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Meglumine Sulfate as an Effective Catalyst for the Preparation of some Indeno[1,2-b]indole-9,10-dione Derivatives

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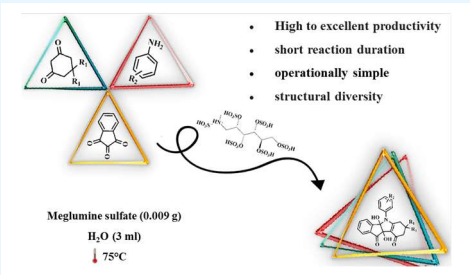
Abstract: Aligned with advancements in synthetic chemistry, this work presents the synthesis and characterization of meglumine sulfate using FT-IR and CHNS analysis. Subsequently, the potential of the synthesized catalyst is explored in the preparation of a class of nitrogen-containing heterocyclic compounds (indenoindolones), which possess significant medicinal and biological properties. The study also reports the optimization of reaction conditions to enable the efficient synthesis and characterization of a broad spectrum of these compounds. Notably, the utilization of meglumine sulfate offers several advantages, including exceptional yields, cost-effectiveness, rapid reaction times, and facile product purification.



Keywords: Meglumine sulfate, Indeno[1,2-b]indole-9,10-diones, Multicomponent reaction, Acid catalyst

1. Introduction

Multicomponent reactions (MCRs) are one-pot reactions in which the multiple reactants (at least three) combine to form a final product that shows maximum structural parts of the starting materials. In these reactions, all the steps of synthesis take place continuously without the need for the separation of intermediates and can be used to synthesize diverse molecular scaffolds such as heterocycles. MRCs have some merits including high efficiency, low purification cost and minimal formation of by-products.[1-7]

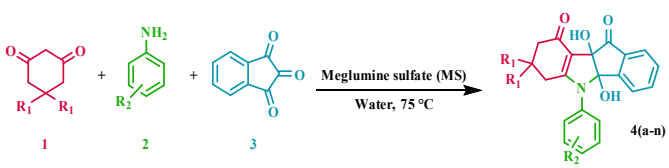
Heterocyclic compounds, defined as 5- or 6-membered rings containing at least one heteroatom (S, O, N), attracted most significant attention of researchers due to their immense potential against pathogens.[8-10] Nitrogen-consisted heterocycles have particular importance in medicinal chemistry due to their superior therapeutic potential compared to others.[11-13]

Indo-indolones as one of the N-containing heterocycle compounds are polyhydroxylated alkaloids with small, smooth ring structures. These compounds have been the subject of much attention from researchers in the chemical community, and also studies have shown that they have a variety of biological and biomedical properties, including the ability to kill breast and colon cancer cells, reduce neurological disorders, and treat bacterial, fungal, and viral infections.[14-18] Continuous efforts to synthesize indo[2,1-b]indole-10,9-dione derivatives have led to significant advances in this field. In 2015, Pardhan et al. successfully synthesized these compounds using the SnO_2QDS catalyst.[16]

In 2016, Chen et al. reported a solvent-free synthesis of these compounds using lactic acid.[19] In 2018, Safari et al. reported a method using the MMT@Fe_3O_4 catalyst in aqueous solution.[15] Finally, Moradi et al. introduced MSA-2/MO as an efficient catalyst for the same reaction in an aqueous media in 2021.[14]

Meglumine or N-methyl-D-glucamine as a sugar alcohol with a tertiary amine group, is a derivative of sorbitol. Meglumine is a versatile chemical compound with a wide range of applications due to its unique properties. It is commonly used as an excipient in pharmaceutical formulations, and recent research has highlighted its potential as a sustainable and accessible catalyst in organic reactions.[20,21]

In this research, the use of meglumine sulfate as an efficient catalyst was presented. The prepared catalyst was used in water solvent for the synthesis of indole[1,2-b]indole-9,10-dione by aniline derivatives, dimedone/1,3-cyclohexadione and ninhydrin with high yield and short reaction time (Scheme 1).



Scheme 1. Preparation of indeno[1,2-b]indole-9,10-diones using meglumine sulfate

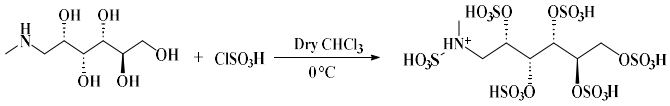
2. Experimental

Instrumentation and reagent

All necessary reagents and solvents were procured (from Merck and Fluka companies) and utilized without any additional purification. Melting points were obtained using an open capillary tube on an Electrothermal MK3 apparatus. ^{1}H and ^{13}C NMR spectra were acquired at 400 and 100 MHz, respectively, using a Bruker spectrometer. The FT-IR spectra were obtained in the wavenumber range of 400-4000 cm^{-1} using a Perkin-Elmer 550 spectrometer and KBr pellets. The elemental analyses were accomplished on a Perkin-Elmer 240c analyzer.

Preparation of meglumine sulfate

Meglumine (1 g) was added to dry chloroform (20 ml) at 0 °C. The reaction mixture was stirred at the aforementioned temperature. Chlorosulfonic acid (0.6 ml) was added dropwise to the mixture over 60 minutes, and then the mixture was stirred continuously for 6 hours. After the reaction was complete, the product was collected by centrifugation, washed twice with ethanol (20 mL each time) and dried in an oven at 90 °C (Scheme 2).



Scheme 2. Synthesis of meglumine sulfate (MS)

General procedure for the synthesis of indeno indolones

Dimedone (1 mmol), various anilines (1 mmol) and 0.009 g of meglumine sulfate were added to a 5 mL round-bottom flask containing 3 mL of H_2O. Stirring the mixture at 75 °C for a prescribed time (monitored by TLC), produced an enaminone intermediate (I). Ninhydrin (1 mmol) was subsequently added to the mixture and the reaction was allowed to proceed at 75 °C for the appropriate time. Upon completion of the reaction (confirmed by TLC), the mixture was filtered and the crude product was separated by filtration and for further purification, recrystallized from hot ethanol.

Selected spectral data

5-(3-Nitrophenyl)-4b,9b-dihydroxy-7,7-dimethyl-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione (4h). Cream solid; M.P. = 201-204 °C (201-205 °C) [16]; FT-IR (KBr, $\bar{\nu}$ (cm^{-1}): 3382 (OH), 2950 (C-H sp^2), 2946 (C-H sp^3), 1722 (C=O), 1605, 1447 (C=C aromatic), 1533, 1347 (N-O), 1287 (C-N), 1159 (C-O); ^{1}H NMR (400 MHz, DMSO-d_6) (δ ppm): 0.90 (3H, s, CH$_3$), 0.96 (3H, s, CH$_3$), 1.83 (1H, d, J = 16.0 Hz, CH), 1.92 (1H, d, J = 16.0 Hz, CH), 2.18 (1H, d, J = 16.0 Hz, CH), 2.56-2.54 (1H, m, CH), 6.16 (1H, s, OH), 6.68-6.66 (1H, m, Ar-H), 7.58-7.55 (3H, m, Ar-H, OH), 7.63 (1H, d, J = 8.0 Hz, Ar-H), 7.80-7.73 (2H, m, Ar-H), 8.36-8.31 (2H, m, Ar-H).

5-(3-Chloro-4-fluorophenyl)-4b,9b-dihydroxy-7,7-dimethyl-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione (4i). White solid; M.P. = 238-240 °C; FT-IR (KBr, $\bar{\nu}$ (cm^{-1}): 3469 (OH), 3102 (C-H sp^2), 2951 (C-H sp^3), 1723 (C=O), 1634, 1446 (C=C aromatic), 1449, 1395 (CH$_3$), 1325 (C-N), 1204 (C-F), 1148 (C-O), 765 (C-Cl); ^{1}H NMR (400 MHz, DMSO-d_6) (δ ppm): 0.91 (3H, s, CH$_3$), 0.96 (3H, s, CH$_3$), 1.82 (1H, d, J = 16.0 Hz, CH), 1.90 (1H, d, J = 16.0 Hz, CH), 2.14 (1H, d, J = 16.0 Hz, CH$_2$), 2.46 (1H, d, J = 16.0 Hz, CH$_2$), 6.10 (1H, s, OH), 6.72 (1H, d, J = 8.0 Hz, Ar-H), 7.27-7.23 (1H, m, Ar-H), 7.37 (1H, s, OH), 7.69-7.54 (4H, m, Ar-H), 7.74 (1H, d, J = 8.0 Hz, Ar-H); ^{13}C NMR (101 MHz, DMSO-d_6) (δ ppm): 18.5, 26.2, 29.5, 33.4, 36.6, 51.2, 56.0, 83.3, 96.6, 106.0, 117.0, 117.2, 119.5, 119.7, 124.0 (d, J = 150.0 Hz), 130.8 (d, J = 98.0 Hz), 133.1, 133.2, 134.9 (d, J = 40.0 Hz), 146.8, 155.5, 157.9, 163.0, 189.4, 197.4.

5-(3-Chloro-4-fluorophenyl)-4b,9b-dihydroxy-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione (4j). White solid; M.P. = 185-187 °C (180-190 °C)[15]; FT-IR (KBr, $\bar{\nu}$ (cm^{-1}): 3412 (OH), 2952 (C-H sp^2), 2876 (C-H sp^3), 1725 (C=O), 1555, 1498 (C=C aromatic), 1258 (C-N), 1154 (C-O), 994 (C-F), 768 (C-Cl); ^{1}H NMR (400 MHz, DMSO-d_6) (δ ppm): 1.87-1.72 (2H, m, CH$_2$), 2.05-1.99 (1H, m, CH), 2.18-2.08 (2H, m, CH$_2$), 2.49-2.42 (1H, m, CH), 6.06 (1H, s, OH), 6.73 (1H, d, J = 8.0 Hz, Ar-H), 7.31 (2H, s, Ar-H, OH), 7.64-7.54 (3H, m, Ar-H), 7.69 (1H, d, J = 8.0 Hz, Ar-H), 7.74 (1H, d, J = 8.0 Hz, Ar-H).

5-(Naphthalen-1-yl)-4b,9b-dihydroxy-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione (4k). Yellow solid; M.P. = 187-189 °C (186-188 °C)[22]; ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 0.77 (3H, s, CH$_3$), 0.90 (3H, s, CH$_3$), 1.78 (1H, s, CH$_2$), 2.19-1.93 (3H, m, CH$_2$, CH), 6.04 (1H, s, OH), 6.29 (1H, d, J = 8.0 Hz, Ar-H), 7.17-7.05 (2H, m, Ar-H), 7.45-7.42 (2H, m, Ar-H, OH), 7.63-7.59 (1H, m, Ar-H), 7.8-7.7 (2H, m, Ar-H), 8.12-7.87 (4H, m, Ar-H).

5,5'-(1,4-Phenylene)bis(4b,9b-dihydroxy-7,7-dimethyl-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione) (4l). Yellow solid; M.P. = 268-270 °C; FT-IR (KBr, $\bar{\nu}$ (cm^{-1}): 3422 (OH), 2956 (C-H sp^2), 2894 (C-H sp^3), 1723 (C=O), 1605, 1437 (C=C aromatic), 1404, 1356 (CH$_3$), 1279 (C-N), 1162 (C-O); ^{1}H NMR (400 MHz, DMSO-d_6) (δ ppm): 0.96 (6H, d, J = 8.0 Hz, CH$_3$), 1.015 (6H, d, J = 12.0 Hz, CH$_3$), 1.97-1.91 (4H, m, CH$_2$), 2.22-2.16 (2H, m, CH$_2$), 2.56 (2H, t, J = 12.0 Hz, CH$_2$), 6.04 (2H, s, OH), 6.78 (2H, t, J = 8.0 Hz, Ar-H), 7.53-7.28 (6H, m, Ar-H, OH), 7.66-7.56 (4H, m, Ar-H), 7.77 (2H, d, J = 8.0 Hz, Ar-H); ^{13}C NMR (100 MHz, DMSO-d$_6$) (δ ppm): 26.3, 26.4, 27.9, 29.5, 33.6, 37.0, 51.2, 83.4, 96.8, 106.0, 106.1, 123.4, 124.6, 129.5, 130.4, 134.7, 135.4, 147.1, 163.1, 189.2, 189.4, 197.5, 197.5.

3. Results and Discussion

In this section, the characteristics of meglumine sulfate were investigated using spectroscopic and pattern recognition methods. By utilizing the prepared catalyst, a sustainable and efficient one-pot, multi-component synthesis of indeno[1,2-b]indol-9,10-diones was achieved. To establish the ideal conditions for the synthesis, the impact of diverse parameters such as temperature, solvent, and amount of catalyst was meticulously evaluated. Finally, the synthesized derivatives were thoroughly identified and characterized using melting point determination, FT-IR, ^{1}H NMR and ^{13}C NMR spectroscopy.

Structural characterization of meglumine sulfate

FT-IR Analysis. Meglumine sulfate catalyst was synthesized following the procedure outlined in the experimental section and analyzed by Fourier transform infrared (FT-IR) to prove its structure. By examining the infrared spectra of meglumine, the characteristic absorptions related to OH and NH groups can be identified. These absorptions can be seen in the region of 3100 cm^{-1} to 3400 cm^{-1}. After the sulfonation, the absorptions related to the sulfonic acid group can be well

observed in the spectrum of meglumine sulfate. The broadening in absorption peak in the region of 3364 cm^{-1} indicates the presence of acidic OH. The sharp and intense absorption observed at 1256 cm^{-1} is related to the S=O group and the absorption at 649 cm^{-1} can be attributed to S-O stretching vibration (Figure 1). Acid-base titration revealed a concentration of approximately 3.5 mmol Hþ per gram on the meglumine sulfate.

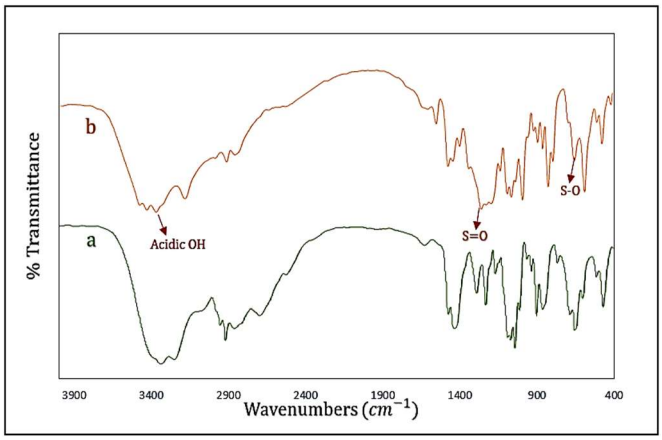


Figure 1. FT-IR of meglumine (a) and meglumine sulfate (b).

Elemental analysis (CHNS). Elemental analysis (CHNS) confirmed the successful synthesis of meglumine sulfate. Table 1 shows the elemental composition of the synthesized meglumine sulfate.

Table 1. Elemental analysis (CHNS) results of MS

Element	C	H	N	S
Percentage of each elements	24%	5.3%	4.3%	17%

Investigating the effects of various factors on the synthesis of indeno[1,2-b]indole-9,10-dione derivatives in the presence of MS

After the synthesis of meglumine sulfate, 4-methylaniline, dimedone, and ninhydrin were selected as starting materials to the optimize the reaction conditions. For this purpose, the effects of various factors on the synthesis of **4j** was investigated.

At first, the reaction was carried out in H_2O as a green solvent using different amounts of meglumine sulfate (Table 2). The best results were obtained when the reaction was carried out in the presence of 0.009 g of the catalyst at 75 ºC (Table 2, Entry 2). Further amount of catalyst (0.01 g) has no significant effect on reaction efficiency.

Table 2. Optimization of MS amount[a]

Entry	Catalyst (g)	Time (s)	Yield[b] (%)
1	0.007	75	41
2	**0.009**	**75**	**97**
3	0.01	75	97

[a]*Reaction conditions:* 4-methylaniline (1 mmol), dimedone (1 mmol) and ninhydrin (1 mmol), different amounts of MS, H_2O (3 ml) at 75 °C.
[b]Isolated yield.

In continue, an investigation was undertaken to identify the optimal solvent for the aforementioned reaction. To this regard, the reaction was carried out in H_2O, EtOH, EtOH: H_2O (1:1), and CH_3CN solvents (Table 3). As can be seen, the most desirable result was obtained when the reaction was carried out in H_2O (Table 3, Entry 1).

Table 3. Suitable reaction medium[a]

Entry	Solvent	Time (s)	Yield[b] (%)
1	**H_2O**	**75**	**97**
2	EtOH	75	25
3	EtOH/H_2O (1:1)	75	29
4	Toluene	75	31

[a]Reaction conditions*:* 4-methylaniline (1 mmol), dimedone (1 mmol) and ninhydrin (1 mmol), MS (0.009 g), different solvents (3 ml), at 75 °C.
[b]Isolated yield.

In addition to the factors mentioned above, temperature plays a crucial role in multicomponent reactions. To investigate its impact, the reaction was conducted at room temperature, 65, 75, and 85 °C (Table 4). While the reaction proceeded slowly at room temperature with trace amount of product (Table 4, Entry 1), increasing the temperature to 65 and 75 °C led to a significant increase in the reaction rate (Table 4, Entries 2, 3). However, the increasing of temperature to 85 °C, show no changes in the reaction efficiency (Table 4, Entry 4). Therefore, 75 °C was identified as the optimal temperature for the reaction (Table 4, Entry 3).

Table 4. Determination of the best temperature[a]

Entry	Temp. (°C)	Time (s)	Yield (%)
1	r.t	75	trace
2	65	75	20
3	**75**	**75**	**97**
4	80	75	97

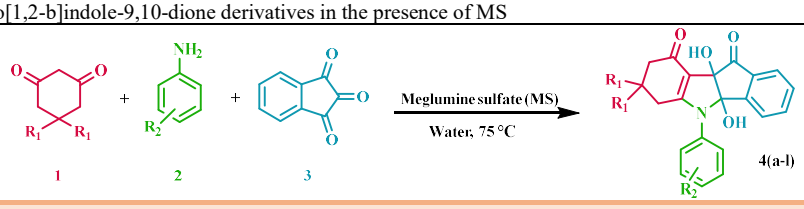
[a]*Reaction conditions:* 4-methylaniline (1 mmol), dimedone (1 mmol) and ninhydrin (1 mmol), MS (0.009 g), in H_2O (3 ml), under thermal conditions.
[b]Isolated yield.

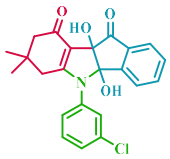
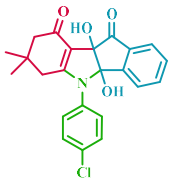
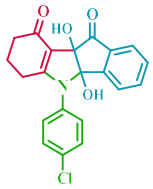
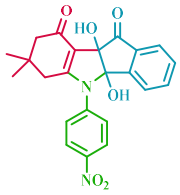
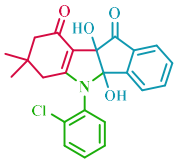
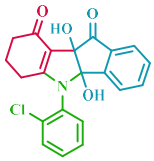
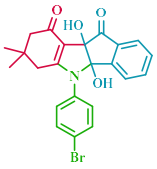
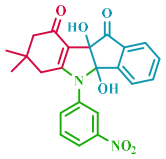
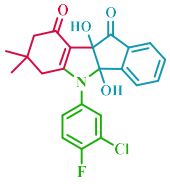
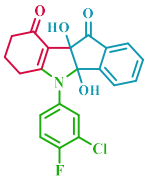
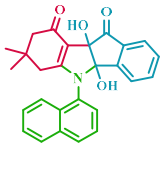
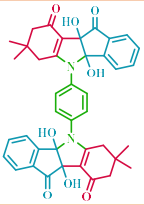
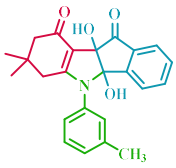
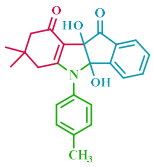
Consequently, the optimized conditions for the synthesis of this class of derivatives involve the use of 0.009 g of meglumine sulfate in H_2O at 75 °C.

Synthesis of indeno[1,2-b]indole-9,10-dione derivatives with MS under optimized conditions

After investigating various parameters and obtaining the optimal conditions, some of the indeno[1,2-b]indole-9,10-dione derivatives were synthesized in the presence of 0.009 g of meglumine sulfate in H_2O at 75 °C. Table 5 shows the time and yield for the synthesis of these derivatives. All products were obtained with high to excellent yields without any by-products. In this reaction, derivatives of aniline with electron-donating substituents showed faster and more efficient reactions than those with electron-withdrawing substituents.

Table 5. Synthesis of indeno[1,2-b]indole-9,10-dione derivatives in the presence of MS

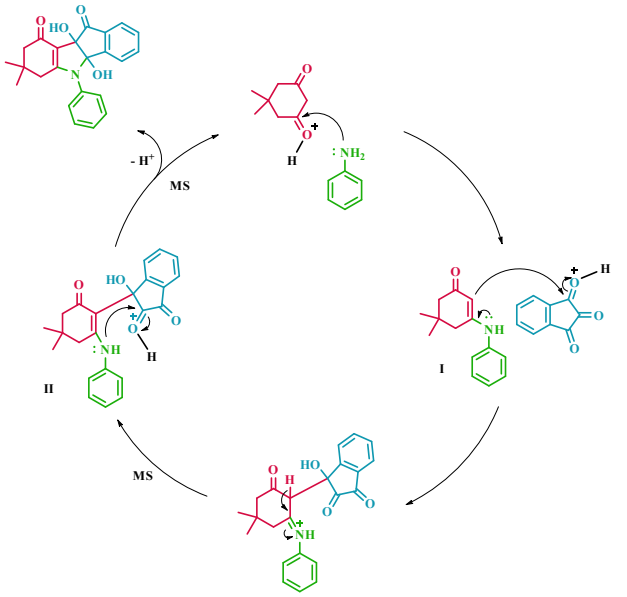


Compound (4a-4n)[a], Yield (%)[b], Time (min) m.p (°C), m.p (°C) [Ref.]			
			
4a, 89%, 80 min 223-225, 223-226 [19]	4b, 92%, 90 min 240-241, 240-242 [15]	4c, 92%, 90 min 199-201, 198-200 [14]	4d, 92%, 95 min 175-177, 175-177 [15]
			
4e, 89%, 85 min 241-242, 241-243 [15]	4f, 87%, 85 min 208-212, 205-220 [14]	4g, 92%, 90 min 160-162, 160-162 [14]	4h, 92%, 90 min 201-204, 201-205 [15]
			
4i, 91%, 85 min 238-240°	4j, 92%, 85 min 185-187, 180-190 [14]	4k, 83%, 105 min 187-189, 186-188 [22]	4l, 80%, 115 min 268-270
			
4m, 96%, 75 min 118-121, 118-120 [23]	4n, 97%, 75 min 234-237, 234-236 [15]		

[a]Isolated yield.

Proposed mechanism

Scheme 2 shows a suggested mechanism for the synthesis of indeno[1,2-b]indole-9,10-diones. Initially, enaminone (intermediate I) is produced by Knoevenagel condensation of aniline derivatives and dimedone which activated by meglumine sulfate. Subsequently, the reaction between intermediate I and ninhydrin produce intermediate II (iminium ion). The iminium ion undergoes tautomerization to form enamine, and the product is finally synthesized by intramolecular electrophilic cyclization.



Scheme 2. Reaction mechanism for the preparation of indeno[1,2-b]indole-9,10-diones using meglumine sulfate

Comparison of the performance of MS with other catalysts for the synthesis of 4b

A comparison between the performance of the presented method and other reported results (for the synthesis of 4b) was studied. As seen in Table 6, the meglumine sulfate catalyst outperforms other reported catalysts in terms of yield and reaction time. This superior performance is indicative of the catalyst's superior efficiency and effectiveness.

Table 6. Comparison of the catalytic efficiency of meglumine sulfate with other reported catalysts

Entry	Catalyst (amount)	Reaction conditions	Time (min)	Yield (%)[b]	Ref.
1	MMT@Fe_3O_4 (20 g)	H_2O, 70 °C	35	90	[15]
2	C@TiO_2 -SO_3H-IL1 (0.1g, 2.7 mol%)	H_2O, 60 °C	120	91	[24]
3	SnO_2QDs (10 mol %)	H_2O, 70 °C	150	87	[16]
4	Meglumine sulfate	H_2O, 75 °C	90	92	This work

[a]*Reaction conditions:* 4-chloroaniline (1 mmol), dimedone (1 mmol) and ninhydrin (1 mmol).
[b]Isolated yield.

Spectroscopic analysis of compound 4i

To confirm the structure of the prepared compounds, the ^{1}H NMR and ^{13}C NMR spectra with related data of 5-(3-chloro-4-fluorophenyl)-4b,9b-dihydroxy-7,7-dimethyl-4b,5,6,7,8,9b-hexahydroindeno[1,2-b]indole-9,10-dione (**4i**) has been displayed as follow:

^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 0.91 (3H, s, CH_3), 0.96 (3H, s, CH_3), 1.82 (1H, d, J = 16.0 Hz, CH), 1.90 (1H, d, J = 16.0 Hz, CH) ,2.14 (1H, d, J = 16.0 Hz, CH_2), 2.46 (1H, d, J = 16.0 Hz, CH_2), 6.10 (1H, s, OH), 6.72 (1H, d, J = 8.0 Hz, Ar-H), 7.27-7.23 (1H, m, Ar-H), 7.37 (1H, s, OH), 7.69-7.54 (4H, m, Ar-H), 7.74 (1H, d, J = 8.0 Hz, Ar-H).

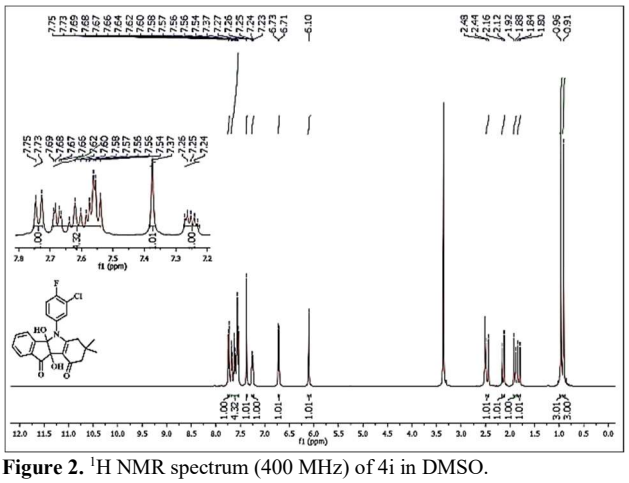


Figure 2. ^{1}H NMR spectrum (400 MHz) of 4i in DMSO.

^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 18.5, 26.2, 29.5, 33.4, 36.6, 51.2, 56.0, 83.3, 96.6, 106.0, 117.0, 117.2, 119.5, 119.7, 124.0 (d, J = 150.0 Hz), 130.8 (d, J = 98.0 Hz), 133.1, 133.2, 134.9 (d, J = 40.0 Hz), 146.8, 155.5, 157.9, 163.0, 189.4, 197.4.

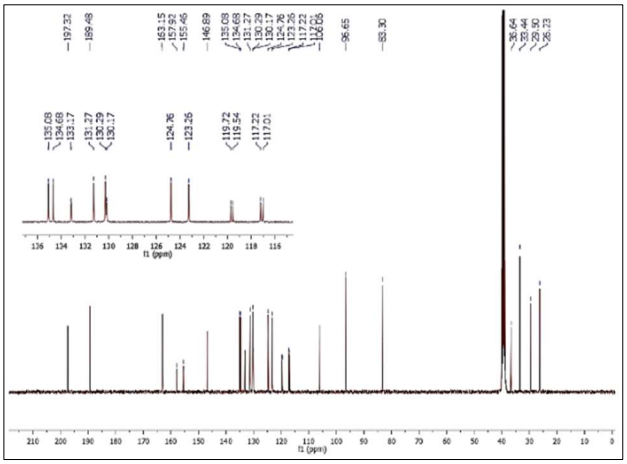


Figure 3. ^{13}C NMR spectrum (400 MHz) of 4i in DMSO.

4. Conclusions

In this research, we focused on synthesizing and characterizing meglumine sulfate using FT-IR and CHNS analysis. Subsequently, this homogeneous catalyst enabled the development of a facile, efficient, and straightforward process for synthesizing diverse indeno[1,2-b]indole-9,10-dione derivatives under mild thermal conditions (75 °C). By fine-tuning reaction parameters, we achieved high to excellent yields (80-97%) for various indeno[1,2-b]indole-9,10-dione derivatives, demonstrating the effectiveness of the optimized process. This study demonstrated the superior performance of meglumine sulfate in the synthesis of these compounds compared to existing methods.

Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contributions

Azam Moazeni Bistgani: Investigation, Writing-Original draft preparation, Visualization.
Leila Moradi: Supervision, Conceptualization, Writing-Reviewing and Editing.

Supporting Information

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