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A VERSATILE AND EFFICIENT METHODOLOGY FOR THE PREPARATION OF CHOLINE ESTER AUXIN CONJUGATES

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Abstract—A two-step synthetic strategy affords indole-3-acetylcholine (IAC) from indole-3-acetic acid (IAA) via the intermediacy of 2-dimethylaminoethyl indole-3-acetate. Thus, treatment of indole-3-acetate with 1-dimethylamino-2-chloroethane, followed by methylation with methyl iodide, yields 55–70% recrystallized IAC from IAA. Using the same methodology, six analogues of IAC have been prepared in overall yields ranging from 40 to 60%.

INTRODUCTION

Derivatives of choline serve important functions in plant physiology. Phosphatidylcholine is a key component of plant cellular membranes, and acetylcholine has been found in the tissues of many plants [1]. Indole-3-acetic acid (IAA) is a potent plant growth hormone, and examples of IAA conjugates with plant activity abound in the literature [2]. According to the 'hormonal homeostasis theory', IAA conjugates play a vital role in regulating the level of free IAA available to a growing plant [3]. IAA conjugates have been implicated in other plant hormone metabolic functions as well: (1) transport of IAA, (2) storage and subsequent re-use of IAA, and (3) protection of IAA from enzymic destruction [4].

RESULTS AND DISCUSSION

We have recently reported that indole-3-acetylcholine iodide (**1**), a novel analogue of acetylcholine and an IAA conjugate, promotes pea stem elongation at concentrations as low as 1 μM [5]. We now wish to report on the synthetic strategy and methodology that we have devised for the preparation of **1**. Our synthetic strategy (Scheme 1) identifies 2-dimethylaminoethyl indole-3-acetate (**2**) as a key intermediate that can be accessed *via* esterification of indole-3-acetic acid (IAA). Although our initial synthetic route to the intermediate involved boron trifluoride-catalysed Fischer esterification of IAA with 2-dimethylaminoethanol [5], we have obtained a significant yield improvement by treatment of indole-3-acetate with 1-dimethylamino-2-chloroethane (DMAC). The new methodology affords routine overall yields of 55–70% recrystallized IAC (**1**) from IAA.

The structure proofs of **2** and **1**, both hitherto unreported in the literature,‡ rest primarily on NMR spectroscopy, both ^{1}H and ^{13}C (Tables 1 and 2). The NMR assignments for the aromatic portion of IAA were based on indole. The acid portion of esters **1** and **2** was modelled by IAA; the alcohol portion of **2** by 2-dimethylaminoethanol (DMAE), and the corresponding portion of **1** by choline iodide (ChI). The only significant deviation between the ^{1}H NMR spectrum of **2** and that of DMAE involves the 0.65 ppm downfield shift of the OCH_2 hydrogens (δ3.45 in DMAE versus δ4.1 in **2**). In the ^{13}C NMR spectrum of **2** the CH_2O carbon is shifted downfield by 3.0 ppm whereas the CH_2N^+ carbon is shifted upfield by 4.2 ppm. The methylene carbons in both DMAE and **2** have been differentiated by means of a 2D HETCOR pulse sequence.

The ^{1}H NMR spectrum of **1** is not appreciably different from those of the two constituents IAA and ChI except that the CH_2O hydrogens in **1** are shifted downfield 0.65 ppm relative to the corresponding hydrogens in ChI. Two carbons in the alcohol portion of **1** have ^{13}C NMR chemical shifts slightly different from the analogous carbons in ChI. The CH_2O carbon in **1** is shifted downfield by 3.0 ppm whereas the CH_2N+ carbon in **1** is shifted upfield by 3.1 ppm. The carbons adjacent to the quaternary nitrogen have been unambiguously identified by an equal intensity triplet structure due to splitting by ^{14}N.

The two-step methodology outlined in Scheme 2 for the synthesis of IAC from IAA is extremely versatile. Thus, we have extended this methodology to the prep-

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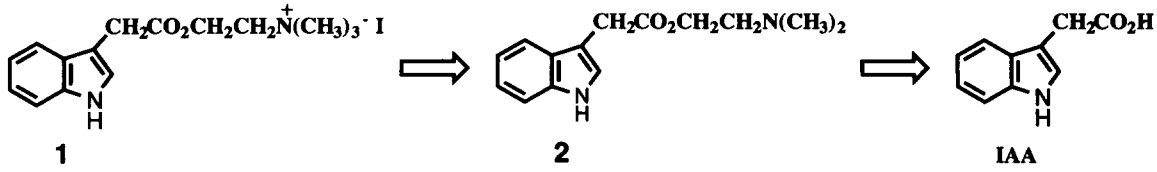
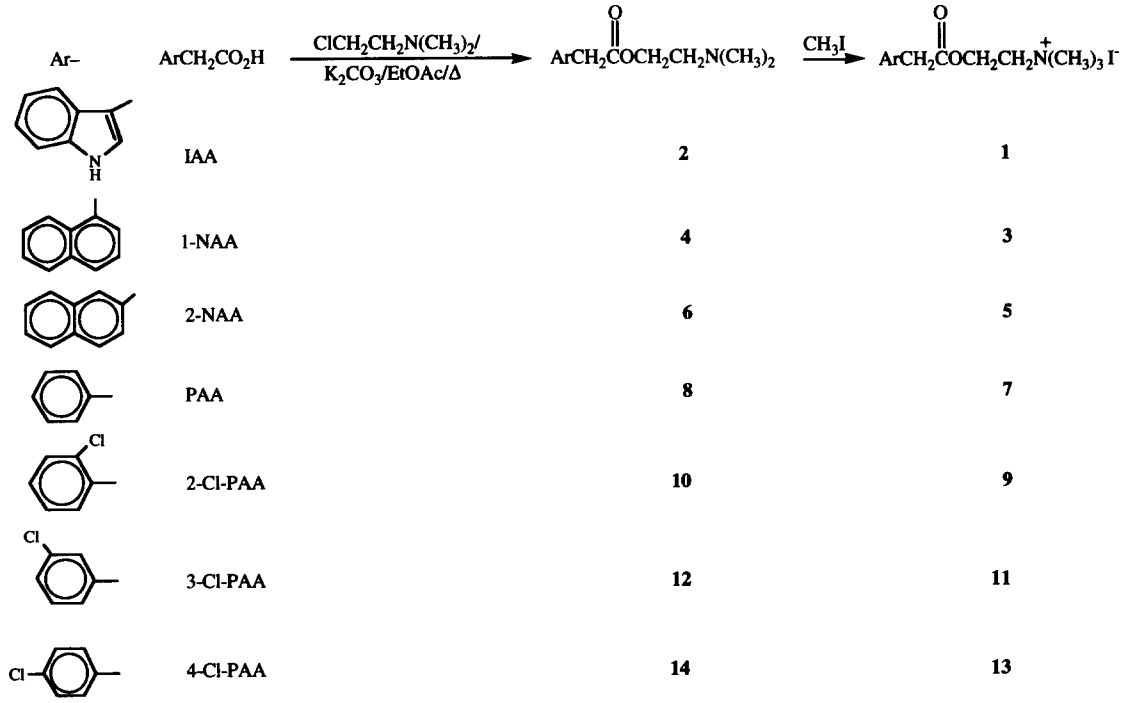
‡The hydrochloride salt of compound **2** has been cited in one previous reference: Thuillier, G. and Rumpf, P. (1960) *Bull. Soc. Chim. Fr.* 1789. 'Indolylacetylcholine', presumably the bromide salt, was prepared according to the method of Fourneau and Page (1914) *Bull. Soc. Chim. Fr.* **15**, 544; Keyl, M. J., Michaelson, I. A. and Whittaker, V. P. (1957) *J. Physiol.* **139**, 434. However, the only characterization of the compound reported was the ion exchange chromatographic retention volume.



Scheme 1. Strategy for IAC (1) synthesis.



Scheme 2. Synthetic methodology for conversion of auxins to choline ester conjugates.

aration of other choline esters of auxin-like acids, such as naphthalene-1-acetic acid (1-NAA) and phenylacetic acid (PAA). We have also prepared the choline conjugate of the anti-auxin 2-NAA and of the three isomeric monochloro PAAs. The effect of a halogen, especially chlorine, on the auxin activity of PAA is well documented [6]. Routine yields of 40–60% of choline esters were obtained after modification of the methylation procedure by substituting diethyl ether for ethyl acetate as the solvent to minimize the formation of the tetramethylammonium iodide side product.

We have previously proposed a theoretical model to account for the observed pea stem segment elongation induced by IAC [5]. In this model, IAC is hydrolysed by cholinesterase (ChE) in the plant, and subsequently IAA, one of the two resulting hydrolysis products, stimulates plant growth. Hydrolysis catalysed by ChE can be differentiated from other catalysed hydrolysis processes by incubating pea stem segments with neostigmine, a competitive inhibitor of ChE activity. While neostigmine does not inhibit the action of IAA, it does suppress IAC-promoted growth.

However, based on our prior experimental work [5], we were unable to determine if non-enzymic hydrolysis of IAC to IAA was operative. To ascertain the extent of non-enzymic hydrolysis of IAC, we obtained a quantitative measure of the amount of IAA liberated from IAC non-enzymically using the method of isotopic dilution with $^{13}C_6$-IAA serving as the internal standard [7]. The percentage hydrolysis was computed from the amounts of IAA present after a 4 hr incubation of IAC in buffers at pH 6.1, 7.0 and 8.0. Sample preparation prior to gas chromatography-selected ion monitoring-mass spectrographic (GC-SIM-MS) analysis was performed as previously described by Chen *et al.* [8]. From the values of percentage hydrolysis reported in Table 3, it is apparent that no appreciable hydrolysis of IAC occurs during 4 hr in MES buffer under conditions identical to those employed in our pea stem growth studies. Thus, we have definitively shown that non-enzymic hydrolysis of IAC to IAA is inconsequential at pH 6.1.

This conclusion is strengthened by an examination of the relative biological activity of three representative choline esters subjected to pea stem segment elongation

Table 1. Comparative ^{1}H NMR data (300 MHz, d_6-DMSO)

IAA	DMAE	2	ChI	1	Assignment
12.2 (*br s*, 1H)					
10.9 (*s*, 1H)		10.9 (*br s*, 1H)		11.0 (*br s*, 1H)	H-1
7.5 (*d*, 1H)*		7.5 (*d*, 1H)*		7.5 (*d*, 1H)*	H-4
7.35 (*d*, 1H)*		7.35 (*d*, 1H)*		7.35 (*d*, 1H)*	H-7
7.2 (*s*, 1H)		7.25 (*s*, 1H)		7.3 (*s*, 1H)	H-2
7.1 (*t*, 1H)*		7.1 (*t*, 1H)*		7.1 (*t*, 1H)*	H-5
7.0 (*t*, 1H)*		7.0 (*t*, 1H)*		7.0 (*t*, 1H)*	H-6
	4.4 (*br s*, 1H)		5.2 (*t*, 1H)†		
	3.45 (*t*, 2H)†	4.1 (*t*, 2H)†	3.85 (*m*, 2H)	4.5 (*br s*, 2H)	H-4′
3.6 (*s*, 2H)		3.7 (*s*, 2H)		3.8 (*s*, 2H)	H-1′
	2.3 (*t*, 2H)†	2.45 (*t*, 2H)†	3.45 (*t*, 2H)†	3.65 (*m*, 2H)	H-5′
	2.25 (*s*, 6H)	2.25 (*s*, 6H)	3.2 (*s*, 9H)	3.1 (*s*, 9H)	H-7′

*J = 8 Hz.
†J = 6 Hz.

Table 2. Comparative ^{13}C NMR data (22.5 MHz, d_6-DMSO)

IAA	DMAE	2	ChI	1	Assignment
173.4 (C=O)		171.5 (C=O)		170.9 (C=O)	C-2′
136.3 (C=)		136.2 (C=)		136.0 (C=)	C-8
127.4 (C=)		127.1 (C=)		127.0 (C=)	C-9
124.0 (CH=)		124.1 (CH=)		124.3 (CH=)	C-2
121.1 (CH=)		121.1 (CH=)		121.1 (CH=)	C-5
118.7 (CH=)		118.5 (CH=)		118.5 (CH=)	C-4*
118.6 (CH=)		118.4 (CH=)		118.4 (CH=)	C-6*
111.5 (CH=)		111.4 (CH=)		111.4 (CH=)	C-7
107.8 (C=)		107.0 (C=)		106.4 (C=)	C-3
	61.5 (CH_2N)	57.3 (CH_2N^+)	66.8 (CH_2N^+)	63.7 (CH_2N)	C-5′
	59.0 (CH_2O)	62.0 (CH_2O)	55.0 (CH_2O)	58.0 (CH_2O)	C-4′
	45.5 $(CH_3)_2$	45.3 $(CH_3)_2$	53.1 $(CH_3)_3$	52.9 $(CH_3)_3$	C-7′
31.2 (CH_2)		31.0 (CH_2)		30.6 (CH_2)	C-1′

*Assignments could be reversed.

Table 3. Extent of non-enzymic hydrolysis of IAC

Buffer	pH	C_f*	y†	Percentage hydrolysis‡	Mean value (% hydrolysis)
MES	6.10	0.988	0.896	0.045	0.02
		0.989	0.000	0.000	
MOPS	7.00	0.977	10.9	0.548	0.52
		0.978	9.95	0.500	
Tris	8.00	0.756	273	13.7	14
		0.745	290	14.6	

*C_i (initial concentration) = 0.989 ± 0.001 was obtained by subjecting three t = 0 hr samples in MES buffer to standard work-up and GC-SIM-MS quantitative analysis. C_f (final concentration) values were determined similarly after a 4 hr incubation period.

†$y = [(C_f/C_i) - 1)]\,X/R$, where X is the amount of $^{13}C_6$-IAA present in each trial; here, 1 μg. R is an empirically determined weighting factor that takes into account the fact that, while predominantly present as quinolinium ions with only values of 136 and 130, respectively, $^{13}C_6$-IAA and IAA are also present in small percentages at neighbouring values of $m/z \pm n$, where $n = \{1, 2, 3\}$. As such, R is the ratio of the fraction of IAA at 130 to the fraction of its labelled counterpart at 136, where R = (0.89/0.79) = 1.13 [6].

‡Percentage hydrolysis is relative to the amount of unlabelled IAA that would be generated if all the IAC present were hydrolysed.

studies as compared to the effect of the parent auxins in the same plant bioassay: IAC > 3-Cl-PAC (**11**) ≫ 1-NAC (**3**), whereas IAA > 1-NAA > 3-Cl-PAA. After we have completed our growth studies of the six IAC analogues, we will provide a comprehensive report of these results.

EXPERIMENTAL

Mp: uncorr. IR: KBr. NMR in $CDCl_3$ or d_6-DMSO with TMS as int. standard: ^1H at 300 MHz and ^{13}C at 22.5 MHz (HETCOR 2D NMR at 75.4 MHz for ^{13}C). Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN.

Analysis of free IAA by GC-SIM-MS. A standard soln of IAC was prepd by dissolving 20.2 mg IAC in 10 ml double-distilled H_2O. To 50 μl of soln was added 50 μl standard soln of $^{13}C_6$-IAA in MeOH (5.0 mg $^{13}C_6$-IAA, 10 ml). To each 1 ml sample of buffer was pipetted 4 μl IAC soln doped with $^{13}C_6$-IAA int. standard. Thus, each 1 ml of buffer soln contained *ca* 4 μg IAC (*ca* 10 μM) and exactly 1 μg $^{13}C_6$-IAA.

The amino anion exchange column (PrepSep 0.3 g, Fisher) was conditioned as previously reported [8]. Following application of buffer soln, the column was washed with 4–5 ml double-distilled H_2O. The IAA fr. was then eluted with 5% AcOH in MeOH, evapd to dryness, methylated in 100 μl MeOH using ethereal diazomethane [9], and analysed by GC-SIM-MS [7].

Purification of 1-dimethylamino-2-chloroethane (DMAC). To 11.52 g DMAC·HCl (80.0 mmol) was added sufficient H_2O to dissolve the solid (*ca* 50 ml). After cooling the soln in an ice bath, 50 ml Et_2O was added, followed by 11.06 g K_2CO_3 (80.0 mmol) and 4–5 ml 50% (w/v) aq. NaOH. After separating the layers, the aq. layer was immediately extracted with 4 × 50 ml Et_2O, and the combined organic extracts were dried overnight over K_2CO_3. The solvent was removed under red. pres. and DMAC was distilled from CaH_2 just prior to use. Yield: 51% (bp 98°).

2-Dimethylaminoethyl indole-3-acetate (**2**). To a soln of 1.76 g IAA (10.0 mmol) and 2.15 g DMAC (20.0 mmol) in 100 ml EtOAc was added 1.38 g finely ground K_2CO_3 (10.0 mmol). After refluxing for 5 hr, the reaction was quenched with 100 ml ice-cold 5% (w/v) aq. Na_2CO_3 followed by extraction with 4 × 75 ml Et_2O. After drying the combined Et_2O extracts over $MgSO_4$, the drying agent was removed by gravity filtration and the solvent via rotoevaporation. Crystals were allowed to form in the desiccator. Crude yield: 94%. Recrystallization from cyclohexane afforded 53% yield (mp 62.2–62.8°). IR (cm^{-1}): 3120 (m), 3060 (m), 2949 (m), 2820 (m), 1715 (s), 1480 (m), 1210 (s), 1160 (s), 1000 (m), 950 (m), 785 (m), 725 (s). Found: C, 68.15; H, 7.47; N, 11.15. $C_{14}H_{18}N_2O_2$ requires C, 68.27; H, 7.37; N, 11.37.

2-Dimethylaminoethyl naphthalene-1-acetate (**4**). IR (cm^{-1}): 3025 (w), 2940 (s), 1730 (s), 1450 (m), 1240 (s), 1040 (m), 775 (s). ^1H NMR ($CDCl_3$): 8.0 (*m*, 1H), 7.8 (*m*, 1H), 7.7 (*m*, 1H), 7.5 (*t*, 2H, J = 7 Hz), 7.4 (*m*, 2H), 4.2 (*t*, 2H, J = 6 Hz), 4.1 (*s*, 2H), 2.5 (*t*, 2H, J = 6 Hz), 2.2 (*s*, 6H). ^{13}C NMR ($CDCl_3$): 171.4 (C=O), 134.1 (C=), 132.5 (C=), 130.9 (C=), 128.8 (CH=), 128.2 (CH=), 128.1 (CH=), 126.4 (CH=), 125.8 (CH=), 125.6 (CH=), 124.1 (CH=), 62.8 (CH_2O), 57.9 (CH_2N), 45.6 (2Me), 39.4 (CH_2).

2-Dimethylaminoethyl naphthalene-2-acetate (**6**). IR (cm^{-1}): 3020 (w), 2950 (s), 1730 (s), 1360 (m), 1235 (s), 1040 (s), 800 (m). ^1H NMR ($CDCl_3$): 7.8 (*m*, 3H), 7.7 (*s*, 1H), 7.4–7.5 (*m*, 3H), 4.4 (*t*, 2H, J = 6 Hz), 3.8 (*s*, 2H), 2.5 (*t*, 2H, J = 6 Hz), 2.2 (*s*, 6H). ^{13}C NMR ($CDCl_3$): 171.5 (C=O), 133.4 (C=), 132.5 (C=), 131.5 (C=), 128.1 (CH=), 128.0 (CH=), 127.63 (CH=), 127.59 (CH=), 127.4 (CH=), 126.1 (CH=), 125.7 (CH=), 62.7 (CH_2O), 57.7 (CH_2N), 45.6 (2Me), 41.4 (CH_2).

2-Dimethylaminoethyl phenylacetate (**8**). IR (cm^{-1}): 3040 (m), 2970 (s), 1730 (s), 1460 (s), 1250 (s), 1155 (s), 760 (m), 730 (s). ^1H NMR (d_6-DMSO): 7.3 (*m*, 5H), 4.1 (*t*, 2H, J = 6 Hz), 3.6 (*s*, 2H), 2.4 (*t*, 2H, J = 6 Hz), 2.1 (*s*, 6H). ^{13}C NMR (d_6-DMSO): 171.5 (C=O), 134.9 (C=), 129.8 (CH=), 128.7 (CH=), 127.3 (CH=), 62.7 (CH_2O), 57.7 (CH_2N), 45.8 (2Me), 40.9 (CH_2).

2-Dimethylaminoethyl 2-chlorophenylacetate (**10**). IR (cm^{-1}): 3060 (m), 2950 (s), 1730 (s), 1440 (m), 1240 (s), 1155 (s), 740 (s). ^1H NMR (d_6-DMSO): 7.3 (*m*, 4H), 4.2 (*t*, 2H, J = 6 Hz), 3.8 (*s*, 2H), 2.4 (*t*, 2H, J = 6 Hz), 2.1 (*s*, 6H). ^{13}C NMR (d_6-DMSO): 170.5 (C=O), 134.2 (C=), 133.2 (C=), 132.6 (CH=), 129.6 (CH=), 129.5 (CH=), 127.7 (CH=), 63.0 (CH_2O), 57.6 (CH_2N), 45.8 (2Me), 39.0 (CH_2).

2-Dimethylaminoethyl 3-chlorophenylacetate (**12**). IR (cm^{-1}): 3040 (w), 2950 (s), 1725 (s), 1460 (s), 1240 (s), 1150 (s), 860 (w), 775 (s), 680 (s). ^1H NMR (d_6-DMSO): 7.3 (*m*, 4H), 4.2 (*t*, 2H, J = 6 Hz), 3.7 (*s*, 2H), 2.4 (*t*, 2H, J = 6 Hz), 2.1 (*s*, 6H). ^{13}C NMR (d_6-DMSO): 171.1 (C=O), 137.3 (C=), 133.4 (C=), 130.5 (CH=), 129.8 (CH=), 128.6 (CH=), 127.3 (CH=), 62.3 (CH_2O), 57.6 (CH_2N), 45.7 (2Me), 40.2 (CH_2).

2-Dimethylaminoethyl 4-chlorophenylacetate (**14**). IR (cm^{-1}): 3030 (w), 2970 (s), 1720 (s), 1460 (m), 1240 (s), 1150 (s), 800 (m). ^1H NMR (d_6-DMSO): 7.35 (*dd*, 4H), 4.1 (*t*, 2H, J = 6 Hz), 3.7 (*s*, 2H), 2.5 (*t*, 2H, J = 6 Hz), 2.1 (*s*, 6H). ^{13}C NMR (d_6-DMSO): 170.7 (C=O), 133.4 (C=), 131.6 (C=), 131.2 (CH=), 128.2 (CH=), 62.2 (CH_2O), 57.1 (CH_2N), 45.2 (2Me), 39.5 (CH_2).

Indole-3-acetylcholine (**1**). Into a 5 ml vial was added 246 mg (1.00 mmol) **2**. After passage of N_2, 3 ml dry EtOAc was added and the vial was heated at 90°. On dissolution of **2**, 62 μl (1.0 mmol) MeI was added and the reaction mixt. was refluxed for 1 h. Another 1.0 mmol aliquot of MeI was added, and reflux was continued for a further 0.5 h. On cooling, a crystalline solid was formed, the supernatant liquid was removed, and the solid was dried *in vacuo* at 40°. Yield: 60% (mp 171.5–173.0°). When impure **2** was converted directly into **1**, the product was recrystallized from dry MeOH (mp 172.3°). IR (cm^{-1}): 3260 (s), 3020 (m), 2949 (m), 1710 (m), 1460 (m), 1325 (m), 1190 (m), 1140 (s), 1080 (m), 930 (m), 735 (m). Found: C, 46.19; H, 5.55; N, 7.10; I, 32.76. $C_5H_{21}N_2O_2I$ requires: C, 46.39; H, 5.41; N, 7.22; I, 32.73.

Naphthalene-1-acetylcholine (**3**). Mp 112–113°. IR (cm^{-1}): 3020 (w), 2980 (m), 1740 (s), 1465 (s), 1155 (s), 930

(m), 765 (s). ^{1}H NMR (d_6-DMSO): 8.0 (*t*, 2H, J = 9 Hz), 7.9 (*dd*, 1H), 7.6 (*m*, 2H), 7.5 (*m*, 2H), 4.5 (*br s*, 2H), 3.7 (*m*, 2H), 3.2 (*s*, 2H), 3.1 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 170.4 (C=O), 133.2 (C=), 131.6 (C=), 130.3 (C=), 128.4 (CH=), 128.1 (CH=), 127.6 (CH=), 126.3 (CH=), 125.7 (CH=), 125.4 (CH=), 123.9 (CH=), 63.5 (CH_2N+), 58.2 (CH_2O), 52.8 (3Me), 37.8 (CH_2).

Naphthalene-2-acetylcholine (**5**). Mp 103–104°. IR (cm^{-1}): 3020 (w), 2980 (m), 1735 (s), 1460 (m), 1140 (s), 930 (s), 815 (s), 760 (s). ^{1}H NMR (d_6-DMSO): 7.9 (*m*, 3H), 7.8 (*s*, 1H), 7.5 (*m*, 3H), 4.4 (*br s*, 2H), 3.9 (*s*, 2H), 3.6 (*m*, 2H), 3.1 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 170.4 (C=O), 132.8 (C=), 131.8 (C=), 131.5 (C=), 127.8 (CH=), 127.76 (CH=), 127.7 (CH=), 127.4 (CH=), 127.3 (CH=), 126.1 (CH=), 125.8 (CH=), 63.5 (CH_2N+), 58.2 (CH_2O), 52.9 (3Me), 40.2 (CH_2).

Phenylacetylcholine (**7**). Mp 147–148°. IR (cm^{-1}): 3030 (w), 2950 (w), 1730 (s), 1460 (m), 1215 (s), 1155 (s), 760 (m), 720 (m). ^{1}H NMR (d_6-DMSO): 7.3 (*m*, 5H), 4.4 (*br s*, 2H), 3.7 (*s*, 2H), 3.66 (*m*, 2H), 3.1 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 171.1 (C=O), 134.4 (C=), 127.8 (CH=), 127.76 (CH=), 130.1 (CH=), 128.9 (CH=), 127.5 (CH=), 64.2 (CH_2N+), 58.7 (CH_2O), 53.5 (3Me), 40.8 (CH_2).

2-Chlorophenylacetylcholine (**9**). Mp 105–106°. IR (cm^{-1}): 2990 (m), 1730 (s), 1460 (m), 1245 (s), 1145 (s), 740 (s). ^{1}H NMR (d_6-DMSO): 7.5 (*m*, 2H), 7.3 (*m*, 2H), 4.45 (*br s*, 2H), 3.9 (*s*, 2H), 3.7 (*m*, 2H), 3.1 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 170.0 (C=O), 134.1 (C=), 132.8 (CH=), 132.7 (C=), 129.7 (CH=), 129.6 (CH=), 127.8 (CH=), 64.1 (CH_2N+), 59.0 (CH_2O), 53.5 (3Me), 38.9 (CH_2).

3-Chlorophenylacetylcholine (**11**). Mp 120–122°. IR (cm^{-1}): 3030 (w), 2950 (w), 1730 (s), 1460 (s), 1210 (m), 1155 (s), 875 (m), 780 (m), 705 (m), 675 (m). ^{1}H NMR (d_6-DMSO): 7.4 (*m*, 4H), 4.5 (*br s*, 2H), 3.8 (*s*, 2H), 3.7 (*br m*, 2H), 3.2 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 170.0 (C=O), 136.2 (C=), 132.7 (C=), 130.0 (CH=), 129.4 (CH=), 128.3 (CH=), 126.8 (CH=), 63.5 (CH_2N+), 58.2 (CH_2O), 52.9 (3Me), 40.0 (CH_2).

4-Chlorophenylacetylcholine (**13**). Mp 123°. IR (cm^{-1}): 3030 (w), 2950 (w), 1720 (s), 1460 (s), 1210 (m), 1155 (s), 800 (m), 730 (m). ^{1}H NMR (d_6-DMSO): 7.4 (*d*, 2H), 7.35 (*d*, 2H), 4.5 (*br s*, 2H), 3.8 (*s*, 2H), 3.77 (*m*, 2H), 3.2 (*s*, 9H). ^{13}C NMR (d_6-DMSO): 170.1 (C=O), 132.8 (C=), 131.5 (C=), 131.4 (CH=), 128.1 (CH=), 63.5 (CH_2N+), 58.2 (CH_2O), 52.9 (3Me), 39.2 (CH_2).

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