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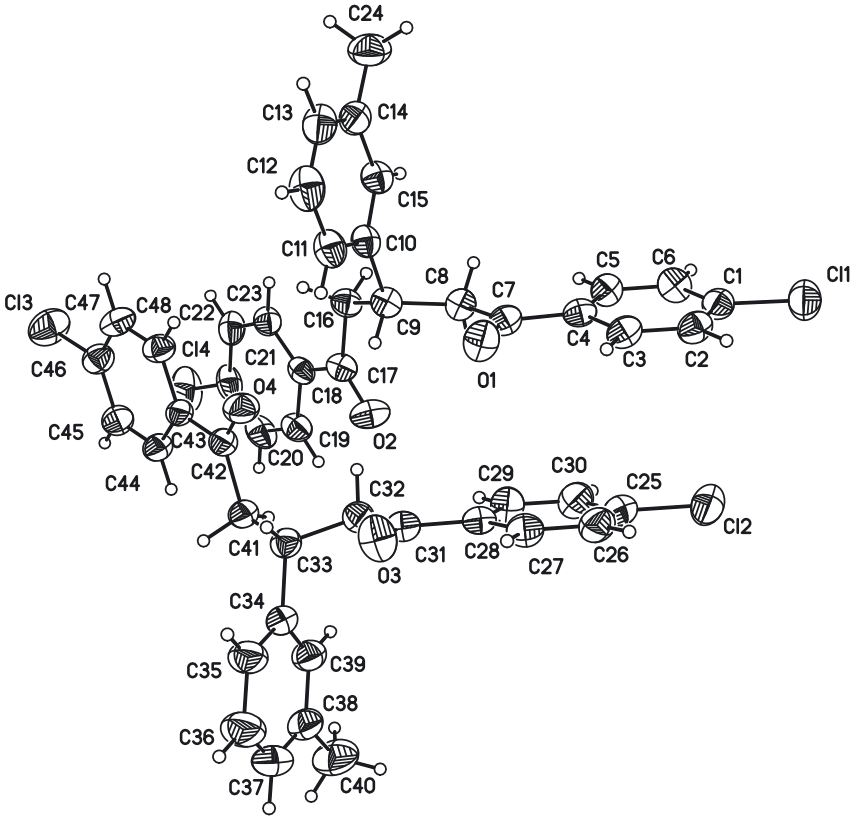
The crystal structure of 1,5-bis(4-chlorophenyl)-3-(3-methylphenyl)pentane- 1,5-dione, $C_{48}H_{40}Cl_4O_4$



https://doi.org/10.1515/ncrs-2022-0335
Received June 28, 2022; accepted July 20, 2022;
published online August 24, 2022

Abstract

$C_{48}H_{40}Cl_4O_4$, triclinic, $P\bar{1}$ (no. 2), a = 7.1407(5) Å, b = 12.5905(8) Å, c = 24.6662(17) Å, α = 103.442(6)°, β = 93.551(5)°, γ = 101.219(5)°, V = 2102.3(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0595, $wR_{ref}(F^2)$ = 0.1641, T = 293 K.

CCDC no.: 2191527

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Table 1: Data collection and handling.

Crystal:	White block
Size:	0.12 × 0.12 × 0.11 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	2.90 mm^{-1}
Diffractometer, scan mode:	Xcalibur
θ_{max}, completeness:	67.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13195, 7526, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4091
$N(param)_{refined}$:	507
Programs:	CrysAlis^{PRO} [1], SHELX [2]

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

In a 10 mL round-bottom flask, 3-methylbenzaldehyde (0.25 mmol) was reacted with 4-chloroacetophenone (0.5 mmol) in the presence of NaOH (0.5 mmol), the

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.37169 (17)	0.23627 (10)	0.34813 (5)	0.0996 (4)
Cl2	0.16802 (19)	0.47885 (13)	0.45542 (5)	0.1200 (5)
Cl3	0.6981 (2)	1.13654 (12)	0.03486 (5)	0.1165 (5)
Cl4	0.2511 (2)	1.25145 (10)	0.13280 (7)	0.1307 (6)
O1	0.3895 (4)	0.4913 (3)	0.24819 (12)	0.0866 (9)
O2	0.3625 (4)	0.8374 (2)	0.24890 (11)	0.0812 (8)
O3	0.9398 (4)	0.6977 (3)	0.34703 (13)	0.0959 (9)
O4	0.8306 (4)	0.7987 (2)	0.19795 (10)	0.0810 (8)
C1	−0.2012 (6)	0.3104 (3)	0.31555 (16)	0.0703 (10)
C2	−0.0128 (6)	0.3042 (3)	0.32383 (15)	0.0718 (10)
H2	0.023986	0.259357	0.345816	0.086*
C3	0.1217 (5)	0.3658 (3)	0.29894 (15)	0.0694 (10)
H3	0.250186	0.362490	0.304702	0.083*
C4	0.0704 (5)	0.4325 (3)	0.26560 (14)	0.0595 (9)
C5	−0.1206 (5)	0.4370 (3)	0.25815 (15)	0.0676 (10)
H5	−0.158076	0.482008	0.236327	0.081*
C6	−0.2573 (6)	0.3755 (3)	0.28276 (17)	0.0761 (11)
H6	−0.386079	0.378298	0.277094	0.091*
C7	0.2237 (5)	0.4990 (3)	0.24094 (15)	0.0650 (9)
C8	0.1707 (5)	0.5776 (3)	0.20816 (15)	0.0638 (9)
H8A	0.133918	0.639272	0.233705	0.077*
H8B	0.059722	0.538484	0.181313	0.077*
C9	0.3318 (5)	0.6250 (3)	0.17666 (15)	0.0603 (9)
H9	0.449255	0.652443	0.202993	0.072*
C10	0.3722 (5)	0.5382 (3)	0.12705 (15)	0.0599 (9)
C11	0.5550 (5)	0.5177 (3)	0.12352 (18)	0.0767 (11)
H11	0.653139	0.554201	0.152219	0.092*
C12	0.5905 (7)	0.4416 (4)	0.0763 (2)	0.0935 (14)
H12	0.712716	0.426784	0.073921	0.112*
C13	0.4488 (7)	0.3889 (4)	0.0339 (2)	0.0881 (13)
H13	0.476326	0.339130	0.002688	0.106*
C14	0.2666 (6)	0.4074 (3)	0.03591 (17)	0.0739 (11)
C15	0.2325 (5)	0.4825 (3)	0.08336 (16)	0.0669 (10)
H15	0.109244	0.495755	0.085673	0.080*
C16	0.2874 (5)	0.7243 (3)	0.15571 (15)	0.0624 (9)
H16A	0.367540	0.735735	0.126341	0.075*
H16B	0.154606	0.704992	0.139155	0.075*
C17	0.3184 (5)	0.8327 (3)	0.20021 (16)	0.0595 (9)
C18	0.2946 (4)	0.9346 (3)	0.18164 (15)	0.0565 (8)
C19	0.3247 (5)	1.0360 (3)	0.22233 (17)	0.0695 (10)
H19	0.355503	1.038116	0.259813	0.083*
C20	0.3090 (6)	1.1325 (3)	0.2072 (2)	0.0834 (13)
H20	0.326372	1.199566	0.234416	0.100*
C21	0.2677 (6)	1.1295 (3)	0.1520 (2)	0.0798 (12)
C22	0.2377 (5)	1.0322 (3)	0.11116 (18)	0.0762 (11)
H22	0.207362	1.031482	0.073833	0.091*
C23	0.2530 (5)	0.9343 (3)	0.12608 (16)	0.0635 (9)
H23	0.235031	0.867786	0.098437	0.076*
C24	0.1077 (7)	0.3477 (4)	−0.01057 (19)	0.1072 (16)
H24A	0.155154	0.347369	−0.046179	0.161*
H24B	0.003083	0.385677	−0.007116	0.161*
H24C	0.063788	0.272186	−0.008021	0.161*
C25	0.3466 (6)	0.5494 (4)	0.42423 (17)	0.0813 (12)
C26	0.5219 (6)	0.5220 (4)	0.42325 (17)	0.0838 (12)
H26	0.546157	0.465101	0.439022	0.101*
C27	0.6630 (6)	0.5793 (3)	0.39871 (16)	0.0741 (11)
H27	0.782951	0.560930	0.398242	0.089*
C28	0.6288 (5)	0.6643 (3)	0.37463 (14)	0.0609 (9)
C29	0.4474 (5)	0.6881 (3)	0.37540 (17)	0.0764 (11)
H29	0.419823	0.743320	0.358825	0.092*
C30	0.3078 (6)	0.6313 (4)	0.40027 (17)	0.0859 (13)
H30	0.187015	0.648631	0.400806	0.103*
C31	0.7850 (5)	0.7244 (3)	0.34860 (15)	0.0651 (9)
C32	0.7482 (5)	0.8167 (3)	0.32373 (15)	0.0653 (9)
H32A	0.677068	0.861371	0.348652	0.078*
H32B	0.667111	0.784017	0.288430	0.078*
C33	0.9267 (5)	0.8937 (3)	0.31313 (14)	0.0623 (9)
H33	1.011162	0.846981	0.294966	0.075*
C34	1.0378 (5)	0.9710 (3)	0.36721 (15)	0.0671 (10)
C35	1.2293 (6)	0.9742 (4)	0.37943 (18)	0.0932 (14)
H35	1.291089	0.928070	0.355033	0.112*
C36	1.3307 (7)	1.0465 (5)	0.4283 (2)	0.121 (2)
H36	1.460442	1.048640	0.436429	0.145*
C37	1.2413 (8)	1.1141 (5)	0.4642 (2)	0.1145 (18)
H37	1.311170	1.161712	0.496772	0.137*
C38	1.0507 (8)	1.1136 (4)	0.45343 (18)	0.0896 (13)
C39	0.9524 (6)	1.0414 (4)	0.40456 (16)	0.0787 (11)
H39	0.822949	1.040058	0.396479	0.094*
C40	0.9502 (8)	1.1877 (5)	0.4934 (2)	0.134 (2)
H40A	0.898836	1.149108	0.520286	0.201*
H40B	0.847510	1.205814	0.472736	0.201*
H40C	1.040168	1.255175	0.512500	0.201*
C41	0.8739 (5)	0.9625 (3)	0.27333 (14)	0.0645 (9)
H41A	0.762385	0.991377	0.284964	0.077*
H41B	0.979340	1.026002	0.276525	0.077*
C42	0.8305 (5)	0.8976 (3)	0.21258 (15)	0.0608 (9)
C43	0.7904 (4)	0.9587 (3)	0.16953 (13)	0.0563 (8)
C44	0.7918 (5)	1.0727 (3)	0.18295 (15)	0.0671 (10)
H44	0.812761	1.112598	0.220370	0.080*
C45	0.7626 (5)	1.1275 (3)	0.14182 (16)	0.0728 (10)
H45	0.763270	1.203577	0.151172	0.087*
C46	0.7324 (5)	1.0674 (4)	0.08683 (16)	0.0726 (10)
C47	0.7281 (6)	0.9549 (4)	0.07216 (16)	0.0785 (11)
H47	0.706220	0.915416	0.034663	0.094*
C48	0.7565 (5)	0.9012 (3)	0.11364 (15)	0.0668 (10)
H48	0.752887	0.824810	0.103889	0.080*

mixutre was ground together for 40 min, and the reaction was monitored by TLC. After the reaction was finished, the mixture was washed three times with hot water and recystallized from ethanol, the colourless crystals were obtained, yield: 62%.

Experimental details

All hydrogen atomic positions were taken from a difference Fourier map. Their U_{iso} values were set to 1.2U_{eq} (C, phenyl

ring and methylene), 1.5U_{eq}(C, methyl) of the parent atoms, respectively. All the H atoms were refined as riding on their parent atoms.

Comment

Up to now, the efficient, economical and environmentally friendly approaches for the preparation of high value-added 1,5-diketones derivatives from low-cost organic raw materials have attracted wide attention because these 1,5-diketones are very important structural units in many natural products and other biologically active molecules [3–6]. Thus, the synthetic strategy for 1,5-diketones and several 1,5-diones have been developed [7, 8]. For instance, Nagaraj *et al.* have synthesized four 3-(4-aryl)-2,4-bis(cyclohexylsulfanyl)-1,5-diphenylpentane-1,5-diones under solvent-free conditions [9]. Nevertheless, many efforts about the synthesis of 1,5-diketones have been made, to date, the crystal structure of 3-(3-methylphenyl)-1,5-di-/p-chloropentane-1,5-dione has not been reported. In this paper, we report the synthesis of the title compound using 3-methylbenzaldehyde and 4-chloroacetophenone under solvent-free condition and its crystal structure is described.

In the crystal structure, the asymmetric unit contains two similar 3-(3-methylphenyl)-1,5-di-/p-chloropentane-1,5-dione structure motifs, which cause the slightly different bond lengths and bond angles. Thus, only the structure of the upper (C1–C24) unit is described in detail here. Thus, the bond lengths and angles are normal and correspond to those observed in 3-(2-chlorophenyl)-1,5-bis(4-chlorophenyl) pentane-1,5-dione [10], 1,5-bis(4-chlorophenyl)-3-(2,5-dimethoxyphenyl)pentane-1,5-dione [11], 1,5-bis(4-chlorophenyl)-3-(4-methylphenyl)pentane-1,5-dione [12]. The pentane-1,5-dione unit (C7/C17/O1/O2) is puckered with the torsion angles C7–C8–C9–C16 = –166.60(3)° and C8–C9–C16–C17 = 76.91(3)°, making the two ketone groups pointing towards different directions. The dihedral angles between the central benzene ring and the benzene rings C1–C6 and C18–C23 are 81.26(1)° and 69.24(1)°, respectively. The crystal packing exhibits weak intermolecular π···π interactions and C—H···O hydrogen bonds.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: This work was supported by the Research on Technology of Liaocheng University (K22LD10).
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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