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Solvent-free Promoted One-pot Synthesis of ٤H-quinolizine, pyrido[٢،١-a]isoquinoline and pyrido[١،٢-a] quinoline Derivatives

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Abstract

This work describes a fast, mild, convenient and simple method for preparing of nitrogen heterocyclic derivatives by MCR reaction under solvent-free condition.

Keywords: *Solvent free reaction, Multi-component reactions, Acetylenic esters, ٤H-quinolizine, ٤H-pyrido[٢،١-a]isoquinoline, ٤aH-pyrido[١،٢-a]quinoline.*

Introduction

Quinolizines are of considerable interest due to their widespread occurrence in natural products, particularly in the field of alkaloids [١]. The importance of these nitrogen heterocyclic derivatives to the pharmaceutical industry has spurred a great amount of research, and numerous methods have been devised for their construction [٢]. Although many routes to the basic ring systems are known, new general synthetic approaches are still highly desirable [٣].

The possibility of performing chemical reactions in the absence of solvent has been receiving more attention now-a-days [٤-٨]. The examples reported [٩-١٤], demonstrate that solvent-free reactions are generally faster giving higher selectivities and excellent yields. A large variety of nitrogen heterocycles are known to form zwitterionic species on addition of activated olefins or acetylenes. Pyridine deserves special mention owing to the variety of transformations that it mediates. The earliest work in the area was reported by Diels and Alder, and their study [١٥] and subsequently the structure elucidation of Acheson [١٦-٢٠]

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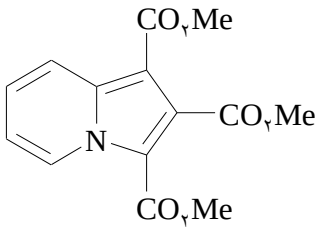
showed that pyridine reacts smoothly with dimethyl acetylenedicarboxylate (DMAD) to form indolizine-۱،۲،۳-tricarboxylate **۱** and ۴*H*-quinolizine **۲** in methanol as a solvent (Scheme ۱) [۲۱].



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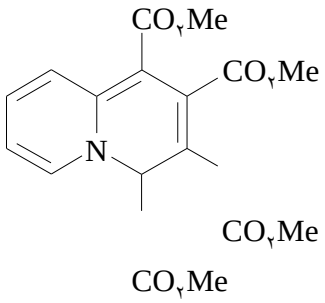

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Scheme ۱

However, the above method suffers from drawbacks such as longer reaction time, the need for unfriendly solvent, and moderate yield. Recently H. Valizadeh and *et.all* have reported an addition reaction of Nitrogen-containing heterocyclic compounds with DMAD under neat condition [۲۲]. Following, as part of our ongoing research program on the development of new protocols in heterocylic synthesis [۲۳-۲۶], herein, we applied this methodology to describe the synthesis of ۴*H*-pyrido[۲،۱-a]isoquinoline, ۴H-quinolizine, and ۴*aH*-pyrido[۱،۲-a] quinoline derived from the reaction between diethyl acetylenedicarboxylate, di-*tert*-butyl acetylenedicarboxylate and isoquinoline, pyridine, and quinoline under the same reaction conditions.

Experimental

General

Chemicals were purchased from Fluka and used without further purification. Melting points were measured on an Electrothermal ۹۱۰۰ apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer, and the results agreed favorably with the calculated values. Mass spectra were recorded on a Finnigan MAT ۸۴۳۰ spectrometer operating at an ionization potential of ۷۰ eV. IR spectra were measured on a Shimadzu IR-۴۶۰ spectrometer. ^{۱}H and ^{۱۳}C NMR spectra were measured on a Bruker Avance DRX-۳۰۰ spectrometer using $CDCl_۳$ as applied solvent and TMS as internal standard at ۳۰۰ and ۷۵ MHz, respectively.

General procedure for the preparation of compound **۵**

In a typical reaction, a mixture of isoquinoline (۰،۲۶ g, ۲ mmol) and dimethyl acetylenedicarboxylate (۰،۳۲ ml, ۲mmol) under solvent free condition was stirred for ۱ hour. The progress of reaction was

monitored by TLC. The resulting precipitate was separated by filtration and recrystallized from diethyl ether (Et_2O) to afford the pure compounds.

*Tetraethyl ٤H-pyrido[٢،١-a]isoquinoline-١،٢،٣،٤-tetracarboxylate (**٥a**)*

Yellow powder; yield: ٠,٨٤ g (٩٠٪), mp ١٥٢-١٥٤°C.IR (KBr) (ν_{max}/cm^{-1}): ١٧٣٤, ١٧١٥, and ١٦٦٦ (C=O), ١٦٢٦-١٤٧٨ (C=C). ^{1}H-NMR (٣٠٠ MHz, $CDCl_3$): δ = ١,١٤ (٣H, t, *J* = ٧,١ Hz, CH_3), ١,٢١ (٣H, t, *J* = ٧,١ Hz, CH_3), ١,٣٥ (٣H, t, *J* = ٧,١ Hz, CH_3), ١,٣٨ (٣H, t, *J* = ٧,١ Hz, CH_3), ٤,٠٨ (٢H, q, *J* = ٧,١ Hz, OCH_2), ٤.١١ (٢H, q, *J* = ٧,١ Hz, OCH_2), ٤,٣١(٢H, q, *J* = ٧,١ Hz, OCH_2), ٤,٣٨ (٢H, q, *J* = ٧,١ Hz, OCH_2), ٥,٧٧ (١H, s, CH), ٦,٤٤ (١H, d, *J* = ٧,٢ Hz, CH), ٦,٥٢ (١H, d, *J* = ٧,٢ Hz, CH), ٧,٠٠ (١H, m, CH), ٧,١٥ (١H, m, CH), ٧,٢٨ (٢H, m, ٢ CH). ^{13}C-NMR (٧٥ MHz, $CDCl_3$): δ = ١٣,٧, ١٣,٨, ١٤,٠, and ١٤,٢ (٤ CH_3), ٦٠,٨, ٦١,٠, ٦١,٥, and ٦٢,٦ (٤ OCH$_2$) ٩٦,٨ (CH), ١١١,٣ (C), ١١٩,٥ (CH), ١٢٣,٨ (CH), ١٢٣,٩ (CH), ١٢٦,٠ (CH), ١٢٧,٤ (CH), ١٢٨,٠ (CH), ١٢٩,٨ (C), ١٣٠,٠ (C), ١٣٩,٠ (C), ١٤٧,٧ (C), ١٥١,٨ (C), ١٦٥٨ (C=O), ١٦١٨-١٤٧٠ (C=C). ^{1}H-NMR (٣٠٠ MHz, $CDCl_3$): δ = ١,٤٤, ١,٤٨, ١,٥٧ and ١,٥٨ (٣٦H, ٤ s, ١٢ CH_3), ٥,٦٤ (١H, s, CH), ٦,٤١ (١H, d, *J* = ٧,٢ Hz, CH), ٦,٥٥ (١H, d, *J* = ٧,٢ Hz, CH), ٧,٠٤ (١H, m, CH), ٧.١١ (١H, m, CH), ٧,٣٠ (٢H, m, ٢ CH). ^{13}C-NMR (٧٥ MHz, $CDCl_3$): δ = ٢٨,٠, ٢٨,١, ٢٨,٣ and ٢٨,٤ (٤ CMe_3), ٧٩,٩, ٨٠,١, ٨٣,٨ and ٨٤,٢ (٤ O-CMe_3), ٩٦,٤ (CH), ١١٢,٨ (C), ١٢٠,٣ (CH), ١٢٥,٠ (CH), ١٢٥,٢ (CH), ١٢٧,٢ (CH), ١٢٨,٦ (CH), ١٢٩,٧ (CH), ١٣٠,١ (C), ١٣١,٥ (C), ١٤١,٢ (C), ١٥٠,٨ (C), ١٥٣,٤ (C), ١٦٢,٧, ١٦٣,٣, ١٦٤,١, and ١٦٦,٣ (٤ C=O). Anal.Calcd for $C_{٣١}H_{٣٧}NO_٨$ (٥٥١,٦٣): C, ٦٧,٥٠; H, ٦,٧٦; N, ٢,٥٤; Found: C, ٦٧,٣٤; H, ٦,٧٣; N, ٢,٥٠ %.

Tetraethyl ٤H-quinolizine-١،٢،٣،٤- tetracarboxylate (٢a)

Yellow powder; yield: ٠,٨٨ g (٩٥٪), mp١٩٥-١٩٦°C.IR (KBr) (ν_{max}/cm^{-1}): ١٧٤١, ١٧١٥, and ١٦٦٦ (C=O), ١٦١٩-١٤٨١ (C=C). ^{1}H-NMR (٣٠٠ MHz, $CDCl_3$): δ = ١,١٨ (٣H, t, *J* =٧,١ Hz, CH_3), ١,٢٨ (٣H, t, *J* = ٧,١ Hz, CH_3), ١,٣٦ (٣H, CH_3), ١٦٢,٤,

t, *J* = ۷,۱ Hz, CH), ۱,۳۹ (۳H, t, *J* =۷,۱ Hz, CH), ٤,۱۰ (۲H, q, *J* = ۷,۱ Hz, OCH), ٤,۲۳ (۲H, q, *J* = ۷,۱ Hz, OCH), ٤,۳۳ (۲H, q, *J* = ۷,۱ Hz, OCH), ٤,٤۲ (۲H, q, *J* = ۷,۱ Hz, OCH), ٥,۹٦ (۱H, s, CH), ٦,۷۹ (۱H, dt, ^{۳}J = ٦,۷ Hz, ^{٤}J = ۱,۳ Hz, CH), ۷,٤٦ (۲H, m, ۲ CH), ۸,٦۳ (۱H, dd, ^{۳}J = ۹,۷ Hz, ٤J = ۱,۳ Hz, CH). C-NMR (۷٥ MHz, $CDCl_{۳}$): δ = ۱۳,٤, ۱۳,۸, ۱٤,۰, and ۱٤,۱ (٤ $CH_{۳}$), ٦۰,٥, ٦۱,۲, ٦۱,٦, and ٦۲,۰ (٤ $OCH_{۲}$), ۹٥,۸ (C), ۱۱٥,٥ (CH), ۱۲۳,۸ (CH), ۱۳٦,۳ (C),

Anal.Calcd for $C_{۲٥}H_{۲۷}NO_{۸}$ (٤٦۹,٤۹): C, ٦۳,۹٦; H, ٥,۸۰; N, ۲,۹۸; Found: C, ٦۳,۹۰; H, ٥,۸۳; N, ۲,۹٦ %.

Tetra-tert-butyl ٤*H-pyrido[*۲،۱*-a]isoquinoline* **-** ۱،۲،۳،٤*-tetracarboxylate (***0b***)*

Yellow powder; yield: ۰,۹٤ g (۸٥%), mp ۱۷٥-۱۷۷°C.IR (KBr) (ν_{max}/$cm^{-۱}$): ۱۷۳٥, ۱۷۱۱, and

۱۳۷,۹ (C), ۱۴۲,۷ (CH), ۱۴۶,۲ (C), ۱۴۹,۳ (CH),
۱۵۷,۰ (C), ۱۶۳,۸, ۱۶۴,۸, ۱۶۸,۱, and ۱۶۹,۸ (۴
C=O). Anal.Calcd for $C_{۲۱}H_{۲۵}NO_{۸}$ (۴۱۹,۴): C, ۶۰,۱۴; H, ۶,۰۱; N, ۳,۳۴; Found: C, ۶۰,۱۷; H,
۵,۹۷; N, ۳,۳۰ %.

*Tetra-tert-butyl ۴H-quinolizine-۱،۲،۳،۴- tetracarboxylate (**۳b**)*

Yellow powder; yield: ۱,۰۱ g (۹۸٪), mp۱۹۵-۱۹۷°C.IR (KBr) (ν_{max}/cm$^{-۱}$): ۱۷۴۳, ۱۷۱۲, and ۱۶۸۶ (C=O), ۱۶۲۵-۱۴۳۶ (C=C). ^{۱}H-NMR (۳۰۰
MHz, $CDCl_{۳}$): δ = ۱,۴۰, ۱,۴۶, ۱,۵۳ and ۱,۵۵
(۳۶H, ۴ s, ۱۲ $CH_{۳}$), ۵,۵۸ (۱H, s, CH), ۶,۷۹-
۶,۸۲ (۱H, dt, ^{۳}J = ۶,۷ Hz, ^{۴}J = ۱,۳ Hz, CH),
۷,۴۷-۷,۵۰ (۲H, m, ۲ CH), ۸,۶۲-۸,۶۴ (۱H, dd,
۳J = ۹,۶ Hz, ^{۴}J = ۱,۲ Hz, CH). ^{۱۳}C-NMR (۷۵
MHz, $CDCl_{۳}$): δ = ۲۷,۶, ۲۷,۹, ۲۸,۰ and ۲۸,۳ (۴
$CMe_{۳}$), ۷۹,۵, ۸۰,۰, ۸۳,۷ and ۸۴,۳ (۴ O-$CMe_{۳}$),
۹۴,۷ (CH), ۱۱۴,۹ (CH), ۱۲۲,۸ (CH), ۱۳۶,۴ (C),
۱۳۸,۵ (C), ۱۴۲,۶ (CH), ۱۴۵,۵ (C), ۱۴۸,۴ (CH),
۱۵۷,۴ (C), ۱۶۳,۷, ۱۶۵,۲, ۱۶۶,۸, and ۱۶۷,۳ (۴
C=O). Anal.Calcd for $C_{۲۹}H_{۴۱}NO_{۸}$ (۵۳۱,۶۴): C, ۶۵,۵۲; H, ۷,۷۷; N, ۲,۶۳; Found: C, ۶۵,۴۹; H,
۷,۷۶; N, ۲,۶۰
%.

$CH_{۳}$), ۳,۹۳ (q, ۲H, *J* = ۷,۱ Hz, $OCH_{۲}$), ۴,۰۹ (q, ۲H, *J* = ۷,۱ Hz, $OCH_{۲}$), ۴,۱۸ (q, ۲H, *J* = ۷,۱ Hz, $OCH_{۲}$), ۴,۳۲ (q, ۲H, *J* = ۷,۱ Hz, $OCH_{۲}$),
۵,۱۷ (۱H, dd, ^{۳}J = ۲,۸ Hz, ^{۴}J = ۲,۸ Hz, CH),
۶,۰۳ (۱H, dd, ^{۳}J = ۹,۴ Hz, ^{۴}J = ۲,۸ Hz, CH),
۶,۵۶ (۱H, dd, ^{۳}J = ۹,۴ Hz, ^{۴}J = ۲,۸ Hz, CH),
۷,۱۴-۷,۱۷ (۳H, m, CH), ۷,۲۳-۷,۲۸ (۱H, m, CH). ^{۱۳}C-NMR (۷۵ MHz, $CDCl_{۳}$): δ = ۱۳,۲,
۱۳,۴, ۱۳,۸, and ۱۴,۰ (۴ $CH_{۳}$), ۶۰,۷, ۶۱,۵, ۶۱,۷,
and ۶۲,۱ (۴ $OCH_{۲}$), ۹۷,۸ (CH), ۱۱۱,۱ (CH),
۱۲۱,۷ (CH), ۱۲۵,۴ (C), ۱۲۷,۰ (C), ۱۲۷,۵ (C),
۱۲۸,۲ (CH), ۱۲۹,۷ (CH), ۱۳۰,۸ (CH),
۱۳۱,۶ (C), ۱۳۶,۵ (CH), ۱۳۸,۶ (C), ۱۵۱,۶ (C), ۱۶۲,۷,
۱۶۳,۶, ۱۶۳,۸, and ۱۶۷,۶ (۴ C=O). Anal.Calcd for $C_{۲۵}H_{۲۷}NO_{۸}$ (۴۶۹,۴۹): C, ۶۳,۹۶; H, ۵,۸۰; N, ۲,۹۸; Found: C, ۶۳,۹۱; H, ۵,۸۳; N, ۳,۰۱ %.

*Tetra-tert-butyl ۴aH-pyrido[۱،۲-a]quinolone-۱،۲،۳،۴-tetracaboxylate (**۸b**)*

Yellow powder; yield: ۱,۰۴ g (۹۵٪), mp۱۵۸-۱۶۰°C. IR (KBr) (ν_{max}/cm$^{-۱}$): ۱۷۴۱, ۱۷۲۰, and ۱۶۹۵ (C=O), ۱۶۲۳-۱۴۴۵ (C=C), ^{۱}H-NMR (۳۰۰

Tetraethyl ۴aH-pyrido[۱،۲-a]quinolone-۱،۲،۳،۴-tetracaboxylate (۸a)

Yellow powder; yield: ۱,۰ g (۹۰٪), mp۱۴۳-

۱۴۵°C. IR (KBr) (νmax/cm^{-1}): ۱۷۳۸, ۱۷۰۹, and ۱۶۸۶ (C=O), ۱۴۹۲,۱۴۸۰ (C=C), ^{1}H-NMR (۳۰۰ MHz, CDCl$_3$): δ = ۰,۹۳ (t, ۳H, *J* =۷,۱ Hz, CH$_3$), ۱,۱۷ (t, ۳H, *J* = ۷,۱ Hz, CH$_3$), ۱,۲۲ (t, ۳H, *J* = ۷.۱ Hz, CH$_3$), ۱,۳۵ (t, ۳H, *J* =۷,۱ Hz, MHz, CDCl$_3$): δ = ۱,۴۰, ۱,۴۳, ۱,۵۲ and (۳۶H, ۴ s, ۱۲ CH$_3$) ۵,۵۴ (۱H, dd, ^{3}*J* = ۳.۰ Hz, ^{4}*J* = ۲.۷ Hz, CH), ۶,۱۹ (۱H, dd, ^{3}*J* = ۹.۴ Hz, ^{4}*J* = ۲.۷ Hz, CH), ۶,۶۱-۶,۶۲ (۱H, dd, ۳J = ۹.۳ Hz, ^{4}*J* = ۲.۷ Hz, CH), ۷,۱۲-۷,۲۴ (۳H, m, CH), ۷,۲۸-۷,۳۰ (۱H, m, CH). ^{13}C-NMR (۷۵ MHz, CDCl$_3$): δ = ۲۸,۳, ۲۸,۵, ۲۸.۹ and ۳۰.۱ (۴ C(Me)), ۸۰,۱, ۸۰,۵, ۸۴.۰ and ۸۴.۴ (۴ O-C(Me)), ۹۷.۲ (CH), ۱۱۰,۸ (CH), ۱۲۲.۳ (CH), ۱۲۵.۹ (C), ۱۲۷.۳ (C), ۱۲۷,۸ (C), ۱۲۸.۵ (CH), ۱۳۰.۲ (CH), ۱۳۰.۶ (CH), ۱۳۱,۱ (C), ۱۳۶.۱ (CH), ۱۳۸.۴ (C), ۱۵۱.۹ (C), ۱۶۲,۳,

۱۶۳,۷, ۱۶٤,۲, and ۱۶۷.۹ (٤ C=O). Anal.Calcd for

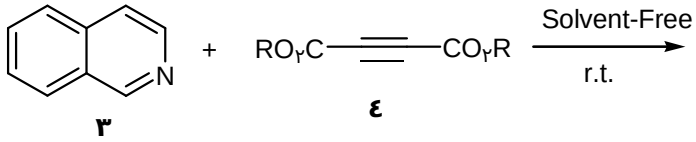
C H NO (۵۵۱,۶۳): C, ۶۷,۵۰; H, ۶,۷۶; N, ۳۱ ۳۷ ۸ ۲,۵٤;

Found: C, ۶۷,۵۲; H, ۶,۷۹; N, ۲,۵۷ %.

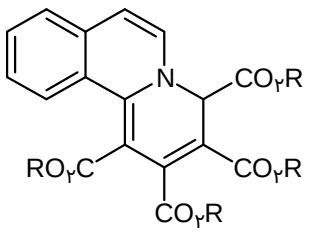
acetylenedicarboxylate ٤ in the absence of solvent at ambient temperature produces ٤H-pyrido[۲،۱-a]isoquinoline ٥ in an excellent yields (Scheme ۲).

Results and Discussion

The reaction of isoquinoline ۳ and dialkyl



۳ **٤**



٥

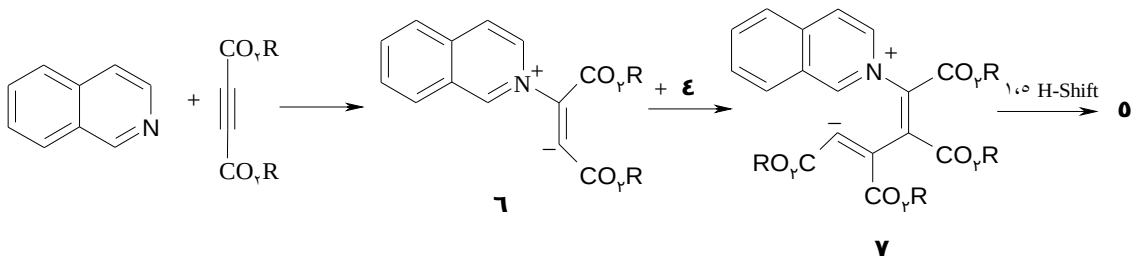
٥	R	%Yield
a	Et	۹۰
b	t-Bu	۸۵

Scheme ۲.

Isoquinoline undergoes a smooth reaction with dialkyl acetylenedicarboxylates ٤ in the absence of solvent at ambient temperature to produce functionalized ۱۱bH-pyrido[**۲،۱-a**] isoquinoline (**٥ a-b**) in an excellent yields.

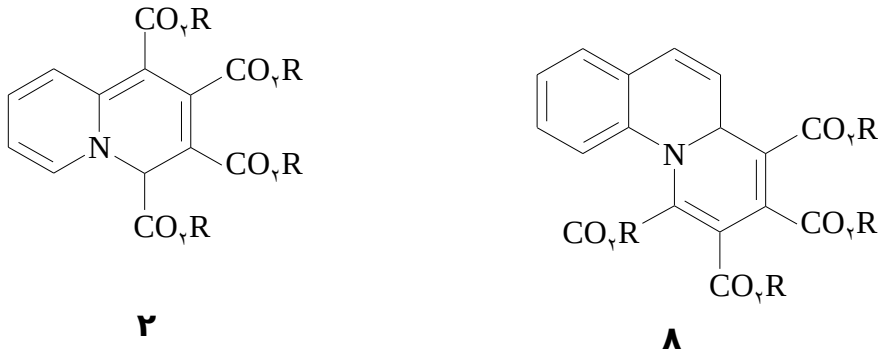
The reaction was completed within an hour (monitored by TLC). The ^{1}H and ^{13}C NMR spectra of the crude products clearly indicated the formation of ٥. The structures of compounds **٥a-b** were deduced from their elemental analyses and their IR, ^{1}H and ^{13}C NMR spectra. The mass spectra of these compounds displayed molecular ion peaks at the appropriate *m/z* values.

Although the mechanistic details of the reaction are not clearly known, a plausible rationalization may be advanced to explain the product formation. Presumably, the zwitterions [۲۷-۲۸] **٦** formed from isoquinoline and dialkyl acetylenedicarboxylate, adds to second acetylenic compound to furnish intermediate ۷. This intermediate undergoes cyclization and then ۱،٥ H-shift to furnish the fused structure ٥ (Scheme ۳).



Scheme ٣.

Reaction of pyridin and quinoline with dialkyl acetylenedicarboxylates ٤ under above conditions produce ٤H-quinolizine ٢ and ٤aH-pyrido[١،٢-a]quinoline ٨ respectively (Scheme ٤).



Scheme ٤.

Conclusion

The presented reaction provides a simple entry to the one-pot synthesis of ٤H-pyrido[٢،١-a] isoquinoline, ٤H-quinolizine, and ٤aH-pyrido[١،٢-a]quinoline derivatives of potential synthetic interest. The present procedure carries the advantage that, not only is the reaction performed under neutral conditions, but also the substances can be mixed without any activation or modification.

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