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1-(2′-Benzimidazolylmethyl)-pyridinium ylide in the one-pot synthesis of indolizine and benzimidazo[1,2-*a*]pyridine derivatives

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Abstract: Four indolizine derivatives were obtained *via* 1,3-dipolar cycloaddition reaction of 1-(2′-benzimidazolylmethyl)pyridinium ylide with various electron-deficient alkynes. The reaction of this pyridinium *N*-ylide with dimethyl maleate gave an unexpected methyl 1-oxobenzimidazo[1,2-*a*]pyridine-3-carboxylate. The structures of all reported compounds have been examined by X-ray crystallography and NMR spectroscopy.

Keywords: 1,3-dipolar cycloaddition; crystal structure; indolizine; pyridinium *N*-ylide.

1 Introduction

N-Phenacylpyridinium salts have found applications as reagents in the organic synthesis of diverse classes of substances [1–3]. Their heteroanalogues, with a heterocyclic ring such as pyridine or pyrazine instead of a phenacyl group, have been used successfully to extend the classical synthesis of indolizine derivatives [4, 5]. The use of these salts offers the advantage of removing a few reaction steps to reach the target molecule and a simple workup. Furthermore, it has recently been reconsidered as a one-pot multicomponent process [6, 7].

Previously, we have reported that bicyclic mesoionic thiazolo[3,2-*a*]imidazole derivatives can be formed by a reaction involving carbon disulfide and imidazolium *N*-ylide generated in situ from *N*-phenacyl-2-bromoimidazolium salts, in the presence of Et_3N [8].

In continuation of our research interest in heteroaromatic *N*-ylides involving multicomponent reactions, we report here our results concerning the reactivity of (2′-benzimidazolylmethyl)pyridinium ylide towards both dimethyl maleate and some alkynes as dipolarophiles. Pyridone and indolizine derivatives containing a benzimidazole unit were achieved as a result of these reactions.

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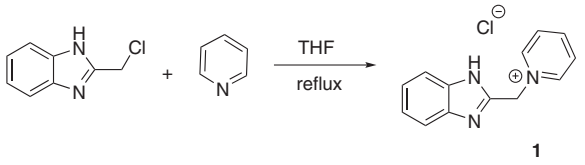
2 Results and discussion

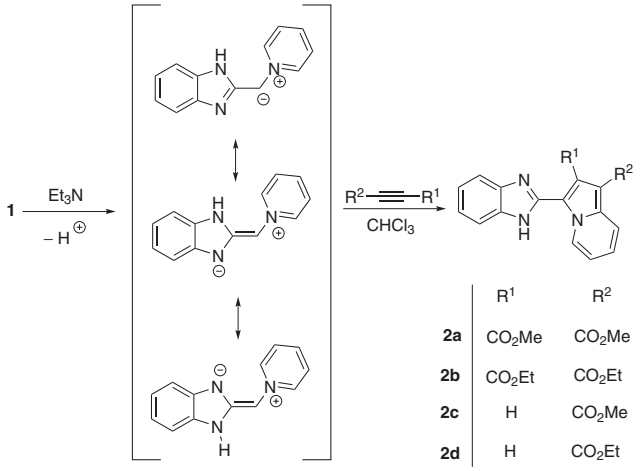
In a preliminary exploratory study, a mixture of 1-[(1*H*-benzo[*d*]imidazol-2-yl)methyl] pyridinium chloride **1** (Scheme 1), which was prepared from the reaction of pyridine with 2-(chloromethyl)-1*H*-benzo[*d*]imidazole as described previously [9–11], and dimethyl acetylenedicarboxylate (DMAD) was stirred at ambient temperature using chloroform as solvent and in the presence of triethylamine for 24 h (Scheme 2).

After general workup, the obtained compound was fully characterized by ^1H, ^{13}C NMR, and mass spectra. The formation of indolizine **2a** was further confirmed by crystal structure determination (Fig. 1). Compound **2a** comes from the dehydrogenation of the initially formed cycloadduct in air. The intermediates, dihydroindolizines and tetrahydroindolizine, were not stable enough to be isolated.

This result clearly showed that, due to the electron withdrawing ability of the pyridinium moiety, the



Scheme 1: Preparation of 1-[(1*H*-benzo[*d*]imidazol-2-yl)methyl] pyridinium chloride **1**.



Scheme 2: Formation of indolizines derivatives **2a-d**.

methylene is acidic, and a 1,3-dipolar cycloaddition of the in situ generated ylide with DMAD took place. The favorable effect of the benzimidazole substituent on the intermediate carbanion stability was confirmed by comparison with a phenyl substituent. The presence of the benzimidazole group is essential, since no adducts were reported in the literature using *N*-benzylpyridinium chloride.

To further extend the utility of this reaction, the reactivity of other activated dipolarophiles was explored using the same procedure. Four functionalized indolizines were successfully prepared in 30–45 % yields, and their structures were confirmed by X-ray crystallography and NMR spectroscopy (Scheme 2, Fig. 1, Tables 1 and 2).

It should be mentioned that the cyclization reactions of **1** proceed to yield **2c** and **2d** instead of the others possible regioisomers. The position of the ester group at the indolizine unit in compounds **2c** and **2d** was confirmed by the structure determinations in the solid state by X-ray diffraction (Fig. 1). It could be noted that the formation of an indolizine was not observed when pyridinium *N*-ylide was stirred with other alkynes, such as phenylacetylene, propyne, pentyne, or hexyne.

In the following, the crystal and molecular structure of **2a** shall be described in more detail (Tables 3–5). Information on the other solid-state structures may be taken from the deposited crystallographic CIF files (see below). Compound **2a** crystallizes in the monoclinic crystal system (space group $P2_1/a$ with $Z = 4$). The indolizine ring is close to planarity with a maximum deviation of 0.023(1) Å at atom C(3). The benzimidazole ring is almost planar, with a maximum deviation of 0.031(1) Å at C(19) and forms a dihedral angle with the indolizine ring of 29.88(2)°. The carbonyl group (C(7)/O(3)–O(4)), attached to C(6), forms a dihedral angle of 25.83(7)° with the plane defined by the atoms N(1), C(5)–C(6), C(11)–C(12), while the second carbonyl group (C(10)/O(1)–O(2)) attached to the C(11) atom is rotated by 47.94(5)° out of the same plane. Bond lengths and angles are in the expected range [12, 13]. The C–N bonds (N(1)–C(1) 1.382(1), N(1)–C(5) 1.393(1), N(1)–C(12) 1.392(1) Å) in compound **2a** indicate partial double bond character (Table 3, Fig. 2). The packing of the molecule **2a** in the crystal can be described as alternating layers parallel to the $(30\bar{1})$ plane. It is stabilized by N–H···O hydrogen bonding interactions (Table 4, Fig. 2). The carbomethoxy methyl group forms a C–H···π contact to both imidazole and benzene rings of an adjacent benzimidazole unit. In addition, the interaction C(18)–H(18)···π with an approximately 3.513(1) Å distance has also been observed (Table 5). The packing is consolidated by slipped π–π stacking with a centroid-to-centroid distance of 3.593(1) between pyrrole and pyridine rings of indolizine with symmetry operation $\frac{1}{2} + x, \frac{1}{2} - y, z$.

Next, we tried the utility of pyridinium *N*-ylide for the construction of other heterocyclic moieties. We decided to test this 1,3-dipolar addition reaction in the presence of an alkene as dipolarophile. In this regard, *N*-pyridinium salt **1** was treated with dimethyl maleate in the presence of triethylamine as base using chloroform as solvent (Scheme 3).

Surprisingly, and in contrast to the foregoing reaction results, when the pyridinium *N*-ylide was treated with dimethyl maleate, a polycyclic product **3** was generated in about 25 % of yield. Compound **3** was the main product which could be easily isolated and identified. The structure of **3** was unambiguously confirmed by a crystal structure determination (Fig. 3, Table 2).

Although the exact mechanism of this reaction is not very clear, a plausible reaction course of pyridinium ylide with dimethyl maleate is proposed in Scheme 4. The 1-(1-(1*H*-benzo [*d*]imidazol-2-yl)-4-methoxy-2-(methoxycarbonyl)-4-oxobutyl)pyridinium chloride **A** was formed by a Michael addition reaction of pyridinium ylide to the dimethyl maleate. This intermediate undergoes an elimination reaction to generate the dimethyl 2-((1*H*-benzo[*d*]imidazol-2-yl)methylene)succinate **B**. This latter could be deprotonated by Et_3N giving, *via* a

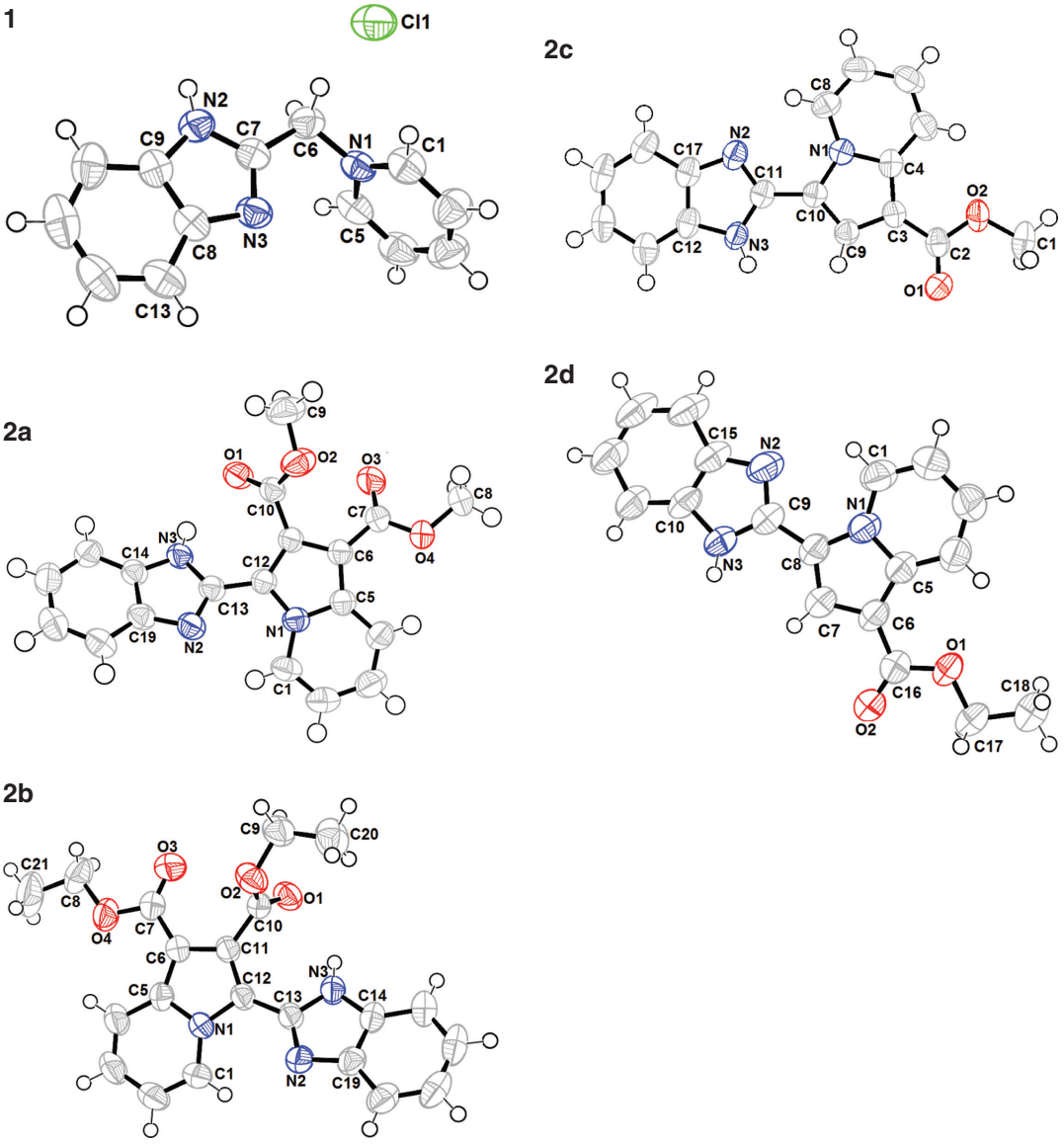


Fig. 1: Oak ridge thermal ellipsoid plots (ORTEP) of the molecular structures of **1**, **2a**, **2b**, **2c**, **2d** in the crystal and atom numbering scheme adopted (displacement ellipsoids at the 50 % probability level; H atoms with arbitrary radii; blue: nitrogen, red: oxygen, yellow: chloride).

six-membered ring *N*-cyclization process, the corresponding 1-oxo-benzimidazo[1,2-*a*]pyridine derivative **3**.

3 Conclusion

In conclusion, the 1,3-dipolar cycloaddition reactions of 1-(2′-benzimidazolylmethyl)pyridinium ylide with various electron-deficient alkynes and an alkene as dipolarophiles were investigated. Thus, the corresponding substituted indolizine derivatives were obtained in the presence of activated alkynes. Unexpectedly, the reaction of pyridinium *N*-ylide with dimethyl maleate gave an unexpected methyl 1-oxo-benzimidazo[1,2-*a*]pyridine-3-carboxylate. Further investigations of these reactions using others reagents were carried out in our laboratory.

4 Experimental section

Melting points were determined on an Electrothermal Digital Melting Point Apparatus (IA9200, Electrothermal, Stone, Staffordshire, UK) and are uncorrected. ^{1}H NMR and ^{13}C NMR spectra were recorded on a BRUKER 250 DXP (Bruker Instruments, Germany) and VARIAN Mercury 300 spectrometers (Varian Medical Systems, Palo Alto, CA,

Table 1: Crystal structure data for **1**, **2a**, **2b**.

	1	**2a**	**2b**
Formula	$C_{13}H_{12}ClN_3$	$C_{19}H_{15}N_3O_4$	$C_{21}H_{19}N_3O_4$
M_r	245.71	349.34	377.39
Crystal size, mm³	0.15 × 0.09 × 0.012	0.2 × 0.13 × 0.09	0.2 × 0.15 × 0.11
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/a$	$P2_1/c$
a, Å	9.6358(6)	7.3528(17)	7.4268(2)
b, Å	9.6452(5)	17.486(4)	22.2753(6)
c, Å	13.1453(7)	12.406(3)	11.2956(3)
β, deg	91.813(2)	94.332(11)	94.9470(10)
V, Å³	1221.10(12)	1590.5(6)	1861.72(9)
Z	4	4	4
D_{calcd}, g cm^{-3}	1.337	1.459	1.346
μ(MoK_α), cm^{-1}	0.293	0.105	0.095
F(000), e	512	728	792
hkl range	$-11 \le h \le +11$	$-11 \le h \le +11$	$-9 \le h \le +9$
	$-9 \le k \le +11$	$-26 \le k \le +26$	$-29 \le k \le +29$
	$-16 \le l \le +15$	$-18 \le l \le +14$	$-11 \le l \le +14$
$((\sin\theta)/\lambda)_{max}$, Å^{-1}	0.618	0.754	0.657
Refl. measured	9207	39412	17988
Refl. unique/R_{int}	2413/0.0519	5667/0.0292	4425/0.0490
Param. refined	154	237	255
$R(F)/wR(F^2)$ ($I > 2\sigma(I)$)	0.0430/0.0853	0.0418/0.1099	0.0482/0.1052
$R(F)/wR(F^2)$ (all data)	0.0878/0.1016	0.0576/0.1204	0.0966/0.1250
Goodness-of-fit on F^2	1.000	1.029	1.012
$\Delta\rho_{fin}$ (max/min), e Å^{-3}	0.199/−0.235	0.456/−0.273	0.208/−0.199

Table 2: Crystal structure data for **2c**, **2d**, **3**.

	2c	**2d**	**3**
Formula	$C_{17}H_{13}N_3O_2$	$C_{18}H_{15}N_3O_2$	$C_{13}H_{10}N_2O_3$
M_r	291.3	305.33	242.23
Crystal size, mm³	0.19 × 0.14 × 0.1	0.22 × 0.17 × 0.09	0.14 × 0.1 × 0.08
Crystal system	Monoclinic	Triclinic	Orthorhombic
Space group	$P2_1/n$	$P\bar{1}$	$Pca2_1$
a, Å	6.7660(17)	6.9876(4)	25.034(7)
b, Å	16.097(4)	9.6542(6)	5.7033(15)
c, Å	12.805(3)	12.0140(7)	7.7384(16)
α, deg	90	99.948(5)	90
β, deg	99.752(11)	100.096(3)	90
γ, deg	90	105.380(4)	90
V, Å³	1374.4(6)	748.53(8)	1104.9(5)
Z	4	2	4
D_{calcd}, g cm^{-3}	1.408	1.355	1.456
μ(MoK_α), cm^{-1}	0.095	0.091	0.106
F(000), e	608	320	504
hkl range	$-9 \le h \le +8$	$-8 \le h \le +8$	$-32 \le h \le +32$
	$-21 \le k \le +17$	$-11 \le k \le +11$	$-7 \le k \le +4$
	$-17 \le l \le +17$	$-14 \le l \le +15$	$-7 \le l \le +10$
$((\sin\theta)/\lambda)_{max}$, Å^{-1}	0.688	0.630	0.668
Refl. measured	14618	8928	5778
Refl. unique/R_{int}	3724/0.0628	2915/0.0432	2224/0.0347
Param. refined	200	210	164
$R(F)/wR(F^2)$ ($I > 2\sigma(I)$)	0.0405/0.0896	0.0499/0.1134	0.0391/0.0856
$R(F)/wR(F^2)$ (all data)	0.0939/0.1192	0.1187/0.1426	0.0528/0.0919
Goodness-of-fit on F^2	0.879	0.982	1.033
$\Delta\rho_{fin}$ (max/min), e Å^{-3}	0.251/−0.285	0.201/−0.153	0.235/−0.180

Table 3: Selected bond lengths (Å), angles (deg), and dihedral angles (deg) for **2a** with estimated standard deviations in parentheses.

	2a
Distances	
N(1)–C(1)	1.3818(12)
N(1)–C(5)	1.3933(13)
N(1)–C(12)	1.3917(12)
Angles	
C(12)–N(1)–C(5)	109.60(8)
C(1)–N(1)–C(5)	121.62(8)
N(1)–C(12)–C(11)	106.81(8)
N(1)–C(5)–C(6)	107.36(8)

USA). Chemical shifts (δ values) are expressed in ppm. High-resolution mass spectra were recorded on an LC-MS instrument (Agilent 1200 LC-Agilent 6500 Accurate Mass) and were carried out at the Instituto de Química Orgánica General (Spanish National Research Council, CSIC, Madrid, Spain). Thin layer chromatography (TLC) was carried out on precoated Merck silica gel aluminum sheets 60 F_{254}.

4.1 General reaction procedure for the synthesis of indolizines 2

A suspension of ylide (1.0 mmol) and alkyne (1.1 mmol) in chloroform was stirred at 0 °C. Triethylamine (1.3 mmol) was added dropwise, and the mixture was stirred at room temperature for 24 h. The solution was evaporated to dryness under reduced pressure, and the brown residue was chromatographed on a silica gel column.

4.2 Dimethyl 3-(1*H*-benzo[*d*]imidazol-2-yl) indolizine-1,2-dicarboxylate (2a)

Yield: 32 %; yellow solid; m.p. 189 °C. – ^{1}H NMR ([D_5]pyridine, 250 MHz): δ = 13.12 (br, 1H), 10.03 (td, 1H, *J* = 7.1, 7.1,

Table 4: Distances (Å) and angles (deg) of intra- and intermolecular hydrogen bonds for **2a**.

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D–A)	∠(D–H···A)	Symm. op.
N(3)–H(3N)···O(1)	0.86	2.24	2.9612(13)	142	–*x*, –*y* + 1, –*z*
N(3)–H(3N)···O(1)	0.86	2.15	2.8149(14)	134	*x*, *y*, *z*

Table 5: Intermolecular interactions C–H···Cg (C–H···π; Å, deg) operating in the crystal structure of **2a**.

C–H···Cg	*d*(C–H)	*d*(H···Cg)	*d*(C–Cg)	∠(D–H···Cg)	Symm. op.
C(9)–H(9B)···Cg1 (N(2)/C(13)/N(3)/C(14)/C(19))	0.96	2.76	3.3609(15)	121	–*x*, –*y* + 2, –*z* + 2
C(9)–H(9B)···Cg2 (C(14)–C(18))	0.96	2.96	3.8876(16)	164	–*x*, –*y* + 2, –*z* + 2
C(18)–H(18)···Cg2 (C(14)–C(18))	0.93	2.75	3.5131(14)	140	*x* – 3/2, –*y* – 1/2, *z*

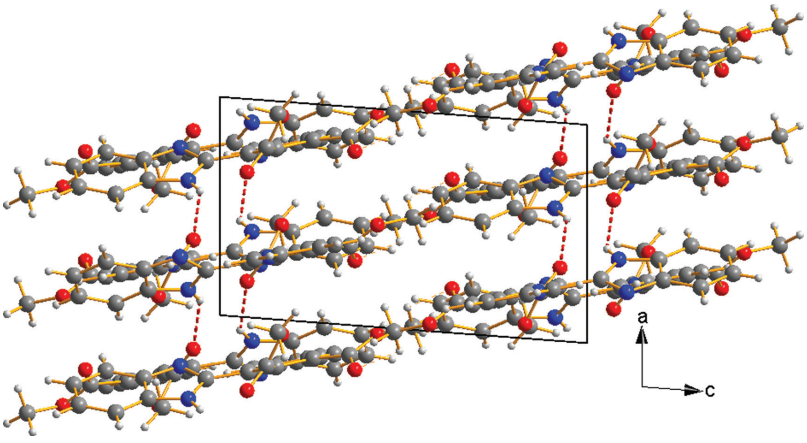
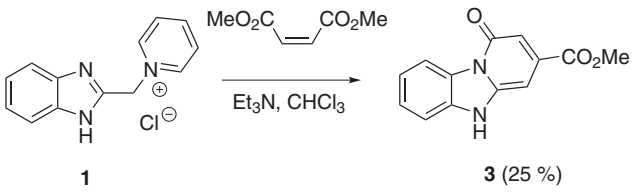


Fig. 2: View of the crystal structure of **2a** in a projection along [010] showing the formation of layers parallel to the (30$\bar{1}$) plane of **2a**. Hydrogen bonds are shown as red dashed lines (N–H···O) connecting these layers.



Scheme 3: Formation of 1-oxo-benzimidazo[1,2-*a*] pyridine-3-carboxylate.

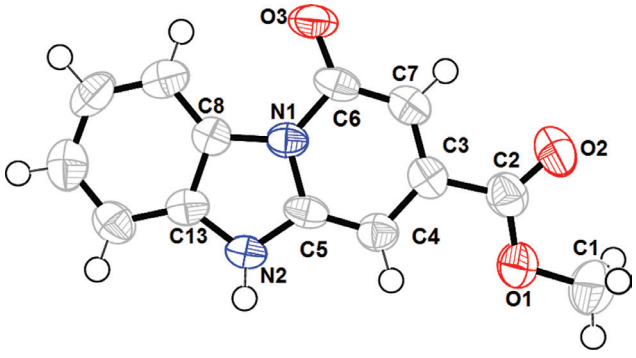
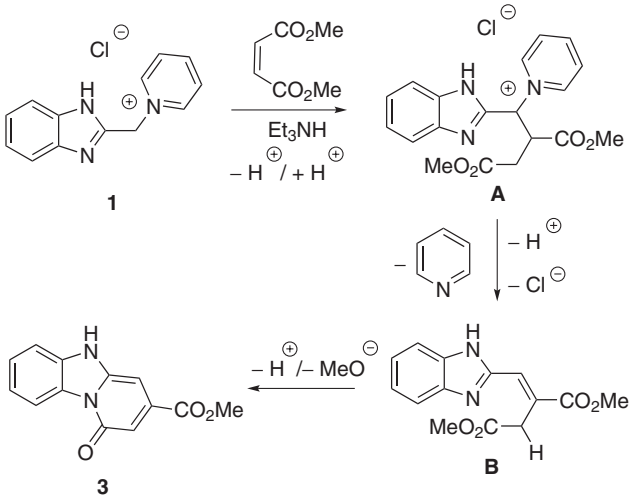


Fig. 3: ORTEP plot of the molecular structure of **3** in the crystal and atom numbering scheme adopted (displacement ellipsoids at the 50 % probability level; H atoms with arbitrary radii).



Scheme 4: Proposed mechanism for the formation of **3**.

1.0 Hz), 8.26 (td, 1H, *J* = 9.0, 1.1 Hz), 7.86–8.02 (m, 2H), 7.45–7.37 (m, 2H), 7.21–7.15 (m, 1H), 6.88 (td, 1H, *J* = 7.0, 1.3 Hz), 3.88 (s, 6H). – ^{13}C NMR ([D$_5$]pyridine, 62.9 MHz): δ = 167.9, 164.3, 144.2, 136.9, 136.2, 128.3, 126.0, 124.1, 123.7, 120.2, 116.2, 114.9, 104.3, 53.3, 51.8. – HRMS (ESI): *m*/*z* = 350.1138 (calcd. 350.1135 for $C_{19}H_{16}N_3O_4$, [M+H]$^+$).

4.3 Diethyl 3-(1*H*-benzo[*d*]imidazol-2-yl) indolizine-1,2-dicarboxylate (2b)

Yield: 30 %; yellow solid; m.p. 161 °C. – ^{1}H NMR ([D$_6$]DMSO, 250 MHz): δ = 12.32 (br, 1H), 9.42 (d, 1H, *J* = 7.1 Hz), 8.23 (d, 1H, *J* = 10.9 Hz), 7.74–7.70 (m, 2H), 7.42 (td, 1H, *J* = 6.8, 0.8 Hz), 7.32–7.28 (m, 2H), 7.14 (td, 1H, *J* = 6.9, 1.1 Hz), 4.43–4.22 (m, 4H), 1.33 (t, 3H, *J* = 7.0 Hz), 1.24 (t, 3H, *J* = 7.1 Hz). – ^{13}C NMR ([D$_6$]DMSO, 62.9 MHz): δ = 165.0, 162.7, 142.2, 139.6, 135.6, 126.9, 125.9, 123.5, 122.6, 119.0, 115.3, 114.9, 114.8, 102.3, 61.6, 60.0, 14.3, 13.8. – HRMS (ESI): *m*/*z* = 378.1454 (calcd. 378.1454 for $C_{21}H_{20}N_3O_4$, [M+H]$^+$).

4.4 Methyl 3-(1*H*-benzo[*d*]imidazol-2-yl) indolizine-1-carboxylate (2c)

Yield: 45 %; white solid; m.p. 268 °C. – ^{1}H NMR ([D$_6$]DMSO, 300 MHz): δ = 10.23 (d, 1H, *J* = 7.2 Hz), 8.23 (d, 1H, *J* = 8.7 Hz), 8.17 (s, 1H), 7.72–7.69 (m, 1H), 7.52–7.50 (m, 1H), 7.42 (td, 1H, *J* = 9.3, 0.9 Hz), 7.24–7.19 (m, 3H), 3.85 (s, 3H). – ^{13}C NMR ([D$_6$]DMSO, 75 MHz): δ = 164.5, 145.9, 144.0, 137.5, 134.3, 128.5, 125.6, 123.3, 122.3, 119.4, 119.1, 118.4, 116.2, 114.9, 111.4, 104.4, 51.7. – HRMS (ESI): *m*/*z* = 293.1105 (calcd. 293.1111 for $C_{17}H_{14}N_3O_2$, [M+H]$^+$).

4.5 Ethyl 3-(1*H*-benzo[*d*]imidazol-2-yl) indolizine-1-carboxylate (2d)

Yield: 45 %; white solid; m.p. 165 °C. – ^{1}H NMR ([D$_6$]DMSO, 250 MHz): δ = 12.96 (br, 1H), 10.25 (d, 1H, *J* = 7.0 Hz), 8.24 (d, 1H, *J* = 8.9 Hz), 8.16 (s, 1H), 7.74–7.71 (m, 1H), 7.54–7.51 (m, 1H), 7.40 (td, 1H, *J* = 9.8, 0.9 Hz), 7.25–7.18 (m, 3H), 4.32 (q, 2H, *J* = 7.1 Hz), 1.35 (t, 3H, *J* = 4.0 Hz). – ^{13}C NMR ([D$_6$] DMSO, 62.5 MHz): δ = 163.5, 145.3, 143.4, 136.9, 133.7, 127.9, 124.8, 122.7, 121.6, 118.7, 118.5, 117.7, 115.5, 114.2, 110.8, 104.0, 59.5, 14.2. – HRMS (ESI): *m*/*z* = 306.1237 (calcd. 306.1243 for $C_{18}H_{16}N_3O_2$, [M+H]$^+$).

4.6 Methyl 1-oxo-benzimidazo[1,2-*a*]pyridine-3-carboxylate (3)

Yield: 25 %; white solid; m.p. 192 °C. – ^{1}H NMR ([D$_6$]DMSO, 300 MHz): δ = 12.94 (br, 1H), 8.87 (d, 1H, *J* = 13.7 Hz), 7.67–7.55 (m, 2H), 7.38 (td, 1H, *J* = 8.3, 1.7 Hz), 6.67 (d, 1H, *J* = 1.6 Hz), 6.43 (d, 1H, *J* = 1.5 Hz), 3.93 (s, 3H). – ^{13}C NMR ([D$_6$] DMSO, 75 MHz): δ = 166.1, 159.1, 144.8, 139.4, 132.5, 127.8, 127.1, 121.7, 116.9, 111.0, 101.9, 83.7, 53.0, 114. – HRMS (ESI): *m*/*z* = 243.0760 (calcd. 243.0764 for $C_{13}H_{11}N_2O_3$, [M+H]$^+$).

4.7 X-ray structure determinations

X-ray data were collected with a Bruker Apex II CCD area detector diffractometer with graphite-monochromated MoK_α radiation (λ = 0.71073 Å) at 298 K. Tables 1 and 2 summarize important crystal structure data. Figures 1–3 show views of the molecular and crystal structures. Additional details of the structure determinations may be taken from the crystallographic CIF files (see the next paragraph).

CCDC 1041006, 1007307, 1041347, 1007303, 1041920, and 1007302 contain the supplementary crystallographic data for compounds **1**, **2a**, **2b**, **2c**, **2d**, and **3**, respectively. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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Graphical synopsis

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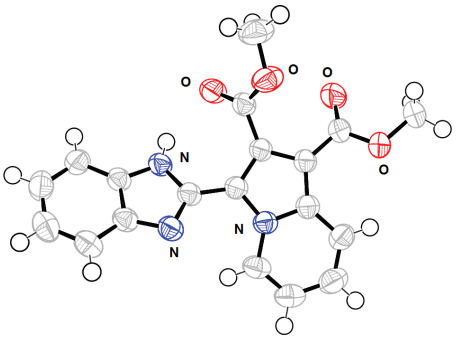
1-(2′-Benzimidazolylmethyl)-pyridinium ylide in the one-pot synthesis of indolizine and benzimidazo[1,2-*a*] pyridine derivatives



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