1-(4-Acetylphenyl)-2,5-dimethyl-1H-pyrrole

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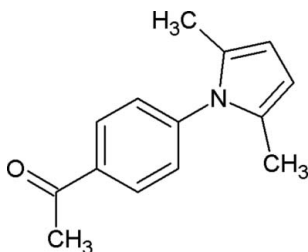Correspondence e-mail: manuela@pollux.fis.uc.pt

In the title compound, $C_{14}H_{15}NO$, the molecules are joined *via* C—H$\cdots$O interactions into chains along the *b* axis. The planes of the pyrrole and benzene rings are at an angle of 62.17 (10)° with respect to each other.

Related literature

For the use of polypyrroles as sensors, see: Anzenbacher *et al.* (2006); Zanganeh & Amini (2007). For investigations of other polypyrrole properties, see: Biagiotti *et al.* (2007); Bonfiglio *et al.* (1998); Dutta & De (2006); Kaianak (1998); Stejskal *et al.* (2004); Wang *et al.* (2001). For the synthesis of the title compound, see: Gerrius (1990); Banik *et al.* (2004).

Experimental

Crystal data

$C_{14}H_{15}NO$	V = 1184.8 (2) Å^3
M_r = 213.27	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 10.9476 (13) Å	μ = 0.08 mm^{-1}
b = 7.4005 (6) Å	T = 293 (2) K
c = 14.8518 (14) Å	$0.29 \times 0.22 \times 0.10$ mm
β = 100.043 (6)°	

Data collection

Bruker APEX CCD area-detector diffractometer	8847 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	2945 independent reflections
T_{min} = 0.984, T_{max} = 0.993	1461 reflections with $I > 2\sigma(I)$
	R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	149 parameters
$wR(F^2)$ = 0.131	H-atom parameters constrained
S = 1.00	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
2945 reflections	$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$\cdots$O1^{i}	0.93	2.47	3.338 (2)	155
C14—H14$B\cdots$O1^{ii}	0.96	2.51	3.465 (2)	172

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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supplementary materials

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A. C. Santos, A. T. Marques, M. Ramos Silva, A. Matos Beja and A. J. F. N. Sobral

Comment

Pyrroles are important compounds in the fields of macromolecular, environmental and medical chemistry, optics and nano-technologies (Kaianak, 1998; Dutta & De, 2006; Stejskal *et al.*, 2004; Bonfiglio *et al.*, 1998; Biagiotti *et al.*, 2007; Zanganeh & Amini, 2007; Anzenbacher *et al.*, 2006). For many applications pyrroles have to be able to be polymerized electrochemically and therefore a better understanding of the kinetic and thermodynamic polymerization parameters is needed (Wang *et al.*, 2001). Such parameters are affected by the acid/base characteristics of the nitrogen atom and the α and β positions, characteristics that have been determined with the help of many *N*-phenyl substituted pyrroles. (Wang *et al.*, 2001). The title compound (Fig. 1), obtained by the Paal-Knorr method (Gerrius, 1990), is such a *N*-phenyl substituted pyrrole. In each molecule the non H-atoms are distributed over two planes: the pyrrolic and the benzene phenyl ring plane. The atoms O1 and C14 are located basically within the mean plane of the six-membered aromatic ring with deviations of only 0.158 (1) and 0.113 (2) Å. The dihedral angle between the two planes is 62.2 (1)°. Due to the lack of conventional donors there are no conventional hydrogen bonds between the molecules. However, intermolecular interactions of the type C—H···O join the molecules in chains that run along the *b* axis (Fig. 2).

Experimental

2,5-Dimethyl-1-(4-acetophenyl)-1*H*-pyrrole was synthesized by the method of Paal-Knorr (Gerrius, 1990) in an way analogous to that described by Banik (Banik *et al.*, 2004) using iodine as catalyst.

Refinement

All H-atoms were positioned geometrically and refined using a riding model with C—H=0.95 Å, $U_{iso}(H)=1.2U_{eq}(C)$ for aromatic and C—H=0.96 Å, $U_{iso}(H)=1.5U_{eq}(C)$ for the methyl groups. The methyl groups were idealized based on difference electron density synthesis, then refined as a rigid group allowed to rotate but not tip.

Figures
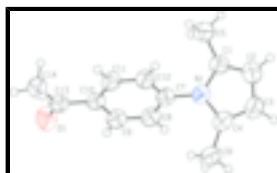

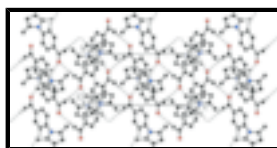Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

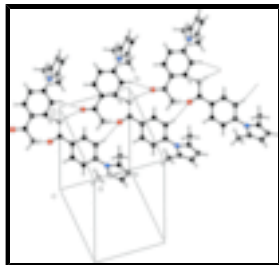Fig. 2. Packing diagram of the title compound. C—H···O interactions are shown as dashed lines.

supplementary materials

Fig. 3. One of the chains in which the molecules aggregate *via* C—H···O intermolecular interactions.

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Crystal data

$C_{14}H_{15}NO$	$F_{000} = 456$
$M_r = 213.27$	$D_x = 1.196$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.9476$ (13) Å	Cell parameters from 1357 reflections
$b = 7.4005$ (6) Å	$\theta = 2.8–22.2°$
$c = 14.8518$ (14) Å	$\mu = 0.08$ mm^{-1}
$\beta = 100.043$ (6)°	$T = 293$ (2) K
$V = 1184.8$ (2) Å^3	Plate, colourless
$Z = 4$	$0.29 \times 0.22 \times 0.10$ mm

Data collection

Bruker APEX CCD area-detector diffractometer	2945 independent reflections
Radiation source: fine-focus sealed tube	1461 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 293(2)$ K	$\theta_{max} = 28.4°$
φ and ω scans	$\theta_{min} = 2.8°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$h = -7 \rightarrow 14$
$T_{min} = 0.984$, $T_{max} = 0.993$	$k = -8 \rightarrow 9$
8847 measured reflections	$l = -19 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
2945 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}

149 parameters

$\Delta\rho_{min} = -0.14$ e Å^{-3}

Primary atom site location: structure-invariant direct methods

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.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.02792 (13)	−0.15368 (19)	0.36051 (9)	0.0795 (4)
N1	0.31347 (12)	0.52600 (19)	0.56347 (9)	0.0533 (4)
C1	0.37686 (15)	0.6603 (2)	0.52528 (12)	0.0557 (5)
C2	0.43942 (17)	0.7583 (3)	0.59568 (13)	0.0686 (5)
H2	0.4906	0.8567	0.5905	0.082*
C3	0.41374 (17)	0.6856 (3)	0.67830 (13)	0.0709 (6)
H3	0.4447	0.7284	0.7367	0.085*
C4	0.33637 (16)	0.5430 (3)	0.65807 (12)	0.0598 (5)
C5	0.37593 (17)	0.6748 (3)	0.42511 (12)	0.0690 (5)
H5A	0.4332	0.7669	0.4139	0.103*
H5B	0.2940	0.7057	0.3945	0.103*
H5C	0.4000	0.5612	0.4024	0.103*
C6	0.27643 (18)	0.4236 (3)	0.71888 (13)	0.0768 (6)
H6A	0.3174	0.3084	0.7248	0.115*
H6B	0.1906	0.4071	0.6928	0.115*
H6C	0.2828	0.4785	0.7781	0.115*
C7	0.23376 (15)	0.3954 (2)	0.51268 (11)	0.0498 (4)
C8	0.25979 (16)	0.2123 (3)	0.52272 (12)	0.0571 (5)
H8	0.3294	0.1734	0.5632	0.069*
C9	0.18239 (15)	0.0885 (2)	0.47270 (12)	0.0563 (5)
H9	0.2006	−0.0339	0.4797	0.068*
C10	0.07785 (15)	0.1421 (2)	0.41203 (10)	0.0484 (4)
C11	0.05271 (15)	0.3253 (2)	0.40330 (11)	0.0566 (5)
H11	−0.0173	0.3642	0.3632	0.068*
C12	0.12968 (15)	0.4513 (2)	0.45298 (11)	0.0560 (5)
H12	0.1114	0.5738	0.4462	0.067*
C13	−0.00238 (17)	0.0043 (3)	0.35785 (11)	0.0563 (5)
C14	−0.12147 (16)	0.0631 (3)	0.30037 (13)	0.0713 (6)
H14A	−0.1657	−0.0409	0.2731	0.107*
H14B	−0.1042	0.1428	0.2531	0.107*

H14C	−0.1711	0.1252	0.3379	0.107*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0932 (10)	0.0530 (9)	0.0885 (10)	0.0083 (8)	0.0056 (8)	−0.0106 (7)
N1	0.0544 (8)	0.0552 (9)	0.0492 (8)	0.0022 (7)	0.0064 (6)	0.0009 (7)
C1	0.0548 (10)	0.0524 (11)	0.0596 (11)	0.0059 (9)	0.0092 (9)	0.0057 (9)
C2	0.0651 (12)	0.0636 (13)	0.0757 (14)	−0.0036 (10)	0.0079 (10)	−0.0033 (11)
C3	0.0683 (12)	0.0824 (15)	0.0596 (12)	−0.0050 (12)	0.0050 (10)	−0.0136 (11)
C4	0.0558 (10)	0.0724 (14)	0.0503 (10)	0.0060 (10)	0.0070 (8)	−0.0021 (9)
C5	0.0699 (12)	0.0733 (14)	0.0652 (12)	0.0011 (11)	0.0156 (10)	0.0100 (10)
C6	0.0792 (13)	0.0970 (16)	0.0543 (11)	−0.0027 (12)	0.0122 (10)	0.0058 (11)
C7	0.0528 (10)	0.0504 (11)	0.0472 (9)	0.0053 (9)	0.0116 (8)	0.0006 (8)
C8	0.0543 (10)	0.0583 (13)	0.0564 (10)	0.0128 (9)	0.0033 (8)	0.0064 (9)
C9	0.0626 (10)	0.0493 (11)	0.0572 (10)	0.0122 (9)	0.0110 (9)	0.0025 (9)
C10	0.0547 (10)	0.0493 (11)	0.0423 (9)	0.0079 (8)	0.0117 (8)	0.0027 (8)
C11	0.0557 (10)	0.0553 (12)	0.0555 (10)	0.0092 (9)	0.0001 (8)	0.0054 (9)
C12	0.0600 (10)	0.0465 (11)	0.0592 (11)	0.0108 (9)	0.0037 (9)	0.0043 (9)
C13	0.0681 (11)	0.0545 (12)	0.0487 (10)	0.0028 (10)	0.0173 (9)	0.0023 (9)
C14	0.0754 (13)	0.0674 (15)	0.0659 (12)	−0.0088 (11)	−0.0025 (10)	0.0052 (10)

Geometric parameters (Å, °)

O1—C13	1.214 (2)	C6—H6C	0.9600
N1—C1	1.389 (2)	C7—C12	1.380 (2)
N1—C4	1.389 (2)	C7—C8	1.387 (2)
N1—C7	1.427 (2)	C8—C9	1.375 (2)
C1—C2	1.356 (2)	C8—H8	0.9300
C1—C5	1.490 (2)	C9—C10	1.385 (2)
C2—C3	1.412 (2)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.385 (2)
C3—C4	1.353 (2)	C10—C13	1.487 (2)
C3—H3	0.9300	C11—C12	1.381 (2)
C4—C6	1.495 (3)	C11—H11	0.9300
C5—H5A	0.9600	C12—H12	0.9300
C5—H5B	0.9600	C13—C14	1.493 (2)
C5—H5C	0.9600	C14—H14A	0.9600
C6—H6A	0.9600	C14—H14B	0.9600
C6—H6B	0.9600	C14—H14C	0.9600
C1—N1—C4	109.47 (15)	C12—C7—C8	119.55 (17)
C1—N1—C7	124.90 (14)	C12—C7—N1	119.81 (16)
C4—N1—C7	125.60 (14)	C8—C7—N1	120.64 (15)
C2—C1—N1	106.80 (16)	C9—C8—C7	119.81 (16)
C2—C1—C5	130.24 (17)	C9—C8—H8	120.1
N1—C1—C5	122.86 (16)	C7—C8—H8	120.1
C1—C2—C3	108.41 (18)	C8—C9—C10	121.48 (17)
C1—C2—H2	125.8	C8—C9—H9	119.3

C3—C2—H2	125.8	C10—C9—H9	119.3
C4—C3—C2	108.41 (17)	C11—C10—C9	117.98 (17)
C4—C3—H3	125.8	C11—C10—C13	122.10 (16)
C2—C3—H3	125.8	C9—C10—C13	119.92 (16)
C3—C4—N1	106.91 (16)	C12—C11—C10	121.20 (16)
C3—C4—C6	130.57 (17)	C12—C11—H11	119.4
N1—C4—C6	122.46 (17)	C10—C11—H11	119.4
C1—C5—H5A	109.5	C7—C12—C11	119.97 (16)
C1—C5—H5B	109.5	C7—C12—H12	120.0
H5A—C5—H5B	109.5	C11—C12—H12	120.0
C1—C5—H5C	109.5	O1—C13—C10	120.68 (17)
H5A—C5—H5C	109.5	O1—C13—C14	120.36 (18)
H5B—C5—H5C	109.5	C10—C13—C14	118.96 (17)
C4—C6—H6A	109.5	C13—C14—H14A	109.5
C4—C6—H6B	109.5	C13—C14—H14B	109.5
H6A—C6—H6B	109.5	H14A—C14—H14B	109.5
C4—C6—H6C	109.5	C13—C14—H14C	109.5
H6A—C6—H6C	109.5	H14A—C14—H14C	109.5
H6B—C6—H6C	109.5	H14B—C14—H14C	109.5
C4—N1—C1—C2	0.61 (18)	C4—N1—C7—C8	−63.3 (2)
C7—N1—C1—C2	178.52 (15)	C12—C7—C8—C9	0.5 (2)
C4—N1—C1—C5	177.38 (15)	N1—C7—C8—C9	−179.52 (14)
C7—N1—C1—C5	−4.7 (2)	C7—C8—C9—C10	−0.1 (2)
N1—C1—C2—C3	−0.6 (2)	C8—C9—C10—C11	−0.3 (2)
C5—C1—C2—C3	−177.04 (17)	C8—C9—C10—C13	178.87 (14)
C1—C2—C3—C4	0.4 (2)	C9—C10—C11—C12	0.4 (2)
C2—C3—C4—N1	0.0 (2)	C13—C10—C11—C12	−178.77 (14)
C2—C3—C4—C6	−177.10 (18)	C8—C7—C12—C11	−0.4 (2)
C1—N1—C4—C3	−0.38 (18)	N1—C7—C12—C11	179.61 (14)
C7—N1—C4—C3	−178.27 (15)	C10—C11—C12—C7	−0.1 (2)
C1—N1—C4—C6	177.01 (16)	C11—C10—C13—O1	173.12 (16)
C7—N1—C4—C6	−0.9 (3)	C9—C10—C13—O1	−6.0 (2)
C1—N1—C7—C12	−60.9 (2)	C11—C10—C13—C14	−7.2 (2)
C4—N1—C7—C12	116.66 (18)	C9—C10—C13—C14	173.60 (15)
C1—N1—C7—C8	119.11 (18)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C12—H12⋯O1[i]	0.93	2.47	3.338 (2)	155
C14—H14B⋯O1[ii]	0.96	2.51	3.465 (2)	172

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

Fig. 1

Fig. 2

Fig. 3