organic compounds

1,3,5-Tri-2-pyridylpentane-1,5-dione

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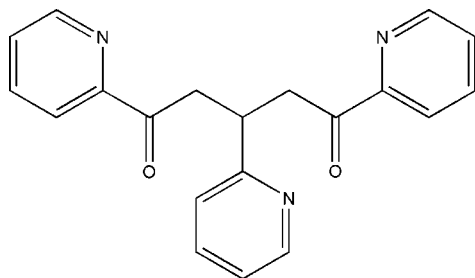Correspondence e-mail: ffj2003@163169.net

The title compound, $C_{20}H_{17}N_3O_2$, was prepared by the reaction of 1-(2-pyridyl)ethanone and pyridine-2-carbaldehyde in an ethanol solution at room temperature. The dihedral angles between the pyridine rings are 14.69 (12), 89.2 (1) and 87.1 (1)°. There are weak intermolecular C—H···O and intramolecular C—H···N hydrogen bonds in the crystal structure.

Related literature

For general background, see: Franzen (2000); Gelen *et al.* (2003); Hu *et al.* (2001); Kong *et al.* (2004); Krchna & Holladay (2002); Phoon & Sim (2002).

Experimental

Crystal data

$C_{20}H_{17}N_3O_2$	$\alpha = 101.92$ (3)°
$M_r = 331.37$	$\beta = 104.71$ (3)°
Triclinic, $P\overline{1}$	$\gamma = 111.51$ (3)°
$a = 8.3830$ (17) Å	$V = 837.3$ (3) Å^3
$b = 10.750$ (2) Å	$Z = 2$
$c = 10.950$ (2) Å	Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}	$0.20 \times 0.15 \times 0.11$ mm
$T = 295$ (2) K	

Data collection

Enraf–Nonius CAD-4 diffractometer	2353 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.018$
4623 measured reflections	3 standard reflections
3084 independent reflections	every 100 reflections
	intensity decay: 0.1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	226 parameters
$wR(F^2) = 0.113$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{max} = 0.13$ e Å^{-3}
3084 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O1^i	0.93	2.51	3.322 (3)	146
C11—H11A···O2^{ii}	0.93	2.55	3.415 (2)	155
C14—H14B···N2	0.97	2.48	2.836 (2)	101
C14—H14B···N3	0.97	2.56	2.908 (2)	101

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

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supplementary materials

1,3,5-Tri-2-pyridylpentane-1,5-dione

F.-F. Jian, J.-H. Wang, X.-Y. Yang and Y.-X. Wei

Comment

In the previous research on Dracena cochinchinensit, we found that chalcone and it derivative have significant antifungal activities (Hu *et al.*, 2001). They are important constituents commonly existing in biologically active natural products and synthetic compounds of medicinal interest (Kong *et al.*, 2004). In recent years, various protocols for the construction of the compounds *via* solid-phase strategies have been reported (Gelen *et al.*, 2003; Krchna & Holladay, 2002; Phoon & Sim, 2002; Franzen, 2000). The title compound has been synthesized and its crystal structure is presented here.

In the molecular structure (Fig. 1), the dihedral angles between pyridine (N1, C1, C2, C3, C4 and C5) and pyridine (N2, C16, C17, C18, C19 and C20) rings with the plane through the pyridine ring (N3, C9, C10, C11, C12 and C13) are 87.13 (10) and 89.15 (10)°, respectively. The C6═O1 bond length of 1.2078 (19) Å and C15═O2 bond length of 1.2066 (18) Å indicate the typical double bonds. Intramolecular C—H⋯N as well as intermolecular C—H⋯O hydrogen bonds are observed in the crystal structure.

Experimental

A mixture of 1-pyridin-2-yl-ethanone (0.02 mol) and pyridine-2-carbaldehyde (0.01 mol) and sodium hydroxide (0.034 mol) in ethanol (30 ml) was stirred at 293 K for 3 h. The hydrochloric acid solution (0.05 *M*) was dropped into the above solution to pH = 6 to get the title compound (1.0 g, yield 30%). Single crystals were obtained by recrystallization from an ethanol solution at room temperature.

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.93–0.98 Å, U_{iso}(H) = 1.2U_{eq}(C).
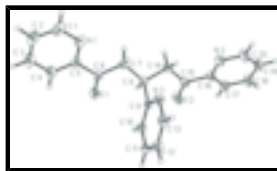
Figures

Fig. 1. The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

1,3,5-Tri-2-pyridylpentane-1,5-dione

Crystal data

$C_{20}H_{17}N_3O_2$	$Z = 2$

supplementary materials

$M_r = 331.37$	$F_{000} = 348$
Triclinic, $P\bar{1}$	$D_x = 1.314$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 124 K
$a = 8.3830$ (17) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.750$ (2) Å	Cell parameters from 25 reflections
$c = 10.950$ (2) Å	$\theta = 4-14°$
$\alpha = 101.92$ (3)°	$\mu = 0.09$ mm^{-1}
$\beta = 104.71$ (3)°	$T = 295$ (2) K
$\gamma = 111.51$ (3)°	Block, colourless
$V = 837.3$ (3) Å^3	$0.20 \times 0.15 \times 0.11$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.018$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.5°$
Monochromator: graphite	$\theta_{min} = 2.0°$
$T = 295(2)$ K	$h = -10 \rightarrow 9$
$\omega/2\theta$ scans	$k = -7 \rightarrow 13$
Absorption correction: none	$l = -13 \rightarrow 13$
4623 measured reflections	3 standard reflections
3084 independent reflections	every 100 reflections
2353 reflections with $I > 2\sigma(I)$	intensity decay: 0.1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o{}^2) + (0.0477P)^2 + 0.1482P]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3084 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.21$ 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 > 2sigma(F^2) is used only for calculat-

ing *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	0.57178 (18)	−0.27893 (12)	0.07393 (13)	0.0640 (4)
O2	0.78779 (19)	0.19726 (12)	0.04347 (12)	0.0612 (4)
N1	0.41992 (19)	−0.30815 (15)	0.34078 (14)	0.0560 (4)
N2	0.8417 (2)	0.45083 (15)	0.32752 (14)	0.0557 (4)
N3	0.97442 (18)	0.09329 (14)	0.36515 (13)	0.0478 (3)
C1	0.3457 (3)	−0.4064 (2)	0.3933 (2)	0.0721 (6)
H1B	0.3208	−0.3782	0.4697	0.087*
C2	0.3047 (3)	−0.5456 (2)	0.3409 (3)	0.0811 (7)
H2B	0.2529	−0.6102	0.3810	0.097*
C3	0.3399 (3)	−0.5888 (2)	0.2301 (3)	0.0846 (7)
H3B	0.3126	−0.6835	0.1927	0.101*
C4	0.4162 (3)	−0.49125 (19)	0.1741 (2)	0.0672 (5)
H4A	0.4424	−0.5181	0.0980	0.081*
C5	0.4536 (2)	−0.35289 (16)	0.23193 (16)	0.0453 (4)
C6	0.5366 (2)	−0.24400 (16)	0.17207 (16)	0.0440 (4)
C7	0.5694 (2)	−0.09482 (16)	0.23362 (16)	0.0423 (4)
H7A	0.6284	−0.0657	0.3300	0.051*
H7B	0.4520	−0.0912	0.2152	0.051*
C8	0.6889 (2)	0.00837 (15)	0.18096 (15)	0.0383 (3)
H8A	0.6417	−0.0323	0.0831	0.046*
C9	0.8847 (2)	0.02844 (14)	0.23367 (14)	0.0360 (3)
C10	0.9652 (2)	−0.01886 (17)	0.15183 (16)	0.0453 (4)
H10A	0.8993	−0.0647	0.0605	0.054*
C11	1.1434 (2)	0.00199 (19)	0.20588 (19)	0.0542 (4)
H11A	1.2002	−0.0286	0.1516	0.065*
C12	1.2357 (2)	0.06803 (18)	0.34003 (19)	0.0555 (5)
H12A	1.3565	0.0834	0.3795	0.067*
C13	1.1469 (2)	0.11107 (19)	0.41502 (18)	0.0544 (4)
H13A	1.2104	0.1557	0.5068	0.065*
C14	0.6836 (2)	0.15105 (16)	0.21789 (16)	0.0433 (4)
H14A	0.5566	0.1351	0.1969	0.052*
H14B	0.7474	0.1990	0.3136	0.052*
C15	0.7697 (2)	0.24470 (16)	0.14624 (15)	0.0417 (4)
C16	0.8333 (2)	0.40088 (16)	0.20360 (15)	0.0416 (4)
C17	0.8833 (3)	0.48560 (18)	0.12857 (18)	0.0564 (5)
H17A	0.8748	0.4463	0.0418	0.068*
C18	0.9460 (3)	0.62891 (19)	0.1835 (2)	0.0691 (6)
H18A	0.9799	0.6887	0.1347	0.083*
C19	0.9575 (3)	0.68178 (19)	0.3110 (2)	0.0690 (6)
H19A	0.9998	0.7787	0.3511	0.083*
C20	0.9062 (3)	0.59109 (19)	0.3790 (2)	0.0690 (6)
H20A	0.9167	0.6290	0.4667	0.083*

supplementary materials

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0827 (9)	0.0397 (7)	0.0708 (9)	0.0201 (6)	0.0441 (7)	0.0133 (6)
O2	0.0957 (10)	0.0497 (7)	0.0500 (7)	0.0367 (7)	0.0365 (7)	0.0185 (6)
N1	0.0529 (9)	0.0523 (9)	0.0566 (9)	0.0150 (7)	0.0204 (7)	0.0205 (7)
N2	0.0775 (10)	0.0419 (8)	0.0516 (9)	0.0264 (7)	0.0299 (8)	0.0148 (7)
N3	0.0463 (8)	0.0487 (8)	0.0425 (8)	0.0206 (6)	0.0123 (6)	0.0094 (6)
C1	0.0720 (13)	0.0758 (15)	0.0686 (13)	0.0214 (11)	0.0322 (11)	0.0367 (11)
C2	0.0843 (15)	0.0638 (14)	0.0990 (18)	0.0202 (12)	0.0395 (14)	0.0501 (13)
C3	0.1021 (18)	0.0452 (11)	0.1096 (19)	0.0241 (12)	0.0468 (15)	0.0376 (12)
C4	0.0813 (14)	0.0426 (10)	0.0817 (14)	0.0249 (10)	0.0372 (11)	0.0238 (10)
C5	0.0383 (8)	0.0394 (9)	0.0525 (10)	0.0130 (7)	0.0130 (7)	0.0162 (7)
C6	0.0394 (8)	0.0369 (8)	0.0507 (10)	0.0137 (7)	0.0159 (7)	0.0116 (7)
C7	0.0408 (8)	0.0378 (8)	0.0492 (9)	0.0171 (7)	0.0183 (7)	0.0145 (7)
C8	0.0416 (8)	0.0340 (8)	0.0394 (8)	0.0174 (7)	0.0143 (7)	0.0113 (6)
C9	0.0417 (8)	0.0261 (7)	0.0396 (8)	0.0133 (6)	0.0156 (7)	0.0122 (6)
C10	0.0515 (9)	0.0423 (9)	0.0436 (9)	0.0210 (8)	0.0204 (7)	0.0133 (7)
C11	0.0514 (10)	0.0532 (10)	0.0703 (12)	0.0281 (9)	0.0328 (9)	0.0222 (9)
C12	0.0428 (9)	0.0536 (10)	0.0694 (12)	0.0219 (8)	0.0165 (9)	0.0226 (9)
C13	0.0492 (10)	0.0532 (10)	0.0482 (10)	0.0195 (8)	0.0070 (8)	0.0114 (8)
C14	0.0481 (9)	0.0390 (8)	0.0500 (9)	0.0231 (7)	0.0212 (7)	0.0175 (7)
C15	0.0473 (9)	0.0408 (9)	0.0398 (9)	0.0233 (7)	0.0138 (7)	0.0143 (7)
C16	0.0464 (9)	0.0387 (8)	0.0425 (9)	0.0211 (7)	0.0155 (7)	0.0153 (7)
C17	0.0724 (12)	0.0475 (10)	0.0527 (10)	0.0242 (9)	0.0280 (9)	0.0210 (8)
C18	0.0915 (15)	0.0420 (10)	0.0781 (14)	0.0245 (10)	0.0367 (12)	0.0298 (10)
C19	0.0864 (15)	0.0355 (10)	0.0810 (14)	0.0229 (10)	0.0340 (12)	0.0145 (10)
C20	0.1003 (16)	0.0443 (11)	0.0637 (12)	0.0304 (11)	0.0393 (11)	0.0116 (9)

Geometric parameters (Å, °)

O1—C6	1.2078 (19)	C8—C14	1.523 (2)
O2—C15	1.2066 (18)	C8—H8A	0.9800
N1—C5	1.329 (2)	C9—C10	1.374 (2)
N1—C1	1.336 (2)	C10—C11	1.372 (2)
N2—C16	1.325 (2)	C10—H10A	0.9300
N2—C20	1.334 (2)	C11—C12	1.360 (3)
N3—C9	1.332 (2)	C11—H11A	0.9300
N3—C13	1.334 (2)	C12—C13	1.360 (2)
C1—C2	1.362 (3)	C12—H12A	0.9300
C1—H1B	0.9300	C13—H13A	0.9300
C2—C3	1.351 (3)	C14—C15	1.494 (2)
C2—H2B	0.9300	C14—H14A	0.9700
C3—C4	1.364 (3)	C14—H14B	0.9700
C3—H3B	0.9300	C15—C16	1.495 (2)
C4—C5	1.371 (2)	C16—C17	1.373 (2)
C4—H4A	0.9300	C17—C18	1.369 (2)
C5—C6	1.495 (2)	C17—H17A	0.9300

C6—C7	1.496 (2)	C18—C19	1.358 (3)
C7—C8	1.520 (2)	C18—H18A	0.9300
C7—H7A	0.9700	C19—C20	1.358 (3)
C7—H7B	0.9700	C19—H19A	0.9300
C8—C9	1.510 (2)	C20—H20A	0.9300
C5—N1—C1	116.29 (17)	C11—C10—C9	119.52 (16)
C16—N2—C20	116.38 (15)	C11—C10—H10A	120.2
C9—N3—C13	117.34 (14)	C9—C10—H10A	120.2
N1—C1—C2	123.4 (2)	C12—C11—C10	118.97 (16)
N1—C1—H1B	118.3	C12—C11—H11A	120.5
C2—C1—H1B	118.3	C10—C11—H11A	120.5
C3—C2—C1	119.29 (19)	C13—C12—C11	118.30 (16)
C3—C2—H2B	120.4	C13—C12—H12A	120.9
C1—C2—H2B	120.4	C11—C12—H12A	120.9
C2—C3—C4	118.8 (2)	N3—C13—C12	124.06 (17)
C2—C3—H3B	120.6	N3—C13—H13A	118.0
C4—C3—H3B	120.6	C12—C13—H13A	118.0
C3—C4—C5	118.7 (2)	C15—C14—C8	112.94 (13)
C3—C4—H4A	120.6	C15—C14—H14A	109.0
C5—C4—H4A	120.6	C8—C14—H14A	109.0
N1—C5—C4	123.38 (16)	C15—C14—H14B	109.0
N1—C5—C6	116.83 (14)	C8—C14—H14B	109.0
C4—C5—C6	119.79 (16)	H14A—C14—H14B	107.8
O1—C6—C5	119.43 (14)	O2—C15—C14	121.65 (14)
O1—C6—C7	121.65 (14)	O2—C15—C16	119.61 (14)
C5—C6—C7	118.90 (14)	C14—C15—C16	118.74 (13)
C6—C7—C8	112.50 (13)	N2—C16—C17	123.20 (15)
C6—C7—H7A	109.1	N2—C16—C15	117.27 (14)
C8—C7—H7A	109.1	C17—C16—C15	119.52 (14)
C6—C7—H7B	109.1	C18—C17—C16	118.98 (17)
C8—C7—H7B	109.1	C18—C17—H17A	120.5
H7A—C7—H7B	107.8	C16—C17—H17A	120.5
C9—C8—C7	110.17 (12)	C19—C18—C17	118.47 (18)
C9—C8—C14	110.11 (13)	C19—C18—H18A	120.8
C7—C8—C14	111.77 (12)	C17—C18—H18A	120.8
C9—C8—H8A	108.2	C20—C19—C18	119.05 (17)
C7—C8—H8A	108.2	C20—C19—H19A	120.5
C14—C8—H8A	108.2	C18—C19—H19A	120.5
N3—C9—C10	121.81 (14)	N2—C20—C19	123.91 (18)
N3—C9—C8	115.90 (13)	N2—C20—H20A	118.0
C10—C9—C8	122.28 (14)	C19—C20—H20A	118.0

Hydrogen-bond geometry (Å, °)

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C4—H4A$\cdots$O1[i]	0.93	2.51	3.322 (3)	146
C11—H11A$\cdots$O2[ii]	0.93	2.55	3.415 (2)	155
C14—H14B$\cdots$N2	0.97	2.48	2.836 (2)	101

C14—H14B···N3	0.97	2.56	2.908 (2)	101

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

Fig. 1