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Crystal structure of (Z)-3-(6-bromo-1*H*-indol-3-yl)-1,3-diphenylprop-2-en-1-one, C₂₃H₁₆BrNO

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Abstract

C₂₃H₁₆BrNO, triclinic, *P* $\bar{1}$ (no. 2), *a* = 9.914(3) Å, *b* = 10.141(3) Å, *c* = 10.179(3) Å, α = 90.177(5)°, β = 112.202(5)°, γ = 95.167(5)°, *V* = 942.9(4) Å³, *Z* = 2, *R*_{gt}(*F*) = 0.0520, *wR*_{ref}(*F*²) = 0.1351, *T* = 296 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.

Source of material

1,3-Diphenylpropane-1,3-dione (2.0 mmol), 6-bromo-1*H*-indole (6.0 mmol) and CF₃SO₃H (4.0 mmol) were mixed and

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.25 × 0.23 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.19 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	4715, 3262, 0.018
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2338
<i>N</i> (<i>param</i>) _{refined} :	235
Programs:	Bruker [1], SHELX [2], Olex2 [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.73990 (7)	0.98733 (5)	0.30366 (7)	0.0742 (3)
O1	0.6867 (3)	0.3230 (3)	0.2735 (3)	0.0397 (7)
N1	1.0055 (3)	0.6059 (3)	0.6315 (3)	0.0342 (8)
H1A	1.093526	0.642946	0.671364	0.041*
C1	0.7589 (4)	0.3744 (4)	0.8329 (4)	0.0349 (10)
H1	0.767345	0.464771	0.818313	0.042*
C2	0.7628 (5)	0.3314 (5)	0.9635 (4)	0.0441 (11)
H2	0.772361	0.392605	1.035432	0.053*
C3	0.7524 (5)	0.1992 (5)	0.9856 (5)	0.0521 (13)
H3	0.753335	0.170343	1.072558	0.063*
C4	0.7408 (6)	0.1089 (5)	0.8818 (5)	0.0526 (13)
H4	0.737032	0.018928	0.899214	0.063*
C5	0.7346 (5)	0.1503 (4)	0.7506 (5)	0.0412 (11)
H5	0.724803	0.087905	0.679605	0.049*
C6	0.7427 (4)	0.2843 (4)	0.7239 (4)	0.0285 (9)
C7	0.7225 (4)	0.3315 (4)	0.5801 (4)	0.0266 (8)
C8	0.6183 (4)	0.2668 (4)	0.4662 (4)	0.0313 (9)
H8	0.558949	0.199055	0.484508	0.038*
C9	0.5880 (4)	0.2917 (4)	0.3156 (4)	0.0294 (9)
C10	0.4332 (4)	0.2755 (4)	0.2137 (4)	0.0288 (9)
C11	0.3161 (4)	0.2491 (4)	0.2565 (4)	0.0357 (10)
H11	0.333769	0.238204	0.352058	0.043*
C12	0.1745 (5)	0.2389 (5)	0.1585 (5)	0.0443 (11)
H12	0.096971	0.219706	0.187817	0.053*
C13	0.1472 (5)	0.2571 (5)	0.0171 (5)	0.0494 (12)
H13	0.051381	0.251643	-0.048932	0.059*
C14	0.2611 (5)	0.2831 (6)	-0.0258 (5)	0.0565 (14)
H14	0.242556	0.293710	-0.121607	0.068*
C15	0.4031 (5)	0.2939 (5)	0.0709 (4)	0.0455 (12)
H15	0.479643	0.313807	0.040270	0.055*
C16	0.8109 (4)	0.4537 (4)	0.5749 (4)	0.0264 (8)
C17	0.7670 (4)	0.5677 (4)	0.4906 (4)	0.0251 (8)
C18	0.8919 (4)	0.6600 (4)	0.5302 (4)	0.0285 (9)

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Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C19	0.9560 (4)	0.4845 (4)	0.6583 (4)	0.0316 (9)
H19	1.013011	0.428911	0.724742	0.038*
C20	0.6351 (4)	0.6038 (4)	0.3914 (4)	0.0300 (9)
H20	0.551228	0.544434	0.362642	0.036*
C21	0.6306 (5)	0.7273 (4)	0.3371 (4)	0.0372 (10)
H21	0.542875	0.752413	0.272124	0.045*
C22	0.7560 (5)	0.8153 (4)	0.3782 (5)	0.0390 (10)
C23	0.8894 (5)	0.7858 (4)	0.4752 (5)	0.0393 (10)
H23	0.972466	0.846126	0.502269	0.047*

stirred overnight in 1-butanol (3.0 mL). When the reaction was completed, the solvent was removed under reduced pressure.

Then the crude residue was purified by silica gel column chromatography using a gradient of ethyl acetate/hexane (0–100%) to afford the title product.

Experimental details

A suitable crystal was selected and mounted on a Bruker APEX-II CCD. The crystal was kept at 296 K during data collection. The structure was solved with the ShelXS refinement package using Least-Squares minimization [2]. All hydrogen atoms were added using a riding model using the default parameters of the SHELX and Olex program [3].

Comment

Indols are widely employed in drug discovery and typically exhibit a wide range of important biological activities [4]. The asymmetric unit of the title compound contains one molecule. The bond lengths and angles are in the normal ranges [5–7]. In conclusion, we have developed a facile

process for the synthesis of (Z)-3-(6-bromo-1H-indol-3-yl)-1,3-diphenylprop-2-en-1-one using 1,3-diphenylpropane-1,3-dione and 6-bromo-1H-indole. This scaffold with more possible spaces for further modifications provides great benefit toward medicinal chemistry.

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