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3-(1,3-Dioxolan-2-yl)-6,7-dimethoxyquinoline

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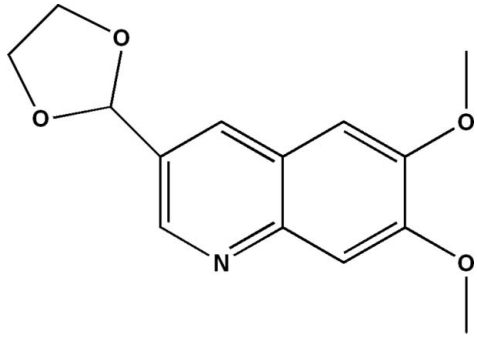
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 13.8.

In the molecule of the title compound, $C_{14}H_{15}NO_4$, the quinoline ring system and methoxy substituents are located on a crystallographic mirror plane. The 1,3-dioxolane ring adopts an envelope conformation.

Related literature

For general background, see: Kayirere *et al.* (1998); Kidwai *et al.* (2000); Zhao *et al.* (2005); Charris *et al.* (2005); Cunico *et al.* (2006); Musiol *et al.* (2006); Chen *et al.* (2006). For synthesis see: Meth-Cohn *et al.* (1981); Meth-Cohn (1993). For related structures, see: Bouraiou *et al.* (2007*a*,*b*).

For related literature, see: Bailey *et al.* (1979); Burkhaller & Edgerton (1951); Dade *et al.* (2001); Garden *et al.* (2007); Jain *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{15}NO_4$
M_r = 261.27
Orthorhombic, *Pnma*
a = 13.847 (2) Å
b = 7.0960 (12) Å
c = 12.467 (2) Å
V = 1225.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.7 × 0.65 × 0.6 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 2002)
T_{min} = 0.921, T_{max} = 0.939
12283 measured reflections
1504 independent reflections
1378 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.07
1504 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2203).

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supplementary materials

Acta Cryst. (2007). E**63**, o3315-o3316 [**doi:10.1107/S160053680703019X**]

3-(1,3-Dioxolan-2-yl)-6,7-dimethoxyquinoline

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Comment

Quinolines are an important group of heterocyclic compounds. Among these, 2-chloro-3-formylquinolines occupy a prominent position as they are key intermediates for further (β)-annelation of a wide variety of rings and for various functional group interconversions (Meth-Cohn, 1993).

Literature survey revealed that substituted quinolines possess diverse chemotherapeutic activities including antibacterial, (Kayirere *et al.*, 1998, Kidwai *et al.*, 2000) antifungal, (Musiol *et al.*, 2006) antiamoebic, (Burkhaller *et al.*, 1951; Bailey *et al.*, 1979) antileishmanial, (Dade *et al.*, 2001; Jain *et al.*, 2005) antimalarial, (Charris *et al.*, 2005; Cunico *et al.*, 2006) and antitumor activities. (Zhao *et al.*, 2005; Chen *et al.*, 2006).

The title compound, (I) consists of a −6,7-dimethoxyquinoline unit linked to a (1,3-dioxolan-2-yl)ring (Fig. 1). The geometric parameters of (I) are in agreement with those of other structures possessing a quinolyl substituent and dioxolane previously reported in the literature (Bouraiou *et al.*, 2007*a,b*; Garden *et al.*, 2007).

The 1,3-dioxolane ring adopts a twisted conformation. The crystal structure can be described by intercalled layers stacked along the *b* axis in 1/4, 3/4. No classical hydrogen bonds were found in the crystal structure.

Experimental

The title compound was synthesized by refluxing 2-chloro-3-formyl-6,7-dimethoxyquinoline(3.6 mmol) according to the literature procedure of Meth-Cohn *et al.* (1981) with ethelyne glycol(7.1 mmol)in toluene(35 ml) for 3 h. The contents were then evoporated dryness and added the solution of $NiCl_2$, 6 H_2O(3.6 mmol),triphenylphosphine(14.4 mmol) and zinc powder(3.8 mmol) in DMF(20 ml) was stirring at 70° C for 2 h. A White solid was obtained that was purifed by chromatography ehtylacetate-pentane (3:2) Crystals suitable for X-ray analysis were obtained by slow evaporation of a toluene solution of (I).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), O.96Å (methyl), 0.97 Å (methylene) and 0.98Å (methine) with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(methyl).

Figures

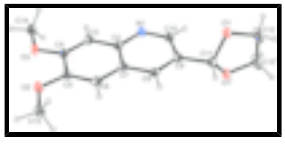

Fig. 1. The molecular structure of (I), with atom labelling scheme. Ellipsoids are drawn at the 50% probability level·H atoms are represented as small spheres of arbitrary radii. [Symmetry codes: (i) *x*, 1/2 − *y*, *z*].

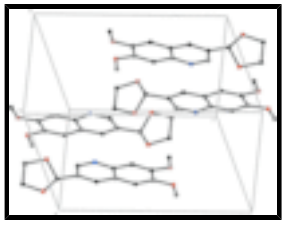

Fig. 2. Partial packing view showing the intercalled layers. H atoms have been omitted for clarity.

3-(1,3-Dioxolan-2-yl)-6,7-dimethoxyquinoline

Crystal data

$C_{14}H_{15}NO_4$	F_{000} = 552
M_r = 261.27	D_x = 1.417 Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 5615 reflections
a = 13.847 (2) Å	θ = 2.2–27.4º
b = 7.0960 (12) Å	μ = 0.10 mm^{-1}
c = 12.467 (2) Å	T = 100 (2) K
V = 1225.0 (3) Å^3	Prism, colourless
Z = 4	0.7 × 0.65 × 0.6 mm

Data collection

Bruker APEXII diffractometer	1378 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
T = 100(2) K	θ_{max} = 27.5º
CCD rotation images, thin slices scans	θ_{min} = 2.9º
Absorption correction: multi-scan SADABS (Sheldrick, 2002)	h = 0→17
T_{min} = 0.921, T_{max} = 0.939	k = 0→9
12283 measured reflections	l = 0→16
1504 independent reflections	Standard reflections: ?

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.131	$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.8241P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$
1508 reflections	$\Delta\rho_{max}$ = 0.52 e Å^{-3}
109 parameters	$\Delta\rho_{min}$ = −0.37 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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
N1	0.25658 (12)	0.2500	0.28355 (13)	0.0178 (4)	
O1	0.39995 (7)	0.08969 (14)	0.56778 (7)	0.0197 (3)	
O2	0.48367 (10)	0.2500	−0.08196 (11)	0.0216 (3)	
O3	0.30037 (10)	0.2500	−0.09922 (11)	0.0208 (3)	
C2	0.31740 (13)	0.2500	0.19680 (14)	0.0149 (4)	
C3	0.27537 (13)	0.2500	0.09301 (15)	0.0156 (4)	
H3	0.2085	0.2500	0.0857	0.019*	
C4	0.33269 (14)	0.2500	0.00341 (14)	0.0157 (4)	
C5	0.43621 (13)	0.2500	0.01377 (15)	0.0157 (4)	
C6	0.47828 (13)	0.2500	0.11308 (15)	0.0158 (4)	
H6	0.5452	0.2500	0.1193	0.019*	
C7	0.41971 (13)	0.2500	0.20700 (15)	0.0147 (4)	
C8	0.45894 (13)	0.2500	0.31164 (15)	0.0153 (4)	
H8	0.5255	0.2500	0.3216	0.018*	
C9	0.39822 (13)	0.2500	0.39823 (15)	0.0158 (4)	
C10	0.29715 (14)	0.2500	0.37939 (15)	0.0177 (4)	
H10	0.2566	0.2500	0.4388	0.021*	
C11	0.43506 (14)	0.2500	0.51138 (14)	0.0163 (4)	
H11	0.5058	0.2500	0.5115	0.020*	
C12	0.38835 (16)	0.1471 (2)	0.67633 (12)	0.0391 (5)	
H12A	0.4410	0.0995	0.7199	0.047*	
H12B	0.3280	0.0995	0.7050	0.047*	
C13	0.58676 (14)	0.2500	−0.07773 (17)	0.0233 (5)	
H13A	0.6123	0.2500	−0.1493	0.035*	
H13B	0.6086	0.1395	−0.0405	0.035*	0.50
H13C	0.6086	0.3605	−0.0405	0.035*	0.50
C14	0.19740 (14)	0.2500	−0.11326 (15)	0.0226 (4)	
H14A	0.1824	0.2500	−0.1884	0.034*	
H14B	0.1705	0.3605	−0.0804	0.034*	0.50
H14C	0.1705	0.1395	−0.0804	0.034*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0164 (8)	0.0218 (8)	0.0152 (8)	0.000	0.0019 (6)	0.000
O1	0.0308 (6)	0.0158 (5)	0.0125 (5)	−0.0005 (4)	0.0011 (3)	0.0000 (4)
O2	0.0157 (7)	0.0348 (8)	0.0142 (7)	0.000	0.0021 (5)	0.000
O3	0.0154 (7)	0.0350 (8)	0.0118 (6)	0.000	−0.0013 (5)	0.000
C2	0.0164 (9)	0.0142 (8)	0.0142 (8)	0.000	0.0008 (6)	0.000
C3	0.0139 (8)	0.0173 (9)	0.0157 (9)	0.000	0.0000 (7)	0.000
C4	0.0173 (9)	0.0163 (9)	0.0136 (8)	0.000	−0.0026 (7)	0.000
C5	0.0150 (9)	0.0174 (9)	0.0147 (9)	0.000	0.0017 (7)	0.000
C6	0.0132 (8)	0.0178 (9)	0.0165 (9)	0.000	0.0001 (6)	0.000
C7	0.0160 (9)	0.0130 (8)	0.0151 (8)	0.000	−0.0004 (7)	0.000
C8	0.0149 (8)	0.0142 (8)	0.0168 (9)	0.000	−0.0010 (7)	0.000
C9	0.0185 (9)	0.0155 (9)	0.0134 (8)	0.000	−0.0012 (7)	0.000
C10	0.0175 (9)	0.0221 (9)	0.0135 (8)	0.000	0.0030 (7)	0.000
C11	0.0182 (9)	0.0177 (9)	0.0130 (8)	0.000	−0.0007 (7)	0.000
C12	0.0831 (14)	0.0203 (9)	0.0137 (7)	−0.0020 (8)	0.0098 (7)	−0.0011 (6)
C13	0.0159 (9)	0.0344 (12)	0.0196 (9)	0.000	0.0049 (7)	0.000
C14	0.0158 (9)	0.0361 (12)	0.0158 (9)	0.000	−0.0029 (7)	0.000

Geometric parameters (Å, °)

N1—C10	1.320 (2)	C8—C9	1.368 (3)
N1—C2	1.371 (2)	C8—H8	0.9300
O1—C12	1.4225 (17)	C9—C10	1.419 (3)
O1—C11	1.4229 (15)	C9—C11	1.500 (2)
O2—C5	1.362 (2)	C10—H10	0.9300
O2—C13	1.428 (2)	C11—O1^{i}	1.4229 (15)
O3—C4	1.356 (2)	C11—H11	0.9800
O3—C14	1.437 (2)	C12—C12^{i}	1.460 (3)
C2—C3	1.419 (2)	C12—H12A	0.9700
C2—C7	1.422 (3)	C12—H12B	0.9700
C3—C4	1.370 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.439 (3)	C13—H13C	0.9600
C5—C6	1.368 (3)	C14—H14A	0.9600
C6—C7	1.424 (2)	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
C7—C8	1.413 (2)		
C10—N1—C2	116.91 (16)	N1—C10—C9	124.70 (17)
C12—O1—C11	106.24 (11)	N1—C10—H10	117.6
C5—O2—C13	116.73 (15)	C9—C10—H10	117.6
C4—O3—C14	116.27 (14)	O1—C11—O1^{i}	106.15 (14)
N1—C2—C3	117.88 (16)	O1—C11—C9	110.40 (10)
N1—C2—C7	122.77 (17)	O1^{i}—C11—C9	110.40 (10)
C3—C2—C7	119.35 (16)	O1—C11—H11	109.9

C4—C3—C2	120.39 (17)	O1^{i}—C11—H11	109.9
C4—C3—H3	119.8	C9—C11—H11	109.9
C2—C3—H3	119.8	O1—C12—C12^{i}	106.65 (8)
O3—C4—C3	125.33 (17)	O1—C12—H12A	110.4
O3—C4—C5	114.42 (16)	C12^{i}—C12—H12A	110.4
C3—C4—C5	120.25 (16)	O1—C12—H12B	110.4
O2—C5—C6	125.96 (17)	C12^{i}—C12—H12B	110.4
O2—C5—C4	113.69 (16)	H12A—C12—H12B	108.6
C6—C5—C4	120.35 (16)	O2—C13—H13A	109.5
C5—C6—C7	120.09 (17)	O2—C13—H13B	109.5
C5—C6—H6	120.0	H13A—C13—H13B	109.5
C7—C6—H6	120.0	O2—C13—H13C	109.5
C8—C7—C2	117.74 (17)	H13A—C13—H13C	109.5
C8—C7—C6	122.69 (17)	H13B—C13—H13C	109.5
C2—C7—C6	119.57 (16)	O3—C14—H14A	109.5
C9—C8—C7	119.48 (17)	O3—C14—H14B	109.5
C9—C8—H8	120.3	H14A—C14—H14B	109.5
C7—C8—H8	120.3	O3—C14—H14C	109.5
C8—C9—C10	118.39 (17)	H14A—C14—H14C	109.5
C8—C9—C11	122.21 (16)	H14B—C14—H14C	109.5
C10—C9—C11	119.41 (16)		
C12—O1—C11—O1^{i}	−27.90 (19)	C8—C9—C11—O1	−121.47 (10)
C12—O1—C11—C9	−147.55 (14)	C10—C9—C11—O1	58.53 (10)
C8—C9—C11—O1^{i}	121.47 (10)	C11—O1—C12—C12^{i}	17.06 (11)
C10—C9—C11—O1^{i}	−58.53 (10)		

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

Fig. 1

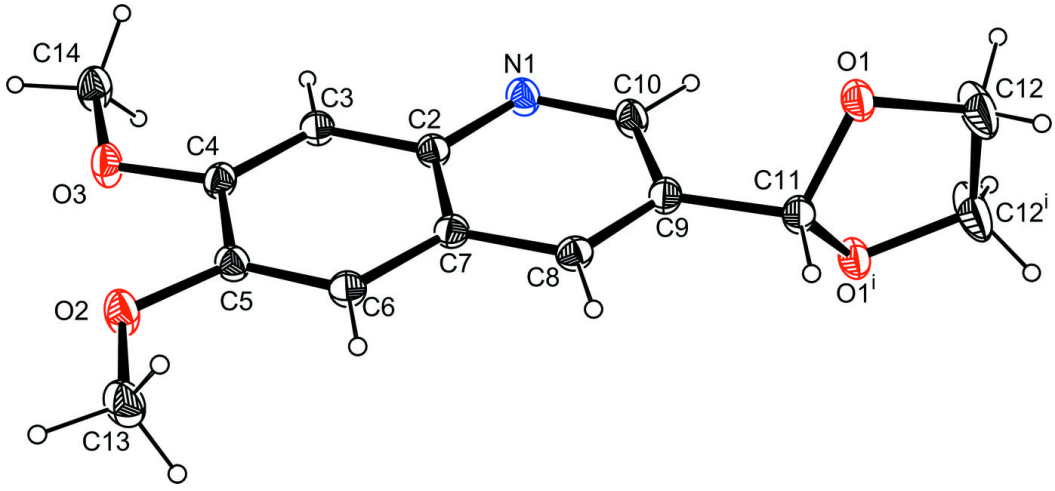

Fig. 2