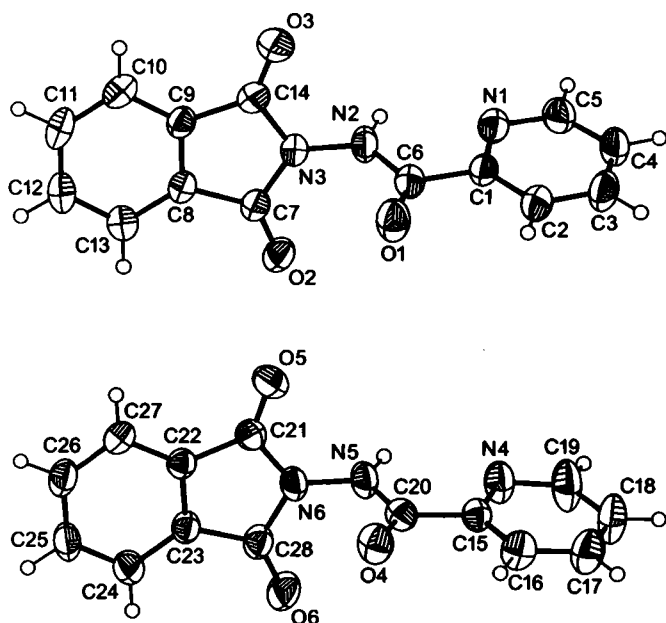


Crystal structure of *N,N*-phthaloylpicoloylhydrazide, $C_{14}H_9N_3O_3$

L.-X. Shi, W.-S. Wu*, J.-C. Dai and J.-M. Lin

Huaqiao University, College of Materials, Quanzhou, Fujian, 362021 P. R. China

Received December 17, 2004, accepted and available on-line January 13, 2005; CCDC no. 1267/1447



Abstract

$C_{14}H_9N_3O_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 8.8812(5)$ Å, $b = 20.295(1)$ Å, $c = 14.3945(8)$ Å, $\beta = 107.882(1)^\circ$, $V = 2469.2$ Å³, $Z = 8$, $R_{gt}(F) = 0.042$, $wR_{ref}(F^2) = 0.123$, $T = 293$ K.

Source of material

The title compound was synthesized by refluxing an equimolar mixture of picoloylhydrazine and phthalic anhydride in ethanol for 1 h. Single crystals suitable for the X-ray structure analysis were obtained from ethanol.

Discussion

Hydrazides form an important class of organic compounds with many applications in organic synthesis and in industry. They are useful starting materials for the synthesis of biologically active heterocycles [1]. Compared to simple hydrazide, acyl, aroyl and heteroaryl hydrazide have an additional O-donor atom from the carbonyl group, whose presence introduces a wider range of properties. Therefore, these compounds may function as polydentate ligands and some reports of their complexes have appeared [2,3].

The X-ray analysis of title compound indicated that both crystallographically independent molecules are non-planar and twisted in the form of planes of picolonybonyl group (r.m.s. devi-

ations 0.008 Å) and phthaloyl group (r.m.s. deviations 0.023 Å), the dihedral angles between them are $74.95(3)^\circ$ and $80.25(5)^\circ$, respectively. Due to their structural equivalence the discussion focuses only on the first molecule. The C6—N2 bond length of 1.351(2) Å is essentially the same as C7—N3 of 1.392(2) Å and C14—N3 of 1.405(2) Å, indicating that C6—N2 is single bond. The N2—N3 bond length of 1.375(2) Å is in good agreement with the N—N distances in the compounds of *N*-phenylpicoloylhydrazide [3] and propionylpicoloylhydrazide [4]. To form the one-dimensional structure along *a*-axis, the molecules are connected to each other via intermolecular hydrogen bonds with $d(N2\cdots O4^i) = 2.865$ Å and $\angle N2-HO2A\cdots O4^i = 132.4^\circ$, $d(N5\cdots O2^{ii}) = 2.875$ Å and $\angle N5-HO5A\cdots O2^{ii} = 130.4^\circ$ (symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$).

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.35 \times 0.48 \times 0.63$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.05 cm^{-1}
Diffractionmeter, scan mode:	Rigaku R-Axis RAPID, ω
$2\theta_{\text{max}}$:	54.98°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	9336, 5140
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3633
$N(\text{param})_{\text{refined}}$:	362
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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(2A)	4e	0.2132	0.4704	−0.0346	0.074
H(3A)	4e	0.0644	0.4312	−0.1870	0.088
H(4A)	4e	−0.1316	0.3542	−0.1958	0.083
H(5A)	4e	−0.1778	0.3203	−0.0560	0.075
H(10A)	4e	0.1670	0.5099	0.5567	0.068
H(11A)	4e	0.3791	0.4899	0.6973	0.073
H(12A)	4e	0.5911	0.4280	0.6899	0.071
H(13A)	4e	0.6020	0.3845	0.5430	0.063
H(16A)	4e	−0.1797	0.7122	0.5528	0.079
H(17A)	4e	−0.2785	0.7218	0.3840	0.095
H(18A)	4e	−0.1138	0.6981	0.2917	0.103
H(19A)	4e	0.1372	0.6624	0.3681	0.110
H(24A)	4e	0.5104	0.7761	1.0267	0.072
H(25A)	4e	0.5903	0.7238	1.1783	0.077
H(26A)	4e	0.5701	0.6117	1.1884	0.077
H(27A)	4e	0.4585	0.5480	1.0504	0.067
H(02A)	4e	0.0645	0.3863	0.1971	0.070
H(05A)	4e	0.3001	0.6393	0.6567	0.068

* Correspondence author (e-mail: wuwenshi@sohu.com)

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	4e	0.1009(2)	0.41590(9)	0.0413(1)	0.051(1)	0.055(1)	0.0379(9)	0.0051(8)	0.0133(7)	0.0015(7)
C(2)	4e	0.1335(2)	0.4395(1)	−0.0403(1)	0.066(1)	0.075(1)	0.046(1)	−0.002(1)	0.0196(9)	0.0046(9)
C(3)	4e	0.0452(3)	0.4163(1)	−0.1305(2)	0.088(2)	0.094(2)	0.039(1)	0.013(1)	0.020(1)	0.008(1)
C(4)	4e	−0.0712(3)	0.3710(1)	−0.1358(2)	0.071(1)	0.089(2)	0.042(1)	0.010(1)	0.008(1)	−0.013(1)
C(5)	4e	−0.0975(2)	0.3507(1)	−0.0514(2)	0.059(1)	0.068(1)	0.055(1)	−0.0002(9)	0.0109(9)	−0.013(1)
C(6)	4e	0.1937(2)	0.43972(9)	0.1410(1)	0.054(1)	0.058(1)	0.042(1)	−0.0007(8)	0.0130(8)	0.0008(8)
C(7)	4e	0.3632(2)	0.39881(9)	0.3617(1)	0.0510(9)	0.0478(9)	0.0400(9)	−0.0030(7)	0.0160(8)	−0.0010(7)
C(8)	4e	0.3910(2)	0.42138(8)	0.4638(1)	0.0523(9)	0.0431(9)	0.0381(9)	−0.0052(7)	0.0166(7)	−0.0024(7)
C(9)	4e	0.2623(2)	0.45879(9)	0.4677(1)	0.052(1)	0.051(1)	0.0389(9)	−0.0030(7)	0.0179(7)	0.0017(7)
C(10)	4e	0.2537(2)	0.48505(9)	0.5541(1)	0.069(1)	0.056(1)	0.055(1)	0.0032(9)	0.032(1)	−0.0021(9)
C(11)	4e	0.3806(3)	0.4728(1)	0.6376(1)	0.093(2)	0.058(1)	0.039(1)	−0.010(1)	0.028(1)	−0.0064(8)
C(12)	4e	0.5082(3)	0.4356(1)	0.6331(1)	0.074(1)	0.059(1)	0.038(1)	−0.007(1)	0.0069(9)	0.0015(8)
C(13)	4e	0.5156(2)	0.40940(9)	0.5459(1)	0.058(1)	0.051(1)	0.047(1)	−0.0016(8)	0.0119(8)	0.0005(8)
C(14)	4e	0.1501(2)	0.4639(1)	0.3676(1)	0.051(1)	0.068(1)	0.050(1)	0.0036(9)	0.0204(8)	0.0033(9)
C(15)	4e	0.0345(2)	0.67963(9)	0.5544(1)	0.052(1)	0.053(1)	0.041(1)	−0.0056(8)	0.0133(8)	0.0010(7)
C(16)	4e	−0.1170(2)	0.7017(1)	0.5137(2)	0.055(1)	0.081(1)	0.062(1)	0.003(1)	0.020(1)	0.005(1)
C(17)	4e	−0.1751(3)	0.7079(1)	0.4138(2)	0.058(1)	0.107(2)	0.063(2)	0.009(1)	0.006(1)	0.009(1)
C(18)	4e	−0.0784(3)	0.6935(1)	0.3593(2)	0.071(2)	0.133(2)	0.044(1)	0.008(1)	0.001(1)	0.009(1)
C(19)	4e	0.0722(3)	0.6722(2)	0.4060(2)	0.071(1)	0.163(3)	0.040(1)	0.021(2)	0.015(1)	−0.002(1)
C(20)	4e	0.1003(2)	0.67375(9)	0.6632(1)	0.057(1)	0.050(1)	0.046(1)	−0.0057(8)	0.0220(8)	0.0000(8)
C(21)	4e	0.3461(2)	0.59843(9)	0.8574(1)	0.053(1)	0.051(1)	0.044(1)	0.0022(8)	0.0183(8)	−0.0015(8)
C(22)	4e	0.4225(2)	0.62504(8)	0.9564(1)	0.0455(9)	0.0496(9)	0.0411(9)	0.0028(7)	0.0137(7)	0.0019(7)
C(23)	4e	0.4389(2)	0.69249(8)	0.9499(1)	0.052(1)	0.050(1)	0.0391(9)	−0.0034(7)	0.0103(7)	0.0040(7)
C(24)	4e	0.5008(2)	0.7307(1)	1.0315(1)	0.069(1)	0.052(1)	0.050(1)	−0.0092(9)	0.0066(9)	−0.0026(8)
C(25)	4e	0.5487(3)	0.6992(1)	1.1216(1)	0.072(1)	0.073(1)	0.038(1)	−0.006(1)	0.0025(9)	−0.0036(9)
C(26)	4e	0.5348(2)	0.6319(1)	1.1275(1)	0.072(1)	0.075(1)	0.040(1)	0.006(1)	0.0092(9)	0.0127(9)
C(27)	4e	0.4698(2)	0.59333(9)	1.0455(1)	0.065(1)	0.051(1)	0.049(1)	0.0056(9)	0.0130(9)	0.0106(8)
C(28)	4e	0.3775(2)	0.71259(9)	0.8463(1)	0.060(1)	0.055(1)	0.043(1)	−0.0043(8)	0.0114(8)	0.0065(8)
N(1)	4e	−0.0142(2)	0.37209(8)	0.0373(1)	0.0572(9)	0.0601(9)	0.0447(9)	−0.0027(7)	0.0144(7)	−0.0045(7)
N(2)	4e	0.1441(2)	0.41268(9)	0.2124(1)	0.0572(9)	0.082(1)	0.0329(8)	−0.0164(8)	0.0086(7)	−0.0028(7)
N(3)	4e	0.2191(2)	0.42675(8)	0.3090(1)	0.0498(8)	0.069(1)	0.0360(8)	0.0006(7)	0.0108(6)	−0.0036(7)
N(4)	4e	0.1315(2)	0.6645(1)	0.5027(1)	0.060(1)	0.104(1)	0.0416(9)	0.0112(9)	0.0133(8)	−0.0013(9)
N(5)	4e	0.2517(2)	0.65293(8)	0.6965(1)	0.0593(9)	0.077(1)	0.0327(8)	0.0052(8)	0.0125(7)	−0.0027(7)
N(6)	4e	0.3270(2)	0.65392(8)	0.7956(1)	0.0652(9)	0.0592(9)	0.0325(8)	−0.0022(7)	0.0129(7)	−0.0004(6)
O(1)	4e	0.3019(2)	0.47885(8)	0.1558(1)	0.081(1)	0.090(1)	0.0548(9)	−0.0322(9)	0.0176(7)	−0.0024(8)
O(2)	4e	0.4410(2)	0.36316(7)	0.32771(9)	0.0669(8)	0.0662(8)	0.0493(8)	0.0089(7)	0.0222(6)	−0.0072(6)
O(3)	4e	0.0258(2)	0.49246(9)	0.3385(1)	0.0621(9)	0.119(1)	0.066(1)	0.0304(9)	0.0180(7)	0.0105(9)
O(4)	4e	0.0222(2)	0.68684(7)	0.7172(1)	0.0700(9)	0.087(1)	0.0560(9)	0.0026(7)	0.0335(7)	0.0040(7)
O(5)	4e	0.3061(2)	0.54323(7)	0.8309(1)	0.087(1)	0.0547(8)	0.0619(9)	−0.0048(7)	0.0215(8)	−0.0113(7)
O(6)	4e	0.3700(2)	0.76615(7)	0.8090(1)	0.100(1)	0.0620(9)	0.0549(9)	−0.0110(8)	0.0119(8)	0.0175(7)

Acknowledgments. We are grateful for financial support from the National Science Foundation of Fujian Province, China (grant no. E0210026) and Research Funding of Fujian Province, China (grant no.2003F006).

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