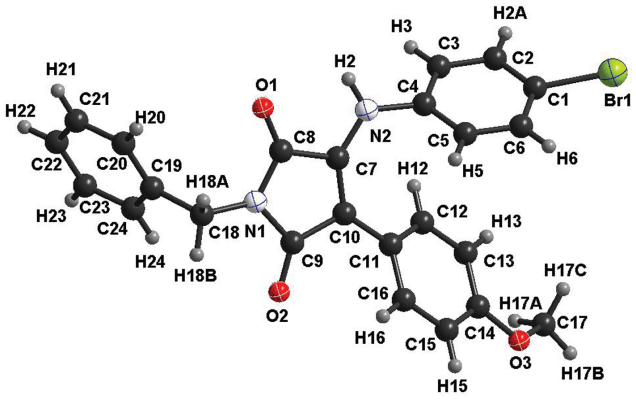
Meng Lan Lv*

Crystal structure of 1-benzyl-3-((4-bromophenyl) amino)-4-(4-methoxyphenyl)-1*H*-pyrrole-2,5-dione, $C_{24}H_{19}BrN_2O_3$



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Abstract

$C_{24}H_{19}BrN_2O_3$, monoclinic, $P\bar{1}$ (no. 2), $a = 9.1003(5)$ Å, $b = 9.9785(6)$ Å, $c = 12.9831(6)$ Å, $\alpha = 74.611(5)°$, $\beta = 76.063(4)°$, $\gamma = 64.264(5)°$, $V = 1013.17(10)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0467$, $wR_{ref}(F^2) = 0.1259$, $T = 294$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Atoms are shown with arbitrary radii. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A solution of *p*-methoxybenzoylformic acid (1.0 mmol), benzyl isocyanide (1.0 mmol), aniline (1.0 mmol) and ethyl glyoxylate (1.0 mmol) was stirred overnight in MeOH (2.0 mL) at room temperature. The reaction mixture was monitored by TLC. When no isonitrile was left, the solvent was removed under nitrogen blowing and the crude residue was dissolved in DIPA (2.0 equiv.) and DMF (3.0 mL). This solution was

***Corresponding author: Meng Lan Lv,** School of Chemical Engineering, Guizhou Institute of Technology, Guizhou 550003, P.R. China, e-mail: lvmenglan@git.edu.cn

Table 1: Data collection and handling.

Crystal:	Block, clear yellow
Size:	0.7 × 0.6 × 0.4 mm
Wavelength:	Cu *Kα* radiation (1.54184 Å)
μ:	3.01 mm^{-1}
Diffractometer, scan mode:	Gemini Dual, φ and ω-scans
θ_{max}, completeness:	67°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10717, 3605, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3264
$N(param)_{refined}$:	582
Programs:	CrysAlis^{PRO} [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.28626(4)	0.45593(3)	1.02761(2)	0.05582(16)
O1	0.0180(3)	0.9399(2)	0.38815(17)	0.0506(5)
O2	0.2081(3)	0.4771(2)	0.30033(17)	0.0536(5)
O3	0.5223(3)	−0.0465(2)	0.6896(2)	0.0614(6)
N1	0.1055(3)	0.7279(2)	0.31493(18)	0.0422(5)
N2	0.1655(3)	0.7488(2)	0.56683(18)	0.0447(5)
H2	0.1414	0.8446	0.5532	0.054*
C7	0.1650(3)	0.6888(3)	0.4858(2)	0.0361(5)
C11	0.3002(3)	0.3948(3)	0.5247(2)	0.0364(5)
C10	0.2177(3)	0.5479(3)	0.4630(2)	0.0367(6)
C3	0.3138(4)	0.6938(3)	0.7146(2)	0.0452(6)
H3	0.3690	0.7539	0.6727	0.054*
C8	0.0870(3)	0.8043(3)	0.3921(2)	0.0380(5)
C1	0.2606(3)	0.5374(3)	0.8794(2)	0.0399(6)
C9	0.1809(3)	0.5696(3)	0.3542(2)	0.0385(5)
C4	0.2013(3)	0.6720(3)	0.6721(2)	0.0375(5)
C2	0.3434(4)	0.6259(3)	0.8195(2)	0.0469(6)
H2A	0.4181	0.6400	0.8489	0.056*
C16	0.2626(3)	0.2752(3)	0.5181(2)	0.0430(6)
H16	0.1855	0.2935	0.4749	0.052*
C12	0.4195(3)	0.3604(3)	0.5884(2)	0.0411(6)
H12	0.4491	0.4367	0.5932	0.049*
C19	0.1193(3)	0.8912(3)	0.1351(2)	0.0414(6)
C18	0.0359(4)	0.7963(3)	0.2144(2)	0.0463(6)
H18A	−0.0802	0.8592	0.2307	0.056*
H18B	0.0440	0.7167	0.1810	0.056*
C6	0.1521(4)	0.5113(3)	0.8376(2)	0.0455(6)
H6	0.0998	0.4485	0.8790	0.055*
C13	0.4956(3)	0.2149(3)	0.6452(2)	0.0447(6)
H13	0.5738	0.1953	0.6880	0.054*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C14	0.4554(3)	0.0997(3)	0.6383(2)	0.0456(6)
C15	0.3380(4)	0.1310(3)	0.5745(3)	0.0479(6)
H15	0.3098	0.0540	0.5697	0.057*
C5	0.1225(3)	0.5798(3)	0.7335(2)	0.0444(6)
H5	0.0489	0.5639	0.7042	0.053*
C20	0.0385(4)	1.0475(3)	0.1137(2)	0.0514(7)
H20	−0.0658	1.0939	0.1509	0.062*
C22	0.2639(4)	1.0690(4)	−0.0185(3)	0.0603(8)
H22	0.3119	1.1280	−0.0706	0.072*
C21	0.1118(4)	1.1348(3)	0.0376(3)	0.0584(8)
H21	0.0567	1.2396	0.0245	0.070*
C24	0.2747(4)	0.8254(3)	0.0805(3)	0.0583(8)
H24	0.3325	0.7211	0.0954	0.070*
C23	0.3452(4)	0.9150(4)	0.0030(3)	0.0692(10)
H23	0.4492	0.8695	−0.0349	0.083*
C17	0.6671(5)	−0.0933(4)	0.7358(3)	0.0702(10)
H17A	0.7511	−0.0735	0.6813	0.105*
H17B	0.7049	−0.1998	0.7649	0.105*
H17C	0.6431	−0.0385	0.7925	0.105*

treated in microwave at 130 °C for 10 min. After the microwave vial was cooled to room temperature, the residue was purified by silica gel column chromatography using a gradient of ethyl acetate/hexane (20–100%) to afford the relative targeted title product.

Experimental details

All hydrogen atoms were added using a riding model using the default parameters [2].

Discussion

The Ugi four-component reaction (U-4CR), followed by various post-condensation transformations, is well known as a versatile and highly efficient synthetic strategy for the preparation of heterocyclic compounds [3]. Moreover, these multicomponent reactions (MCRs) provide facile access to complex products with high iterative efficiency potential [4], for the design of novel drug scaffolds from commercially available starting materials. We previously reported the use of this strategy to achieve the construction of various heterocyclic systems, including benzodiazepines [5], benzimidazoles [6], pyrazoles [7] and quinoxalines [8], as well as several other systems. This new method represents a facile and efficient one-pot procedure for the preparation of these ring systems under basic conditions. Furthermore, this sequence could be readily tailored to the design of a wide range of biologically active scaffolds by varying the nature of the starting materials used in the Ugi reaction.

The title molecule is depicted in the figure. The geometric parameters are in expected ranges especially with regard to its parent compound [9].

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