 (2)
C171—C176	1.401 (2)	C271—C276	1.396 (2)
C172—C173	1.388 (3)	C272—C273	1.381 (3)
C172—H172	0.9500	C272—H272	0.9500
C173—C174	1.396 (3)	C273—C274	1.400 (3)
C173—H173	0.9500	C273—H273	0.9500
C174—C175	1.388 (3)	C274—C275	1.394 (3)
C174—C177	1.506 (3)	C274—C277	1.500 (3)
C175—C176	1.385 (3)	C275—C276	1.383 (3)
C175—H175	0.9500	C275—H275	0.9500
C176—H176	0.9500	C276—H276	0.9500
C177—H17A	0.9800	C277—H27A	0.9800
C177—H17B	0.9800	C277—H27B	0.9800
C177—H17C	0.9800	C277—H27C	0.9800
N151—C157	1.270 (2)	N251—C257	1.265 (2)
C157—C151	1.469 (2)	C257—C251	1.471 (2)
C157—H157	0.9500	C257—H257	0.9500
C151—C156	1.390 (2)	C251—C252	1.386 (3)
C151—C152	1.394 (2)	C251—C256	1.388 (3)
C152—C153	1.381 (3)	C252—C253	1.385 (3)
C152—H152	0.9500	C252—H252	0.9500
C153—C154	1.393 (3)	C253—C254	1.394 (3)
C153—H153	0.9500	C253—H253	0.9500
C154—C155	1.386 (3)	C254—C255	1.374 (3)
C154—C158	1.511 (3)	C254—C258	1.516 (3)
C155—C156	1.387 (3)	C255—C256	1.392 (3)
C155—H155	0.9500	C255—H255	0.9500
C156—H156	0.9500	C256—H256	0.9500
C158—H18A	0.9800	C258—H28A	0.9800

C158—H18B	0.9800	C258—H28B	0.9800
C158—H18C	0.9800	C258—H28C	0.9800
O161—C161	1.438 (2)	O261—C261	1.445 (2)
C161—H16A	0.9800	C261—H26A	0.9800
C161—H16B	0.9800	C261—H26B	0.9800
C161—H16C	0.9800	C261—H26C	0.9800

C16—N11—C12	116.72 (14)	C26—N21—C22	117.16 (14)
N121—C12—N13	116.87 (15)	N221—C22—N21	118.06 (15)
N121—C12—N11	118.51 (15)	N221—C22—N23	117.31 (15)
N13—C12—N11	124.62 (15)	N21—C22—N23	124.62 (15)
C14—N13—C12	116.05 (14)	C24—N23—C22	115.76 (14)
N13—C14—C15	125.55 (15)	N23—C24—C25	125.61 (15)
N13—C14—N141	114.32 (14)	N23—C24—N241	114.69 (14)
C15—C14—N141	120.07 (14)	C25—C24—N241	119.70 (14)
C14—C15—N151	117.75 (14)	C24—C25—N251	117.35 (14)
C14—C15—C16	112.34 (14)	C24—C25—C26	112.54 (14)
N151—C15—C16	129.44 (15)	N251—C25—C26	130.07 (15)
N11—C16—O161	118.58 (15)	N21—C26—O261	118.71 (14)
N11—C16—C15	124.63 (15)	N21—C26—C25	124.04 (15)
O161—C16—C15	116.75 (14)	O261—C26—C25	117.25 (14)
C12—N121—H12A	116.8 (14)	C22—N221—H22A	116.1 (15)
C12—N121—H12B	121.6 (14)	C22—N221—H22B	122.1 (14)
H12A—N121—H12B	121.2 (19)	H22A—N221—H22B	120.9 (19)
C142—N141—C17A	106.79 (13)	C242—N241—C27A	106.55 (13)
C142—N141—C14	129.12 (14)	C242—N241—C24	127.15 (14)
C17A—N141—C14	122.06 (13)	C27A—N241—C24	123.66 (14)
N143—C142—N141	112.34 (15)	N243—C242—N241	112.63 (15)
N143—C142—C171	123.40 (15)	N243—C242—C271	124.05 (15)
N141—C142—C171	123.85 (15)	N241—C242—C271	123.13 (14)
C142—N143—C13A	105.71 (14)	C242—N243—C23A	105.55 (14)
N143—C13A—C144	130.21 (16)	N243—C23A—C244	130.79 (17)
N143—C13A—C17A	110.21 (15)	N243—C23A—C27A	110.18 (15)
C144—C13A—C17A	119.58 (17)	C244—C23A—C27A	119.02 (17)
C145—C144—C13A	117.65 (18)	C245—C244—C23A	118.35 (19)
C145—C144—H144	121.2	C245—C244—H244	120.8
C13A—C144—H144	121.2	C23A—C244—H244	120.8
C144—C145—C146	122.00 (18)	C244—C245—C246	121.92 (19)
C144—C145—H145	119.0	C244—C245—H245	119.0
C146—C145—H145	119.0	C246—C245—H245	119.0
C147—C146—C145	121.37 (18)	C245—C246—C247	121.5 (2)
C147—C146—H146	119.3	C245—C246—H246	119.3
C145—C146—H146	119.3	C247—C246—H246	119.3
C146—C147—C17A	116.47 (17)	C246—C247—C27A	116.30 (18)
C146—C147—H147	121.8	C246—C247—H247	121.8
C17A—C147—H147	121.8	C27A—C247—H247	121.8
C147—C17A—N141	132.13 (16)	C247—C27A—N241	132.04 (16)
C147—C17A—C13A	122.93 (16)	C247—C27A—C23A	122.92 (17)

N141—C17A—C13A	104.94 (14)	N241—C27A—C23A	105.04 (15)
C172—C171—C176	118.23 (17)	C272—C271—C276	118.67 (17)
C172—C171—C142	122.14 (15)	C272—C271—C242	121.31 (15)
C176—C171—C142	119.39 (16)	C276—C271—C242	119.90 (16)
C173—C172—C171	120.46 (16)	C273—C272—C271	121.17 (16)
C173—C172—H172	119.8	C273—C272—H272	119.4
C171—C172—H172	119.8	C271—C272—H272	119.4
C172—C173—C174	121.40 (18)	C272—C273—C274	120.61 (17)
C172—C173—H173	119.3	C272—C273—H273	119.7
C174—C173—H173	119.3	C274—C273—H273	119.7
C175—C174—C173	117.80 (18)	C275—C274—C273	117.86 (18)
C175—C174—C177	121.12 (19)	C275—C274—C277	121.32 (18)
C173—C174—C177	121.1 (2)	C273—C274—C277	120.80 (19)
C176—C175—C174	121.52 (17)	C276—C275—C274	121.53 (17)
C176—C175—H175	119.2	C276—C275—H275	119.2
C174—C175—H175	119.2	C274—C275—H275	119.2
C175—C176—C171	120.58 (18)	C275—C276—C271	120.13 (17)
C175—C176—H176	119.7	C275—C276—H276	119.9
C171—C176—H176	119.7	C271—C276—H276	119.9
C174—C177—H17A	109.5	C274—C277—H27A	109.5
C174—C177—H17B	109.5	C274—C277—H27B	109.5
H17A—C177—H17B	109.5	H27A—C277—H27B	109.5
C174—C177—H17C	109.5	C274—C277—H27C	109.5
H17A—C177—H17C	109.5	H27A—C277—H27C	109.5
H17B—C177—H17C	109.5	H27B—C277—H27C	109.5
C157—N151—C15	124.28 (15)	C257—N251—C25	125.63 (15)
N151—C157—C151	121.44 (16)	N251—C257—C251	120.39 (16)
N151—C157—H157	119.3	N251—C257—H257	119.8
C151—C157—H157	119.3	C251—C257—H257	119.8
C156—C151—C152	118.54 (16)	C252—C251—C256	118.14 (17)
C156—C151—C157	118.95 (16)	C252—C251—C257	121.64 (16)
C152—C151—C157	122.50 (16)	C256—C251—C257	120.22 (17)
C153—C152—C151	120.03 (17)	C253—C252—C251	120.47 (18)
C153—C152—H152	120.0	C253—C252—H252	119.8
C151—C152—H152	120.0	C251—C252—H252	119.8
C152—C153—C154	121.56 (18)	C252—C253—C254	121.40 (19)
C152—C153—H153	119.2	C252—C253—H253	119.3
C154—C153—H153	119.2	C254—C253—H253	119.3
C155—C154—C153	118.31 (17)	C255—C254—C253	118.02 (17)
C155—C154—C158	120.72 (18)	C255—C254—C258	120.8 (2)
C153—C154—C158	120.97 (18)	C253—C254—C258	121.2 (2)
C154—C155—C156	120.45 (17)	C254—C255—C256	120.87 (19)
C154—C155—H155	119.8	C254—C255—H255	119.6
C156—C155—H155	119.8	C256—C255—H255	119.6
C155—C156—C151	121.08 (18)	C251—C256—C255	121.1 (2)
C155—C156—H156	119.5	C251—C256—H256	119.4
C151—C156—H156	119.5	C255—C256—H256	119.4
C154—C158—H18A	109.5	C254—C258—H28A	109.5

C154—C158—H18B	109.5	C254—C258—H28B	109.5
H18A—C158—H18B	109.5	H28A—C258—H28B	109.5
C154—C158—H18C	109.5	C254—C258—H28C	109.5
H18A—C158—H18C	109.5	H28A—C258—H28C	109.5
H18B—C158—H18C	109.5	H28B—C258—H28C	109.5
C16—O161—C161	116.64 (14)	C26—O261—C261	116.05 (13)
O161—C161—H16A	109.5	O261—C261—H26A	109.5
O161—C161—H16B	109.5	O261—C261—H26B	109.5
H16A—C161—H16B	109.5	H26A—C261—H26B	109.5
O161—C161—H16C	109.5	O261—C261—H26C	109.5
H16A—C161—H16C	109.5	H26A—C261—H26C	109.5
H16B—C161—H16C	109.5	H26B—C261—H26C	109.5
C16—N11—C12—N121	177.86 (17)	C26—N21—C22—N221	176.98 (17)
C16—N11—C12—N13	−3.2 (3)	C26—N21—C22—N23	−4.1 (3)
N121—C12—N13—C14	−177.56 (16)	N221—C22—N23—C24	−176.37 (16)
N11—C12—N13—C14	3.5 (3)	N21—C22—N23—C24	4.7 (3)
C12—N13—C14—C15	−1.1 (3)	C22—N23—C24—C25	−0.4 (3)
C12—N13—C14—N141	175.97 (15)	C22—N23—C24—N241	179.33 (15)
N13—C14—C15—N151	171.78 (16)	N23—C24—C25—N251	174.37 (16)
N141—C14—C15—N151	−5.2 (2)	N241—C24—C25—N251	−5.4 (2)
N13—C14—C15—C16	−1.1 (2)	N23—C24—C25—C26	−3.7 (3)
N141—C14—C15—C16	−178.06 (15)	N241—C24—C25—C26	176.54 (15)
C12—N11—C16—O161	−177.18 (16)	C22—N21—C26—O261	179.78 (15)
C12—N11—C16—C15	0.5 (3)	C22—N21—C26—C25	−0.8 (3)
C14—C15—C16—N11	1.4 (3)	C24—C25—C26—N21	4.4 (3)
N151—C15—C16—N11	−170.40 (17)	N251—C25—C26—N21	−173.41 (17)
C14—C15—C16—O161	179.17 (15)	C24—C25—C26—O261	−176.22 (15)
N151—C15—C16—O161	7.4 (3)	N251—C25—C26—O261	6.0 (3)
N13—C14—N141—C142	129.84 (18)	N23—C24—N241—C242	130.72 (18)
C15—C14—N141—C142	−52.9 (2)	C25—C24—N241—C242	−49.5 (3)
N13—C14—N141—C17A	−68.6 (2)	N23—C24—N241—C27A	−70.2 (2)
C15—C14—N141—C17A	108.71 (18)	C25—C24—N241—C27A	109.57 (19)
C17A—N141—C142—N143	−0.61 (19)	C27A—N241—C242—N243	−1.01 (19)
C14—N141—C142—N143	163.14 (16)	C24—N241—C242—N243	160.92 (16)
C17A—N141—C142—C171	172.24 (15)	C27A—N241—C242—C271	174.24 (15)
C14—N141—C142—C171	−24.0 (3)	C24—N241—C242—C271	−23.8 (3)
N141—C142—N143—C13A	−0.1 (2)	N241—C242—N243—C23A	−0.25 (19)
C171—C142—N143—C13A	−172.96 (16)	C271—C242—N243—C23A	−175.45 (15)
C142—N143—C13A—C144	−179.7 (2)	C242—N243—C23A—C244	−179.77 (19)
C142—N143—C13A—C17A	0.7 (2)	C242—N243—C23A—C27A	1.43 (19)
N143—C13A—C144—C145	−178.55 (19)	N243—C23A—C244—C245	−178.18 (18)
C17A—C13A—C144—C145	1.0 (3)	C27A—C23A—C244—C245	0.5 (3)
C13A—C144—C145—C146	−0.6 (3)	C23A—C244—C245—C246	−0.4 (3)
C144—C145—C146—C147	−0.3 (3)	C244—C245—C246—C247	0.3 (3)
C145—C146—C147—C17A	0.8 (3)	C245—C246—C247—C27A	−0.4 (3)
C146—C147—C17A—N141	179.90 (18)	C246—C247—C27A—N241	−179.03 (18)
C146—C147—C17A—C13A	−0.3 (3)	C246—C247—C27A—C23A	0.6 (3)

C142—N141—C17A—C147	−179.17 (19)	C242—N241—C27A—C247	−178.57 (19)
C14—N141—C17A—C147	15.7 (3)	C24—N241—C27A—C247	18.7 (3)
C142—N141—C17A—C13A	1.01 (18)	C242—N241—C27A—C23A	1.78 (18)
C14—N141—C17A—C13A	−164.14 (15)	C24—N241—C27A—C23A	−160.94 (15)
N143—C13A—C17A—C147	179.06 (16)	N243—C23A—C27A—C247	178.30 (16)
C144—C13A—C17A—C147	−0.6 (3)	C244—C23A—C27A—C247	−0.7 (3)
N143—C13A—C17A—N141	−1.10 (19)	N243—C23A—C27A—N241	−2.02 (19)
C144—C13A—C17A—N141	179.26 (17)	C244—C23A—C27A—N241	179.02 (16)
N143—C142—C171—C172	150.51 (17)	N243—C242—C271—C272	149.00 (17)
N141—C142—C171—C172	−21.6 (3)	N241—C242—C271—C272	−25.7 (2)
N143—C142—C171—C176	−23.8 (3)	N243—C242—C271—C276	−26.8 (3)
N141—C142—C171—C176	164.11 (16)	N241—C242—C271—C276	158.47 (16)
C176—C171—C172—C173	−0.3 (3)	C276—C271—C272—C273	−1.6 (3)
C142—C171—C172—C173	−174.73 (16)	C242—C271—C272—C273	−177.48 (16)
C171—C172—C173—C174	0.5 (3)	C271—C272—C273—C274	1.3 (3)
C172—C173—C174—C175	0.0 (3)	C272—C273—C274—C275	0.0 (3)
C172—C173—C174—C177	−179.11 (19)	C272—C273—C274—C277	−178.37 (19)
C173—C174—C175—C176	−0.7 (3)	C273—C274—C275—C276	−1.1 (3)
C177—C174—C175—C176	178.4 (2)	C277—C274—C275—C276	177.32 (19)
C174—C175—C176—C171	0.8 (3)	C274—C275—C276—C271	0.8 (3)
C172—C171—C176—C175	−0.3 (3)	C272—C271—C276—C275	0.6 (3)
C142—C171—C176—C175	174.23 (17)	C242—C271—C276—C275	176.49 (17)
C14—C15—N151—C157	171.48 (17)	C24—C25—N251—C257	173.80 (18)
C16—C15—N151—C157	−17.1 (3)	C26—C25—N251—C257	−8.5 (3)
C15—N151—C157—C151	176.28 (16)	C25—N251—C257—C251	178.60 (17)
N151—C157—C151—C156	176.25 (18)	N251—C257—C251—C252	3.8 (3)
N151—C157—C151—C152	−2.9 (3)	N251—C257—C251—C256	−177.1 (2)
C156—C151—C152—C153	−0.6 (3)	C256—C251—C252—C253	0.9 (3)
C157—C151—C152—C153	178.49 (17)	C257—C251—C252—C253	179.97 (19)
C151—C152—C153—C154	−1.3 (3)	C251—C252—C253—C254	−0.6 (3)
C152—C153—C154—C155	1.8 (3)	C252—C253—C254—C255	0.1 (3)
C152—C153—C154—C158	−177.42 (19)	C252—C253—C254—C258	179.8 (2)
C153—C154—C155—C156	−0.3 (3)	C253—C254—C255—C256	0.2 (4)
C158—C154—C155—C156	178.87 (19)	C258—C254—C255—C256	−179.6 (2)
C154—C155—C156—C151	−1.6 (3)	C252—C251—C256—C255	−0.6 (4)
C152—C151—C156—C155	2.1 (3)	C257—C251—C256—C255	−179.7 (2)
C157—C151—C156—C155	−177.08 (18)	C254—C255—C256—C251	0.1 (4)
N11—C16—O161—C161	3.5 (3)	N21—C26—O261—C261	−3.2 (2)
C15—C16—O161—C161	−174.4 (2)	C25—C26—O261—C261	177.32 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N121—H12*A*···N23	0.90 (2)	2.13 (2)	3.022 (2)	171.5 (19)
N121—H12*B*···N243^{i}	0.92 (2)	2.18 (2)	3.062 (2)	160 (2)
N221—H22*A*···N13	0.84 (2)	2.19 (2)	3.023 (2)	173.4 (19)
N221—H22*B*···N143^{ii}	0.90 (2)	2.11 (2)	2.994 (2)	169 (2)
C146—H146···N151^{iii}	0.95	2.57	3.390 (3)	145

C176—H176···N21^{ii}	0.95	2.58	3.464 (2)	154
C155—H155···*Cg*1^{iv}	0.95	2.60	3.465 (2)	151
C255—H255···*Cg*2^{v}	0.95	2.87	3.784 (2)	163

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

(*E*)-4-Methoxy-5-[(4-chlorobenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine (VIa)

Crystal data

$C_{26}H_{21}ClN_6O$
M_r = 468.94
Triclinic, $P\overline{1}$
a = 10.2298 (7) Å
b = 14.8344 (9) Å
c = 16.5321 (10) Å
α = 99.672 (2)°
β = 92.038 (2)°
γ = 106.704 (2)°
V = 2359.4 (3) Å^3
Z = 4
F(000) = 976
D_x = 1.320 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 10821 reflections
θ = 2.2–27.5°
μ = 0.19 mm^{-1}
T = 100 K
Block, yellow
0.18 × 0.13 × 0.11 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.926, T_{max} = 0.979
126128 measured reflections
10821 independent reflections
8240 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −13→13
k = −19→19
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.164
S = 1.04
10821 reflections
629 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 2.9524P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.5093 (2)	0.73559 (13)	0.78242 (12)	0.0295 (4)
C12	0.4121 (2)	0.64984 (16)	0.77238 (13)	0.0277 (5)

N13	0.32778 (19)	0.60766 (13)	0.70269 (11)	0.0261 (4)
C14	0.3497 (2)	0.65317 (15)	0.63966 (13)	0.0232 (4)
C15	0.4491 (2)	0.73995 (15)	0.64043 (13)	0.0235 (4)
C16	0.5266 (2)	0.77777 (15)	0.71803 (14)	0.0266 (4)
N121	0.3950 (2)	0.60280 (16)	0.83527 (13)	0.0364 (5)
H12A	0.340 (3)	0.546 (2)	0.8284 (19)	0.044*
H12B	0.451 (3)	0.624 (2)	0.881 (2)	0.044*
N141	0.26435 (18)	0.60411 (12)	0.56618 (11)	0.0236 (4)
C142	0.1848 (2)	0.63769 (15)	0.51649 (13)	0.0248 (4)
N143	0.1480 (2)	0.58204 (14)	0.44356 (12)	0.0315 (4)
C13A	0.2064 (2)	0.50812 (16)	0.44425 (14)	0.0300 (5)
C144	0.2013 (3)	0.42957 (19)	0.38317 (17)	0.0417 (6)
H144	0.1546	0.4205	0.3304	0.050*
C145	0.2665 (3)	0.36576 (19)	0.40220 (18)	0.0448 (7)
H145	0.2639	0.3116	0.3616	0.054*
C146	0.3361 (3)	0.37778 (19)	0.47877 (18)	0.0427 (6)
H146	0.3788	0.3314	0.4893	0.051*
C147	0.3447 (3)	0.45575 (17)	0.53994 (16)	0.0335 (5)
H147	0.3934	0.4650	0.5922	0.040*
C17A	0.2781 (2)	0.51979 (15)	0.52076 (14)	0.0264 (4)
C171	0.1326 (2)	0.71894 (15)	0.54459 (14)	0.0273 (5)
C172	0.1197 (2)	0.74991 (17)	0.62741 (15)	0.0299 (5)
H172	0.1519	0.7212	0.6680	0.036*
C173	0.0601 (3)	0.82244 (19)	0.65130 (17)	0.0381 (6)
H173	0.0534	0.8432	0.7081	0.046*
C174	0.0100 (3)	0.86500 (19)	0.59310 (19)	0.0411 (6)
C175	0.0223 (3)	0.83344 (18)	0.51088 (18)	0.0416 (6)
H175	−0.0118	0.8612	0.4702	0.050*
C176	0.0833 (3)	0.76219 (17)	0.48645 (16)	0.0356 (6)
H176	0.0915	0.7426	0.4296	0.043*
C177	−0.0521 (4)	0.9448 (3)	0.6201 (3)	0.0657 (10)
H17A	−0.0838	0.9651	0.5715	0.099*
H17B	0.0169	0.9992	0.6543	0.099*
H17C	−0.1299	0.9220	0.6520	0.099*
N151	0.46905 (18)	0.77248 (13)	0.56570 (11)	0.0248 (4)
C157	0.5450 (3)	0.85499 (17)	0.55919 (14)	0.0336 (5)
H157	0.5893	0.8995	0.6074	0.040*
C151	0.5654 (2)	0.88248 (17)	0.47830 (14)	0.0315 (5)
C152	0.5123 (3)	0.81888 (18)	0.40506 (15)	0.0332 (5)
H152	0.4634	0.7542	0.4061	0.040*
C153	0.5305 (3)	0.84949 (19)	0.33039 (15)	0.0372 (6)
H153	0.4964	0.8057	0.2801	0.045*
C154	0.5988 (3)	0.9443 (2)	0.32981 (15)	0.0357 (5)
C155	0.6541 (3)	1.0089 (2)	0.40099 (16)	0.0394 (6)
H155	0.7022	1.0736	0.3995	0.047*
C156	0.6376 (3)	0.97672 (19)	0.47544 (16)	0.0404 (6)
H156	0.6765	1.0200	0.5253	0.049*
Cl14	0.61513 (8)	0.98406 (6)	0.23613 (4)	0.0535 (2)

O161	0.62680 (18)	0.86076 (12)	0.72414 (10)	0.0371 (4)
C161	0.7048 (4)	0.8990 (2)	0.8030 (2)	0.0758 (13)
H16A	0.6432	0.9094	0.8452	0.114*
H16B	0.7732	0.9600	0.8005	0.114*
H16C	0.7512	0.8535	0.8168	0.114*
N21	−0.01076 (19)	0.29134 (13)	0.72456 (11)	0.0264 (4)
C22	0.0905 (2)	0.37540 (16)	0.73184 (13)	0.0276 (5)
N23	0.2062 (2)	0.40131 (13)	0.78392 (11)	0.0282 (4)
C24	0.2215 (2)	0.33553 (16)	0.82574 (13)	0.0256 (4)
C25	0.1298 (2)	0.24447 (15)	0.82064 (13)	0.0255 (4)
C26	0.0090 (2)	0.22894 (15)	0.76805 (13)	0.0250 (4)
N221	0.0750 (2)	0.43796 (15)	0.68594 (13)	0.0327 (5)
H22A	0.137 (3)	0.484 (2)	0.6879 (18)	0.039*
H22B	0.009 (3)	0.424 (2)	0.6519 (19)	0.039*
N241	0.34453 (19)	0.36443 (13)	0.87967 (11)	0.0262 (4)
C242	0.3554 (2)	0.35658 (15)	0.96201 (13)	0.0252 (4)
N243	0.4798 (2)	0.35935 (14)	0.98802 (12)	0.0320 (4)
C23A	0.5563 (2)	0.36898 (17)	0.92006 (15)	0.0321 (5)
C244	0.6928 (3)	0.37458 (19)	0.9110 (2)	0.0435 (6)
H244	0.7501	0.3694	0.9554	0.052*
C245	0.7429 (3)	0.3877 (2)	0.8368 (2)	0.0522 (8)
H245	0.8365	0.3926	0.8306	0.063*
C246	0.6619 (3)	0.3940 (2)	0.7710 (2)	0.0486 (7)
H246	0.7010	0.4033	0.7206	0.058*
C247	0.5242 (3)	0.38698 (19)	0.77644 (16)	0.0391 (6)
H247	0.4676	0.3906	0.7310	0.047*
C27A	0.4740 (2)	0.37435 (16)	0.85209 (15)	0.0303 (5)
C271	0.2428 (2)	0.35166 (16)	1.01447 (14)	0.0289 (5)
C272	0.1396 (2)	0.39084 (16)	0.99886 (14)	0.0302 (5)
H272	0.1404	0.4201	0.9519	0.036*
C273	0.0352 (3)	0.38865 (18)	1.04979 (17)	0.0386 (6)
H273	−0.0356	0.4150	1.0368	0.046*
C274	0.0327 (3)	0.3479 (2)	1.12034 (17)	0.0413 (6)
C275	0.1385 (3)	0.3107 (2)	1.13687 (15)	0.0411 (6)
H275	0.1400	0.2840	1.1851	0.049*
C276	0.2419 (3)	0.31137 (18)	1.08503 (14)	0.0341 (5)
H276	0.3123	0.2844	1.0974	0.041*
C277	−0.0822 (4)	0.3425 (3)	1.1745 (2)	0.0579 (8)
H27A	−0.0463	0.3802	1.2294	0.087*
H27B	−0.1258	0.2756	1.1787	0.087*
H27C	−0.1499	0.3682	1.1508	0.087*
N251	0.1688 (2)	0.18238 (13)	0.86433 (11)	0.0271 (4)
C257	0.0960 (3)	0.09961 (17)	0.87066 (15)	0.0358 (6)
H257	0.0050	0.0754	0.8451	0.043*
C251	0.1510 (3)	0.04070 (17)	0.91710 (14)	0.0326 (5)
C252	0.2832 (3)	0.07450 (18)	0.95538 (15)	0.0337 (5)
H252	0.3394	0.1366	0.9514	0.040*
C253	0.3335 (3)	0.0185 (2)	0.99919 (18)	0.0423 (6)

H253	0.4244	0.0415	1.0249	0.051*
C254	0.2507 (3)	−0.07150 (19)	1.00544 (16)	0.0395 (6)
C255	0.1211 (3)	−0.10679 (19)	0.96876 (18)	0.0506 (8)
H255	0.0654	−0.1688	0.9734	0.061*
C256	0.0713 (3)	−0.04997 (19)	0.9240 (2)	0.0538 (9)
H256	−0.0192	−0.0740	0.8978	0.065*
Cl24	0.31387 (9)	−0.14186 (6)	1.06192 (5)	0.0601 (2)
O261	−0.08870 (16)	0.14522 (11)	0.76251 (10)	0.0321 (4)
C261	−0.2151 (3)	0.13423 (19)	0.71455 (18)	0.0435 (7)
H26A	−0.1977	0.1342	0.6566	0.065*
H26B	−0.2511	0.1875	0.7352	0.065*
H26C	−0.2822	0.0735	0.7193	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0323 (10)	0.0255 (9)	0.0273 (9)	0.0019 (8)	−0.0106 (8)	0.0102 (8)
C12	0.0291 (11)	0.0273 (11)	0.0250 (11)	0.0036 (9)	−0.0073 (9)	0.0107 (9)
N13	0.0280 (9)	0.0239 (9)	0.0240 (9)	0.0019 (7)	−0.0078 (7)	0.0101 (7)
C14	0.0233 (10)	0.0225 (10)	0.0236 (10)	0.0057 (8)	−0.0060 (8)	0.0072 (8)
C15	0.0247 (10)	0.0216 (10)	0.0241 (10)	0.0049 (8)	−0.0038 (8)	0.0085 (8)
C16	0.0270 (11)	0.0209 (10)	0.0294 (11)	0.0025 (8)	−0.0070 (9)	0.0078 (8)
N121	0.0422 (12)	0.0314 (11)	0.0278 (10)	−0.0049 (9)	−0.0160 (9)	0.0158 (9)
N141	0.0273 (9)	0.0193 (8)	0.0220 (8)	0.0028 (7)	−0.0068 (7)	0.0070 (7)
C142	0.0279 (11)	0.0212 (10)	0.0227 (10)	0.0016 (8)	−0.0078 (8)	0.0088 (8)
N143	0.0375 (11)	0.0261 (10)	0.0267 (10)	0.0044 (8)	−0.0121 (8)	0.0055 (8)
C13A	0.0321 (12)	0.0251 (11)	0.0285 (11)	0.0032 (9)	−0.0055 (9)	0.0045 (9)
C144	0.0479 (15)	0.0337 (13)	0.0349 (13)	0.0056 (11)	−0.0082 (11)	−0.0028 (11)
C145	0.0470 (16)	0.0310 (13)	0.0494 (16)	0.0092 (12)	−0.0005 (13)	−0.0068 (12)
C146	0.0422 (15)	0.0306 (13)	0.0557 (17)	0.0143 (11)	−0.0017 (12)	0.0039 (12)
C147	0.0336 (12)	0.0263 (11)	0.0390 (13)	0.0076 (9)	−0.0055 (10)	0.0064 (10)
C17A	0.0260 (11)	0.0220 (10)	0.0275 (11)	0.0012 (8)	−0.0031 (8)	0.0061 (8)
C171	0.0269 (11)	0.0218 (10)	0.0308 (11)	0.0015 (8)	−0.0065 (9)	0.0103 (9)
C172	0.0282 (11)	0.0306 (11)	0.0330 (12)	0.0071 (9)	0.0024 (9)	0.0148 (9)
C173	0.0365 (13)	0.0372 (13)	0.0452 (15)	0.0125 (11)	0.0133 (11)	0.0159 (11)
C174	0.0379 (14)	0.0328 (13)	0.0587 (17)	0.0135 (11)	0.0089 (12)	0.0191 (12)
C175	0.0427 (14)	0.0329 (13)	0.0521 (16)	0.0115 (11)	−0.0113 (12)	0.0188 (12)
C176	0.0444 (14)	0.0271 (11)	0.0334 (12)	0.0072 (10)	−0.0122 (10)	0.0097 (10)
C177	0.070 (2)	0.057 (2)	0.094 (3)	0.0417 (18)	0.031 (2)	0.0359 (19)
N151	0.0259 (9)	0.0249 (9)	0.0239 (9)	0.0059 (7)	−0.0024 (7)	0.0097 (7)
C157	0.0403 (13)	0.0285 (12)	0.0266 (11)	0.0009 (10)	−0.0059 (10)	0.0083 (9)
C151	0.0313 (12)	0.0330 (12)	0.0286 (11)	0.0043 (10)	−0.0017 (9)	0.0118 (9)
C152	0.0342 (12)	0.0324 (12)	0.0320 (12)	0.0079 (10)	−0.0001 (10)	0.0078 (10)
C153	0.0404 (14)	0.0433 (14)	0.0284 (12)	0.0133 (11)	0.0001 (10)	0.0074 (10)
C154	0.0349 (13)	0.0484 (15)	0.0297 (12)	0.0139 (11)	0.0064 (10)	0.0200 (11)
C155	0.0378 (14)	0.0399 (14)	0.0381 (14)	0.0015 (11)	0.0023 (11)	0.0186 (11)
C156	0.0449 (15)	0.0354 (13)	0.0323 (13)	−0.0038 (11)	−0.0048 (11)	0.0123 (10)
Cl14	0.0599 (5)	0.0689 (5)	0.0327 (3)	0.0115 (4)	0.0078 (3)	0.0250 (3)

O161	0.0395 (9)	0.0280 (8)	0.0334 (9)	−0.0084 (7)	−0.0169 (7)	0.0133 (7)
C161	0.088 (3)	0.0500 (18)	0.0565 (19)	−0.0356 (17)	−0.0506 (19)	0.0287 (16)
N21	0.0276 (9)	0.0257 (9)	0.0229 (9)	0.0016 (7)	−0.0065 (7)	0.0091 (7)
C22	0.0308 (11)	0.0257 (11)	0.0229 (10)	0.0016 (9)	−0.0076 (9)	0.0094 (8)
N23	0.0304 (10)	0.0268 (9)	0.0248 (9)	0.0020 (8)	−0.0066 (7)	0.0109 (7)
C24	0.0267 (11)	0.0295 (11)	0.0190 (10)	0.0047 (9)	−0.0041 (8)	0.0079 (8)
C25	0.0299 (11)	0.0238 (10)	0.0208 (10)	0.0031 (8)	−0.0061 (8)	0.0089 (8)
C26	0.0277 (11)	0.0229 (10)	0.0208 (10)	0.0015 (8)	−0.0045 (8)	0.0064 (8)
N221	0.0327 (11)	0.0282 (10)	0.0317 (10)	−0.0033 (8)	−0.0160 (9)	0.0159 (8)
N241	0.0247 (9)	0.0273 (9)	0.0244 (9)	0.0026 (7)	−0.0046 (7)	0.0094 (7)
C242	0.0263 (10)	0.0251 (10)	0.0217 (10)	0.0025 (8)	−0.0079 (8)	0.0082 (8)
N243	0.0301 (10)	0.0310 (10)	0.0329 (10)	0.0056 (8)	−0.0089 (8)	0.0092 (8)
C23A	0.0278 (11)	0.0277 (11)	0.0364 (12)	0.0031 (9)	−0.0048 (9)	0.0048 (10)
C244	0.0276 (12)	0.0381 (14)	0.0624 (18)	0.0076 (11)	−0.0061 (12)	0.0088 (13)
C245	0.0324 (14)	0.0437 (16)	0.070 (2)	0.0025 (12)	0.0078 (14)	−0.0026 (15)
C246	0.0403 (15)	0.0429 (15)	0.0545 (17)	0.0007 (12)	0.0153 (13)	0.0055 (13)
C247	0.0393 (14)	0.0391 (14)	0.0356 (13)	0.0040 (11)	0.0046 (11)	0.0107 (11)
C27A	0.0246 (11)	0.0271 (11)	0.0355 (12)	0.0018 (9)	−0.0015 (9)	0.0065 (9)
C271	0.0308 (11)	0.0261 (11)	0.0253 (11)	0.0022 (9)	−0.0061 (9)	0.0056 (9)
C272	0.0353 (12)	0.0289 (11)	0.0255 (11)	0.0081 (9)	−0.0005 (9)	0.0055 (9)
C273	0.0392 (14)	0.0342 (13)	0.0427 (14)	0.0126 (11)	0.0016 (11)	0.0053 (11)
C274	0.0444 (15)	0.0402 (14)	0.0350 (13)	0.0068 (12)	0.0103 (11)	0.0038 (11)
C275	0.0490 (15)	0.0463 (15)	0.0245 (12)	0.0061 (12)	−0.0006 (11)	0.0118 (11)
C276	0.0351 (13)	0.0369 (13)	0.0269 (11)	0.0042 (10)	−0.0063 (9)	0.0098 (10)
C277	0.061 (2)	0.068 (2)	0.0447 (17)	0.0172 (17)	0.0191 (15)	0.0109 (15)
N251	0.0332 (10)	0.0251 (9)	0.0225 (9)	0.0058 (8)	−0.0051 (7)	0.0099 (7)
C257	0.0379 (13)	0.0274 (12)	0.0373 (13)	0.0001 (10)	−0.0152 (10)	0.0136 (10)
C251	0.0429 (14)	0.0257 (11)	0.0280 (11)	0.0075 (10)	−0.0071 (10)	0.0083 (9)
C252	0.0351 (13)	0.0351 (12)	0.0345 (12)	0.0109 (10)	0.0025 (10)	0.0158 (10)
C253	0.0341 (13)	0.0527 (16)	0.0484 (15)	0.0159 (12)	0.0023 (11)	0.0270 (13)
C254	0.0558 (16)	0.0397 (14)	0.0338 (13)	0.0247 (12)	0.0019 (11)	0.0182 (11)
C255	0.073 (2)	0.0254 (12)	0.0481 (16)	0.0057 (13)	−0.0201 (15)	0.0126 (12)
C256	0.0602 (19)	0.0310 (13)	0.0588 (18)	−0.0055 (12)	−0.0335 (15)	0.0182 (13)
Cl24	0.0683 (5)	0.0630 (5)	0.0691 (5)	0.0343 (4)	0.0049 (4)	0.0419 (4)
O261	0.0283 (8)	0.0267 (8)	0.0357 (9)	−0.0027 (6)	−0.0132 (7)	0.0133 (7)
C261	0.0320 (13)	0.0377 (14)	0.0531 (16)	−0.0069 (10)	−0.0200 (12)	0.0225 (12)

Geometric parameters (Å, º)

N11—C16	1.315 (3)	N21—C26	1.318 (3)
N11—C12	1.351 (3)	N21—C22	1.356 (3)
C12—N121	1.336 (3)	C22—N221	1.332 (3)
C12—N13	1.356 (3)	C22—N23	1.358 (3)
N13—C14	1.326 (3)	N23—C24	1.327 (3)
C14—C15	1.390 (3)	C24—C25	1.393 (3)
C14—N141	1.425 (3)	C24—N241	1.430 (3)
C15—N151	1.401 (3)	C25—N251	1.394 (3)
C15—C16	1.421 (3)	C25—C26	1.424 (3)

C16—O161	1.342 (3)	C26—O261	1.338 (3)
N121—H12A	0.85 (3)	N221—H22A	0.79 (3)
N121—H12B	0.89 (3)	N221—H22B	0.82 (3)
N141—C142	1.382 (3)	N241—C242	1.389 (3)
N141—C17A	1.393 (3)	N241—C27A	1.392 (3)
C142—N143	1.316 (3)	C242—N243	1.316 (3)
C142—C171	1.467 (3)	C242—C271	1.459 (3)
N143—C13A	1.393 (3)	N243—C23A	1.396 (3)
C13A—C144	1.395 (3)	C23A—C244	1.390 (4)
C13A—C17A	1.398 (3)	C23A—C27A	1.407 (3)
C144—C145	1.374 (4)	C244—C245	1.368 (4)
C144—H144	0.9500	C244—H244	0.9500
C145—C146	1.389 (4)	C245—C246	1.377 (5)
C145—H145	0.9500	C245—H245	0.9500
C146—C147	1.382 (4)	C246—C247	1.390 (4)
C146—H146	0.9500	C246—H246	0.9500
C147—C17A	1.387 (3)	C247—C27A	1.390 (3)
C147—H147	0.9500	C247—H247	0.9500
C171—C172	1.393 (3)	C271—C272	1.379 (3)
C171—C176	1.396 (3)	C271—C276	1.396 (3)
C172—C173	1.392 (3)	C272—C273	1.380 (4)
C172—H172	0.9500	C272—H272	0.9500
C173—C174	1.393 (4)	C273—C274	1.398 (4)
C173—H173	0.9500	C273—H273	0.9500
C174—C175	1.384 (4)	C274—C275	1.387 (4)
C174—C177	1.510 (4)	C274—C277	1.495 (4)
C175—C176	1.386 (4)	C275—C276	1.384 (4)
C175—H175	0.9500	C275—H275	0.9500
C176—H176	0.9500	C276—H276	0.9500
C177—H17A	0.9800	C277—H27A	0.9800
C177—H17B	0.9800	C277—H27B	0.9800
C177—H17C	0.9800	C277—H27C	0.9800
N151—C157	1.272 (3)	N251—C257	1.263 (3)
C157—C151	1.467 (3)	C257—C251	1.465 (3)
C157—H157	0.9500	C257—H257	0.9500
C151—C152	1.388 (3)	C251—C256	1.384 (4)
C151—C156	1.391 (3)	C251—C252	1.387 (3)
C152—C153	1.386 (3)	C252—C253	1.379 (3)
C152—H152	0.9500	C252—H252	0.9500
C153—C154	1.381 (4)	C253—C254	1.383 (4)
C153—H153	0.9500	C253—H253	0.9500
C154—C155	1.373 (4)	C254—C255	1.355 (4)
C154—Cl14	1.744 (2)	C254—Cl24	1.747 (2)
C155—C156	1.392 (3)	C255—C256	1.395 (4)
C155—H155	0.9500	C255—H255	0.9500
C156—H156	0.9500	C256—H256	0.9500
O161—C161	1.440 (3)	O261—C261	1.446 (3)
C161—H16A	0.9800	C261—H26A	0.9800

C161—H16B	0.9800	C261—H26B	0.9800
C161—H16C	0.9800	C261—H26C	0.9800
C16—N11—C12	116.73 (18)	C26—N21—C22	117.12 (18)
N121—C12—N11	118.6 (2)	N221—C22—N21	118.11 (19)
N121—C12—N13	116.8 (2)	N221—C22—N23	117.3 (2)
N11—C12—N13	124.63 (19)	N21—C22—N23	124.56 (19)
C14—N13—C12	116.07 (18)	C24—N23—C22	115.75 (19)
N13—C14—C15	125.32 (19)	N23—C24—C25	125.62 (19)
N13—C14—N141	114.47 (18)	N23—C24—N241	114.59 (19)
C15—C14—N141	120.19 (18)	C25—C24—N241	119.78 (18)
C14—C15—N151	117.77 (18)	C24—C25—N251	117.34 (19)
C14—C15—C16	112.69 (18)	C24—C25—C26	112.81 (18)
N151—C15—C16	129.17 (19)	N251—C25—C26	129.83 (19)
N11—C16—O161	118.61 (19)	N21—C26—O261	118.82 (18)
N11—C16—C15	124.46 (19)	N21—C26—C25	123.90 (19)
O161—C16—C15	116.89 (19)	O261—C26—C25	117.28 (18)
C12—N121—H12A	120 (2)	C22—N221—H22A	117 (2)
C12—N121—H12B	122 (2)	C22—N221—H22B	120 (2)
H12A—N121—H12B	117 (3)	H22A—N221—H22B	122 (3)
C142—N141—C17A	106.95 (17)	C242—N241—C27A	106.73 (18)
C142—N141—C14	129.10 (18)	C242—N241—C24	126.70 (18)
C17A—N141—C14	121.96 (18)	C27A—N241—C24	123.42 (19)
N143—C142—N141	112.19 (19)	N243—C242—N241	112.5 (2)
N143—C142—C171	123.28 (19)	N243—C242—C271	124.16 (19)
N141—C142—C171	124.13 (19)	N241—C242—C271	123.27 (19)
C142—N143—C13A	105.69 (18)	C242—N243—C23A	105.59 (19)
N143—C13A—C144	130.3 (2)	C244—C23A—N243	131.1 (2)
N143—C13A—C17A	110.1 (2)	C244—C23A—C27A	118.9 (2)
C144—C13A—C17A	119.6 (2)	N243—C23A—C27A	110.0 (2)
C145—C144—C13A	117.5 (2)	C245—C244—C23A	118.6 (3)
C145—C144—H144	121.2	C245—C244—H244	120.7
C13A—C144—H144	121.2	C23A—C244—H244	120.7
C144—C145—C146	122.2 (2)	C244—C245—C246	122.0 (3)
C144—C145—H145	118.9	C244—C245—H245	119.0
C146—C145—H145	118.9	C246—C245—H245	119.0
C147—C146—C145	121.5 (2)	C245—C246—C247	121.5 (3)
C147—C146—H146	119.3	C245—C246—H246	119.2
C145—C146—H146	119.3	C247—C246—H246	119.2
C146—C147—C17A	116.2 (2)	C246—C247—C27A	116.2 (3)
C146—C147—H147	121.9	C246—C247—H247	121.9
C17A—C147—H147	121.9	C27A—C247—H247	121.9
C147—C17A—N141	132.0 (2)	C247—C27A—N241	132.1 (2)
C147—C17A—C13A	123.0 (2)	C247—C27A—C23A	122.7 (2)
N141—C17A—C13A	105.05 (19)	N241—C27A—C23A	105.2 (2)
C172—C171—C176	118.1 (2)	C272—C271—C276	118.4 (2)
C172—C171—C142	122.30 (19)	C272—C271—C242	121.6 (2)
C176—C171—C142	119.4 (2)	C276—C271—C242	120.0 (2)

C173—C172—C171	120.7 (2)	C271—C272—C273	121.6 (2)
C173—C172—H172	119.6	C271—C272—H272	119.2
C171—C172—H172	119.6	C273—C272—H272	119.2
C172—C173—C174	121.0 (3)	C272—C273—C274	120.5 (2)
C172—C173—H173	119.5	C272—C273—H273	119.7
C174—C173—H173	119.5	C274—C273—H273	119.7
C175—C174—C173	118.0 (2)	C275—C274—C273	117.6 (2)
C175—C174—C177	121.6 (3)	C275—C274—C277	121.4 (3)
C173—C174—C177	120.4 (3)	C273—C274—C277	120.9 (3)
C174—C175—C176	121.5 (2)	C276—C275—C274	121.8 (2)
C174—C175—H175	119.2	C276—C275—H275	119.1
C176—C175—H175	119.2	C274—C275—H275	119.1
C175—C176—C171	120.6 (2)	C275—C276—C271	120.0 (2)
C175—C176—H176	119.7	C275—C276—H276	120.0
C171—C176—H176	119.7	C271—C276—H276	120.0
C174—C177—H17A	109.5	C274—C277—H27A	109.5
C174—C177—H17B	109.5	C274—C277—H27B	109.5
H17A—C177—H17B	109.5	H27A—C277—H27B	109.5
C174—C177—H17C	109.5	C274—C277—H27C	109.5
H17A—C177—H17C	109.5	H27A—C277—H27C	109.5
H17B—C177—H17C	109.5	H27B—C277—H27C	109.5
C157—N151—C15	124.16 (19)	C257—N251—C25	126.0 (2)
N151—C157—C151	121.0 (2)	N251—C257—C251	120.4 (2)
N151—C157—H157	119.5	N251—C257—H257	119.8
C151—C157—H157	119.5	C251—C257—H257	119.8
C152—C151—C156	119.0 (2)	C256—C251—C252	118.5 (2)
C152—C151—C157	122.6 (2)	C256—C251—C257	120.5 (2)
C156—C151—C157	118.4 (2)	C252—C251—C257	121.0 (2)
C153—C152—C151	120.2 (2)	C253—C252—C251	120.4 (2)
C153—C152—H152	119.9	C253—C252—H252	119.8
C151—C152—H152	119.9	C251—C252—H252	119.8
C154—C153—C152	119.3 (2)	C252—C253—C254	119.5 (2)
C154—C153—H153	120.3	C252—C253—H253	120.2
C152—C153—H153	120.3	C254—C253—H253	120.2
C155—C154—C153	122.0 (2)	C255—C254—C253	121.6 (2)
C155—C154—Cl14	118.7 (2)	C255—C254—Cl24	118.9 (2)
C153—C154—Cl14	119.3 (2)	C253—C254—Cl24	119.5 (2)
C154—C155—C156	118.1 (2)	C254—C255—C256	118.5 (3)
C154—C155—H155	121.0	C254—C255—H255	120.8
C156—C155—H155	121.0	C256—C255—H255	120.8
C151—C156—C155	121.3 (2)	C251—C256—C255	121.4 (3)
C151—C156—H156	119.3	C251—C256—H256	119.3
C155—C156—H156	119.3	C255—C256—H256	119.3
C16—O161—C161	116.70 (19)	C26—O261—C261	116.35 (17)
O161—C161—H16A	109.5	O261—C261—H26A	109.5
O161—C161—H16B	109.5	O261—C261—H26B	109.5
H16A—C161—H16B	109.5	H26A—C261—H26B	109.5
O161—C161—H16C	109.5	O261—C261—H26C	109.5

H16A—C161—H16C	109.5	H26A—C261—H26C	109.5
H16B—C161—H16C	109.5	H26B—C261—H26C	109.5
C16—N11—C12—N121	177.3 (2)	C26—N21—C22—N221	177.4 (2)
C16—N11—C12—N13	−3.7 (4)	C26—N21—C22—N23	−3.8 (4)
N121—C12—N13—C14	−177.2 (2)	N221—C22—N23—C24	−176.8 (2)
N11—C12—N13—C14	3.8 (4)	N21—C22—N23—C24	4.4 (4)
C12—N13—C14—C15	−1.2 (3)	C22—N23—C24—C25	−0.4 (4)
C12—N13—C14—N141	176.64 (19)	C22—N23—C24—N241	179.6 (2)
N13—C14—C15—N151	172.6 (2)	N23—C24—C25—N251	174.9 (2)
N141—C14—C15—N151	−5.1 (3)	N241—C24—C25—N251	−5.1 (3)
N13—C14—C15—C16	−1.0 (3)	N23—C24—C25—C26	−3.5 (3)
N141—C14—C15—C16	−178.74 (19)	N241—C24—C25—C26	176.5 (2)
C12—N11—C16—O161	−176.7 (2)	C22—N21—C26—O261	179.9 (2)
C12—N11—C16—C15	1.1 (4)	C22—N21—C26—C25	−0.8 (3)
C14—C15—C16—N11	1.1 (3)	C24—C25—C26—N21	4.1 (3)
N151—C15—C16—N11	−171.7 (2)	N251—C25—C26—N21	−174.1 (2)
C14—C15—C16—O161	178.9 (2)	C24—C25—C26—O261	−176.6 (2)
N151—C15—C16—O161	6.1 (4)	N251—C25—C26—O261	5.2 (4)
N13—C14—N141—C142	129.0 (2)	N23—C24—N241—C242	130.7 (2)
C15—C14—N141—C142	−53.0 (3)	C25—C24—N241—C242	−49.3 (3)
N13—C14—N141—C17A	−69.3 (3)	N23—C24—N241—C27A	−72.1 (3)
C15—C14—N141—C17A	108.7 (2)	C25—C24—N241—C27A	107.9 (3)
C17A—N141—C142—N143	−0.4 (3)	C27A—N241—C242—N243	−0.9 (3)
C14—N141—C142—N143	163.5 (2)	C24—N241—C242—N243	159.4 (2)
C17A—N141—C142—C171	172.5 (2)	C27A—N241—C242—C271	175.6 (2)
C14—N141—C142—C171	−23.7 (3)	C24—N241—C242—C271	−24.1 (3)
N141—C142—N143—C13A	−0.4 (3)	N241—C242—N243—C23A	−0.3 (3)
C171—C142—N143—C13A	−173.3 (2)	C271—C242—N243—C23A	−176.9 (2)
C142—N143—C13A—C144	−179.7 (3)	C242—N243—C23A—C244	−179.2 (3)
C142—N143—C13A—C17A	1.0 (3)	C242—N243—C23A—C27A	1.5 (3)
N143—C13A—C144—C145	−178.2 (3)	N243—C23A—C244—C245	−177.5 (3)
C17A—C13A—C144—C145	1.0 (4)	C27A—C23A—C244—C245	1.7 (4)
C13A—C144—C145—C146	−0.3 (4)	C23A—C244—C245—C246	−1.0 (4)
C144—C145—C146—C147	−0.7 (5)	C244—C245—C246—C247	−0.2 (5)
C145—C146—C147—C17A	1.0 (4)	C245—C246—C247—C27A	0.6 (4)
C146—C147—C17A—N141	179.6 (2)	C246—C247—C27A—N241	−179.8 (3)
C146—C147—C17A—C13A	−0.4 (4)	C246—C247—C27A—C23A	0.1 (4)
C142—N141—C17A—C147	−179.0 (2)	C242—N241—C27A—C247	−178.4 (3)
C14—N141—C17A—C147	15.8 (4)	C24—N241—C27A—C247	20.6 (4)
C142—N141—C17A—C13A	1.0 (2)	C242—N241—C27A—C23A	1.7 (2)
C14—N141—C17A—C13A	−164.29 (19)	C24—N241—C27A—C23A	−159.4 (2)
N143—C13A—C17A—C147	178.7 (2)	C244—C23A—C27A—C247	−1.3 (4)
C144—C13A—C17A—C147	−0.7 (4)	N243—C23A—C27A—C247	178.1 (2)
N143—C13A—C17A—N141	−1.3 (3)	C244—C23A—C27A—N241	178.6 (2)
C144—C13A—C17A—N141	179.4 (2)	N243—C23A—C27A—N241	−2.0 (3)
N143—C142—C171—C172	150.7 (2)	N243—C242—C271—C272	150.1 (2)
N141—C142—C171—C172	−21.4 (3)	N241—C242—C271—C272	−26.0 (3)

N143—C142—C171—C176	−23.8 (3)	N243—C242—C271—C276	−26.2 (3)
N141—C142—C171—C176	164.1 (2)	N241—C242—C271—C276	157.7 (2)
C176—C171—C172—C173	−0.5 (3)	C276—C271—C272—C273	−1.7 (3)
C142—C171—C172—C173	−175.0 (2)	C242—C271—C272—C273	−178.1 (2)
C171—C172—C173—C174	0.9 (4)	C271—C272—C273—C274	1.3 (4)
C172—C173—C174—C175	−0.5 (4)	C272—C273—C274—C275	0.3 (4)
C172—C173—C174—C177	−178.9 (3)	C272—C273—C274—C277	−178.1 (3)
C173—C174—C175—C176	−0.5 (4)	C273—C274—C275—C276	−1.4 (4)
C177—C174—C175—C176	178.0 (3)	C277—C274—C275—C276	176.9 (3)
C174—C175—C176—C171	0.9 (4)	C274—C275—C276—C271	1.0 (4)
C172—C171—C176—C175	−0.5 (4)	C272—C271—C276—C275	0.6 (3)
C142—C171—C176—C175	174.3 (2)	C242—C271—C276—C275	177.0 (2)
C14—C15—N151—C157	171.8 (2)	C24—C25—N251—C257	175.7 (2)
C16—C15—N151—C157	−15.8 (4)	C26—C25—N251—C257	−6.2 (4)
C15—N151—C157—C151	177.4 (2)	C25—N251—C257—C251	178.5 (2)
N151—C157—C151—C152	−4.9 (4)	N251—C257—C251—C256	−180.0 (3)
N151—C157—C151—C156	173.3 (3)	N251—C257—C251—C252	0.4 (4)
C156—C151—C152—C153	−0.5 (4)	C256—C251—C252—C253	0.1 (4)
C157—C151—C152—C153	177.8 (2)	C257—C251—C252—C253	179.7 (3)
C151—C152—C153—C154	−1.7 (4)	C251—C252—C253—C254	−0.6 (4)
C152—C153—C154—C155	2.5 (4)	C252—C253—C254—C255	0.7 (4)
C152—C153—C154—Cl14	−177.2 (2)	C252—C253—C254—Cl24	−179.3 (2)
C153—C154—C155—C156	−1.2 (4)	C253—C254—C255—C256	−0.2 (5)
Cl14—C154—C155—C156	178.6 (2)	Cl24—C254—C255—C256	179.8 (3)
C152—C151—C156—C155	1.9 (4)	C252—C251—C256—C255	0.4 (5)
C157—C151—C156—C155	−176.4 (3)	C257—C251—C256—C255	−179.2 (3)
C154—C155—C156—C151	−1.1 (4)	C254—C255—C256—C251	−0.3 (5)
N11—C16—O161—C161	−2.2 (4)	N21—C26—O261—C261	−5.9 (3)
C15—C16—O161—C161	179.8 (3)	C25—C26—O261—C261	174.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N121—H12*A*···N23	0.86 (3)	2.18 (3)	3.016 (3)	168 (3)
N121—H12*B*···N243^{i}	0.89 (3)	2.21 (3)	3.043 (3)	158 (3)
N221—H22*A*···N13	0.78 (3)	2.23 (3)	3.012 (3)	173 (3)
N221—H22*B*···N143^{ii}	0.82 (3)	2.18 (3)	2.988 (3)	168 (3)
C146—H146···N151^{iii}	0.95	2.58	3.404 (4)	146
C176—H176···N21^{ii}	0.95	2.57	3.449 (3)	154
C155—H155···*Cg*1^{iv}	0.95	2.55	3.391 (3)	147
C255—H255···*Cg*2^{v}	0.95	2.85	3.754 (3)	160

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

(*E*)-4-Methoxy-5-[(4-chlorobenzylidene)amino]-6-[2-(4-methylphenyl)-1*H*-benzo[*d*]imidazol-1-yl]pyrimidin-2-amine (VIb)

Crystal data

$C_{26}H_{21}ClN_6O$
$M_r = 468.94$
Triclinic, $P\overline{1}$
$a = 9.6520$ (8) Å
$b = 9.7408$ (10) Å
$c = 14.1445$ (12) Å
$\alpha = 98.183$ (4)°
$\beta = 104.638$ (3)°
$\gamma = 90.059$ (4)°
$V = 1272.6$ (2) Å^3
$Z = 2$
$F(000) = 488$
$D_x = 1.224$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5851 reflections
$\theta = 2.1$–$27.5°$
$\mu = 0.18$ mm^{-1}
$T = 100$ K
Plate, yellow
0.12 × 0.09 × 0.08 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: INCOATEC high brilliance microfocus sealed tube
Multilayer mirror monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
$T_{min} = 0.916$, $T_{max} = 0.986$
60695 measured reflections
5849 independent reflections
4653 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.115$
$S = 1.03$
5849 reflections
315 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 1.4226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.03612 (15)	0.68107 (16)	0.74950 (11)	0.0171 (3)
C2	0.03059 (18)	0.61398 (18)	0.65740 (13)	0.0160 (3)
N3	0.14638 (15)	0.57773 (15)	0.62317 (10)	0.0153 (3)
C4	0.27199 (17)	0.59837 (17)	0.69062 (12)	0.0140 (3)
C5	0.29105 (17)	0.65195 (18)	0.78952 (12)	0.0153 (3)
C6	0.16281 (18)	0.69867 (18)	0.81240 (12)	0.0163 (3)
N21	−0.09903 (16)	0.58655 (18)	0.59557 (12)	0.0213 (3)

H21A	−0.107 (2)	0.542 (2)	0.5380 (18)	0.026*
H21B	−0.179 (2)	0.600 (2)	0.6164 (16)	0.026*
N41	0.39498 (14)	0.56386 (15)	0.65574 (10)	0.0137 (3)
C42	0.52077 (17)	0.64373 (18)	0.67274 (12)	0.0141 (3)
N43	0.62314 (15)	0.57292 (15)	0.64527 (10)	0.0156 (3)
C43A	0.56511 (18)	0.43932 (18)	0.60946 (12)	0.0157 (3)
C44	0.6288 (2)	0.32167 (19)	0.57234 (13)	0.0198 (4)
H44	0.7251	0.3253	0.5674	0.024*
C45	0.5454 (2)	0.1998 (2)	0.54317 (14)	0.0233 (4)
H45	0.5855	0.1182	0.5176	0.028*
C46	0.4037 (2)	0.19366 (19)	0.55027 (14)	0.0219 (4)
H46	0.3503	0.1079	0.5294	0.026*
C47	0.33895 (19)	0.30883 (18)	0.58677 (13)	0.0176 (4)
H47	0.2430	0.3045	0.5922	0.021*
C47A	0.42253 (18)	0.43100 (18)	0.61490 (12)	0.0149 (3)
C71	0.53513 (18)	0.79222 (18)	0.71213 (12)	0.0156 (3)
C72	0.42003 (18)	0.87996 (18)	0.69585 (12)	0.0162 (3)
H72	0.3282	0.8437	0.6579	0.019*
C73	0.4387 (2)	1.01970 (19)	0.73461 (13)	0.0193 (4)
H73	0.3588	1.0777	0.7236	0.023*
C74	0.5723 (2)	1.07686 (19)	0.78945 (13)	0.0216 (4)
C75	0.6867 (2)	0.9883 (2)	0.80476 (15)	0.0263 (4)
H75	0.7788	1.0249	0.8419	0.032*
C76	0.6694 (2)	0.8482 (2)	0.76705 (14)	0.0233 (4)
H76	0.7491	0.7901	0.7786	0.028*
C77	0.5936 (2)	1.2303 (2)	0.82597 (15)	0.0281 (4)
H77A	0.6322	1.2757	0.7800	0.042*
H77B	0.6610	1.2455	0.8915	0.042*
H77C	0.5015	1.2696	0.8301	0.042*
N51	0.42618 (15)	0.64448 (16)	0.85395 (11)	0.0176 (3)
C57	0.47980 (19)	0.7367 (2)	0.92717 (13)	0.0211 (4)
H57	0.4284	0.8173	0.9403	0.025*
C51	0.62249 (19)	0.7181 (2)	0.99163 (13)	0.0201 (4)
C52	0.6904 (2)	0.5935 (2)	0.98118 (15)	0.0267 (4)
H52	0.6457	0.5202	0.9308	0.032*
C53	0.8225 (2)	0.5750 (2)	1.04349 (17)	0.0323 (5)
H53	0.8687	0.4894	1.0362	0.039*
C54	0.8864 (2)	0.6820 (2)	1.11626 (15)	0.0279 (4)
Cl54	1.05226 (6)	0.65619 (7)	1.19494 (5)	0.04823 (18)
C55	0.8227 (2)	0.8073 (3)	1.12703 (17)	0.0386 (6)
H55	0.8687	0.8807	1.1768	0.046*
C56	0.6902 (2)	0.8253 (2)	1.06430 (16)	0.0344 (5)
H56	0.6455	0.9117	1.0711	0.041*
O61	0.17378 (13)	0.76209 (14)	0.90545 (9)	0.0213 (3)
C61	0.0420 (2)	0.8045 (2)	0.92910 (14)	0.0286 (5)
H61A	0.0000	0.8759	0.8894	0.043*
H61B	0.0617	0.8422	0.9994	0.043*
H61C	−0.0252	0.7242	0.9146	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0137 (7)	0.0224 (8)	0.0150 (7)	0.0013 (6)	0.0044 (6)	0.0009 (6)
C2	0.0123 (8)	0.0201 (9)	0.0156 (8)	0.0001 (6)	0.0038 (6)	0.0021 (7)
N3	0.0106 (6)	0.0198 (7)	0.0147 (7)	0.0002 (5)	0.0028 (5)	0.0007 (6)
C4	0.0109 (7)	0.0149 (8)	0.0167 (8)	0.0012 (6)	0.0044 (6)	0.0021 (6)
C5	0.0124 (8)	0.0191 (9)	0.0140 (8)	−0.0004 (6)	0.0026 (6)	0.0026 (7)
C6	0.0169 (8)	0.0182 (9)	0.0138 (8)	0.0006 (7)	0.0043 (7)	0.0012 (7)
N21	0.0101 (7)	0.0362 (10)	0.0159 (8)	0.0004 (6)	0.0041 (6)	−0.0038 (7)
N41	0.0108 (6)	0.0157 (7)	0.0134 (7)	0.0005 (5)	0.0023 (5)	−0.0005 (5)
C42	0.0095 (7)	0.0196 (8)	0.0121 (8)	0.0005 (6)	0.0012 (6)	0.0018 (6)
N43	0.0123 (7)	0.0193 (7)	0.0144 (7)	0.0014 (5)	0.0025 (5)	0.0015 (6)
C43A	0.0145 (8)	0.0189 (9)	0.0133 (8)	0.0034 (6)	0.0020 (6)	0.0034 (7)
C44	0.0196 (9)	0.0226 (9)	0.0189 (9)	0.0073 (7)	0.0071 (7)	0.0044 (7)
C45	0.0318 (10)	0.0189 (9)	0.0200 (9)	0.0091 (8)	0.0086 (8)	0.0022 (7)
C46	0.0285 (10)	0.0157 (9)	0.0195 (9)	−0.0007 (7)	0.0031 (8)	0.0014 (7)
C47	0.0174 (8)	0.0183 (9)	0.0154 (8)	−0.0010 (7)	0.0017 (7)	0.0016 (7)
C47A	0.0159 (8)	0.0170 (8)	0.0110 (8)	0.0032 (6)	0.0027 (6)	0.0011 (6)
C71	0.0146 (8)	0.0177 (8)	0.0143 (8)	−0.0005 (6)	0.0040 (6)	0.0013 (7)
C72	0.0139 (8)	0.0197 (9)	0.0142 (8)	0.0001 (6)	0.0027 (6)	0.0016 (7)
C73	0.0216 (9)	0.0206 (9)	0.0158 (8)	0.0044 (7)	0.0050 (7)	0.0032 (7)
C74	0.0291 (10)	0.0183 (9)	0.0163 (9)	−0.0025 (7)	0.0048 (7)	0.0005 (7)
C75	0.0192 (9)	0.0239 (10)	0.0286 (10)	−0.0050 (7)	−0.0030 (8)	−0.0029 (8)
C76	0.0166 (9)	0.0228 (10)	0.0268 (10)	0.0007 (7)	0.0001 (7)	0.0009 (8)
C77	0.0366 (11)	0.0189 (10)	0.0251 (10)	−0.0032 (8)	0.0039 (9)	−0.0014 (8)
N51	0.0117 (7)	0.0265 (8)	0.0140 (7)	0.0002 (6)	0.0018 (6)	0.0033 (6)
C57	0.0177 (9)	0.0251 (10)	0.0191 (9)	0.0033 (7)	0.0036 (7)	0.0004 (7)
C51	0.0161 (8)	0.0285 (10)	0.0147 (8)	−0.0004 (7)	0.0020 (7)	0.0028 (7)
C52	0.0247 (10)	0.0235 (10)	0.0270 (10)	−0.0032 (8)	−0.0018 (8)	0.0026 (8)
C53	0.0257 (10)	0.0277 (11)	0.0387 (12)	0.0023 (8)	−0.0030 (9)	0.0092 (9)
C54	0.0132 (8)	0.0424 (12)	0.0246 (10)	−0.0013 (8)	−0.0052 (7)	0.0109 (9)
Cl54	0.0240 (3)	0.0607 (4)	0.0478 (4)	0.0011 (3)	−0.0163 (2)	0.0141 (3)
C55	0.0267 (11)	0.0459 (14)	0.0303 (12)	0.0004 (10)	−0.0069 (9)	−0.0114 (10)
C56	0.0246 (10)	0.0383 (12)	0.0309 (11)	0.0072 (9)	−0.0017 (9)	−0.0098 (10)
O61	0.0155 (6)	0.0319 (7)	0.0144 (6)	0.0027 (5)	0.0037 (5)	−0.0035 (5)
C61	0.0202 (9)	0.0452 (13)	0.0185 (9)	0.0072 (9)	0.0074 (8)	−0.0057 (9)

Geometric parameters (Å, º)

N1—C6	1.312 (2)	C72—H72	0.9500
N1—C2	1.360 (2)	C73—C74	1.394 (3)
C2—N21	1.335 (2)	C73—H73	0.9500
C2—N3	1.354 (2)	C74—C75	1.395 (3)
N3—C4	1.334 (2)	C74—C77	1.507 (3)
C4—C5	1.388 (2)	C75—C76	1.387 (3)
C4—N41	1.420 (2)	C75—H75	0.9500
C5—N51	1.398 (2)	C76—H76	0.9500

C5—C6	1.416 (2)	C77—H77A	0.9800
C6—O61	1.350 (2)	C77—H77B	0.9800
N21—H21A	0.85 (2)	C77—H77C	0.9800
N21—H21B	0.90 (2)	N51—C57	1.269 (2)
N41—C42	1.391 (2)	C57—C51	1.474 (2)
N41—C47A	1.395 (2)	C57—H57	0.9500
C42—N43	1.312 (2)	C51—C52	1.387 (3)
C42—C71	1.467 (2)	C51—C56	1.388 (3)
N43—C43A	1.390 (2)	C52—C53	1.383 (3)
C43A—C44	1.400 (2)	C52—H52	0.9500
C43A—C47A	1.401 (2)	C53—C54	1.378 (3)
C44—C45	1.385 (3)	C53—H53	0.9500
C44—H44	0.9500	C54—C55	1.374 (3)
C45—C46	1.398 (3)	C54—Cl54	1.7446 (19)
C45—H45	0.9500	C55—C56	1.387 (3)
C46—C47	1.384 (3)	C55—H55	0.9500
C46—H46	0.9500	C56—H56	0.9500
C47—C47A	1.388 (2)	O61—C61	1.441 (2)
C47—H47	0.9500	C61—H61A	0.9800
C71—C72	1.395 (2)	C61—H61B	0.9800
C71—C76	1.396 (2)	C61—H61C	0.9800
C72—C73	1.387 (3)		

C6—N1—C2	116.86 (14)	C72—C73—C74	121.34 (17)
N21—C2—N3	118.02 (16)	C72—C73—H73	119.3
N21—C2—N1	117.05 (15)	C74—C73—H73	119.3
N3—C2—N1	124.89 (15)	C73—C74—C75	117.70 (17)
C4—N3—C2	114.98 (14)	C73—C74—C77	121.00 (18)
N3—C4—C5	125.61 (15)	C75—C74—C77	121.22 (18)
N3—C4—N41	115.85 (14)	C76—C75—C74	121.58 (17)
C5—C4—N41	118.54 (14)	C76—C75—H75	119.2
C4—C5—N51	118.29 (15)	C74—C75—H75	119.2
C4—C5—C6	113.10 (15)	C75—C76—C71	120.17 (17)
N51—C5—C6	128.42 (15)	C75—C76—H76	119.9
N1—C6—O61	119.20 (15)	C71—C76—H76	119.9
N1—C6—C5	123.85 (16)	C74—C77—H77A	109.5
O61—C6—C5	116.93 (15)	C74—C77—H77B	109.5
C2—N21—H21A	119.7 (15)	H77A—C77—H77B	109.5
C2—N21—H21B	121.4 (14)	C74—C77—H77C	109.5
H21A—N21—H21B	118 (2)	H77A—C77—H77C	109.5
C42—N41—C47A	106.54 (13)	H77B—C77—H77C	109.5
C42—N41—C4	127.65 (14)	C57—N51—C5	123.74 (16)
C47A—N41—C4	124.64 (14)	N51—C57—C51	119.55 (17)
N43—C42—N41	112.40 (15)	N51—C57—H57	120.2
N43—C42—C71	123.74 (15)	C51—C57—H57	120.2
N41—C42—C71	123.79 (14)	C52—C51—C56	119.12 (18)
C42—N43—C43A	105.65 (14)	C52—C51—C57	120.69 (17)
N43—C43A—C44	129.49 (16)	C56—C51—C57	120.19 (18)

N43—C43A—C47A	110.40 (15)	C53—C52—C51	120.58 (19)
C44—C43A—C47A	120.11 (16)	C53—C52—H52	119.7
C45—C44—C43A	117.06 (17)	C51—C52—H52	119.7
C45—C44—H44	121.5	C54—C53—C52	119.2 (2)
C43A—C44—H44	121.5	C54—C53—H53	120.4
C44—C45—C46	121.81 (17)	C52—C53—H53	120.4
C44—C45—H45	119.1	C55—C54—C53	121.33 (18)
C46—C45—H45	119.1	C55—C54—Cl54	120.07 (16)
C47—C46—C45	121.95 (17)	C53—C54—Cl54	118.59 (17)
C47—C46—H46	119.0	C54—C55—C56	119.2 (2)
C45—C46—H46	119.0	C54—C55—H55	120.4
C46—C47—C47A	116.02 (16)	C56—C55—H55	120.4
C46—C47—H47	122.0	C55—C56—C51	120.6 (2)
C47A—C47—H47	122.0	C55—C56—H56	119.7
C47—C47A—N41	131.90 (16)	C51—C56—H56	119.7
C47—C47A—C43A	123.04 (16)	C6—O61—C61	116.52 (14)
N41—C47A—C43A	105.01 (14)	O61—C61—H61A	109.5
C72—C71—C76	118.74 (16)	O61—C61—H61B	109.5
C72—C71—C42	122.49 (15)	H61A—C61—H61B	109.5
C76—C71—C42	118.76 (16)	O61—C61—H61C	109.5
C73—C72—C71	120.46 (16)	H61A—C61—H61C	109.5
C73—C72—H72	119.8	H61B—C61—H61C	109.5
C71—C72—H72	119.8		

C6—N1—C2—N21	175.07 (17)	C42—N41—C47A—C43A	−0.19 (18)
C6—N1—C2—N3	−7.4 (3)	C4—N41—C47A—C43A	−168.60 (15)
N21—C2—N3—C4	−175.16 (16)	N43—C43A—C47A—C47	−178.13 (16)
N1—C2—N3—C4	7.3 (3)	C44—C43A—C47A—C47	1.4 (3)
C2—N3—C4—C5	0.2 (3)	N43—C43A—C47A—N41	−0.29 (19)
C2—N3—C4—N41	−178.67 (15)	C44—C43A—C47A—N41	179.26 (15)
N3—C4—C5—N51	168.95 (16)	N43—C42—C71—C72	147.26 (17)
N41—C4—C5—N51	−12.2 (2)	N41—C42—C71—C72	−29.4 (3)
N3—C4—C5—C6	−6.5 (3)	N43—C42—C71—C76	−32.0 (3)
N41—C4—C5—C6	172.36 (15)	N41—C42—C71—C76	151.33 (17)
C2—N1—C6—O61	−178.62 (16)	C76—C71—C72—C73	−0.8 (3)
C2—N1—C6—C5	−0.1 (3)	C42—C71—C72—C73	179.98 (16)
C4—C5—C6—N1	6.4 (3)	C71—C72—C73—C74	0.9 (3)
N51—C5—C6—N1	−168.45 (18)	C72—C73—C74—C75	−0.5 (3)
C4—C5—C6—O61	−175.07 (16)	C72—C73—C74—C77	176.20 (18)
N51—C5—C6—O61	10.1 (3)	C73—C74—C75—C76	0.0 (3)
N3—C4—N41—C42	131.91 (18)	C77—C74—C75—C76	−176.67 (19)
C5—C4—N41—C42	−47.0 (2)	C74—C75—C76—C71	0.1 (3)
N3—C4—N41—C47A	−62.2 (2)	C72—C71—C76—C75	0.3 (3)
C5—C4—N41—C47A	118.89 (18)	C42—C71—C76—C75	179.58 (18)
C47A—N41—C42—N43	0.64 (19)	C4—C5—N51—C57	145.43 (18)
C4—N41—C42—N43	168.59 (15)	C6—C5—N51—C57	−39.9 (3)
C47A—N41—C42—C71	177.66 (15)	C5—N51—C57—C51	179.10 (16)
C4—N41—C42—C71	−14.4 (3)	N51—C57—C51—C52	−8.5 (3)

N41—C42—N43—C43A	−0.80 (19)	N51—C57—C51—C56	171.95 (19)
C71—C42—N43—C43A	−177.82 (16)	C56—C51—C52—C53	1.3 (3)
C42—N43—C43A—C44	−178.82 (18)	C57—C51—C52—C53	−178.24 (19)
C42—N43—C43A—C47A	0.67 (19)	C51—C52—C53—C54	−0.1 (3)
N43—C43A—C44—C45	178.78 (17)	C52—C53—C54—C55	−1.1 (4)
C47A—C43A—C44—C45	−0.7 (3)	C52—C53—C54—Cl54	179.36 (17)
C43A—C44—C45—C46	0.0 (3)	C53—C54—C55—C56	1.1 (4)
C44—C45—C46—C47	0.0 (3)	Cl54—C54—C55—C56	−179.40 (19)
C45—C46—C47—C47A	0.7 (3)	C54—C55—C56—C51	0.2 (4)
C46—C47—C47A—N41	−178.56 (17)	C52—C51—C56—C55	−1.3 (3)
C46—C47—C47A—C43A	−1.4 (3)	C57—C51—C56—C55	178.2 (2)
C42—N41—C47A—C47	177.38 (18)	N1—C6—O61—C61	0.9 (2)
C4—N41—C47A—C47	9.0 (3)	C5—C6—O61—C61	−177.71 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*A*···N3^{i}	0.85 (2)	2.35 (2)	3.196 (2)	175.9 (19)
N21—H21*B*···N43^{ii}	0.90 (2)	2.08 (2)	2.946 (2)	163.6 (19)
C72—H72···*Cg*5	0.95	2.97	3.5865 (19)	124

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*−1, *y*, *z*.