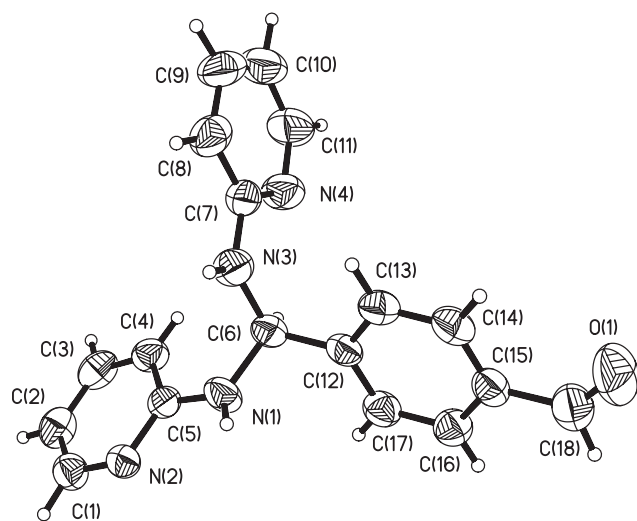


Crystal structure of 4-di(2-pyridinoamino)methylbenzaldehyde, $C_{18}H_{16}N_4O$

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Abstract

$C_{18}H_{16}N_4O$, monoclinic, $P12_1/c1$ (No. 14), $a = 10.7420(6)$ Å, $b = 12.7228(7)$ Å, $c = 11.8769(7)$ Å, $\beta = 105.495(1)^\circ$, $V = 1564.2$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.045$, $wR_{\text{ref}}(F^2) = 0.125$, $T = 298$ K.

Source of material

Reagents and solvents used were of commercially available quality. The title compound was prepared by dissolving terephthalaldehyde (1 mmol, 134 mg) and 2-aminopyridine (2 mmol, 94 mg) in methanol, and then removing about two thirds of the solvents. The yellow rhombus crystals precipitated were filtered and washed with methanol for three times and dried in a vacuum under CaCl_2 (yield 63%). Elemental analysis found: C, 70.69%; H, 5.36%; N, 18.26%; calc. for $C_{18}H_{16}N_4O$: C, 71.04%; H, 5.30%; N, 18.41%.

Discussion

The design and preparation of intermediates for organic synthesis are very important in order to get various new complexes. After having tried many methods, we succeeded in isolating a new unsymmetry organic intermediate, 4-di(2-pyridylamino)methylbenzaldehyde, in high yield.

The C(1) atom in the compound is in a distorted tetrahedral geometry environment, connected to C(2) (from the phenyl ring), N(2) and N(4) atoms (from the two 2-aminopyridine groups), and one hydrogen atom. The angles around C(1) are from $108.6(1)^\circ$ to

$114.4(1)^\circ$, indicating a slightly distorted tetrahedron of C(1) atom. All the three aromatic rings in the compound are well-defined planes, which are located at the angles of 70.9° , 85.0° , and 88.3° , respectively. The intermolecular hydrogen bonds between N(2) and N(1) atoms ($3.064(3)$ Å, $176(2)^\circ$), and between N(4) and O(1) atoms ($2.952(3)$ Å, $174(2)^\circ$) join the molecules to form a zig-zag chain.

Table 1. Data collection and handling.

Crystal:	yellow block, size $0.80 \times 0.50 \times 0.32$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.84 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, φ/ω
$2\theta_{\text{max}}$:	56.60°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	9667, 3836
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2754
$N(\text{param})_{\text{refined}}$:	272
Programs:	SHELXTL [1], SHELXTL-plus [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	4e	0.873(2)	0.187(2)	0.644(2)	0.064(5)
H(2)	4e	0.676(2)	0.167(2)	0.689(2)	0.073(6)
H(3)	4e	0.560(2)	0.008(2)	0.636(2)	0.066(5)
H(4)	4e	0.650(2)	−0.116(1)	0.540(2)	0.058(5)
H(6)	4e	0.767(1)	−0.240(1)	0.499(1)	0.042(4)
H(8)	4e	0.546(2)	−0.177(2)	0.133(2)	0.067(5)
H(9)	4e	0.357(2)	−0.279(2)	0.074(2)	0.094(7)
H(10)	4e	0.306(2)	−0.416(2)	0.201(2)	0.094(7)
H(11)	4e	0.455(3)	−0.440(2)	0.394(3)	0.13(1)
H(13)	4e	0.853(2)	−0.322(1)	0.264(2)	0.060(5)
H(14)	4e	1.016(2)	−0.442(1)	0.261(2)	0.066(5)
H(16)	4e	1.177(2)	−0.397(1)	0.600(2)	0.055(5)
H(17)	4e	1.016(2)	−0.274(1)	0.607(2)	0.062(5)
H(18)	4e	1.282(2)	−0.521(2)	0.510(2)	0.067(5)
H(1A)	4e	0.940(2)	−0.093(1)	0.472(1)	0.051(5)
H(3A)	4e	0.734(2)	−0.151(1)	0.285(2)	0.058(5)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1)	4e	1.2210(2)	−0.5587(1)	0.3466(2)	0.104(1)	0.083(1)	0.104(1)	0.0174(9)	0.049(1)	−0.0206(9)
N(1)	4e	0.8727(1)	−0.1116(1)	0.4911(1)	0.0417(7)	0.0416(7)	0.0764(9)	−0.0109(6)	0.0246(7)	−0.0124(6)
N(2)	4e	0.8761(1)	0.05308(9)	0.5690(1)	0.0405(6)	0.0403(6)	0.0496(7)	−0.0024(5)	0.0118(5)	−0.0016(5)
N(3)	4e	0.7160(1)	−0.1924(1)	0.3344(1)	0.0507(8)	0.0530(8)	0.0583(8)	−0.0078(6)	0.0069(6)	0.0113(7)
N(4)	4e	0.5825(1)	−0.3238(1)	0.3691(1)	0.0527(8)	0.0625(9)	0.0716(9)	−0.0140(7)	0.0070(7)	0.0039(7)
C(1)	4e	0.8235(2)	0.1260(1)	0.6232(1)	0.0543(9)	0.0458(8)	0.0511(9)	0.0009(7)	0.0135(7)	−0.0027(7)
C(2)	4e	0.7084(2)	0.1141(2)	0.6510(2)	0.058(1)	0.065(1)	0.0500(9)	0.0128(8)	0.0198(8)	−0.0013(8)
C(3)	4e	0.6414(2)	0.0213(2)	0.6189(2)	0.0423(8)	0.077(1)	0.0555(9)	0.0066(8)	0.0203(7)	0.0086(8)
C(4)	4e	0.6910(2)	−0.0547(1)	0.5620(1)	0.0395(8)	0.0535(9)	0.0558(9)	−0.0060(7)	0.0121(7)	0.0044(7)
C(5)	4e	0.8112(1)	−0.0374(1)	0.5397(1)	0.0360(7)	0.0422(7)	0.0412(7)	−0.0001(6)	0.0076(5)	0.0041(6)
C(6)	4e	0.8132(1)	−0.2096(1)	0.4441(1)	0.0421(7)	0.0408(8)	0.0525(8)	−0.0095(6)	0.0101(6)	−0.0013(6)
C(7)	4e	0.6065(1)	−0.2516(1)	0.2966(1)	0.0425(8)	0.0497(8)	0.0540(9)	0.0044(7)	0.0099(7)	−0.0074(7)
C(8)	4e	0.5233(2)	−0.2350(2)	0.1844(2)	0.058(1)	0.077(1)	0.055(1)	0.0162(9)	0.0091(8)	−0.0098(9)
C(9)	4e	0.4136(2)	−0.2951(2)	0.1511(2)	0.055(1)	0.107(2)	0.066(1)	0.018(1)	−0.006(1)	−0.037(1)
C(10)	4e	0.3863(2)	−0.3694(2)	0.2263(2)	0.053(1)	0.086(2)	0.101(2)	−0.012(1)	0.010(1)	−0.035(1)
C(11)	4e	0.4729(2)	−0.3805(2)	0.3321(2)	0.057(1)	0.075(1)	0.100(2)	−0.020(1)	0.014(1)	−0.011(1)
C(12)	4e	0.9190(1)	−0.2868(1)	0.4364(1)	0.0454(8)	0.0356(7)	0.0474(8)	−0.0107(6)	0.0101(6)	−0.0032(6)
C(13)	4e	0.9194(2)	−0.3376(1)	0.3330(1)	0.0537(9)	0.0531(9)	0.0457(8)	−0.0053(7)	0.0017(7)	−0.0085(7)
C(14)	4e	1.0154(2)	−0.4087(1)	0.3298(2)	0.064(1)	0.0530(9)	0.0489(9)	−0.0067(8)	0.0133(8)	−0.0157(8)
C(15)	4e	1.1121(2)	−0.4313(1)	0.4301(1)	0.0519(9)	0.0395(7)	0.0552(9)	−0.0055(6)	0.0173(7)	−0.0038(6)
C(16)	4e	1.1120(2)	−0.3809(1)	0.5335(2)	0.0529(9)	0.0515(9)	0.0469(8)	−0.0005(7)	0.0065(7)	0.0005(7)
C(17)	4e	1.0168(2)	−0.3091(1)	0.5365(1)	0.0558(9)	0.0484(8)	0.0421(8)	−0.0032(7)	0.0097(7)	−0.0075(7)
C(18)	4e	1.2122(2)	−0.5106(2)	0.4307(2)	0.064(1)	0.055(1)	0.078(1)	−0.0002(8)	0.028(1)	−0.0023(9)

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