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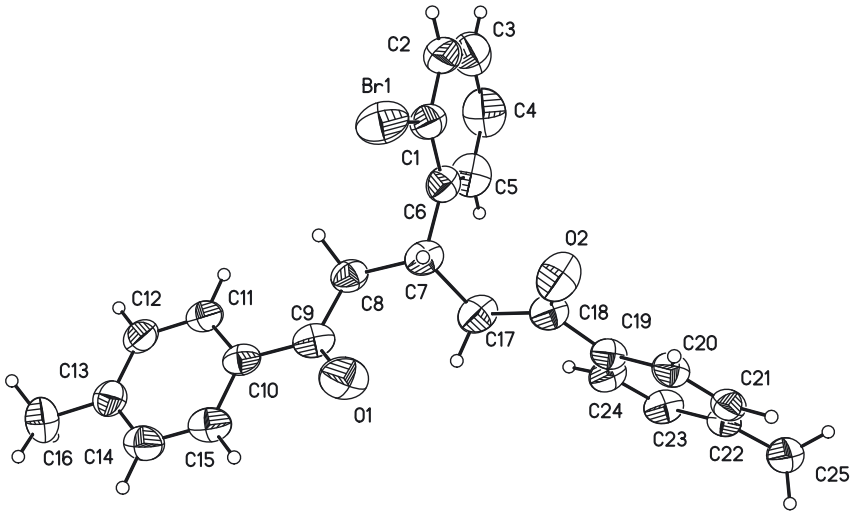
The crystal structure of 3-(2-bromophenyl)-1,5-di-*p*-tolylpentane-1,5-dione, $C_{25}H_{23}BrO_2$



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Abstract

$C_{25}H_{23}BrO_2$, monoclinic, $P2_1/c$ (no. 14), a = 9.6229(7) Å, b = 21.7953(16) Å, c = 10.9104(6) Å, β = 109.330(7)°, V = 2159.3(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0499, $wR_{ref}(F^2)$ = 0.1385, T = 293 K.

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The molecular 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.

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.12 × 0.12 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	2.72 mm^{-1}
Diffractometer, scan mode:	Xcalibur
θ_{max}, completeness:	67.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7926, 3856, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2419
$N(param)_{refined}$:	255
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3]

Source of material

In a 25 mL round-bottom flask, 2-bromobenzaldehyde (0.25 mmol), 4-methylacetophenone (0.5 mmol) and NaOH (0.5 mmol) were ground together for 40 min. After the reaction was completed, the mixture was washed three times with hot water and recystallized from ethanol, the colourless crystals were obtained, yield: 68%.

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 atom.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.31890 (6)	0.61708 (3)	0.51571 (5)	0.1179 (3)
O1	0.7750 (3)	0.61008 (16)	0.4402 (3)	0.1142 (11)
O2	0.5713 (3)	0.77499 (14)	0.5150 (2)	0.1077 (10)
C1	0.2475 (4)	0.66468 (18)	0.3636 (4)	0.0796 (10)
C2	0.0970 (5)	0.6748 (2)	0.3157 (5)	0.1022 (14)
H2	0.036177	0.659220	0.358997	0.123*
C3	0.0391 (6)	0.7079 (3)	0.2041 (6)	0.1221 (18)
H3	−0.061785	0.715180	0.171327	0.147*
C4	0.1279 (8)	0.7303 (3)	0.1410 (5)	0.1294 (19)
H4	0.087854	0.752790	0.064983	0.155*
C5	0.2769 (6)	0.7199 (2)	0.1887 (4)	0.1069 (14)
H5	0.335921	0.734977	0.143149	0.128*
C6	0.3426 (4)	0.68737 (17)	0.3036 (3)	0.0737 (9)
C7	0.5061 (4)	0.67364 (18)	0.3517 (3)	0.0753 (10)
H7	0.535334	0.665952	0.445288	0.090*
C8	0.5342 (4)	0.61458 (17)	0.2872 (4)	0.0745 (10)
H8A	0.460899	0.584494	0.289677	0.089*
H8B	0.519606	0.623305	0.196613	0.089*
C9	0.6837 (4)	0.5865 (2)	0.3465 (3)	0.0781 (10)
C10	0.7179 (4)	0.52850 (18)	0.2910 (3)	0.0683 (9)
C11	0.6123 (4)	0.49516 (19)	0.1947 (3)	0.0759 (10)
H11	0.515749	0.509290	0.162501	0.091*
C12	0.6508 (4)	0.44140 (19)	0.1475 (4)	0.0832 (11)
H12	0.579295	0.419614	0.083783	0.100*
C13	0.7920 (5)	0.41940 (19)	0.1923 (4)	0.0826 (11)
C14	0.8958 (4)	0.4521 (2)	0.2880 (4)	0.0886 (12)
H14	0.991881	0.437481	0.320422	0.106*
C15	0.8604 (4)	0.5056 (2)	0.3362 (4)	0.0832 (11)
H15	0.932713	0.526928	0.400111	0.100*
C16	0.8334 (6)	0.3616 (2)	0.1368 (6)	0.1235 (17)
H16A	0.755972	0.331974	0.122578	0.185*
H16B	0.922857	0.345313	0.196675	0.185*
H16C	0.847915	0.370848	0.055829	0.185*
C17	0.6001 (4)	0.72661 (19)	0.3314 (3)	0.0867 (12)
H17A	0.698556	0.711459	0.343596	0.104*
H17B	0.558926	0.740797	0.242364	0.104*
C18	0.6108 (4)	0.77991 (19)	0.4205 (3)	0.0736 (10)
C19	0.6741 (3)	0.83842 (17)	0.3948 (3)	0.0653 (9)
C20	0.7215 (4)	0.88109 (18)	0.4930 (3)	0.0729 (10)
H20	0.717680	0.871504	0.574918	0.088*
C21	0.7741 (4)	0.9372 (2)	0.4732 (4)	0.0778 (10)
H21	0.807197	0.964635	0.541989	0.093*
C22	0.7787 (4)	0.95366 (18)	0.3520 (4)	0.0752 (10)
C23	0.7333 (4)	0.9111 (2)	0.2538 (4)	0.0825 (11)
H23	0.736603	0.921037	0.171908	0.099*
C24	0.6832 (4)	0.8540 (2)	0.2739 (3)	0.0768 (10)
H24	0.655238	0.825764	0.206287	0.092*
C25	0.8297 (5)	1.0167 (2)	0.3283 (5)	0.1040 (14)
H25A	0.934830	1.019291	0.366574	0.156*
H25B	0.784635	1.047111	0.366669	0.156*
H25C	0.801979	1.023914	0.236493	0.156*

Comment

In the past few decades, the efficient, mild, economical and environmentally friendly approaches have been developed for the generation of value-added 1,5-dione molecules from inexpensive, readily accessible organic feedstocks [4]. Generally, the 1,5-dione molecules were achieved by the Aldol and Michael addition cascade reactions using the inorganic and organic base as catalysts and the preparation of 1,5-dione derivatives has received considerable attention because they could facilitate the efficient construction of complex bioactive molecules in an atom/step economic manner [5]. In this regard, several 1,5-diones have been synthesized and their crystal structure have been described in detail [6–9]. Regardless of these above results, to date, the crystal structure of 3-(2-bromophenyl)-1,5-di-/p-tolylpentane-1,5-dione has not been presented. In this paper, the title compound was achieved and its crystal structure is reported.

In the title compound, the bond lengths and angles are normal and correspond to those observed in 1,3,5-tri-*p*-tolylpentane-1,5-dione and 1,5-bis(4-methylphenyl)-3-phenylpentane-1,5-dione [10, 11]. The title molecule is non-planar, the benzene ring (C1–C6) and the two *p*-methylphenyl rings (C10–C15 and C19–C24) on both sides form the dihedral angles of 88.248(1)° and 76.726(1)°, respectively, which suggests that the *p*-methylphenyl rings are almost perpendicular to the central benzene ring, and the results are similar with those of the reported 3-(4-chlorophenyl)-1,5-di-/p-tolylpentane-1,5-dione [12]. The dihedral angle between the plane (C17–C19) and the plane (C8–C10) is 85.739(3)°, is larger than that of the reported 3-(3-bromo-4-methoxyphenyl)-1,5-diphenylpentane-1,5-dione [13] and the result indicates two planes are also perpendicular to each other, which may be caused by the a larger steric hindrance of bromo substituent at the 2-position of the central phenyl moiety. Furthermore, the methylene carbon atoms interact with carbonyl oxygen atoms of the adjacent molecule through weak C—H···O intermolecular interactions, forming 1D chains.

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