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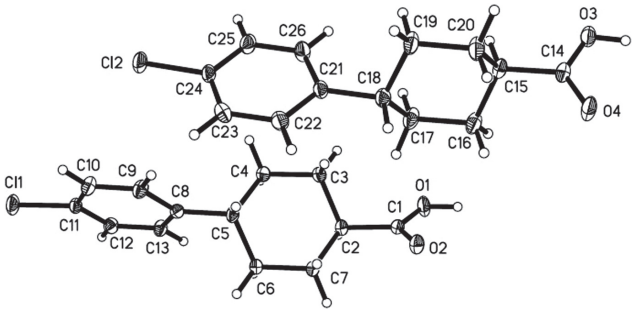
The crystal structure of 4-(4-chlorophenyl) cyclohexane-1-carboxylic acid, $C_{13}H_{15}ClO_2$



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Abstract

$C_{13}H_{15}ClO_2$, monoclinic, $P2_1/n$ (no. 14), $a = 14.4696(7)$ Å, $b = 9.5385(4)$ Å, $c = 18.7042(11)$ Å, $\beta = 112.619(2)°$, $V = 2383.0(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0414$, $wR_{ref}(F^2) = 0.1090$, $T = 150(2)$ K.

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The asymmetric unit of the title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All of starting materials are used without further purification. Under stirring, 4-(4-chlorophenyl)cyclohexanecarboxylic acid (2.38 g, 10 mmol) was added to 25 mL THF in a warm water bath for 30 min. After cooling, the solution was filtered

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.15 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.30 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	32366, 4858, 0.053
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3550
$N(param)_{refined}$:	297
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.46667(13)	0.32592(19)	0.60064(11)	0.0271(4)
C2	0.36168(13)	0.3092(2)	0.54110(11)	0.0270(4)
H2	0.360699	0.226519	0.507961	0.032*
C3	0.33404(15)	0.4400(2)	0.48926(12)	0.0369(5)
H3A	0.339503	0.524152	0.521612	0.044*
H3B	0.381685	0.450708	0.463263	0.044*
C4	0.22744(14)	0.4291(2)	0.42815(11)	0.0356(5)
H4A	0.224037	0.351055	0.392308	0.043*
H4B	0.210311	0.516826	0.397581	0.043*
C5	0.15106(13)	0.40370(19)	0.46515(11)	0.0269(4)
H5	0.155691	0.484692	0.500313	0.032*
C6	0.17985(13)	0.2720(2)	0.51536(11)	0.0290(4)
H6A	0.176114	0.189572	0.482185	0.035*
H6B	0.131634	0.257527	0.540530	0.035*
C7	0.28581(13)	0.2837(2)	0.57758(11)	0.0313(4)
H7A	0.288406	0.361829	0.613119	0.038*
H7B	0.302924	0.196038	0.608278	0.038*
C8	0.04537(13)	0.40065(19)	0.40497(10)	0.0251(4)
C9	−0.01749(14)	0.5152(2)	0.39584(11)	0.0321(5)
H9	0.007062	0.595282	0.427758	0.038*
C10	−0.11520(14)	0.5160(2)	0.34136(11)	0.0339(5)
H10	−0.156693	0.595827	0.335660	0.041*
C11	−0.15101(13)	0.3993(2)	0.29577(10)	0.0274(4)
C12	−0.09056(14)	0.2846(2)	0.30130(11)	0.0294(4)
H12	−0.115340	0.205688	0.268465	0.035*
C13	0.00756(14)	0.2865(2)	0.35598(11)	0.0290(4)
H13	0.049641	0.207979	0.359890	0.035*
C14	0.76258(14)	0.8498(2)	0.74946(11)	0.0285(4)
C15	0.65660(14)	0.8546(2)	0.69085(11)	0.0298(4)
H15	0.658294	0.869561	0.638450	0.036*
C16	0.60240(15)	0.7184(2)	0.68970(13)	0.0417(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H16A	0.601022	0.701209	0.741480	0.050*
H16B	0.638919	0.639940	0.677719	0.050*
C17	0.49465(16)	0.7233(2)	0.62884(13)	0.0430(6)
H17A	0.496615	0.728924	0.576592	0.052*
H17B	0.459852	0.635356	0.631621	0.052*
C18	0.43559(14)	0.8471(2)	0.64015(11)	0.0313(4)
H18	0.426949	0.832554	0.690164	0.038*
C19	0.49281(14)	0.9834(2)	0.64682(13)	0.0400(5)
H19A	0.456383	1.060209	0.660317	0.048*
H19B	0.495456	1.005874	0.595965	0.048*
C20	0.59961(15)	0.9761(2)	0.70797(14)	0.0414(6)
H20A	0.634831	1.065223	0.708272	0.050*
H20B	0.597458	0.963259	0.759827	0.050*
C21	0.33156(14)	0.85609(19)	0.57613(11)	0.0277(4)
C22	0.24625(15)	0.8430(2)	0.59257(12)	0.0370(5)
H22	0.253102	0.826309	0.644442	0.044*
C23	0.15104(15)	0.8537(2)	0.53482(13)	0.0405(5)
H23	0.093473	0.844932	0.547178	0.049*
C24	0.14089(14)	0.8771(2)	0.45974(12)	0.0311(4)
C25	0.22375(15)	0.8890(2)	0.44093(12)	0.0351(5)
H25	0.216150	0.903759	0.388739	0.042*
C26	0.31826(15)	0.8790(2)	0.49909(12)	0.0347(5)
H26	0.375424	0.887880	0.486225	0.042*
Cl1	−0.27574(3)	0.39643(6)	0.23058(3)	0.04083(16)
Cl2	0.02171(4)	0.89427(6)	0.38669(4)	0.04856(18)
H1	0.599(2)	0.314(3)	0.6135(18)	0.086(10)*
H3	0.892(2)	0.875(3)	0.7604(18)	0.081(10)*
O1	0.53649(10)	0.30102(18)	0.57425(8)	0.0404(4)
O2	0.48521(10)	0.36194(14)	0.66807(8)	0.0339(3)
O3	0.83116(11)	0.87893(16)	0.72272(8)	0.0383(4)
O4	0.78200(10)	0.82055(18)	0.81785(8)	0.0427(4)

and evaporated in air at room temperature. Colorless crystals were harvested after one week, yield: 82.6%.

Experimental details

The structure was solved by direct methods with the SHELXS program. All of H-atoms from C atoms were positioned with idealized geometry with $U_{iso}(H) = 1.2U_{eq}(C)$. The two hydrogen atoms from O atoms were positioned with Q peaks and refined freely.

Comment

It's known that the methyl or ethyl esters of 4-arylcyclohexanecarboxylic acid and its derivatives are one of the most common kinds of the products of Diels-Alder reactions [5–8]. These compounds mentioned above could although come from other organic reactions [9, 10]. Especially, these asymmetrically substituted products, are usually synthesized by the Michael addition [11–15]. However, the corresponding acids are rare [16].

As shown in the figure, X-ray diffraction indicates that the asymmetric unit contains two molecules of the title compound. All atoms of the chlorophenyl moiety are nearly coplanar, the cyclohexyl group adapts the chair conformation. A dimer is generated by the O—H···O hydrogen bond with a head to head model. All of the bond lengths and angles of the title compound are comparable with its analogues.

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