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3-(5-*tert*-Butyl-2-hydroxybenzoyl)-propanoic acid

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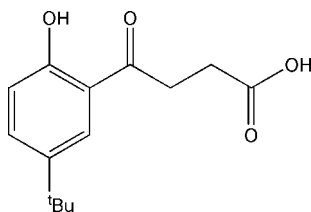
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.052; wR factor = 0.142; data-to-parameter ratio = 23.4.

The title compound, $\text{C}_{14}\text{H}_{18}\text{O}_4$, was synthesized as a part of an investigation into corrosion inhibitors, as it is structurally related to the commercial reagent Ircagor 419. The molecules form centrosymmetric dimers due to hydrogen bonding involving the carboxyl groups, typical for the crystal structures of carboxylic acids. There is also an intramolecular hydrogen bond between the phenol hydroxyl group and the *ortho* keto O atom. π - π interactions between benzene rings link the molecules into stacks running along the a axis (distances between the planes of adjacent molecules are 3.305 and 3.389 Å). Intermolecular hydrogen bonds, together with the stacking interactions, give rise to infinite sheets parallel to the ac plane of the crystal structure.

Related literature

Similar 4-keto acids based on 3-benzoylpropionic acid, which does not contain the phenolic hydroxyl group, have been characterized crystallographically. These include two polymorphs of 3-benzoylpropionic acid (Selladurai *et al.*, 1990; Thompson *et al.*, 1991), 4-(4-biphenyl)-4-oxobutyric acid (Kim *et al.*, 1988), 3-[4-(methylsulfonyl)benzoyl]propionic acid (Lynch & McClenaghan, 2002) and 3-(4-methylbenzoyl)propionic acid (Ircagor 419; Frey *et al.*, 2000), which is used as a corrosion inhibitor for mild steel. All structures contain the dimeric carboxylic acid hydrogen bonding seen in the title compound.



Experimental

Crystal data

$\text{C}_{14}\text{H}_{18}\text{O}_4$
 $M_r = 250.28$
 Triclinic, $P\bar{1}$
 $a = 7.0938$ (2) Å
 $b = 8.5918$ (3) Å
 $c = 11.4966$ (3) Å
 $\alpha = 76.917$ (2)°
 $\beta = 85.205$ (2)°
 $\gamma = 83.318$ (2)°
 $V = 676.69$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 150$ (2) K
 $0.34 \times 0.26 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.807$, $T_{\max} = 0.990$
 14490 measured reflections
 3998 independent reflections
 2956 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.142$
 $S = 1.03$
 3998 reflections
 171 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1-H1 \cdots O29	0.86 (2)	1.80 (2)	2.5777 (14)	150 (2)
O4-H4 \cdots O3 ⁱ	0.87 (2)	1.78 (2)	2.6417 (15)	175.8 (19)

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

Data collection: SMART (Siemens, 1993); cell refinement: SAINT; data reduction: SAINT (Siemens, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg, 2004); software used to prepare material for publication: PLATON (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2053).

References

- Brandenburg, K. (2004). DIAMOND. Release 3.0. Crystal Impact GbR, Bonn, Germany. <http://www.crystalimpact.com/diamond>.
- Frey, M., Harris, S. G., Holmes, J. M., Nation, D. A., Parsons, S., Tasker, P. A. & Winpenny, R. E. P. (2000). *Chem. Eur. J.* **6**, 1407–1415.
- Kim, Y. B., Park, I. Y. & Park, Y. H. (1988). *Arch. Pharmacol. Res.* **11**, 127–133.
- Lynch, D. E. & McClenaghan, I. (2002). *Acta Cryst.* **E58**, o729–o730.
- Selladurai, S., Kumar, M. S. & Subramanian, K. (1990). *Proc. Indian Acad. Sci. Chem. Sci.* **102**, 39–43.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2004). SADABS. Version 2004/1. University of Göttingen, Germany.
- Siemens (1993). SMART. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1995). SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Thompson, H. W., Vanderhoff, P. A. & Lalancette, R. A. (1991). *Acta Cryst.* **C47**, 1443–1445.

supplementary materials

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3-(5-*tert*-Butyl-2-hydroxybenzoyl)propanoic acid

R. S. Forgan, S. Parsons, P. A. Tasker and F. J. White

Comment

The title compound was synthesized as a potential corrosion inhibitor as it has a structure similar to that of successful reagent Ircagor 419TM (Frey *et al.*, 2000) and, in addition, includes a phenolic hydroxyl group which may coordinate to a metal or form hydrogen bonds to the surface. The molecules in the crystal structure form dimers *via* H-bonding between two adjacent carboxylate moieties (O4—H4 \cdots O3 = 2.642 (1) Å; Figure 1) which is typical for carboxylate structures. An intramolecular hydrogen bond is also formed between the phenolic hydroxyl group and the *ortho* keto oxygen (O1 \cdots O29 = 2.578 (1) Å).

Each molecule is involved in π - π stacking interactions with one molecule above and one below, in the direction of the *a* axis. The distances between the planes of the molecules are 3.305 Å and 3.389 Å. An isotactic arrangement of these π - π stacked chains occurs, with each chain linked to the next one through the carboxylate H-bonds described above, thus forming two-dimensional sheets parallel to the *ac* plane (Figure 2).

Experimental

4-*tert*-Butylanisole (3.001 g, 18.2 mmol) and succinic anhydride (1.832 g, 18.3 mmol) were dissolved in nitrobenzene and aluminium chloride (5.1 g, 38.2 mmol) was added in portions over ice. The mixture was heated to 60 °C for 4 h and then treated with aqueous HCl (100 ml, 1 *M*). Extraction into chloroform (100 ml) and removal of solvent yielded a solid which was dissolved in NaOH (1 *M*, 100 ml). The pH was adjusted to 2 with conc HCl and the precipitate collected and recrystallized from hexane/chloroform to yield the title compound (2.350 g, 9.4 mmol, 52%). Colourless rods suitable for XRD analysis were grown by slow evaporation of a hexane/chloroform solution.

Refinement

Hydroxyl H atoms were found in a Fourier difference map and their positional and isotropic displacement parameters refined [O1—H1 0.86 (2) Å; O4—H4 0.87 (2) Å]. The H atoms bound to carbon were placed in idealized positions and allowed to ride on their parent atoms with bond lengths of 0.95–0.99 Å and U_{iso} values set at 1.2 U_{eq} of the corresponding carrier atom (1.5 U_{eq} for methyl H atoms).

Figures

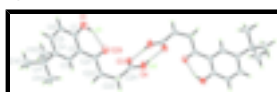


Fig. 1. The molecular structure of (I) showing atom labels and 50% probability displacement ellipsoids for non-H atoms. The H-atoms are shown as dashed lines. The unlabelled atoms are related to corresponding labelled atoms by an inversion centre ($-x, 1 - y, 1 - z$).

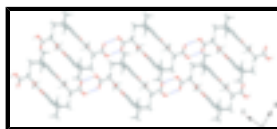


Fig. 2. Crystal packing of (I) as viewed along the *b* axis, showing the formation of two-dimensional sheets *via* combination of H-bonding and π - π stacking interactions. H-bonds are shown as dotted lines.

3-(5-*tert*-Butyl-2-hydroxybenzoyl)propanoic acid

Crystal data

$C_{14}H_{18}O_4$	$Z = 2$
$M_r = 250.28$	$F_{000} = 268$
Triclinic, $P\bar{1}$	$D_x = 1.228 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 7.0938 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.5918 (3) \text{ \AA}$	Cell parameters from 3804 reflections
$c = 11.4966 (3) \text{ \AA}$	$\theta = 30.5\text{--}2.4^\circ$
$\alpha = 76.917 (2)^\circ$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 85.205 (2)^\circ$	$T = 150 (2) \text{ K}$
$\gamma = 83.318 (2)^\circ$	Block, colourless
$V = 676.69 (4) \text{ \AA}^3$	$0.34 \times 0.26 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3998 independent reflections
Radiation source: fine-focus sealed tube	2956 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.034$
$T = 150(2) \text{ K}$	$\theta_{\text{max}} = 30.6^\circ$
ω and φ scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -10 \rightarrow 9$
$T_{\text{min}} = 0.807$, $T_{\text{max}} = 0.990$	$k = -12 \rightarrow 12$
14490 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.142$	$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.0859P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3998 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
171 parameters	$\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

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.

Refinement. Checkcif output:

912_ALERT_3_B # Missing FCF Reflections Above STH/L=0.6 159 910_ALERT_3_C # Missing FCF Reflections Below TH(Min) 1 911_ALERT_3_C # Missing FCF Refl Between THmin & STh/l= 0.600 1

The data collection strategy used aimed to achieve a complete data set to $2\theta = 53^\circ$. Some higher angle data were collected in the process and these have been included in the refinement. Please refer to the completeness statistics below

=====
Resolution & Completeness Statistics (Cumulative)

=====
Complete Expected Measured Missing ----- Theta sin(th)/Lambda
1417 1 23.01 0.550 0.999 1883 1882 1 25.24 0.600 0.999 2457 2455 2 ----- ACTA Min.
Res. --- 27.51 0.650 0.997 3116 3108 8 29.84 0.700 0.977 3877 3787 90 30.57 0.716 0.961 4159 3998 161

061_ALERT_3_C Tmax/Tmin Range Test RR' too Large 0.83

SADABS corrects for all systematic errors that lead to disparities in the intensities of symmetry-equivalent data. These may include absorption by the mount, crystal decay *etc.* The crystal was a rather irregular shape, having been cleaved from a larger block; this precluded integration methods for correcting for absorption.

094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ... 2.37

Highest peak = 0.43 Deepest Hole = -0.18 These are not unreasonable. They are localized in the region of an aromatic ring

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.25675 (15)	0.27572 (10)	0.04192 (9)	0.0344 (2)
C1	0.23777 (17)	0.42613 (13)	-0.02693 (10)	0.0256 (2)
C2	0.26835 (17)	0.56259 (13)	0.01488 (10)	0.0235 (2)
O3	0.04561 (13)	0.62039 (11)	0.37232 (8)	0.0318 (2)
C3	0.25301 (18)	0.71372 (13)	-0.06456 (10)	0.0249 (2)
H3A	0.2753	0.8056	-0.0367	0.030*
C4	0.20655 (18)	0.73325 (14)	-0.18182 (10)	0.0261 (3)
O4	0.25919 (15)	0.47532 (13)	0.49478 (8)	0.0392 (3)
H4	0.157 (3)	0.443 (2)	0.5352 (19)	0.059 (6)*
C5	0.16996 (19)	0.59523 (15)	-0.21840 (10)	0.0281 (3)
H5A	0.1331	0.6057	-0.2976	0.034*
C6	0.18548 (18)	0.44515 (14)	-0.14380 (11)	0.0280 (3)
H6A	0.1605	0.3543	-0.1720	0.034*
C21	0.31001 (17)	0.54528 (14)	0.14063 (10)	0.0260 (2)
C22	0.33448 (19)	0.69232 (15)	0.18742 (10)	0.0285 (3)

supplementary materials

H22A	0.2174	0.7676	0.1762	0.034*
H22B	0.4408	0.7476	0.1411	0.034*
C23	0.3750 (2)	0.64759 (17)	0.31958 (11)	0.0332 (3)
H23A	0.4881	0.5677	0.3312	0.040*
H23B	0.4046	0.7444	0.3449	0.040*
C24	0.21078 (19)	0.57950 (15)	0.39652 (10)	0.0280 (3)
O29	0.32127 (14)	0.41288 (11)	0.21016 (8)	0.0333 (2)
C41	0.1914 (2)	0.89572 (15)	-0.27057 (11)	0.0334 (3)
C42	-0.0136 (3)	0.93891 (19)	-0.30816 (15)	0.0495 (4)
H42A	-0.0987	0.9467	-0.2376	0.074*
H42B	-0.0502	0.8554	-0.3450	0.074*
H42C	-0.0229	1.0422	-0.3659	0.074*
C43	0.3241 (3)	0.8850 (2)	-0.38152 (14)	0.0554 (5)
H43A	0.3144	0.9889	-0.4386	0.083*
H43B	0.2872	0.8022	-0.4189	0.083*
H43C	0.4555	0.8571	-0.3578	0.083*
C44	0.2471 (3)	1.02985 (17)	-0.21712 (15)	0.0532 (5)
H44A	0.1630	1.0390	-0.1464	0.080*
H44B	0.2350	1.1316	-0.2766	0.080*
H44C	0.3790	1.0051	-0.1941	0.080*
H1	0.284 (3)	0.287 (3)	0.1107 (19)	0.065 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0409 (6)	0.0241 (4)	0.0364 (5)	-0.0035 (4)	-0.0020 (4)	-0.0029 (3)
C1	0.0223 (6)	0.0244 (5)	0.0290 (5)	-0.0017 (4)	0.0019 (5)	-0.0054 (4)
C2	0.0204 (6)	0.0262 (5)	0.0236 (5)	-0.0012 (4)	0.0004 (4)	-0.0058 (4)
O3	0.0267 (5)	0.0390 (5)	0.0274 (4)	-0.0007 (4)	-0.0024 (4)	-0.0035 (3)
C3	0.0260 (6)	0.0248 (5)	0.0245 (5)	-0.0020 (4)	0.0000 (4)	-0.0074 (4)
C4	0.0282 (7)	0.0260 (5)	0.0234 (5)	-0.0016 (4)	-0.0005 (5)	-0.0049 (4)
O4	0.0284 (6)	0.0598 (6)	0.0245 (4)	-0.0027 (5)	-0.0047 (4)	0.0009 (4)
C5	0.0285 (7)	0.0329 (6)	0.0247 (5)	-0.0023 (5)	-0.0024 (5)	-0.0099 (4)
C6	0.0279 (7)	0.0273 (5)	0.0314 (6)	-0.0033 (5)	-0.0002 (5)	-0.0125 (4)
C21	0.0205 (6)	0.0331 (6)	0.0233 (5)	-0.0007 (4)	0.0009 (4)	-0.0055 (4)
C22	0.0293 (7)	0.0352 (6)	0.0215 (5)	-0.0039 (5)	-0.0009 (5)	-0.0073 (4)
C23	0.0289 (7)	0.0478 (7)	0.0243 (5)	-0.0071 (5)	-0.0030 (5)	-0.0086 (5)
C24	0.0297 (7)	0.0353 (6)	0.0213 (5)	-0.0030 (5)	-0.0029 (5)	-0.0108 (4)
O29	0.0366 (6)	0.0332 (4)	0.0267 (4)	-0.0013 (4)	-0.0023 (4)	-0.0007 (3)
C41	0.0438 (8)	0.0282 (6)	0.0266 (5)	-0.0041 (5)	-0.0044 (5)	-0.0017 (4)
C42	0.0538 (11)	0.0377 (7)	0.0515 (9)	0.0022 (7)	-0.0158 (8)	0.0028 (6)
C43	0.0701 (13)	0.0504 (9)	0.0362 (7)	-0.0051 (8)	0.0119 (8)	0.0044 (6)
C44	0.0850 (14)	0.0297 (6)	0.0452 (8)	-0.0146 (7)	-0.0201 (9)	0.0016 (6)

Geometric parameters (\AA , $^\circ$)

O1—C1	1.3524 (14)	C22—C23	1.5238 (16)
O1—H1	0.86 (2)	C22—H22A	0.99
C1—C6	1.3925 (17)	C22—H22B	0.99

C1—C2	1.4087 (16)	C23—C24	1.4945 (19)
C2—C3	1.4067 (15)	C23—H23A	0.99
C2—C21	1.4709 (16)	C23—H23B	0.99
O3—C24	1.2199 (15)	C41—C44	1.526 (2)
C3—C4	1.3832 (16)	C41—C42	1.532 (2)
C3—H3A	0.95	C41—C43	1.534 (2)
C4—C5	1.4022 (17)	C42—H42A	0.98
C4—C41	1.5296 (16)	C42—H42B	0.98
O4—C24	1.3156 (14)	C42—H42C	0.98
O4—H4	0.87 (2)	C43—H43A	0.98
C5—C6	1.3757 (17)	C43—H43B	0.98
C5—H5A	0.95	C43—H43C	0.98
C6—H6A	0.95	C44—H44A	0.98
C21—O29	1.2318 (14)	C44—H44B	0.98
C21—C22	1.5131 (17)	C44—H44C	0.98
C1—O1—H1	105.5 (14)	C22—C23—H23A	109.2
O1—C1—C6	117.98 (10)	C24—C23—H23B	109.2
O1—C1—C2	122.74 (11)	C22—C23—H23B	109.2
C6—C1—C2	119.28 (10)	H23A—C23—H23B	107.9
C3—C2—C1	118.83 (10)	O3—C24—O4	122.72 (12)
C3—C2—C21	121.44 (10)	O3—C24—C23	123.00 (11)
C1—C2—C21	119.71 (10)	O4—C24—C23	114.24 (11)
C4—C3—C2	122.29 (10)	C44—C41—C4	112.10 (11)
C4—C3—H3A	118.9	C44—C41—C42	108.46 (13)
C2—C3—H3A	118.9	C4—C41—C42	109.44 (12)
C3—C4—C5	117.00 (10)	C44—C41—C43	108.31 (14)
C3—C4—C41	123.41 (10)	C4—C41—C43	109.30 (11)
C5—C4—C41	119.59 (10)	C42—C41—C43	109.19 (13)
C24—O4—H4	108.8 (13)	C41—C42—H42A	109.5
C6—C5—C4	122.40 (11)	C41—C42—H42B	109.5
C6—C5—H5A	118.8	H42A—C42—H42B	109.5
C4—C5—H5A	118.8	C41—C42—H42C	109.5
C5—C6—C1	120.12 (10)	H42A—C42—H42C	109.5
C5—C6—H6A	119.9	H42B—C42—H42C	109.5
C1—C6—H6A	119.9	C41—C43—H43A	109.5
O29—C21—C2	121.18 (11)	C41—C43—H43B	109.5
O29—C21—C22	118.85 (10)	H43A—C43—H43B	109.5
C2—C21—C22	119.95 (10)	C41—C43—H43C	109.5
C21—C22—C23	111.33 (10)	H43A—C43—H43C	109.5
C21—C22—H22A	109.4	H43B—C43—H43C	109.5
C23—C22—H22A	109.4	C41—C44—H44A	109.5
C21—C22—H22B	109.4	C41—C44—H44B	109.5
C23—C22—H22B	109.4	H44A—C44—H44B	109.5
H22A—C22—H22B	108.0	C41—C44—H44C	109.5
C24—C23—C22	112.24 (11)	H44A—C44—H44C	109.5
C24—C23—H23A	109.2	H44B—C44—H44C	109.5

supplementary materials

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···O29	0.86 (2)	1.80 (2)	2.5777 (14)	150 (2)
O4—H4···O3 ⁱ	0.87 (2)	1.78 (2)	2.6417 (15)	175.8 (19)

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

Fig. 1

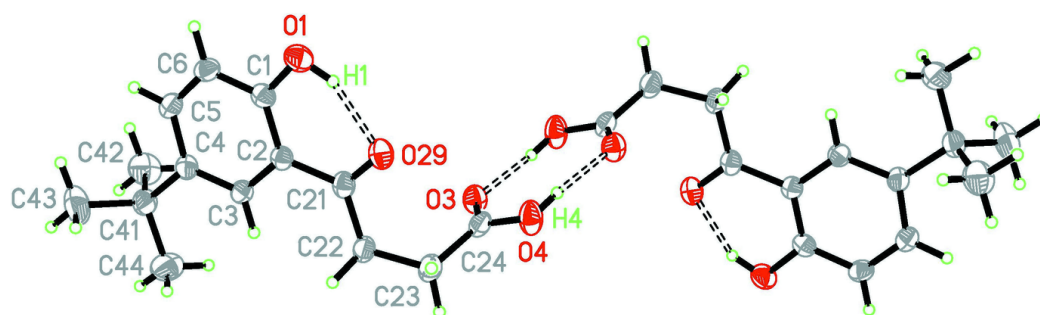


Fig. 2

