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Structure Reports

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N-[4-(Di-2-pyrrolylmethyl)phenyl]-acetamide

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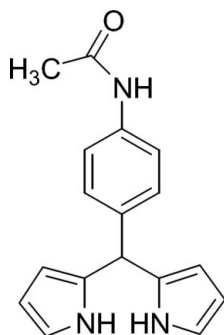
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Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.051; wR factor = 0.131; data-to-parameter ratio = 24.4.

The crystal structure of the title compound, $\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}$, shows the formation of infinite chains of molecules that are linked *via* hydrogen bonds involving the acetamide group. The carbonyl O atom forms hydrogen bonds with both a pyrrole NH group and another acetamide NH unit. In addition, the carbonyl O atom is involved in a weak intramolecular interaction with the neighboring aryl group, thus connecting different chains.

Related literature

For related literature, see: Bonnett *et al.* (1972); Lin *et al.* (1996); Bennis & Gallagher (1998); Gallagher & Moriarty (1999); Patra *et al.* (2002); Senge (2000, 2005*a,b*); Senge & Smith (2005); Shin *et al.* (2004). The compound was prepared as described by Lazzeri & Durantini (2003); crystals were handled as described by Hope (1994). The hydrogen-bonding analysis followed the criteria set out by Steiner (1997).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{17}\text{N}_3\text{O}$
 $M_r = 279.34$
 Monoclinic, $P2_1/c$
 $a = 7.4007$ (6) Å

$b = 24.858$ (2) Å
 $c = 8.2690$ (7) Å
 $\beta = 112.490$ (2)°
 $V = 1405.5$ (2) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹

$T = 90$ (2) K
 $0.50 \times 0.40 \times 0.01$ mm

Data collection

Bruker SMART APEX II diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.96$, $T_{\max} = 0.99$

20557 measured reflections
 4656 independent reflections
 3167 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.131$
 $S = 1.01$
 4656 reflections

191 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.41$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.88	2.02	2.8914 (15)	171
$\text{N3}-\text{H3B}\cdots\text{O1}^{\text{ii}}$	0.88	2.03	2.8936 (15)	169
$\text{C55}-\text{H55A}\cdots\text{O1}$	0.95	2.55	2.9708 (17)	107

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL (Siemens, 1995); software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RT2013).

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supplementary materials

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N-[4-(Di-2-pyrrolylmethyl)phenyl]acetamide

M. O. Senge

Comment

Dipyrromethanes are central synthetic building blocks for porphyrins which exhibit a rich structural chemistry (Senge, 2005*a*; Shin *et al.*, 2004). In continuation of our studies on the hydrogen bonding pattern in porphyrins and related building blocks (Senge, 2000, 2005*b*; Senge & Smith, 2005). We report here the structure of a dipyrromethane with an acceptor substituted *meso* position. Using the criteria defined by Steiner (1997) the title compound was investigated for N–H···O and C–H···O interactions. The compound forms infinite chains through hydrogen bonding of O1 to a neighboring pyrrole NH [N1–H1···O1, 2.891 (2) Å, 171°] and the acetamido NH [N3–H3···O2, 2.894 (2) Å, 169°]. Thus, the acetamido group on the *meso* substituent functions as the connecting unit in the supramolecular structure, primarily through the 3-center hydrogen bond system involving O1.

The pyrrole N–H unit of ring 2 is not involved in any hydrogen bonding. As can be seen in Fig. 1 the two pyrrole rings point in opposite directions and the two pyrrole planes are tilted by 60.1°. As shown in Fig. 2, the N2 unit is effectively shielded by the backbone of the neighboring chain of molecules and points towards the back of pyrrole ring 1 in the neighboring unit, where no acceptor atoms are present. In addition, the carbonyl oxygen atom is involved in a weak intramolecular interaction with the neighboring aryl group [C55–H55A···O1 = 2.55 (2) Å]. Related structures have been reported (Bonnert *et al.*, 1972; Lin *et al.*, 1996; Bennis & Gallagher, 1998; Gallagher & Moriarty, 1999; Patra *et al.*, 2002).

Experimental

Crystals were handled as described by Hope (1994). The compound was prepared as described earlier (Lazzeri & Durantini, 2003) and crystallized from CH₂Cl₂/n-hexane.

Refinement

H atoms were mostly placed in geometrically idealized positions and constrained to ride on their parent atoms with C–H distances in the range of 0.88–1.00 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for CH and NH groups and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl groups.

Figures

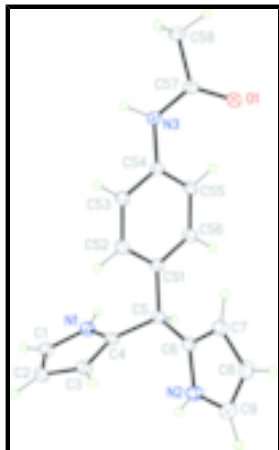


Fig. 1. A view of (I). Displacement ellipsoids for the non-hydrogen atoms are drawn at the 50% probability level.

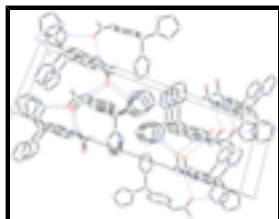


Fig. 2. View of the molecular arrangement of (I) in the crystal. Dashed lines indicate intermolecular hydrogen bonds.

N-[4-(Di-2-pyrrolylmethyl)phenyl]acetamide

Crystal data

$C_{17}H_{17}N_3O$

$M_r = 279.34$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.4007$ (6) Å

$b = 24.858$ (2) Å

$c = 8.2690$ (7) Å

$\beta = 112.490$ (2)°

$V = 1405.5$ (2) Å³

$Z = 4$

$F_{000} = 592$

$D_x = 1.320$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3237 reflections

$\theta = 5.6$ – 62.6 °

$\mu = 0.09$ mm⁻¹

$T = 90$ (2) K

Plate, yellow

$0.50 \times 0.40 \times 0.01$ mm

Data collection

Bruker SMART APEX II
diffractometer

Radiation source: sealed tube

Monochromator: graphite

Detector resolution: 8.3 pixels mm⁻¹

$T = 90$ (2) K

φ and ω scans

4656 independent reflections

3167 reflections with $I > 2\sigma(I)$

$R_{int} = 0.061$

$\theta_{max} = 31.5$ °

$\theta_{min} = 2.8$ °

$h = -10 \rightarrow 10$

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996) $k = -36 \rightarrow 36$
 $T_{\min} = 0.96, T_{\max} = 0.99$ $l = -12 \rightarrow 12$
 20557 measured reflections

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 0.3541P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
4656 reflections	$(\Delta/\sigma)_{\max} = 0.001$
191 parameters	$\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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. All hydrogen atoms were located in difference maps. All hydrogen atoms were located in difference maps. No H-acceptor was found for N2.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.09109 (16)	0.41540 (5)	0.30239 (15)	0.0171 (2)
H1A	0.0414	0.3836	0.2631	0.020*
N2	0.29445 (17)	0.43496 (5)	0.84943 (16)	0.0194 (2)
H2A	0.1802	0.4496	0.7890	0.023*
C1	0.0471 (2)	0.46203 (6)	0.2072 (2)	0.0195 (3)
H1B	-0.0407	0.4655	0.0887	0.023*
C2	0.1523 (2)	0.50284 (6)	0.31309 (19)	0.0189 (3)
H2B	0.1509	0.5396	0.2817	0.023*
C3	0.2638 (2)	0.47978 (5)	0.47852 (19)	0.0181 (3)
H3A	0.3503	0.4984	0.5784	0.022*
C4	0.22382 (18)	0.42563 (5)	0.46783 (18)	0.0154 (3)
C5	0.30492 (19)	0.38041 (5)	0.59659 (18)	0.0163 (3)
H5A	0.1932	0.3562	0.5868	0.020*

supplementary materials

C6	0.39253 (19)	0.40006 (5)	0.78285 (18)	0.0165 (3)
C7	0.5625 (2)	0.38611 (6)	0.9176 (2)	0.0205 (3)
H7A	0.6598	0.3624	0.9105	0.025*
C8	0.5673 (2)	0.41349 (6)	1.0705 (2)	0.0226 (3)
H8A	0.6677	0.4113	1.1840	0.027*
C9	0.4008 (2)	0.44351 (6)	1.0236 (2)	0.0217 (3)
H9A	0.3651	0.4663	1.0986	0.026*
C51	0.45508 (19)	0.34645 (5)	0.55772 (18)	0.0155 (3)
C52	0.5895 (2)	0.37005 (6)	0.4991 (2)	0.0202 (3)
H52A	0.5821	0.4075	0.4752	0.024*
C53	0.7340 (2)	0.33949 (6)	0.4753 (2)	0.0202 (3)
H53A	0.8263	0.3563	0.4375	0.024*
C54	0.74457 (19)	0.28430 (5)	0.50661 (17)	0.0159 (3)
C55	0.6086 (2)	0.25983 (5)	0.56148 (19)	0.0176 (3)
H55A	0.6123	0.2221	0.5802	0.021*
C56	0.46718 (19)	0.29128 (5)	0.58853 (19)	0.0177 (3)
H56A	0.3767	0.2747	0.6290	0.021*
C57	0.98507 (19)	0.21091 (5)	0.55646 (18)	0.0158 (3)
C58	1.1101 (2)	0.18235 (6)	0.4755 (2)	0.0226 (3)
H58A	1.2343	0.1717	0.5677	0.034*
H58B	1.1358	0.2066	0.3932	0.034*
H58C	1.0414	0.1503	0.4130	0.034*
N3	0.88981 (17)	0.25458 (5)	0.47060 (15)	0.0174 (2)
H3B	0.9199	0.2661	0.3833	0.021*
O1	0.97034 (14)	0.19367 (4)	0.69187 (13)	0.0187 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0165 (5)	0.0150 (5)	0.0188 (6)	-0.0017 (4)	0.0058 (5)	-0.0015 (4)
N2	0.0149 (5)	0.0209 (6)	0.0222 (6)	0.0029 (4)	0.0070 (5)	-0.0013 (5)
C1	0.0168 (6)	0.0206 (7)	0.0200 (7)	0.0026 (5)	0.0060 (6)	0.0030 (5)
C2	0.0171 (6)	0.0155 (6)	0.0242 (8)	0.0025 (5)	0.0080 (6)	0.0032 (5)
C3	0.0158 (6)	0.0144 (6)	0.0221 (7)	-0.0003 (5)	0.0051 (5)	-0.0009 (5)
C4	0.0131 (6)	0.0143 (6)	0.0191 (7)	0.0016 (5)	0.0063 (5)	0.0007 (5)
C5	0.0138 (6)	0.0140 (6)	0.0211 (7)	0.0012 (5)	0.0066 (5)	0.0008 (5)
C6	0.0158 (6)	0.0155 (6)	0.0206 (7)	0.0014 (5)	0.0095 (5)	0.0023 (5)
C7	0.0174 (6)	0.0234 (7)	0.0213 (7)	0.0050 (5)	0.0082 (6)	0.0049 (6)
C8	0.0205 (7)	0.0282 (8)	0.0182 (7)	0.0013 (6)	0.0065 (6)	0.0037 (6)
C9	0.0224 (7)	0.0240 (7)	0.0210 (7)	-0.0018 (5)	0.0107 (6)	-0.0029 (6)
C51	0.0144 (6)	0.0148 (6)	0.0161 (6)	0.0020 (5)	0.0047 (5)	-0.0003 (5)
C52	0.0210 (6)	0.0138 (6)	0.0278 (8)	0.0032 (5)	0.0117 (6)	0.0051 (6)
C53	0.0206 (6)	0.0178 (7)	0.0265 (8)	0.0037 (5)	0.0140 (6)	0.0065 (6)
C54	0.0165 (6)	0.0163 (6)	0.0151 (6)	0.0038 (5)	0.0063 (5)	0.0010 (5)
C55	0.0188 (6)	0.0122 (6)	0.0220 (7)	0.0014 (5)	0.0080 (6)	0.0009 (5)
C56	0.0157 (6)	0.0148 (6)	0.0236 (7)	-0.0005 (5)	0.0086 (6)	0.0013 (5)
C57	0.0153 (6)	0.0141 (6)	0.0184 (7)	0.0005 (5)	0.0068 (5)	-0.0019 (5)
C58	0.0239 (7)	0.0201 (7)	0.0281 (8)	0.0057 (6)	0.0147 (6)	-0.0004 (6)

N3	0.0193 (5)	0.0183 (6)	0.0173 (6)	0.0049 (4)	0.0101 (5)	0.0030 (4)
O1	0.0218 (5)	0.0170 (5)	0.0185 (5)	0.0042 (4)	0.0090 (4)	0.0026 (4)

Geometric parameters (Å, °)

N1—C4	1.3686 (18)	C8—H8A	0.9500
N1—C1	1.3685 (18)	C9—H9A	0.9500
N1—H1A	0.8800	C51—C56	1.3914 (18)
N2—C9	1.368 (2)	C51—C52	1.3917 (18)
N2—C6	1.3741 (17)	C52—C53	1.3859 (19)
N2—H2A	0.8800	C52—H52A	0.9500
C1—C2	1.371 (2)	C53—C54	1.3926 (19)
C1—H1B	0.9500	C53—H53A	0.9500
C2—C3	1.420 (2)	C54—C55	1.3921 (18)
C2—H2B	0.9500	C54—N3	1.4265 (16)
C3—C4	1.3737 (19)	C55—C56	1.3913 (18)
C3—H3A	0.9500	C55—H55A	0.9500
C4—C5	1.5061 (19)	C56—H56A	0.9500
C5—C6	1.506 (2)	C57—O1	1.2421 (16)
C5—C51	1.5245 (18)	C57—N3	1.3402 (17)
C5—H5A	1.0000	C57—C58	1.5110 (18)
C6—C7	1.368 (2)	C58—H58A	0.9800
C7—C8	1.424 (2)	C58—H58B	0.9800
C7—H7A	0.9500	C58—H58C	0.9800
C8—C9	1.364 (2)	N3—H3B	0.8800
C4—N1—C1	109.84 (12)	C8—C9—N2	107.93 (13)
C4—N1—H1A	125.1	C8—C9—H9A	126.0
C1—N1—H1A	125.1	N2—C9—H9A	126.0
C9—N2—C6	109.90 (12)	C56—C51—C52	118.34 (12)
C9—N2—H2A	125.1	C56—C51—C5	120.55 (12)
C6—N2—H2A	125.1	C52—C51—C5	121.04 (12)
N1—C1—C2	107.90 (13)	C53—C52—C51	120.76 (13)
N1—C1—H1B	126.0	C53—C52—H52A	119.6
C2—C1—H1B	126.0	C51—C52—H52A	119.6
C1—C2—C3	107.16 (13)	C52—C53—C54	120.35 (12)
C1—C2—H2B	126.4	C52—C53—H53A	119.8
C3—C2—H2B	126.4	C54—C53—H53A	119.8
C4—C3—C2	107.58 (13)	C55—C54—C53	119.65 (12)
C4—C3—H3A	126.2	C55—C54—N3	122.55 (12)
C2—C3—H3A	126.2	C53—C54—N3	117.70 (12)
N1—C4—C3	107.51 (12)	C54—C55—C56	119.26 (12)
N1—C4—C5	120.50 (12)	C54—C55—H55A	120.4
C3—C4—C5	131.91 (13)	C56—C55—H55A	120.4
C4—C5—C6	112.35 (11)	C51—C56—C55	121.61 (12)
C4—C5—C51	112.94 (11)	C51—C56—H56A	119.2
C6—C5—C51	109.38 (11)	C55—C56—H56A	119.2
C4—C5—H5A	107.3	O1—C57—N3	123.18 (12)
C6—C5—H5A	107.3	O1—C57—C58	121.15 (12)
C51—C5—H5A	107.3	N3—C57—C58	115.66 (12)

supplementary materials

C7—C6—N2	107.19 (13)	C57—C58—H58A	109.5
C7—C6—C5	130.77 (12)	C57—C58—H58B	109.5
N2—C6—C5	121.86 (12)	H58A—C58—H58B	109.5
C6—C7—C8	107.76 (12)	C57—C58—H58C	109.5
C6—C7—H7A	126.1	H58A—C58—H58C	109.5
C8—C7—H7A	126.1	H58B—C58—H58C	109.5
C9—C8—C7	107.22 (13)	C57—N3—C54	126.48 (11)
C9—C8—H8A	126.4	C57—N3—H3B	116.8
C7—C8—H8A	126.4	C54—N3—H3B	116.8
C4—N1—C1—C2	-0.28 (15)	C7—C8—C9—N2	0.71 (17)
N1—C1—C2—C3	-0.04 (15)	C6—N2—C9—C8	-0.77 (16)
C1—C2—C3—C4	0.34 (15)	C4—C5—C51—C56	-144.69 (13)
C1—N1—C4—C3	0.49 (15)	C6—C5—C51—C56	89.39 (15)
C1—N1—C4—C5	-176.75 (11)	C4—C5—C51—C52	38.56 (18)
C2—C3—C4—N1	-0.50 (15)	C6—C5—C51—C52	-87.36 (15)
C2—C3—C4—C5	176.30 (13)	C56—C51—C52—C53	-1.2 (2)
N1—C4—C5—C6	-160.97 (11)	C5—C51—C52—C53	175.61 (14)
C3—C4—C5—C6	22.6 (2)	C51—C52—C53—C54	1.2 (2)
N1—C4—C5—C51	74.73 (15)	C52—C53—C54—C55	0.2 (2)
C3—C4—C5—C51	-101.74 (17)	C52—C53—C54—N3	176.64 (13)
C9—N2—C6—C7	0.50 (16)	C53—C54—C55—C56	-1.7 (2)
C9—N2—C6—C5	176.08 (12)	N3—C54—C55—C56	-177.90 (13)
C4—C5—C6—C7	-136.77 (15)	C52—C51—C56—C55	-0.3 (2)
C51—C5—C6—C7	-10.5 (2)	C5—C51—C56—C55	-177.10 (13)
C4—C5—C6—N2	48.81 (16)	C54—C55—C56—C51	1.7 (2)
C51—C5—C6—N2	175.06 (12)	O1—C57—N3—C54	-7.1 (2)
N2—C6—C7—C8	-0.05 (16)	C58—C57—N3—C54	171.41 (13)
C5—C6—C7—C8	-175.09 (14)	C55—C54—N3—C57	-36.2 (2)
C6—C7—C8—C9	-0.41 (17)	C53—C54—N3—C57	147.52 (14)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O1 ⁱ	0.88	2.02	2.8914 (15)	171
N3—H3B \cdots O1 ⁱⁱ	0.88	2.03	2.8936 (15)	169
C55—H55A \cdots O1	0.95	2.55	2.9708 (17)	107

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

Fig. 1

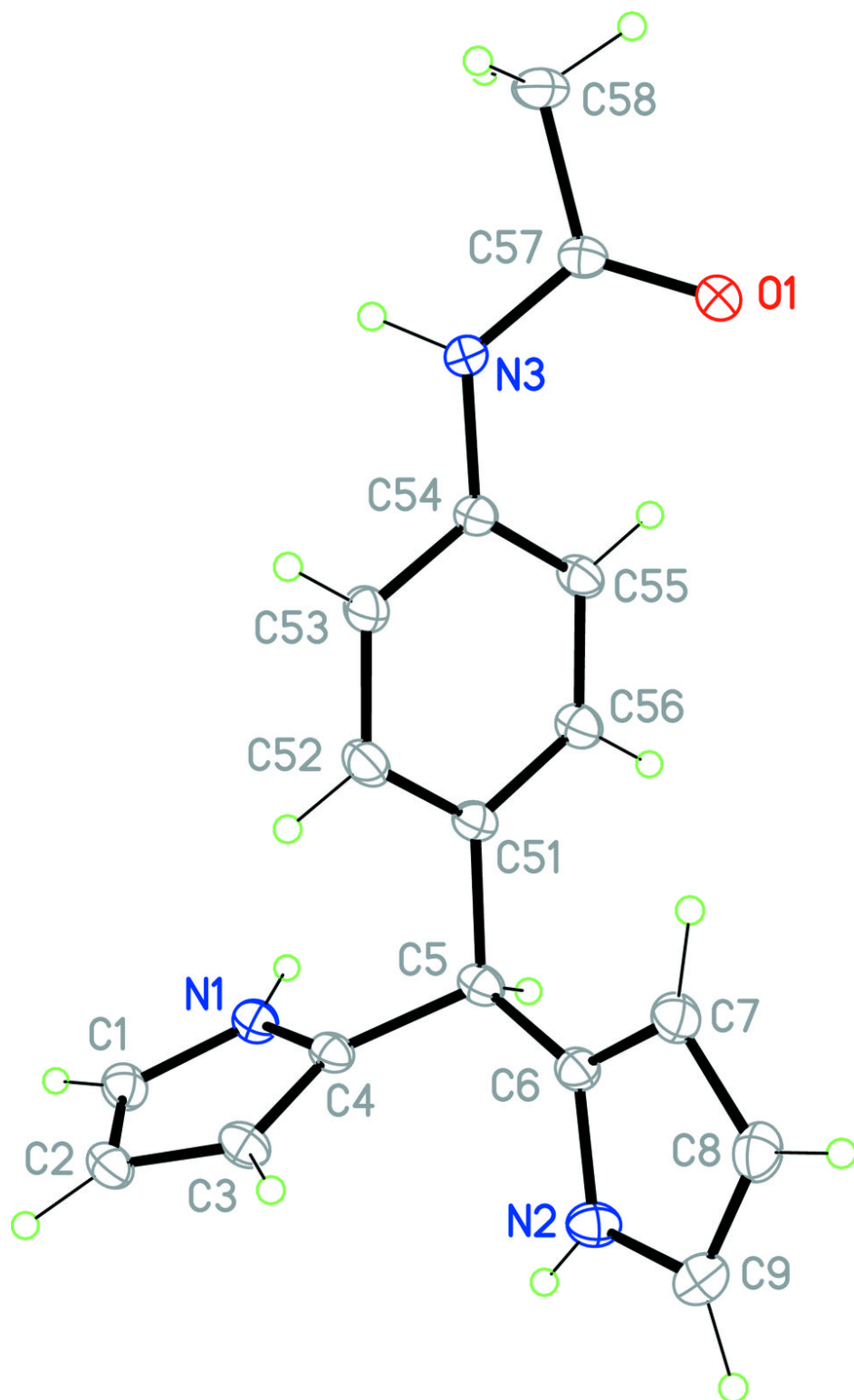


Fig. 2

