RESEARCH LETTER

An expeditious, highly efficient, catalyst and solvent-free synthesis of 9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinimide derivatives

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A simple, fast, and highly efficient protocol has been developed for the synthesis of 9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinic imide derivatives by simple mixing and grinding of 9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinic anhydride with various aliphatic, aromatic amines, and hydrazines. This catalyst and solvent-free green approach provided the condensation products within few minutes at room temperature in quantitative yield.

Keywords: anthracene; imides; green synthesis; solvent- and catalyst-free synthesis

Introduction

Solvent- and catalyst-free green synthesis occupies a unique segment in chemical space. With the growing awareness in academic, research, and industrial laboratories for sustainable development, the research community is under increasing pressure to alter current working practices and to find greener alternatives (1, 2). Therefore, it is not surprising and is reflected in an exponential increase in the productivity of scientific papers, books (3, 4), and reviews (5, 6) related to the use of green chemistry. In recent years, researchers have applied green technology as a tool in order to reduce reaction time, diminish side products, increase yield, and simplify the course of reactions for combinatorial chemistry (7). This solvent- and catalyst-free synthetic technology has shown broad applications as a very efficient way to develop the course of many organic reactions; so as a result, it has been promoted worldwide by an increasing number of research groups because it provides transformations that are environmentally benign (8, 9).

In the past decade, aromatic compounds bearing imide moiety are significant organic species (10–16), which promote a wide range of applications as optoelectronic devices (17), chemical sensors (18), supramolecular assemblies (19), and organic semiconductors (20). Along with the photophysical and chemical properties, organic imides and diimides also play significant role in pharmacology. Imides possess wide range of biological activities such as anti-inflammatory (21–24), anticancer (25, 26), antimicrobial (27), DNA binding, and apoptotic-inducing activities (28), etc. They also act as selective \(\alpha\)1-adrenergic receptor antagonists which modulate intercellular biochemical processes in response to changes in extracellular concentrations of the neurotransmitter norepinephrine and the circulating hormone epinephrine, leading to widespread physiological actions that make them attractive targets for

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drug discovery (29, 30). Well-known drug thalidomide, a synthetic glutamic acid derivative, bearing two imide moieties, has interesting pharmacological properties (31).

Due to the immense importance of imide molecules, it is necessary to review the synthesis of imide derivatives. Various methods are reported by several research groups in literature for synthesis of imide derivatives (32–35). Current interest includes developing new imide molecules to broaden the imides applications and further understand the relationship between molecular structure and properties. It is necessary to develop novel synthetic methodology to synthesize novel types of aromatic imides with π-conjugated cores. N-substituted imide bond formation frequently requires the low-yielding condensation of an amine with a free anhydride at high temperature for long time.

As part of our ongoing research work on the development of new molecules of biological interest using green approaches in organic synthesis (36, 37), herein we report our results on near absolute green procedure for the synthesis of N-substituted 9,10-dihydro-anthracene-9,10-imide. Herein we report our results on near absolute green approach in organic synthesis (38). Current interest includes developing new imide molecules to broaden the imides applications and further understand the relationship between molecular structure and properties. It is necessary to develop novel synthetic methodology to synthesize novel types of aromatic imides with π-conjugated cores. N-substituted imide bond formation frequently requires the low-yielding condensation of an amine with a free anhydride at high temperature for long time.

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Results and discussion

9,10-Dihydro-anthracene-9,10-β-succinic anhydride (3) has been synthesized by reaction of anthracene (1) and maleic anhydride (2) (Scheme 1) under conventional heating (38) in 85% yield, (mp 261–263°C).

Reaction between 9,10-dihydroanthracene-9,10-β-succinic anhydride and various amines: As a prelude to this objective, we carried out the reaction between liquid reactants. Thus, equimolar amounts of 9,10-dihydroanthracene-9,10-β-succinic anhydride (3) and cyclopropanamine (4a) having n = 0, (Method A, Scheme 2) were taken in a petri dish and mixed thoroughly with spatula followed by vigorously grinding for 2 minutes and then the mixture was allowed to stand in a desiccator for 5 minutes. The progress of the reaction was monitored by thin-layer chromatography (TLC) over silica gel using ethyl acetate/methanol (19:1) as mobile phase. During this period, complete conversion from the reactants to imide took place in this atom economy reaction.

After completion of the reaction, product was purified via washing with diethyl ether followed by simple crystallization from methanol to obtain product 5a in good yield. The purity of the product 5a was good enough for spectroscopic analysis. Compound 5a was fully characterized by IR, 1H NMR, GC-MS spectroscopic data, and elemental analysis. For further verification, this reaction was performed under conventional heating. Equimolar amounts of 9,10-dihydroanthracene-9,10-β-succinic anhydride (3) and cyclopropanamine (4a) (Method B, Scheme 2) were dissolved in toluene and refluxed on sand bath. Reaction progress was monitored by TLC over silica gel using ethyl acetate: methanol (19:1) as mobile phase. After completion of the reaction in 2 hour, product was purified by washing with diethyl ether followed by crystallization from methanol to give pure product 5a. Compound 5a was fully characterized by IR, 1H NMR, GC-MS spectroscopic data, and elemental analysis. Spectroscopic data of compound 5a reveals that the product obtained via Method A and Method B was pure and same.

After a successful attempt with amine in which value of n is 0, we further explored the reaction of 9,10-dihydroanthracene-9,10-β-succinic anhydride (3) with various amines (n = 0, 1, 2, 3 and three- to six-membered ring system). A series of product 5a–i have been synthesized via condensation of compound 3 with various amines (4a–i). All the compounds 5a–i were synthesized by following Method A (grinding the equimolar ratio of both reactant, solvent- and catalyst-free condition) and Method B (by refluxing both the reactants in toluene for 2–5 h, conventional method).

In further exploration of this method, anthracene imides 7a and 7b (Scheme 3) have been synthesized by both the methods grinding method and conventional heating method and results are summarized in Table 1. This method is also applicable to hydrazine and heterocyclic hydrazine, that is phenylhydrazine and 2-hydrazinopyridine with, 9,10-dihydroanthracene-9,10-β-succinic anhydride. Both the compounds 7a and 7b were characterized by IR, H NMR, GC-MS spectroscopic data, and elemental analysis. Reaction time taken, percentage yield, and melting point (mp) of all the compounds (5a–i, 7a, and 7b) synthesized are mentioned in Table 1.

In the course of our investigations reactions were carried out by fast grinding of stoichiometric molar
ratio of both the reactants with a spatula in a petri dish for different time intervals (2–5 min) and allowing the contents to stand for 5 minutes in desiccator. In all these cases, the reaction proceeded smoothly to produce the condensation products in quantitative yield.

**Experimental**

Melting points were determined on a JSGW apparatus and are uncorrected. IR spectra were recorded using a Perkin–Elmer 1600 FT spectrometer. ^1^H NMR spectra were recorded on a Bruker WH-500 spectrometer at a ca 5–15% (w/v) solution in DMSO-\(\text{d}_6\). GC-MS was recorded on Perkin–Elmer Clarus 500 gas chromatograph where built in MS detector was used. Elemental analysis was carried out on a Vario EL III elementor. TLC was performed on silica gel G for TLC (Merck) and spots were visualized by iodine vapor or by irradiation with ultraviolet light (short wave length, 254 nm).

**General procedure for synthesis of 9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinimide derivatives**

**Method A**

9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinic anhydride (3; 552 mg, 2 mmol) and cyclopropanamine \(4a\) (114 mg, 2 mmol) were mixed together thoroughly and ground vigorously with a spatula for 2 minutes in a petri dish to form a homogeneous paste and then the mixture was allowed to stand for 5 minutes in a desiccator. Completion of reaction was monitored by TLC over silica gel using ethyl acetate: methanol (19:1) as mobile phase. The solid product so obtained was purified by washing with diethyl ether followed by crystallization from methanol to give pure product \(5a\) in 92% yield.

**Method B**

9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinic anhydride (3; 276 mg, 1 mmol) and cyclopropanamine \(4a\) (57 mg, 2 mmol) were dissolved in dry tetrahydrofuran and kept on refluxing on sand bath.

Scheme 2. Synthesis of 9,10-dihydro-anthracene-9,10-\(\alpha,\beta\)-succinimide derivatives.

Scheme 3. Synthesis of anthracene-imide derivatives by using hydrazines.
progress was monitored by TLC over silica gel using ethyl acetate: methanol (19:1) as mobile phase. After completion of the reaction in 3 hour, reaction contents were cooled in cold water bath. So obtained solid was filtered and this crude product was purified by washing with diethyl ether followed by crystallization from methanol to give pure product 5a in 72% yield. Similarly compounds 5b–i, 7a, and 7b were synthesized.

9,10-Dihydro-anthracene-9,10-α,β-succinimide: (3)
IR (KBr) ν max: 1650 (CO–C=O), 1601, 1470 (Ar) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ: 3.659–3.666 (t, 2H, 2 CH₂), 4.881 (s, 2H, 2 CH₂), 7.178–7.211 (d, 4H, Ar), 7.335–7.353 (q, 2H, Ar), and 7.475–7.492 (q, 2H, Ar). GC-MS: m/z 276 (5%).

N-(Cyclopropyl)-9,10-dihydro-anthracene-9,10-α,β-succinimide, (5a)
IR (KBr) ν max: 1711 (–CO–N–CO–), 1461, 1403 (Ar) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ: 0.501–0.509 (m, 2H, 2 CH₃), 0.521–0.573 (m, 2H, CH₂), 2.047–2.091 (m, 1H, CH), 3.133–3.150 (d, 2H, J = 8.5 Hz), 4.715–4.732 (d, 2H, J = 8.5 Hz), 7.137–7.230 (m, 6H, Ar), 7.446–7.479 (m, 2H, Ar). GC-MS: m/z 315 (M⁺, 25%). Analysis calculated for C₂₁H₁₇NO₂: C, 80.00; H, 5.39; N, 4.44. Found: C, 79.96; H, 5.38; N, 4.40.

N-(4-(2,2,6,6-tetramethyl)piperadine)-9,10-dihydro anthracene-9,10-α,β-succinimide, (5b)
IR (KBr) ν max: 1699 (CO–C=O), 1498, 1401 (Ar) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ: 0.544–0.564 (d, 2H, CH₂), 0.919 (s, 6H, 2 CH₃), 0.971–1.011 (s, 6H, 4 CH₃), 1.450–1.500 (t, 2H, J = 12.5 Hz, CH₂), 3.184 (s, 2H, 2 CH₂), 3.67–3.899 (t, 1H, J = 3.5 Hz, CH₂), 4.755 (s, 2H, 2 CH₂), 7.125–7.142 (m, 2H, Ar), 7.160–7.185 (m, 2H, Ar), 7.227–7.252 (m, 2H, Ar), 7.465–7.489 (m, 2H, Ar). GC-MS: m/z 414 (M⁺, 8%). Analysis calculated for C₂₇H₃₀N₂O₂: C, 78.26; H, 7.24; N, 6.76. Found: C, 78.25; H, 7.24; N, 6.73.

N-(2-Picolyl)-9,10-dihydro-anthracene-9,10-α,β-succinimide, (5c)
IR (KBr) ν max: 1698 (–CO–N–CO–), 1462, 1397 (Ar) cm⁻¹; ¹H NMR (DMSO-d₆, 500 MHz) δ: 3.452 (s, 2H, 2 CH₂), 4.330 (s, 2H, CH₂), 3.868 (s, 2H, CH₂), 5.359–5.375 (t, 1H, J = 8 Hz, py). 7.160–7.182 (m, 3H, 2H of Ar +1H of py), 7.218–7.251 (m, 2H, Ar), 7.288–7.314 (m, 2H, Ar), 7.400–7.435 (m, 1H, py), 7.473–7.505 (m, 2H, Ar). GC-MS: m/z 366 (M⁺, 28%).

Table 1. Grinding time (in min, Method A), refluxing time (hours, Method B), percentage yield and melting point of anthracene derivatives 5a–i and 7a, 7b.

| No. | N | Reaction time | % Yield | Melting point |
|-----|---|----------|-------|--------------|
|     |   | Method A (min) | Method B (h) | Method A | Method B |
| 5a  | 0 | 2        | 2     | 220–221 | 92 | 72 |
| 5b  | 0 | 3        | 2     | 245–247 | 96 | 86 |
| 5c  | 1 | 4        | 1     | 240–241 | 91 | 87 |
| 5d  | 1 | 4        | 2     | 237–240 | 98 | 85 |
| 5e  | 1 | 4        | 3     | 225–228 (228–230) | 95 | 86 |
| 5f  | 2 | 5        | 4     | 241–243 | 90 | 81 |
| 5g  | 2 | 4        | 3     | 218–220 (217–220) | 90 | 82 |
| 5h  | 2 | 3        | 4     | 153–159 | 94 | 83 |
| 5i  | 3 | 4        | 5     | 242–249 | 94 | 80 |
| 7a  | – | 3        | 2     | 280–283 (279) (40) | 92 | 80 |
| 7b  | – | 5        | 4     | 165–168 (168–170) (41) | 86 | 95 |

Table 1. Grinding time (in min, Method A), refluxing time (hours, Method B), percentage yield and melting point of anthracene derivatives 5a–i and 7a, 7b.
N-(4-Picolyl)-9,10-dihydro-anthracene-9,10-α,β-succinimide: (5d)
IR (KBr) ν max: 1715 (C=O), 1651 (C=O), 1548, 1319 (Ar) cm⁻¹. 1H NMR (DMSO-d₆, 500 MHz) δ: 8.265 (s, 1H, Ar), 7.240 (m, 2H, Ar), 7.274–7.292 (m, 2H, Ar), 7.475–7.492 (m, 2H, Ar), 8.265–8.277 (d, 2H, J = 6 Hz, py). GC-MS: m/z 366 (M⁺, 10%). Analysis calculated for C₂₅H₁₈N₂O₂: C, 76.88; H, 4.92; N, 7.65. Found: C, 76.85; H, 4.91; N, 7.65.

N-(2-Methylmorpholin)-9,10-dihydro-anthracene-9,10-α,β-succinimide: (5e)
IR (KBr) ν max: 1705 (C=O), 1641, 1413, 1395 (Ar) cm⁻¹. 1H NMR (DMSO-d₆, 500 MHz) δ: 2.184 (bs, 4H, 2 × CH₂), 2.354 (s, 2H, CH₂), 2.344 (s, 2H, 2 × CH₂), 3.663 (s, 4H, 2 × CH₂), 4.817 (s, 2H, 2 × CH), 7.122–7.140 (m, 2H, Ar), 7.160–7.176 (m, 2H, Ar), 7.260–7.257 (m, 2H, Ar), 7.465–7.481 (m, 2H, Ar). GC-MS: m/z 374 (M⁺, 22%). Analysis calculated for C₂₄H₁₈N₂O₂: C, 78.68; H, 4.92; N, 7.64. Found: C, 78.64; H, 4.92; N, 7.64.

N-(2-Pyrrolidin-2-one)propyl-9,10-dihydro-anthracene-9,10-α,β-succinimide: (5f)
IR (KBr) ν max: 1707 (C=O), 1646(CO), 1546, 1459, 1319 (Ar) cm⁻¹. 1H NMR (DMSO-d₆, 500 MHz) δ: 1.388–1.599 (m, 6H, aliphatic), 2.226 (bs, 4H, aliphatic), 3.037–3.067 (t, 2H, CH₂), 3.251 (s, 2H, 2 × CH₂), 4.771 (s, 2H, 2 × CH), 7.113–7.138 (m, 2H, Ar), 7.158–7.175 (m, 2H, Ar), 7.223–7.248 (m, 2H, Ar), 7.463–7.480 (q, 2H, J = 3, J = 5, J = 8.5, Ar). GC-MS: m/z 372 (M⁺, 5%). Analysis calculated for C₂₅H₂₂N₂O₂: C, 77.42; H, 6.45; N, 7.52. Found: C, 77.41; H, 6.40; N, 7.52.

N-(3-(Pyridin-2-yl)hydrazino)-9,10-dihydro-anthracene-9,10-α,β-succinimide: (7b)
IR (KBr) ν max: 3098 (NH), 1701 (C=O), 1520, 1495, 1400 (Ar) cm⁻¹. 1H NMR (DMSO-d₆, 500 MHz) δ: 3.658 (s, 2H, 2 × CH), 4.873(s, 2H, 2 × CH), 6.665–6.688 (t, 1H, Ar), 7.172–7.203(m, 2H, Ar), 7.266–7.295 (d, 2H, Ar), 7.329–7.345 (t, 3H, Ar), 7.468–7.495 (d, 2H, Ar), 7.942–7.952 (d, 1H, Ar), 8.746 (s, 1H, Ar). GC-MS: m/z 367 (M⁺, 6%). Analysis calculated for C₂₅H₁₈N₄O₂: C, 75.20; H, 6.43; N, 11.44. Found: C, 75.11; H, 4.57; N, 11.52.
Conclusion

In conclusion, we have demonstrated a simple, atom economy, fast, and highly efficient solvent-free green protocol for synthesis of anthracene imide derivatives. To the best of our knowledge, this green protocol is not known in literature. Further detailed studies on the biological evaluation of these anthracene imide derivatives are in progress.

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Electronic Supporting Information

An expeditious, highly efficient, catalyst and solvent-free synthesis of 9,10-dihydro-anthracene-9,10-a,b-succiniimide derivatives

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Copies of $^1$ H (500 MHz) and GC-MS Spectra: Pages 2–25
$^1$H NMR spectrum of compound 3: 9,10-dihydro-anthracene-9,10-α,β-Succinic Anhydride
GC and Mass spectra of compound 3: 9,10-dihydro-anthracene-9,10-a,b-Succinic Anhydride

GC spectrum

Mass spectrum

Molecular formula: C_{18}H_{12}O_{3}
Molecular weight: 276
$^1$H NMR spectrum of compound 5a

Molecular formula: C$_{21}$H$_{17}$NO$_2$
GC and Mass spectra of compound 5a

GC spectrum

Mass spectrum

Molecular formula: C_{21}H_{17}NO_{2}
Molecular weight: 315
$^1$H NMR spectrum of compound 5b

Molecular formula: C$_{27}$H$_{30}$N$_2$O$_2$
GC and Mass spectra of compound 5b

**GC spectrum**

![GC spectrum diagram]

**Mass spectrum**

Molecular formula: C_{27}H_{30}N_2O_2
Molecular weight: 414
$^1$H NMR spectrum of compound 5c

Molecular formula: $C_{24}H_{18}N_2O_2$
GC and Mass spectra of compound 5c

GC spectrum

Mass spectrum

Molecular formula: C_{24}H_{18}N_{2}O_{2}
Molecular weight: 366
$^1$H NMR spectrum of compound 5d

Molecular formula: $C_{24}H_{18}N_2O_2$
GC and Mass spectra of compound 5d

**GC spectrum**

![GC spectrum graph](image)

**Mass spectrum**

![Mass spectrum graph](image)

Molecular formula: C_{24}H_{18}N_{2}O_{2}
Molecular weight: 366
$^1$H NMR spectrum of compound 5e

Molecular formula: C$_{23}$H$_{22}$N$_2$O$_2$
GC and Mass spectra of compound 5e

GC spectrum

Mass spectrum

Molecular formula: C_{23}H_{22}N_{2}O_{2}
Molecular weight: 374
$^1$H NMR spectrum of compound 5f

Molecular formula: C$_{2d}$H$_{24}$N$_2$O$_2$
GC and Mass spectra of compound 5f

GC spectrum

Molecular formula: C_{24}H_{24}N_{2}O_{2}
Molecular weight: 372
$^1$H NMR spectrum of compound 5g

Molecular formula: $C_{25}H_{26}N_2O_2$
GC and Mass spectra of compound 5g

GC spectrum

Mass spectrum

Molecular formula: C_{25}H_{26}N_{2}O_{2}
Molecular weight: 386
$^1$H NMR spectrum of compound 5h

Molecular formula: C$_{24}$H$_{22}$N$_2$O$_3$
GC and Mass spectra of compound 5h

GC spectrum

Mass spectrum

Molecular formula: C_{24}H_{22}N_{2}O_{3}
Molecular weight: 386
$^1$H NMR spectrum of compound 5i

Molecular formula: $C_{25}H_{21}N_2O_2$
GC and Mass spectra of compound 5i

GC spectrum

Mass spectrum

Molecular formula: C_{25}H_{21}N_{2}O_{2}
Molecular weight: 381
$^1$H NMR spectrum of compound 7a

Molecular formula: C_{20}H_{18}N_{2}O_{2}
GC and Mass spectra of compound 7a

GC spectrum

Mass spectrum

Molecular formula: C_{24}H_{18}N_{2}O_{2}
Molecular weight: 366
$^1H$ NMR spectrum of compound 7b

Molecular formula: $C_{23}H_{17}N_3O_2$
GC and Mass spectra of compound 7b

GC spectrum

Mass spectrum

Molecular formula: C_{23} H_{17} N_{3} O_{2}
Molecular weight: 367