Crystal structure of 4-benzoylpyridinium-1-squarate, $C_{16}H_9NO_4$

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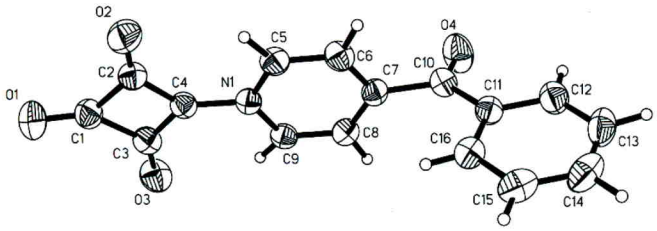

Abstract

$C_{16}H_9NO_4$, orthorhombic, $Pna2_1$ (No. 33), a = 14.671(1) Å, b = 8.1155(9) Å, c = 10.494(1) Å, V = 1249.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.095, T = 291 K.

Source of material

Squaric acid reacted with equimolecular amounts 4-benzoylpyridine in acetic anhydride to give the monocondensation product (yield: 91%, mp 569 K – 571 K with decomposition) [1]. The purity of the compound was confirmed by elemental analysis, IR, UV-vis and mass spectrometry. Orange-red transparent crystals, suitable for X-ray diffraction were grown from ethanol by slow evaporation.

Discussion

Organic nonlinear optical (NLO) materials have attracted much attention in the past years due to potential applications in various fields such as telecommunication, optical data storage and optical information processing. Because of their notable chemical flexibility that allows for molecular engineering of the nonlinear optical responses and their fast electronic responses, organic materials are particularly interesting candidates for the elaboration of optimized NLO materials [2, 3, 4].

The conversion of N atom of 4-benzoylpyridin into the corresponding pyridinium betaine is a way to enhance the charge transfer transition on the molecular level - a requisite for a design of efficient second- and third-order nonlinear optical materials.

Table 1. Data collection and handling.

Crystal:	orange needle, size 0.13 × 0.20 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.09 cm^{-1}
Diffractometer, scan mode:	Nonius MACH3, 2θ/ω
$2\theta_{max}$:	54.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2962, 1513
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1143
$N(param)_{refined}$:	227
Programs:	SHELXS-97 [6], SHELXTL-Plus [7], SHELXL-97 [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(5)	4*a*	0.286(2)	0.162(3)	0.108(3)	0.037(7)
H(6)	4*a*	0.446(2)	0.165(4)	0.113(4)	0.07(1)
H(8)	4*a*	0.446(2)	–0.185(3)	–0.170(3)	0.040(8)
H(9)	4*a*	0.282(2)	–0.153(4)	–0.170(3)	0.045(9)
H(12)	4*a*	0.741(2)	0.006(5)	–0.009(4)	0.07(1)
H(13)	4*a*	0.836(2)	–0.128(4)	0.131(4)	0.061(9)
H(14)	4*a*	0.770(2)	–0.313(4)	0.282(4)	0.056(9)
H(15)	4*a*	0.611(3)	–0.341(6)	0.291(5)	0.08(1)
H(16)	4*a*	0.521(2)	–0.200(3)	0.153(3)	0.040(7)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*a*	–0.0391(1)	0.0061(3)	–0.0293(3)	0.0355(8)	0.073(1)	0.082(2)	0.002(1)	–0.002(1)	–0.001(1)
O(2)	4*a*	0.1219(1)	0.1906(3)	0.1309(3)	0.054(1)	0.052(1)	0.057(2)	0.0037(8)	0.005(1)	–0.015(1)
O(3)	4*a*	0.1177(1)	–0.1762(3)	–0.1998(3)	0.053(1)	0.061(1)	0.046(1)	–0.0081(9)	–0.007(1)	–0.012(1)
N(1)	4*a*	0.2781(1)	0.0024(3)	–0.0333(3)	0.0331(8)	0.0330(8)	0.0285(8)	–0.0003(9)	0.001(1)	0.0021(7)
C(1)	4*a*	0.0428(2)	0.0093(4)	–0.0331(4)	0.038(1)	0.043(1)	0.046(1)	0.001(1)	–0.003(2)	0.004(1)
C(2)	4*a*	0.1190(2)	0.0942(3)	0.0421(3)	0.039(1)	0.036(1)	0.043(2)	0.0010(9)	0.002(1)	0.003(1)
C(4)	4*a*	0.1824(1)	0.0086(3)	–0.0360(3)	0.035(1)	0.034(1)	0.034(1)	–0.001(1)	–0.001(2)	0.002(1)
C(3)	4*a*	0.1172(2)	–0.0763(3)	–0.1126(3)	0.041(1)	0.040(1)	0.036(2)	–0.003(1)	–0.004(1)	0.005(1)
O(4)	4*a*	0.5982(2)	0.0819(3)	–0.1282(3)	0.046(1)	0.081(2)	0.058(1)	–0.008(1)	0.005(1)	0.026(1)
C(5)	4*a*	0.3239(2)	0.0974(3)	0.0513(3)	0.041(1)	0.038(1)	0.032(1)	–0.001(1)	0.003(1)	–0.008(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	4*a*	0.4169(2)	0.0950(3)	0.0544(3)	0.041(1)	0.043(1)	0.034(1)	–0.004(1)	–0.005(1)	–0.006(1)
C(7)	4*a*	0.4643(1)	–0.0050(3)	–0.0312(3)	0.0343(9)	0.036(1)	0.033(1)	0.002(1)	–0.003(1)	0.0057(9)
C(8)	4*a*	0.4167(2)	–0.1039(3)	–0.1153(3)	0.038(1)	0.037(1)	0.033(1)	0.002(1)	–0.001(1)	0.001(1)
C(9)	4*a*	0.3231(2)	–0.0993(3)	–0.1155(3)	0.038(1)	0.033(1)	0.033(1)	–0.003(1)	0.000(1)	–0.002(1)
C(10)	4*a*	0.5671(1)	0.0043(4)	–0.0393(4)	0.036(1)	0.041(1)	0.037(1)	–0.003(1)	–0.001(1)	–0.000(1)
C(11)	4*a*	0.6230(2)	–0.0807(3)	0.0561(3)	0.037(1)	0.038(1)	0.034(1)	0.0004(9)	–0.003(1)	–0.006(1)
C(12)	4*a*	0.7179(2)	–0.0643(4)	0.0519(3)	0.040(1)	0.050(2)	0.049(2)	–0.001(1)	–0.001(1)	–0.009(2)
C(13)	4*a*	0.7715(2)	–0.1492(4)	0.1368(4)	0.040(1)	0.064(2)	0.059(2)	0.011(1)	–0.013(2)	–0.020(2)
C(14)	4*a*	0.7322(2)	–0.2527(5)	0.2244(4)	0.067(2)	0.057(2)	0.044(2)	0.025(2)	–0.018(2)	–0.015(2)
C(15)	4*a*	0.6388(2)	–0.2692(4)	0.2309(3)	0.071(2)	0.045(2)	0.037(1)	0.010(1)	–0.006(2)	–0.002(1)
C(16)	4*a*	0.5837(2)	–0.1817(3)	0.1473(3)	0.042(1)	0.043(1)	0.036(1)	0.003(1)	–0.001(1)	–0.002(1)

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