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Crystal structure of 4-(2-bromo-4-(6-morpholino-3-phenyl-3*H*-benzo[*f*]chromen-3-yl) cyclohexa-2,5-dien-1-yl)morpholine, C₃₃H₃₁BrN₂O

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.18 × 0.17 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.58 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	24.7°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18753, 4521, 0.072
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2866
$N(\text{param})_{\text{refined}}$:	352
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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Abstract

C₃₁H₃₁BrN₂O₃, monoclinic, $P2_1/c$ (no. 14), $a = 10.2014(15)$ Å, $b = 21.499(3)$ Å, $c = 12.1885(18)$ Å, $\beta = 95.175(2)^\circ$, $V = 2662.3(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0521$, $wR_{\text{ref}}(F^2) = 0.1993$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal 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

4-Morpholinonaphthalen-2-ol (2.4 mmol), 1-(3-bromo-4-morpholinophenyl)-1-phenylprop-2-yn-1-ol (1.2 mmol), toluene

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(50 mL) and *p*-toluenesulfonic acid (0.24 mmol) respectively were added to a 100 mL single-mouth flask. The resultant reaction mixture was stirred at 80 °C, and TLC was used to monitor the progress of the reaction. After the reaction was completed, toluene was removed under reduced pressure. A saturated sodium carbonate solution (100 mL) was added, and the mixture was extracted with ethyl acetate (50 mL × 3). The organic phases were combined, dried over anhydrous MgSO₄, and concentrated under reduced pressure. Purification of the residue furnished the title compound.

Experimental details

Hydrogen atoms were positioned geometrically.

Discussion

Photochromism is referred to as a reversible photoisomerization process induced at least in one direction by an electromagnetic radiation between two isomers having different absorption spectra such as biindenylidenedione compounds, quaterthiophene compound, benzofuran compound [5–8]. In 1992, the family of biradical compounds was first explored by Toda and Tanaka [9]. During the past years, various types of organic photochromic compounds have aroused lots of interests because of their potential applications as photoswitchable molecular devices, optical memory storage media, as well as multi-color display [10–17]. For practical applications, some groups try to modulate

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.31773(5)	0.50108(3)	0.92376(5)	0.0332(2)
N1	0.3167(5)	0.5022(2)	0.6718(4)	0.0295(11)
N2	0.2058(4)	0.8100(2)	1.0609(3)	0.0240(11)
O1	0.1261(4)	0.4444(2)	0.5230(4)	0.0459(12)
O2	0.6065(3)	0.71007(18)	0.9798(3)	0.0249(9)
O3	−0.0051(4)	0.78428(19)	1.1924(3)	0.0355(11)
C1	0.7913(5)	0.6469(3)	0.9447(5)	0.0254(13)
C2	0.4268(5)	0.8373(3)	0.8329(4)	0.0229(13)
C3	0.5240(5)	0.7909(3)	0.8576(4)	0.0226(13)
C4	0.5134(5)	0.7531(3)	0.9478(5)	0.0217(12)
C5	0.3118(5)	0.8019(2)	0.9921(4)	0.0218(12)
C6	0.3188(5)	0.8438(3)	0.9001(4)	0.0211(12)
C7	0.6712(5)	0.6817(3)	0.8903(5)	0.0260(13)
C8	0.6435(5)	0.7830(3)	0.7999(5)	0.0279(14)
H8	0.6679	0.8136	0.7518	0.034*
C9	0.4089(5)	0.7585(3)	1.0148(4)	0.0216(12)
H9	0.4055	0.7324	1.0753	0.026*
C10	0.4148(5)	0.5555(3)	0.8373(5)	0.0256(13)
C11	0.2405(6)	0.9336(3)	0.7924(5)	0.0307(14)
H11	0.1797	0.9655	0.7790	0.037*
C12	0.2290(5)	0.8931(3)	0.8779(4)	0.0257(13)
H12	0.1603	0.8983	0.9223	0.031*
C13	0.4027(5)	0.5475(3)	0.7221(5)	0.0266(13)
C14	0.7153(5)	0.7331(3)	0.8162(4)	0.0257(14)
H14	0.7932	0.7290	0.7827	0.031*
C15	0.4972(5)	0.5985(3)	0.8897(5)	0.0266(13)
H15	0.5019	0.6022	0.9660	0.032*
C16	0.9570(6)	0.6311(3)	1.0962(5)	0.0377(16)
H16	0.9915	0.6402	1.1676	0.045*
C17	0.8421(5)	0.6604(3)	1.0528(5)	0.0279(14)
H17	0.7994	0.6886	1.0951	0.033*
C18	0.3435(6)	0.9268(3)	0.7256(5)	0.0308(15)
H18	0.3512	0.9543	0.6676	0.037*
C19	0.2297(6)	0.7820(3)	1.1723(5)	0.0299(14)
H19A	0.3140	0.7956	1.2072	0.036*
H19B	0.2307	0.7370	1.1669	0.036*
C20	0.9684(6)	0.5754(3)	0.9282(6)	0.0417(17)
H20	1.0110	0.5469	0.8864	0.050*
C21	0.0748(5)	0.7902(3)	1.0108(5)	0.0275(14)
H21A	0.0728	0.7454	1.0020	0.033*
H21B	0.0574	0.8090	0.9385	0.033*
C22	0.4331(5)	0.8802(3)	0.7447(5)	0.0267(14)
H22	0.5005	0.8763	0.6986	0.032*
C23	0.4810(6)	0.5866(3)	0.6648(5)	0.0321(15)
H23	0.4766	0.5830	0.5886	0.039*
C24	0.1206(6)	0.8025(3)	1.2393(5)	0.0357(16)
H24A	0.1353	0.7850	1.3128	0.043*
H24B	0.1229	0.8475	1.2462	0.043*
C25	0.5656(5)	0.6308(3)	0.7155(5)	0.0283(14)
H25	0.6158	0.6559	0.6735	0.034*
C26	0.2595(6)	0.4245(3)	0.5321(6)	0.0403(17)
H26A	0.2815	0.4086	0.4616	0.048*
H26B	0.2711	0.3911	0.5856	0.048*
C27	0.8554(5)	0.6041(3)	0.8846(5)	0.0366(16)
H27	0.8214	0.5944	0.8133	0.044*
C28	0.5746(5)	0.6370(3)	0.8291(5)	0.0257(13)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C29	−0.0293(5)	0.8103(3)	1.0843(5)	0.0332(15)
H29A	−0.0300	0.8553	1.0895	0.040*
H29B	−0.1151	0.7969	1.0521	0.040*
C30	1.0192(6)	0.5893(3)	1.0354(5)	0.0345(15)
H30	1.0958	0.5699	1.0653	0.041*
C31	0.3496(6)	0.4771(3)	0.5670(6)	0.0384(16)
H31A	0.4400	0.4626	0.5740	0.046*
H31B	0.3418	0.5096	0.5115	0.046*
C32	0.1770(5)	0.5212(3)	0.6631(5)	0.0335(15)
H32A	0.1633	0.5554	0.6114	0.040*
H32B	0.1534	0.5352	0.7344	0.040*
C33	0.0926(6)	0.4676(3)	0.6249(5)	0.0397(16)
H33A	0.1021	0.4348	0.6796	0.048*
H33B	0.0012	0.4807	0.6177	0.048*

the photochromic properties of diarylethenes, which has recently been reported. Huang's groups gated the spectral properties of dithienylethene by means of Lewis acid-base interaction [18, 19].

In this work, we report on the synthesis of the title compound in high yield, which is a key method for the synthesis of photochromic compounds. There is one crystallographically independent molecule in the asymmetric unit, in which all bond lengths are in normal ranges. In the title crystal structure including a pyran ring, several important bond lengths are as follows: O2–C7 = 1.459 Å, C7–C14 = 1.521 Å, C14–C8 = 1.304 Å, C8–C3 = 1.471 Å, C3–C4 = 1.380 Å, C4–O2 = 1.357 Å.

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