Crystal structure of (6-methyl-2-pyridinyl)acetoacetamide, $C_{10}H_{12}N_2O_2$

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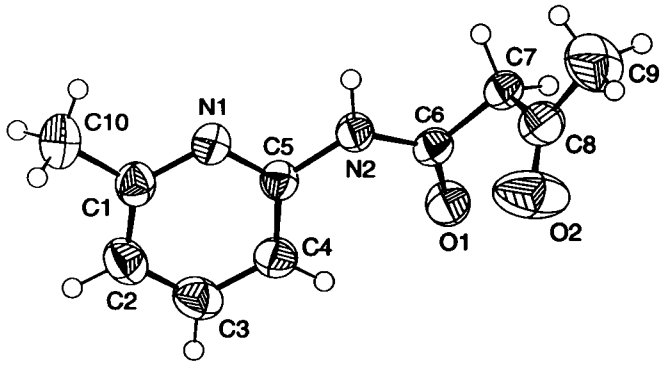
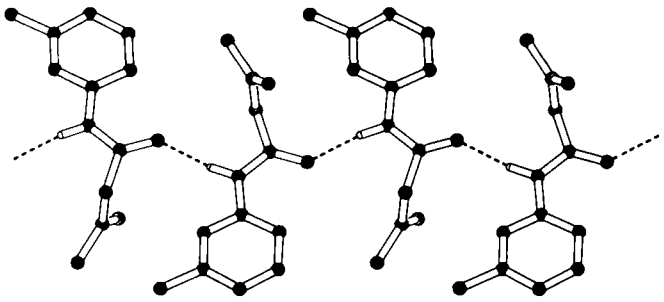
Received April 8, 2005, accepted and available on-line April 27, 2005; CCDC no. 1267/1525





Abstract

$C_{10}H_{12}N_2O_2$, orthorhombic, *Pbca* (no. 61), $a = 9.474(2)$ Å, $b = 13.303(3)$ Å, $c = 16.365(3)$ Å, $V = 2062.5$ Å^3, $Z = 8$, $R_{gt}(F) = 0.079$, $wR_{ref}(F^2) = 0.199$, $T = 291$ K.

Source of material

The title compound was prepared by a method similar to that of [1]: a solution of 2-amino-6-methylpyridine (10 mmol) in benzene (30 ml) was added to a solution of ethyl acetoacetate (10 mmol). The reaction mixture was refluxed for 2 h with stirring, then the resulting pale precipitate was collected by filtration, washed several times with benzene and dried in vacuo (yield 78 %). An ethanol solution of product was slowly evaporated and pale crystals were obtained after a week. All commercially available reagents were used as supplied.

Elemental analysis: found – C, 62.40 %; H, 6.40 %; N, 14.48 %, calc. for $C_{10}H_{12}N_2O_2$ – C, 62.49 %; H, 6.25 %; N, 14.57 %. MS, IR and ^{1}H NMR data are available in the CIF file.

Experimental details

The hydrogen atom bound to the nitrogen atom was freely refined in order to show its significance.

Discussion

The europium(III) and terbium(III) complexes with conjugated ligands such as β-diketonato have been studied as emitting materials for organic electroluminescent diodes (OLEDs) [2,3] due to the initial report [4]. However, the quantum efficiency of most these complexes is unfortunately still low. This may be mostly caused by the inefficiency of the energy transfer, in particular triplet-triplet transfer. It is essential to design ligands, which have better energy-transfer properties to the lanthanide metal ion.

As part of our studies on the synthesis and characterization of β-diketonato and its complexes, we report the crystal structure of the new β-diketonate-type ligand, *N*-(2-amino-6-methyl-pyridinyl)ketoacetamide. In the structure, the two C=O bonds lengths are 1.231(3) Å and 1.188(5) Å, which suggests that the title compound is in the *keto* form (figure, top). Other bond distances and angles are in normal ranges. The molecules are linked to each other by an N–H···O intermolecular hydrogen bond to form ribbons running along *a* axis (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.17 × 0.18 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS IP, ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4805, 1668
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1219
$N(param)_{refined}$:	132
Program:	SHELXTL [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	8*c*	0.2548	1.1504	0.3222	0.066
H(3A)	8*c*	0.0397	1.0863	0.3665	0.067
H(4A)	8*c*	0.0325	0.9347	0.4356	0.058
H(7A)	8*c*	0.1307	0.6203	0.5530	0.050
H(7B)	8*c*	0.2888	0.6508	0.5389	0.050
H(9A)	8*c*	0.2926	0.6672	0.7549	0.128
H(9B)	8*c*	0.3864	0.6336	0.6808	0.128
H(9C)	8*c*	0.2481	0.5743	0.7019	0.128
H(10A)	8*c*	0.5871	1.0294	0.3628	0.109
H(10B)	8*c*	0.5315	1.1401	0.3697	0.109
H(10C)	8*c*	0.5196	1.0821	0.2866	0.109
H(2E)	8*c*	0.348(4)	0.793(3)	0.484(2)	0.07(1)

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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)	8c	0.0255(2)	0.7747(2)	0.4874(2)	0.030(1)	0.059(2)	0.077(2)	−0.002(1)	−0.004(1)	0.016(1)
O(2)	8c	0.1557(4)	0.7820(3)	0.6701(2)	0.137(3)	0.109(3)	0.061(2)	0.061(2)	−0.010(2)	−0.021(2)
N(1)	8c	0.3697(2)	0.9493(2)	0.4107(2)	0.040(1)	0.044(2)	0.041(2)	−0.003(1)	0.001(1)	0.002(1)
N(2)	8c	0.2559(3)	0.8211(2)	0.4767(2)	0.031(1)	0.044(2)	0.049(2)	0.000(1)	0.000(1)	0.010(1)
C(1)	8c	0.3721(3)	1.0367(3)	0.3698(2)	0.056(2)	0.044(2)	0.037(2)	−0.005(2)	0.004(1)	−0.001(2)
C(2)	8c	0.2505(4)	1.0903(3)	0.3511(2)	0.071(2)	0.046(2)	0.046(2)	0.003(2)	−0.002(2)	0.006(2)
C(3)	8c	0.1228(4)	1.0515(3)	0.3771(2)	0.057(2)	0.055(3)	0.055(2)	0.012(2)	−0.008(2)	0.004(2)
C(4)	8c	0.1179(3)	0.9618(3)	0.4184(2)	0.042(2)	0.053(2)	0.051(2)	0.007(2)	0.000(1)	0.007(2)
C(5)	8c	0.2444(3)	0.9130(3)	0.4337(2)	0.038(2)	0.041(2)	0.036(2)	−0.001(1)	−0.002(1)	0.002(2)
C(6)	8c	0.1509(3)	0.7608(3)	0.5036(2)	0.033(2)	0.043(2)	0.043(2)	0.000(1)	0.002(1)	−0.001(2)
C(7)	8c	0.1978(3)	0.6751(3)	0.5575(2)	0.032(2)	0.046(2)	0.048(2)	0.000(1)	0.003(1)	0.005(2)
C(8)	8c	0.2093(4)	0.7071(3)	0.6456(2)	0.053(2)	0.062(3)	0.054(3)	0.007(2)	0.005(2)	0.006(2)
C(9)	8c	0.2914(5)	0.6395(4)	0.7007(3)	0.105(3)	0.093(4)	0.058(3)	0.020(3)	−0.014(2)	0.017(3)
C(10)	8c	0.5156(4)	1.0756(3)	0.3450(3)	0.071(3)	0.065(3)	0.082(3)	−0.012(2)	0.017(2)	0.016(2)

Acknowledgments. The authors would like to thank the National Natural Science Foundation of China (no. 20441002), Ningxia Natural Gas Transferring Key Laboratory (no. 2004007) and Weifang University for research grants.

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