

Andrew Duthie,^a Peter Scammells,^a Andrew Katsifis^b and Edward R. T. Tiekink^{c*}^aCentre for Chiral and Molecular Technologies, Deakin University, Geelong, Victoria 3217, Australia, ^bRadiopharmaceuticals, ANSTO, Private Bag 1, Menai, NSW 2234, Australia, and ^cDepartment of Chemistry, The University of Adelaide, Australia 5005Correspondence e-mail:
edward.tiekink@adelaide.edu.au

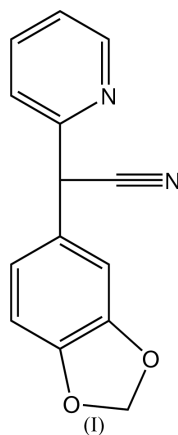
Key indicators

Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C})$ = 0.002 Å
R factor = 0.039
wR factor = 0.139
Data-to-parameter ratio = 15.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.(1,3-Benzo[*d*]dioxol-5-yl)(2-pyridyl)methyl cyanide

In the title compound, $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2$, the dihedral angle formed between the substituted pyridine ring and 1,3-benzodioxole group is $67.73(6)^\circ$. The crystal features chains of molecules held together by alternating $\pi \cdots \pi$ and $\text{C}-\text{H} \cdots \pi$ interactions.

Comment

The title compound, (I), is an intermediate in the synthesis of potential cocaine antagonists. It was prepared by a nucleophilic substitution reaction involving 2-bromopyridine and 1,3-benzodioxol-5-ylmethyl cyanide. The mean deviation of the atoms from the 1,3-benzodioxo group is 0.054 Å with the major deviation of 0.100 (2) Å associated with C2. The dihedral angle between this plane and that through the pyridyl group is $67.73(6)^\circ$. Molecules associate in the crystal to form chains held by alternating $\pi \cdots \pi$ and $\text{C}-\text{H} \cdots \pi$ interactions. Centrosymmetrically $(1-x, 1-y, -z)$ related 1,3-benzodioxole groups are aligned so as to place the six-membered rings in close proximity. The distance separating the ring centroids is calculated to be 3.584 (2) Å (Spek, 1990). These pairs are capped on each sides by a symmetry related H5' atom $(-x, -y, -z)$ which forms an interaction of the type $\text{C}-\text{H} \cdots \pi$ with the C3a–C7a aromatic ring so that the $\text{H} \cdots \pi$ ring centroid separation is 2.73 Å and the angle at H5' is 157° .



Experimental

To a stirred suspension of 1.12 g (46.5 mmol) of NaH in dry THF (30 ml) under dry argon gas was added a solution of 5.00 g (161.2 mmol) of 3,4-(methylenedioxy)phenylacetonitrile and 4.90 g (31.0 mmol) of 2-bromopyridine in dry THF (30 ml). The mixture was stirred at room temperature for 1 h and then at reflux overnight. After cooling, the THF was removed and water (50 ml) added while cooling in an ice bath. The aqueous layer was extracted with 3×50 ml of EtOAc and the combined organic layer washed with water

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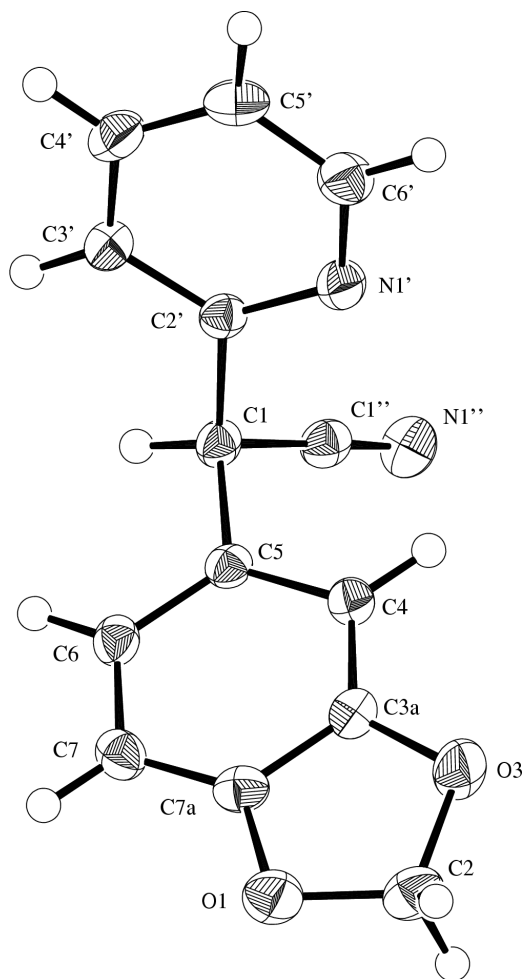


Figure 1

The molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

and then extracted with 4×30 ml of 6 M HCl solution. The combined aqueous layer was adjusted to pH 11 with 15% NaOH and extracted with 3×50 ml EtOAc. The organic layer was washed with water, dried over Na_2SO_4 , filtered and the solvent removed *in vacuo*. Column chromatography with EtOAc–hexane (1:4) gave 5.83 g (79%) of the title compound as a pale yellow powder with m.p. 338–340 K. ^1H NMR (CDCl_3) δ 5.22 (1H, s), 5.94 (2H, s), 6.78 (1H, d), 6.87 (1H, d), 6.91 (1H, dd), 7.24 (1H, ddd), 7.36 (1H, d), 7.69 (1H, ddd), 8.59 (1H, ddd). ^{13}C NMR (CDCl_3) δ 44.9, 101.4, 108.1, 108.6, 119.0, 121.2, 121.8, 123.0, 128.2, 137.5, 147.7, 148.3, 149.9, 155.4. Analysis calculated for $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2$ (238.24): C 70.58, H 4.23%; found: C 70.80, H 4.20%. ES–MS $[M + \text{H}]^+ = 239.1$. Crystals were obtained from the slow evaporation of a CDCl_3 solution of the compound.

Crystal data

$\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_2$
 $M_r = 238.25$
 Triclinic, $P\bar{1}$
 $a = 8.851(2) \text{ \AA}$
 $b = 11.435(4) \text{ \AA}$
 $c = 5.831(1) \text{ \AA}$
 $\alpha = 94.42(2)^\circ$
 $\beta = 92.79(2)^\circ$
 $\gamma = 107.65(2)^\circ$
 $V = 559.1(3) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.415 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 8.0\text{--}30.0^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 173 \text{ K}$
 Block, colourless
 $0.48 \times 0.48 \times 0.11 \text{ mm}$

Data collection

Rigaku AFC-7R diffractometer
 ω – 2θ scans
 2731 measured reflections
 2564 independent reflections
 1911 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.05$
 $\theta_{\text{max}} = 27.5^\circ$

$h = -11 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = 0 \rightarrow 7$
 3 standard reflections every 400 reflections
 intensity decay: 0.2%

Refinement

Refinement on F^2
 $R(F) = 0.039$
 $wR(F^2) = 0.139$
 $S = 1.05$
 2564 reflections
 163 parameters

H-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

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