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2-(3,4,5-Trimethoxyphenyl)-1*H*-pyrrolo-[2,3-*b*]pyridine

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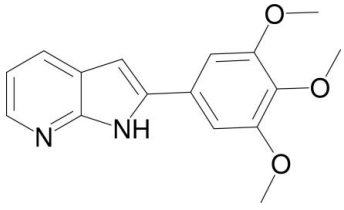
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.125; data-to-parameter ratio = 13.8.

In the title compound, $C_{16}H_{16}N_2O_3$, the 3,4,5-trimethoxyphenyl group makes a dihedral angle of 10.04 (7)° toward the 1*H*-pyrrolo[2,3-*b*]pyridine system. The crystal structure displays intermolecular N—H···N hydrogen bonds, forming inversion dimers.

Related literature

For the synthesis of the title copmpound, see: Davis *et al.* (1992)



Experimental

Crystal data

$C_{16}H_{16}N_2O_3$
M_r = 284.31
Monoclinic, $P2_1/c$
a = 7.6283 (9) Å
b = 10.1745 (4) Å
c = 18.604 (2) Å
β = 104.778 (6)°
V = 1396.2 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.78 mm^{-1}
T = 193 K
0.40 × 0.40 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2865 measured reflections
2657 independent reflections
2352 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.07
2657 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N7^{i}	0.95	2.12	3.061 (2)	171

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CORINC* (Dräger & Gattow, 1971); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2158).

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supporting information

Acta Cryst. (2009). E**65**, o3097 [doi:10.1107/S1600536809047540]

2-(3,4,5-Trimethoxyphenyl)-1*H*-pyrrolo[2,3-*b*]pyridine

Roland Selig, Dieter Schollmeyer, Wolfgang Albrecht and Stefan Laufer

S1. Comment

7-Azaindoles are found in natural and synthetic compounds of biological interest. The interest in 7-azaindoles as indole analogues has arisen in recent years due to their improved physicochemical and pharmacological properties. The substitution of this heterocycle is widely studied and used in synthesis of many compounds of potential pharmaceutical interest. The 3,4,5-trimethoxyphenyl moiety encloses a dihedral angle of 10.04 (7)° toward the 1*H*-pyrrolo[2,3-*b*]pyridine system. The crystal structure of 2-(3,4,5-trimethoxyphenyl)-1*H*-pyrrolo[2,3-*b*]pyridine, $C_{16}H_{16}N_2O_3$, is characterized by an intermolecular hydrogen bond N1–H1···N7 (2.12 Å).

S2. Experimental

3-Methylpyridine (0.68 g 7.25 mmol) was added dropwise to a freshly prepared solution of LDA in THF (2*M*) (3.6 ml 7.25 mmol) at 273 K. The resulting suspension was stirred at 273 K for 30 min. Trimethoxybenzonitrile (1.4 g 7.25 mmol) was added dropwise at such a rate that the temperature did not rise above 283 K. Stirring was continued for 60 min. at 273 K. Another portion of LDA solution (3.6 ml 7.25 mmol) was added and stirring was continued for 10 h at 353 K. The final reaction mixture was allowed to cool and ice-water was added. The mixture was extracted with ethylacetate and the combined extracts were dried (Na_2SO_4) and the solvent was evaporated under reduced pressure. The residue was subjected to flash chromatography. The title compound was obtained in a yield of 67% (1.37 g 4.83 mmol). Crystals suitable for X-ray diffraction were obtained by slow evaporation of the solvent (methanol) during several weeks.

S3. Refinement

Hydrogen atoms attached to carbon were placed at calculated positions with C—H = 0.95 Å (aromatic) or 0.98–0.99 Å (sp^3 C-atom). The H atom attached to N1 was located in difference Fourier maps. All H atoms were refined using the riding-model approximation with isotropic displacement parameters (set at 1.2–1.5 times of the U_{eq} of the parent atom).

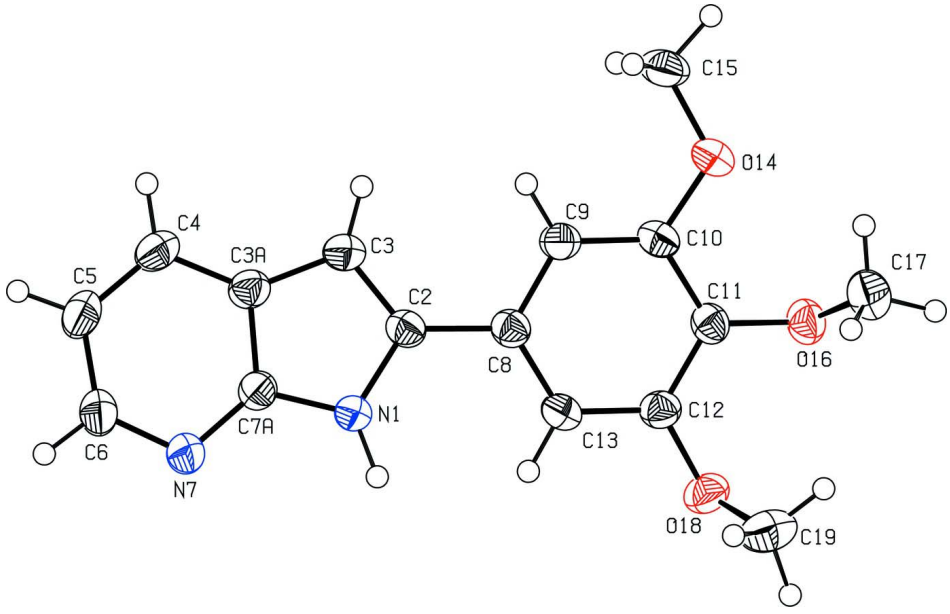

Figure 1

View of compound **I**. Displacement ellipsoids are drawn at the 50% probability level.

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$c = 18.604\ (2)$ Å
$\beta = 104.778\ (6)°$
$V = 1396.2\ (2)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.353$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 66–70°$
$\mu = 0.78$ mm^{-1}
$T = 193$ K
Plate, colourless
0.40 × 0.40 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: rotating anode
Graphite monochromator
$\omega/2\theta$ scans
2865 measured reflections
2657 independent reflections
2352 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 70.0°$, $\theta_{min} = 4.9°$
$h = -9\rightarrow0$
$k = 0\rightarrow12$
$l = -21\rightarrow22$
3 standard reflections every 60 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.125$
$S = 1.07$
2657 reflections
193 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.5259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.62152 (18)	0.53967 (13)	0.42466 (7)	0.0312 (3)
H1	0.6399	0.4802	0.4653	0.037*
C2	0.7269 (2)	0.55266 (16)	0.37403 (8)	0.0308 (3)
C3	0.6670 (2)	0.65831 (17)	0.32906 (9)	0.0360 (4)
H3	0.7158	0.6888	0.2899	0.043*
C3A	0.5187 (2)	0.71430 (17)	0.35131 (9)	0.0344 (4)
C4	0.4017 (3)	0.82022 (19)	0.32915 (10)	0.0451 (4)
H4	0.4120	0.8762	0.2896	0.054*
C5	0.2705 (3)	0.8406 (2)	0.36686 (11)	0.0524 (5)
H5	0.1888	0.9123	0.3536	0.063*
C6	0.2568 (3)	0.7568 (2)	0.42413 (11)	0.0492 (5)
H6	0.1629	0.7736	0.4481	0.059*
N7	0.3663 (2)	0.65397 (15)	0.44823 (8)	0.0381 (4)
C7A	0.4931 (2)	0.63661 (16)	0.41105 (9)	0.0310 (3)
C8	0.8710 (2)	0.45896 (16)	0.37109 (8)	0.0307 (3)
C9	0.9838 (2)	0.48432 (16)	0.32401 (9)	0.0330 (4)
H9	0.9711	0.5640	0.2966	0.040*
C10	1.1137 (2)	0.39363 (17)	0.31741 (9)	0.0324 (4)
C11	1.1358 (2)	0.27617 (16)	0.35828 (9)	0.0313 (3)
C12	1.0243 (2)	0.25251 (16)	0.40535 (9)	0.0326 (4)
C13	0.8925 (2)	0.34277 (16)	0.41142 (9)	0.0338 (4)
H13	0.8164	0.3249	0.4435	0.041*
O14	1.22631 (17)	0.40749 (13)	0.27123 (7)	0.0439 (3)
C15	1.1984 (3)	0.5172 (2)	0.22231 (10)	0.0454 (5)
H15A	1.0720	0.5186	0.1929	0.068*
H15B	1.2787	0.5103	0.1889	0.068*
H15C	1.2253	0.5984	0.2513	0.068*
O16	1.25139 (16)	0.17927 (12)	0.34718 (7)	0.0376 (3)
C17	1.4407 (2)	0.2090 (2)	0.37068 (11)	0.0458 (4)
H17A	1.4722	0.2711	0.3357	0.069*

H17B	1.5110	0.1281	0.3721	0.069*
H17C	1.4684	0.2484	0.4204	0.069*
O18	1.03203 (16)	0.13515 (12)	0.44277 (7)	0.0399 (3)
C19	1.1896 (3)	0.1164 (2)	0.50170 (11)	0.0518 (5)
H19A	1.2953	0.1062	0.4813	0.078*
H19B	1.1753	0.0372	0.5297	0.078*
H19C	1.2073	0.1928	0.5349	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0357 (7)	0.0303 (7)	0.0307 (7)	0.0044 (5)	0.0140 (5)	0.0038 (5)
C2	0.0334 (8)	0.0312 (8)	0.0295 (7)	−0.0014 (6)	0.0112 (6)	−0.0015 (6)
C3	0.0419 (9)	0.0371 (9)	0.0310 (8)	0.0024 (7)	0.0134 (7)	0.0049 (7)
C3A	0.0397 (9)	0.0348 (8)	0.0283 (8)	0.0025 (7)	0.0082 (6)	0.0019 (6)
C4	0.0566 (11)	0.0429 (10)	0.0369 (9)	0.0136 (9)	0.0135 (8)	0.0129 (8)
C5	0.0596 (12)	0.0519 (12)	0.0470 (10)	0.0276 (10)	0.0161 (9)	0.0138 (9)
C6	0.0513 (11)	0.0563 (12)	0.0445 (10)	0.0241 (9)	0.0203 (8)	0.0092 (9)
N7	0.0427 (8)	0.0400 (8)	0.0345 (7)	0.0116 (6)	0.0149 (6)	0.0044 (6)
C7A	0.0346 (8)	0.0298 (8)	0.0281 (7)	0.0025 (6)	0.0072 (6)	−0.0008 (6)
C8	0.0328 (8)	0.0304 (8)	0.0301 (7)	−0.0012 (6)	0.0101 (6)	−0.0018 (6)
C9	0.0383 (8)	0.0310 (8)	0.0317 (8)	−0.0001 (7)	0.0127 (7)	0.0024 (6)
C10	0.0358 (8)	0.0356 (9)	0.0289 (7)	−0.0028 (7)	0.0138 (6)	−0.0018 (6)
C11	0.0334 (8)	0.0300 (8)	0.0318 (8)	0.0003 (6)	0.0104 (6)	−0.0045 (6)
C12	0.0361 (8)	0.0287 (8)	0.0339 (8)	−0.0024 (6)	0.0109 (6)	0.0016 (6)
C13	0.0347 (8)	0.0360 (9)	0.0346 (8)	−0.0002 (7)	0.0162 (7)	0.0027 (7)
O14	0.0502 (7)	0.0475 (8)	0.0431 (7)	0.0089 (6)	0.0285 (6)	0.0092 (6)
C15	0.0417 (9)	0.0565 (12)	0.0424 (10)	−0.0042 (8)	0.0184 (8)	0.0123 (8)
O16	0.0388 (6)	0.0343 (6)	0.0422 (6)	0.0041 (5)	0.0148 (5)	−0.0042 (5)
C17	0.0393 (10)	0.0520 (11)	0.0466 (10)	0.0033 (8)	0.0121 (8)	−0.0037 (9)
O18	0.0414 (7)	0.0327 (6)	0.0457 (7)	−0.0015 (5)	0.0113 (5)	0.0095 (5)
C19	0.0540 (12)	0.0544 (12)	0.0426 (10)	−0.0083 (9)	0.0041 (9)	0.0139 (9)

Geometric parameters (Å, º)

N1—C7A	1.367 (2)	C10—O14	1.3681 (19)
N1—C2	1.3917 (19)	C10—C11	1.403 (2)
N1—H1	0.9499	C11—O16	1.3729 (19)
C2—C3	1.367 (2)	C11—C12	1.389 (2)
C2—C8	1.467 (2)	C12—O18	1.3758 (19)
C3—C3A	1.419 (2)	C12—C13	1.387 (2)
C3—H3	0.9500	C13—H13	0.9500
C3A—C4	1.393 (2)	O14—C15	1.422 (2)
C3A—C7A	1.417 (2)	C15—H15A	0.9800
C4—C5	1.377 (3)	C15—H15B	0.9800
C4—H4	0.9500	C15—H15C	0.9800
C5—C6	1.389 (3)	O16—C17	1.430 (2)
C5—H5	0.9500	C17—H17A	0.9800

C6—N7	1.343 (2)	C17—H17B	0.9800
C6—H6	0.9500	C17—H17C	0.9800
N7—C7A	1.336 (2)	O18—C19	1.418 (2)
C8—C13	1.387 (2)	C19—H19A	0.9800
C8—C9	1.400 (2)	C19—H19B	0.9800
C9—C10	1.382 (2)	C19—H19C	0.9800
C9—H9	0.9500		

C7A—N1—C2	108.43 (13)	O14—C10—C11	114.85 (14)
C7A—N1—H1	124.1	C9—C10—C11	120.66 (14)
C2—N1—H1	127.2	O16—C11—C12	119.38 (15)
C3—C2—N1	109.17 (14)	O16—C11—C10	121.53 (14)
C3—C2—C8	128.68 (14)	C12—C11—C10	118.76 (14)
N1—C2—C8	122.09 (14)	O18—C12—C13	118.16 (14)
C2—C3—C3A	107.69 (14)	O18—C12—C11	121.02 (14)
C2—C3—H3	126.2	C13—C12—C11	120.64 (15)
C3A—C3—H3	126.2	C12—C13—C8	120.57 (14)
C4—C3A—C7A	117.21 (16)	C12—C13—H13	119.7
C4—C3A—C3	136.27 (16)	C8—C13—H13	119.7
C7A—C3A—C3	106.52 (14)	C10—O14—C15	117.87 (13)
C5—C4—C3A	117.38 (16)	O14—C15—H15A	109.5
C5—C4—H4	121.3	O14—C15—H15B	109.5
C3A—C4—H4	121.3	H15A—C15—H15B	109.5
C4—C5—C6	120.36 (17)	O14—C15—H15C	109.5
C4—C5—H5	119.8	H15A—C15—H15C	109.5
C6—C5—H5	119.8	H15B—C15—H15C	109.5
N7—C6—C5	124.90 (17)	C11—O16—C17	116.03 (13)
N7—C6—H6	117.5	O16—C17—H17A	109.5
C5—C6—H6	117.6	O16—C17—H17B	109.5
C7A—N7—C6	113.64 (15)	H17A—C17—H17B	109.5
N7—C7A—N1	125.32 (14)	O16—C17—H17C	109.5
N7—C7A—C3A	126.50 (15)	H17A—C17—H17C	109.5
N1—C7A—C3A	108.18 (14)	H17B—C17—H17C	109.5
C13—C8—C9	119.24 (15)	C12—O18—C19	115.25 (14)
C13—C8—C2	121.36 (14)	O18—C19—H19A	109.5
C9—C8—C2	119.33 (14)	O18—C19—H19B	109.5
C10—C9—C8	120.12 (15)	H19A—C19—H19B	109.5
C10—C9—H9	119.9	O18—C19—H19C	109.5
C8—C9—H9	119.9	H19A—C19—H19C	109.5
O14—C10—C9	124.47 (15)	H19B—C19—H19C	109.5

C7A—N1—C2—C3	0.71 (18)	C13—C8—C9—C10	0.7 (2)
C7A—N1—C2—C8	−176.55 (14)	C2—C8—C9—C10	−176.40 (14)
N1—C2—C3—C3A	−0.21 (19)	C8—C9—C10—O14	177.84 (15)
C8—C2—C3—C3A	176.82 (16)	C8—C9—C10—C11	−0.8 (2)
C2—C3—C3A—C4	179.8 (2)	O14—C10—C11—O16	−5.2 (2)
C2—C3—C3A—C7A	−0.35 (19)	C9—C10—C11—O16	173.52 (14)
C7A—C3A—C4—C5	−0.2 (3)	O14—C10—C11—C12	−178.56 (14)

C3—C3A—C4—C5	179.6 (2)	C9—C10—C11—C12	0.2 (2)
C3A—C4—C5—C6	−0.6 (3)	O16—C11—C12—O18	2.1 (2)
C4—C5—C6—N7	1.1 (4)	C10—C11—C12—O18	175.55 (14)
C5—C6—N7—C7A	−0.7 (3)	O16—C11—C12—C13	−172.96 (15)
C6—N7—C7A—N1	179.77 (17)	C10—C11—C12—C13	0.5 (2)
C6—N7—C7A—C3A	−0.1 (3)	O18—C12—C13—C8	−175.80 (14)
C2—N1—C7A—N7	179.17 (15)	C11—C12—C13—C8	−0.6 (2)
C2—N1—C7A—C3A	−0.92 (18)	C9—C8—C13—C12	0.0 (2)
C4—C3A—C7A—N7	0.6 (3)	C2—C8—C13—C12	177.04 (15)
C3—C3A—C7A—N7	−179.31 (16)	C9—C10—O14—C15	−5.3 (2)
C4—C3A—C7A—N1	−179.33 (15)	C11—C10—O14—C15	173.38 (15)
C3—C3A—C7A—N1	0.79 (18)	C12—C11—O16—C17	−117.52 (17)
C3—C2—C8—C13	−167.47 (17)	C10—C11—O16—C17	69.2 (2)
N1—C2—C8—C13	9.2 (2)	C13—C12—O18—C19	−113.18 (18)
C3—C2—C8—C9	9.5 (3)	C11—C12—O18—C19	71.7 (2)
N1—C2—C8—C9	−173.77 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N7^{i}	0.95	2.12	3.061 (2)	171

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