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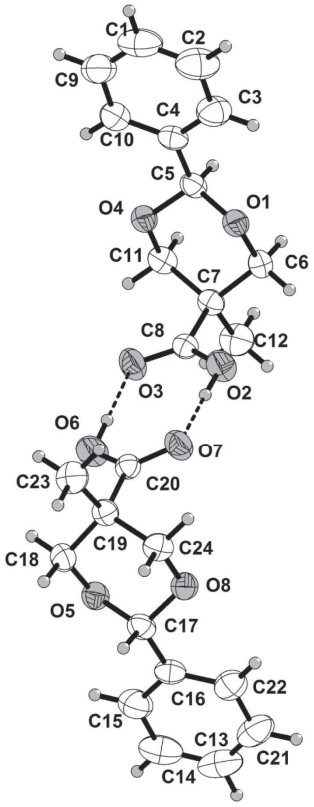
Xian-You Yuan*, Guo-Kai Jia, Min Zhang and Lin Yuan

# Crystal structure of 5-methyl-2-phenyl-1,3-dioxane-5-carboxylic acid, $C_{12}H_{14}O_4$

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## Abstract

$C_{12}H_{14}O_4$, monoclinic, $C2/c$, $a = 32.6674(8)$ Å, $b = 6.0061(2)$ Å, $c = 23.2786(6)$ Å, $\alpha = 90°$, $\beta = 103.368(2)°$, $\gamma = 90°$, $V = 4443.6(2)$ Å$^3$, $Z = 16$, $R_{gt}(F) = 0.0459$, $wR_{ref}(F^2) = 0.1412$, $T = 298(2)$ K.

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***Corresponding author: Xian-You Yuan,** Key Laboratory of Comprehensive Utilization of Advantage Plants Resources in Hunan South, Hunan University of Science and Engineering, Yongzhou Hunan 425199, P. R. China, e-mail: yuanxianzhangmin@163.com
**Guo-Kai Jia, Min Zhang and Lin Yuan:** Key Laboratory of Comprehensive Utilization of Advantage Plants Resources in Hunan South, Hunan University of Science and Engineering, Yongzhou Hunan 425199, P. R. China

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

| | |
|---|---|
| Crystal: | Colorless, block, size 0.23×0.41×0.46 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.00 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II CCD, $\varphi$ and $\omega$ scans |
| $2\theta_{max}$: | 55° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 17586, 5049 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 4119 |
| $N(param)_{refined}$: | 297 |
| Programs: | SHELXS-97 [7, 8] |

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

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 8*f* | 0.2916(6) | 0.473(3) | 0.3740(8) | 0.073 |
| H(16) | 8*f* | 0.2744(6) | 0.559(4) | 0.4621(8) | 0.078 |
| H(1) | 8*f* | 0.0603 | 1.0532 | 0.1470 | 0.082 |
| H(12) | 8*f* | 0.1091 | 0.8789 | 0.1057 | 0.084 |
| H(11) | 8*f* | 0.1492 | 0.5924 | 0.1548 | 0.068 |
| H(10) | 8*f* | 0.1337 | 0.2556 | 0.2367 | 0.043 |
| H(9) | 8*f* | 0.2386 | 0.1893 | 0.2602 | 0.048 |
| H(8) | 8*f* | 0.1953 | 0.0633 | 0.2448 | 0.048 |
| H(14) | 8*f* | 0.0517 | 0.9414 | 0.2379 | 0.080 |
| H(13) | 8*f* | 0.0917 | 0.6536 | 0.2879 | 0.064 |
| H(4) | 8*f* | 0.1509 | 0.0591 | 0.3246 | 0.047 |
| H(3) | 8*f* | 0.1691 | 0.1864 | 0.3839 | 0.047 |
| H(6) | 8*f* | 0.2573 | −0.0909 | 0.3465 | 0.079 |
| H(7) | 8*f* | 0.2114 | −0.1865 | 0.3300 | 0.079 |
| H(5) | 8*f* | 0.2300 | −0.0940 | 0.3937 | 0.079 |
| H(15) | 8*f* | 0.5200 | −0.0561 | 0.6442 | 0.083 |
| H(17) | 8*f* | 0.4751 | 0.0870 | 0.6976 | 0.087 |
| H(18) | 8*f* | 0.4312 | 0.3818 | 0.6611 | 0.070 |
| H(26) | 8*f* | 0.4401 | 0.7503 | 0.5825 | 0.046 |
| H(19) | 8*f* | 0.3797 | 0.9379 | 0.5895 | 0.049 |
| H(25) | 8*f* | 0.3367 | 0.8101 | 0.5788 | 0.049 |
| H(28) | 8*f* | 0.5189 | 0.0827 | 0.5517 | 0.075 |
| H(27) | 8*f* | 0.4755 | 0.3770 | 0.5145 | 0.060 |
| H(22) | 8*f* | 0.3325 | 1.1335 | 0.4471 | 0.075 |
| H(21) | 8*f* | 0.3569 | 1.2104 | 0.5101 | 0.075 |
| H(20) | 8*f* | 0.3112 | 1.1143 | 0.5008 | 0.075 |
| H(24) | 8*f* | 0.3926 | 0.8616 | 0.4407 | 0.049 |
| H(23) | 8*f* | 0.4157 | 0.9710 | 0.5007 | 0.049 |

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

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 8*f* | 0.18934(3) | 0.3912(2) | 0.24551(4) | 0.0400(5) | 0.0446(6) | 0.0323(5) | 0.0006(4) | 0.0098(4) | 0.0024(4) |
| O(2) | 8*f* | 0.27339(3) | 0.3853(2) | 0.34901(5) | 0.0436(6) | 0.0597(7) | 0.0444(6) | −0.0171(5) | 0.0132(5) | −0.0083(5) |
| O(3) | 8*f* | 0.23661(3) | 0.3879(2) | 0.41775(5) | 0.0466(6) | 0.0644(7) | 0.0405(6) | −0.0149(5) | 0.0138(5) | −0.0170(5) |
| O(4) | 8*f* | 0.14921(3) | 0.3867(2) | 0.31569(4) | 0.0386(5) | 0.0452(6) | 0.0332(5) | 0.0003(4) | 0.0097(4) | 0.0012(4) |
| O(5) | 8*f* | 0.38551(3) | 0.6119(2) | 0.58090(4) | 0.0391(5) | 0.0463(6) | 0.0367(5) | −0.0026(4) | 0.0115(4) | 0.0016(4) |
| O(6) | 8*f* | 0.29423(4) | 0.6389(2) | 0.48855(5) | 0.0433(6) | 0.0640(7) | 0.0491(6) | −0.0205(5) | 0.0121(5) | −0.0083(5) |
| O(7) | 8*f* | 0.32670(3) | 0.6565(2) | 0.41481(5) | 0.0470(6) | 0.0646(7) | 0.0415(6) | −0.0116(5) | 0.0101(5) | −0.0153(5) |
| O(8) | 8*f* | 0.41766(3) | 0.6423(2) | 0.50202(4) | 0.0376(5) | 0.0466(6) | 0.0377(5) | 0.0000(4) | 0.0110(4) | 0.0022(4) |
| C(1) | 8*f* | 0.07647(6) | 0.9375(3) | 0.16692(9) | 0.065(1) | 0.0454(9) | 0.077(1) | 0.0000(8) | −0.019(1) | 0.0060(9) |
| C(2) | 8*f* | 0.10546(6) | 0.8336(4) | 0.14244(9) | 0.065(1) | 0.072(1) | 0.067(1) | −0.004(1) | 0.0017(9) | 0.028(1) |
| C(3) | 8*f* | 0.12947(6) | 0.6615(3) | 0.17187(8) | 0.0518(9) | 0.066(1) | 0.0506(9) | 0.0020(8) | 0.0103(7) | 0.0151(8) |
| C(4) | 8*f* | 0.12460(4) | 0.5904(2) | 0.22634(6) | 0.0360(7) | 0.0416(7) | 0.0375(7) | −0.0058(6) | −0.0016(5) | −0.0026(6) |
| C(5) | 8*f* | 0.14822(4) | 0.3897(2) | 0.25489(6) | 0.0347(7) | 0.0414(7) | 0.0305(6) | −0.0055(5) | 0.0038(5) | −0.0030(5) |
| C(6) | 8*f* | 0.21027(5) | 0.1876(2) | 0.26667(6) | 0.0430(8) | 0.0416(7) | 0.0346(7) | 0.0026(6) | 0.0089(6) | −0.0070(6) |
| C(7) | 8*f* | 0.21231(4) | 0.1572(2) | 0.33236(5) | 0.0378(7) | 0.0296(6) | 0.0343(7) | −0.0035(5) | 0.0053(5) | −0.0011(5) |
| C(8) | 8*f* | 0.24208(4) | 0.3246(2) | 0.36905(5) | 0.0345(6) | 0.0298(6) | 0.0315(6) | −0.0001(5) | 0.0060(5) | 0.0023(5) |
| C(9) | 8*f* | 0.07134(6) | 0.8705(3) | 0.22111(9) | 0.059(1) | 0.062(1) | 0.067(1) | 0.0151(9) | −0.0086(9) | −0.0181(9) |
| C(10) | 8*f* | 0.09534(5) | 0.6975(3) | 0.25106(7) | 0.0518(9) | 0.059(1) | 0.0447(8) | 0.0068(8) | 0.0022(7) | −0.0077(7) |
| C(11) | 8*f* | 0.16801(4) | 0.1847(2) | 0.34186(6) | 0.0401(7) | 0.0396(7) | 0.0365(7) | −0.0091(6) | 0.0074(5) | 0.0030(5) |
| C(12) | 8*f* | 0.22935(6) | −0.0753(3) | 0.35250(8) | 0.062(1) | 0.0319(7) | 0.060(1) | 0.0020(7) | 0.0059(8) | 0.0025(7) |
| C(13) | 8*f* | 0.50190(6) | 0.0598(3) | 0.6288(1) | 0.047(1) | 0.054(1) | 0.093(2) | −0.0001(8) | −0.0133(9) | 0.006(1) |
| C(14) | 8*f* | 0.47540(7) | 0.1471(4) | 0.66086(9) | 0.069(1) | 0.075(1) | 0.060(1) | −0.003(1) | −0.0104(9) | 0.0182(9) |
| C(15) | 8*f* | 0.44907(6) | 0.3241(3) | 0.63907(7) | 0.057(1) | 0.070(1) | 0.0454(9) | 0.0028(9) | 0.0034(7) | 0.0058(8) |
| C(16) | 8*f* | 0.44948(4) | 0.4149(3) | 0.58440(6) | 0.0330(7) | 0.0445(8) | 0.0428(8) | −0.0077(6) | −0.0010(6) | −0.0013(6) |
| C(17) | 8*f* | 0.42429(4) | 0.6204(2) | 0.56379(6) | 0.0343(7) | 0.0433(8) | 0.0355(7) | −0.0082(5) | 0.0045(5) | −0.0027(5) |
| C(18) | 8*f* | 0.36336(5) | 0.8184(2) | 0.56731(6) | 0.0406(7) | 0.0440(8) | 0.0376(7) | −0.0011(6) | 0.0093(6) | −0.0078(6) |
| C(19) | 8*f* | 0.35547(4) | 0.8682(2) | 0.50144(6) | 0.0365(7) | 0.0328(6) | 0.0372(7) | −0.0040(5) | 0.0075(5) | −0.0027(5) |
| C(20) | 8*f* | 0.32389(4) | 0.7070(2) | 0.46566(6) | 0.0310(6) | 0.0321(6) | 0.0351(6) | −0.0002(5) | 0.0066(5) | −0.0011(5) |
| C(21) | 8*f* | 0.50156(5) | 0.1443(3) | 0.5740(1) | 0.0373(8) | 0.056(1) | 0.092(2) | −0.0004(7) | 0.0089(8) | −0.0078(9) |
| C(22) | 8*f* | 0.47550(5) | 0.3209(3) | 0.55177(8) | 0.0382(8) | 0.0523(9) | 0.060(1) | −0.0072(7) | 0.0103(7) | −0.0011(7) |
| C(23) | 8*f* | 0.33733(6) | 1.1036(2) | 0.48868(8) | 0.0557(9) | 0.0346(8) | 0.0589(9) | −0.0004(6) | 0.0102(7) | −0.0024(6) |
| C(24) | 8*f* | 0.39745(4) | 0.8494(2) | 0.48327(7) | 0.0371(7) | 0.0417(7) | 0.0427(7) | −0.0091(6) | 0.0085(6) | 0.0041(6) |

## Source of material

2,2-Bis(hydroxymethyl)propionic acid (0.5008 g, 3.7337 mmol), benzaldehyde (0.4351 g, 4.1000 mmol), cyclohexane (12 mL), N,N-dimethytformamide (2 mL) and p-toluene sulfonic acid (0.0281 g, 0.1630 mmol) were heated and stirred at 373 K for four hours. Then sodium bicarbonate (0.0151 g, 0.1793 mmol) was added to dissolve the residue after the solvent was evaporated under reduced pressure. The solution was washed with water (20 mL), and dried with anhydrous sodium sulfate. The resulting solution was filtered and evaporated, and the product was recrystallized from ethyl acetate to afford colorless crystals (0.6359 g, 2.8611 mmol; yield 76.63%; m. p. 462.3 K).

## Discussion

The title compound was synthesized to be used as a reactive intermediate in organic syntheses [1]. This class of compounds has insecticidal as well as anti-foaming properties [2, 3]. The title compound 5-methyl-2-phenyl-1,3-dioxane-5-carboxylic acid was synthesized by the reaction of 2,2-Bis(hydroxymethyl) propionic acid with benzaldehyde and p-toluene sulfonic acid as catalyst. The crystal structures of some similar 1,3-dioxanes have been reported in the last years [4–6]. The literature known 4-chloro derivative [5] is isotypic to the title structure.

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