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Short Communication

A facile synthesis of deuteroporphyrins derivatives under ultrasound irradiation

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ABSTRACT

A facile, efficient and general method for preparing deuteroporphyrin derivatives by using concentrated H_2SO_4 and alcohol under ultrasound irradiation has been developed. A series of new deuteroporphyrin derivatives bearing different propionic ester groups have been synthesized in good yields starting from readily accessible deuterohemin. The characterization of these compounds confirms the synthetic methodology. Compared with conventional methods, the main advantages of the present procedure are shorter reaction time and higher yields.

1. Introduction

Porphyrins and phthalocyanines peripherally bearing with different substituents are of great interest in areas [1–6] as diverse as biomimetic reactions, biological and photobiological processes, photodynamic therapy, energy migration, analytical chemistry and so on. The nature and location of the substituents in porphyrin rings have a great influence on their properties. For this reason considerable efforts have been devoted to the synthesis of porphyrin derivatives bearing various substituents to improve their performance [7–11]. However, to the best of our knowledge, the reported methods are all inconvenient and time-consuming. As described in the literature, deuteroporphyrin IX dimethyl ester (DPDME) is generally synthesized from deuterohemin by the procedure of Chu and Dolphin [12,13]. The procedure involves two steps: demetalation of deuterohemin by the mixture of glacial CH_3COOH and concentrated HCl and esterification of deuteroporphyrin with CH_3OH–H_2SO_4 (20:1) overnight, with the total yield of 46.5–80% (Scheme 1). And other deuteroporphyrins bearing two propionic ester groups are always prepared from DPDME [14,15]. Another method for the synthesis of these deuteroporphyrin derivatives was reported by Caughey and coworkers [16], these compounds could be prepared from deuterohemin using anhydrous $FeSO_4$, dry HCl and methanol in one-pot, with a yield of only 66%. Thus, the development of a simple, highly efficient methodology for the synthesis of deuteroporphyrin derivatives remains desired.

Recently, ultrasound has been successfully applied in various organic reactions [17]. Many papers have indicated that some organic reactions can be carried out in high yields, short reaction times and mild conditions under ultrasound irradiation [18–20]. Therefore, "sonochemistry" has been a new trend in organic chemistry, offering a versatile and facile pathway for a large variety of synthesis [21,22]. We have now developed a convenient method for the synthesis of deuteroporphyrin derivatives directly from deuterohemin under ultrasound irradiation in one-pot based on alcohols and concentrated H_2SO_4 (Scheme 1). We describe here the successful use of this reaction procedure to prepare a series of deuteroporphyrins bearing different propionic ester groups in high yields.

2. Method

2.1. Apparatus and analysis

All reagents were analytical purity obtained from commercial sources and used without further purification unless otherwise stated. Deuterohemin chloride was synthesized from hemin chloride following the reference procedures [12,16]. Ultrasonication was performed in a KQ-250B ultrasound cleaner with a frequency of 40 kHz and an output power of 250 W. High speed stirring was carried out with the Biaoma JB50-D series aggregating heat constant temperature blender. Melting points were determined on a XT4 micro hot-stage apparatus and was uncorrected; ^1H NMR was recorded on a Bruker 500 MHz spectrometers in $CDCl_3$ with tetramethylsilane (TMS) as an internal standard. Elemental analysis was conducted on an PE-2004 (Perkin–Elmer) elemental

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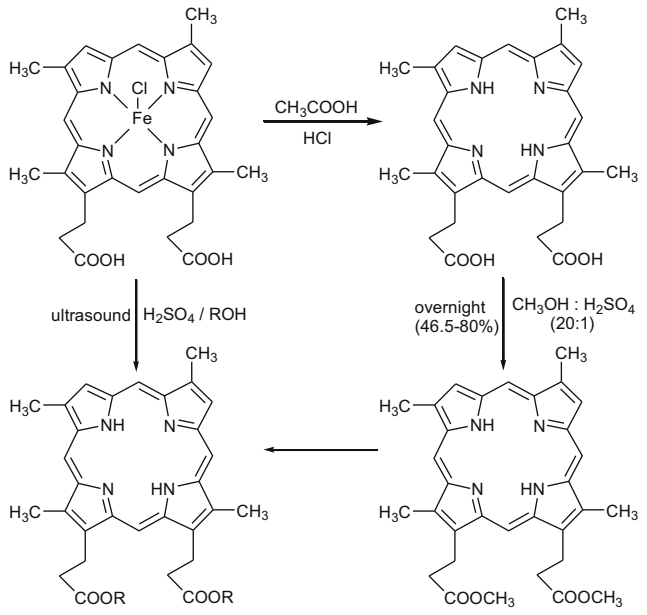

Scheme 1. Synthesis of deuteroporphyrin derivatives bearing different propionic ester groups.

analyzer. ESI-MS/MS mass spectra were recorded on a Finnigan TSQ Quantum ultra AM mass spectrometer. Infrared spectra were obtained on a Perkin–Elmer 681 instrument.

2.2. Conventional synthesis of deuteroporphyrin derivatives

To the mixture of deuterohemin chloride (1.0 g, 1.67 mmol) and concentrated H_2SO_4 (15 ml, 0.28 mol) in a boiling florence 3-neck-flask of 150 ml under the condition of mechanical stirring, excess alcohol (0.6 mol) was added dropwise (15 min) at refluxing. The reaction was monitored by TLC. After the reaction, the result mixture was stored in refrigeratory for more than 2 h to cool down. After neutralization by cooled ammonia, the mixture was then extracted with CH_2Cl_2 (100 ml, three times). The organic layer was washed with brine, dried over anhydrous Na_2SO_4. After solvent removal, the residue was further purified by column chromatography on silica gel with dichloromethane: ethyl acetate = 10:1 to afford product as a pure solid.

2.3. Ultrasound-promoted synthesis of deuteroporphyrin derivatives

To the mixture of deuterohemin chloride (1.0 g, 1.67 mmol) and concentrated H_2SO_4 (15 ml, 0.28 mol) in a boiling florence 3-neck-flask of 150 ml, excess alcohol (0.6 mol) was added dropwise (15 min) at room temperature in an ultrasound bath having a frequency of 40 kHz. After the addition, the mixture was irradiated by ultrasound for another 1 h. Then the result mixture was stored in refrigeratory for more than 2 h to cool down. After neutralization by cooled ammonia, the mixture was then extracted with CH_2Cl_2 (100 ml, three times). The organic layer was washed with brine, dried over anhydrous Na_2SO_4. After solvent removal, the residue was further purified by column chromatography on silica gel with dichloromethane: ethyl acetate = 10:1 to afford product as a pure solid.

Deuteroporphyrins IX dimethyl ester (1) mp 224–225 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ/ppm = −3.87 (s, 2H), 3.30, 3.29,3.27(t, J = 7.25, 4H), 3.73, 3.75 (2s, 6H), 3.63–3.66 (4s, 12H), 4.41, 4.42, 4.44 (t, J = 7.25, 4H), 9.08, 9.09 (2s, 2H), 10.03, 10.07, 10.10, 10.13 (4s, 4H); **IR** (KBr, cm^{-1}): 3400 (m, N–H), 2900 (w), 1733 (s, C=O), 1435 (m), 1361 (m), 1300 (w), 1235 (w), 1196 (m), 1165 (s, C–O), 1125 (m), 1055 (w), 1016 (m), 970 (m), 894 (w), 845 (s); ESI$^+$-MS (42 eV, m/z): 539.1 [M+H]$^+$, 524.1 [M+H−CH_3]$^+$, 451.1 [M+H−$CH_2CH_2COOCH_3$]$^+$. Anal. Calcd. for $C_{32}H_{34}N_4O_4$: C, 71.36; H, 6.36; N, 10.40. Found: C, 71.28; H, 6.40; N, 10.29.

Deuteroporphyrins IX diethyl ester (2) mp 199–200 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.92 (s, 2H), 1.12, 1.14, 1.15 (t, J = 7.25 Hz 6H), 3.26, 3.27, 3.29 (t, J = 7.75, 4H), 3.63, 3.65, 3.73, 3.74 (4s, 12H), 4.12, 4.13, 4.15, 4.16 (m, J = 7.25, 4 H), 4.43, 4.42, 4.4 (t, J = 7.75, 4H), 9.08 (s, 2H), 10.02, 10.06, 10.11, 10.12 (s, 4H). **IR** (KBr, cm^{-1}): 3452 (w, N–H), 3309 (m, C–H($C_{3,8}$)); 2911 (w, –CH_3), 1730 (s, C=O), 1444 (m, –CH_2–), 1172(s, C–O), 839(m, C–H($C_{5,10,15,20}$)); ESI$^+$-MS (40 eV, m/z): 567.1 [M+H]$^+$, 552.1 [M+H−CH_3]$^+$, 466.0 [M+H−$CH_2CH_2COOCH_2CH_3$]$^+$. Anal. Calcd for $C_{34}H_{38}N_4O_4$: C, 72.06; H, 6.76; N, 9.89. Found: C, 71.98; H, 6.80; N, 9.82.

Deuteroporphyrins IX dipropyl ester (3) mp 194–195 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.93 (s, 2H), 0.76–0.79 (m, J = 6.67 Hz, 6H), 1.50–1.57 (m, 4H), 1.12–1.15 (m, J = 8.75 Hz, 4H), 3.26, 3.27, 3.29 (t, J = 7.25, 2H), 3.62, 3.64, 3.72, 3.73 (4s, 12H), 4.03, 4.05, 4.06 (t, J = 6.75 Hz, 4H), 4.40, 4.41, 4.42 (t, J = 7.25 Hz, 4H), 9.07 (s, 2H), 10.00, 10.04, 10.08, 10.09 (4s, 4H). **IR** (KBr, cm^{-1}): 3566 (w, N–H), 3306 (m, C–H($C_{3,8}$)); 2964 (w, –CH_3), 1735 (s, C=O), 1455 (m, –CH_2–), 1170 (s, C–O), 839 (m, C–H($C_{5,10,15,20}$)); ESI$^+$-MS (40 eV, m/z): 595.1 [M+H]$^+$, 553.1 [M+H−$CH_2CH_2CH_3$]$^+$, 479.3 [M+H−$CH_2CH_2COOCH_2CH_2CH_3$]$^+$. Anal. Calcd for $C_{36}H_{42}N_4O_4$: C, 72.70; H, 7.12; N, 9.42. Found: C, 72.56; H, 7.26; N, 9.55.

Deuteroporphyrins IX diisopropyl ester (4) mp 219–220 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.91 (s, 2H), 1.10–1.12 (d, J = 6.0 Hz, 12H), 3.24, 3.25, 3.27 (t, J = 7.25 Hz, 4H), 3.64, 3.67, 3.73, 3.76 (4s, 12H), 4.40, 4.42, 4.43(t, J = 7.25 Hz, 4H), 5.03–5.10 (m, J = 6.0 Hz, 2H), 9.09 (d, 2H), 10.03, 10.08, 10.13 10.14(4s, 4H). **IR** (KBr, cm^{-1}): 3567 (w, N–H), 3311 (m, C–H($C_{3,8}$)); 2973 (w, –CH_3), 1771 (s, C=O), 1456 (m, –CH_2–), 1174 (s, C–O), 840 (m, C–H($C_{5,10,15,20}$)); ESI$^+$-MS (45 eV, m/z): 595.1 [M+H]$^+$, 553.1 [M+H−$CH_2CH_2CH_3$]$^+$, 479.3 [M+H−$CH_2CH_2COOCH_2CH_2CH_3$]$^+$. Anal. Calcd for $C_{36}H_{42}N_4O_4$: C, 72.70; H, 7.12; N, 9.42. Found: C, 72.60; H, 7.22; N, 9.53.

Deuteroporphyrins IX dibutyl ester (5) mp 189.5–190 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.90 (s, 2H), 0.70, 0.71, 0.73 (t, J = 7.25 Hz, 6H), 1.15–1.22 (m, J = 7.75 Hz, 4H), 1.44–1.50 (m, J = 7.25 Hz, 4H), 3.26, 3.27, 3.29 (t, J = 7.75 Hz, 4H), 3.62, 3.64, 3.72, 3.74 (4s, 12H), 4.06, 4.07, 4.08 (t, J = 7.25 Hz, 4H), 4.40, 4.41, 4.43(t, J = 7.25 Hz, 4H), 9.07, 9.08 (2s, 2H), 10.01, 10.05, 10.10 10.11 (s, 4H). **IR** (KBr, cm^{-1}): 3447 (w, N–H), 2959 (w, –CH_3), 1730 (s, C=O), 1462 (m, –CH_2–), 1169 (s, C–O), 804 (m, C–H($C_{5,10,15,20}$)); ESI$^+$-MS (45 eV, m/z): 623.1 [M+H]$^+$, 567.1 [M+H−$(CH_2)_3CH_3$]$^+$, 492.8 [M+H−$CH_2CH_2COO(CH_2)_3CH_3$]$^+$. Anal. Calcd. for $C_{38}H_{46}N_4O_4$: C, 73.28; H, 7.44; N, 9.00. Found: C, 73.14; H, 7.56; N, 9.12.

Deuteroporphyrins IX diisobutyl ester (6) mp 188–188.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.89 (s, 2H), 0.78–0.80 (m, 12H), 1.79–1.87 (m, 2H), 3.28, 3.39, 3.41 (t, J = 8.0 Hz, 4 H), 3.64, 3.66, 3.73, 3.76 (m, 12H), 3.88–3.89 (d, J = 8.0 Hz, 4H), 4.42, 4.43, 4.44 (t, 4H), 9.09, 9.10 (d, 2H), 10.03, 10.07, 10.11, 10.14 (4s, 4H). **IR** (KBr, cm^{-1}): 3452 (w, N–H), 3312 (m, C–H($C_{3,8}$)); 2959 (w, –CH_3), 1736(s, C=O), 1379 (m, –CH_2–), 1169 (s, C–O), 845 (m, C–H($C_{5,10,15,20}$)); ESI$^+$-MS (45 eV, m/z): 623.1 [M+H]$^+$, 567.2 [M+H−$CH(CH_3)_2$]$^+$, 433.0 [M+H−CH_2CH_2COO $CH(CH_3)_2$−$4CH_3$]$^+$. Anal. Calcd. for $C_{38}H_{46}N_4O_4$: C, 73.28; H, 7.44; N, 9.00. Found: C, 73.16; H, 7.55; N, 9.08.

Deuteroporphyrins IX diisooctyl ester (7) mp 110–111 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.90 (s, 2H), 0.84–0.88 (m, 12H), 1.02–1.06 (m, 12H), 1.12–1.15 (m, 4H), 1.25 (s, 2H), 1.38–1.40 (m, 4H), 3.26, 3.28, 3.29 (t, J = 7.25 Hz, 4H), 3.62–3.74

(m, 12H), 3.95–4.01 (m, 4H), 4.40, 4.41, 4.43 (t, J = 7.25 Hz, 4H), 9.07, 9.08 (d, 2H), 10.01, 10.05, 10.09, 10.11 (s, 4H). **IR** (KBr, cm^{-1}): 3524 (w, N–H), 3316 (m, C–H($C_{3,8}$)); 2928 (w, $-CH_3$), 1736 (s, C=O), 1460 (m, $-CH_2-$), 1167 (s, C–O), 860 (m, C–H($C_{5,10,15,20}$)); ESI^+-MS (50 eV, m/z): 735.5 $[M+H]^+$, 623.1 $[M+H-CH(CH_3)(CH_2)_5CH_3]^+$, 511.1 $[M+H-2CH(CH_3)(CH_2)_5CH_3]^+$. Anal. Calcd. for $C_{46}H_{62}N_4O_4$: C, 75.17; H, 8.50; N, 7.62. Found: C, 75.08; H, 8.64; N, 7.79.

Deuteroporphyrins IX tert-butyl ester (8) mp 236.5–237.5 °C; ^1H NMR (500 MHz, $CDCl_3$) δ/ppm = −3.94 (s, 2H), 0.64–0.68 (m, 12H), 1.24–1.29 (m, 9H), 3.70, 3.72, 3.73, 3.74 (m, J = 7.0 Hz, 4H), 4.50, 4.51, 4.52, 4.54 (m, J = 6.67 Hz, 4H), 9.06, 9.08 (d, 2H), 10.03, 10.05, 10.08, 10.12(s, 4H), 12.11(s, 1H). **IR** (KBr, cm^{-1}): 3526 (w, N–H), 3423 (w, O–H), 3317 (m, C–H($C_{3,8}$)); 2925 (w, $-CH_3$), 1734(s, C=O), 1462 (m, $-CH_2-$), 1164 (s, C–O), 861 (m, C–H($C_{5,10,15,20}$)); ESI^+-MS (38 eV, m/z): 567.0 $[M+H]^+$, 511.0 $[M+H-C(CH_3)_3]^+$, 493.1 $[M+H-C(CH_3)_5CH_3-H_2O]^+$. Anal. Calcd. for $C_{46}H_{62}N_4O_4$: C, 72.05; H, 6.76; N, 9.89. Found: C, 71.68; H, 6.64; N, 9.96.

Table 1
Synthesis of deuteroporphyrin IX dimethyl ester.[a]

Entry	V^b	r^c(mol)	Yield[d] (%)
1	10	110	88
2	15	165	97
3	20	220	96
4	25	275	92
5	30	330	91

[a] Reaction conditions: deuterohemin chloride (1.0 g, 1.67 mmol), methanol (25 ml, 0.6 mol), reaction time 60 min, rt.
[b] The volume of concentrated H_2SO_4.
[c] The mol ratio of H_2SO_4 to deuterohemin chloride.
[d] Yields refer to the isolated products.

Table 2
Effect of the power of ultrasound irradiation on the reaction.[a]

Ultrasonic power (W)	100	150	200	250
Yield[b](%)	41	56	75	97

[a] Reaction conditions: deuterohemin chloride (1.0 g, 1.67 mmol), methanol (25 ml, 0.6 mol), concentrated H_2SO_4 (15 ml, 0.28 mol), reaction time 60 min, rt.
[b] Yields refer to the isolated products.

3. Results and discussion

To optimize the reaction conditions, the ratio of reactants was investigated in the synthesis of DPDME (1) as model compound. The best result was obtained by the reaction of deuterohemin (1.0 g), methanol (25 ml) and concentrated H_2SO_4 (15 ml) at room temperature under silent condition by ultrasound irradiation (40 kHz) to produce DPDME in 1 h with 97% yield. In the reaction, the concentrated H_2SO_4 played an important role as demetalation reagent and absorber of H_2O produced in the reaction. The effect of the content of concentrated H_2SO_4 is shown in Table 1. The yields of deuteroporphyrin increased with the increase of H_2SO_4 at the beginning, when the volume of H_2SO_4 was 15 ml, the yield reached the highest (97%). However, the yields decreased when the content of H_2SO_4 unceasingly increased, the phenomenon is due to the fact that the esterification is reversible and the superfluous H_2SO_4 may help the hydrolysis of the product.

In addition, the influence of the power of ultrasound irradiation on the reaction has also been studied. Raising the ultrasound power in our study by from 100, 150, 200 to 250 W, we could identify an increase of the yield of DPDME (Table 2). The sonication power influences the level of cavitations produced by ultrasound in the liquid. Higher power ultrasound generates the larger number of cavitation events due to more transient cavitations bubbles being formed and causes the reaction rapidly.

To investigate the scope and generality of the present method, various alcohols were tested for the reactions with deuterohemin under the optimum conditions. The results showed that these reactions proceeded smoothly to give the expected compounds in good to excellent yields. In addition, the comparative study of the one-pot reaction under conventional high speed stirring conditions was also carried out to investigate the specific effect of ultrasonic on these reactions and the repre-

Table 3
Synthesis of deuteroporphyrin derivatives under ultrasound and conventional stirring.[a]

Entry	Alcohol	Ultrasound		Conventional	
		t/min	Yield[b](%)	t/h	Yield[b](%)
1	H_3C-OH	60	97	7	90
2	H_3C-CH_2-OH	60	95	7	89
3	$H_3C-CH_2-CH_2-OH$	60	96	7	88
4	$H_3C-CH(CH_3)-OH$	100	89	14	69
5	$H_3C-CH_2-CH_2-CH_2-OH$	100	94	12	76
6	$H_3C-CH_2-CH(CH_3)-OH$	120	88	16	56
7	$H_3C-CH_2-CH_2-CH_2-CH_2-CH(CH_3)-OH$	120	84	16	52
8	$H_3C-C(CH_3)_2-OH$	120	68[c]	16	31[c]

[a] Reaction conditions: deuterohemin chloride (1.0 g, 1.67 mmol), concentrated H_2SO_4 (15 ml, 0.28 mol), alcohol (0.6 mol), rt.
[b] Yields refer to the isolated products.
[c] Yields of monoester.

sentative examples are summarized in Table 3. From Table 3, it is found that under conventional conditions the yield of deuteroporphyrins varied with the structures of alcohols. For example, when the alcohol was isooctanol, the yields of deuteroporphyrins IX diisooctyl ester (7) was only 52%, markedly lower than others. That is to say, steric factors played a key role in affecting the yield and rate of reaction under conventional conditions. However, the yields of products changed little under ultrasound irradiation conditions. These results indicate that ultrasound irradiation could evidently reduce the steric influence of reactant on the reaction compared with conventional conditions, in accord with the results of Zengs' [23]. To gain further insight, we performed the reactions with tert-butyl alcohol under the same reaction conditions. The central iron has been absolutely taken off after irradiation for 2 h, but the esterification was incompleted. The monoester was the dominant reaction product, suggesting that the reduction of dimensional effect is limited for the reaction with more hindered alcohols.

Another influence of ultrasound on chemical reaction is that it can markedly shorten the reaction time compared with conventional conditions [24–26]. The similar effect was also observed in our experiments. The results in Table 3 show that ultrasound is much more efficient than magnetic stirring. Clearly, sluggish reactions were observed under conventional conditions and longer reaction time was required to achieve better yield. However, to our delight, ultrasound irradiation efficiently accelerated the reaction and markedly shortened the reaction time. In case of methanol reaction time was reduced from 7 h to 60 min to get the similar yield (Table 3, entry 1). Likewise with other alcohols with more carbon atoms, a considerable shortening of reaction time is observed (Table 3, entries 2–7). These results indicated that there was remarkable ultrasonic effect on the one-pot reaction. We presume that the efficiency using ultrasound irradiation is due to the cavitations phenomena. Cavitation was the production of microbubble in liquid when a large negative pressure was applied to it. In succeeding compression cycles bubbles can collapse violently with the release of large amounts of energy. The "hot-spot" theory suggested that temperature of up to 5000 K and pressures of several thousand atmospheres were produced during this collapse [27,28]. The energy being more efficiently transmitted to the substrates compared to the reactions performed at conventional conditions and cause reaction rapidly. Also, the collapse of bubbles induces the efficient mixing, increase in mass transfer and mechanical stress that can be transmitted to a target single bond and accelerates the reaction.

4. Conclusion

In conclusion, we have developed a new general and convenient method for synthesis of a series of deuteroporphyrin derivatives in high yields under ultrasound irradiation. The method leads to a series of porphyrins bearing different propionic ester groups. Ultrasounds induce a remarkable acceleration for these reactions, the reaction times decreasing dramatically and the yields increasing considerably.

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