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Synthesis of Several Derivatives of Bicyclic Aziridine

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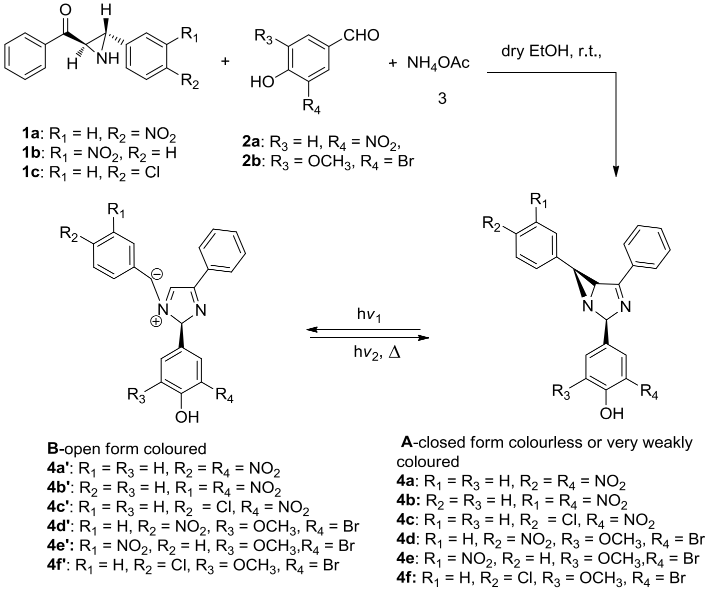
ABSTRACT
As a result of one-pot three-component reaction of ketoaziridines with two aromatic aldehydes in presence of ammonium acetate 1,3-diazabicylo[3.1.0]hex-3-ene derivatives were achieved. These compounds exhibit good photochromic behavior in solution and crystalline phase. The photochromic behavior of compounds 4(a-e) was investigated using absorption spectral changes.

Introduction

Aziridines are valuable and versatile synthetic intermediates for the synthesis of a variety of nitrogen-containing functional compounds.[1] According to photochromism definition, *i.e.*, reversible colour change upon irradiation with UV light and having different absorption spectra,[2] bicyclic aziridines exhibit these properties. Bicyclic aziridines have been established to be very fascinating family of organic photochromic compounds having exclusive photochromic properties which create bistable molecules and undergo photochromism in the crystalline phase. This property allows us to consider them as nominees in the search for intelligent photochromic materials.[3] Of course, photochromic materials that show signs of photochromism in solid state are rare.[4] Typical compounds include bicyclic aziridines, dithienylethenes,[5] Biindenylidenedione,[6] hexaarylbiimidazoles,[7] *N*-salicylidene-anilines,[8] spiropyrans,[9] spirooxazines,[10] dihydroindolizines[11], fulgides[12], and 2-(2'-4'-dinitrobenzyl)pyridine (DNBP).[13] Also some of bicyclic aziridines applied for the detection of Sn^{II} and Sr^{II} in real samples.[14] With regard to the applications of these eye-catching compounds, herein we wish to report synthesis of some derivatives of 1,3-diazabicyclo[3.1.0]hex-3-enes **4(a-f)** (Scheme 1).

Results and Discussion

Bicyclic aziridines **4(a-f)** was synthesized from premade ketoaziridine compound **1(a-c)**[3d,15] and premade two aryl aldehyde (3-bromo-4-hydroxy-5-methoxy benzaldehyde[16] and 4-hydroxy-3-nitrobenzaldehyde[17]) in presence of ammonium acetate **3** (Scheme 1). The stuctures of these compounds were charactrised with spectroscopic methods. The NMR spectra of synthesized compounds in $CDCl_3$ are showed signals at ambient temperature as expected. In the ^1H NMR spectra of purified compounds, protons that can help them to undderstand the formation of these compounds, the protons are in positions 2, 5 and 6. For example, in the ^1H NMR spectrum of **4b**, the H-2, H-5 and H-6 protons appeared as a singlet at δ 6.74, 3.83 and 2.47 ppm, respectively. No proton-proton coupling was observed for these protons, which probably is owing to the dihedral angle between them (It is possible to suggest that, which dihedral angle approximately 90˚, be that as it may, direct assessment of the dihedral angel from the amount of the *J* is indecisive[3d]). The signals of other protons are shown in the figure 1.



Scheme 1 synthesis route of bicyclic aziridines 4a-f and their photochromic reaction

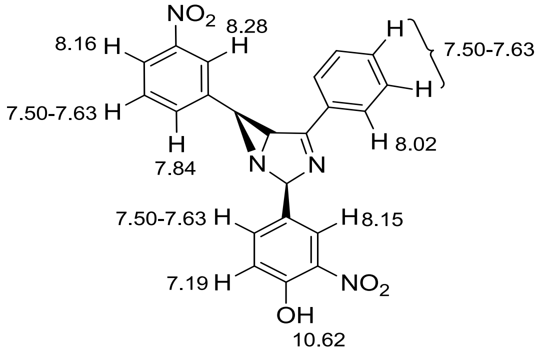


Figure 1. Structure of 4b

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The corresponding signals in the other compounds were appeared in the same region of the ^{1}H NMR spectra. It can be mentioned that formation of *endo*- and *exo*-isomers with orientation of substituent in C-2 position is possible. The ^{1}H NMR spectra of purified **4b** and **4c** are indicated that signals belong to one of the isomers. With consideration of chemical shifts for the H-2, H-5 and H-6 protons, and comparison with those obtained by reported in literature[3], *endo*-isomer is major compound. This is only a suggestion. In the nmr spectra, it is likely that both **A**-closed and **B**-open forms are also observed. In the nmr spectra of compounds **4a** and **4d-e**, two sets of signals were appeared that show two different structures of these compounds are present. These signals can be attributed in two different orientations *exo* or *endo* in C-2 position. If this statement is correct, should have seen two spots in TLC of compounds. Even by changing the solvent was observed only one spot. Similar results were obtained after recrystallization twice. Since the preparation of the samples was carried out in order to recording spectra in the presence of fluorescent room light, two sets of signals can be attributed to both closed and open forms. Of course, this comment is a suggestion that the perviously litrature[3] have been proposed. The photochromic behaviour of synthesized compounds **4(a-f)** are based on a reversible aziridine ring opening, stimulated by UV light, which transform a colourless form (closed ring, **A**) to the coloured form (open ring, **B**) (Scheme 1).3

According to UV/Vis spectra, all target compounds **4a-4f** show photochromic reaction. To search the photochromic behaviour of target compounds, the solution of material synthesized was exposed to 365 nm UV light for a given time at ambient temperature. For example, a solution of **4a** in EtOH is colourless (**4a-A** form). Upon irradiation at 365 nm to this solution, the closed form of **4a-A** was changed to the open form of **4a-B**, go along with by noticeable changes in the absorption spectra as shown in Figure 1.

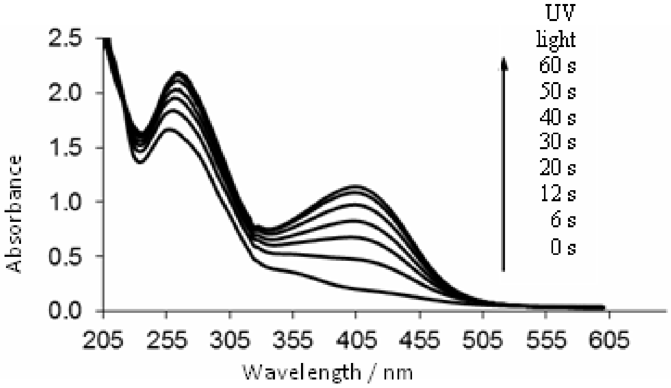

Figure 2. Overlay spectra of 4a in EtOH (2.0 × 10^{-4} M) under 365 nm irradiation at ambient temperature

As shown in Figure 1, irradiation of a solution of **4a** with UV light at 365 nm led to the appearance of new absorption bands at 265 and 418 nm, which indicated that zwitterionic species **4a**-**B** was produced and exhibited photochromic behaviour. As expected, continuous irradiation time resulted in intensity the absorption band in the visible region is increased and in the ultraviolet is decreased. The same results were obtained for the other compounds. Colour change in solid and solution state was observed by eye-naked, when compounds exposed to light (UV light from mercury, xenon lamp, ordinary room light or sunlight). Similar results were also attained when the other synthesized compounds were irradiated with UV light. A study of the photochromic behaviour the solution of synthesized compounds identified that these compounds are sensitive to the position of nitro group and type of substituent on phenyl ring in aziridine moiety. By changing substituent from nitro to chlorine, and also change from *para* to the *meta* position, photochromic sensitivity was decreased and blue shift was observed. It is noteworthy to mention that nitro group at *para* position on phenyl ring in aziridine moiety is much more photochromic behaviour than chlorine group at *para* or nitro at *meta* position. This property attributed to π-conjugation for zwitterionic species, which possess nitro group at *para* position.

Experimental

Melting

Melting points were measured on a Buchi 510 melting point apparatus and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature on a Brucker AVANCE DRX-500 MHz and 400 MHz using $CDCl_3$ or DMSO-d_6 as solvent. FT-IR spectra were recorded on a PerkinElmer RXI spectrometer. UV spectra were recorded using Analytik Jena UV/Vis spectrometer (Specord 205) or Perkin-Elmer Lambda spectrophotometer. Chemicals were obtained from Merck and Fluka. The development of reactions was monitored by thin layer chromatography (TLC) on silica gel 60 GF$_{254}$ aluminium sheets, using ethyl acetate: petroleum ether (1: 3) as mobile phase. The spots were exposed by UV light and iodine vapour.

General procedure for Preparation of 1,3-diazabicyclo[3.1.0]hex-3-enes, (4a-f)

The mixture of ketoaziridine (1 mmol), aldehyde (1 mmol) and ammonium acetate (10 mmol) was stirred at ambient temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the cold reaction mixture was treated with ethanol; solid products was filtered, dried and recrystallized from ethanol or purified by column chromatography to afford the title compounds.

2-Nitro-4-[6-(4-nitrophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-en-2-yl]-phenol (4a): As a yellow solid, yield: 75%, m. p 214-215 °C; IR (KBr, *v*/cm^{-1}): 3230, 3097, 1633, 1540, 1514, 1423, 1348, 1307, 1177, 1047, 895.6, 763.6, 747, 668.1. Closed form, 68%, ^{1}H NMR (400 MHz, CDCl3, ppm): δ 2.45 (s, H), 3.83 (s, 1H), 6.74 (s, H) 7.18 (d, *J* = 8.8 Hz, 1H), 7.44 (*J* = 8.8 Hz, 2H), 7.52-7.63 (m, 3H), 7.83 (dd, *J* = 8.8, 1.6 Hz, 1H), 8.01 (d, *J* = 8.4, 1.6 Hz, 2H), 8.21 (d, *J* = 8.8 Hz, 2H), 8.28 (s, 1H), 10.61 (s, 1H); ^{13}C NMR (100 MHz, CDCl3, ppm): δ 41.7, 58.1, 94.6, 120.5, 123.3, 123.8, 127.3, 128.6, 128.7, 129.0, 131.1, 132.2, 133.3, 136.6, 144.7, 147.5, 154.7, 171.6. After irradiation with UV light converted to blue (open-form, 32%); ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.87 (s, 1H), 3.80 (s, 1H), 6.26 (s, 1H) 7.25 (d, *J* = 8.4 Hz, 1H), 7.52-7.63 (m, 5H), 7.89 (dd, *J* = 8.8, 1.6 Hz, 1H), 8.01 (dd, *J* = 8.4, 1.6 Hz, 2H), 8.27 (d, *J* = 8.8 Hz, 2H), 8.38 (d, *J* = 2 Hz, 1H), 10.66 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$ ppm): δ 47.9, 57.0, 94.6, 120.5,123.9, 127.3, 128.6, 128.7, 129.0, 131.2, 132.2, 133.3, 136.3, 144.7, 147.5, 154.7, 171.6; UV-Vis (EtOH) λ_{max}/nm: 250 before irradiation and 265, 418 after irradiation.

2-Nitro-4-[6-(3-nitrophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-en-2-yl]phenol (4b): As a pale yellow solid (closed form), which after irradiation with UV light converted to deep-yellow (open-form) , yield: 80%, m. p 215-216 °C; IR (KBr, *v*/cm^{-1}): 3200, 3097, 1630, 1608, 1576, 1534.5, 1425, 1349, 1243, 1183, 1026, 776.5, 690.5. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.47 (s, 1H), 3.83 (s, 1H), 6.74 (s, 1H), 7.50-7.63 (m, 5H), 7.19 (d, *J* = 8.8, 1H), 7.84 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.02 (d, *J* = 7.2, 2H), 8.16 (d, *J* = 8.4, 1H), 8.18 (s, 1H), 8.28 (s, 1H), 10.62 (s, 1H).^{13}C NMR (400 MHz, $CDCl_3$, ppm): 41.4, 57.9, 94.5, 120.5, 121.4, 122.7, 123.8, 128.6, 129.0, 129.5, 131.2, 131.3, 132.2, 132.6, 133.3, 136.7, 139.7, 148.5, 154.7, 171.6. UV-Vis (EtOH) λ_{max}/nm: 245 before irradiation and 372 after irradiation.

4-[6-(4-Chlorophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-en-2-yl]-2-nitrophenol (4c):

As a pale yellow solid (closed form), which after irradiation with UV light converted to dark-yellow (open-form), yield: 70%, m. p 197-198 ºC; IR (KBr, v/cm^{-1}): 3200, 3097, 1629, 1607, 1577, 1531.8, 1495.7, 1421.2, 1325.2, 1242, 1178.2, 1049, 1025.3, 806, 787.9, 537.4. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.37 (s, 1H), 3.77 (s, 1H), 6.71 (s, 1H), 7.18 (t, *J* = 6.8 Hz 3H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.54-7.61 (m, 3H), 7.83 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.01 (d, *J* = 7.2 Hz, 2H), 8.29 (s, 1H), 10.62 (s, 1H). ^{13}C NMR (400 MHz, $CDCl_3$, ppm): 42.0, 57.7, 94.5, 120.3, 123.9, 127.8, 128.6, 128.7, 128.9, 131.3, 131.6, 132.1, 133.3, 133.7, 135.8, 136.8, 154.6, 171.9. UV-vis (EtOH) λ_{max}/nm: 240, 365 before irradiation and 260, 365 after irradiation.

2-Bromo-3-methoxy-5-[6-(4-nitro-phenyl)-4-phenyl-1,3-diaza-bicyclo[3.1.0]hex-3-en-2-yl]phenol (4d): As a white solid, yield: 80%, m. p 187-188 ºC; IR (KBr, v/cm^{-1}): 3200, 3097, 1603, 1578, 1515, 1449, 1346, 1279, 1026, 1051, 770, 692.7. Closed form, 72%, ^{1}H NMR (400 MHz, $CDCl_3$, ppm) colorless form: δ 2.51 (s, 1H), 3.80 (s, 1H), 3.85 (s, 3H), 5.97 (s, 1H), 6.72 (s, 1H), 7.04 (s, 1H), 7.26 (m, 1H), 7.44 (dt, *J* = 8.8, 2.4 Hz, 2H), 7.51-7.61 (m, 3H), 8.00 (dt, *J* = 6.8, 1.6 Hz, 2H), 8.21 (dt, *J* = 8.8, 2.0 Hz, 2H). ^{13}C NMR (400 MHz, $CDCl_3$, ppm): 41.7, 56.5, 56.8, 95.2, 108.2, 109.2, 123.6, 123.8, 127.3, 128.6, 128.9, 131.4, 132.1, 142.7, 145.2, 147.2, 147.4, 170.9. After irradiation with UV light converted to blue (open-form, 28%), ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.87 (s, 1H) 3.80 (s, 1H), 3.98 (s, 3H), 6.05 (s, 1H), 6.20 (d, *J* = 2.4 Hz, 1H), 7.07 (s, 1H), 7.34 (d, *J* = 1.6 Hz, 1H), 7.51-7.61 (m, 5H), 8.00 (dt, *J* = 6.8, 1.6 Hz, 2H), 8.26 (dt, *J* = 8.8, 2.0 Hz, 2H). ^{13}C NMR (400 MHz, $CDCl_3$, ppm): 48.1, 56.4, 56.9, 95.2, 108.8, 109.2, 122.9, 123.8, 127.3, 128.8, 129.0, 131.3, 132.1, 142.7, 145.2, 147.2, 147.6, 170.9. UV-Vis (EtOH) λ_{max}/nm: 265 before irradiation and 255, 291, 420 after irradiation.

2-Bromo-6-methoxy-5-[6-(3-nitrophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-en-2-yl]phenol (4e): As a white solid, yield: 75%, m. p 207-208 ºC; IR (KBr, v/cm^{-1}): 3029, 1602, 1576, 1521, 1498, 1414, 1348, 1285, 1142, 1047, 819.4, 735.7, 698.5. Closed form, 33%, ^{1}H NMR (400 MHz, $CDCl_3$, ppm): colorless form: δ 2.55 (s, 1H), 3.74 (s, 1H), 3.87 (s, 3H), 5.94 (s, 1H), 6.73 (s, 1H), 7.08 (s, 1H), 7.28 (s, 1H), 7.52-7.60 (m, 4H), 7.75 (d, *J* = 7.6 Hz, 1H),

8.04 (d, *J* = 8.2 Hz, 2H), 8.21 (d, *J* = 8.0 Hz, 1H), 8.27 (s, 1H). After irradiation with UV light converted to green (open-form), ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.86 (s, 1H), 3.83 (s, 1H), 3.99 (s, 3H), 6.00 (s, 1H), 6.21 (s, 1H), 7.11 (s, 1H), 7.34 (s, 1H), 7.52-7.60 (m, 5H), 8.04 (d, *J* = 8.2 Hz, 2H), 8.16 (d, *J* = 6.4 Hz, 1H), 8.17 (s, 1H); UV-Vis (EtOH) λ_{max}/nm: 261 before irradiation and 271, 290 (shoulder), 370 after irradiation.

2-Bromo-4-[6-(4-chlorophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-en-2-yl]-6-methoxyphenol (4f): As a white solid, yield: 75%, m. p 158-159 ºC; IR (KBr, v/cm^{-1}): 3200, 3097, 2933, 1608, 1576, 1496, 1448, 1417, 1346, 1279, 1048, 1014.2, 879.6, 768.7, 695. Closed form, 64%, ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.44 (s, 1H), 3.76 (s, 1H), 3.84 (s, 1H), 5.96 (s, 1H), 6.69 (s, 1H), 7.06 (s, 1H), 7.20 (d, *J* = 8.4 Hz, 2H), 7.31-7.38 (m, 3H), 7.50-7.58 (m, 3H), 8.01 (d, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 42.0, 56.4, 57.3, 95.2, 108.1, 109.3, 123.6, 127.8, 128.6, 128.7, 128.9, 131.7, 131.8, 133.5, 134.0, 136.2, 142.6, 147.2, 171.2. After irradiation with UV light turns to pinkish (open-form, 36%) ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 2.72 (s, 1H), 3.74 (s, 1H), 3.97 (s, 3H), 6.02 (s, 1H), 6.16 (s, 1H), 7.09 (s, 1H), 7.31-7.38 (m, 5H), 7.50-7.58 (m, 3H), 8.01 (d, *J* = 2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 48.5, 56.4, 56.6, 96.7, 108.4, 108.8, 123.0, 127.6, 128.5, 128.6, 128.9, 131.4, 131.6, 131.8, 133.7, 136.4, 142.9, 147.2, 170.5; UV-Vis (EtOH) λ_{max}/nm: 231, 290 (Shoulder) before irradiation and 306, 374 after irradiation.

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