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4,5,6,7,8,9-Hexamethoxyindeno[1,2,3-*ij*]isoquinoline

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Abstract

The crystal and molecular structure of the title compound (I), has been determined to confirm the molecular conformation. We established by *x*-ray investigations the planarity of the polyaromatic ring-system, which is necessary for the formation of discotic mesophases.

Comment

Aiming to develop new approaches to discotic liquid crystals, which form tilted columnar phases, exhibiting ferroelectric properties (Helfrich 1992 & Schrowsky 1994, 1995) a new heterocyclic compound (1), containing nitrogen in the aromatic core was synthesized. 4,5,6,7,8,9-Hexamethoxy-indeno[1,2,3-ij]isoquinoline (1) is a derivative of the two natural alkaloids imelutein (2) and rufescine (3) which were isolated as the first azafluoranthene alkaloids from the Amazonian vines Abuta imene and Abuta rufescine (Cava 1972,1975).

Experimental

The title compound (1) was synthesized follow the procedures described in E. Frackowiak PhD thesis (Frackowiak 1997).

Computing details

Data collection: Siemens *XSCANS*; cell refinement: Siemens *XSCANS*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

(cl)

Crystal data

$C_{21}H_{21}NO_6$	$V = 3745.9\ (6)$ Å^3
$M_r = 383.39$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$
$a = 19.133\ (2)$ Å	$\mu = 0.10$ mm^{-1}
$b = 10.2247\ (10)$ Å	$T = 293\ (2)$ K
$c = 20.0835\ (14)$ Å	0.2 × 0.15 × 0.15 mm
$\beta = 107.55\ (2)^o$	

Data collection

Siemens P4 diffractometer	$R_{int} = 0.117$

CIF access

Absorption correction: none	3 standard reflections
7131 measured reflections	every 60 min
3236 independent reflections	intensity decay: 2%
1655 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.069$	253 parameters
$wR(F^2) = 0.218$	H-atom parameters not refined
$S = 1.19$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
3235 reflections	$\Delta\rho_{min} = -0.33$ e Å^{-3}

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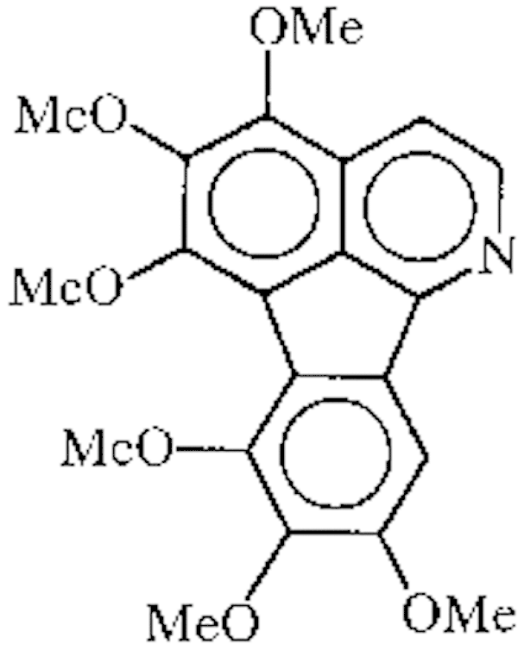
Scheme 1



supplementary materials

(cl)

Crystal data

$C_{21}H_{21}NO_6$	F_{000} = 1616
M_r = 383.39	D_x = 1.360 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 19.133 (2) Å	Cell parameters from 24 reflections
b = 10.2247 (10) Å	θ = 2.6–26.3º
c = 20.0835 (14) Å	μ = 0.10 mm^{-1}
β = 107.55 (2)º	*T* = 293 (2) K
V = 3745.9 (6) Å^3	Prism, clear
Z = 8	0.2 × 0.15 × 0.15 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.117
Radiation source: fine-focus sealed tube	θ_{max} = 24.9º
Monochromator: graphite	θ_{min} = 2.7º
T = 293(2) K	*h* = −22→22
ω scans	*k* = −11→12
Absorption correction: none	*l* = −13→23
7131 measured reflections	3 standard reflections
3236 independent reflections	every 60 min
1655 reflections with $I > 2\sigma(I)$	intensity decay: 2%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.069	H-atom parameters not refined
$wR(F^2)$ = 0.218	Calculated $w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?
S = 1.19	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
3235 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
253 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 on F^2 for ALL reflections except for 1 with very negative F^2 or flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating _R_factor_obs *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}
C1	0.8356 (2)	0.8534 (4)	0.4863 (3)	0.0610 (12)
C2	0.7804 (2)	0.8199 (3)	0.4290 (3)	0.0567 (11)
C3	0.7721 (2)	0.6870 (3)	0.4074 (2)	0.0505 (10)
C4	0.8229 (2)	0.6028 (3)	0.4491 (2)	0.0467 (9)
C5	0.8786 (2)	0.6465 (3)	0.5080 (2)	0.0487 (10)
C6	0.7184 (2)	0.6299 (3)	0.3499 (2)	0.0556 (11)
C7	0.7192 (2)	0.4967 (4)	0.3403 (2)	0.0537 (11)
C8	0.7724 (2)	0.4119 (3)	0.3869 (2)	0.0501 (10)
C9	0.8253 (2)	0.4647 (3)	0.4413 (2)	0.0477 (10)
C10	0.8884 (2)	0.4195 (3)	0.4993 (2)	0.0475 (10)
C11	0.9212 (2)	0.5296 (3)	0.5408 (2)	0.0488 (10)
C12	0.9817 (2)	0.5198 (4)	0.5980 (2)	0.0565 (11)
C13	1.0122 (2)	0.3962 (4)	0.6178 (2)	0.0590 (11)
C14	0.9812 (2)	0.2877 (3)	0.5784 (2)	0.0544 (11)
C15	0.9202 (2)	0.2969 (3)	0.5203 (2)	0.0487 (10)
C16	0.5945 (2)	0.6911 (4)	0.2927 (3)	0.0767 (15)
C17	0.6890 (2)	0.4123 (5)	0.2266 (3)	0.084 (2)
C18	0.7182 (2)	0.2090 (4)	0.3954 (3)	0.083 (2)
C19	1.1041 (3)	0.4769 (5)	0.7171 (3)	0.086 (2)
C20	0.9834 (3)	0.0968 (5)	0.6436 (3)	0.091 (2)
C21	0.9252 (2)	0.1383 (4)	0.4383 (3)	0.0758 (14)
N1	0.8869 (2)	0.7694 (3)	0.5290 (2)	0.0580 (10)
O1	0.67040 (14)	0.7125 (3)	0.3040 (2)	0.0738 (10)
O2	0.66578 (13)	0.4386 (3)	0.2854 (2)	0.0609 (8)
O3	0.76913 (14)	0.2805 (2)	0.3702 (2)	0.0623 (8)
O4	1.0722 (2)	0.3717 (3)	0.6738 (2)	0.0819 (10)
O5	1.01356 (14)	0.1661 (2)	0.5983 (2)	0.0666 (9)
O6	0.88939 (13)	0.1854 (2)	0.4861 (2)	0.0580 (8)
H11	0.8461	0.9394	0.5062	0.050*
H21	0.7461	0.8874	0.4032	0.050*
H121	1.0021	0.5962	0.6275	0.050*
H161	0.5838	0.6558	0.3281	0.050*
H162	0.5817	0.7338	0.2545	0.050*
H163	0.5812	0.6124	0.2456	0.080*
H171	0.7281	0.3759	0.2428	0.050*
H172	0.6552	0.3827	0.1973	0.050*

H173	0.7011	0.5056	0.2088	0.080*
H181	0.7409	0.1855	0.4425	0.050*
H182	0.6734	0.2268	0.3678	0.050*
H183	0.7084	0.1421	0.3508	0.080*
H191	1.1169	0.5495	0.6786	0.050*
H192	1.1403	0.4385	0.7507	0.050*
H193	1.0672	0.5221	0.7448	0.080*
H201	1.0200	0.0332	0.6784	0.050*
H202	0.9451	0.1066	0.6436	0.050*
H203	0.9689	0.0267	0.6096	0.080*
H211	0.9215	0.1856	0.3944	0.050*
H212	0.9770	0.1474	0.4605	0.080*
H213	0.9059	0.0520	0.4325	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.067 (3)	0.034 (2)	0.084 (4)	−0.001 (2)	0.025 (2)	0.002 (2)
C2	0.056 (2)	0.034 (2)	0.075 (3)	0.002 (2)	0.013 (2)	0.012 (2)
C3	0.047 (2)	0.037 (2)	0.063 (3)	0.001 (2)	0.009 (2)	0.008 (2)
C4	0.046 (2)	0.036 (2)	0.056 (3)	0.000 (2)	0.012 (2)	0.001 (2)
C5	0.045 (2)	0.037 (2)	0.062 (3)	0.000 (2)	0.014 (2)	0.002 (2)
C6	0.042 (2)	0.047 (2)	0.072 (3)	0.005 (2)	0.009 (2)	0.016 (2)
C7	0.042 (2)	0.048 (2)	0.064 (3)	−0.003 (2)	0.006 (2)	0.001 (2)
C8	0.047 (2)	0.035 (2)	0.062 (3)	−0.001 (2)	0.006 (2)	−0.002 (2)
C9	0.042 (2)	0.035 (2)	0.061 (3)	0.0014 (15)	0.007 (2)	0.002 (2)
C10	0.042 (2)	0.035 (2)	0.059 (3)	−0.0007 (15)	0.006 (2)	0.002 (2)
C11	0.042 (2)	0.043 (2)	0.057 (3)	0.001 (2)	0.010 (2)	0.002 (2)
C12	0.044 (2)	0.047 (2)	0.070 (3)	−0.003 (2)	0.005 (2)	−0.001 (2)
C13	0.044 (2)	0.058 (2)	0.064 (3)	0.004 (2)	0.001 (2)	0.004 (2)
C14	0.046 (2)	0.044 (2)	0.070 (3)	0.007 (2)	0.011 (2)	0.010 (2)
C15	0.040 (2)	0.037 (2)	0.066 (3)	−0.001 (2)	0.011 (2)	0.004 (2)
C16	0.050 (2)	0.074 (3)	0.089 (4)	0.004 (2)	−0.005 (2)	0.031 (3)
C17	0.053 (3)	0.104 (4)	0.079 (4)	0.000 (2)	−0.003 (2)	−0.022 (3)
C18	0.064 (3)	0.062 (3)	0.105 (4)	−0.020 (2)	−0.001 (3)	0.008 (3)
C19	0.076 (3)	0.092 (3)	0.067 (3)	0.001 (3)	−0.013 (3)	−0.002 (3)
C20	0.075 (3)	0.080 (3)	0.122 (5)	0.027 (3)	0.037 (3)	0.049 (3)
C21	0.073 (3)	0.054 (2)	0.101 (4)	−0.005 (2)	0.026 (3)	−0.010 (3)
N1	0.057 (2)	0.037 (2)	0.077 (3)	−0.0051 (14)	0.017 (2)	−0.005 (2)
O1	0.048 (2)	0.057 (2)	0.103 (3)	0.0035 (12)	0.003 (2)	0.031 (2)
O2	0.048 (2)	0.066 (2)	0.061 (2)	−0.0029 (12)	0.0045 (13)	−0.007 (2)
O3	0.057 (2)	0.0432 (15)	0.077 (2)	0.0028 (12)	0.0057 (14)	−0.0094 (14)
O4	0.060 (2)	0.077 (2)	0.083 (2)	0.0114 (15)	−0.016 (2)	0.003 (2)
O5	0.055 (2)	0.050 (2)	0.092 (2)	0.0154 (12)	0.017 (2)	0.021 (2)
O6	0.0490 (14)	0.0365 (13)	0.084 (2)	−0.0005 (11)	0.0127 (14)	−0.0015 (14)

Geometric parameters (Å, °)

C1—C2	1.351 (6)	C10—C15	1.401 (5)

C1—N1	1.388 (5)	C10—C11	1.429 (5)
C2—C3	1.421 (5)	C11—C12	1.366 (5)
C3—C4	1.377 (5)	C12—C13	1.399 (5)
C3—C6	1.420 (6)	C13—O4	1.365 (5)
C4—C5	1.405 (5)	C13—C14	1.388 (6)
C4—C9	1.423 (5)	C14—O5	1.393 (4)
C5—N1	1.320 (5)	C14—C15	1.382 (6)
C5—C11	1.485 (5)	C15—O6	1.370 (4)
C6—O1	1.377 (4)	C16—O1	1.418 (5)
C6—C7	1.376 (5)	C17—O2	1.409 (6)
C7—O2	1.389 (5)	C18—O3	1.427 (5)
C7—C8	1.446 (5)	C19—O4	1.402 (6)
C8—C9	1.358 (5)	C20—O5	1.408 (5)
C8—O3	1.381 (4)	C21—O6	1.421 (5)
C9—C10	1.476 (5)		
C2—C1—N1	126.5 (3)	C15—C10—C11	117.0 (3)
C1—C2—C3	119.3 (3)	C15—C10—C9	134.0 (3)
C4—C3—C6	116.2 (3)	C11—C10—C9	108.9 (3)
C4—C3—C2	114.8 (3)	C12—C11—C10	123.0 (3)
C6—C3—C2	129.0 (3)	C12—C11—C5	130.2 (3)
C3—C4—C5	122.0 (3)	C10—C11—C5	106.8 (3)
C3—C4—C9	126.5 (4)	C11—C12—C13	118.8 (3)
C5—C4—C9	111.5 (3)	O4—C13—C14	115.6 (3)
N1—C5—C4	124.0 (4)	O4—C13—C12	125.1 (4)
N1—C5—C11	128.9 (4)	C14—C13—C12	119.3 (3)
C4—C5—C11	107.1 (3)	C13—C14—O5	118.3 (3)
O1—C6—C7	123.1 (4)	C13—C14—C15	122.3 (3)
O1—C6—C3	117.7 (3)	O5—C14—C15	119.5 (4)
C7—C6—C3	119.1 (3)	O6—C15—C14	119.5 (3)
C6—C7—O2	119.9 (3)	O6—C15—C10	120.8 (3)
C6—C7—C8	122.5 (4)	C14—C15—C10	119.6 (3)
O2—C7—C8	117.6 (3)	C5—N1—C1	113.4 (3)
C9—C8—O3	123.4 (3)	C6—O1—C16	117.2 (3)
C9—C8—C7	119.4 (3)	C7—O2—C17	113.8 (3)
O3—C8—C7	117.0 (3)	C8—O3—C18	113.4 (3)
C8—C9—C4	116.2 (3)	C13—O4—C19	118.1 (3)
C8—C9—C10	138.1 (3)	C14—O5—C20	113.6 (3)
C4—C9—C10	105.7 (3)	C15—O6—C21	113.5 (3)