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# Chromium-mediated aldol and homoaldol reactions on solid support directed towards an iterative polyol strategy

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**Abstract**—Chromium-Reformatsky and chromium-homoaldol reactions run under neutral and mild reaction conditions. They are highly chemoselective, tolerant towards most common functional groups, and are not prone to retroaldol reactions. Initial studies directed to transfer these homogeneous chromium-mediated solution-phase reactions to solid phase are presented. The main objective was to develop a methodology to aid a combinatorial iterative strategy to polyols (polyketides) on solid phase. A general reactivity problem was observed with polystyrene based resins compared to the solution-phase reactions, independent if the electrophilic (aldehyde) or nucleophilic (bromide) end of the polyol chain was supported to the resin. A complicated penetration, or loss of the polar solvent environment after penetration into the resin, might be responsible for the reduced reactivity. Application of either a soluble polystyrene resin or a polystyrene resin with a polar polyethylene glycol tether resulted in improved yields.


A method of crucial importance for the construction of carbon–carbon bonds in natural and artificial synthetic chemistry is the aldol reaction.[1,2] One of the oldest selective methods for cross-coupling aldol reactions is the Reformatsky reaction.[3] This redox-initiated aldol reaction allows the enolate formation at a predefined position, independent of thermodynamic and kinetic C–H acidities. Especially attractive is the chromium-Reformatsky[4,5] reaction, which tolerates most common functional groups, and has a high chemoselectivity towards aldehydes.[6] Even more, this reaction, performed under mild and neutral reaction conditions, is not effected by retroaldol reactions.[7,8] The kinetic *anti*-aldol products of esters are the result of a 'kinetic quench' of the formed chromium-alcoholate, and are normally difficult to obtain.[4,8] Chromium-Reformatsky reactions using Evans-type oxazolidinones as chiral auxiliaries combine this high *anti*-selectivity with a high stereoselectivity, and have been successfully applied in the total synthesis of epothilone B.[9–11] By this method one is able to overcome the limitations of the classical Evans *syn*-aldol chemistry allowing direct access to *anti*-Evans aldols with 'non-Evans' absolute orientation. In addition, the highly stereoselective transfer of carboxylmethyl groups (so called 'acetate anion' transfer of $CH_2$–$CO_2R$) is possible.[4,9,10] A closely related and complementary chromium-mediated reaction to form 1,*n*-polyols is the Takai–Utimoto reaction, which allows the direct formation of chromium alkyls under cobalt-catalysis in the presence of many functional groups including homoenolates.[4,12] Transfer of these powerful chromium reactions from solution to solid phase would be an enrichment of the present aldol chemistry for solid-phase reactions,[13–17] and result in the first homoaldol method available for solid-phase reactions. Here we wish to report our progress to transfer the solution-phase chromium-Reformatsky and Takai–Utimoto reaction to solid phase, which consists of optimization of the resin, linker and reaction conditions. Finally, we hope that this work will form a basis for the implementation of an iterative polyol strategy to synthesize polyketides.

An iterative polyol synthesis consists of a sequence of aldol reactions in combination with the reestablishment of a key reactive functionality (Fig. 1). Development of an iterative polyol strategy is highly challenging and complicated, which is evident from the pioneering works

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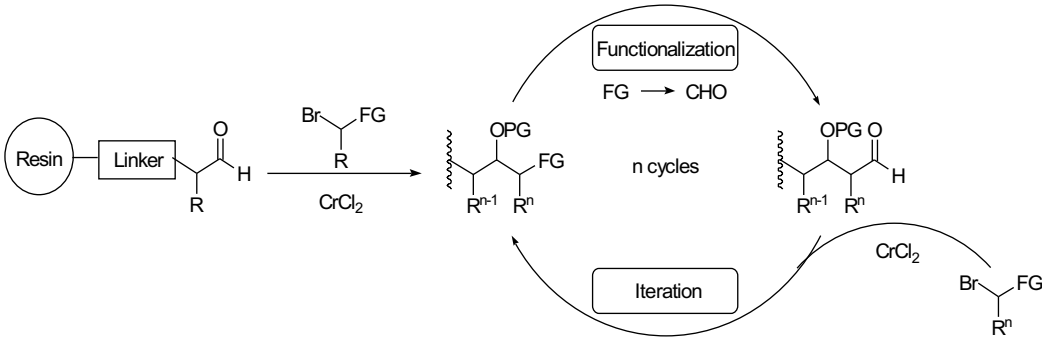



**Figure 1.** Principle of the iterative polyol synthesis utilizing chromium(II)-mediated reactions.

of Reggelin et al.,[18–20] and Paterson et al.[21,22] An important advantage of the stereoselective chromium-Reformatsky reaction is its insensitivity to matched and mismatched interactions, that is, the induction usually is not much effected by the chirality in the aldehyde.[4] For practical purposes it is also important that many chromium(II)-mediated reactions to some extent tolerate residual water or OH-groups, and do not react with, for example, keto, ester, amide or other groups important for natural products like polyketides. In principle, two inverse strategies are possible for chromium(II)-mediated reactions on solid phase: the iteration of the aldehyde (electrophile) or that of the bromide (to be converted to the organochromium nucleophile) supported to the resin. Theoretical considerations suggest to use the aldehyde supported strategy. The aldehyde key functionality can be reestablished directly from the activating carbonyl functional group, for example, Evans amide (Fig. 1). Other advantages are better yields through the suppression of pinacol coupling by spatial separation of the aldehyde groups, and a better acceptance of traces of water since the chromium(III) enolate is present in excess.

A set of aldehyde- and bromide-functionalized resins (Fig. 2) were synthesized to screen the potential of the chromium-Reformatsky- and Takai–Utimoto reaction in solid-phase assisted reactions. Coupling of butanediol with sodium hydride to the Merrifield resin followed by an oxidation with Dess–Martin Periodinane resulted in the formation of aldehyde-functionalized resin **1** (FT-IR: 1721 $cm^{-1}$).[23] Introduction of the aldehyde functionality together with an easy cleavable linker on the Merrifield resin was achieved in three steps. A nucleophilic substitution with the sodium salt of 2,2-dimethyl-4-hydroxymethyl-1,3-dioxolane, cleavage of the acetal in a one to one mixture of 1 M-HCl (aq) and dioxane, and a condensation with terephthaldehyde afforded **2** (0.86 mmol/g).[24] The direct coupling of *p*-hydroxybenzaldehyde with 2-chlorotrityl chloride resin in the presence of pyridine gave resin **3**. Resin **4** was synthesized by an etherification of the trityl chloride resin with *p*-bis(hydroxymethyl)benzene, followed by an oxidation with the pyridine complex of sulfur trioxide.[25] Functionalization of soluble chloromethyl polystyrene (1.04 mmol/g based on chlorine analysis)[26] via a nucleophilic substitution with the sodium salt of 2-(4-hydroxyphenyl)-1,3-dioxalane, and acid catalyzed cleavage of the acetal afforded resin **5**, which was confirmed by NMR.

Access to bromide-functionalized resins was more convenient and straightforward compared to the synthesis of aldehyde resins, because the desired substrates are commercially available. Synthesis was achieved in a straightforward manner by an acylation of OH- or $NH_2$-functionalized resins with an activated acid. Thus,

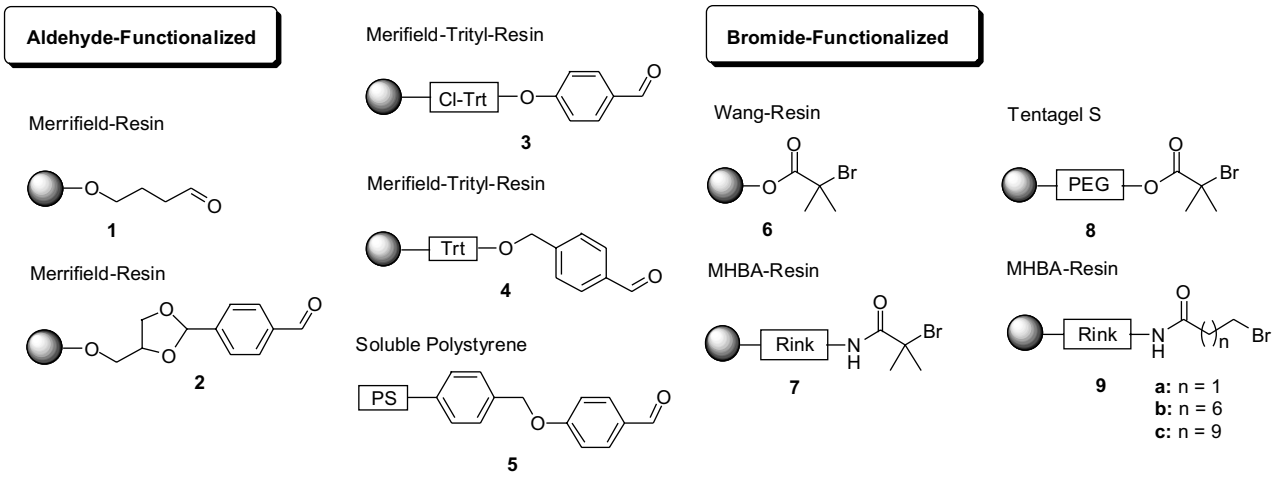


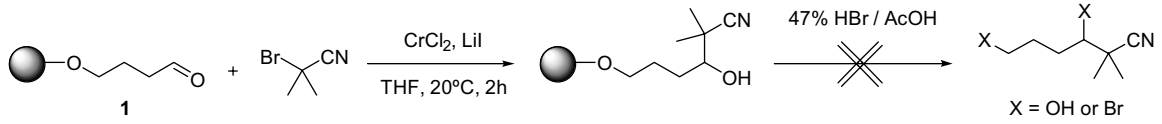

**Figure 2.** Functionalized resins.

reaction of the Wang resin with 2-bromoisobutyryl bromide and diisopropylethylamine afforded **6** with a loading of 0.68 mmol/g based on bromide analysis. The same reaction was performed on the deprotected MHBA-Rink amide- and Tentagel S OH resin, to yield **7** (Kaisertest: negative, 0.61 mmol/g) and **8** (0.25 mmol/g). Aliphatic bromide-functionalized resin **9** (Kaisertest: negative. a: 0.59 mmol/g; b: 0.61 mmol/g; c: 0.61 mmol/g) was synthesized by amidation of the corresponding diisopropylcarbodiimide activated acid with the deprotected MHBA-Rink amide resin.

Reaction of Merrifield resin **1** (Scheme 1) with 2-bromoisobutyronitrile resulted into successful coupling, based on the disappearance of the carbonyl- and appearance of the nitrile-band. Unfortunately, the ether linker was too stable, and the product could not be cleaved from the resin without severe decomposition.

Methyl-2-bromoisobutyrate was chosen as model substrate for the further development of the chromium-Reformatsky reaction (Table 1). Introduction of a suitable linker was necessary to enable cleavage of the product. The 1,3-dioxolane linker in Merrifield resin **2** is stable towards many reaction conditions and could be cleaved with 2 M aqueous HCl/dioxane (1:1). A mixture of coupling product **10b** and aldehyde **11b** was isolated in a ratio of 60:40. The absolute isolated yield, based on the theoretical loading as given by the producer of the resin is calculated to 23% of **10b** (Table 1, entry 1). Cleavage from trityl resin **3** (Table 1, entry 2) resulted only in the recovery of unreacted aldehyde, which might be explained by the low electrophilicity of *p*-methoxybenzaldehyde. Trityl resin **4** was chosen to overcome this reactivity problem, and resulted into a mixture of coupling product **10c** (6% isolated yield) and aldehyde **11c** (Table 1, entry 3). The low absolute yields do not correspond to our experience in solution phase with yields >95%. They might indicate that the reactivity of the aldehyde as such or the linker/cleavage properties are not the only reason for the low purity and yield. Another reason might be the apolar polystyrene backbone of the resins. Chromium(II)-mediated reactions require polar reaction media like THF and DMF. Even the addition of 10% toluene inhibits the chromium reaction efficiently. Thus, soluble polystyrene support **5** was applied to assure a higher interaction with the polar solvent without loss of the advantages of solid-phase synthesis.[27] Coupling product **10a** was the only isolated product in an absolute yield of 44% (Table 1, entry 4), supporting our notion that the polymer backbone bears importance for the reactivity. Unexpectedly, the soluble resin became insoluble after the first chromium-Reformatsky reaction was performed, rendering this principally suitable system useless for further iterative steps.

The complementary Takai–Utimoto reaction of methyl-3-bromopropionate with trityl resin **4** resulted in the same reaction profile as for the chromium-Reformatsky reaction (Scheme 2). An incomplete reaction with a 30:70 ratio of product **12** to aldehyde, and a low absolute yield of 17%.

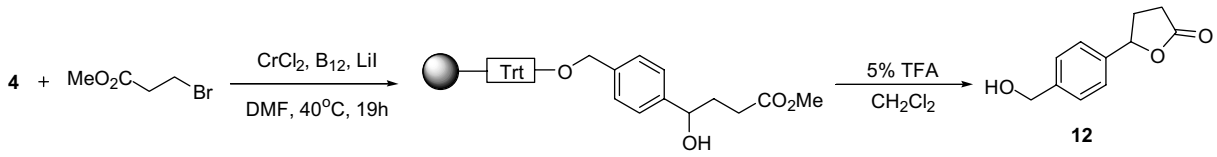



**Scheme 1.** Chromium-Reformatsky reaction of 2-bromoisobutyronitrile (3.6 equiv) with resin **1** (1.0 equiv), $CrCl_2$ (7.5 equiv) and LiI (0.5 equiv).

**Table 1.** Chromium-Reformatsky reaction of methyl-2-bromoisobutyrate with aldehyde-functionalized resins


| Entry | Resin | *T* (°C) | *t* (h) | **10** | Purity (%)[a] |
|---|---|---|---|---|---|
| 1 | **2**[b] | 20 | 6 | **10a** | 60[c] |
| 2 | **3**[d] | 40 | 16 | **10b** | —[e] |
| 3 | **4**[f] | 20–45 | 1–1.25 | **10c** | 47[g] |
| 4 | **5**[h] | 20 | 2 | **10b** | 100[i] |

[a] Purity of **10** related to **11**.
[b] **2** (1.0 equiv), methyl-2-bromoisobutyrate (2.7 equiv), $CrCl_2$ (5.9 equiv) and LiI (0.3 equiv) in DMF.
[c] Product cleaved by a 1:1 mixture of 2 M HCl and dioxane.
[d] **3** (1.0 equiv), methyl-2-bromoisobutyrate (3.2 equiv) and $CrCl_2$ (7.2 equiv) in DMF.
[e] Product cleaved with 30% trifluoroacetic acid in DCM.
[f] **4** (1.0 equiv), methyl-2-bromoisobutyrate (2.7 equiv) and $CrCl_2$ (6.6 equiv) in THF. Resin **4** was pretreated with TMSCl and $NEt_3$ in THF to protect non-oxidized free alcohol groups.
[g] Product cleaved with 5% trifluoroacetic acid in dichloromethane.
[h] **5** (1.0 equiv), methyl-2-bromoisobutyrate (2.5 equiv), $CrCl_2$ (4.5 equiv) and LiI (0.3 equiv) in THF.
[i] Product cleaved with TMSI in chloroform.[28]

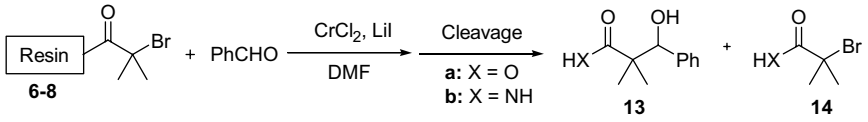



**Scheme 2.** Takai–Utimoto reaction of methyl-3-bromopropionate (2.1 equiv) with aldehyde resin **4** (1.0 equiv), CrCl2 (5.2 equiv), cyanocobalamine (0.1 equiv) and LiI (0.7 equiv).

The nucleophile supported strategy has the disadvantage that inorganic Cr(II) has to penetrate the polymer and that the bromide key functionality is troublesome to reestablish when applied in an iterative synthesis. Advantages are the direct accessability to bromide-functionalized resins, the usually cheaper aldehydes are applied in excess, and the loading of the bromide-functionalized resins can be determined conveniently by elemental analysis. The methyl-2-bromoisobutyrate functionalized resins for the Reformatsky reaction were screened for their efficiency in the coupling with benzaldehyde. A first attempt with Wang resin **6** resulted in the isolation of 24% of the desired product **13a** (Table 2, entry 1), but with the presence of a considerable amount of unreacted bromide. Higher temperature and longer reaction time led to complete reaction of the bromide (Table 2, entry 2) at the cost of a lower absolute yield of 11%. Application of MHBA resin **7** resulted in the same trend (Table 2, entry 3), a low absolute yield of 12%, without complete consumption of the bromide. These observations are in agreement with the previously obtained results of the aldehyde-functionalized resins. The apolarity of the polystyrene backbone seems to reduce the reactivity of the chromium reagent. Tentagel S resin **8** offers the possibility to study this reaction in the more polar, cation complexing environment of polyethylene glycol side chains, with the reactive centre remote from the polystyrene backbone. This resulted in a complete conversion of the bromide, an increased yield (35% over all steps, based on theoretical loading) and high purity of the desired product (Table 2, entry 4). Substitution of the bulk solvent DMF by THF did not effect the reaction (Table 2, entry 5).

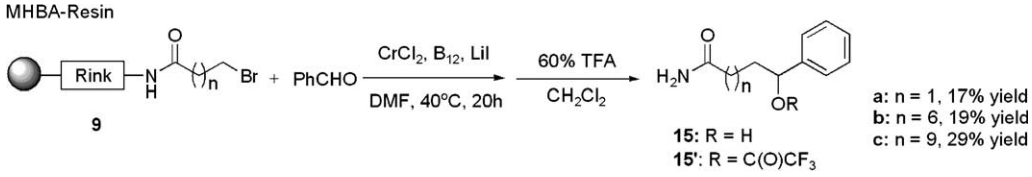
A longer distance to the polystyrene backbone appears to be the key to higher yields in chromium-mediated reactions on solid phase. Reaction of MHBA resins **9a–c** with benzaldehyde resulted, after cleavage with trifluoroacetic acid, in a mixture of alcohol and trifluoroacetate products (Scheme 3). The increased total yields

**Table 2.** Chromium-Reformatsky reaction of benzaldehyde with bromide-functionalized resins




| Entry | Resin | $T$ (°C) | $t$ (h) | 13 | Purity (%)[a,b] |
|---|---|---|---|---|---|
| 1 | **6**[c] | 20 | 1.50 | **13a** | 65[d] |
| 2 | **6**[e] | 60 | 3.00 | **13a** | 100[d] |
| 3 | **7**[f] | 20 | 1.50 | **13b** | 34[g] |
| 4 | **8**[h] | 40 | 17.0 | **13a** | 100[i] |
| 5 | **8**[h] | 40 | 17.0 | **13a** | 100[i,j] |

[a] Purity of **13** related to **14**.
[b] Low isolated yields are probably the result of low active loading or the volatility of debrominated **14**.
[c] **6** (1.0 equiv), PhCHO (5.6 equiv) and $CrCl_2$ (4.8 equiv) in DMF.
[d] Cleavage performed with 60% trifluoroacetic acid in dichloromethane. Transesterification/cleavage did not succeed with catalytic NaOMe in MeOH/THF.
[e] **6** (1.0 equiv), PhCHO (3.9 equiv), $CrCl_2$ (3.9 equiv) and LiI (1.0 equiv) in DMF.
[f] **7** (1.0 equiv), PhCHO (6.2 equiv) and $CrCl_2$ (5.2 equiv) in DMF.
[g] Cleavage performed with 80% trifluoroacetic acid in dichloromethane. Transamidation/cleavage did not occur with *n*-butylamine.
[h] **8** (1.0 equiv), PhCHO (8.0 equiv), $CrCl_2$ (9.8 equiv) and LiI (1.12 equiv) in DMF. Treated **8** with $TMSCl/NEt_3$ to remove $H_2O$.
[i] Cleavage performed with 1 M-NaOH. Elemental analysis of **8** after Reformatsky reaction: 0% Br.
[j] Reaction performed in THF, THF/DMF mixture formed in a ratio of 3/1 to 4/1 as the $CrCl_2$ was added as a DMF-solution.




**Scheme 3.** Takai–Utimoto reaction of benzaldehyde (5.0 equiv) with resins **9** (1.0 equiv), $CrCl_2$ (6.0 equiv), cyanocobalamine (0.1 equiv) and LiI (0.5 equiv). Yields are based on theoretical loading and thus should only be considered in relative terms.

(based on theoretical loading) of alcohol and trifluoroacetate products with an increased length of the aliphatic chain again indicate that the presence of the polar solvent medium is crucial for high conversion and purity (and higher yields).

Chromium-Reformatsky and chromium-homoaldol reactions performed on solid phase are severely effected by polymer backbone, spacer and linker type. The reactivity generally depends very much on the polarity and complexation properties of the polymer and spacer. Also, an electron poor linker is preferred. This is independent of the substrate directionality, that is, from the bromide or aldehyde functionality being attached to the solid support. Three possible reasons may explain this effect. First, penetration of the chromium reagent into the resin may be the problem. Second, chromium-mediated reactions do not work in apolar solvents like toluene or even in polar solvents mixed with toluene, whereby toluene is a good mimic for the Merrifield resin environment. Third, chromium complexes and reactions are extremely sensitive to steric effects, and thus may be effected by substrates close to the backbone. The presence of the very lipophilic polystyrene backbone, together with an eventual loss of coordinating polar solvent molecules after penetration into the solid phase may cause the reduced reactivity. Application of a soluble linear polystyrene supported aldehyde (**5**) in the chromium-Reformatsky reaction released the steric strain and allowed somewhat more influence of polar solvent, and solved the reactivity problem. The insolubility of the soluble polystyrene resin after reaction, however, constitutes a severe disadvantage with respect to its application in an iterative polyol strategy.

Separation of the polystyrene backbone and the bromide functionality by a polar polyethylene glycol spacer (**8**), or an apolar aliphatic spacer (**9c**), resulted in both cases in an increased yield, and a completely converted bromide substrate. These first results will be used for a further development of chromium-mediated reactions on solid support, and optimized reaction conditions will be used to introduce the enantioselective chromium-Reformatsky reaction on solid phase. The future focus will be on the application of novel polar resins and easily cleavable linkers to bring this method to a level at which it can be applied in an iterative polyol strategy.[29]

*General reaction procedure for the chromium-mediated reactions with a resin supported substrate*: All reactions were carried out under an argon atmosphere in flame-dried glassware using standard syringe, septa and glove-box techniques. Absolute solvent (10 mL/g resin) was added to the resin, followed by benzaldehyde or bromide. The required quantities of $CrCl_2$, LiI and cyanocobalamine were dissolved in DMF (1 mL/g $CrCl_2$) and added to the resin. This mixture was shaked at the temperature and time mentioned in the article. The resin was filtered, and washed several times with DMF, DMF/$H_2O$, THF/$H_2O$ and THF. Standard reaction conditions were used for each linker type to cleave the products from the resins. A general problem was the removal of chromium residues from the resin under the applied washing procedure, with the exception of Tentagel S. If removal of chromium occurs under cleavage conditions, this resulted in considerably decreased yield because of additional purification requirements.

*Methyl-2,2-dimethyl-3-hydroxy-3-(4-formylphenyl)-propionate* (**10a**): $^1$H NMR (300 MHz): $\delta$ 9.93 (s, 1H), 7.77 (d, $J = 7.9$, 2H), 7.39 (d, $J = 7.9$, 2H), 4.89 (s, 1H), 3.63 (s, 3H), 1.10 (s, 3H), 1.05 (s, 3H).

*Methyl-2,2-dimethyl-3-(4-hydroxyphenyl)-propionate* (**10b**): $^1$H NMR (300 MHz): $\delta$ 7.12 (d, $J = 8.7$, 2H), 6.82 (d, $J = 8.7$, 2H), 4.41 (s, 1H), 3.72 (s, 3H), 1.11 (s, 3H), 1.01 (s, 3H).

*Methyl-2,2-dimethyl-3-hydroxy-3-(4-hydroxymethylphenyl)-propionate* (**10c**): $^1$H NMR (250 MHz): $\delta$ 7.40–7.10 (m, 5H), 4.9 (s, 1H), 4.8 (s, 2H), 3.7 (s, 3H), 1.1 (s, 6H).

*4-[4-(Hydroxymethyl)phenyl]-butyrolactone* (**12**): $^1$H NMR (250 MHz): $\delta$ 7.5–7.2 (m, 4H), 5.6–5.5 (m, 1H), 4.75 (s, 2H), 2.8–2.6 (m, 3H), 2.3–2.1 (m, 1H).

*2,2-Dimethyl-3-hydroxy-3-phenyl propionic acid* (**13a**): $^1$H NMR (250 MHz): $\delta$ 7.40–7.30 (m, 5H), 4.91 (s, 1H), 4.60 (br, 1H), 1.17 (s, 3H), 1.15 (s, 3H).

*2,2-Dimethyl-3-hydroxy-3-phenylpropionamide* (**13b**): MS-ESI (positive), *m/z* (%): 194 (22) $[M+H]^+$, 211 (29) $[M+NH_4]^+$.

*4-Hydroxy-4-phenyl-butanamide* (**15a**): $^1$H NMR (250 MHz): $\delta$ 7.5–7.3 (m, 5H), 5.52 (t, $J = 8$, 1H), 2.8-2.6 (m, 3H), 2.3–2.1 (m, 1H).

*9-Hydroxy-9-phenyl-nonaamide* (**15b**): $^1$H NMR (250 MHz): $\delta$ 7.4–7.2 (m, 5H), 4.85 (t, $J = 9$, 1H), 2.20 (t, $J = 8$, 2H), 2.1–1.6 (m, 4H), 1.4–1.1 (m, 8H).

*12-Hydroxy-12-phenyl-dodecanamide* (**15c**): $^1$H NMR (250 MHz): $\delta$ 7.4–7.2 (m, 5H), 5.9 (br, 1H), 5.7 (br, 1H), 4.67 (t, $J = 8$, 1H), 2.24 (t, $J = 8$, 2H), 1.8–1.5 (m, 4H), 1.5–1.2 (m, 14H).

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