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One-step synthesis, characterization and X-ray analysis of benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-ones

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HIGHLIGHTS

- One-step ring-closure synthesis of benzo[4,5]imidazo[2,1-*a*]-isoquinolin-7-ones.
- The isomeric compounds were characterized by HRMS, NMR and X-ray crystallography.
- Substituent position influences non-classical intermolecular C–H···O interactions.
- Substituent position causes obviously different modes of structure stabilization.

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ABSTRACT

Two isomeric compounds of methoxy substituted benzo[*de*]benzo[4,5]imidazo[2,1-*a*] -isoquinolin-7-one were obtained from the one-step ring closure reaction of 2-(2-acetamino-5-methoxyphenyl)benzo[*de*]isoquinoline-1,3-dione under acidic condition. Their structures were characterized by using HRMS, NMR spectra analyses together with single crystal X-ray diffraction. Compound **2** crystallizes in the monoclinic P2 (1)/n space group with the crystal cell parameters $a = 8.3311(11)$ Å, $b = 7.6646(10)$ Å, $c = 21.201(2)$ Å, $\alpha = 90.00°$, $\beta = 95.7380(10)°$, $\gamma = 90.00°$, $V = 1347.0(3)$ Å^3 and $Z = 4$. Compound **3** crystallizes in the monoclinic space group P2 (1)/c with the crystal cell parameters $a = 3.8270(3)$ Å, $b = 15.8361(14)$ Å, $c = 28.559(3)$ Å, $\alpha = 90.00°$, $\beta = 94.1090(10)°$, $\gamma = 90.00°$, $V = 1726.4(3)$ Å^3 and $Z = 4$. X-ray results indicated that it exist non-classically intermolecular interactions in the molecules of the two compounds. The substitution position of methoxy group has no obvious influence on the whole molecular planarity, but results in great different formation modes of their intermolecular interactions and structural stabilization.

1. Introduction

Naphthalimide derivatives have attracted much attention in various fields of industry as fluorescent brightening agents [1,2], fluorescent colorants [3,4], drugs and biological agents [5–7], fluorescence and luminescent materials [8–10]. Furthermore, in recent years naphthalimide derivatives have emerged as organic light-emitting diode materials because of their good optical, thermal and chemical stabilities, as well as their high photoluminescence quantum efficiency in solution [11,12]. It is reported that 1,8-naphthalimides substituted at the 4-position with electron-donating groups have high fluorescent quantum yield, and their photoluminescence emission color can be readily changed [13–16]. Because of the existence of an electron-deficient center of naphthalimide derivatives, they usually have high electron affinity [17,18], which should have good capability of electron transportation and is suitable for organic light-emitting diode materials.

It is well known that properties of chemical compounds are usually dependent not only on their molecular structures but also on their inter- and intra-molecular interactions in crystalline state [19,20]. In addition to a more intuitive show of molecular structure, X-ray analysis can also provide full crystallography details, including inter- and intra-molecular interactions, morphology, as well as crystal stacking and arrangement, which are generally related to their technical performance and functionality [21]. Therefore, the structural investigation of different compounds in solid state has always been an area of devoted interest [22–25].

Regarding our continuous research interest on molecular structures and their properties, we reported several X-ray analyses of azo compounds [26,27], anthraquinone derivatives [28,29] and benzophenones [30]. Following this interest, we report here spectroscopic analysis and X-ray diffraction of two methoxy substituted benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-ones, as shown in Scheme 1.

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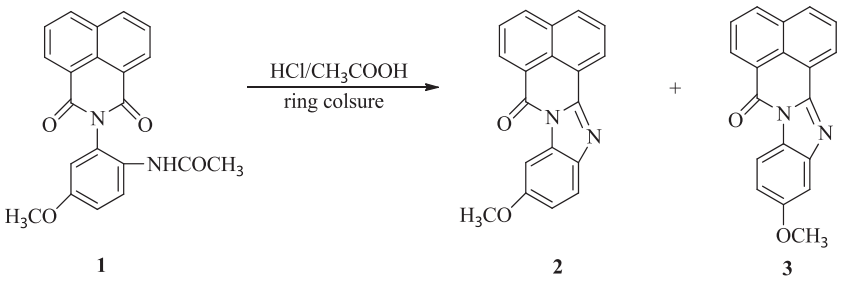

Scheme 1. Synthetic route for the titled isoquinolinones.

Table 1
Crystallographic data for methoxy substituted benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-ones (**2** and **3**).

Data points	Compound **2**	Compound **3**
Formula	$C_{19}H_{12}N_2O_2$	$C_{19}H_{12}N_2O_2$
Formula weight (g/mol)	300.31	300.31
Crystal dimensions (mm)	0.38 × 0.36 × 0.35	0.4 × 0.30 × 0.16
Crystal system	Monoclinic	Monoclinic
Space group	P2 (1)/*n*	P2 (1)/*c*
Temperature (K)	293(2)	293(2)
a (Å)	8.3311(11)	3.8270(3)
b (Å)	7.6646(10)	15.8361(14)
c (Å)	21.201(2)	28.559(3)
α (°)	90.00	90.00
β (°)	95.7380(10)	94.1090(10)
γ (°)	90.00	90.00
V (Å^3)	1347.0(3)	1726.4(3)
Number of reflections to determine final unit cell	1125	917
Min and max 2θ for cell determination (°)	5.46, 48.0	5.002, 38.634
Z	4	4
F(000)	624	624
ρ (g/cm)	1.481	1.155
λ (Å), (Mo Kα)	0.71073	0.71073
μ (cm^{-1})	0.098	0.077
Diffractometer type	CCD area detector	CCD area detector
Scan type(s)	Phi and omega scans	Phi and omega scans
Max 2θ for data collection (°)	50.02	50.04
Measured fraction of data	0.998	0.985
Number of reflections measured	6455	8822
Unique reflections measured	2363	3016
R_{merge}	0.0412	0.0735
Number of reflections included in refinement	2363	3016
Cut off threshold expression	>2sigma(I)	>2sigma(I)
Structure refined using	Full matrix least-squares using F^2	Full matrix least-squares using F^2
Weighting scheme	Calc w = 1/[\s ∧ 2 ∧ (Fo ∧ 2∧) + (0.1008P) ∧ 2 ∧ +0.7491P] where P = (Fo ∧ 2 ∧ +2Fc ∧ 2∧)/3	Calc w = 1/[\s ∧ 2 ∧ (Fo ∧ 2∧) + (0.0853P) ∧ 2 ∧ +0.0000P] where P = (Fo ∧ 2 ∧ +2Fc ∧ 2∧)/3
Number of parameters in least-squares	209	211
R_1	0.0675	0.0826
wR_2	0.1789	0.1867
R_1 (all data)	0.1050	0.1477
wR_2 (all data)	0.2047	0.2077
GOF	1.048	1.020
Maximum shift/error	0.000	0.001
Min and max peak heights on final Δ*F* map (*e*$^-$/Å)	−0.239, 0.486	−0.225, 0.388

2. Experimental

2.1. General

2-(2-Acetamino-5-methoxyphenyl)benzo[*de*]isoquinoline-1,3-dione (**1**) was provided by Jihua Group (Hangzhou, China). Common reagent grade chemicals were commercially available and were used without further purification. High resolution mass spectra (HRMS) were recorded on a UPLC/Q-Tof Mass Spectrometer (Micromass, UK). Nuclear magnetic resonance (NMR) spectra were recorded on an AVANCE II 400 spectrometer (Bruker, Switzerland) using TMS as internal standard.

2.2. Synthesis

The 1.80 g of 2-(2-acetamino-5-methoxyphenyl)benzo[*de*]isoquinoline-1,3-dione (**1**) was added slowly to the mixture of 4.68 g acetic acid, 2.45 g hydrochloric acid (30% aqueous) and 10 mL water. Then the mixture was refluxed for 4 h. After cooled to room temperature, the yellow powder was collected by filtration. The crude was purified by preparative thin layer chromatography in silica gel using toluene/ethyl acetate (5/1, v/v) as eluent.

*Compound **2:*** yellow needles, 85.0% yield. M.p.: 210 °C. HRMS (ES Positive): m/z 301.0984 (M + H, 100%), Calc. 301.0977 (M + H). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.65 (s, 1H, Ar), 8.63

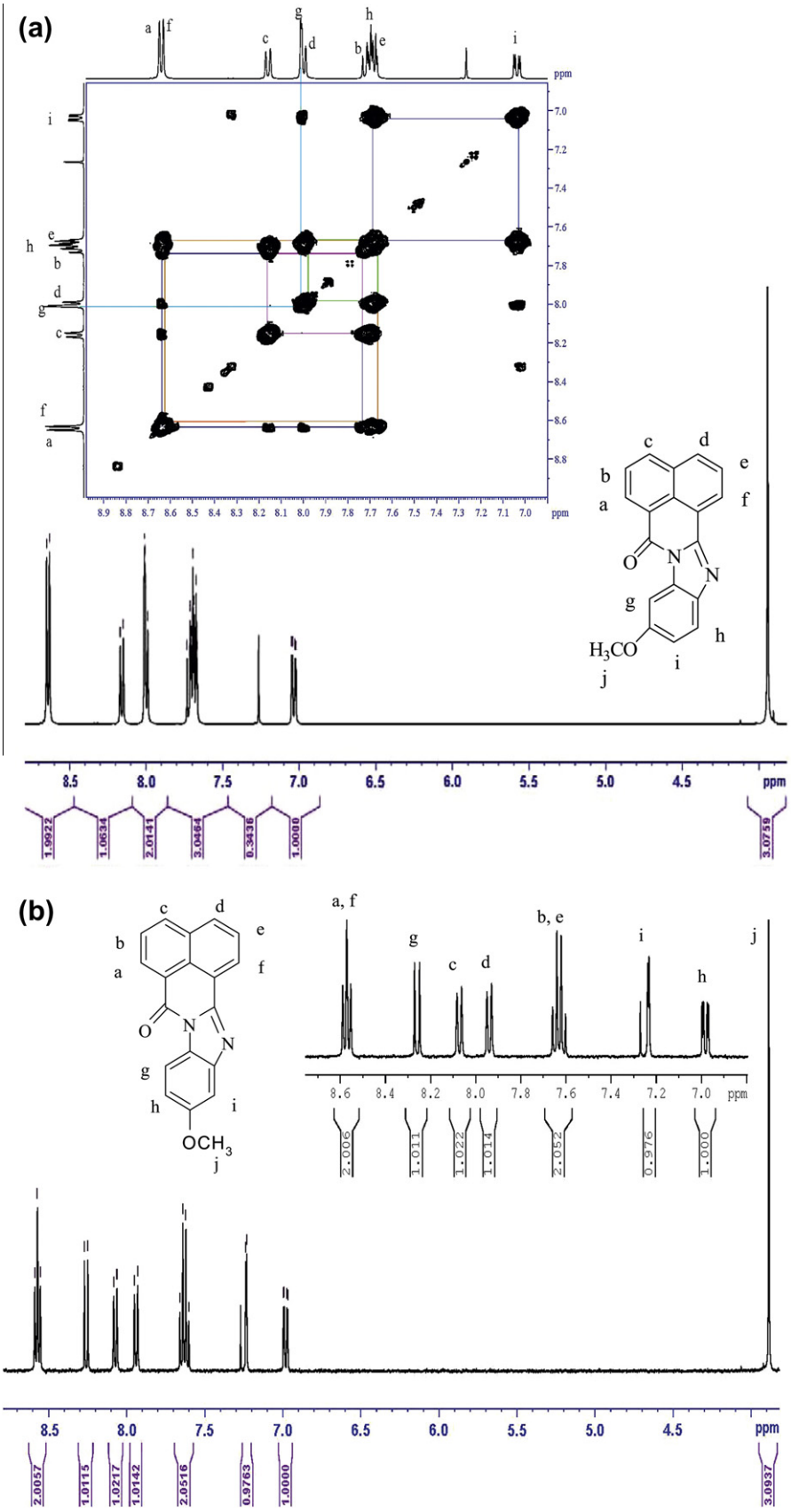

Fig. 1. ^{1}H NMR spectra in $CDCl_3$ for compound **2** (a, inset: ^{1}H–^{1}H COSY spectra) and compound **3** (b, inset: enlarged part of aromatic protons.).

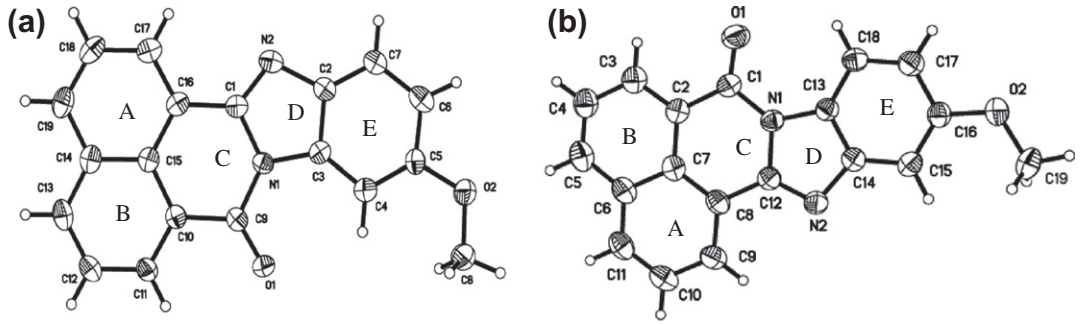

Fig. 2. ORTEP drawing of compounds **2** (a) and **3** (b) showing numbering scheme.

(s, 1H, Ar), 8.16 (d, 1H, Ar, J = 8.0), 8.01 (s, 1H, Ar), 7.99 (s, 1H, Ar), 7.70 (m, 3H, Ar), 7.04 (d–d, 1H, Ar, *J* = 8.8), 3.94 (s, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.97, 158.43, 148.32, 138.07, 135.31, 132.74, 132.33, 131.44, 131.26, 127.43, 127.07, 126.86, 126.37, 123.23, 120.96, 120.50, 115.10, 99.45, 56.11.

Compound ***3:*** yellow needles, 9.7% yield. M.p.: 194 °C. HRMS (ES Positive): m/z 301.0975 (M + H, 100%), Calc. 301.0977 (M + H). ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.57 (t, 2H, Ar), 8.26 (d, 1H, Ar, *J* = 8.8), 8.07 (d, 1H, Ar, *J* = 8.0), 7.94 (d, 1H, Ar, *J* = 8.0), 7.63 (q, 2H, Ar), 7.24 (s, 1H, Ar), 6.99 (d–d, 1H, Ar, *J* = 8.8), 3.89 (s, 3H, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.20, 158.33, 149.64, 144.93, 135.13, 132.08, 131.70, 131.52, 127.27, 126.95, 126.80, 126.08, 123.00, 120.47, 116.24, 114.20, 102.65, 55.80.

2.3. X-ray analysis

Their suitable crystals for X-ray were achieved by dissolving the samples in 1,2-dicholorethane at room temperature. The resulting solution was covered with a Parafilm plastic containing pinholes and kept for several days, allowing slow evaporation of solvent.

The suitable each crystal was mounted on a nylon loop using a small amount of Paratone N oil. All X-ray measurements were made on a Bruker SMART diffractometer equipped with a graphite monochromated Mo Ka (λ = 0.71073) radiation source and a CCD detector. The frame integration was performed using the program SAINT [31]. The structures were solved by direct method provided by the program package SHELXTL-97 [32] and refined a full matrix least square against F^2 for all data. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were introduced at idealized positions and were allowed to refine isotropically. Crystallographic data of compounds **2** and **3** are summarized in Table 1.

3. Results and discussion

3.1. Synthesis

In very earlier study, it was reported that two isomeric mixture of **2** and **3** were obtained by using 1,8-naphthalic anhydride and 3,4-diaminoanisole as starting materials [33,34]. In this reaction, the two amino groups in 3,4-diaminoanisole have similar competitive ability of condensation with 1,8-naphthalic anhydride. Because of their different relative positions to the methoxy group, it was expected to obtain two different condensation products, which were subsequently converted to the above-mentioned isomeric mixture (**2** and **3**) via ring-closure reaction. But, in present case, it was undertaken the ring closure reaction with 2-(2-acetamino-5-methoxyphenyl)benzo[*de*]isoquinoline-1,3-dione (**1**), which was the only one compound derived from the condensation reaction of 1,8-naphthalic anhydride. After the reaction was over, it was unexpected to obtain the isomeric 11-methoxy-benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-one (**3**) in 9.7% yield, besides the target of 10-methoxy-benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-one (**2**). The exact mechanism of its appearance is not absolutely clear and still in the further study.

Table 2
Selected bond lengths (Å) and bond angles (°) for compound **2**.

N1–C1	1.403(4)	N1–C3	1.404(4)	N1–C9	1.390(4)
N2–C1	1.307(4)	N2–C2	1.442(4)	O1–C9	1.213(4)
O2–C5	1.380(4)	O2–C8	1.427(5)	C1–C16	1.435(5)
C2–C3	1.408(5)	C2–C7	1.372(5)	C3–C4	1.384(5)
C4–C5	1.369(5)	C5–C6	1.391(5)	C6–C7	1.393(5)
C9–C10	1.484(5)	C10–C11	1.368(5)	C10–C15	1.418(5)
C11–C12	1.392(5)	C12–C13	1.370(5)	C13–C14	1.408(5)
C14–C15	1.419(5)	C14–C19	1.420(5)	C15–C16	1.413(5)
C16–C17	1.373(5)	C17–C18	1.388(5)	C18–C19	1.368(6)
C9–N1–C1	125.5(3)	C9–N1–C3	126.8(3)	C1–N1–C3	107.6(3)
C1–N2–C2	104.9(3)	C5–O2–C8	117.2(3)	N2–C1–N1	112.8(3)
N2–C1–C16	127.9(3)	N1–C1–C16	119.3(3)	C7–C2–C3	120.5(3)
C7–C2–N2	129.2(3)	C3–C2–N2	110.3(3)	C4–C3–N1	133.8(3)
C4–C3–C2	121.8(3)	N1–C3–C2	104.4(3)	C5–C4–C3	116.3(3)
C4–C5–O2	123.6(3)	C4–C5–C6	123.5(3)	O2–C5–C6	112.9(3)
C5–C6–C7	119.5(3)	C2–C7–C6	118.4(4)	O1–C9–N1	120.6(3)
O1–C9–C10	124.5(3)	N1–C9–C10	114.9(3)	C11–C10–C15	120.8(3)
C11–C10–C9	119.1(3)	C15–C10–C9	120.2(3)	C10–C11–C12	120.7(4)
C13–C12–C11	120.0(4)	C12–C13–C14	121.1(4)	C13–C14–C15	118.9(4)
C13–C14–C19	122.5(4)	C15–C14–C19	118.5(4)	C16–C15–C10	122.0(3)
C16–C15–C14	119.6(3)	C10–C15–C14	118.4(3)	C17–C16–C15	119.9(3)
C17–C16–C1	122.1(3)	C15–C16–C1	118.0(3)	C16–C17–C18	120.7(4)
C19–C18–C17	121.1(4)	C18–C19–C14	120.1(4)		

Table 3
Selected bond lengths (Å) and bond angles (°) for compound **3**.

N1–C1	1.379(4)	N1–C12	1.406(4)	N1–C13	1.389(4)
N2–C12	1.284(4)	N2–C14	1.408(4)	O1–C1	1.200(4)
O2–C16	1.363(4)	O2–C19	1.414(4)	C1–C2	1.457(5)
C2–C3	1.345(5)	C2–C7	1.412(5)	C3–C4	1.408(5)
C4–C5	1.337(5)	C5–C6	1.392(5)	C6–C7	1.410(5)
C6–C11	1.400(5)	C7–C8	1.392(5)	C8–C9	1.381(5)
C8–C12	1.430(5)	C9–C10	1.408(5)	C10–C11	1.344(5)
C13–C14	1.358(5)	C13–C18	1.387(5)	C14–C15	1.376(5)
C15–C16	1.367(5)	C16–C17	1.386(5)	C17–C18	1.353(5)
C1–N1–C12	125.4(3)	C1–N1–C13	129.7(3)	C13–N1–C12	104.9(3)
C12–N2–C14	105.2(3)	C16–O2–C19	117.9(3)	O1–C1–N1	120.4(3)
O1–C1–C2	124.2(3)	N1–C1–C2	115.5(3)	C3–C2–C1	118.9(3)
C3–C2–C7	120.5(3)	C7–C2–C1	120.5(3)	C2–C3–C4	120.2(4)
C5–C4–C3	120.5(4)	C4–C5–C6	121.2(4)	C5–C6–C7	119.0(4)
C5–C6–C11	123.0(4)	C11–C6–C7	117.9(4)	C8–C7–C2	121.5(3)
C8–C7–C6	119.9(3)	C6–C7–C2	118.6(3)	C9–C8–C7	120.8(3)
C7–C8–C12	119.1(3)	C9–C8–C12	120.1(3)	C8–C9–C10	118.8(4)
C10–C11–C6	121.9(4)	C11–C10–C9	120.7(4)	N2–C12–N1	113.0(3)
N1–C12–C8	118.0(3)	N2–C12–C8	129.0(3)	C14–C13–C18	121.7(3)
C14–C13–N1	106.6(3)	C18–C13–N1	131.7(3)	C13–C14–C15	122.0(4)
C13–C14–N2	110.3(3)	C15–C14–N2	127.7(3)	C16–C15–C14	116.8(3)
O2–C16–C15	124.0(3)	O2–C16–C17	115.3(3)	C15–C16–C17	120.7(3)
C17–C18–C13	116.0(4)	C18–C17–C16	122.7(4)		

3.2. Spectral analysis

The HRMS of the two compounds were checked by using ES positive ionization mode. In this case, the base peaks were detected as the specie of $[M + H]^+$. Results indicated that compounds **2** and **3** had m/z 301.0984 and 301.0975, respectively. These were consistent with their calculated values of 301.0977, which revealed the correct molecular ion peaks. The representative NMR spectra of compound **2** are shown in Fig. 1a. The peak assignment was assisted by peak integrations in its ^{1}H NMR spectrum and by correlation analysis in its ^{1}H–1H COSY spectrum (Fig. 1a, inset). Its ^{1}H NMR spectrum contains a singlet of 3H at δ 3.94 ppm for CH_3 group. The two doublets at δ 7.04 ppm and 8.16 ppm arise from aromatic H_i and H_c, while the three singlets at δ 7.99 ppm, 8.63 ppm and 8.65 ppm arise from aromatic H_d, H_f and H_a, respectively. The multiplet of 3H at δ 7.70 ppm is assigned to the protons of H_b, H_e and H_h. The 1H singlet at δ 8.01 ppm is attributable to aromatic H_g. The ^{1}H NMR spectrum of compound **3** is also exampled in Fig. 1b. Besides a 3H singlet at δ 3.89 ppm for CH_3 group, the 1H singlet at δ 7.24 ppm is assigned to H_i. The 1H doublets at δ 6.99 ppm, 7.94 ppm, 8.07 ppm and 8.26 ppm are assigned to H_h, H_d, H_c and H_g, respectively. In addition, the 2H quartet at δ 7.63 ppm and the 2H triplet at δ 8.57 ppm arise from H_b and H_e, H_a and H_f, respectively.

3.3. X-ray of the crystal structures

As far as known, the single crystal structural data of the two compounds are still absent. This may be, at least in part, due to the difficulty in culturing their crystals acceptable for X-ray diffraction analysis. Our attempts to culture their single crystals from most commonly-used organic solvents such as methanol, acetone, acetonitrile, tetrahydrofuran, and N,N-dimethylformamide always provided needle-shape or too thin crystals, none of which diffracted well enough to afford their structural evidences. After a long time work, their yellow crystals suitable for X-ray diffraction were obtained by slow evaporation of their 1,1-dichloroethane solutions for several days at room temperature. As listed in Table 1, the two compounds have the same molecular formula and formula weight. In addition, they are also characterized by monoclinic space group with one molecule in the asymmetric unit. Although each of them has 4 molecules per unit cell, the molecular volume of compound **2** is nearly 30% smaller than that of compound **3**. And compound **2** crystallizes in space group P2(1)/*n*, while compound **3** crystallizes in space group P2(1)/*c*. Their crystal structures were given in Fig. 2. Their selected bond lengths and angles were also listed in Tables 2 and 3.

Indicated by X-ray diffraction results, the substitution position of methoxy group doesn't cause obvious influence on their molecular planarity. Both of the two molecular structures are essentially planar as evidenced by the small torsion angles among their different rings. For instance, in compound **2** the bigger torsion between

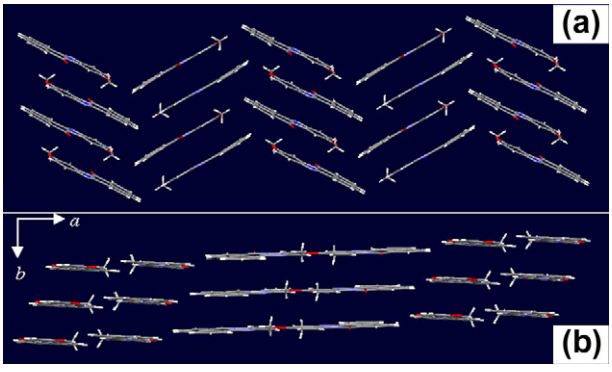

Fig. 3. Stacking arrangements for compounds **2** (a) and **3** (b).

Table 4
Intermolecular H-bonds geometry (Å, °) for compound **2**.

D–H···A	D–H	H···A	D···A	D–H···A
C8–H8B$^{\alpha}$···O1	0.96	2.708	3.547(5)	146.3
C13–H13$^{\beta}$···O2	0.93	2.672	3.225(5)	118.8
C8–H8A$^{\gamma}$···N2	0.96	2.699	3.524(5)	144.4

Table 5
Intermolecular H-bonds geometry (Å, °) for compound **3**.

D–H···A	D–H	H···A	D···A	D–H···A
C5–H5$^{\alpha}$···O1	0.93	2.481	3.372(5)	160.7
C19–H19A$^{\beta}$···N2	0.96	2.662	3.563(5)	156.3

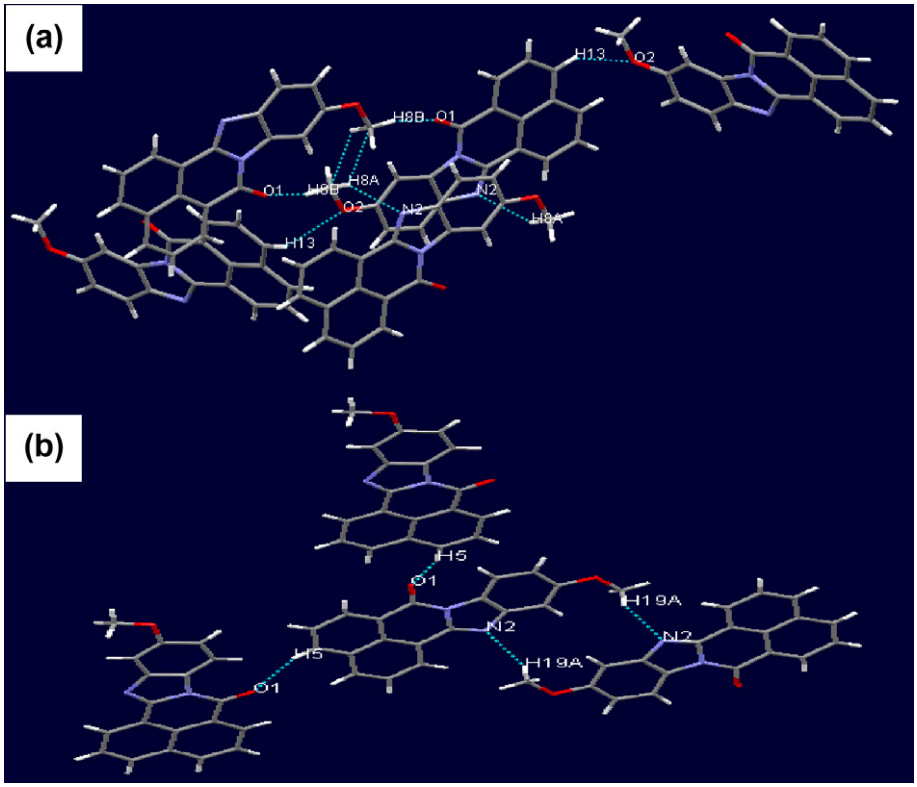

Fig. 4. Intermolecular interactions by non-classic H-bonds for compounds **2** (a) and **3** (b).

rings A and B is only 2.1(3)° (C13–C14–C15–C16). The torsion of 0.9(3)° (C14–C15–C10–C9) between rings B and C is smaller than the torsion of 2.8(3)° (C14–C15–C16–C1) between rings A and C, which shows that ring C has a better co-planarity with ring B. The torsions between rings C and D, rings D and E are 1.3(3)° (C9–N1–C1–N2) and 1.8(3)° (N2–C2–C3–C4), respectively. This indicates that ring D has a little bit better co-planarity with ring C. Also, the carbonyl group is not largely twisted out of ring C as evidenced by the torsion of 3.0(3)° (O1–C9–N1–C1). And another non-ring part of methoxy group is only slightly twisted out of the benzene ring to which it connected, which is indicated by the small torsion of 1.7(3)° (C8–O2–C5–C6). For compound **3**, ring D has a little bit higher co-planarity with ring C, as the same for compound **2**. But opposite to that for compound 2, ring C has a better co-planarity with ring A, not ring B. And the torsion of methoxy group is 1.8(5)° (C19–O2–C16–C15), which is only 0.1° bigger than that in compound **2**. Except this, all the torsions in compound **3**, including torsions concerned to carbonyl group, are slightly smaller than those in compound **2**. This shows that compound **3** have a better molecular planarity than compound **2**. In addition, it's interesting to note that the methoxy group in compound **2** locates in the direction of the carbonyl group, while it departs from the carbonyl group in compound **3**.

The position of the substituted methoxy group has some influences on their bond lengths and bond angles, as listed in Tables 2 and 3. Except bonds C3–C4, C9–C10, N1–C12 and C16–C17, compound **3** has shorter corresponding bond lengths than those for compound **2**. Especially, in compound **3** the bond length of N2–C12 (ring D) is 1.284(4) Å, which is relatively much shorter than the corresponding bond length of 1.307(4) Å (N2–C1) in compound **2**. This leads to the bigger deviation of rings D and E to the direction of carbonyl group, as evidenced by the 2.9° bigger in bond angle of C1–N1–C13 in compound **3** than the corresponding bond angle of C9–N1–C3 in compound **2**. Except this, the two compounds have similar bond angles. As listed in Tables 4 and 5, it exist three kinds of non-classic C–H···O hydrogen bonds for compound **2** and two kinds of non-classic hydrogen bonds for compound **3**, which have the bond lengths of ca. 2.4–2.8 Å. These intermolecular hydrogen bonds take part in stabilizing the molecular structure and stacking. It is worthy to note that one oxygen atom of methoxy group (O2) in compound **3**, not like O2 in compound **2**, does not participate in any intermolecular C–H···O interaction. This difference, to some extent, influences their molecular stacking modes.

Concerned the network stacking of the two compounds, it is observed that there are obvious differences in their π–π stacking interactions (Fig. 3). In the stacking mode of compound **2**, it includes the head-to-tail mode in its *a* axis, which displays in a wave type. In the *b* axis, it is in an anti-parallel π–π stacking. For compound **3**, it linearly stacks in a way of head-to-head mode in its *a* axis, while in the *b* axis it exists in a parallel π–π stacking. These molecular stacking differences are caused by their different intermolecular interactions, as presented in Fig. 4. In Fig. 4a, each molecule of compound **2** forms 6 intermolecular interactions with 4 nearby molecules. The O1 and H8B atoms in compound **2** participate in the formation of intermolecular hydrogen bonds with the nearby $H8B^{\alpha}$ and $O1^{\alpha}$, respectively. Furthermore, both O2 and N2 form the intermolecular interactions with the close $H13^{\beta}$ and $H8A^{\gamma}$, respectively. In addition, its H13 also takes part in forming C13–H13···O2 interaction with the 4rd molecule. However, in Fig. 4b each molecule of compound **3** forms intermolecular interactions only with 3 nearby molecules. Similarly, the N2 and H19A atoms form the intermolecular interactions with $H19A^{\beta}$ and $N2^{\beta}$, respectively. And its O1 and H5 atoms form the interactions with two different molecules nearby. Finally, it's interesting that the oxygen and two hydrogen atoms of methoxy group in compound **2** take part in the formation of intermolecular interactions, whereas in compound **3** only one hydrogen atom takes part in the interaction and the oxygen atom doesn't participate in any intermolecular interactions formation.

4. Conclusions

Two methoxy substituted benzo[*de*]benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-ones were synthesized and characterized by spectroscopic and X-ray diffraction. Based on their X-ray analyses, the methoxy location has no obvious influence on the molecular planarity, but has great influence on their formation modes of intermolecular interaction and molecular stacking. These structural analyses will provide helpful knowledge for these compounds.

Supplementary data

Crystallographic data for the structural analysis are in the Cambridge Crystallographic Data Centre, and CCDC 872808 for 10-methoxy-benzo[*de*]benzo- [4,5]imidazo[2,1-*a*]isoquinolin-7-one (2) and 872809 for 11-methoxy-benzo[*de*]-benzo[4,5]imidazo[2,1-*a*]isoquinolin-7-one (3). Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1FZ, UK (email:deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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