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Crystal structure of 2,2'-(*p*-tolylazanediy)bis(1-phenylethan-1-one), C₂₃H₂₁NO₂

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Abstract

C₂₃H₂₁NO₂, monoclinic, *C*2/*c* (no. 15), *a* = 20.163(3) Å, *b* = 10.1693(16) Å, *c* = 8.8032(19) Å, β = 95.992(12)°, *V* = 1795.2(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0624, *wR*_{ref}(*F*²) = 0.1411, *T* = 150 K.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	29.4°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	2111, 2111
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1425
<i>N</i> (<i>param</i>) _{refined} :	122
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Diamond [4]

CCDC no.: 2108404

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.

Source of material

The mixture of α -acetophenone bromide (49.8 mg, 0.25 mmol), *p*-toluidine (10.7 mg, 0.1 mmol), anhydrous sodium carbonate (15.9 mg, 0.1 mmol), and ethanol solvent (10 ml) was added into a 50 ml three mouth flask. The reaction solution was heated and refluxed at 90 °C for 48 h, then cooled to room temperature and filtered. Subsequently, the filter residue was washed three times with ethanol and water and dried to obtain a powder with a yield of 64.5%. Moreover, put the powdered product into a single mouth bottle and add an appropriate amount of methanol to form a suspension by ultrasound. Afterward, the bottle placed in the oil bath is heated to boiling and then cooled to room temperature. After the reaction, the reaction solution was cooled to room temperature to obtain light yellow crystals.

Experimental details

Comment

N,N-diacyl aromatic amine has played a vital role as an intermediate in the synthesis of pesticides, medicine, fine

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
C1	0.500000	1.0609 (3)	0.750000	0.0476 (10)
H1A ^a	0.542913	1.092370	0.792123	0.071*
H1B ^a	0.466430	1.092370	0.810699	0.071*
H1C ^a	0.490657	1.092370	0.647178	0.071*
C2	0.500000	0.9124 (3)	0.750000	0.0289 (7)
C3	0.54704 (11)	0.8408 (2)	0.6809 (2)	0.0278 (5)
H3	0.579037	0.885868	0.632481	0.033*
C4	0.54817 (10)	0.7049 (2)	0.6812 (2)	0.0241 (5)
H4	0.581198	0.660691	0.635253	0.029*
C5	0.500000	0.6328 (3)	0.750000	0.0194 (6)
C6	0.54436 (10)	0.42012 (19)	0.6661 (2)	0.0222 (5)
H6A	0.556256	0.471462	0.580111	0.027*
H6B	0.520915	0.342290	0.625783	0.027*
C7	0.60772 (10)	0.3781 (2)	0.7621 (2)	0.0213 (5)
C8	0.65091 (10)	0.27674 (19)	0.6994 (2)	0.0225 (5)
C9	0.70461 (11)	0.2267 (2)	0.7929 (2)	0.0332 (6)
H9	0.713725	0.258651	0.891864	0.040*
C10	0.74458 (12)	0.1304 (2)	0.7410 (3)	0.0396 (6)
H10	0.780408	0.097503	0.804850	0.047*
C11	0.73155 (12)	0.0827 (2)	0.5946 (3)	0.0386 (6)
H11	0.758561	0.017679	0.559449	0.046*
C12	0.67846 (12)	0.1315 (2)	0.5005 (3)	0.0386 (6)
H12	0.669769	0.099266	0.401513	0.046*
C13	0.63794 (11)	0.2279 (2)	0.5518 (2)	0.0319 (5)
H13	0.602031	0.260045	0.487664	0.038*
N1	0.500000	0.4970 (2)	0.750000	0.0224 (5)
O1	0.62217 (7)	0.42488 (14)	0.88885 (15)	0.0305 (4)

^aOccupancy: 0.5.

chemicals and other fields [5–7]. The reason is that *N,N*-diacyl aromatic amines can synthesize 1,4-dihydropyrazine [8], piperidine [9], oxazine [10] and other heterocyclic compounds through dehydration and ring closure. So far, researchers have made many attempts to synthesize *N,N*-diacyl arylamine derivatives, using the heating reflux method [11], grinding method [12], ultrasonic-assisted method [13] and so on. Heating reflux has the advantages of low cost and easy operation, but is limited by simple equipment, resulting in slow reaction speed and low yield. The grinding method can greatly reduce the pressure on the environment caused by solvent discharge because it does not use any solvent. Unfortunately, this method is only suitable for the preparation of a small amount of products. Moreover, the ultrasonic-assisted method significantly shortens the reaction time and improves the yield of *N,N*-diacyl aromatic amines, but this technology has been developed slowly and needs further research before it can be used for industrialization. However, regardless of the advantages and disadvantages of these methods, the products obtained are basically amorphous powder with low purity.

In addition, some products have specific requirements for the crystal form of the raw materials during the synthesis process. Therefore, in order to improve the purity of the product, we use the recrystallization method to quickly and easily convert the powdered product into a crystal.

The title molecule is located on a two fold axis (see the figure). The bond lengths and bond angles of the crystal structure are within a reasonable range. Meanwhile, there is no hydrogen bonding interaction between molecules. The torsion angles of N1–C6–C7–O1 and N1–C6–C7–C8 are 10.95° and –167.80° respectively. In addition, the dihedral angle between half of the benzene ring plane (C2–C3–C4–C5) and the other benzene ring plane (C8–C9–C10–C11–C12–C13) is 87.87(7)°.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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