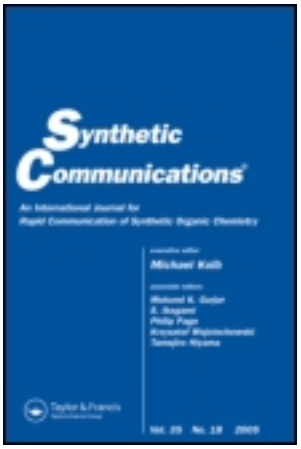

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One-Pot Formylation and Dimerization of p-Alkyl Phenols Using DCMT-Activated DMSO

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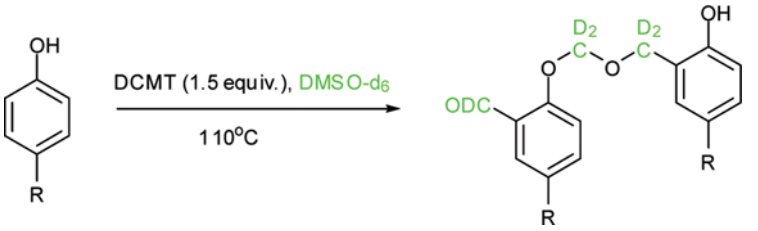
GRAPHICAL ABSTRACT



Abstract *Dimerization and formylation of p-alkyl-substituted phenols have been successfully achieved in one pot, using 2,4-dichloro-6-methoxy[1,3,5]triazine (DCMT)-activated dimethylsulfoxide. Similarly, the d5-labeled products have been prepared in high selectivity. To the best of our knowledge, the products are of new skeletons, which have not been reported to date. A plausible mechanism is proposed based on the experiments.*

Keywords 2,4-Dichloro-6-methoxy[1,3,5]triazine; dimerization; DMSO; formylation; phenols

INTRODUCTION

Cyanuric chloride (CC) and its derivatives have been reported as efficient reagents in synthesis.[1] Recently there has been a considerable growth of interest in the use of CC and its derivatives in the processes involving functional group transformations; notable examples include activation of carboxylic acids,[2] preparation of sulfonyl and alkyl chlorides,[3] selective protection of primary alcohols by a formyl residue,[4] catalysis of the Beckmann rearrangement,[5] deprotection of 1,3-dithioacetals and 1,3-oxathiolanes to their corresponding carbonyl compounds,[6] activation of dimethylsulfoxide (DMSO) in the Swern oxidation,[7] conversion of formamides to isonitriles,[8] and catalysis of Friedel–Crafts acylations.[9] We have reported the

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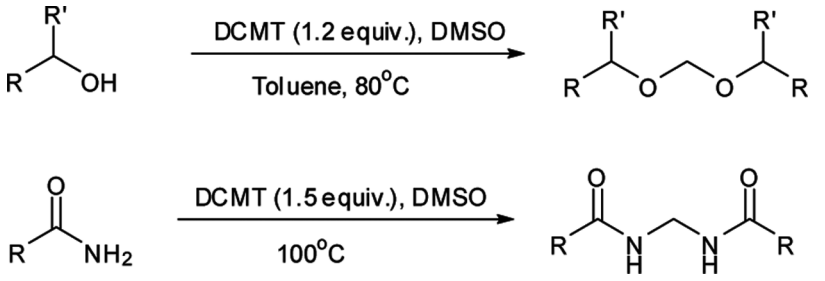


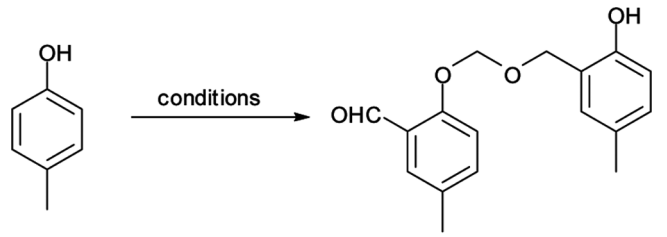
Scheme 1.

preparation of methylene acetal[10] and methylenebisaminde[11] using 2,4-dichloro-6-methoxy[1,3,5]triazine (DCMT)–activated DMSO (Scheme 1).

RESULTS AND DISCUSSION

In an attempted synthesis of bis(p-tolyloxy)methane from p-cresol using DCMT-activated DMSO, we discovered the rather surprising formation of 2-((2-hydroxy-5-methylbenzyloxy)methoxy)-5-methylbenzaldehyde (Table 1) when the reaction temperature was elevated to 110 °C. Therefore, we started optimizing the reaction conditions for this interesting new product. No product was observed when the reaction was carried out at lower temperature (Table 1, entry 6) or without DCMT (Table 1, entry 1). The reaction was performed in various solutions, such as

Table 1. Optimization of the reaction of p-cresol and DCMT-activated DMSO[a]



1a 2a

Entry	DCMT (equiv)	T (°C)	Yield (%)[b]
1	0	110	Trace
2	0.6	110	10
3	1.2	110	26
4	1.5	110	36
5	2	110	35
6	1.5	90	Trace

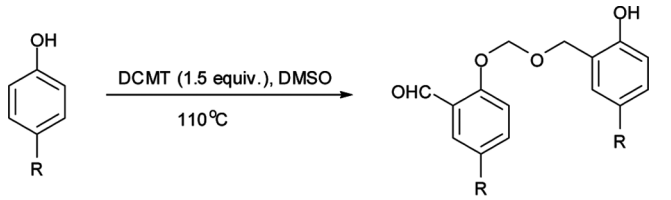
[a]Reaction conditions: p-cresol (500 mg, 1 equiv.), DMSO (10 mL), 90–110 °C.
[b]Isolated yields.

toluene, xylene, and DMSO, and DMSO gave the best result. Decreasing the amount of the DCMT resulted in reduced yield, while increasing the amount to 2 equiv. did not make the reaction system complicated and the yield was not notably changed.

Under these optimized conditions, the scope of p-substituted phenols were investigated (Table 2). Altogether seven p-substituted phenols have been successfully transformed into the dimerized aldehydes. In a typical precedure, p-cresol and DCMT (1.5 equiv.) were heated in 10 mL DMSO at 110 °C for 6 h followed by usual workup to get the products. However, no reaction took place for phenols bearing electron withdrawing groups (Table 2, entries 8 and 9). It is obvious that only alkyl- and alkoxy-substituted phenols are capable of reacting with DCMT-activated DMSO to produce the desired products. The yields range from 35% to 58% and the reaction durations from 4 h to 6 h. The dimerized phenolic aldehydes are of new skeletons, not known in the literature. In one pot, we have realized formylation and dimerization of phenols. Formylation reactions represented[12] by the Gatermann–Koch reaction, the Friedel–Crafts reaction, and the Vilsmeier–Haack reaction are important organic reactions. Our procedure not only formylates the phenols as the known reactions do but also dimerizes the phenols. Although the yields of the reactions are poor, this reaction has provided a simple route to the multifunctional group products. It is noteworthy that the products possess two active functional groups, which could be used for further transformations.

When the reaction was carried out in DMSO-d_6 (99.9%), d_5-labeled products were obtained (Table 3). The reaction conditions and the yields of these reactions were similar to the reactions in DMSO. ^{1}HNMR indicates that deuterated formyl

Table 2. Reaction of p-substituted phenols and DCMT-activated DMSO[a]

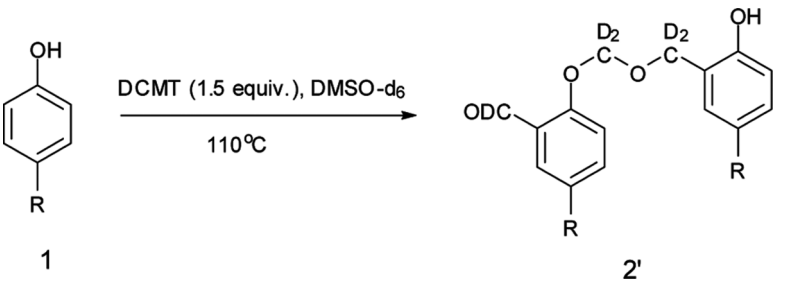


1 2

Entry	R	Compound	Time (h)	Yield (%)[b]
1	-CH_3	**2a**	6	36
2	-OCH_3	**2b**	4	35
3	-$C(CH_3)_3$	**2c**	4	56
4	-$C(CH_3)_2CH_2CH_3$	**2d**	4	58
5	Cyclohexyl	**2e**	6	47
6	n-C_9H_{19}	**2f**	5	52
7	-CH_2COOCH_3	**2g**	6	45
8	-NO_2	**2h**	12	Trace
9	-*$COCH_3$*	**2i**	12	Trace

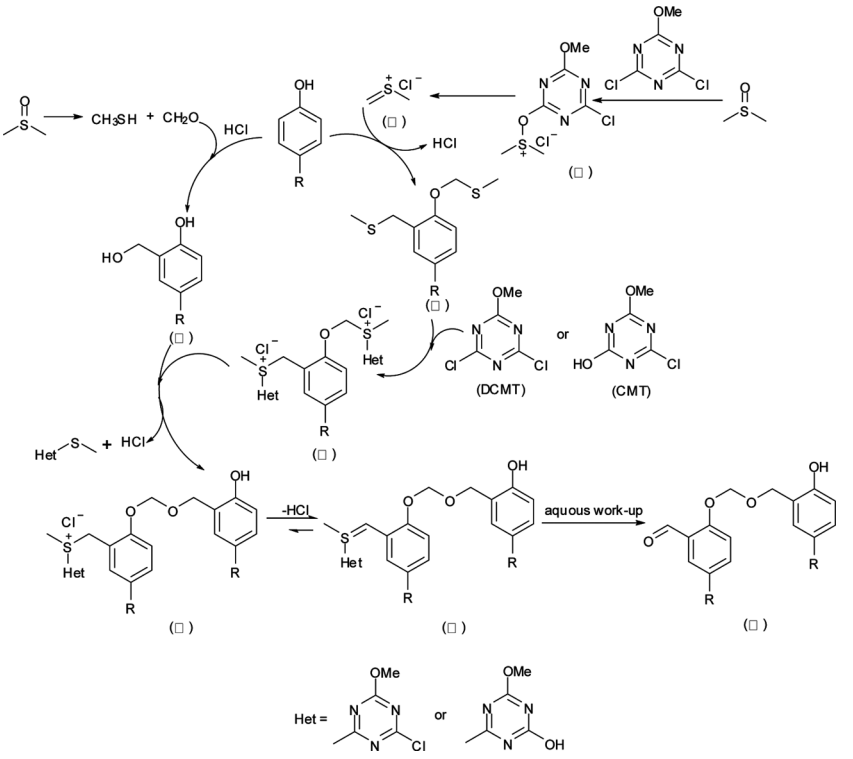
[a]Reaction conditions: p-substituted phenol (500 mg, 1 equiv.), DCMT (1.5 equiv.), DMSO (10 mL), 110 °C.
[b]Isolated yields.

Table 3. Reaction of p-substituted phenols and DCMT-activated DMSO-d_6[a]



Entry	R	Compound	Time (h)	Yield (%)[b]
1	$-CH_3$	**2a′**	6	37
2	$-OCH_3$	**2b′**	4	39
3	$-C(CH_3)_2CH_2CH_3$	**2d′**	4	55
4	$-CH_2COOCH_3$	**2g′**	6	48

[a]Reaction conditions: p-substituted phenol (500 mg, 1 equiv.), DCMT (1.5 equiv.), DMSO-d_6 (10 mL), 110 °C.
[b]Isolated yields.



Scheme 2.

and methylene groups of the products originate from DMSO-d_6. According to ^{1}HNMR, the deuterations are highly regioselective and the nonlabeled products are not detected. The reaction has fully utilized the DMSO-d_6 in building the CD_2 and deuterated formyl (CDO) groups. In the literature,[13] only the deuterated hydrogen atoms of DMSO-d_6 have been used in building the labeled products. Deuterated compounds are useful in the studies of metabolism and movement of drugs and toxic substances in humans and animals, and reaction mechanisms.[14] We have provided a convenient route to d_5-labeled compounds from the inexpensive DMSO-d_6 in one pot.

On the basis of these experiments, a plausible mechanism is proposed (Scheme 2). Reacting with DCMT, DMSO yields intermediate II via intermediate I. Addition of intermediate II to both the phenolic ring and the phenolic hydroxyl generate thioester III. The bisulfide is a good nucleophile to form sulfonium salt IV with DCMT or 4-chloro-6-methoxy-1,3,5-triazin-2-ol (CMT). On the other hand, formaldehyde formed from the thermal decomposition of DMSO[15] hydroxymethylates the phenol in the presence of HCl, leading to V. This hydroxymethylated product can polymerize with another phenol in the presence of HCl, which may account for the moderate yields of the reactions. Nucleophilic attack of V on sulfonium IV leads to intermediate VI, which is in equilibrium with VII. After aqueous workup, VI hydrolyzed into the final product VIII. This mechanism is supported by the d_5-labeled products from the reaction run in DMSO-d_6 (Table 3). It is further supported by the fact that phenols substituted with electron-withdrawing groups cannot afford desired products (Table 2, entries 8 and 9). The reason is that these phenols are not electron-rich enough to react with II, which is not sufficiently electrophilic.

CONCLUSION

In conclusion, we have demonstrated that the dimerization and formylation of p-alkyl-substituted phenols can be performed in one pot. Similarly, the d_5-labeled products have been prepared with the same procedure when DMSO-d_6 was used. The procedure reported herein is operationally simple and requires inexpensive reagents. A plausible mechanism of the reaction is proposed and supported by the experiments. Further study into the synthetic applications is ongoing in our laboratory.

EXPERIMENTAL

All of the chemicals were obtained from commercial sources or prepared according to standard methods. The ^{1}HNMR (500 MHz) and ^{13}CNMR (125 MHz) spectra were recorded on a Varian Oxford 500 spectrometer. The ^{1}HNMR (400 MHz) and ^{13}CNMR (100 MHz) spectra were recorded on a Bruker AM-400 spectrometer. Chemical shifts (δ) are reported relative to tetramethylsilane (TMS) (^{1}H) or $CDCl_3$ (^{13}C). Mass spectra [electrospray ionization (ESI)] were obtained on a Finnigan LCQ Advantage MAX spectrometer. Elemental analyses for C and H were performed on a Yanaco CHNCORNER MF-3 elemental analyzer, and the analytical results were within $\pm 0.4\%$ of the theoretical values.

Typical Experimental Procedure

The procedure for the reaction of DMCT-activated DMSO and p-cresol (Table 2, entry 1) is representative for all p-substituted phenols. p-Cresol (500 mg, 4.63 mmol) was added to the solution of DCMT (1250 mg, 6.94 mmol) in dry DMSO (5 mL). The mixture was stirred at 110 °C and monitored by TLC until completion (6 h). Then, it was neutralized with saturated aqueous $NaHCO_3$ (20 mL) and extracted with CH_2Cl_2 (3×10 mL). The extract was washed with water (5×5 mL) and dried over anhydrous Na_2SO_4. The solvent was concentrated in vacuo to give the crude product, which was further purified by silica-gel column chromatography (PE/EA = 30/1) to afford 2-((2-hydroxy-5-methylbenzyloxy)methoxy)-5-methylbenzaldehyde **(2a)** (238 mg, yield 36%).

Compound 2a

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.85 (s, 1H), 9.85 (s, 1H), 7.33–7.35 (m, 2H), 6.97 (dd, $J = 7.6$ Hz, $J = 8.3$ Hz, 1H), 6.90 (d, $J = 2.0$ Hz, 1H), 6.77 (m, 2H), 5.22 (s, 2H), 4.87 (s, 2H), 2.34 (s, 3H), 2.27 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 196.8, 159.8, 150.7, 138.3, 133.6, 130.8, 129.4, 128.8, 125.6, 121.2, 120.6, 117.6, 116.8, 91.5, 66.4, 20.9, 20.5; MS (ESI): m/z (%) = 325 $[M + K]^+$ (100): Anal. calcd. for $C_{17}H_{18}O_4$: C, 71.31; H, 6.34. Found: C, 71.74; H, 6.10.

2-((2-Hydroxy-5-methoxybenzyloxy)methoxy)-5-methoxybenzaldehyde (2b)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.66 (s, 1H), 9.86 (s, 1H), 7.15 (dd, $J = 3.1$ Hz, $J = 9.1$ Hz, 1H), 7.01 (d, $J = 3.1$ Hz, 1H), 6.94 (d, $J = 9.1$ Hz, 1H), 6.82 (d, $J = 8.9$ Hz, 1H), 6.74 (dd, $J = 2.9$ Hz, $J = 8.9$ Hz, 1H), 6.50 (d, $J = 2.9$ Hz, 1H), 5.20 (s, 2H), 4.88 (s, 2H), 3.82 (s, 1H), 3.75 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 196.4, 156.3, 154.3, 152.9, 146.8, 1(m, 2H), 6.817.15 (m, 4H), 5.24 (s, 2H), 4.91 (s, 2H), 25.5, 122.0, 120.3, 118.9, 117.8, 115.4, 114.2, 109.9, 91.4, 66.4, 56.2, 55.9; MS (ESI): m/z 318.3(%) = 341 $[M + Na]^+$ (100): Anal. calcd. for $C_{17}H_{18}O_6$: C, 64.14; H, 5.70. Found: C, 64.32; H, 5.92.

5-*tert*-Butyl-2-((5-*tert*-butyl-2-hydroxybenzyloxy)methoxy) benzaldehyde (2c)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.88 (s, 1H), 9.90 (s, 1H), 7.60 (dd, $J = 2.5$ Hz, $J = 8.7$ Hz, 1H), 7.52 (d, $J = 2.5$ Hz, 1H), 7.20 (dd, $J = 2.4$ Hz, $J = 8.6$ Hz, 1H), 6.96 (m, 2H), 6.83 (d, $J = 8.6$ Hz, 1H), 5.24 (s, 2H), 4.92 (s, 2H), 1.34 (s, 9H), 1.29 (s, 9H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 197.1, 159.7, 150.6, 144.3, 143.0, 134.9, 130.0, 125.3, 121.9, 120.7, 120.2, 117.5, 116.4, 91.4, 66.7, 34.5, 34.3, 31.8, 31.4; MS (ESI): m/z $(\%)^+ = 409$ $[M + K]$ (100): Anal. calcd. for $C_{23}H_{30}O_4$: C, 74.56; H, 8.16. Found: C, 74.74; H, 8.10.

2-((2-Hydroxy-5-*tert*-pentylbenzyloxy)methoxy)-5-*tert*-pentylbenzaldehyde (2d)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.87 (s, 1H), 9.89 (s, 1H), 7.52 (dd, $J = 2.2$ Hz, $J = 8.7$ Hz, 1H), 7.46 (d, $J = 2.2$ Hz, 1H), 7.13 (dd, $J = 2.2$ Hz, $J = 8.6$ Hz, 1H), 6.95

(d, $J = 8.7$ Hz, 1H), 6.89 (d, $J = 2.2$ Hz, 1H), 6.82 (d, $J = 8.6$ Hz, 1H), 5.24 (s, 2H), 4.91 (s, 2H), 1.62 (m, 4H), 1.29 (s, 6H), 1.24 (s, 6H), 0.69 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 197.1, 159.6, 150.5, 142.6, 141.2, 135.4, 130.9, 125.9, 122.6, 120.6, 120.3, 117.4, 116.4, 91.5, 66.7, 37.6, 37.5, 37.1, 36.9, 28.8, 28.6, 9.36, 9.29; MS (ESI): m/z (%) = 437 $[M + K]^+$ (100): Anal. calcd. for $C_{25}H_{34}O_4$: C, 75.34; H, 8.60. Found: C, 74.98; H, 8.79.

2-((2-Hydroxy-5-nonylbenzyloxy)methoxy)-5-nonylbenzaldehyde (2e)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.88 (s, 1H), 9.89 (s, 1H), 7.39–7.53 (m, 2H), 6.81–7.15 (m, 4H), 5.24 (s, 2H), 4.91 (s, 2H), 0.66–1.71 (m, 38H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 197.2, 159.6, 150.3, 143.8, 142.4, 135.5, 131.0, 126.6, 126.0, 122.7, 120.4, 117.2, 116.2, 91.5, 66.8, 41.6, 40.6, 31.6, 31.0, 30.9, 30.1, 30.0, 29.4, 25.7, 24.9, 24.6, 23.7, 23.1, 22.2, 21.6, 20.2, 14.7, 14.3; MS (ESI): m/z (%) = 534 $[M + Na]^+$ (100): Anal. calcd. for $C_{33}H_{50}O_4$: C, 77.60; H, 9.87. Found: C, 77.84; H, 9.60.

5-Cyclohexyl-2-((5-cyclohexyl-2-hydroxybenzyloxy)methoxy) benzaldehyde (2f)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.87 (s, 1H), 9.89 (s, 1H), 7.38–7.42 (m, 2H), 6.81–7.05 (m, 4H), 5.24 (s, 2H), 4.91 (s, 2H), 2.40–2.51 (m, 2H), 1.74–1.88 (m, 10H), 1.23–1.47 (m, 10H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.7, 159.8, 150.7, 141.1, 139.6, 136.1, 131.2, 126.4, 123.1, 120.9, 120.4, 117.4, 116.5, 91.2, 66.3, 43.9, 43.3, 34.7, 34.5, 26.9, 26.8, 26.1, 26.0; MS (ESI): m/z (%) = 446 $[M + Na]^+$ (100): Anal. calcd. for $C_{33}H_{50}O_4$: C, 76.74; H, 8.11. Found: C, 76.34; H, 8.40.

Methyl-2-(3-formyl-4-((2-hydroxy-5-(2-methoxy-2-oxoethyl) benzyloxy)methoxy)phenyl)acetate (2g)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.95 (s, 1H), 9.88 (s, 1H), 7.48 (d, $J = 2.1$ Hz, 1H), 7.45 (dd, $J = 2.2$ Hz, $J = 8.5$ Hz, 1H), 7.06 (dd, $J = 2.2$ Hz, $J = 8.4$ Hz, 1H), 6.97 (d, $J = 8.5$ Hz, 1H), 6.88 (d, $J = 2.2$ Hz, 1H), 6.83 (d, $J = 8.4$ Hz, 1H), 5.23 (s, 2H), 4.89 (s, 2H), 3.71 (s, 3H), 3.69 (s, 3H), 3.62 (s, 2H), 3.54 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 196.6, 172.3, 171.7, 160.9, 151.8, 140.4, 138.1, 134.2, 128.9, 125.9, 125.4, 121.4, 120.5, 118.0, 117.1, 91.3, 66.2, 52.2, 52.1, 40.4, 39.8; MS (ESI): m/z (%) = 441 $[M + K]^+$ (100): Anal. calcd. for $C_{21}H_{22}O_8$: C, 62.68; H, 5.51. Found: C, 62.84; H, 5.40.

2-((2-Hydroxy-5-methylbenzyloxy)methoxy)-5-methylbenzaldehyde-D_5 (2a′)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.85 (s, 1H), 7.33–7.35 (m, 2H), 6.97 (dd, $J = 7.6$ Hz, $J = 8.3$ Hz, 1H), 6.90 (m, 1H), 6.77 (m, 2H), 2.34 (s, 3H), 2.27 (s, 3H); MS (ESI): m/z (%) = 330 $[M + K]^+$ (100).

2-((2-Hydroxy-5-methoxybenzyloxy)methoxy)-5-methoxybenzaldehyde-D_5 (2b′)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.66 (s, 1H), 7.15 (dd, $J = 3.1$ Hz, $J = 9.1$ Hz, 1H), 7.01 (d, $J = 3.1$ Hz, 1H), 6.94 (d, $J = 9.1$ Hz, 1H), 6.82 (d, $J = 8.9$ Hz, 1H),

6.74 (dd, $J = 2.9$ Hz, $J = 8.9$ Hz, 1H), 6.50 (d, $J = 2.9$ Hz, 1H), 3.82 (s, 3H), 3.75 (s, 3H); MS (ESI): m/z (%) = 346 $[M + Na]^+$ (100).

2-((2-Hydroxy-5-tert-Pentylbenzyloxy)methoxy)-5-tert-pentylbenzaldehyde-d_5 (2d′)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.92 (s, 1H), 7.55 (d, $J = 8.7$ Hz, 1H), 7.53 (s, 1H), 7.15 (d, $J = 8.7$ Hz, 1H), 6.96 (d, $J = 8.4$ Hz, 1H), 6.91 (s, 1H), 6.83(d, $J = 8.4$ Hz, 1H), 1.62 (m, 4H), 1.29 (s, 6H), 1.24 (s, 6H), 0.69 (m, 6H); MS (ESI): m/z (%) = 426 $[M + Na]^+$ (100).

Methyl-2-(3-formyl-4-((2-hydroxy-5-(2-methoxy-2-oxoethyl)benzyloxy)methoxy)phenyl)acetate-D_5 (2g′)

^{1}H NMR ($CDCl_3$, 500 MHz) δ 10.99 (s, 1H), 7.50 (s, 1H), 7.45 (d, $J = 8.5$ Hz, 1H), 7.09 (d, $J = 8.4$ Hz, 1H), 6.99 (d, $J = 8.5$ Hz, 1H), 6.90 (s, 1H), 6.85 (d, $J = 8.4$ Hz, 1H), 3.72 (s, 3H), 3.70 (s, 3H), 3.64 (s, 2H), 3.56 (s, 2H); MS (ESI): m/z (%) = 446 $[M + K]^+$ (100).

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