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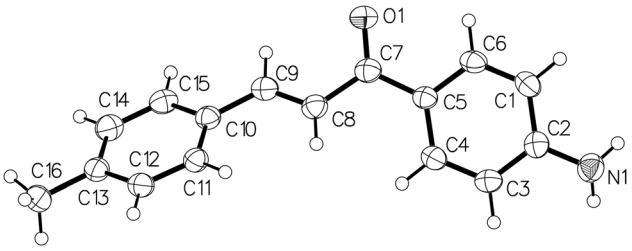
The crystal structure of (*E*)-1-(4-aminophenyl)-3-(*p*-tolyl)prop-2-en-1-one, $C_{16}H_{15}NO$



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Abstract

$C_{16}H_{15}NO$, monoclinic, $P2_1/c$ (no. 14), a = 5.9233(3) Å, b = 14.5788(9) Å, c = 14.7095(9) Å, β = 99.073(2)°, V = 1254.34(13) Å^3, Z = 4, $R_{gt}(F)$ = 0.0600, $wR_{ref}(F^2)$ = 0.1512, T = 170 K.

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The molecular structure is shown in 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:	Colourless block
Size:	0.15 × 0.08 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9288, 2525, 0.062
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1561
$N(\text{param})_{refined}$:	165
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

The substrate 4-methylbenzaldehyde (12 mmol) was initially added to 10 mL ethanol in a 100 mL three-mouth flask. Then 1-(4-aminophenyl)ethan-1-one (10 mmol) was added to the solution and stirred at room temperature until the reactants were mixed evenly. Subsequently, 10 mL potassium hydroxide solution (20%) was slowly added to the reaction mixture and continued stirring for 30 min with solid precipitates. After the disappearance of raw materials monitored by thin-layer chromatography (TLC), the reactants were poured into 50 mL water. The solids were filtered by suction and washed successively with 50 mL water and 50 mL 30% ethanol. The crystals of the title compound were obtained after further recrystallization.

Experimental details

All hydrogen atoms were included in the refinement in the riding model approximation. The U_{iso} values of the hydrogen atoms of phenolic hydroxyl groups were set to $1.5U_{eq}$(C), and the U_{iso} values of all other hydrogen atoms were arranged to $1.2U_{eq}$(C).

Comment

Chalcones are aromatic ketones whose configurations are α, β-unsaturated ketones substituted with diaryl groups [5–7]. They are widely distributed in nature and have various biological activities [8–11]. Because of their multiple reaction centers, they can bind to a variety of receptors. They have many pharmacological properties, [12–14]. Furthermore chalcones serve as an essential intermediate in organic synthesis and novel drug discovery in medicinal chemistry. The research and development of chalcones have become a hot research field of pharmaceutical chemistry [15–17], and many relative structures were reported [18–21]. chalcones can be extracted from natural products and synthesized by chemical and biological methods [5, 15, 17].

The asymmetric unit of the title structure consists of one (*E*)-1-(4-aminophenyl)-3-(*p*-tolyl)prop-2-en-1-one molecule.

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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}
C1	0.0665 (4)	0.65406 (17)	0.73260 (18)	0.0368 (6)
H1	−0.025558	0.686228	0.769335	0.044*
C2	0.2691 (4)	0.69388 (17)	0.71327 (17)	0.0339 (6)
C3	0.4017 (4)	0.64440 (17)	0.65972 (18)	0.0348 (6)
H3	0.538568	0.670646	0.645369	0.042*
C4	0.3368 (4)	0.55821 (17)	0.62747 (17)	0.0342 (6)
H4	0.431085	0.525377	0.592139	0.041*
C5	0.1338 (4)	0.51812 (16)	0.64597 (16)	0.0298 (6)
C6	0.0006 (4)	0.56888 (17)	0.69874 (18)	0.0344 (6)
H6	−0.138946	0.543599	0.711388	0.041*
C7	0.0561 (4)	0.42645 (17)	0.61382 (18)	0.0337 (6)
C8	0.2148 (4)	0.36410 (16)	0.57528 (18)	0.0350 (6)
H8	0.374422	0.375960	0.587377	0.042*
C9	0.1389 (4)	0.29222 (17)	0.52436 (18)	0.0353 (6)
H9	−0.022393	0.285782	0.509914	0.042*
C10	0.2762 (4)	0.22144 (16)	0.48778 (18)	0.0330 (6)
C11	0.5047 (4)	0.20595 (17)	0.52362 (18)	0.0344 (6)
H11	0.578029	0.243631	0.572153	0.041*
C12	0.6266 (4)	0.13633 (17)	0.48949 (18)	0.0374 (6)
H12	0.781757	0.126501	0.515750	0.045*
C13	0.5266 (4)	0.08044 (17)	0.41751 (18)	0.0362 (6)
C14	0.3003 (5)	0.09620 (18)	0.38163 (19)	0.0407 (7)
H14	0.228638	0.059223	0.332185	0.049*
C15	0.1750 (4)	0.16482 (18)	0.41610 (18)	0.0379 (6)
H15	0.018811	0.173386	0.390669	0.046*
C16	0.6631 (5)	0.00734 (18)	0.3791 (2)	0.0466 (8)
H16A	0.769847	0.035940	0.343057	0.070*
H16B	0.559583	−0.033613	0.339385	0.070*
H16C	0.748961	−0.028048	0.429739	0.070*
N1	0.3355 (4)	0.77931 (14)	0.74641 (16)	0.0428 (6)
H1A	0.410274	0.806343	0.706709	0.051*
H1B	0.214020	0.812081	0.752672	0.051*
O1	−0.1388 (3)	0.39956 (12)	0.62087 (14)	0.0459 (5)

In the title compound, as displayed in the figure, a methyl group and an amino group replace the hydrogen atoms on the opposite positions of the two benzene rings in the chalcone structure, respectively. Among them, the bond length of C13–C16 is 1.501(4) Å, and the bond length of C2–N1 is 1.372(4) Å. The bond angles of C12–C13–C16 and C14–C13–C16 are 120.8(3)° and 121.5(3)°, respectively [22]. In addition, the angle in the C10···C15 ring is in the range of 117.7°–121.5°, and the bond angles at substitutions sites are the smallest, indicating that the substitution on the benzene ring reduces the bond angle. The dihedral angle between the C10···C15 ring plane and the ketone plane is 12.4°, and the dihedral angle between the C1···C6 benzene ring plane and the ketone plane is 26.0°. Weak NH···O hydrogen bonds connects neighboring molecules to form chains along [10].

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