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Crystal structure of 3-(2,5-dimethoxyphenyl)propionic acid

Bernhard Bugenhagen,[a] Yosef Al Jasem,[b] Mariam AlAzani[c] and Thies Thiemann[c]*

[a]Institute of Inorganic Chemistry, University of Hamburg, Hamburg, Germany, [b]Department of Chemical Engineering, and [c]Department of Chemistry, United Arab Emirates University, AL Ain, Abu Dhabi, United Arab Emirates. *Correspondence e-mail: thies@uaeu.ac.ae

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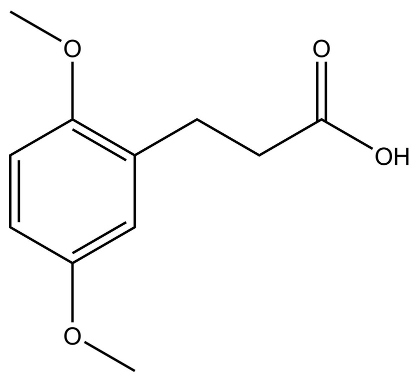
In the crystal of the title compound, $C_{11}H_{14}O_4$, the aromatic ring is almost coplanar with the 2-position methoxy group with which it subtends a dihedral of 0.54 (2)°, while the 5-position methoxy group makes a corresponding dihedral angle of just 5.30 (2)°. The angle between the mean planes of the aromatic ring and the propionic acid group is 78.56 (2)°. The fully extended propionic side chain is in a *trans* configuration with a C—C—C—C torsion angle of −172.25 (7)°. In the crystal, hydrogen bonding is limited to dimer formation *via* $R_2^2(8)$ rings. The hydrogen-bonded dimers are stacked along the *b* axis. The average planes of the two benzene rings in a dimer are parallel to each other, but at an offset of 4.31 (2) Å. Within neighbouring dimers along the [101] direction, the average molecular benzene planes are almost perpendicular to each other, with a dihedral angle of 85.33 (2)°.

Keywords: crystal structure; 3-(2,5-dimethoxyphenyl)propionic acid; O—H···O hydrogen bonding.

CCDC reference: 1060285

1. Related literature

For another preparation method of the title compound, see: Anliker *et al.* (1957). For crystal structures of phenylpropionic acids, see: Das *et al.* (2012). For the application of the title compound as a starting material for 19-norsteroidal derivatives, see: Anliker *et al.* (1957); and as a starting material for amidoethylquinones, see: Bremer *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{11}H_{14}O_4$
$M_r = 210.22$
Monoclinic, $C2/c$
$a = 24.3212\ (10)$ Å
$b = 4.6512\ (2)$ Å
$c = 19.7411\ (8)$ Å
$\beta = 109.1782\ (6)^\circ$
$V = 2109.23\ (15)$ Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 100$ K
0.3 × 0.1 × 0.02 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{\min} = 0.604$, $T_{\max} = 0.746$
20284 measured reflections
3224 independent reflections
2927 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.028$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.107$
$S = 1.05$
3224 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.92 (2)	1.75 (2)	2.6624 (11)	172.1 (18)

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2007); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: QM2110).

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supporting information

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Crystal structure of 3-(2,5-dimethoxyphenyl)propionic acid

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S1. Structural commentary

The molecule of the title compound exhibits one conformation (Figure 1), unlike other analogous compounds that exhibit two conformations (e.g. 3-phenylpropionic acid, 3-(3-methylphenyl)propionic acid and 3-(3-methoxyphenyl)propionic acid) (Das *et al.*, 2012). The aromatic ring of the title compound is almost coplanar with the C10 methoxyl with which it has a dihedral of less than 0.54 (2) ° while the C11 methoxyl has a corresponding dihedral of just 5.30 (2)°. The angle between the mean planes of the aromatic ring and the propionic acid group (C7, C8, C9, O3 and O4) is 78.56 (2) °. The fully extended propionic side chain is in a *trans* configuration with (C6—C7—C8—C9) torsion angle of -172.25 (2)°. The O4—H4···O3 hydrogen bonding (Table 1) of the COOH functional groups leads to dimer formation *via* $R^2_2(8)$ rings. The hydrogen bonded dimers are stacked along the *b* axis. The average planes of the two benzene rings in a dimer are parallel to each other, but at an offset of 4.31 (2) Å. Within neighboring dimers along [101] direction, the average molecular benzene planes are almost perpendicular to each other, with an angle of 85.33 (2)°. No other appreciable close contacts were noticed except a very weak C3—H3···π interaction between adjacent dimers along [101], with a bond length of 3.20 (2) Å.

S2. Synthesis and crystallization

3-(2,5-Dimethoxyphenyl)propionic acid. - Ethyl 3-(2,5-dimethoxyphenyl)propionate (3.2 g, 13.4 mmol) in a mixture of aq. NaOH (10 w%, 30 mL) and methanol (8 mL) was heated at reflux for 12h. Then, half. conc. aq. HCl is added to the cooled solution. Thereafter, the mixture is extracted with chloroform (3 X 15 mL). The organic phase is dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The residue is filtered over a small column of silica gel (diethylether–$CHCl_3$, 1:1, v/v) to give the title compound (2.56 g, 89%) as colorless needles, mp. 339 – 340 K [Lit. mp. 339-340 K (Anliker *et al.*, 1957)]; ν_{max} (KBr/cm^{-1}) 3500 – 2050 (bs, OH), 2955, 2835, 1699, 1504, 1449, 1430, 1307, 1281, 1182, 1127, 927, 916, 865, 795, 717, 499; δ_H (400 MHz, $CDCl_3$) 2.65 (2H, t, ^3J = 7.6 Hz), 2.91 (2H, t, ^3J = 7.6 Hz), 6.71 (1H, dd, ^3J = 8.4 Hz, ^4J = 3.2 Hz), 6.75 (1H, d, ^4J = 3.2 Hz), 6.76 (1H, d, ^3J = 8.4 Hz), δ_C (67.8 MHz, $CDCl_3$) 26.0 (CH_2), 33.9 (CH_2), 55.6 (OCH_3), 55.7 (OCH_3), 111.0 (CH), 111.6 (CH), 116.3 (CH), 129.6 (CH), 151.7 (C_{quat}), 153.3 (C_{quat}), 179.7 (C_{quat}, CO).

S3. Refinement

All hydrogen atoms were placed in calculated positions with C—H distances of 0.95- 0.99 Å and refined as riding with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl and x = 1.2 for all other H-atoms.

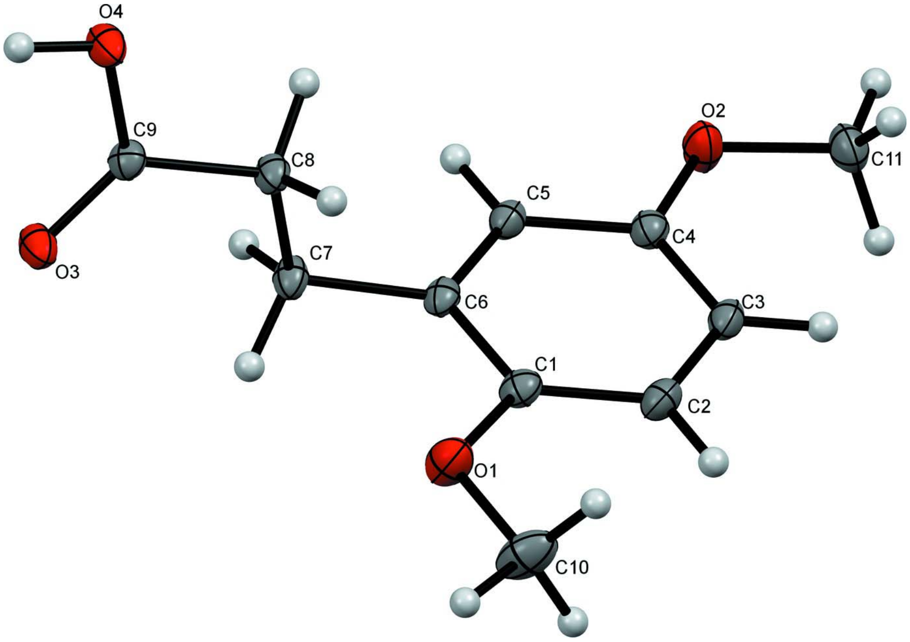

Figure 1

A view of title compound molecule with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

3-(2,5-Dimethoxyphenyl)propionic acid

Crystal data

$C_{11}H_{14}O_4$
$M_r = 210.22$
Monoclinic, $C2/c$
$a = 24.3212$ (10) Å
$b = 4.6512$ (2) Å
$c = 19.7411$ (8) Å
$\beta = 109.1782$ (6)°
$V = 2109.23$ (15) Å^3
$Z = 8$
$F(000) = 896$
$D_x = 1.324$ Mg m^{-3}
Melting point = 339–340 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9914 reflections
$\theta = 2.3–31.2°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Bar, clear light colourless
0.3 × 0.1 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
$T_{\min} = 0.604$, $T_{\max} = 0.746$
20284 measured reflections
3224 independent reflections
2927 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.028$
$\theta_{\max} = 31.3°$, $\theta_{\min} = 1.8°$
$h = -35 \rightarrow 34$
$k = -6 \rightarrow 6$
$l = -27 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.107$
$S = 1.05$
3224 reflections
142 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 1.4383P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Experimental. SADABS-2012/1 (Bruker,2012) was used for absorption correction. wR2(int) was 0.1419 before and 0.0438 after correction. The Ratio of minimum to maximum transmission is 0.8088. The $\lambda/2$ correction factor is 0.0015.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.36178 (4)	0.50978 (18)	0.70073 (5)	0.01639 (17)
C10	0.44839 (4)	0.3697 (3)	0.79439 (5)	0.0289 (2)
C11	0.14931 (4)	0.2622 (2)	0.61260 (5)	0.02223 (19)
C2	0.32768 (4)	0.32755 (19)	0.72601 (5)	0.01806 (17)
C3	0.26774 (4)	0.30632 (18)	0.69042 (5)	0.01692 (17)
C4	0.24216 (4)	0.47152 (18)	0.62979 (5)	0.01575 (16)
C5	0.27657 (4)	0.65750 (18)	0.60495 (4)	0.01568 (16)
C6	0.33612 (4)	0.67778 (17)	0.63904 (4)	0.01468 (16)
C7	0.37340 (4)	0.86255 (18)	0.60859 (5)	0.01664 (16)
C8	0.39755 (4)	0.67941 (18)	0.56036 (5)	0.01553 (16)
C9	0.44172 (3)	0.82948 (18)	0.53467 (4)	0.01464 (16)
H10A	0.4311	0.4146	0.8315	0.043*
H10B	0.4901	0.4129	0.8124	0.043*
H10C	0.4427	0.1653	0.7820	0.043*
H11A	0.1506	0.3038	0.6618	0.033*
H11B	0.1648	0.0690	0.6106	0.033*
H11C	0.1090	0.2724	0.5803	0.033*
H2	0.3452	0.2162	0.7679	0.022*
H3	0.2447	0.1796	0.7077	0.020*
H4	0.4791 (8)	0.795 (4)	0.4682 (10)	0.049 (5)*
H5	0.2587	0.7724	0.5638	0.019*
H7A	0.4059	0.9461	0.6481	0.020*
H7B	0.3498	1.0224	0.5804	0.020*
H8A	0.4156	0.5046	0.5871	0.019*
H8B	0.3647	0.6170	0.5181	0.019*
O1	0.42100 (3)	0.53955 (17)	0.73202 (4)	0.02384 (16)
O2	0.18373 (3)	0.46840 (16)	0.59092 (4)	0.02300 (16)
O3	0.46906 (3)	1.04245 (15)	0.56342 (4)	0.02097 (15)
O4	0.44928 (3)	0.70388 (15)	0.47828 (4)	0.02040 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0167 (4)	0.0191 (4)	0.0150 (4)	0.0013 (3)	0.0074 (3)	−0.0003 (3)
C10	0.0211 (4)	0.0448 (6)	0.0197 (4)	0.0072 (4)	0.0049 (3)	0.0065 (4)
C11	0.0206 (4)	0.0207 (4)	0.0263 (4)	−0.0063 (3)	0.0089 (3)	−0.0017 (3)
C2	0.0221 (4)	0.0190 (4)	0.0149 (4)	0.0013 (3)	0.0086 (3)	0.0031 (3)
C3	0.0218 (4)	0.0159 (4)	0.0162 (4)	−0.0015 (3)	0.0104 (3)	0.0008 (3)
C4	0.0170 (4)	0.0154 (3)	0.0162 (4)	−0.0009 (3)	0.0073 (3)	−0.0010 (3)
C5	0.0196 (4)	0.0144 (3)	0.0148 (3)	0.0004 (3)	0.0081 (3)	0.0015 (3)
C6	0.0190 (4)	0.0130 (3)	0.0155 (4)	0.0002 (3)	0.0103 (3)	−0.0010 (3)
C7	0.0201 (4)	0.0144 (3)	0.0200 (4)	−0.0012 (3)	0.0127 (3)	−0.0008 (3)
C8	0.0166 (4)	0.0159 (4)	0.0172 (4)	−0.0024 (3)	0.0099 (3)	−0.0016 (3)
C9	0.0137 (3)	0.0159 (4)	0.0160 (3)	0.0007 (3)	0.0072 (3)	−0.0003 (3)
O1	0.0167 (3)	0.0334 (4)	0.0207 (3)	0.0005 (3)	0.0053 (2)	0.0055 (3)
O2	0.0176 (3)	0.0258 (3)	0.0242 (3)	−0.0045 (2)	0.0049 (3)	0.0060 (3)
O3	0.0239 (3)	0.0205 (3)	0.0238 (3)	−0.0077 (2)	0.0150 (3)	−0.0069 (2)
O4	0.0208 (3)	0.0229 (3)	0.0233 (3)	−0.0074 (2)	0.0151 (3)	−0.0086 (2)

Geometric parameters (Å, º)

C10—H10C	0.9800	C6—C5	1.3854 (12)
C10—H10B	0.9800	C7—H7B	0.9900
C10—H10A	0.9800	C7—H7A	0.9900
C11—H11C	0.9800	C8—C9	1.5019 (11)
C11—H11B	0.9800	C8—C7	1.5312 (11)
C11—H11A	0.9800	C8—H8B	0.9900
C2—C1	1.3882 (12)	C8—H8A	0.9900
C2—H2	0.9500	O1—C10	1.4299 (12)
C3—C4	1.3858 (12)	O1—C1	1.3751 (10)
C3—C2	1.3985 (12)	O2—C11	1.4278 (11)
C3—H3	0.9500	O2—C4	1.3756 (10)
C5—C4	1.3995 (11)	O3—C9	1.2238 (10)
C5—H5	0.9500	O4—H4	0.917 (18)
C6—C1	1.4085 (12)	O4—C9	1.3224 (10)
C6—C7	1.5101 (11)		
C1—C2—H2	119.7	C9—C8—H8B	108.6
C1—C2—C3	120.65 (8)	C9—C8—H8A	108.6
C1—C6—C7	120.43 (8)	C9—O4—H4	108.5 (11)
C1—O1—C10	117.01 (7)	H10A—C10—H10C	109.5
C2—C1—C6	120.15 (8)	H10A—C10—H10B	109.5
C2—C3—H3	120.2	H10B—C10—H10C	109.5
C3—C4—C5	119.66 (8)	H11A—C11—H11C	109.5
C3—C2—H2	119.7	H11A—C11—H11B	109.5
C4—C5—H5	119.3	H11B—C11—H11C	109.5
C4—C3—C2	119.53 (8)	H7A—C7—H7B	108.2
C4—C3—H3	120.2	H8A—C8—H8B	107.6

C4—O2—C11	116.09 (7)	O1—C10—H10C	109.5
C5—C6—C1	118.54 (7)	O1—C10—H10B	109.5
C5—C6—C7	120.93 (7)	O1—C10—H10A	109.5
C6—C7—H7B	109.8	O1—C1—C2	124.15 (8)
C6—C7—H7A	109.8	O1—C1—C6	115.71 (7)
C6—C7—C8	109.52 (7)	O2—C11—H11C	109.5
C6—C5—C4	121.46 (8)	O2—C11—H11B	109.5
C6—C5—H5	119.3	O2—C11—H11A	109.5
C7—C8—H8B	108.6	O2—C4—C5	115.98 (7)
C7—C8—H8A	108.6	O2—C4—C3	124.36 (8)
C8—C7—H7B	109.8	O3—C9—C8	124.03 (7)
C8—C7—H7A	109.8	O3—C9—O4	122.90 (8)
C9—C8—C7	114.48 (7)	O4—C9—C8	113.05 (7)

C1—C6—C7—C8	83.52 (9)	C5—C6—C1—C2	0.36 (12)
C1—C6—C5—C4	−1.12 (12)	C5—C6—C1—O1	179.97 (7)
C10—O1—C1—C2	0.15 (13)	C5—C6—C7—C8	−92.96 (9)
C10—O1—C1—C6	−179.45 (8)	C6—C5—C4—C3	0.97 (13)
C11—O2—C4—C5	175.08 (8)	C6—C5—C4—O2	−179.31 (7)
C11—O2—C4—C3	−5.21 (13)	C7—C8—C9—O3	19.85 (12)
C2—C3—C4—C5	−0.03 (13)	C7—C8—C9—O4	−161.63 (7)
C2—C3—C4—O2	−179.73 (8)	C7—C6—C1—C2	−176.20 (8)
C3—C2—C1—C6	0.56 (13)	C7—C6—C1—O1	3.41 (11)
C3—C2—C1—O1	−179.02 (8)	C7—C6—C5—C4	175.42 (7)
C4—C3—C2—C1	−0.73 (13)	C9—C8—C7—C6	−172.25 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.92 (2)	1.75 (2)	2.6624 (11)	172.1 (18)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.