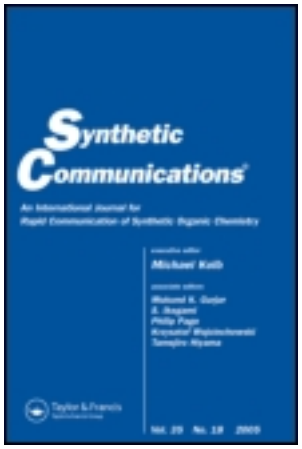

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Ultrasound-Promoted One-Pot Synthesis of 7-Aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one Derivatives

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ULTRASOUND-PROMOTED ONE-POT SYNTHESIS OF 7-ARYL-7,10,11,12-TETRAHYDROBENZO[c]ACRIDIN-8(9H)-ONE DERIVATIVES

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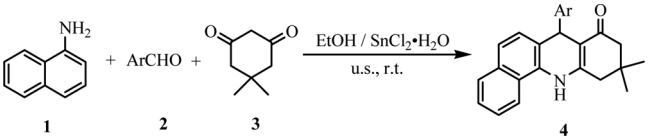
GRAPHICAL ABSTRACT



Abstract *A series of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives were synthesized via the three-component coupling from aromatic aldehydes, 1-naphthylamine, and 5,5-dimethylcyclohexane-1,3-dione catalyzed by stannous chloride dihydrate ($SnCl_2 \cdot 2H_2O$) under ultrasonic irradiation at room temperature. The products were isolated in good yields within short reaction times.*

Keywords Acridine; dimedone; 1-naphthylamine; one-pot reaction; ultrasonic irradiation

INTRODUCTION

The acridine derivatives have been known since the 19th century, when they were first used as pigments and dyes.[1] They are considered to be important chemotherapeutics as they have shown fungicidal, bactericidal, and antimalarial effects,[2] and multihydroacridineone derivatives have been reported to have high fluorescence efficiency and can be used as fluorescent molecular probes for monitoring of polymerization processes.[3] They are also increasingly receiving attention because of their likeness in properties with those of 1,4-dihydropyridines, which have similarities in structure to biologically important compounds such as nicotinamide adenine dinucleotide (NADH) and nicotinamide adenine dinucleotide phosphate (NADPH).[4] As a consequence, the interest of organic chemists in the synthesis or structural modifications of acridinedione derivatives remains high. Recently, some

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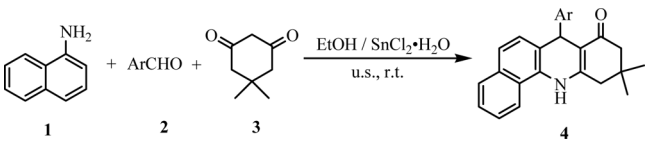

Scheme 1. Synthesis of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives.

developments for the synthesis of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives have been reported, such as by refluxing naphthylamine, dimedone, and aldehyde in benzene,[5] EtOH,[6] H_2O catalyzed by benzyltriethylammonium chloride (TEBA),[7] or under microwave irradiation.[8] These methods all have their own merits and shortcomings. Some methods are not very satisfactory because of drawbacks such as poor yields, high reaction temperature, and long reaction time. Stannous chloride dihydrate ($SnCl_2 \cdot 2H_2O$) is widely used as a reducing agent and as a Lewis acid catalyst, and its dihydrate has been employed as a mild reagent for the deprotection of acetals.[9]

In recent years, ultrasonic irradiation has been extensively applied in organic reactions because of its special sonochemical effect. The advantages of ultrasonic procedures, such as good yields, short reaction times, and mild reaction conditions, are well documented.[10] As we know, the temperature of hot spots caused by the collapse of acoustic caves is generally more than several hundred degrees, and this energy can be transferred to the organic molecules and absorbed to dramatically raise their intrinsic energy. Because of the thermal effect of ultrasonic waves, much larger amounts of molecules can meet the demand for the active energy in a given reaction, leading to the apparent improvement of the reaction efficiency with increased rates and reduced reaction time. It is also observed that reactions under ultrasonic irradiation are commonly easier to work up than those in conventional stirring methods. Herein we describe our example for the synthesis of 7-aryl-7,10,11,12 -tetrahydrobenzo[c]acridin-8(9H)-one derivatives from corresponding aldehydes, 1-naphthylamine, and dimedone using $SnCl_2 \cdot 2H_2O$ in EtOH under ultrasonic irradiation at room temperature (Scheme 1).

RESULTS AND DISCUSSION

We first screened various solvents for the synthesis of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives. The 4-chlorobenzaldehyde was selected as a representative reactant to optimize the reaction conditions (Table 1). We examined the effect of different solvents such as water, $CHCl_3$, CH_2Cl_2, $ClCH_2CH_2Cl$, dimethylformamide (DMF), tetrahydrofuran (THF), CH_3CN, 1,4-dioxane, and EtOH on a model reaction under ultrasonic irradiation at room temperature. The results are listed in Table 1 (entries 1–9). The reaction using EtOH as solvent gave the best result (Table 1, entry 9). To demonstrate the effect of ultrasound, the synthesis of **4a**, which is treated as a model, was investigated under stirring and reflux conditions (Table 1, entries 9–11). Under stirring and reflux conditions, the reaction can be completed within 1 and 2 h respectively, giving **4a** in 77% and 89% yields, whereas under ultrasonic irradiation (Table 1, entry 9), **4a** was

Table 1. Effect of different solvents for synthesis of **4a** using $SnCl_2 \cdot 2H_2O$

Entry[a]	Solvent	Condition	Time (h)	Yield (%)[b]
1	Water	rt	1	47
2	CH_2Cl_2	rt	1	51
3	$CHCl_3$	rt	1	59
4	$ClCH_2CH_2Cl$	rt	1	62
5	DMF	rt	1	63
6	THF	rt	1	56
7	CH_3CN	rt	1	77
8	1,4-Dioxane	rt	1	81
9	EtOH	rt	1	97
10	EtOH	Stirring, rt	1	77
11	EtOH	Reflux, 80 °C	2	89

[a]Reaction conditions: 1 mmol 1-naphthylamine, 1 mmol 4-chlorobenzaldehyde, 1 mmol dimedone, 0.2 mmol $SnCl_2 \cdot 2H_2O$, and 10 ml solvent.
[b]Isolated yield.

obtained in 97% yield within 1 h. It was apparent that the ultrasonic irradiation accelerated this transformation under milder conditions. The reason may be that the phenomenon of cavitation is produced by ultrasound. The effects of various catalysts such as $FeCl_3$, $ZnCl_2$, $NaHSO_4$, NH_2SO_3H, $SnCl_4 \cdot 5H_2O$, and $SnCl_2 \cdot 2H_2O$ or the absence of the catalyst were studied for this conversion, and the results are presented in Table 2. Of these catalysts, $SnCl_2 \cdot 2H_2O$ was found to be the most effective in terms of conversion. We selected the EtOH as solvent, using $SnCl_2 \cdot 2H_2O$ under ultrasonic irradiation conditions for the one-pot reaction of aldehydes, 1-naphthylamine, and dimedone, and put corresponding 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives at room temperature.

Next, various aromatic aldehydes were tested under the optimized reaction conditions (Scheme 1), and the results are listed in Table 3. In all cases, 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives were obtained in good yields. In the present procedure, aromatic aldehydes carrying either electron-donating or electron-withdrawing substituents in the benzene ring reacted very well. The present method was convincingly superior to the reported methods with respect to yield and reaction time.

Table 2. Effects of various catalysts on the preparation of **4a** under ultrasonic irradiation at room temperature

Entry	Catalyst	Time (h)	Yield (%)[a]
1	No	1	90
2	$FeCl_3$	1	89
3	$ZnCl_2$	1	91
4	$NaHSO_4$	1	86
5	NH_2SO_3H	1	90
6	$SnCl_4$	1	89
7	$SnCl_2 \cdot 2H_2O$	1	97

[a]Isolated yield.

Table 3. Synthesis of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives using $SnCl_2 \cdot 2H_2O$ under ultrasonic irradiation at room temperature

No.	Ar	Time (h)	Yield (%)[a]	Mp (°C)/(lit.) (°C)
4a	4-ClC_6H_4	1	97	268–270 (267–269)[5]
4b	2-ClC_6H_4	1	92	266–268 (265–266)[8]
4c	C_6H_5	1	85	261–263 (258–259)[8]
4d	2,4-$Cl_2C_6H_3$	1	89	285–287 (280–282)[7]
4e	2-$CH_3OC_6H_4$	1	95	267–269 (263–265)[5]
4f	4-$CH_3OC_6H_4$	1	88	259–261 (260–262)[5]
4g	2-HOC_6H_4	1	85	218–220 (220–222)[8]
4h	4-HOC_6H_4	1	86	285–287 (280–282)[8]
4i	3-$O_2NC_6H_4$	1	94	271–273 (267–269)[7]
4j	4-$O_2NC_6H_4$	1	88	281–283 (280–282)[5]
4k	3-CH_3O-4-OHC_6H_3	1	83	274–276 (270–273)[7]
4l	4-$(CH_3)_2NC_6H_4$	1	84	281–283 (276–278)[7]

[a]Isolated yield.

CONCLUSION

In conclusion, an efficient and facile method for preparation of 7-aryl-7,10,11,12-tetrahydrobenzo[c]acridin-8(9H)-one derivatives catalyzed by $SnCl_2 \cdot 2H_2O$ under ultrasonic irradiation at room temperature is presented. Ultrasound induces a remarkable acceleration for reactions. The products were isolated in good yields within short reaction times.

EXPERIMENTAL

All the chemicals were obtained from Tianjin Kermel Chemical Reagent Co., Ltd. and used as received. Melting points were recorded on an electrothermal apparatus and were uncorrected. Sonication was performed in Kunshan KQ-250B ultrasonic cleaner with a frequency of 40 kHz and a power of 250 W. The reaction flasks were located in the maximum energy area in the cleaner, and the addition or removal of water controlled the temperature of the water bath.

Typical Procedure for Preparation of Compound 4

In a 100-ml conical flask, a mixture of 1-naphthylamine (1 mmol), dimedone (1 mmol), and respective aldehydes (1 mmol) was mixed with EtOH (10 ml), and 0.2 mmol $SnCl_2 \cdot 2H_2O$ was added. The reaction mixture was irradiated in the water bath of the ultrasonic cleaner by an ultrasonic probe with a frequency of 40 KHz at room temperature (25 °C) for 1 h. The termination of the reaction was monitored by thin-layer chromatography (TLC). When the reaction was over, the mixture was poured into ice water (50 ml) and filtered to give the crude product, which was further purified by recrystallization from EtOH (**4a–4l**). All products were known compounds and were identified by comparison of their physical and spectroscopic data with those reported.[5,7,8]

Melting Point and Spectral Data for Compounds 4a–4l

Compound 4a. White crystal; mp 268–270 °C. IR (KBr, cm^{-1}): 3309, 2951, 1670, 1585, 1520, 1383, 1251, 1154, 1066, 1022, 854, 750; ^1H NMR (300 MHz, DMSO-d_6): 0.98 (3H, s, CH_3), 1.05 (3H, s, CH_3), 2.03–2.25 (2H, dd, $J = 12.5$ Hz, $J = 12.5$ Hz, CH_2), 2.60–2.71 (2H, dd, $J = 15.2$ Hz, $J = 15.2$ Hz, CH_2), 5.32 (1H, s, CH), 7.16–7.33 (5H, m, ArH), 7.48–7.61 (2H, m, ArH), 7.48–7.61 (2H, m, ArH), 8.46 (1H, d, $J = 8.5$ Hz, ArH), 9.38 (1H, s, NH); MS (m/z): 387. Anal. calc. for $C_{25}H_{22}NOCl$: C, 77.41; H, 5.72; N, 3.61. Found: C, 77.43; H, 5.75; N, 3.62.

Compound 4b. White crystal; mp 266–268 °C. IR (KBr, cm^{-1}): 3307, 2947, 2864, 1675, 1584, 1511, 1387, 1262, 1151, 1081, 1020, 846, 748; ^1H NMR (300 MHz, DMSO-d_6): 1.02 (3H, s, CH_3), 1.07 (3H, s, CH_3), 2.05–2.28 (2H, dd, $J = 12.5$ Hz, $J = 12.5$ Hz, CH_2), 2.57–2.73 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.83 (1H, s, CH), 6.86–7.65 (8H, m, ArH), 7.88 (1H, d, $J = 8.5$ Hz, ArH), 8.43 (1H, d, $J = 8.5$ Hz, ArH), 9.28 (1H, s, NH); MS (m/z): 387. Anal. calc. for $C_{25}H_{22}NOCl$: C, 77.41; H, 5.72; N, 3.61. Found: C, 77.45; H, 5.71; N, 3.64.

Compound 4c. White crystal; mp 261–263 °C. IR (KBr, cm^{-1}): 3309, 2949, 2861, 1674, 1595, 1511, 1380, 1253, 1152, 1088, 1022, 750; ^1H NMR (300 MHz, DMSO-d_6): 1.01 (3H, s, CH_3), 1.06 (3H, s, CH_3), 2.03–2.27 (2H, dd, $J = 12.5$ Hz, $J = 12.5$ Hz, CH_2), 2.57–2.78 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.87 (1H, s, CH), 6.85–7.63 (9H, m, ArH), 7.78 (1H, d, $J = 8.5$ Hz, ArH), 8.42 (1H, d, $J = 8.5$ Hz, ArH), 9.22 (1H, s, NH); MS (m/z): 353. Anal. calc. for $C_{25}H_{23}NO$: C, 84.95; H, 6.56; N, 3.96. Found: C, 84.93; H, 56.51; N, 3.93.

Compound 4d. White crystal; mp 285–287 °C. IR (KBr, cm^{-1}): 3310, 2949, 1685, 1586, 1517, 1487, 1397, 1261, 1151, 1087, 1023, 856, 760, 742; ^1H NMR (300 MHz, DMSO-d_6): 1.01 (3H, s, CH_3), 1.08 (3H, s, CH_3), 2.04–2.25 (2H, dd, $J = 12.5$ Hz, $J = 12.5$ Hz, CH_2), 2.67–2.75 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.74 (1H, s, CH), 7.16–7.63 (7H, m, ArH), 7.85 (1H, d, $J = 8.5$ Hz, ArH), 8.46 (1H, d, $J = 8.5$ Hz, ArH), 9.26 (1H, s, NH); MS (m/z): 421. Anal. calc. for $C_{25}H_{21}NOCl_2$: C, 71.10; H, 5.01; N, 3.32. Found: C, 71.11; H, 5.04; N, 3.41.

Compound 4e. White crystal; mp 267–269 °C. IR (KBr, cm^{-1}): 3295, 2952, 2886, 1681, 1589, 1518, 1497, 1412, 1260, 1171, 1143, 1026, 845, 809, 765, 747; ^1H NMR (300 MHz, DMSO-d_6): 1.03 (3H, s, CH_3), 1.10 (3H, s, CH_3), 2.08–2.23 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.59–2.74 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 3.81 (3H, s, CH_3O), 5.84 (1H, s, CH), 6.81–7.65 (8H, m, ArH), 7.83 (1H, d, $J = 8.5$ Hz, ArH), 8.42 (1H, d, $J = 8.5$ Hz, ArH), 9.25 (1H, s, NH); MS (m/z): 383. Anal. calc. for $C_{26}H_{25}NO_2$: C, 81.43; H, 6.57; N, 3.65. Found: C, 81.41; H, 6.51; N, 3.64.

Compound 4f. White crystal; mp 259–261 °C. IR (KBr, cm^{-1}): 3302, 2950, 2885, 1683, 1591, 1510, 1493, 1419, 1385, 1261, 1156, 1020, 835, 809, 762, 749; ^1H NMR (300 MHz, DMSO-d_6): 1.01 (3H, s, CH_3), 1.09 (3H, s, CH_3), 2.05–2.26 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.63–2.75 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 3.78 (3H, s, CH_3O), 5.76 (1H, s, CH), 6.75–7.65 (8H, m, ArH), 7.86 (1H, d, $J = 8.5$ Hz, ArH), 8.43 (1H, d, $J = 8.5$ Hz, ArH), 9.27 (1H, s, NH); MS (m/z):

383. Anal. calc. for $C_{26}H_{25}NO_2$: C, 81.43; H, 6.57; N, 3.65. Found: C, 81.48; H, 6.55; N, 3.68.

Compound 4g. White crystal; mp 218–220 °C. IR (KBr, cm^{-1}): 3305, 3178, 2945, 1685, 1590, 1515, 1491, 1389, 1267, 1154, 1023, 837, 760, 742; ^1H NMR (300 MHz, DMSO-d_6): 1.02 (3H, s, CH_3), 1.10 (3H, s, CH_3), 2.04–2.26 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.62–2.73 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.86 (1H, s, CH), 6.82–7.69 (8H, m, ArH), 7.76 (1H, d, $J = 8.5$ Hz, ArH), 8.35 (1H, d, $J = 8.5$ Hz, ArH), 9.22 (1H, s, NH),13.07 (1H, s, OH); MS (m/z): 383. Anal. calc. for $C_{25}H_{23}NO_2$: C, 81.27; H, 6.27; N, 3.79. Found: C, 81.28; H, 6.25; N, 3.73.

Compound 4h. White crystal; mp 285–287 °C. IR (KBr, cm^{-1}): 3309, 3156, 2942, 1683, 1595, 1522, 1494, 1384, 1265, 1159, 1023, 835, 767, 740; ^1H NMR (300 MHz, DMSO-d_6): 1.00 (3H, s, CH_3), 1.09 (3H, s, CH_3), 2.06–2.25 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.65–2.74 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.82 (1H, s, CH), 6.84–7.66 (8H, m, ArH), 7.78 (1H, d, $J = 8.5$ Hz, ArH), 8.34 (1H, d, $J = 8.5$ Hz, ArH), 9.24 (1H, s, NH), 13.04 (1H, s, OH); MS (m/z): 383. Anal. calc. for $C_{25}H_{23}NO_2$: C, 81.27; H, 6.27; N, 3.79. Found: C, 81.25; H, 6.22; N, 3.77.

Compound 4i. White crystal; mp 271–273 °C. IR (KBr, cm^{-1}): 3308, 2952, 1678, 1591, 1526, 1495, 1381, 1354, 1268, 1152, 1087, 1028, 815, 762, 732; ^1H NMR (300 MHz, DMSO-d_6): 0.99 (3H, s, CH_3), 1.10 (3H, s, CH_3), 2.05–2.28 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.71–2.77 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.74 (1H, s, CH), 7.34–7.86 (7H, m, ArH), 7.98–8.05 (2H, m, ArH), 8.45 (1H, d, $J = 8.5$ Hz, ArH), 9.31 (1H, s, NH); MS (m/z): 398. Anal. calc. for $C_{25}H_{22}N_2O_3$: C, 75.36; H, 5.57; N, 7.03. Found: C, 75.35; H, 5.51; N, 7.09.

Compound 4j. White crystal; mp 281–283 °C. IR (KBr, cm^{-1}): 3310, 2957, 1670, 1594, 1523, 1495, 1384, 1352, 1269, 1149, 1083, 1030, 811, 760, 722; ^1H NMR (300 MHz, DMSO-d_6): 0.99 (3H, s, CH_3), 1.11 (3H, s, CH_3), 2.06–2.26 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.69–2.75 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 5.76 (1H, s, CH), 7.15–7.74 (7H, m, ArH), 7.95–8.03 (2H, m, ArH), 8.40 (1H, d, $J = 8.5$ Hz, ArH), 9.29 (1H, s, NH); MS (m/z): 398. Anal. calc. for $C_{25}H_{22}N_2O_3$: C, 75.36; H, 5.57; N, 7.03. Found: C, 75.30; H, 5.54; N, 7.06.

Compound 4k. White crystal; mp 274–276 °C. IR (KBr, cm^{-1}): 3302, 2959, 1574, 1510, 1498, 1394, 1263, 1144, 1093, 812, 768, 650; ^1H NMR (300 MHz, DMSO-d_6): 1.02 (3H, s, CH_3), 1.10 (3H, s, CH_3), 2.07–2.28 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.65–2.76 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 3.76 (3H, s, CH_3O), 5.48 (1H, s, CH), 6.98–7.51 (7H, m, ArH), 7.85 (1H, d, $J = 8.5$ Hz, ArH), 8.43 (1H, d, $J = 8.5$ Hz, ArH), 8.69 (1H, s, OH), 9.27 (1H, s, NH); MS (m/z): 399. Anal. calc. for $C_{26}H_{25}NO_3$: C, 78.17; H, 6.31; N, 3.51. Found: C, 78.24; H, 6.39; N, 3.42.

Compound 4l. White crystal; mp 281–283 °C. IR (KBr, cm^{-1}): 3298, 2954, 1679, 1578, 1514, 1391, 1261, 1146, 1063, 957, 827, 798, 752; ^1H NMR (300 MHz, DMSO-d_6): 1.01 (3H, s, CH_3), 1.09 (3H, s, CH_3), 2.05–2.26 (2H, dd, $J = 8.5$ Hz, $J = 8.5$ Hz, CH_2), 2.67–2.75 (2H, dd, $J = 15.0$ Hz, $J = 15.0$ Hz, CH_2), 2.86 (6H, s, $2NCH_3$), 5.51 (1H, s, CH), 7.03–7.65 (8H, m, ArH), 7.83 (1H, d, $J = 8.5$ Hz,

ArH), 8.46 (1H, d, $J = 8.5$ Hz, ArH), 9.22 (1H, s, NH); MS (m/z): 396. Anal. calc. for $C_{27}H_{28}N_2O$: C, 81.78; H, 7.12; N, 7.06. Found: C, 81.70; H, 7.14; N, 6.99.

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