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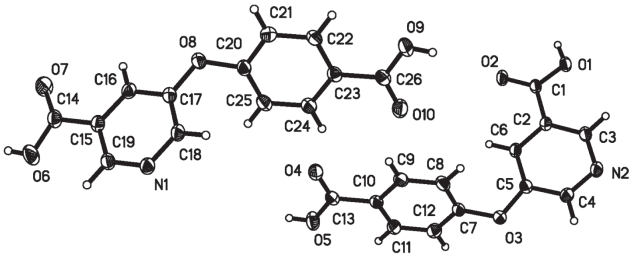
# Crystal structure of 5-(4-carboxyphenoxy)-nicotinic acid, $C_{13}H_9NO_5$

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## Abstract

$C_{13}H_9NO_5$, triclinic, $P\bar{1}$, $a = 7.6228(12)$ Å, $b = 10.5401(16)$ Å, $c = 14.669(2)$ Å, $\alpha = 94.801(2)°$, $\beta = 91.188(2)°$, $\gamma = 105.664(2)°$, $Z = 4$, $Dc = 1.524$ g/cm$^3$, Formula weight = 259.21, $F(000) = 536$, $V = 1129.7(3)$ Å$^3$, $R_{gt}(F) = 0.038$, $wR_{ref}(F^2) = 0.107$, $T = 296$ K.

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## Source of material

A mixture of the 5-(4-carboxyphenoxy)nicotinic acid (0.026 g, 0.1 mmol) and 10 ml distilled water in a 25 ml Teflon-lined autoclave was kept under autogenous pressure at 413 K for 4 days. After cooling to room temperature at a rate of 5 K h$^{-1}$, yellow block crystals suitable for X-ray diffraction were collected by filtration and washed with distilled water in 40% yield. Elemental analysis calculated for $C_{13}H_9NO_5$: C 60.18, H 3.47, N 5.40%. Found C 60.21, H 3.52, N 5.38%.

## Experimental details

The coordinates of the aromatic H atoms were idealized and refined using the riding model (AFIX 43 option of the SHELX-97 program [8]). The OH groups were idealized and refined using rigid groups allowed to rotate about the O—H bond (AFIX 147 option of the SHELX-97 program [8]).

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Yellow, block, size 0.23×0.29×0.38 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.19 cm$^{-1}$ |
| Diffractometer, scan mode: | CCD area detector, $\varphi$ and $\omega$ scans |
| $2\theta_{max}$: | 51° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 9510, 4196 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3218 |
| $N(param)_{refined}$: | 347 |
| Programs: | SHELXS-97 (SHELDRICK, 1990) |

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(3) | 2*i* | −0.353 | 1.0582 | 0.0399 | 0.049 |
| H(4A) | 2*i* | −0.0019 | 1.3133 | 0.2061 | 0.046 |
| H(6A) | 2*i* | 0.0608 | 0.9504 | 0.154 | 0.041 |
| H(8) | 2*i* | 0.0739 | 0.9959 | 0.35 | 0.05 |
| H(9A) | 2*i* | 0.2291 | 0.8686 | 0.4217 | 0.047 |
| H(11) | 2*i* | 0.6891 | 1.0534 | 0.306 | 0.05 |
| H(12) | 2*i* | 0.5336 | 1.1788 | 0.2333 | 0.052 |
| H(16) | 2*i* | 0.4431 | 0.2555 | 0.7755 | 0.046 |
| H(18) | 2*i* | 0.316 | 0.5964 | 0.8332 | 0.048 |
| H(19) | 2*i* | 0.7579 | 0.5472 | 0.9466 | 0.047 |
| H(21) | 2*i* | −0.1287 | 0.3382 | 0.7126 | 0.051 |
| H(22) | 2*i* | −0.2834 | 0.4605 | 0.6358 | 0.048 |
| H(24) | 2*i* | 0.1925 | 0.7008 | 0.5713 | 0.046 |
| H(25) | 2*i* | 0.3481 | 0.5799 | 0.6496 | 0.051 |
| H(1) | 2*i* | −0.3955 | 0.7564 | −0.0417 | 0.068 |
| H(5) | 2*i* | 0.79 | 0.8503 | 0.442 | 0.083 |
| H(6) | 2*i* | 0.9546 | 0.3136 | 0.9362 | 0.107 |
| H(9) | 2*i* | −0.3772 | 0.6862 | 0.5208 | 0.08 |

## Discussion

In recent years, the design and construction of coordination polymers have attracted increasing interest not only owing to their fascinating topological structures, but also due to their potential applications in luminescence, catalysis, gas

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**Table 3:** Atomic displacement parameters (Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| C(1) | 2*i* | −0.2219(2) | 0.8495(2) | 0.0366(1) | 0.0329(8) | 0.0362(9) | 0.0345(8) | 0.0145(7) | −0.0030(7) | 0.0019(7) |
| C(2) | 2*i* | −0.1585(2) | 0.9830(2) | 0.0881(1) | 0.0334(8) | 0.0344(9) | 0.0313(8) | 0.0138(7) | −0.0001(7) | 0.0021(7) |
| C(3) | 2*i* | −0.2487(2) | 1.0793(2) | 0.0784(1) | 0.0388(9) | 0.040(1) | 0.045(1) | 0.0179(8) | −0.0093(8) | −0.0028(8) |
| C(4) | 2*i* | −0.0434(2) | 1.2287(2) | 0.1762(1) | 0.041(1) | 0.0345(9) | 0.0414(9) | 0.0152(8) | −0.0009(8) | −0.0023(7) |
| C(5) | 2*i* | 0.0538(2) | 1.1382(2) | 0.1908(1) | 0.0329(8) | 0.0369(9) | 0.0332(8) | 0.0113(7) | −0.0013(7) | 0.0020(7) |
| C(6) | 2*i* | −0.0028(2) | 1.0128(2) | 0.1459(1) | 0.0360(9) | 0.0345(9) | 0.0363(9) | 0.0161(7) | −0.0017(7) | 0.0028(7) |
| C(7) | 2*i* | 0.2907(2) | 1.0994(2) | 0.2857(1) | 0.0394(9) | 0.0325(9) | 0.0389(9) | 0.0123(7) | −0.0115(7) | −0.0047(7) |
| C(8) | 2*i* | 0.1975(2) | 1.0070(2) | 0.3414(1) | 0.0295(9) | 0.047(1) | 0.048(1) | 0.0109(8) | −0.0032(8) | 0.0017(8) |
| C(9) | 2*i* | 0.2905(2) | 0.9314(2) | 0.3841(1) | 0.0351(9) | 0.0404(9) | 0.0386(9) | 0.0063(8) | −0.0017(7) | 0.0043(7) |
| C(10) | 2*i* | 0.4756(2) | 0.9482(2) | 0.3715(1) | 0.0344(9) | 0.0341(9) | 0.0343(9) | 0.0087(7) | −0.0047(7) | −0.0020(7) |
| C(11) | 2*i* | 0.5655(2) | 1.0416(2) | 0.3148(1) | 0.0318(9) | 0.043(1) | 0.051(1) | 0.0116(8) | 0.0024(8) | 0.0066(8) |
| C(12) | 2*i* | 0.4732(2) | 1.1168(2) | 0.2716(1) | 0.043(1) | 0.040(1) | 0.046(1) | 0.0093(8) | 0.0037(8) | 0.0091(8) |
| C(13) | 2*i* | 0.5749(2) | 0.8681(2) | 0.4182(1) | 0.0328(9) | 0.042(1) | 0.0380(9) | 0.0096(8) | −0.0029(7) | −0.0002(8) |
| C(14) | 2*i* | 0.7412(3) | 0.3006(2) | 0.8812(1) | 0.051(1) | 0.044(1) | 0.043(1) | 0.0238(9) | −0.0072(8) | 0.0009(8) |
| C(15) | 2*i* | 0.6197(2) | 0.3872(2) | 0.8652(1) | 0.0421(9) | 0.0367(9) | 0.0351(9) | 0.0177(8) | −0.0012(7) | 0.0040(7) |
| C(16) | 2*i* | 0.4688(2) | 0.3400(2) | 0.8057(1) | 0.046(1) | 0.0316(9) | 0.0407(9) | 0.0154(8) | −0.0031(8) | 0.0007(7) |
| C(17) | 2*i* | 0.3570(2) | 0.4205(2) | 0.7920(1) | 0.0399(9) | 0.0352(9) | 0.0417(9) | 0.0126(8) | −0.0067(8) | 0.0038(7) |
| C(18) | 2*i* | 0.3952(2) | 0.5441(2) | 0.8403(1) | 0.043(1) | 0.0353(9) | 0.045(1) | 0.0190(8) | −0.0067(8) | 0.0027(8) |
| C(19) | 2*i* | 0.6539(2) | 0.5141(2) | 0.9083(1) | 0.042(1) | 0.0390(9) | 0.0393(9) | 0.0167(8) | −0.0070(8) | 0.0007(7) |
| C(20) | 2*i* | 0.1232(2) | 0.4496(2) | 0.6899(1) | 0.044(1) | 0.0340(9) | 0.0413(9) | 0.0160(8) | −0.0122(8) | −0.0048(7) |
| C(21) | 2*i* | −0.0645(2) | 0.4120(2) | 0.6850(1) | 0.044(1) | 0.0360(9) | 0.046(1) | 0.0071(8) | −0.0043(8) | 0.0028(8) |
| C(22) | 2*i* | −0.1566(2) | 0.4849(2) | 0.6388(1) | 0.0309(9) | 0.042(1) | 0.045(1) | 0.0084(8) | −0.0058(7) | 0.0009(8) |
| C(23) | 2*i* | −0.0615(2) | 0.5944(2) | 0.5967(1) | 0.0361(9) | 0.0381(9) | 0.0302(8) | 0.0118(7) | −0.0042(7) | −0.0035(7) |
| C(24) | 2*i* | 0.1279(2) | 0.6286(2) | 0.6005(1) | 0.0361(9) | 0.042(1) | 0.0370(9) | 0.0089(8) | −0.0014(7) | 0.0030(7) |
| C(25) | 2*i* | 0.2213(2) | 0.5567(2) | 0.6471(1) | 0.0321(9) | 0.048(1) | 0.047(1) | 0.0126(8) | −0.0054(8) | −0.0015(8) |
| C(26) | 2*i* | −0.1614(2) | 0.6719(2) | 0.5472(1) | 0.0351(9) | 0.042(1) | 0.0343(9) | 0.0105(8) | −0.0031(7) | −0.0014(8) |
| N(1) | 2*i* | 0.5427(2) | 0.5905(1) | 0.89671(9) | 0.0457(8) | 0.0351(8) | 0.0401(8) | 0.0174(7) | −0.0059(7) | −0.0007(6) |
| N(2) | 2*i* | −0.1931(2) | 1.2012(1) | 0.1217(1) | 0.0451(9) | 0.0383(8) | 0.0526(9) | 0.0222(7) | −0.0075(7) | −0.0038(7) |
| O(1) | 2*i* | −0.3684(2) | 0.8314(1) | −0.01562(8) | 0.0452(7) | 0.0396(7) | 0.0538(8) | 0.0209(6) | −0.0198(6) | −0.0099(6) |
| O(2) | 2*i* | −0.1418(2) | 0.7650(1) | 0.04310(9) | 0.0503(7) | 0.0391(7) | 0.0642(8) | 0.0257(6) | −0.0187(6) | −0.0071(6) |
| O(3) | 2*i* | 0.2090(2) | 1.1862(1) | 0.24655(9) | 0.0474(7) | 0.0342(7) | 0.0595(8) | 0.0109(6) | −0.0220(6) | −0.0009(6) |
| O(4) | 2*i* | 0.4890(2) | 0.7769(1) | 0.46379(9) | 0.0403(7) | 0.0511(8) | 0.0653(8) | 0.0086(6) | −0.0041(6) | 0.0215(7) |
| O(5) | 2*i* | 0.7471(2) | 0.8954(1) | 0.4097(1) | 0.0383(7) | 0.0676(9) | 0.0673(9) | 0.0221(6) | 0.0040(6) | 0.0261(7) |
| O(6) | 2*i* | 0.8922(2) | 0.3645(1) | 0.9286(1) | 0.070(1) | 0.0555(9) | 0.095(1) | 0.0375(8) | −0.0409(9) | −0.0148(8) |
| O(7) | 2*i* | 0.7065(2) | 0.1867(1) | 0.8516(1) | 0.070(1) | 0.0450(8) | 0.080(1) | 0.0340(7) | −0.0199(8) | −0.0110(7) |
| O(8) | 2*i* | 0.2047(2) | 0.3680(1) | 0.7345(1) | 0.0535(8) | 0.0347(7) | 0.0745(9) | 0.0146(6) | −0.0295(7) | −0.0028(6) |
| O(9) | 2*i* | −0.3349(2) | 0.6363(1) | 0.5488(1) | 0.0358(7) | 0.0616(9) | 0.0659(9) | 0.0152(6) | −0.0084(6) | 0.0169(7) |
| O(10) | 2*i* | −0.0747(2) | 0.7672(1) | 0.50576(9) | 0.0452(7) | 0.0561(8) | 0.0605(8) | 0.0166(6) | 0.0030(6) | 0.0220(7) |

storage, separation and magnetism [1–3]. However, it is a great challenge to control structures with desired properties owing to many complicated factors. To design and construct coordination polymers, the reasonable selection of well designed organic ligand is very important [4, 5] and many multicarboxylate or heterocyclic carboxylic acids are used for this purpose. The V-shaped 5-(4-carboxyphenoxy)nicotinic acid ligand containing a pyridyl and a phenyl ring with structural flexibility and conformation freedom may show versatile coordination modes, which makes it a useful bridge to construct coordination polymers [6, 7]. Presented herein is the crystal structure of the 5-(4-carboxyphenoxy)nicotinic acid.

The title compound, $C_{13}H_9NO_5$, was prepared under hydrothermal conditions. Single crystal X-ray structural analysis reveals that the title compound crystallizes in the triclinic space group $P\bar{1}$ and the asymmetric unit of the crystal structure contains two independent $C_{13}H_9NO_5$ molecules. In crystal structure of the title compound, the phenyl rings and adjacent pyridine rings are not coplanar, making dihedral angles of 69.554(47) and 60.959(47), respectively. There are four different types of intermolecular H-bonds forming a double chain structure. Moreover, the chains are bound together to form a two-dimensional layer supramolecular structure by C–H$\cdots\pi$ interactions with an edge-to-face orientation ($d = 2.750$ Å; $A = 146°$ in the C–H$\cdots\pi$ patterns).

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## References

1. Gao, E. Q.; Bai, S. Q.; Wang, Z. M.; Yan, C. H.: Two-dimensional homochiral manganese(II)-azido frameworks incorporating an achiral ligand: partial spontaneous resolution and weak ferromagnetism. J. Am. Chem. Soc. **125** (2003) 4984–4985.
2. Kitagawa, S.; Kitaura, R.; Noro, S.: Functional porous coordination polymers. Angew. Chem., Int. Ed. **43** (2004) 2334–2375.
3. Bünzli, J. C. G.; Piguet, C.: Lanthanide-containing molecular and supramolecular polymetallic functional assemblies. Chem Rev. **102** (2002) 1897–1928.
4. Sun, C. Y.; Zheng, X. J.; Gao, S.; Li, L. C.; Jin, L. P.: Multiple regulated assembly, crystal structures and magnetic properties of porous coordination polymers with flexible ligands. Eur. J. Inorg. Chem. (2005) 4150–4159.
5. Han, Z. B.; Chen, X. N.; Chen, X. M.: Effect of the size of aromatic chelate ligands on the frameworks of metal dicarboxylate polymers: from helical chains to 2-D networks. Cryst. Growth Des. **5** (2005) 695–700.
6. Xu, J.; Su, W. P.; Hong, M. C.: Two lanthanide-natrium pillared-layer frameworks constructed from 4,4′-oxybis(benzoic acid) and oxalate. Inorg. Chem. Commun. **14** (2011) 1794–1797.
7. Mahata, P.; Draznieks, C. M.; Roy, P.; Natarajan, S.: Solid state and solution mediated multistep sequential transformations in metal-organic coordination networks. Cryst. Growth Des. **13** (2013) 155–168.
8. Sheldrick, G. M.: SHELXL97. Program for crystal structure refinement. University of Göttingen, Germany 1997.