Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2,6-Diisopropylphenyl)-6-[(2,6-diisopropylphenyl)(methacryloyl)aminocarbonyl]pyridine-2-carboximidoyl chloride

Aslıhan Aydın,[a] Muharrem Dinçer,[a]* Osman Dayan[b] and Bekir Çetinkaya[c]

[a]Ondokuz Mayıs University, Arts and Sciences Faculty, Department of Physics, 55139 Samsun, Turkey, [b]Çanakkale Onsekiz Mart University, Arts and Sciences Faculty, Department of Chemistry, 17020 Çanakkale, Turkey, and [c]Ege University, Science Faculty, Department of Chemistry, 35100 Izmir, Turkey
Correspondence e-mail: mdincer@omu.edu.tr

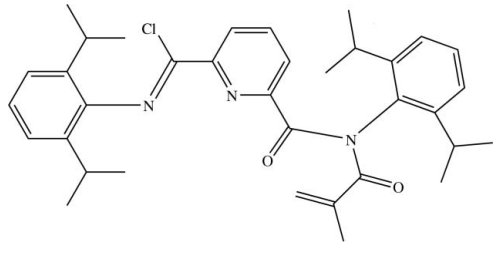
Received 25 October 2007; accepted 28 October 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.142; data-to-parameter ratio = 19.5.

In the title compound, $C_{35}H_{42}ClN_3O_2$, molecules are linked to one another by intermolecular C—H···O interactions, forming a $C(12)$ chain running parallel to the [010] direction. The molecule has a nonplanar conformation. The benzene rings make dihedral angles of 72.72 (6) and 62.72 (6)° with the pyridine ring.

Related literature

For related literature, see: Archer *et al.* (2006); Bacha *et al.* (1987); Bernstein *et al.* (1995); Bonnett (1970); Dayan & Çetinkaya (2005); Shishkin *et al.* (2004); Sladowska *et al.* (1995).



Experimental

Crystal data

$C_{35}H_{42}ClN_3O_2$
M_r = 572.17
Monoclinic, $P2_1/c$
a = 9.4568 (3) Å
b = 14.6356 (7) Å
c = 24.0095 (8) Å
β = 91.391 (3)°
V = 3322.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 296 K
0.69 × 0.58 × 0.30 mm

Data collection

STOE IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.917, T_{max} = 0.960
52993 measured reflections
7398 independent reflections
4940 reflections with $I > 2\sigma(I)$
R_{int} = 0.127

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.06
7398 reflections
380 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14A···O1^{i}	0.96	2.45	3.392 (3)	168

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

This study was supported financially by the Research Center of Ondokuz Mayıs University (project No. F-425).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2180).

References

Archer, A. M., Bouwkamp, M. W., Cortez, M. P., Lobkovsky, E. & Chirik, P. J. (2006). *Organometallics*, **25**, 4269–4278.
Bacha, C., Ferreira, I., Loiseau, P., Schapoval, E., Tarayre, J. P. & Wolf, C. (1987). *Pharm. Acta Helv.* **62**, 292–296.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bonnett, R. (1970). *The Chemistry of the Carbon–Nitrogen Double Bond*, p. 597. New York: Interscience.
Dayan, O. & Çetinkaya, B. (2005). XIXth National Chemistry Congress, Kuşadası, Turkey, 30 September–4 October 2005, Abstracts, p. 133.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shishkin, E. V., Vasil'ev, V. A. & Shishkin, V. E. (2004). *Russ. J. Gen. Chem.* **74**, 475–476.
Sladowska, H., Potoczek, J., Sieklucka-Dziuba, M., Semczuk, A. & Kleinrok, Z. (1995). *Il Farmaco*, **50**, 761–768.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

  doi:10.1107/S1600536807053901

supplementary materials

Acta Cryst. (2007). E**63**, o4538 [**doi:10.1107/S1600536807053901**]

N-(2,6-Diisopropylphenyl)-6-[(2,6-diisopropylphenyl)(methacryloyl)aminocarbonyl]pyridine-2-carboximidoyl chloride

A. Aydin, M. Dinçer, O. Dayan and B. Çetinkaya

Comment

Imide functionality is found as a basic structural element in a wide range of naturally occurring compounds such as uracil and thymine. Moreover, many imide-containing unnatural products have been prepared to test the pharmacological properties as well as their structural properties (Bacha *et al.*, 1987; Sladowska *et al.*, 1995). Imidoyl chlorides react with carboxylic acids and their salts, yielding N-substitue imides with regard to Mumm rearrangement reaction (Bonnett, 1970; Shishkin *et al.*, 2004)

Proceeding with our research into pyridyl containing moties (Dayan & Çetinkaya, 2005), we have studied utility of the above reaction for preparing new functionally substituted pyridine derivatives. In this work, we synthesized (II) from *N*,*N*'-bis(2,6-diisopropylphenyl)pyridine-2,6-dicarboximidoyl dichloride, (I), with sodium methacrylat in THF. The title compound was characterized by elemental analysis, spectroscopic and X-ray crystallographic methods.

The structure of the title compound, (II), is shown in Fig. 1 and selected geometric parameters are listed in Table 1. The interatomic distances and angles show no anomalies. In the molecular structure of (II), (C7—C12) and (C24—C29) phenyl rings make dihedral angles of 72.72 (6) and 62.72 (6)°, respectively, with the pyridine ring.

In the crystal structure of (II), atom C14 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to the O atom in the molecule at (−*x*, *y* + 1/2, −*z* + 1/2), forming a C(12) (Bernstein *et al.*, 1995) chain running parallel to the [010] direction.

Experimental

(I) was prepared a modification of literature method (Archer *et al.*, 2006). A mixture of (I) (523 mg, 1 mmol) and sodium methacrylat (106 mg, 1 mmol) were refluxed in THF (20 ml) for 2 days and was concentrated (5 ml). Et_2O was added with stirring to a final volume of 20 ml causing a yellow powder to precipitate. The precipitate was filtered off, washed with Et_2O and dried. X-ray quality crystals were grown from CH_2Cl_2-hexane (1:2 *v*/*v*, 30 ml) (yield: 410 mg, 72%; m.p. 404–405 K). Analysis calculated for $C_{35}H_{42}ClN_3O_2$: C 73.47, H 7.40, N 7.34%; found: C 73.23, H 7.49, N 7.32%. ^{1}H NMR ($CDCl_3$): δ 8.32 (d, J = 8.0, 1H, py—Hm), 7.99 (d, J = 7.6, 1H, py—Hm), 7.94 (t, J = 7.6, 1H, py—Hp), 7.18–7.13 [m, 6H, $(CH_3)_2CH—C_6H_3$], 5.48 (s, 1H, CH_2-acryl), 5.12 (s, 1H, CH_2-acryl), 2.79–2.66 [m, 4H, $(CH_3)_2CH—C_6H_3$], 1.74 (s, 3H, CH_3-acryl), 1.16–1.05 [m, 24H, $(CH_3)_2CH—C_6H_3$]; ^{13}C NMR ($CDCl_3$): δ 176.75, 168.33, 164.17, 159.49, 155.38, 149.27, 147.86, 145.28, 139.37, 136.14, 132.65; 130.29, 127.35, 126.03, 125.44, 118.14, 112.76, 29.91, 27.75, 22.44, 20.20.

Refinement

H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.98, 0.96 and 0.93 Å for CH, CH_3, aromatic CH and CH_2 groups, respectively. The displacement parameters of the H atoms were constrained

as U_{iso}(H) = 1.2U_{eq} (1.5U_{eq} for methyl) of the parent atom. Riding methyl H atoms were allowed to rotate freely during refinement using the AFIX 137 command of *SHELXL97* (Sheldrick, 1997). Examination of the refined structure using *PLATON* (Spek, 2003) revealed the presence of void spaces having a total volume of 157.8 Å^3 [4.8%] per unit cell, the volume of the individual voids being 39 Å^3.

Figures

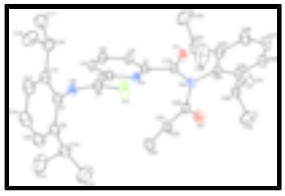


Fig. 1. : A view of (II), with 30% probability displacement ellipsoids and the atom-numbering scheme. The H atoms have been omitted for clarity.

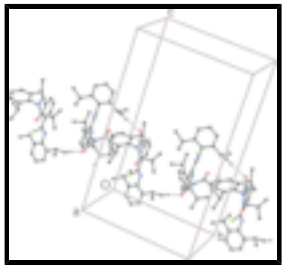


Fig. 2. : A packing diagram of (II), viewed approximately along the *a* axis. For clarity, only H atoms involved in hydrogen bonding have been included.

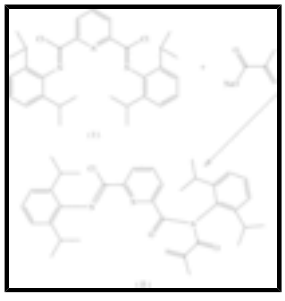


Fig. 3. The formation of the title compound.

N-(2,6-Diisopropylphenyl)-6-[(2,6- diisopropylphenyl)(methacryloyl)aminocarbonyl]pyridine-2-carboximidoyl chloride

Crystal data

$C_{35}H_{42}ClN_3O_2$	F_{000} = 1224
M_r = 572.17	D_x = 1.144 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 35523 reflections
a = 9.4568 (3) Å	θ = 1.4–27.3º
b = 14.6356 (7) Å	μ = 0.15 mm^{-1}
c = 24.0095 (8) Å	T = 296 K
β = 91.391 (3)º	Prism, colorless
V = 3322.1 (2) Å^3	0.69 × 0.58 × 0.30 mm
Z = 4	

Data collection

STOE IPDS 2 diffractometer	7398 independent reflections
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	4940 reflections with $I > 2\sigma(I)$
Monochromator: plane graphite	$R_{int} = 0.127$
Detector resolution: 6.67 pixels mm^{-1}	$\theta_{max} = 27.3°$
$T = 296$ K	$\theta_{min} = 1.6°$
w scans	$h = -12 \rightarrow 12$
Absorption correction: integration X-RED32 (Stoe & Cie, 2002)	$k = -18 \rightarrow 18$
$T_{min} = 0.917$, $T_{max} = 0.960$	$l = -30 \rightarrow 30$
52993 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.1358P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.142$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.19$ e $Å^{-3}$
7398 reflections	$\Delta\rho_{min} = -0.29$ e $Å^{-3}$
380 parameters	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0076 (10)
Secondary atom site location: difference Fourier map	

Special details

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.09937 (5)	0.11683 (5)	0.130226 (18)	0.0925 (2)

O1	0.09310 (12)	0.09776 (10)	0.38351 (4)	0.0686 (3)
O2	0.39007 (13)	0.20674 (11)	0.25418 (6)	0.0835 (4)
N1	0.01045 (12)	0.14281 (9)	0.24386 (5)	0.0471 (3)
N2	−0.16629 (14)	0.16848 (10)	0.11306 (5)	0.0559 (3)
N3	0.24640 (12)	0.14538 (9)	0.31830 (5)	0.0489 (3)
C1	−0.01507 (14)	0.13968 (10)	0.29816 (5)	0.0457 (3)
C2	−0.14984 (15)	0.13780 (12)	0.31944 (6)	0.0562 (4)
H2	−0.1631	0.1347	0.3576	0.067*
C3	−0.26403 (16)	0.14068 (14)	0.28249 (7)	0.0646 (4)
H3	−0.3560	0.1401	0.2954	0.078*
C4	−0.23943 (16)	0.14445 (12)	0.22635 (6)	0.0599 (4)
H4	−0.3145	0.1468	0.2007	0.072*
C5	−0.10126 (15)	0.14471 (11)	0.20865 (6)	0.0483 (3)
C6	−0.07281 (16)	0.14728 (11)	0.14799 (6)	0.0532 (4)
C7	−0.14697 (16)	0.16956 (12)	0.05439 (6)	0.0560 (4)
C8	−0.10439 (18)	0.25004 (13)	0.02938 (7)	0.0632 (4)
C9	−0.0905 (2)	0.24901 (15)	−0.02835 (8)	0.0747 (5)
H9	−0.0597	0.3015	−0.0462	0.090*
C10	−0.1208 (2)	0.17306 (16)	−0.05911 (7)	0.0771 (5)
H10	−0.1105	0.1742	−0.0975	0.092*
C11	−0.16686 (19)	0.09442 (14)	−0.03359 (7)	0.0694 (5)
H11	−0.1886	0.0433	−0.0551	0.083*
C12	−0.18114 (17)	0.09059 (12)	0.02394 (6)	0.0589 (4)
C13	−0.0822 (2)	0.33733 (15)	0.06249 (8)	0.0767 (5)
H13	−0.0890	0.3212	0.1019	0.092*
C14	−0.1990 (4)	0.4046 (2)	0.04950 (17)	0.1455 (14)
H14A	−0.1831	0.4595	0.0706	0.218*
H14B	−0.2882	0.3784	0.0592	0.218*
H14C	−0.2002	0.4187	0.0104	0.218*
C15	0.0599 (3)	0.3802 (2)	0.05534 (16)	0.1359 (11)
H15A	0.1325	0.3359	0.0634	0.204*
H15B	0.0705	0.4310	0.0804	0.204*
H15C	0.0679	0.4011	0.0177	0.204*
C16	−0.2335 (2)	0.00589 (14)	0.05299 (7)	0.0694 (5)
H16	−0.1710	−0.0042	0.0856	0.083*
C17	−0.2292 (2)	−0.08059 (15)	0.01778 (9)	0.0822 (6)
H17A	−0.2963	−0.0755	−0.0127	0.123*
H17B	−0.2527	−0.1324	0.0403	0.123*
H17C	−0.1360	−0.0884	0.0036	0.123*
C18	−0.3825 (2)	0.0218 (2)	0.07462 (10)	0.1040 (8)
H18A	−0.3843	0.0778	0.0954	0.156*
H18B	−0.4082	−0.0281	0.0983	0.156*
H18C	−0.4486	0.0256	0.0437	0.156*
C19	0.10994 (15)	0.12868 (11)	0.33765 (6)	0.0486 (3)
C20	0.28190 (16)	0.21303 (12)	0.28020 (7)	0.0579 (4)
C21	0.19384 (17)	0.29727 (13)	0.27667 (8)	0.0635 (4)
C22	0.1475 (2)	0.33563 (15)	0.32245 (10)	0.0847 (6)
H22A	0.1698	0.3101	0.3570	0.102*
H22B	0.0925	0.3882	0.3202	0.102*

C23	0.1784 (2)	0.33936 (17)	0.21987 (10)	0.0918 (7)
H23A	0.1072	0.3071	0.1986	0.138*
H23B	0.2669	0.3357	0.2013	0.138*
H23C	0.1512	0.4023	0.2234	0.138*
C24	0.35683 (14)	0.08216 (11)	0.33730 (6)	0.0514 (4)
C25	0.37940 (16)	0.00325 (12)	0.30608 (7)	0.0603 (4)
C26	0.4819 (2)	−0.05741 (14)	0.32601 (8)	0.0748 (5)
H26	0.4983	−0.1112	0.3066	0.090*
C27	0.5593 (2)	−0.03942 (15)	0.37385 (8)	0.0784 (5)
H27	0.6276	−0.0808	0.3863	0.094*
C28	0.53648 (18)	0.03889 (14)	0.40326 (7)	0.0694 (5)
H28	0.5908	0.0503	0.4353	0.083*
C29	0.43408 (15)	0.10189 (12)	0.38640 (6)	0.0558 (4)
C30	0.41205 (17)	0.18827 (13)	0.41938 (7)	0.0632 (4)
H30	0.3209	0.2145	0.4074	0.076*
C31	0.4057 (4)	0.1689 (2)	0.48191 (9)	0.1244 (11)
H31A	0.4974	0.1503	0.4957	0.187*
H31B	0.3387	0.1210	0.4883	0.187*
H31C	0.3770	0.2232	0.5010	0.187*
C32	0.5241 (3)	0.2564 (2)	0.40716 (16)	0.1345 (12)
H32A	0.6154	0.2297	0.4143	0.202*
H32B	0.5129	0.3090	0.4305	0.202*
H32C	0.5161	0.2744	0.3688	0.202*
C33	0.29733 (19)	−0.01790 (15)	0.25266 (8)	0.0737 (5)
H33	0.2347	0.0340	0.2445	0.088*
C34	0.2056 (4)	−0.1009 (3)	0.26082 (17)	0.1589 (15)
H34A	0.2645	−0.1531	0.2686	0.238*
H34B	0.1498	−0.1119	0.2276	0.238*
H34C	0.1443	−0.0907	0.2915	0.238*
C35	0.3943 (3)	−0.0290 (3)	0.20401 (10)	0.1347 (13)
H35A	0.4535	−0.0815	0.2100	0.202*
H35B	0.4522	0.0246	0.2008	0.202*
H35C	0.3387	−0.0369	0.1704	0.202*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0634 (3)	0.1692 (6)	0.0451 (2)	0.0331 (3)	0.00353 (18)	−0.0044 (3)
O1	0.0563 (6)	0.1064 (10)	0.0430 (6)	0.0093 (6)	0.0030 (4)	0.0161 (6)
O2	0.0544 (7)	0.1148 (11)	0.0822 (9)	0.0067 (7)	0.0181 (6)	0.0299 (8)
N1	0.0421 (6)	0.0611 (7)	0.0381 (6)	0.0008 (5)	0.0001 (4)	−0.0001 (5)
N2	0.0548 (7)	0.0726 (9)	0.0401 (6)	−0.0004 (6)	−0.0028 (5)	0.0022 (6)
N3	0.0395 (6)	0.0654 (8)	0.0416 (6)	0.0044 (5)	−0.0010 (4)	0.0027 (5)
C1	0.0421 (7)	0.0565 (8)	0.0385 (7)	0.0017 (6)	0.0004 (5)	0.0004 (6)
C2	0.0461 (7)	0.0816 (11)	0.0412 (7)	0.0022 (7)	0.0041 (6)	0.0002 (7)
C3	0.0407 (7)	0.1008 (14)	0.0525 (9)	−0.0005 (8)	0.0038 (6)	0.0018 (8)
C4	0.0436 (7)	0.0867 (12)	0.0489 (8)	−0.0004 (7)	−0.0055 (6)	0.0009 (8)
C5	0.0466 (7)	0.0592 (9)	0.0387 (7)	0.0007 (6)	−0.0025 (5)	−0.0001 (6)

C6	0.0507 (8)	0.0675 (10)	0.0412 (7)	0.0001 (7)	−0.0018 (6)	−0.0013 (6)
C7	0.0513 (8)	0.0770 (11)	0.0395 (7)	0.0052 (7)	−0.0044 (6)	0.0030 (7)
C8	0.0645 (9)	0.0779 (12)	0.0471 (9)	0.0007 (8)	−0.0032 (7)	0.0066 (8)
C9	0.0806 (12)	0.0917 (14)	0.0518 (10)	0.0041 (10)	0.0025 (8)	0.0185 (9)
C10	0.0862 (13)	0.1055 (16)	0.0395 (8)	0.0164 (11)	0.0026 (8)	0.0071 (9)
C11	0.0728 (11)	0.0901 (13)	0.0449 (9)	0.0129 (10)	−0.0040 (7)	−0.0050 (8)
C12	0.0547 (8)	0.0772 (11)	0.0446 (8)	0.0080 (8)	−0.0051 (6)	−0.0004 (7)
C13	0.0839 (13)	0.0801 (13)	0.0657 (11)	−0.0125 (10)	−0.0071 (9)	0.0064 (9)
C14	0.122 (2)	0.112 (2)	0.200 (4)	0.0292 (19)	−0.053 (2)	−0.067 (2)
C15	0.098 (2)	0.139 (3)	0.170 (3)	−0.0331 (18)	−0.0128 (19)	−0.017 (2)
C16	0.0714 (11)	0.0835 (13)	0.0530 (9)	−0.0095 (9)	−0.0034 (8)	−0.0004 (8)
C17	0.0827 (13)	0.0812 (13)	0.0825 (14)	−0.0009 (11)	−0.0039 (10)	−0.0046 (10)
C18	0.0911 (15)	0.122 (2)	0.1003 (17)	−0.0290 (14)	0.0295 (13)	−0.0287 (15)
C19	0.0445 (7)	0.0635 (9)	0.0378 (7)	0.0056 (6)	0.0016 (5)	0.0014 (6)
C20	0.0453 (8)	0.0755 (11)	0.0528 (8)	−0.0019 (7)	−0.0015 (6)	0.0086 (8)
C21	0.0506 (8)	0.0660 (10)	0.0732 (11)	−0.0072 (7)	−0.0093 (7)	0.0122 (9)
C22	0.0927 (15)	0.0691 (12)	0.0911 (15)	0.0129 (11)	−0.0228 (11)	−0.0079 (11)
C23	0.0740 (12)	0.1016 (16)	0.0997 (16)	−0.0031 (11)	−0.0011 (11)	0.0470 (13)
C24	0.0403 (7)	0.0656 (9)	0.0482 (8)	0.0057 (6)	−0.0001 (6)	0.0030 (7)
C25	0.0486 (8)	0.0730 (11)	0.0593 (9)	0.0068 (7)	0.0017 (7)	−0.0054 (8)
C26	0.0680 (11)	0.0775 (12)	0.0787 (12)	0.0210 (9)	−0.0015 (9)	−0.0070 (10)
C27	0.0665 (11)	0.0895 (14)	0.0788 (13)	0.0285 (10)	−0.0066 (9)	0.0053 (11)
C28	0.0574 (9)	0.0916 (13)	0.0586 (10)	0.0159 (9)	−0.0093 (7)	0.0053 (9)
C29	0.0463 (7)	0.0745 (11)	0.0466 (8)	0.0061 (7)	−0.0016 (6)	0.0027 (7)
C30	0.0539 (9)	0.0840 (12)	0.0511 (9)	0.0065 (8)	−0.0091 (7)	−0.0045 (8)
C31	0.175 (3)	0.145 (2)	0.0532 (12)	0.058 (2)	0.0017 (14)	−0.0095 (13)
C32	0.108 (2)	0.107 (2)	0.191 (3)	−0.0304 (16)	0.042 (2)	−0.052 (2)
C33	0.0630 (10)	0.0879 (13)	0.0697 (11)	0.0122 (9)	−0.0074 (8)	−0.0244 (10)
C34	0.153 (3)	0.158 (3)	0.163 (3)	−0.068 (3)	−0.055 (2)	−0.008 (2)
C35	0.0994 (18)	0.230 (4)	0.0745 (15)	0.033 (2)	−0.0008 (13)	−0.053 (2)

Geometric parameters (Å, °)

Cl1—C6	1.7508 (16)	C17—H17B	0.9600
O1—C19	1.2046 (17)	C17—H17C	0.9600
O2—C20	1.2150 (19)	C18—H18A	0.9600
N1—C1	1.3325 (17)	C18—H18B	0.9600
N1—C5	1.3372 (17)	C18—H18C	0.9600
N2—C6	1.2431 (19)	C20—C21	1.489 (3)
N2—C7	1.4251 (19)	C21—C22	1.318 (3)
N3—C20	1.395 (2)	C21—C23	1.500 (3)
N3—C19	1.4035 (18)	C22—H22A	0.9300
N3—C24	1.4599 (18)	C22—H22B	0.9300
C1—C2	1.385 (2)	C23—H23A	0.9600
C1—C19	1.5060 (18)	C23—H23B	0.9600
C2—C3	1.382 (2)	C23—H23C	0.9600
C2—H2	0.9300	C24—C25	1.396 (2)
C3—C4	1.374 (2)	C24—C29	1.402 (2)
C3—H3	0.9300	C25—C26	1.391 (2)

C4—C5	1.384 (2)	C25—C33	1.515 (2)
C4—H4	0.9300	C26—C27	1.372 (3)
C5—C6	1.488 (2)	C26—H26	0.9300
C7—C8	1.386 (2)	C27—C28	1.366 (3)
C7—C12	1.401 (2)	C27—H27	0.9300
C8—C9	1.396 (2)	C28—C29	1.390 (2)
C8—C13	1.517 (3)	C28—H28	0.9300
C9—C10	1.361 (3)	C29—C30	1.509 (2)
C9—H9	0.9300	C30—C32	1.489 (3)
C10—C11	1.379 (3)	C30—C31	1.530 (3)
C10—H10	0.9300	C30—H30	0.9800
C11—C12	1.392 (2)	C31—H31A	0.9600
C11—H11	0.9300	C31—H31B	0.9600
C12—C16	1.512 (3)	C31—H31C	0.9600
C13—C15	1.497 (3)	C32—H32A	0.9600
C13—C14	1.507 (4)	C32—H32B	0.9600
C13—H13	0.9800	C32—H32C	0.9600
C14—H14A	0.9600	C33—C34	1.509 (4)
C14—H14B	0.9600	C33—C35	1.511 (3)
C14—H14C	0.9600	C33—H33	0.9800
C15—H15A	0.9600	C34—H34A	0.9600
C15—H15B	0.9600	C34—H34B	0.9600
C15—H15C	0.9600	C34—H34C	0.9600
C16—C17	1.523 (3)	C35—H35A	0.9600
C16—C18	1.532 (3)	C35—H35B	0.9600
C16—H16	0.9800	C35—H35C	0.9600
C17—H17A	0.9600		
C1—N1—C5	117.40 (12)	H18A—C18—H18C	109.5
C6—N2—C7	124.30 (14)	H18B—C18—H18C	109.5
C20—N3—C19	125.35 (12)	O1—C19—N3	120.72 (12)
C20—N3—C24	118.24 (12)	O1—C19—C1	119.76 (13)
C19—N3—C24	116.28 (12)	N3—C19—C1	119.15 (12)
N1—C1—C2	123.50 (13)	O2—C20—N3	120.17 (16)
N1—C1—C19	117.49 (12)	O2—C20—C21	120.68 (16)
C2—C1—C19	118.75 (12)	N3—C20—C21	118.82 (14)
C3—C2—C1	118.32 (14)	C22—C21—C20	120.12 (17)
C3—C2—H2	120.8	C22—C21—C23	123.8 (2)
C1—C2—H2	120.8	C20—C21—C23	115.70 (18)
C4—C3—C2	118.86 (14)	C21—C22—H22A	120.0
C4—C3—H3	120.6	C21—C22—H22B	120.0
C2—C3—H3	120.6	H22A—C22—H22B	120.0
C3—C4—C5	119.01 (13)	C21—C23—H23A	109.5
C3—C4—H4	120.5	C21—C23—H23B	109.5
C5—C4—H4	120.5	H23A—C23—H23B	109.5
N1—C5—C4	122.90 (13)	C21—C23—H23C	109.5
N1—C5—C6	117.41 (12)	H23A—C23—H23C	109.5
C4—C5—C6	119.69 (12)	H23B—C23—H23C	109.5
N2—C6—C5	121.61 (14)	C25—C24—C29	122.59 (14)
N2—C6—Cl1	123.33 (12)	C25—C24—N3	118.30 (13)

C5—C6—Cl1	115.05 (10)	C29—C24—N3	119.10 (14)
C8—C7—C12	122.73 (14)	C26—C25—C24	117.24 (15)
C8—C7—N2	118.88 (15)	C26—C25—C33	119.96 (16)
C12—C7—N2	118.25 (15)	C24—C25—C33	122.80 (15)
C7—C8—C9	117.14 (17)	C27—C26—C25	121.23 (18)
C7—C8—C13	121.79 (15)	C27—C26—H26	119.4
C9—C8—C13	120.96 (17)	C25—C26—H26	119.4
C10—C9—C8	121.60 (18)	C28—C27—C26	120.40 (17)
C10—C9—H9	119.2	C28—C27—H27	119.8
C8—C9—H9	119.2	C26—C27—H27	119.8
C9—C10—C11	120.33 (16)	C27—C28—C29	121.56 (16)
C9—C10—H10	119.8	C27—C28—H28	119.2
C11—C10—H10	119.8	C29—C28—H28	119.2
C10—C11—C12	120.90 (18)	C28—C29—C24	116.97 (16)
C10—C11—H11	119.5	C28—C29—C30	120.46 (14)
C12—C11—H11	119.5	C24—C29—C30	122.56 (14)
C11—C12—C7	117.24 (17)	C32—C30—C29	110.45 (17)
C11—C12—C16	122.11 (16)	C32—C30—C31	111.2 (2)
C7—C12—C16	120.64 (14)	C29—C30—C31	111.64 (18)
C15—C13—C14	111.0 (2)	C32—C30—H30	107.8
C15—C13—C8	114.0 (2)	C29—C30—H30	107.8
C14—C13—C8	110.51 (17)	C31—C30—H30	107.8
C15—C13—H13	107.0	C30—C31—H31A	109.5
C14—C13—H13	107.0	C30—C31—H31B	109.5
C8—C13—H13	107.0	H31A—C31—H31B	109.5
C13—C14—H14A	109.5	C30—C31—H31C	109.5
C13—C14—H14B	109.5	H31A—C31—H31C	109.5
H14A—C14—H14B	109.5	H31B—C31—H31C	109.5
C13—C14—H14C	109.5	C30—C32—H32A	109.5
H14A—C14—H14C	109.5	C30—C32—H32B	109.5
H14B—C14—H14C	109.5	H32A—C32—H32B	109.5
C13—C15—H15A	109.5	C30—C32—H32C	109.5
C13—C15—H15B	109.5	H32A—C32—H32C	109.5
H15A—C15—H15B	109.5	H32B—C32—H32C	109.5
C13—C15—H15C	109.5	C34—C33—C35	112.1 (3)
H15A—C15—H15C	109.5	C34—C33—C25	109.8 (2)
H15B—C15—H15C	109.5	C35—C33—C25	111.61 (17)
C12—C16—C17	114.32 (15)	C34—C33—H33	107.7
C12—C16—C18	110.27 (18)	C35—C33—H33	107.7
C17—C16—C18	110.57 (17)	C25—C33—H33	107.7
C12—C16—H16	107.1	C33—C34—H34A	109.5
C17—C16—H16	107.1	C33—C34—H34B	109.5
C18—C16—H16	107.1	H34A—C34—H34B	109.5
C16—C17—H17A	109.5	C33—C34—H34C	109.5
C16—C17—H17B	109.5	H34A—C34—H34C	109.5
H17A—C17—H17B	109.5	H34B—C34—H34C	109.5
C16—C17—H17C	109.5	C33—C35—H35A	109.5
H17A—C17—H17C	109.5	C33—C35—H35B	109.5
H17B—C17—H17C	109.5	H35A—C35—H35B	109.5

C16—C18—H18A	109.5	C33—C35—H35C	109.5
C16—C18—H18B	109.5	H35A—C35—H35C	109.5
H18A—C18—H18B	109.5	H35B—C35—H35C	109.5
C16—C18—H18C	109.5		
C5—N1—C1—C2	0.4 (2)	C24—N3—C19—O1	33.8 (2)
C5—N1—C1—C19	174.43 (13)	C20—N3—C19—C1	36.5 (2)
N1—C1—C2—C3	−1.0 (3)	C24—N3—C19—C1	−139.26 (14)
C19—C1—C2—C3	−174.97 (16)	N1—C1—C19—O1	−157.08 (15)
C1—C2—C3—C4	0.6 (3)	C2—C1—C19—O1	17.3 (2)
C2—C3—C4—C5	0.4 (3)	N1—C1—C19—N3	16.1 (2)
C1—N1—C5—C4	0.6 (2)	C2—C1—C19—N3	−169.59 (14)
C1—N1—C5—C6	−179.41 (13)	C19—N3—C20—O2	−159.05 (16)
C3—C4—C5—N1	−1.0 (3)	C24—N3—C20—O2	16.6 (2)
C3—C4—C5—C6	179.04 (16)	C19—N3—C20—C21	27.6 (2)
C7—N2—C6—C5	−178.02 (15)	C24—N3—C20—C21	−156.76 (14)
C7—N2—C6—Cl1	1.0 (2)	O2—C20—C21—C22	−132.8 (2)
N1—C5—C6—N2	−164.47 (16)	N3—C20—C21—C22	40.5 (2)
C4—C5—C6—N2	15.5 (2)	O2—C20—C21—C23	40.1 (2)
N1—C5—C6—Cl1	16.44 (19)	N3—C20—C21—C23	−146.60 (16)
C4—C5—C6—Cl1	−163.61 (13)	C20—N3—C24—C25	−87.66 (18)
C6—N2—C7—C8	−91.8 (2)	C19—N3—C24—C25	88.37 (17)
C6—N2—C7—C12	92.4 (2)	C20—N3—C24—C29	93.46 (17)
C12—C7—C8—C9	−2.7 (3)	C19—N3—C24—C29	−90.50 (17)
N2—C7—C8—C9	−178.26 (15)	C29—C24—C25—C26	0.9 (2)
C12—C7—C8—C13	173.73 (16)	N3—C24—C25—C26	−177.90 (15)
N2—C7—C8—C13	−1.9 (2)	C29—C24—C25—C33	−179.32 (16)
C7—C8—C9—C10	1.7 (3)	N3—C24—C25—C33	1.9 (2)
C13—C8—C9—C10	−174.77 (18)	C24—C25—C26—C27	−1.2 (3)
C8—C9—C10—C11	0.1 (3)	C33—C25—C26—C27	179.06 (19)
C9—C10—C11—C12	−1.0 (3)	C25—C26—C27—C28	0.3 (3)
C10—C11—C12—C7	0.0 (2)	C26—C27—C28—C29	0.8 (3)
C10—C11—C12—C16	179.02 (17)	C27—C28—C29—C24	−1.0 (3)
C8—C7—C12—C11	1.9 (2)	C27—C28—C29—C30	−179.43 (18)
N2—C7—C12—C11	177.47 (14)	C25—C24—C29—C28	0.2 (2)
C8—C7—C12—C16	−177.16 (16)	N3—C24—C29—C28	178.97 (14)
N2—C7—C12—C16	−1.6 (2)	C25—C24—C29—C30	178.50 (15)
C7—C8—C13—C15	126.6 (2)	N3—C24—C29—C30	−2.7 (2)
C9—C8—C13—C15	−57.2 (3)	C28—C29—C30—C32	77.9 (3)
C7—C8—C13—C14	−107.6 (3)	C24—C29—C30—C32	−100.4 (2)
C9—C8—C13—C14	68.7 (3)	C28—C29—C30—C31	−46.5 (2)
C11—C12—C16—C17	16.3 (2)	C24—C29—C30—C31	135.2 (2)
C7—C12—C16—C17	−164.71 (16)	C26—C25—C33—C34	66.1 (3)
C11—C12—C16—C18	−108.98 (19)	C24—C25—C33—C34	−113.6 (3)
C7—C12—C16—C18	70.0 (2)	C26—C25—C33—C35	−58.8 (3)
C20—N3—C19—O1	−150.47 (16)	C24—C25—C33—C35	121.4 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C14—H14A···O1^{i}	0.96	2.45	3.392 (3)	168

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

Fig. 1

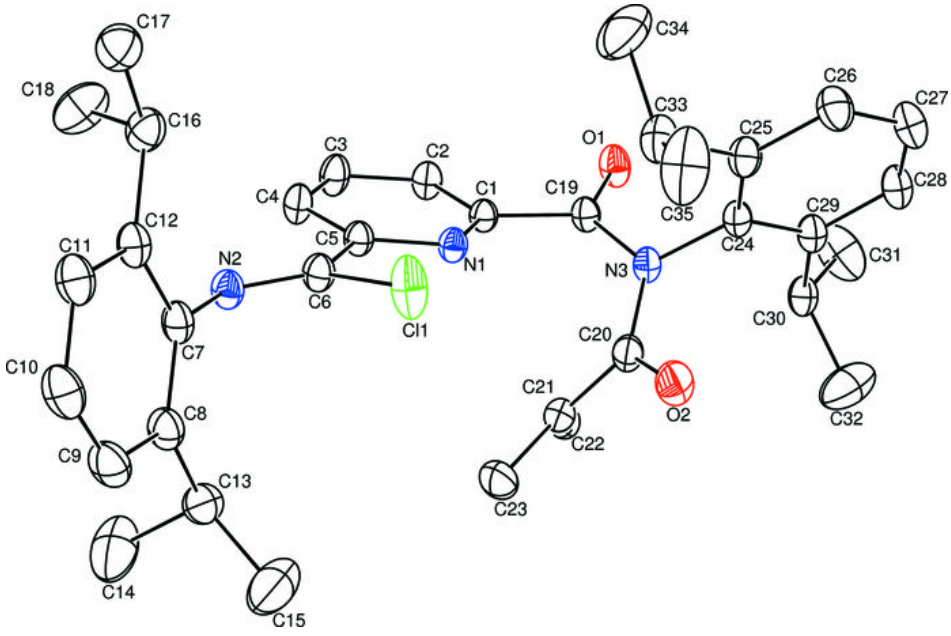


Fig. 2

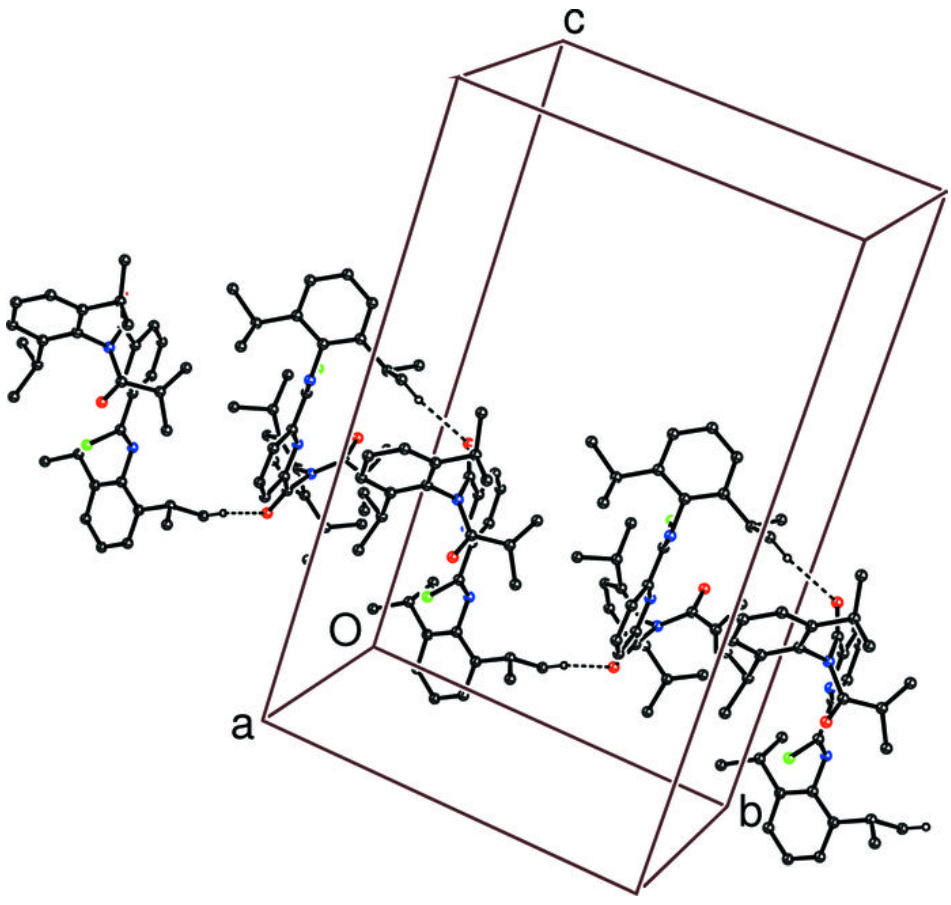


Fig. 3