A Practical Method for *p*-Methoxybenzylation of Hydroxy Groups Using 2,4,6-Tris(*p*-methoxybenzyloxy)-1,3,5-triazine (TriBOT-PM)

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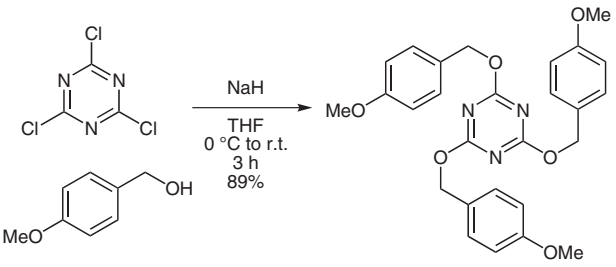
Abstract: A new acid-catalyzed *p*-methoxybenzylating reagent, 2,4,6-tris(*p*-methoxybenzyloxy)-1,3,5-triazine (TriBOT-PM), has been developed. The reaction of acid- and alkali-labile alcohols with TriBOT-PM in the presence of a catalytic amount of various acids (TfOH, $BF_3{\cdot}OEt_2$, CSA, etc.) afforded the corresponding *p*-methoxybenzyl ethers in good yields. Since TriBOT-PM is an air-stable crystalline solid and can be prepared from inexpensive materials, i.e. cyanuric chloride and anisyl alcohol, this route is of practical use.

Key words: benzylation, protecting groups, cations, ethers, alkylation

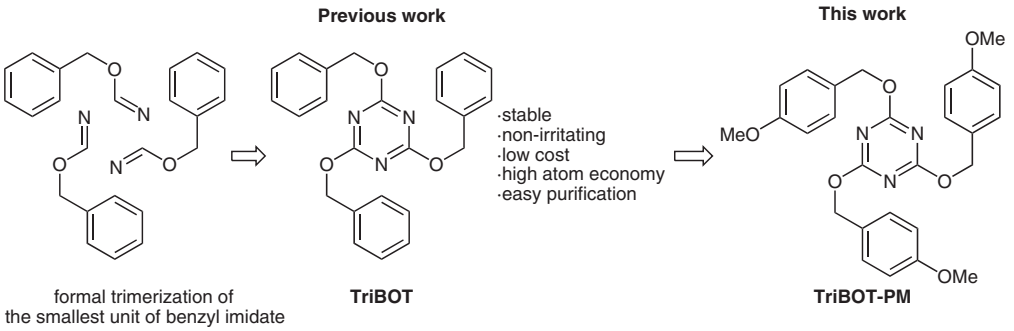
As *p*-methoxybenzyl (PMB) ethers can be selectively deprotected by oxidative cleavage using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in the presence of esters, silyl ethers, and acetals,[1] the PMB group is a versatile protecting group for hydroxy groups.[2] It can be tolerated under various chemical transforming conditions, such as hydrolysis, of these functional groups. Hydroxy groups are most commonly transformed into *p*-methoxybenzyloxy groups by using either a PMB halide and a strong base, such as sodium hydride (the Williamson ether synthesis),[1] or PMB 2,2,2-trichloroacetimidate (PMBTCAI) and catalytic acid, such as trifluoromethanesulfonic acid.[3] However, these reagents are not convenient for use and long-term storage because they are sensitive to moisture and heat. Therefore, a number of alternative methods using stable imidate-surrogate-type reagents,[4] anisyl alcohol,[5] and other reagents[6] have been developed.

Recently, we reported a novel acid-catalyzed benzylating reagent, 2,4,6-tris(benzyloxy)-1,3,5-triazine (TriBOT), the formal trimer of the smallest unit of benzyl imidate (Scheme 1).[7] The key advantages of TriBOT are its reactivity, stability, cost, and excellent atom economy. We strongly believed that a PMB analogue of TriBOT, 2,4,6-tris(*p*-methoxybenzyloxy)-1,3,5-triazine (TriBOT-PM), could act as a reagent for *p*-methoxybenzylation and it would have the aforementioned advantages.[8] In this study, we report the development of TriBOT-PM as a reagent for *p*-methoxybenzylation.

TriBOT-PM was easily synthesized from anisyl alcohol and cyanuric chloride in the presence of sodium hydride in 89% yield (Scheme 2). Similar to TriBOT, TriBOT-PM is a nonhygroscopic crystalline solid without irritating or allergenic properties, and it is stable in air at room temperature for one year.



Scheme 2 Synthesis of TriBOT-PM



Scheme 1 The concept and design of TriBOT-PM

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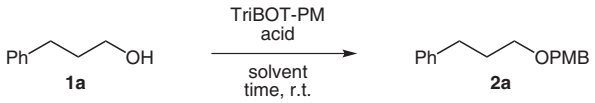
Upon treatment of 3-phenylpropan-1-ol (**1a**, 0.5 mmol) with TriBOT-PM (0.4 equiv) and trifluoromethanesulfonic acid (1 mol%) in 1,4-dioxane (0.2 M) at room temperature, the *p*-methoxybenzylation of **1a** afforded the corresponding PMB ether **2a** in 68% yield (Table 1, entry 1).

In this reaction, *N*-(*p*-methoxybenzyl)isocyanuric acid (**3**, Figure 1) was formed as a byproduct (65% yield based on TriBOT-PM). The reaction of **1a** with **3** under the same conditions did not produce **2a**. Therefore, the competing formation of **3** would be responsible for the moderate yield. We assumed that **3** was mainly generated by the N-*p*-methoxybenzylation of TriBOT-PM,[9] not that of isocyanuric acid, because if the *p*-methoxybenzylation of isocyanuric acid takes place, it should occur at the carbonyl oxygens rather than the nitrogen atoms.[10] Therefore, keeping a low concentration of TriBOT-PM during the reaction would be effective to suppress the production of **3**. For this purpose, a solution of TriBOT-PM in 1,4-dioxane was added dropwise to the reaction mixture over three minutes (initial concentration of **1a**: 0.4 M, final concentration: 0.2 M), and the resulting mixture was quenched after two minutes (total reaction time: 5 min). As a result, the yield of **2a** increased to 91% and the formation of **3** decreased to 11% (entry 2). Interestingly, a prolonged reaction time (dropwise addition time: 7 min; total reaction time: 15 min) resulted in a decrease in the yield of **2a** (entry 3, 73%) and production of symmetric acetal **4** (Figure 1) in 16% yield. Kern et al. reported that the reaction of the PMB ethers with Lewis acids furnished the symmetric acetal through methyleneoxonium intermediate **5** (Scheme 3).[11] Symmetric acetal **4** would be generated through a similar reaction pathway. In comparison with TriBOT, which requires relatively a large amount of trifluoromethanesulfonic acid (as much as 20 mol%) for O-benzylation, TriBOT-PM was more reactive, and underwent O-*p*-methoxybenzylation of alcohols in the presence of only 0.1 mol% of trifluoromethanesulfonic acid (entry 4, 90%). Solvents that exhibit Lewis basic properties (do-

Table 1 Screening of Solvent and Acid Conditions



Entry	Solvent	TriBOT-PM (equiv)	Acid (mol%)	Dropwise addition time	Additional reaction time	Yield[a] (%) of **2a**
1	1,4-dioxane	0.4	TfOH (1)	–[c]	5 min	72 (68[b])
2[d]		0.4	TfOH (1)	3 min	2 min	91
3[d]		0.4	TfOH (1)	7 min	8 min	73
4[d]		0.4	TfOH (0.1)	30 min	30 min	90
5[d]	CH_2Cl_2	0.4	TfOH (0.3)	10 min	5 min	66
6[d]	CH_2Cl_2–Et_2O (1:1)	0.4	TfOH (0.3)	10 min	5 min	90
7[d]	EtOAc	0.4	TfOH (0.3)	10 min	5 min	94
8[d,e,f]	DME	0.4	TfOH (1)	10 min	15 min	95
9[d,e]		0.4	$Sc(OTf)_3$ (5)	10 min	10 min	91
10[d,e]		0.4	$BF_3{\cdot}OEt_2$ (5)	10 min	10 min	92
11[f]	THF	0.7	$BF_3{\cdot}OEt_2$ (0.1)	–[c]	5 h	95
12[g]		0.65	CSA (15)	–[c]	10 h	99
13[g]		0.65	TsOH (15)	–[c]	5.5 h	99

[a] Yields (%) were determined by ^{1}H NMR based on **1a** using an internal standard.
[b] Isolated yield.
[c] TriBOT-PM was added in one portion.
[d] TriBOT-PM solution was prepared in the same solvent as the reaction mixture.
[e] Powdered molecular sieves 5A were added.
[f] The reaction was conducted at 0 °C.
[g] The reaction was conducted at reflux.

nor properties) appear to be suitable for this reaction. The reaction using dichloromethane as a solvent afforded **2a** in 66% yield (entry 5) due to the competing Friedel–Crafts reaction,[7] whereas changing the solvent to a dichloromethane–diethyl ether mixture improved the yield of **2a** (entry 6, 90%). The use of ethyl acetate or 1,2-dimethoxyethane as the solvent also produced **2a** in good yields (entries 7 and 8, 94% and 95%, respectively). We previously reported that O-benzylation with TriBOT proceeds through a benzyl cation intermediate[7] that can be generated by using very strong acids such as trifluoromethanesulfonic acid or trimethylsilyl trifluoromethanesulfonate. If the *p*-methoxybenzylation using TriBOT-PM involves a similar cationic species (PMB cation), the reaction can be catalyzed by acids weaker than trifluoromethanesulfonic acid because a PMB cation is more stable than a benzyl cation.[12] As expected, upon treatment of **1a** with Lewis acids, such as scandium(III) trifluoromethanesulfonate and boron trifluoride–diethyl ether complex, the reaction proceeded to give **2a** in 91% and 92% yields, respectively (entries 9 and 10).

We developed a method for the *p*-methoxybenzylation by dropwise addition of a solution of 0.4 equivalents of TriBOT-PM (equal to 1.2 equiv of the PMB group) to the reaction mixture in the presence of very small amount (minimum 0.1 mol%) of trifluoromethanesulfonic acid.

Although adjustments of the time for dropwise addition and the additional reaction time are required, this method can be considered as a high-atom-economy and time-saving procedure. Further, we developed alternative convenient methods using excess TriBOT-PM (0.65 equiv or more) and weaker acids without dropwise addition of the TriBOT-PM solution. The reaction with TriBOT-PM and boron trifluoride–diethyl ether complex (0.1 mol%) for five hours at 0 °C provided **2a** in excellent yield (entry 11, 95%). The reactions with (+)-10-camphorsulfonic acid (15 mol%) or *p*-toluenesulfonic acid (15 mol%) in tetrahydrofuran under reflux gave almost quantitative yields (99%, entries 12 and 13). In these cases, **4** was scarcely produced (<0.1%), although the production of **3** increased (38%, entry 12) and the N,N′-dibenzylated byproduct **6** (Figure 1, 12%, entry 12) was obtained.

We examined various alcohols for *p*-methoxybenzylation using TriBOT-PM (Table 2). The *p*-methoxybenzylation of **1a** on a gram scale using trifluoromethanesulfonic acid was achieved to give **2a** in an excellent yield (entry 1). However, the reaction of cinnamyl alcohol **1b** (entry 2) with trifluoromethanesulfonic acid resulted in a moderate yield presumably due to decomposition of **1b** and **2b** by trifluoromethanesulfonic acid. The yield of **2b** increased by changing the acid catalyst to (+)-10-camphorsulfonic acid (entry 3). Therefore, (+)-10-camphorsulfonic acid seems to be a suitable catalyst for acid-labile substrates. Moreover, 2-(trimethylsilyl)ethanol (**1c**) and tertiary alcohol **1d** were also converted into **2c** and **2d** in good yields (entries 4–6). The reactions of β-hydroxy ketone **1e** (entry 7) and β-hydroxy ester **1f** (entries 8 and 9), which are prone to undergo retro-aldol reaction and/or β-elimination, afforded **2e** and **2f** in good to excellent yields. No racemization occurred during the reactions of **1f**, ethyl lactate **1g** (entry 10), and serine derivative **1h** (entry 11). The protecting groups such as Boc, TBS, and acetonide in **1h**–**j** (entries 11–13) were tolerated under the reaction conditions. A highly polar sugar tetraol **1k** (entry 14) is soluble in tetrahydrofuran and was thus successfully converted into the tetra-PMB sugar derivative **2k** in 69% yield. The reactions of phenol (**1l**, entry 15 and 16) resulted in poor yields under the conditions with (+)-10-camphorsulfonic acid or boron trifluoride–diethyl ether complex because of the competing Friedel–Crafts reaction at the aromatic rings in both the substrate and the product. In contrast, *p*-chlorophenol (**1m**, entry 17) gave the PMB ether **2m** in an excellent yield, indicating that the electron-withdrawing *p*-chloro substituent would suppress the side reaction.

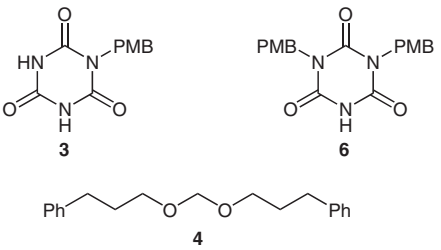
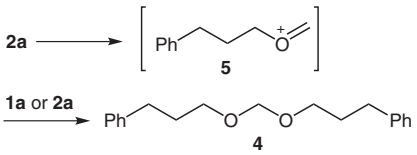
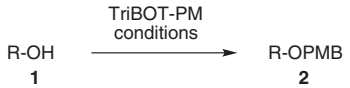


Figure 1 Byproducts in *p*-methoxybenzylation



Scheme 3 Formation of **4** through methyleneoxonium intermediate **5**

Table 2 Scope and Limitations of Various Alcohols for *p*-Methoxybenzylation



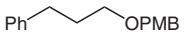
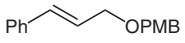
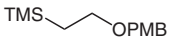
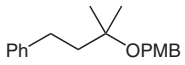
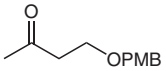
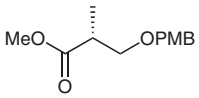
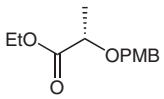
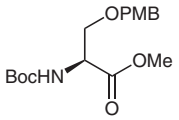
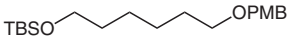
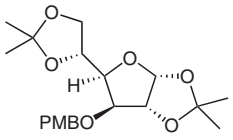
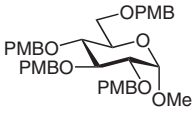
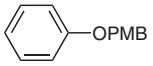
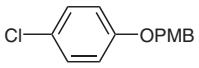
Entry	Product	TriBOT-PM (equiv)	Conditions	Yield (%) of **2**
1[a]	 **2a**	0.37	TfOH (0.3 mol%), EtOAc, r.t., 15 min	94
2[a]	 **2b**	0.4	TfOH (0.3 mol%), THF, r.t., 15 min	66
3		0.65	CSA (10 mol%), THF, reflux, 11 h	85
4[a]	 **2c**	0.4	TfOH (0.3 mol%), EtOAc, r.t., 15 min	83
5		0.6	$BF_3{\cdot}OEt_2$ (0.8 mol%), THF, 0 °C, 45 min	92
6	 **2d**	2.0	CSA (15 mol%), THF, reflux, 8 h	94
7	 **2e**	0.6	CSA (10 mol%), THF, reflux, 6 h	83
8	 **2f**	0.6	CSA (15 mol%), THF, reflux, 11 h	92
9[a]		0.4	TfOH (0.3 mol%), THF, r.t., 15 min	77
10	 **2g**	1.1	CSA (15 mol%), THF, reflux, 13 h	87
11	 **2h**	1.0	CSA (15 mol%), THF, reflux, 9 h	77
12	 **2i**	0.7	CSA (15 mol%), THF, reflux, 12 h	84
13	 **2j**	1.1	CSA (15 mol%), THF, reflux, 7.5 h	90
14	 **2k**	4.8	CSA (60 mol%), THF, reflux, 30 h	69

Table 2 Scope and Limitations of Various Alcohols for *p*-Methoxybenzylation (continued)

Entry	Product	TriBOT-PM (equiv)	Conditions	Yield (%) of **2**
15	 **2l**	0.6	$BF_3{\cdot}OEt_2$ (0.5 mol%), THF, 0 °C, 2 h	15
16		0.6	CSA (20 mol%), THF, reflux, 1.5 h	<1
17	 **2m**	0.65	CSA (15 mol%), THF, reflux, 9 h	94

[a] A solution of TriBOT-PM in the same solvent as the reaction mixture was added dropwise over 10 min for entries 1, 2, 4, and 9.

In conclusion, we successfully developed a new reagent, TriBOT-PM, for *p*-methoxybenzylation that is superior to the conventional reagent (PMBTCAI) with respect to atom economy, stability, and cost. Because TriBOT-PM can be activated by not only trifluoromethanesulfonic acid but also other weaker acids, such as boron trifluoride–diethyl ether complex and (+)-10-camphorsulfonic acid, various alcohols possessing alkali-labile or acid-labile functional groups are converted into PMB ethers in good yields. In addition, common ethereal solvents (1,4-dioxane, DME, and THF) and ethyl acetate can be utilized for the reaction; therefore, various combinations of the solvents and acids are available depending on the requirements of the substrate.

NMR spectra were determined on a Jeol JNM-ECS400 spectrometer [^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz)] or a Jeol JNM-ECA600 [^{13}C NMR (150 MHz)], relative to TMS (^{1}H) as the internal standard or TMS or residual solvents (^{13}C, $CDCl_3$: δ = 77.0, DMSO-d_6: δ = 39.5) as the internal standard. IR spectra were recorded on a Horiba FT-720 FREEXACT-II spectrophotometer. Mass spectra were measured on a Micromass Zq2000 spectrometer (ESI-MS), JMS-SX102A (FAB and EI). Analytical TLC was performed on Merck precoated analytical plates, 0.25 mm, silica gel 60 F_{254}. Preparative TLC separations were performed on Merck analytical plates (0.50 mm) precoated with silica gel 60 F_{254}. Flash chromatography separations were performed on Kanto Chemical Silica Gel 60 N (spherical, neutral, 40–100 mesh) unless otherwise noted. Recycling preparative HPLC was performed with Japan Analytical Industry LC-928 equipped with GPC columns Jaigel-1H and 2H. Reagents were commercial grades and were used without any purification unless otherwise noted. Anhyd Et_2O, 1,4-dioxane, THF, and CH_2Cl_2 were purchased from commercial sources. DME and EtOAc were purchased from commercial sources and distilled before use. All reactions sensitive to O_2 or moisture were conducted under a N_2 atmosphere.

2,4,6-Tris(*p*-methoxybenzyloxy)-1,3,5-triazine (TriBOT-PM)[8a]
To a suspension of NaH (60%) (8.64 g, 216 mmol) in THF (360 mL), anisyl alcohol (24.4 mL, 198 mmol) was added dropwise at 0 °C. The mixture was warmed to r.t., stirred for 50 min, and then cooled to 0 °C. Cyanuric chloride (11.07 g, 60.0 mmol) in THF (120 mL) was added dropwise over 30 min. The mixture was stirred for an additional 30 min at 0 °C and 3 h at r.t., the mixture was poured into H_2O and cooled to 0 °C. After 1 h, the precipitate was filtered, and washed with H_2O (2 × 200 mL), hexane (2 × 100 mL), and H_2O (200 mL). The residue was recrystallized (CH_2Cl_2–MeOH) to give 2,4,6-tris(*p*-methoxybenzyloxy)-1,3,5-triazine (26.2 g, 89%) as colorless crystals; mp 101.5–102.2 °C.

IR (KBr): 2956, 2835, 1614, 1558, 1429, 1340, 1248, 1120, 820 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ = 7.37 (d, *J* = 8.7 Hz, 6 H), 6.89 (d, *J* = 8.7 Hz, 6 H), 5.38 (s, 6 H), 3.81 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 173.0, 159.8, 130.2, 127.5, 113.9, 69.7, 55.3.

HRMS (FAB): *m*/*z* $[M + H]^+$ calcd for $C_{27}H_{28}N_3O_6$: 490.1978; found: 490.1984.

Anal. Calcd for $C_{27}H_{27}N_3O_6$: C, 66.25; H, 5.56; N, 8.58. Found: C, 66.05; H, 5.63; N, 8.48.

***N*-(*p*-Methoxybenzyl)isocyanuric Acid (3)**
Colorless crystals; mp 256.5–258.0 °C.

IR (KBr): 3224, 3093, 2964, 2837, 2789, 1772, 1684, 1469, 1254, 1034 cm^{-1}.

^{1}H NMR (DMSO-d_6): δ = 11.47 (br s, 2 H), 7.24 (d, *J* = 8.7 Hz, 2 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 4.75 (s, 2 H), 3.72 (s, 3 H).

^{13}C NMR (100 MHz, DMSO-d_6): δ = 158.5, 149.9, 148.6, 129.1, 128.8, 113.7, 55.1, 43.0.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{11}H_{11}N_3O_4$: 249.0750; found: 249.0753.

Anal. Calcd for $C_{11}H_{11}N_3O_4$: C, 53.01; H, 4.45; N, 16.86. Found: C, 53.03; H, 4.43; N, 16.79.

***N*,*N*′-Bis(*p*-methoxybenzyl)isocyanuric Acid (6)**
Colorless crystals; mp 183.5–186.2 °C.

IR (KBr): 3203, 3076, 2966, 2839, 1724, 1685, 1454, 1252, 1034 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ = 8.04 (br s, 1 H), 7.41 (d, *J* = 8.7 Hz, 4 H), 6.85 (d, *J* = 8.7 Hz, 4 H), 4.94 (s, 4 H), 3.79 (s, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.5, 149.7, 148.3, 130.8, 127.7, 113.9, 55.3, 45.1.

HRMS (FAB): *m*/*z* $[M + H]^+$ calcd for $C_{19}H_{20}N_3O_5$: 370.1397; found: 370.1410.

Anal. Calcd for $C_{19}H_{19}N_3O_5$: C, 61.78; H, 5.18; N, 11.38. Found: C, 61.73; H, 5.19; N, 11.17.

p-Methoxybenzyl 3-Phenylpropyl Ether (2a)[11]

To a solution of 3-phenylpropan-1-ol (613 μL, 4.50 mmol) in EtOAc (9.2 mL), the dropwise addition of TriBOT-PM (815.1 mg, 1.67 mmol) in EtOAc (12.0 mL, prepared in a test tube) over 10 min was started at r.t. and then TfOH (0.3 mol%) in EtOAc (300 mM, 45.0 μL) was added immediately. After the dropwise addition was complete, the residual TriBOT-PM in the test tube was rinsed with EtOAc (1.3 mL) into the mixture over 1 min. The mixture was stirred for a total of 15 min and washed with sat. $NaHCO_3$ (15 mL), H_2O (15 mL), and brine (15 mL). The organic layer was dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–toluene, 1:1 to hexane to hexane–EtOAc, 19:1) to afford **2a** (1.09 g, 94%) as a clear colorless oil.

IR ($CHCl_3$): 3062, 2937, 2862, 1612, 1508, 1099 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.30–7.23 (m, 4 H), 7.20–7.14 (m, 3 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 4.43 (s, 2 H), 3.80 (s, 3 H), 3.46 (t, *J* = 6.4 Hz, 2 H), 2.70 (t, *J* = 7.8 Hz, 2 H), 1.92 (tt, *J* = 6.4, 7.8 Hz, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.1, 142.0, 130.7, 129.3, 128.5, 128.3, 125.7, 113.8, 72.6, 69.2, 55.3, 32.4, 31.4.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{17}H_{20}O_2$: 256.1463; found: 256.1467.

Anal. Calcd for $C_{17}H_{20}O_2$: C, 79.65; H, 7.86. Found: C, 79.45; H, 7.90.

Bis(3-phenylpropyloxy)methane (4)[11]

^1H NMR ($CDCl_3$): δ = 7.31–7.24 (m, 4 H), 7.22–7.15 (m, 6 H), 4.69 (s, 2 H), 3.56 (t, *J* = 6.4 Hz, 4 H), 2.69 (t, *J* = 7.8 Hz, 4 H), 1.91 (m, 4 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.9, 128.4, 128.3, 125.8, 95.4, 67.1, 32.4, 31.4.

LRMS (ESI): *m*/*z* = 307 ($[M + Na]^+$).

Cinnamyl p-Methoxybenzyl Ether (2b)[13]

High-Atom-Economy Method: To a solution of cinnamyl alcohol (129 μL, 1.00 mmol) in THF (2.50 mL), the dropwise addition of TriBOT-PM (195.8 mg, 0.400 mmol) in THF (2.25 mL, prepared in a test tube) over 10 min was started at r.t. and then TfOH (0.3 mol%) in EtOAc (100 mM, 30.0 μL) was added immediately. After the dropwise addition was complete, the residual TriBOT-PM in the test tube was rinsed with THF (0.25 mL) into the mixture over 1 min. The mixture was stirred for a total of 15 min, diluted with Et_2O (5 mL), washed with sat. $NaHCO_3$ (5 mL), H_2O (50 mL), and brine (5 mL). The organic layer was dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1) to afford **2b** (167.0 mg, 66%) as colorless crystals.

Convenient Method: To a solution of cinnamyl alcohol (77.1 μL, 0.600 mmol) and TriBOT-PM (190.9 mg, 0.390 mmol) in THF (2.00 mL), CSA (13.9 mg, 0.0598 mmol) was added at r.t. The mixture was heated to reflux for 11 h. After cooling to r.t., the mixture was diluted with Et_2O (20 mL), washed with H_2O (40 mL), 10% K_2CO_3 (3 × 20 mL), and brine (20 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1) to afford **2b** (129.5 mg, 85%) as colorless crystals; mp 38.1–40.1 °C.

IR ($CHCl_3$): 3062, 2997, 2839, 1612, 1506, 1466, 1111, 1076 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.42–7.37 (m, 2 H), 7.35–7.21 (m, 5 H), 6.89 (d, *J* = 8.7 Hz, 2 H), 6.62 (dd, *J* = 1.4, 15.6 Hz, 1 H), 6.32 (dt, *J* = 15.6, 6.0 Hz, 1 H), 4.51 (s, 2 H), 4.17 (dd, *J* = 1.4, 6.0 Hz, 2 H), 3.80 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.2, 136.7, 132.5, 130.3, 129.5, 128.5, 127.6, 126.5, 126.2, 113.8, 71.8, 70.5, 55.3.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{17}H_{18}O_2$: 254.1307; found: 254.1311.

Anal. Calcd for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13. Found: C, 80.35; H, 7.11.

(2-Trimethylsilyl)ethyl p-Methoxybenzyl Ether (2c)

High-Atom-Economy Method: To a solution of 2-(trimethylsilyl)ethanol (142 μL, 1.00 mmol) in EtOAc (2.25 mL), the dropwise addition of TriBOT-PM (195.8 mg, 0.400 mmol) in EtOAc (2.20 mL, prepared in a test tube) over 10 min was started at r.t. and then TfOH (0.3 mol%) in EtOAc (100 mM, 30.0 μL) was added immediately. When the dropwise addition was complete, the residual TriBOT-PM in the test tube was rinsed with EtOAc (0.30 mL) into the mixture over 1 min. The mixture was stirred for a total of 15 min and washed with sat. $NaHCO_3$ (5 mL), H_2O (5 mL), and brine (5 mL). The organic layer was dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–toluene–$CHCl_3$, 2:1:1 to 1:1:1) to afford **2c** (198.3 mg, 83%).

Convenient Method: To a solution of 2-(trimethylsilyl)ethanol (142 μL, 1.00 mmol) and TriBOT-PM (293.7 mg, 0.600 mmol) in THF (5.00 mL), $BF_3{\cdot}OEt_2$ in THF (200 mM, 40.0 μL) was added at 0 °C. After 45 min, the mixture was diluted with Et_2O (20 mL) and washed with 2% Na_2CO_3 (100 mL), H_2O (100 mL), and brine (20 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1) to afford **2b** (218.9 mg, 92%) as a clear colorless oil.

IR ($CHCl_3$): 2956, 2860, 1614, 1510, 1074 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.26 (d, *J* = 8.7 Hz, 2 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 4.42 (s, 2 H), 3.81 (s, 3 H), 3.58–3.51 (m, 2 H), 1.02–0.94 (m, 2 H), 0.01 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.0, 130.8, 129.2, 113.7, 72.0, 67.3, 55.2, 18.2, –1.4.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{13}H_{22}O_2Si$: 238.1389; found: 238.1397.

Anal. Calcd for $C_{13}H_{22}O_2Si$: C, 65.50; H, 9.30. Found: C, 65.20; H, 9.49.

p-Methoxybenzyl 2-Methyl-4-phenylbutan-2-yl Ether (2d)[14]

To a solution of 2-methyl-4-phenylbutan-2-ol (74.0 mg, 0.451 mmol) and TriBOT-PM (441.1 mg, 0.901 mmol) in THF (1.50 mL), CSA (15.7 mg, 0.0676 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 1.5 h (132.3 mg, 0.270 mmol), 3 h (66.2 mg, 0.135 mmol), 4.5 h (66.2 mg, 0.135 mmol), and 6 h (44.1 mg, 0.0901 mmol), respectively. The mixture was stirred for an additional 2 h, and then it was cooled to r.t. and diluted with Et_2O (10 mL), washed with H_2O (23 mL), 10% K_2CO_3 (3 × 10 mL), and brine (10 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 40:1 to 19:1) to afford **2d** (107.9 mg, 94%) as a clear colorless oil.

IR ($CHCl_3$): 3060, 2937, 2867, 1614, 1466, 1257 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.32–7.24 (m, 4 H), 7.23–7.15 (m, 3 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 4.40 (s, 2 H), 3.80 (s, 3 H), 2.76–2.68 (m, 2 H), 1.92–1.84 (m, 2 H), 1.32 (s, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.8, 142.9, 131.8, 128.8, 128.34, 128.31, 125.6, 113.7, 74.8, 63.4, 55.3, 42.3, 30.4, 25.8.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{19}H_{24}O_2$: 284.1776; found: 284.1777.

Anal. Calcd for $C_{19}H_{24}O_2$: C, 80.24; H, 8.51. Found: C, 80.19; H, 8.36.

4-(*p*-Methoxybenzyloxy)butan-2-one (2e)[15]
To a solution of 4-hydroxybutan-2-one (86.4 μL, 1.00 mmol) and TriBOT-PM (342.7 mg, 0.700 mmol) in THF (3.33 mL), CSA (23.2 mg, 0.100 mmol) was added at r.t. The mixture was heated to reflux for 6 h. After cooling to r.t., the mixture was diluted with Et_2O (33 mL), washed with H_2O (67 mL), 10% K_2CO_3 (3 × 33 mL), and brine (33 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1 to 9:1) to afford **2e** (174.3 mg, 83%) as a clear colorless oil.

IR ($CHCl_3$): 2866, 1716, 1612, 1363, 1101 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.25 (d, *J* = 8.7 Hz, 2 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 4.44 (s, 2 H), 3.80 (s, 3 H), 3.71 (t, *J* = 6.2 Hz, 2 H), 2.70 (t, *J* = 6.2 Hz, 2 H), 2.17 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 207.3, 159.2, 130.1, 129.3, 113.8, 72.9, 64.9, 55.3, 43.8, 30.5.

HRMS (FAB): *m*/*z* $[M]^+$ calcd for $C_{12}H_{16}O_3$: 208.1099; found: 208.1101.

Anal. Calcd for $C_{12}H_{16}O_3$: C, 69.21; H, 7.74. Found: C, 68.92; H, 7.46.

Methyl (*R*)-3-(*p*-Methoxybenzyloxy)-2-methylpropanoate (2f)[16]
High-Atom-Economy Method: To a solution of methyl (*R*)-3-hydroxy-2-methylpropanoate (110 μL, 1.00 mmol) in THF (2.50 mL), the dropwise addition of TriBOT-PM (195.8 mg, 0.400 mmol) in THF (2.30 mL, prepared in a test tube) over 10 min was started at r.t. and then TfOH (0.3 mol%) in THF (150 mM, 20.0 μL) was added immediately. When the dropwise addition was complete, the residual TriBOT-PM in the test tube was rinsed with THF (0.20 mL) into the mixture over 1 min. The mixture was stirred for a total of 15 min, diluted with Et_2O (15 mL), washed with H_2O (50 mL), sat. $NaHCO_3$ (5 mL), and brine (5 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1 to 9:1) to afford **2f** (183.9 mg, 77%) as a clear colorless oil.

Convenient Method: To a solution of methyl (*R*)-3-hydroxy-2-methylpropanoate (99.4 μL, 0.900 mmol) and TriBOT-PM (264.3 mg, 0.540 mmol) in THF (3.00 mL), CSA (31.4 mg, 0.135 mmol) was added at r.t. The mixture was heated to reflux for 11 h. After cooling to r.t., the mixture was diluted with Et_2O (9 mL), washed with H_2O (45 mL), sat. $NaHCO_3$ (9 mL), and brine (9 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 24:1 to 12:1) to afford **2f** (197.3 mg, 92%) as a clear colorless oil.

^1H NMR ($CDCl_3$): δ = 7.24 (d, *J* = 8.7 Hz, 2 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 4.46 (d, *J* = 11.9 Hz, 1 H), 4.44 (d, *J* = 11.9 Hz, 1 H), 3.80 (s, 3 H), 3.69 (s, 3 H), 3.63 (dd, *J* = 7.3, 9.2 Hz, 1 H), 3.46 (dd, *J* = 6.0, 9.2 Hz, 1 H), 2.82–2.72 (m, 1 H), 1.17 (d, *J* = 7.4 Hz, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 175.3, 159.1, 130.2, 129.2, 113.7, 72.7, 71.6, 55.2, 51.7, 40.1, 14.0.

LRMS (ESI): *m*/*z* = 261 ($[M + Na]^+$).

Ethyl (*S*)-2-(*p*-Methoxybenzyloxy)propanoate (2g)[17]
To a solution of ethyl (*S*)-lactate (102 μL, 0.898 mmol) and TriBOT-PM (264.3 mg, 0.540 mmol) in THF (3.33 mL), CSA (31.4 mg, 0.135 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 6 h (176.2 mg, 0.360 mmol) and 10 h (44.1 mg, 0.0901 mmol), respectively. The mixture was stirred for an additional 3 h, then it was cooled to r.t. and diluted (hexane–EtOAc, 1:1; 53 mL), washed with H_2O (80 mL), sat. $NaHCO_3$ (2 × 30 mL), and brine (30 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 25:1 to 12:1) to afford **2g** (185.4 mg, 87%) as a clear colorless oil.

^1H NMR ($CDCl_3$): δ = 7.29 (d, *J* = 8.7 Hz, 2 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 4.63 (d, *J* = 11.3 Hz, 1 H), 4.39 (d, *J* = 11.3 Hz, 1 H), 4.23 (dq, *J* = 10.8, 7.1 Hz, 1 H), 4.19 (dq, *J* = 10.8, 7.1 Hz, 1 H), 4.03 (q, *J* = 6.9 Hz, 1 H), 3.80 (s, 3 H), 1.43 (d, *J* = 6.9 Hz, 3 H), 1.30 (t, *J* = 7.1 Hz, 3 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 173.4, 159.4, 129.67, 129.66, 113.8, 73.7, 71.6, 60.8, 55.3, 18.7, 14.3.

LRMS (ESI): *m*/*z* = 261 ($[M + Na]^+$).

Boc-L-Ser(OPMB)-OMe (2h)
To a solution of Boc-L-Ser-OMe (232.0 mg, 1.06 mmol) and TriBOT-PM (521.9 mg, 0.107 mmol) in THF (3.52 mL), CSA (36.9 mg, 0.159 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 2 h (33.7 mg, 0.0688 mmol), 3 h (33.7 mg, 0.0688 mmol), 4 h (50.5 mg, 0.103 mmol), 6.5 h (33.7 mg, 0.0688 mmol), and 8 h (33.7 mg, 0.0688 mmol), respectively. The mixture was stirred for an additional 1 h, then it was cooled to r.t., diluted (hexane–EtOAc, 1:1; 53 mL), and washed with H_2O (80 mL), sat. $NaHCO_3$ (2 × 30 mL), and brine (30 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 9:1 to 6:1 to 4:1) followed by recycling preparative HPLC to afford **2h** (276.9 mg, 77%) as a clear colorless oil.

IR ($CHCl_3$): 3442, 2956, 2871, 1749, 1709, 1512, 1173 cm^{-1}.

^1H NMR ($CDCl_3$): δ = 7.20 (d, *J* = 8.7 Hz, 2 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 5.37 (br d, *J* = 8.3 Hz, 1 H), 4.47 (d, *J* = 11.9 Hz, 1 H), 4.42 (d, *J* = 11.9 Hz, 1 H), 4.42 (ddd, *J* = 8.3, 9.2, 9.2 Hz, 1 H), 3.83 (dd, *J* = 3.2, 9.2 Hz, 1 H), 3.81 (s, 3 H), 3.74 (s, 3 H), 3.65 (dd, *J* = 3.2, 9.2 Hz, 1 H), 1.45 (s, 9 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 171.2, 159.3, 155.5, 129.6, 129.3, 113.8, 79.9, 72.9, 69.6, 55.3, 54.0, 52.4, 28.3.

HRMS (FAB): *m*/*z* $[M + H]^+$ calcd for $C_{17}H_{26}NO_6$: 340.1755; found: 340.1762.

Anal. Calcd for $C_{17}H_{25}NO_6$: C, 60.16; H, 7.42; N, 4.13. Found: C, 60.42; H, 7.64; N, 4.12.

6-(*tert*-Butyldimethylsilyloxy)hexyl *p*-Methoxybenzyl Ether (2i)[5f]
To a solution of methyl (6-*tert*-butyldimethylsilyloxy)hexan-1-ol (139.5 mg, 0.600 mmol) and TriBOT-PM (205.6 mg, 0.420 mmol) in THF (2.00 mL), CSA (20.9 mg, 0.0900 mmol) was added at r.t. The mixture was heated to reflux for 12 h. After cooling to r.t., the mixture was diluted with Et_2O (6 mL) and washed with H_2O (30 mL), 10% K_2CO_3 (3 × 6 mL), and brine (6 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 38:1 to 19:1) to afford **2i** (173.8 mg, 84%) as a clear colorless oil.

^1H NMR ($CDCl_3$): δ = 7.26 (d, *J* = 8.7 Hz, 2 H), 6.87 (d, *J* = 8.7 Hz, 2 H), 4.43 (s, 2 H), 3.80 (s, 3 H), 3.59 (t, *J* = 6.4 Hz, 2 H), 3.43 (t, *J* = 6.4 Hz, 2 H), 1.66–1.57 (m, 2 H), 1.57–1.45 (m, 2 H), 1.42–1.26 (m, 4 H), 0.89 (s, 9 H), 0.04 (s, 6 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.1, 130.8, 129.2, 113.7, 72.5, 70.1, 63.2, 55.2, 32.8, 29.8, 26.02, 25.98, 25.7, 18.4, –5.3.

LRMS (ESI): *m*/*z* = 375 ($[M + Na]^+$).

1,2:5,6-Di-*O*-isopropylidene-3-*O*-(*p*-methoxybenzyl)-α-D-glucofuranose (2j)[9,11,18]
To a solution of 1,2:5,6-di-*O*-isopropylidene-α-D-glucofuranose (104.1 mg, 0.400 mmol) and TriBOT-PM (117.5 mg, 0.240 mmol) in THF (1.33 mL), CSA (13.9 mg, 0.0598 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 3.5 h (78.3 mg, 0.160 mmol) and 6.5 h (19.6 mg, 0.0400 mmol), respectively. The mixture was stirred for an additional 1 h, the mixture was cooled to r.t., diluted with Et_2O (25 mL), and

washed with H_2O (40 mL), 10% K_2CO_3 (2 × 27 mL), and brine (27 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 19:1 to 14:1 to 9:1) to afford **2j** (137.4 mg, 90%) as a clear colorless oil.

^{1}H NMR ($CDCl_3$): δ = 7.27 (d, *J* = 8.7 Hz, 2 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 5.89 (d, *J* = 3.7 Hz, 1 H), 4.61 (d, *J* = 11.4 Hz, 1 H), 4.57 (d, *J* = 11.4 Hz, 1 H), 4.56 (d, *J* = 3.7 Hz, 1 H), 4.34 (ddd, *J* = 7.6, 8.7, 8.7 Hz, 1 H), 4.14 (dd, *J* = 3.0, 7.6 Hz, 1 H), 4.10 (d, *J* = 6.0, 8.7 Hz, 1 H), 4.00 (d, *J* = 3.0 Hz, 1 H), 3.99 (d, *J* = 6.0, 8.7 Hz, 1 H), 3.81 (s, 3 H), 1.49 (s, 3 H), 1.43 (s, 3 H), 1.38 (s, 3 H), 1.31 (s, 3 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.4, 129.7, 129.4, 113.8, 111.8, 109.0, 105.3, 82.7, 81.32, 81.31, 72.6, 72.1, 67.4, 55.3, 26.9, 26.8, 26.3, 25.5.

LRMS (ESI): *m*/*z* = 403 ([M + Na]$^+$).

Methyl 2,3,4,6-Tetrakis-*O*-(*p*-methoxybenzyl)-α-D-glucopyranoside (2k)

To a solution of methyl α-D-glucopyranoside (58.3 mg, 0.300 mmol) and TriBOT-PM (235.0 mg, 0.480 mmol) in THF (4.00 mL), CSA (41.8 mg, 0.180 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 1 h (117.5 mg, 0.240 mmol), 5.5 h (33.7 mg, 0.0688 mmol), 12.5 h (58.7 mg, 0.120 mmol), 17 h (58.7 mg, 0.120 mmol), and 21 h (117.5 mg, 0.240 mmol), respectively. The mixture was stirred for an additional 9 h, the mixture was cooled to r.t., diluted with Et_2O (12 mL), and washed with H_2O (60 mL), 10% K_2CO_3 (3 × 12 mL), and brine (12 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–EtOAc, 4:1 to 3:1 to 2:1) followed by recycling preparative HPLC to afford **2k** (139.1 mg, 69%) as a clear pale yellow oil.

IR ($CHCl_3$): 3003, 2937, 2839, 1612, 1510, 1259, 1053 cm^{-1}.

^{1}H NMR ($CDCl_3$): δ = 7.28 (d, *J* = 8.7 Hz, 2 H), 7.28 (d, *J* = 8.7 Hz, 2 H), 7.24 (d, *J* = 8.7 Hz, 2 H), 7.01 (d, *J* = 8.7 Hz, 2 H), 6.86 (d, *J* = 8.7 Hz, 2 H), 6.85 (d, *J* = 8.7 Hz, 2 H), 6.84 (d, *J* = 8.7 Hz, 2 H), 6.80 (d, *J* = 8.7 Hz, 2 H), 4.88 (d, *J* = 10.5 Hz, 1 H), 4.74 (d, *J* = 10.5 Hz, 1 H), 4.73 (d, *J* = 11.4 Hz, 1 H), 4.72 (d, *J* = 10.1 Hz, 1 H), 4.58 (d, *J* = 11.4 Hz, 1 H), 4.56 (d, *J* = 11.9 Hz, 1 H), 4.54 (d, *J* = 3.7 Hz, 1 H), 4.39 (d, *J* = 11.9 Hz, 1 H), 4.33 (d, *J* = 10.1 Hz, 1 H), 3.91 (dd, *J* = 9.2, 9.6 Hz, 1 H), 3.80 (s, 3 H), 3.80 (s, 3 H), 3.78 (s, 3 H), 3.76 (s, 3 H), 3.71–3.63 (m, 2 H), 3.60–3.52 (m, 2 H), 3.50 (dd, *J* = 3.7, 9.6 Hz, 1 H), 3.35 (s, 3 H).

^{13}C NMR (150 MHz, $CDCl_3$): δ = 159.4, 159.24, 159.18, 159.1, 131.1, 130.5, 130.3, 130.0, 129.8, 129.62, 129.60, 129.5, 113.83, 113.79, 113.74, 113.72, 98.3, 81.9, 79.5, 77.4, 75.4, 74.6, 73.05, 73.03, 70.0, 68.0, 55.28, 55.28, 55.27, 55.2, 55.1.

HRMS (FAB): *m*/*z* [M + H]$^+$ calcd for $C_{39}H_{47}O_{10}$: 675.3169; found: 675.3178.

Anal. Calcd for $C_{39}H_{46}O_{10}$: C, 69.42; H, 6.87. Found: C, 69.63; H, 7.10.

***p*-Methoxybenzyl Phenyl Ether (2l)**[19,20]

To a solution of phenol (28.2 mg, 0.300 mmol) and TriBOT-PM (88.1 mg, 0.180 mmol) in THF (1.50 mL), $BF_3{\cdot}OEt_2$ in THF (100 mM, 15.0 μL) was added at 0 °C. The mixture was stirred for 2 h, and it was quenched by solid $NaHCO_3$, filtered through silica gel, and concentrated under reduced pressure. The residue was purified by preparative TLC (hexane–$CHCl_3$, 1:1) to afford **2l** (9.6 mg, 15%) as colorless crystals; mp 90.8–93.0 °C.

^{1}H NMR ($CDCl_3$): δ = 7.36 (d, *J* = 8.7 Hz, 2 H), 7.32–7.26 (m, 2 H), 7.00–6.93 (m, 3 H), 6.92 (d, *J* = 8.7 Hz, 2 H), 4.99 (s, 2 H), 3.82 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.4, 158.8, 129.5, 129.2, 129.1, 120.8, 114.8, 114.0, 69.7, 55.3.

LRMS (EI): 214 ([M]$^+$).

***p*-Chlorophenyl *p*-Methoxybenzyl Ether (2m)**[21]

To a solution of *p*-chlorophenol (119.3 mg, 0.928 mmol) and TriBOT-PM (340.7 mg, 0.603 mmol) in THF (3.10 mL), CSA (32.3 mg, 0.139 mmol) was added at r.t. The mixture was heated to reflux and additional TriBOT-PM was added after 1 h (90.9 mg, 0.186 mmol), 2 h (45.4 mg, 0.0927 mmol), and 5 h (22.7 mg, 0.0464 mmol), respectively. The mixture was stirred for an additional 4 h, and it was cooled to r.t., diluted with Et_2O (9 mL), and washed with H_2O (47 mL), 10% K_2CO_3 (3 × 9 mL), and brine (9 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under reduced pressure. The residue was purified by column chromatography (hexane–$CHCl_3$, 1:1) to afford **2m** (216.6 mg, 94%) as colorless crystals; mp 106.0–107.0 °C.

^{1}H NMR ($CDCl_3$): δ = 7.34 (d, *J* = 8.7 Hz, 2 H), 7.23 (d, *J* = 9.0 Hz, 2 H), 6.91 (d, *J* = 8.7 Hz, 2 H), 6.89 (d, *J* = 8.9 Hz, 2 H), 4.95 (s, 2 H), 3.81 (s, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 159.5, 157.4, 129.29, 129.22, 128.5, 125.7, 116.1, 114.0, 70.1, 55.3.

LRMS (EI): *m*/*z* = 248 ([M]$^+$).

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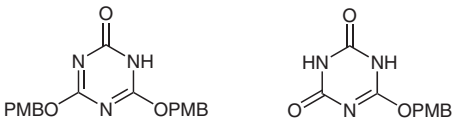

Figure 2 Other intermediates for N-*p*-methoxybenzylation

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