Yong Li* and Jingjing Wang

The crystal structure of 4-(methoxycarbonyl) benzoic acid, $C_9H_8O_4$

Table 1: Data collection and handling.

Crystal:	Plate, colorless
Size:	$1 \times 0.5 \times 0.5$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.12 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	24.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4168, 1381, 0.044
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1234
$N(\text{param})_{\text{refined}}$:	142
Programs:	Bruker programs [1], SHELX [2], Diamond [3]

<https://doi.org/10.1515/ncrs-2018-0408>

Received October 1, 2018; accepted October 24, 2018; available online November 16, 2018

Abstract

$C_9H_8O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 4.8541(19)$ Å, $b = 5.880(2)$ Å, $c = 29.1938(11)$ Å, $\beta = 91.430(5)^\circ$, $V = 833.0(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0477$, $wR_{\text{ref}}(F^2) = 0.1259$, $T = 296(2)$ K.

CCDC no.: 1874996

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The synthetic methods of the title compound is esterification of dicarboxylic acids, reported in the literature [4].

The commercially crude compound was recrystallized two times from MeOH/water (1:1) solution, and then colourless plate crystals were obtained by slow evaporation at room temperature within 3 days.

Experimental details

The hydrogen atoms of phenyl and methyl can be seen as Q-peaks and located in the difference electron density, so they could be refined freely. The hydrogen of hydroxyl is found from the Fourier synthesis at first, and then refined further

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.3318(3)	−0.1037(3)	0.45175(5)	0.0606(5)
O2	1.2710(3)	0.2185(3)	0.49001(5)	0.0641(5)
O3	0.3215(3)	0.2300(3)	0.29061(6)	0.0671(5)
O4	0.3219(3)	0.5704(2)	0.32388(5)	0.0552(5)
C1	0.6029(4)	0.2883(3)	0.35776(6)	0.0384(5)
C2	0.7295(4)	0.0786(3)	0.35459(7)	0.0474(5)
C3	0.9276(4)	0.0128(4)	0.38676(7)	0.0475(5)
C4	0.9980(4)	0.1564(3)	0.42301(6)	0.0393(5)
C5	0.8676(4)	0.3648(4)	0.42667(7)	0.0488(5)
C6	0.6715(4)	0.4310(3)	0.39419(7)	0.0468(5)
C7	1.2139(4)	0.0862(3)	0.45716(6)	0.0426(5)
C8	0.4002(4)	0.3557(3)	0.32077(6)	0.0414(5)
C9	0.1358(5)	0.6526(4)	0.28770(7)	0.0602(6)
H9A	0.2099	0.6165	0.2584	0.090*
H9B	0.1155	0.8144	0.2904	0.090*
H9C	−0.0409	0.5812	0.2905	0.090*
H1	1.411(7)	0.188(10)	0.5079(15)	0.23(3)*
H2	0.679(5)	−0.020(4)	0.3302(8)	0.063(7)*
H3	1.011(5)	−0.130(4)	0.3844(7)	0.052(6)*
H4	0.915(5)	0.469(4)	0.4515(8)	0.062(6)*
H5	0.584(5)	0.573(4)	0.3962(8)	0.060(6)*

with restrained distance 0.85 Å to oxygen atom, standard deviation 0.01.

Comment

The title compound, monomethyl terephthalate (MMT), is used as intermediate to synthesize antineoplastic agent Bexarotene [5], and used to synthesize a super fiber material, Poly (p-phenylene benzothiazole), PBO [6].

*Corresponding author: Yong Li, Department of Chemical Engineering, Ningbo Polytechnic, Beilun District, Ningbo City, Zhejiang Province 315806, P.R. China, e-mail: 25660315@qq.com
Jingjing Wang: Department of Student Affairs, Ningbo Polytechnic, Beilun District, Ningbo City, Zhejiang Province 315806, P.R. China

The asymmetric unit of the title structure contains one molecule (*cf.* left part of the figure). Bond lengths and angles are all in the expected ranges.

Hydrogen bonds of O2—H1···O1 [3-*x*, -*y*, 1-*z*] with H···A = 1.77(4) Å connect two carboxyl groups of neighboring molecules to form an eight-membered ring (*cf.* right part of the figure). This type of eight-membered ring is reported for many carboxylic acids. It would lead to far to mention all of them, thus some instructive examples are listed as references [7–9]. The detail of this eight-membered ring deserves further investigation in the future.

Acknowledgements: We gratefully acknowledge support by Ningbo Polytechnic, P.R. China for financial support.

References

1. Bruker AXS Inc., Bruker SMART and SAINT, Madison, WI, USA (2007).
2. Sheldrick, G. M.: [Crystal structure refinement with SHELXL](#). Acta Crystallogr. **C71** (2015) 3–8.
3. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
4. Ogawa, H.; Chihara, T.; Taya, K.: Selective monomethyl esterification of dicarboxylic acids by use of monocarboxylate chemisorption on alumina. J. Am. Chem. Soc. **107** (1985) 1365–1369.
5. Boehm, M. F.; Zhang, L.; Badea, B. A.; White, S. K.; Mais, D. E.; Berger, E.; Suto, C. M.; Goldman, M. E.; Heyman, R. A.: Synthesis and structure-activity relationships of novel retinoid X receptor-selective retinoids. J. Med. Chem. **37** (1994) 2930–2941.
6. Chow, A. W.; Bitler, S. P.; Penwell, P. E.; Osborne, D. J.; Wolfe, J. F.: Synthesis and solution properties of extended chain poly(2,6-benzothiazole) and poly(2,5-benzoxazole). Macromolecules **22** (1989) 3514–3520.
7. Aakeröy, C. B.; Desper, J.; Helfrich, B. A.: [Heteromeric intermolecular interactions as synthetic tools for the formation of binary co-crystals](#). CrystEngComm **6** (2004) 19–24.
8. García-Rodríguez, R.; Miguel, D.: Molybdenum carbonyl complexes with pyridylimino acidato ligands. Dalton Trans. **9** (2006) 1218–1225.
9. Novoa de Armas, H.; Pomes Hernandez, R.; Duque Rodriguez, J.; Toscano, R. A.: 4,4'-Dichloro-2,2'-iminodibenzoic acid. Acta Crystallogr. Sect. C **52** (1996) 166–168.