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$I_2CHCOCHI_2/PPh_3$: A highly efficient reagent for iodination of alcohols

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ABSTRACT: A new and versatile protocol for converting alcohols into alkyl iodides utilizing $PPh_3/I_2CHCOCHI_2$ is established. Under mild and neutral conditions, this reagent produced higher product yields when compared to PPh_3/CI_4 in the iodination of various alcohols. Competitive reactions with selected iodinating agents and CBr_4 in ^1H NMR studies showed that the reactivity of $I_2CHCOCHI_2$ was greater than general I_2/imidazole and *N*-iodosuccinimide (NIS). Consequently, the $PPh_3/I_2CHCOCHI_2$ condition provides one of the attractive alternatives for iodination.

KEYWORDS: alcohols, alkyl iodides, iodination, tetraiodoacetone, triphenylphosphine

INTRODUCTION

Alkyl iodides are one of the most frequently used intermediates in organic synthesis. They can react with a wide range of nucleophiles such as alcohols, amines, and thiols to provide the corresponding substituted products, which bring about to the synthesis of several bioactive compounds [1–4]. Many functional group interconversions in drug manufacturing involve the iodination of alcohols. The main reason is the straightforward conversion process, together with the wide variety, ease of preparation, low cost, and commercial availability of alcohols [5–7]. For instance, the iodination of alcohols could be accomplished by several reagents such as HI [8] and BINOL-PCl/I_2 [9]. One applicable protocol involves the use of PPh_3 in combination with iodinating agents such as PPh_3/I_2 [10–12], PPh_3/CI_4 [13, 14], PPh_3/NIS [15] and PPh_3/DEAD/MeI [16]. The use of these coupling reagents is recognized since the transformation of alcohols could be performed under mild and acid-free conditions to give the desired alkyl iodides in high to excellent yields. However, some methods require strongly acidic conditions or specialized reagents as shown in Table 1. In this context, the present $PPh_3/I_2CHCOCHI_2$ system provides an alternative protocol that operates under mild and neutral conditions at room temperature and affords high to excellent yields for various alcohol substrates.

Recently, we have studied the reactivity of various halogenating agents for the chlorination and bromination of alcohols to give the corresponding alkyl chlorides and bromides, respectively. The results revealed that the halogenating agents possessing strong electron-withdrawing groups, such as $Cl_3CCOCCl_3$ and $Br_3CCOCBr_3$, showed the highest efficiency and reactivity for chlorination and bromination, respectively [17, 18]. The present study extended our previous reports to explore a new and alternative iodinating agent. Nevertheless, no reports involving the preparation of I_3CCOCI_3 have been addressed, probably due to its stability. Therefore, we have been interested in using $I_2CHCOCHI_2$ because this compound was prepared in 1969 and has never been used as an iodinating agent [19, 20]. Herein, we report the use of $PPh_3/I_2CHCOCHI_2$ for the practical conversion of alcohols into alkyl iodides and a relative reactivity study by comparing with previously reported reagents.

MATERIALS AND METHODS

Chemicals and equipments

All reagent-grade chemicals and solvents were purchased from commercial suppliers (Sigma-Aldrich (St. Louis, MO, USA); TCI Chemicals (Tokyo, Japan); Acros Organics (Geel, Belgium)). Proton (^1H) and carbon (^{13}C) NMR spectra were recorded on a Bruker 400 MHz AVANCE III HD (Bruker, Karlsruhe, Germany) and operating at 400 MHz (^1H) and 100 MHz (^{13}C). High resolution mass spectra (HRMS) were recorded on a Maxis Bruker spectrometer (Bruker).

The preparation of 1,1,3,3-tetraiodoacetone

Citric acid (31.23 mmol), KI (18.07 mmol) and I_2 (5.91 mmol) were dissolved in 10 ml of water and stirred for 10 min at room temperature. Then 5% $KMnO_4$ was added to the mixture until the yellowish precipitate occurred. The mixture was further stirred

Table 1 Comparison of representative methods for the iodination of alcohols.

Method	Condition	Temperature	Yield	Remarks	Ref.
HI	Strongly acidic	Reflux	Moderate–high	Requires strong acid	[8]
BINOL-PCl/I_2	Mild	0 °C to RT	Moderate–high	Reagent preparation required	[9]
PPh_3/I_2	Mild	RT	High	Classical Appel reaction	[10–12]
PPh_3/CI_4	Mild	RT	Fair–high	–	[14]
PPh_3/NIS	Mild	RT	High	Uses NIS as iodine source	[15]
PPh_3/DEAD/MeI	Mild	RT	High	Multi–component system	[16]
$PPh_3/I_2CHCOCHI_2$	Neutral condition	RT	Moderate–high	Simple work-up and no iodoform precipitation	This work

for 10 min and left still for 2 h. After that the precipitate was filtered and dissolved in 5 ml of acetone then 10 ml of water was immediately added while strong stirring. Then the precipitate occurred again and was filtered and washed by water, followed by glacial acetic acid. Finally, the precipitate was dried under vacuum to afford 2.83 g of the desired product [19]. 1,1,3,3-Tetraiodoacetone ($I_2CHCOCHI_2$): yellow solid, (^{1}H) NMR (400 MHz, $CDCl_3$): 6.04 (s, CH). (^{13}C) NMR (100 MHz, $CDCl_3$): 190.6, −37.0. HRMS (ESI) calculated for $C_3H_2I_4NaO^+$ $[M+Na]^+$: 584.6182; found 584.6161.

General procedure for preparation of alkyl iodide

A typical procedure for the preparation of an alkyl iodide: To a stirred solution of alcohol (0.25 mmol) and PPh_3 (2.5 equiv) in dry CH_2Cl_2 (0.25 M) was added $I_2CHCOCHI_2$ (0.75 equiv) or CI_4 (0.75 equiv) at room temperature (30 °C) under an Ar atmosphere. After 30 min, the reaction was quenched with aqueous $Na_2S_2O_3$ solution and dried over anhydrous Na_2SO_4. The presence of the corresponding product in the crude mixture was determined by (^{1}H) NMR analysis using toluene as an internal standard.

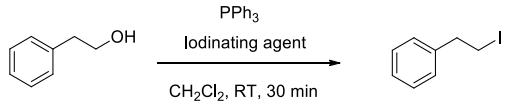
The reactivity study

To a stirred solution of alcohol (0.1 mmol) and a mixture of the iodinating agent (1 equiv) and CBr_4 (1 equiv) in dry CH_2Cl_2 (0.25 M) was added PPh_3 (2 equiv) at room temperature (30 °C) under an Ar atmosphere. After 15 min, the amount of the corresponding products in the crude mixture was determined by (^{1}H) NMR analysis.

RESULTS AND DISCUSSION

The optimized conditions were investigated for the conversion of 2-phenylethanol into 2-phenylethyl iodide using PPh_3 in combination with $I_2CHCOCHI_2$, and the results were compared with CI_4 (Table 2). Additionally, we optimized the general PPh_3/CI_4 conditions for alcohol iodination, which had not been previously reported. In the present study, CH_2Cl_2 was chosen as the reaction medium based on its ability to solubilize both the substrate and reagent, as established in our previous studies [17, 18].

Table 2 Effect of the types of iodinating agent, the ratio of iodinating agent and PPh_3 on the iodination of 2-phenylethanol.



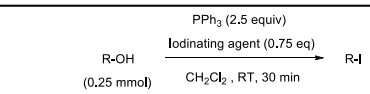
Entry	PPh_3	Iodinating agent		Yield[a]
	(equiv)	Type	Amount (equiv)	(%)
1	1.50	None	–	ND[b]
2	1.50	$I_2CHCOCHI_2$	1.50	95
3	1.50	$I_2CHCOCHI_2$	1.00	Quant
4	1.50	$I_2CHCOCHI_2$	0.75	94
5	1.50	$I_2CHCOCHI_2$	0.50	85
6	2.50	$I_2CHCOCHI_2$	0.75	Quant
7	1.50	CI_4	1.50	Trace
8	1.50	CI_4	1.00	25
9	1.50	CI_4	0.75	57
10	2.50	CI_4	1.50	92
11	2.50	CI_4	0.75	96

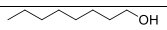
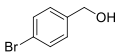
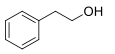
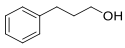
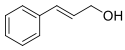
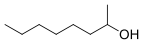
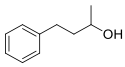
[a] Determined by ^{1}H NMR; [b] Not detected; Quant = Quantitative.

The mild and neutral nature of this protocol arises from the in situ generation of the active iodinating species from PPh_3 and $I_2CHCOCHI_2$ without the need for strong acids, strong bases, or elevated temperatures. Under the optimized conditions, alcohols were converted to the corresponding alkyl iodides at room temperature within 30 min. The operational simplicity and compatibility with structurally diverse alcohols account for the versatility of the $PPh_3/I_2CHCOCHI_2$ protocol.

Comparing the conditions of $I_2CHCOCHI_2$ to CI_4 (Table 2) under the same equivalent of PPh_3 , the results demonstrated that the yield of the desired alkyl iodide in all $I_2CHCOCHI_2$ conditions (entries 2–5) was higher than in CI_4 conditions (entries 7–9). Notably, the ratio of alcohol:PPh_3:iodinating agent at 1.0:1.5:1.0 equivalent in $I_2CHCOCHI_2$ condition (entry 3) achieved a quantitative yield, while CI_4 provided the desired product in a poor yield (entry 8). Interestingly, even reducing the amount of $I_2CHCOCHI_2$ equivalent from 1.5 to 0.5 still resulted in an excellent yield of the desired product.

Table 3 The comparative conversion of alcohols into alkyl iodides.



Entry	Alcohol	Yield[a] (%)		Ref.
		$I_2CHCOCHI_2$	CI_4	
1		Quant, 55[b]	99	[23]
2		Quant, 63[b]	34	[24]
3		Quant	73	[10]
4		Quant	Quant	[25]
5		Quant	78	[26]
6		55	17	[27]
7		95	90	[28]
8		Quant	Quant	[29]
9		Quant	62	[30]
10		65	23	[30]
11		18	ND[c]	[25]
12		24	ND[c]	[31]
13		ND[c]	ND[c]	[25]

[a] Determined by ^{1}H NMR; [b] 1.5 equiv PPh_3 was used; [c] Not detected; Quant = Quantitative.

In the optimization of various equivalent CI_4 conditions with 1.5 equivalents of PPh_3, the yields were consistently poor. However, upon increasing the equivalent of PPh_3 to 2.5, the desired alkyl iodide was obtained in excellent yields (entries 10–11). Similarly, the $I_2CHCOCHI_2$ condition with 2.5 equivalents of PPh_3 exhibited a quantitative yield (entry 6). A noteworthy observation is that, when compared to entry 4 (same equivalent $I_2CHCOCHI_2$), the yield did not increase much, indicating that $I_2CHCOCHI_2$ conditions required less PPh_3 than CI_4 conditions to achieve a quantitative yield in alcohol iodination. Moreover, our developed $I_2CHCOCHI_2$ conditions, the simplified work-up and the purification process make this reagent more practical than CI_4, as it avoids the precipitation of the iodoform by-product.

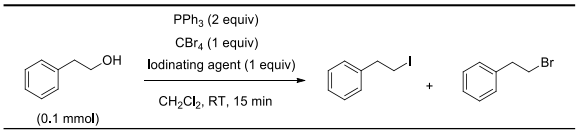
Next, we aim to explore the development conditions for the iodination of various alcohols. To ensure a fair comparison, we have selected conditions under $I_2CHCOCHI_2$ and CI_4 to provide the desired product with an excellent yield. The optimized ratios of alcohol:PPh_3:iodinating agent are 1.0:2.5:0.75. Detailed results are provided in Table 3.

We investigated the impact of various alcohols, including primary, secondary, and tertiary alcohols, as well as aliphatic and benzylic alcohols, on the development conditions. The results demonstrated that the yield of desired alkyl iodide products under all $I_2CHCOCHI_2$ conditions exceeded those obtained with CI_4. Moreover, under $I_2CHCOCHI_2$ conditions, all primary alcohols studied in this context were transformed into the corresponding alkyl iodides with high to excellent yields, except for cinnamyl alcohol, for which the desired product was obtained in moderate yield (entries 1–8). In addition, when comparing primary aliphatic alcohol (entry 1) with benzylic alcohols (entries 2–3), the results indicated that all $I_2CHCOCHI_2$ conditions yielded quantitatively, whereas under CI_4 conditions, the yields ranged from low to good, with benzylic alcohols showing significantly lower yields than aliphatic alcohol. Furthermore, the primary alcohols with and without a vinyl group (entries 5–6), both $I_2CHCOCHI_2$ and CI_4 conditions with vinyl groups, exhibited lower yield of the desired product than those without a vinyl group. The absence of rearrangement products indicates that the substitution proceeded selectively at the hydroxyl-bearing carbon.

The desired products formed from secondary alcohols were similarly obtained in low to high yields (entries 9–11). For secondary alcohols, the conversion of secondary cyclic (entry 11) displayed lower corresponding iodides than secondary alkyl alcohols (entries 9–10). Secondary cyclic exhibited low yields and no reaction under $I_2CHCOCHI_2$ and CI_4 conditions, respectively. In tertiary alcohols, low yield and no reaction were observed under $I_2CHCOCHI_2$ conditions (entries 12–13), and no reaction occurred under CI_4 conditions. This observation, arising from steric hindrance in the starting materials, further supports a direct S_N2-type substitution at the carbinol carbon rather than an S_N1/ionization pathway, as the latter would be expected to favor the formation of more substituted and stabilized carbocation-like intermediates [21, 22]. These findings demonstrate that $I_2CHCOCHI_2$ serves as an efficient iodinating reagent under the developed conditions. The reaction proceeds under mild, neutral conditions at room temperature and is applicable to a wide range of alcohol substrates. Reduced reactivity was observed for sterically hindered secondary cyclic and tertiary alcohols, consistent with steric constraints on the substitution process.

Although no mechanistic investigations were conducted in this study, the observed reactivity patterns, together with precedents from our previously reported PPh_3-mediated halogenation systems containing electron-withdrawing trihalomethyl or dihalomethylene groups [17, 18, 21], provide insight into the possible reaction pathway. The requirement of PPh_3 for efficient conversion suggests that the transformation proceeds through an Appel-type mechanism. A reactive iodophosphonium species, generated from

Table 4 Comparative reactivity study of iodinating agents.



Entry	Iodinating agent	Yield[a] (%)		Ratio of I/Br	Reactivity[b]
		RI	RBr		
1	None	–	Quant	–	–
2	I_2	20	80	0.25	1
3	I_2/Imidazole	44	56	0.79	3.16
4	NIS	41	59	0.70	2.80
5	CI_4	62	38	1.63	6.52
6	$I_2CHCOCHI_2$	57	43	1.33	5.32

[a] Determined by ^{1}H NMR; [b] Based on I_2; Quant = Quantitative.

PPh_3 and $I_2CHCOCHI_2$, is proposed to activate the alcohol substrate to form an alkoxyphosphonium intermediate, which subsequently undergoes nucleophilic displacement by iodide to afford the corresponding alkyl iodide. The reduced reactivity observed for sterically hindered secondary cyclic and tertiary alcohols is consistent with an S_N2 displacement process. Furthermore, the absence of rearrangement products argues against the involvement of carbocationic intermediates. Collectively, these observations suggest that the present transformation is mechanistically related to conventional Appel-type halogenation reactions.

To investigate the reactivity of the iodination of alcohols, the reactivity of various iodinating agents was assessed through a competitive reaction between CBr_4 and the iodinating agent towards 2-phenylethanol. The reactivity was rationalized by the ratio of the yield obtained from 2-phenylethyl iodide and 2-phenylethyl bromide by using ^{1}H NMR (Table 4).

In the absence of an iodinating agent, alkyl bromide was obtained as the sole product in quantitative yield (entry 1). General iodinating agents such as I_2, I_2/imidazole and NIS (entries 2–4) resulted in the conversion of RI less than RBr, whereas CI_4 and $I_2CHCOCHI_2$ (entries 5–6) exhibited yields of RI greater than RBr. Moreover, $I_2CHCOCHI_2$ showed significantly higher reactivity than I_2 and NIS; on the other hand, $I_2CHCOCHI_2$ displayed reactivity close to CI_4. It should be pointed out here that $I_2CHCOCHI_2$, containing four iodine atoms, did not significantly show the highest reactivity, just as was observed in the case of $Cl_3CCOCCl_3$ and $Br_3CCOCBr_3$ for the chlorination and bromination of alcohols, respectively [17, 18]. Although the $I_2CHCOCHI_2$ condition displayed slightly lower reactivity than CI_4, the results obtained from the iodination of various alcohols demonstrated that the $I_2CHCOCHI_2$ condition was still superior to CI_4. Therefore, our new $I_2CHCOCHI_2$ condition provides an attractive alternative for iodination.

To place the $PPh_3/I_2CHCOCHI_2$ protocol in the context of previously reported alcohol iodination methods, several practical advantages are noteworthy. The reaction proceeds under neutral conditions at room temperature and affords moderate to high yields across a broad range of alcohol substrates. Compared with the PPh_3/CI_4 system [13, 14], it provides improved yields under similar conditions, while offering a simpler work-up due to the absence of iodoform byproducts. In contrast to acid-mediated methods, such as those employing HI [8] or BINOL-PCl/I_2 [9], the reaction proceeds without the use of strong acids. These operationally simple and mild conditions make the $PPh_3/I_2CHCOCHI_2$ system an attractive alternative for the preparation of alkyl iodides from alcohols.

CONCLUSION

In summary, the present work introduces a new application of $I_2CHCOCHI_2$ as an iodinating reagent and establishes optimized conditions for the synthesis of alkyl iodides from alcohols using the $PPh_3/I_2CHCOCHI_2$ system. This reagent yielded comparable or higher product yields than PPh_3/CI_4 in the iodination of various alcohols. Notably, $PPh_3/I_2CHCOCHI_2$ exhibited significantly higher reactivity, competing favorably with CBr_4, in contrast to general iodinating agents such as I_2 and NIS.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at https://dx.doi.org/10.2306/scienceasia1513-1874.2026.065.

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Appendix A. Supplementary data

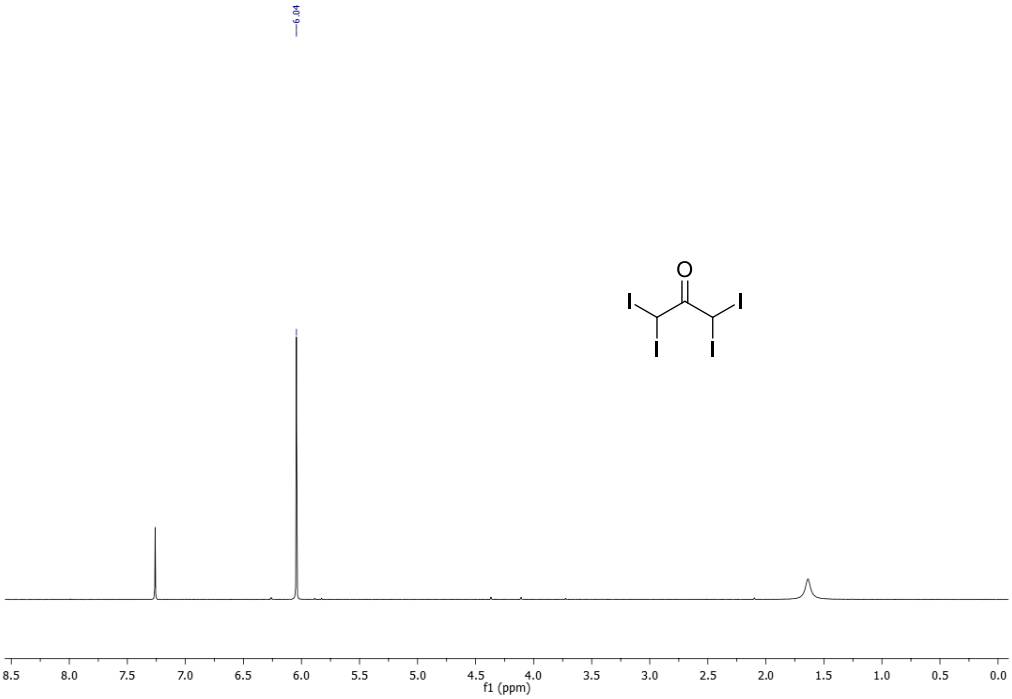


Fig. S1 ^{1}H NMR spectrum of 1,1,3,3-tetraiodoacetone ($CDCl_3$).

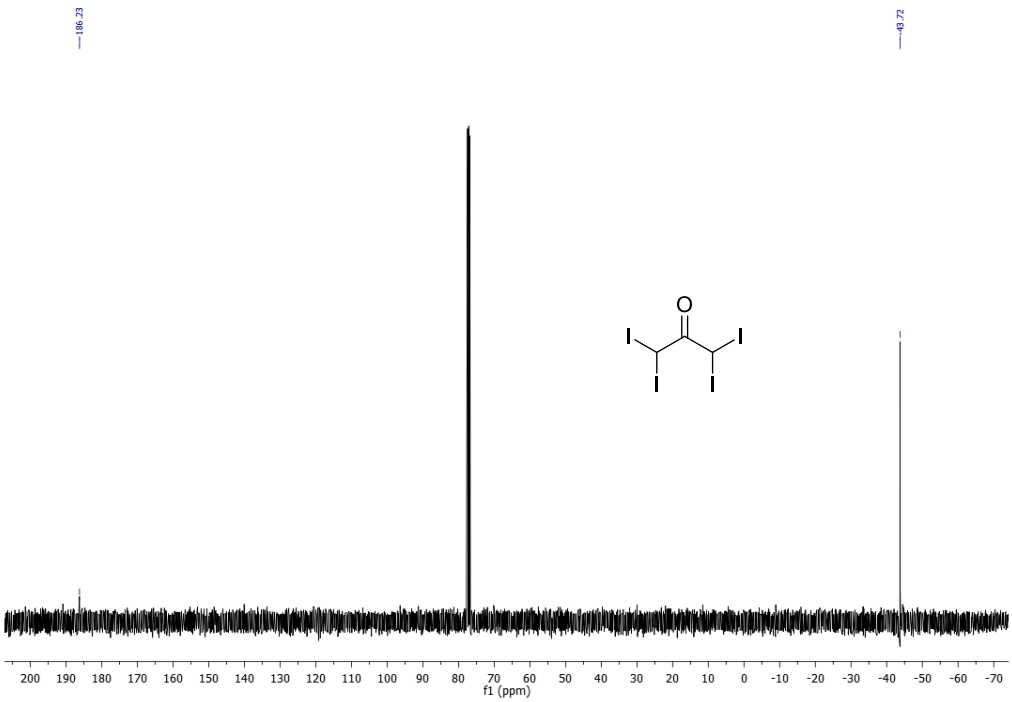
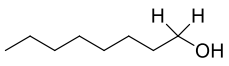
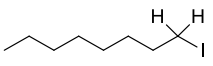
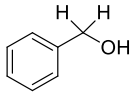
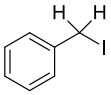
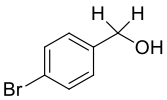
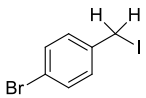
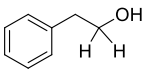
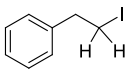
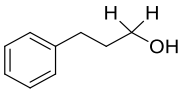
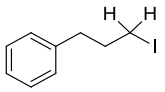
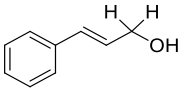
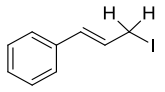
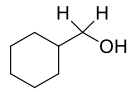
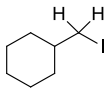
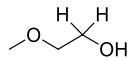
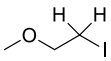
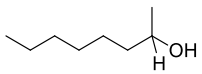
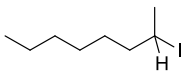
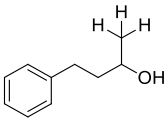
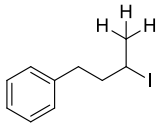
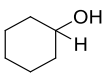
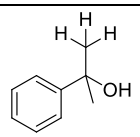
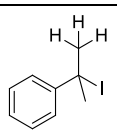
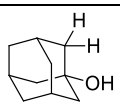
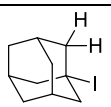


Fig. S2 ^{13}C NMR spectrum of 1,1,3,3-tetraiodoacetone ($CDCl_3$).

Table S1 The selected ^{1}H NMR spectral assignments of the corresponding alkyl iodides for structural determination compared with the literatures.

Alcohol	δ (ppm)	Alkyl iodide	δ (ppm)
	3.67 (t, J = 6.9 Hz)		3.13 (t, J = 7.0 Hz)
	4.65 (s)		4.40 (s)
	4.53 (s)		4.30 (s)
	3.90 (t, J = 7.0 Hz)		3.56 (t, J =7.7 Hz)
	3.71 (t, J = 6.6 Hz)		3.14 (t, J = 6.8 Hz)
	4.26 (dd, J = 5.6, 1.5 Hz)		4.06 (dd, J = 8.1, 0.7 Hz)
	3.82 (d, J = 6.5 Hz)		3.04 (d, J = 6.4 Hz)
	3.71 (t, J =4.5 Hz)		3.59 (t, J = 6.5 Hz)
	4.06 (m)		3.78 (m)
	1.17 (d, J = 6.2 Hz)		1.88 (d, J = 6.9 Hz)
	3.71 (m)		4.29 (m)
	1.55 (s)		2.32 (s)
	1.81 (s)		2.40 (s)