

4-Methylbenzene-1-sulfonic Acid-catalyzed Esterification of Benzoic Acid with Dodecan-1-ol in the Absence of Solvent

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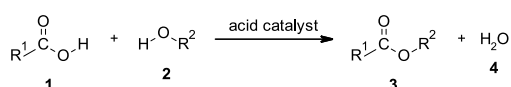
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I. Introduction

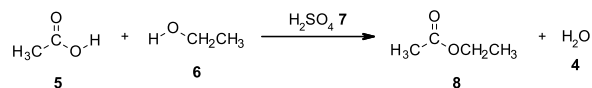
During the Pol Pot regime from 1975 to 1979, Cambodia was badly damaged by the war. The country's infrastructure was destroyed completely and intellectuals were killed. After the end of the regime, the country suffered another civil war with the closure of public sectors. After the civil war, the Royal government of Cambodia's policy was to reform the education system especially for the country development. Ministry of Education Youth and Sports (MoEYS) in Kingdom of Cambodia has given priority to science and mathematics education.¹ Although upper secondary school is usually used in Cambodia, high school is used in this paper.

In order to enhance science education, MoEYS tried to develop the curriculum cooperating with United Nations International Children's Emergency Fund (UNICEF) and United States Agency for International Development (USAID). Furthermore, MoEYS also has tried to develop the curriculum and textbook of science and mathematics cooperating with Japan International Cooperation Agency (JICA) through the Secondary School Teacher Training Project in Science and Mathematics (STEPSAM).² In order to develop science education, National Institute of Education (NIE, Cambodia) has tried to train high school teachers by workshop.³ The quality of science education, especially chemical education, was not improved because not only experimental rooms did not exist but also there were shortages of reagents and solvents in high schools in rural area. Recently, the chemical education in Cambodia has been developed a deep dependence on support from countries such as Japan.^{4,5} It is necessary to create experimental procedures which can be made full or perfect in Cambodia. In order to develop chemistry experiments in Cambodia independently of the others, the ability to plan and perform experiments needs developing through the experiments.



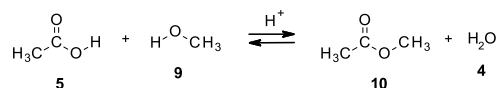
Scheme 1. Esterification of Carboxylic Acid **1** with Alcohol **2**.

In Japan, esterification of carboxylic acid is learned in high school as one of organic reaction (Scheme 1). Moreover, the synthesis of ethyl acetate (**8**) from acetic acid (**5**) and ethanol (**6**) in the presence of sulfuric acid (**7**) is shown in the chemistry textbook as an example of esterification (Scheme 2).



Scheme 2. Synthesis of Ethyl Acetate (**8**).

As an example of experiment for the esterification, carboxylic acid **5** (2.0 mL, 35 mmol) and alcohol **6** (2.0 mL, 34 mmol) are added to a dried test tube. Acid **7** (1.0 mL, 19 mmol) is gradually added to the tube, and the solution is shaken manually for 2–3 min. Then, water (10 mL) is added to the reaction mixture to give a product **8** as upper layer.⁶ In Cambodia, the esterification also exists in high school textbook. As an example of esterification, the textbook in Cambodia shows synthesis of methyl acetate (**10**) from **5** and methanol (**9**) in the presence of sulfuric acid or hydrochloric acid as a Brønsted acid catalyst. The esterification is equilibrium reaction in Cambodian textbook (Scheme 3).



Scheme 3. Synthesis of Methyl Acetate (**10**).

However, the textbook does not show experimental procedure of esterification nor that of other reactions.⁷

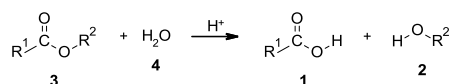
As an experiment conducted in Cambodia, we set esterification of a carboxylic acid and an alcohol in the presence of a Brønsted acid catalyst. In this experiment, if the carboxylic acid and the alcohol have low volatility, they can be used repeatedly through the hydrolysis of the product.

As a starting material, dodecan-1-ol (**13**) and benzoic acid (**12**) were used because they are commercially available, low price, and nonvolatile. Alcohol **13** (500 mL, Extra Pure Reagent of Nacalai tesque Co., Ltd.)⁸ and Acid **12**

(500 g, Extra Pure Grade of Tokyo Chemical Industry Co., Ltd.)⁹ were priced at 2,000 yen and 2,600 yen respectively in Japan. Acid **12** (mp = 124 °C, bp = 262 °C) is a solid at room temperature and have sublimability.¹⁰ Alcohol **13** (mp = 23 °C, bp = 262 °C, d = 0.832) is a liquid at room temperature, hard to dissolve with water, and have low volatility.¹¹ As an acid catalyst, 4-methylbenzene-1-sulfonic acid hydrate (**14**), which was commercially available for 2,700 yen (500 g, Extra Pure Grade of Tokyo Chemical Industry Co., Ltd.) in Japan, was used.¹² Acid **14** (mp = 106 °C, bp = 140 °C/2.7 kPa) is colorless solid at room temperature and has strong acidity.¹³ Catalyst **14** is synthesized by sulfonation of toluene with sulfuric acid. Because toluene and sulfuric acid are commercially available in Cambodia, catalyst **14** may be prepared in Cambodian experimental room.¹⁴ The reaction time was set as short as possible in order to finish the experiment within 50 min which is one period of high school in Cambodia. In the experimental procedure for Cambodian high school, we planned to heat the reaction mixture in water below 100 °C. In order to remove water from the reaction mixture, a beaker was used for the reaction. In this method, a pure product was obtained from the reaction mixture by washing with aqueous solution of sodium hydrogen carbonate.

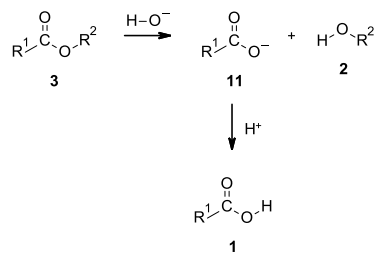
Considerable efforts have been made toward efficient synthetic methods for esters in organic research works. Synthesis of dodecyl benzoate (**15**) from dodecan-1-ol with benzaldehyde,¹⁵ dodecan-1-ol with iodobenzene and bis(cyclopentadienyldicarbonyliron),¹⁶ and dodecan-1-ol with phenylmethanol¹⁷ were reported. Recent studies have shown that efficient catalysts for esterification of carboxylic acid **12** with alcohol **13** were combination of iodine with triphenylphosphane,¹⁸ trichloroacetonitrile with triphenylphosphane,¹⁹ and so on.^{20–25}

Our interest is to develop esterification using renewable starting materials. Carboxylic acids **1** and alcohols **2** are recovered from the reaction mixture by hydrolysis of ester derivatives **3** under acidic reaction conditions (Scheme 4).²⁶



Scheme 4. Hydrolysis of Ester **3** under Acidic Conditions.

Ester derivatives **3** are also hydrolyzed under basic reaction conditions to give carboxylate derivatives **11**. Further, carboxylic acids **1** are obtained by acidification of **11** (scheme 5).²⁶ In this reaction, ester **15** is hydrolyzed with aqueous solution of sodium hydroxide. Then, alcohol **13** is collected from the organic layer and the solid of **12** is collected by acidification of the aqueous layer.

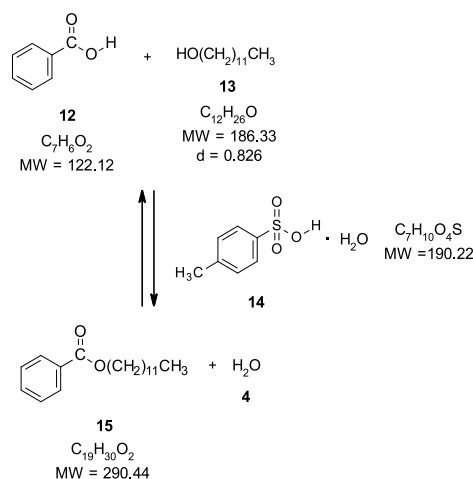


Scheme 5. Hydrolysis of Ester **3** by Hydroxide.

Therefore, in order to re-use starting materials **12** and **13**, we described Brønsted acid-catalyzed esterification of **12** with **13** in a short reaction time and in the absence of solvent with high recovery of remaining starting materials **12** and **13**.

II. Results and discussion

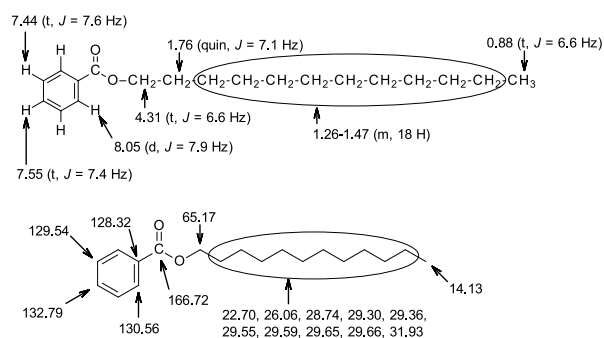
Dodecan-1-ol (**13**) (9.67 mmol), benzoic acid (**12**) (15.0 mmol), and 4-methylbenzene-1-sulfonic acid hydrate (**14**) (0.67 mmol) were added to a beaker (50 mL). The mixture was stirred for 180 min at 110 °C with heating in an oil bath, then cooled to room temperature. The reaction mixture was diluted with chloroform (90 mL). The solution was washed with aqueous solution of sodium hydrogen carbonate (30 mL × 3) and aqueous solution of sodium chloride (30 mL × 3), dried over anhydrous sodium sulfate, and concentrated under reduced pressure to give a crude product as a brown oil. The residue was purified by silica gel column chromatography eluting with hexane to afford dodecyl benzoate (**15**) in quantitative yield as a colorless oil.



Scheme 6. Synthesis of Dodecyl Benzoate (**15**).

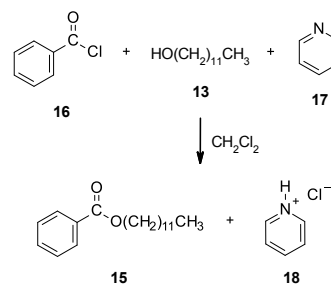
The structure of **15** was supported by the appearance of the signals of dodecyl and benzoyl group protons at 0.88 ppm (t, 3 H, $J = 6.6$ Hz, CH_3), 1.26–1.47 ppm (m, 18 H, $\text{CH}_2 \times 9$), 1.76 ppm (quin, 2 H, $J = 7.1$ Hz, CH_2) 4.31 ppm (t, 2 H, $J =$

6.6 Hz, OCH₂), 7.44 ppm (t, 2 H, $J = 7.6$ Hz, *m*-arom. H), 7.55 ppm (t, 1 H, $J = 7.4$ Hz, *p*-arom. H), 8.05 ppm (d, 2 H, $J = 7.9$ Hz, *o*-arom. H), respectively, in the ¹H NMR (CDCl₃, 400.13 MHz) spectrum. In order to assign ¹³C NMR spectrum, 2D NMR [(¹H-¹³C) HSQC and HMBC] spectra of **15** were measured. The HSQC spectrum of compound **15** showed cross peaks of the *m*-arom. H (7.44 ppm) with *m*-arom. CH (129.54 ppm), *p*-arom. H (7.55 ppm) with *p*-arom. CH (132.79 ppm), and *o*-arom. H (8.05 ppm) with *o*-arom. CH (130.56 ppm). Further, the HMBC spectrum of compound **15** also showed cross peaks (³*J*_{H,C}) of *m*-arom. H (7.44 ppm) with *m*-arom. CH (129.54 ppm), *m*-arom. H (7.44 ppm) with arom. C (128.32 ppm), *p*-arom. H (7.55 ppm) with *o*-arom. CH (130.56 ppm), *o*-arom. H (8.05 ppm) with *o*-arom. CH (130.56 ppm), and *o*-arom. H (8.05 ppm) with *p*-arom. CH (132.79 ppm). In the ¹³C NMR (CDCl₃, 100.62 MHz) spectra, dodecyl and benzoyl group carbons appeared at 14.13 (CH₃), 22.70, 26.06, 28.74, 29.30, 29.36, 29.55, 29.59, 29.65, 29.66, 31.93 (CH₂ × 10), 65.17 (OCH₂), 128.32 (arom. C), 129.54 (*m*-arom. CH), 130.56 (*o*-arom. CH), 132.79 (*p*-arom. CH), 166.72 ppm (C=O) for product **15**.



Scheme 7. Assignment of ¹H and ¹³C Spectra of Dodecyl Benzoate (**15**).

As shown in Scheme 8, an alternative synthesis of **15** was carried out with benzoyl chloride (**16**), alcohol **13**, and pyridine (**17**). The ¹H and ¹³C NMR spectra of the product were



Scheme 8. Alternative Synthesis of Dodecyl Benzoate (**15**).

identical with those of **15** and literature values.¹⁵ There were no assignment of signals in literature.

In an effort to optimize the reaction conditions, we first explored the length of time required for the reaction to stir at 110 °C. The yield of **15** was determined by ERETIC2, which is the function of TopSpin version 3.2.6. ERETIC2 gives concentration of a compound by comparing the integral value of the proton spectrum with that of reference substance.²⁷ The yield of **15**, the recovery of **12** and **13** are shown in Table 1. Time course graph for the esterification of **12** with **13** is shown in Figure 1. After stirring longer than 180 min at 110 °C, color of the reaction mixture was brown. (Table 1, Entries 1 and 2). In addition, the recovery of **12** and **13** were low (Table 1, Entries 1 and 2). Satisfactory yields were obtained for 40, 50, and 60 min (Table 1, Entries 3–5).

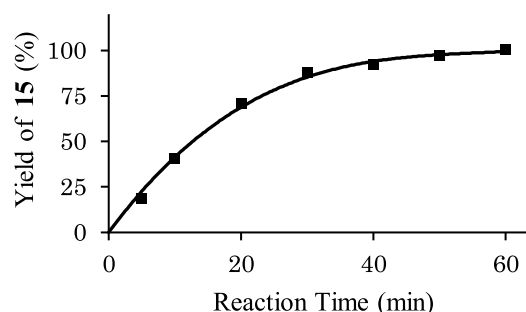


Figure 1. Time Course for the Esterification of Benzoic Acid with Dodecan-1-ol.

Table 1. Time Course for Esterification **12** with **13**.

Entry	12 (equiv)	13 (equiv)	14 (equiv)	Time (min)	Temp. (°C)	Yield of 15 (%)	Recovery of 12 (%) ^a	Recovery of 13 (%) ^b
1	1.50	1.00	0.13	180	110	quant.	10	0.4
2	1.50	1.00	0.13	120	110	quant.	14	1.1
3	1.50	1.00	0.13	60	110	quant.	18	3.8
4	1.50	1.00	0.13	50	110	97	21	5.6
5	1.50	1.00	0.13	40	110	93	26	9.3
6	1.50	1.00	0.13	30	110	82	31	21
7	1.50	1.00	0.13	20	110	71	50	32
8	1.50	1.00	0.13	10	110	41	62	65
9	1.50	1.00	0.13	5	110	18	79	85

^a Recovery of **12** was calculated from starting material of **12**.

^b Recovery of **13** was calculated from starting material of **13**.

In shorter reaction time than 40 min, yields of **15** were not satisfied (Table 1, Entries 7–9). These results indicated that both satisfactory yield and sufficient recovery were obtained after 40 min.

In the next step, we tried to optimize the reaction temperature with stirring for 40 min. The results are shown in Table 2. And the temperature dependence of the yield of **15** is shown in Figure 2. The crude product was brown oil after stirring over 110 °C (Table 2, Entries 1 and 2). The excellent yield was obtained at 110 °C (Table 2, Entry 3). The yields of **15** at 100 and 90 °C were lower than that at 110 °C (Table 2, Entries 4 and 5). Stirring at 80 or 70 °C gave yield much lower than that at 100 and 90 °C. (Table 2, Entries 6 and 7). Trace amount of **15** were obtained at 60 and 50 °C (Table 2, Entries 8 and 9). The results at 40 and 30 °C showed little reaction proceed at around room temperature (Table 2, Entries 10 and 11). These results implied the satisfactory yield was obtained at 110 °C.

Furthermore, the preferred equivalent of **12** was examined to give the results in Table 3, Entries 1–4. The yield of **15** with using **12** (1.0 equiv) was satisfactory (Table 3, Entry 1). Higher yields of **15** were obtained by higher equivalent of

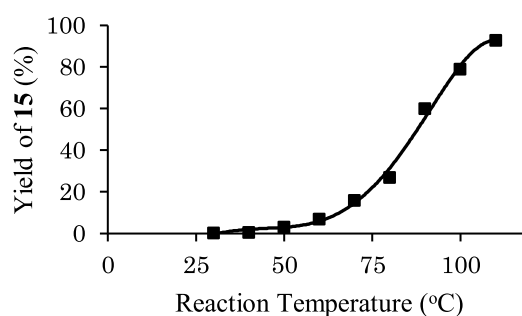


Figure 2. Temperature Dependence of the Yield of **15**.

12 (Table 3, Entries 2–4). The reaction using 1.0 equiv of **12** found to be preferred with a view to reducing costs. Finally, preferred equivalent of **14** was explored. The reaction hardly proceeds without the catalyst **14** (Table 3, Entry 5). Higher yields of **15** were given by higher equivalent of **14** (Table 3, Entries 6–8). Use of 0.10 equiv of **14** gave satisfactory yield (Table 3, Entry 9). Yields of **15** were increased by using larger amount of catalyst than 0.10 equiv (Table 3, Entries 10 and 11). These results revealed that the reaction using **14** (0.10 equiv) was preferred for both satisfactory yield and cost reduction.

Table 2. Temperature Dependence of the Yield of **15**.

Entry	12 (equiv)	13 (equiv)	14 (equiv)	Time (min)	Temp. (°C)	Yield of 15 (%)	Recovery of 12 (%) ^a	Recovery of 13 (%) ^b
1	1.50	1.00	0.13	40	130	quant.	22	1.7
2	1.50	1.00	0.13	40	120	quant.	26	2.1
3	1.50	1.00	0.13	40	110	93	26	9.3
4	1.50	1.00	0.13	40	100	79	27	24
5	1.50	1.00	0.13	40	90	60	44	44
6	1.50	1.00	0.13	40	80	27	67	55
7	1.50	1.00	0.13	40	70	16	91	78
8	1.50	1.00	0.13	40	60	7.1	80	97
9	1.50	1.00	0.13	40	50	3.1	quant.	quant.
10	1.50	1.00	0.13	40	40	0.7	28	quant.
11	1.50	1.00	0.13	40	30	0.4	25	quant.

^a Recovery of **12** was calculated from starting material of **12**.

^b Recovery of **13** was calculated from starting material of **13**.

Table 3. The equivalent of **12**, **13**, and **14** required for synthesis of **15**.

Entry	12 (equiv)	13 (equiv)	14 (equiv)	Time (min)	Temp. (°C)	Yield of 15 (%)	Recovery of 12 (%) ^a	Recovery of 13 (%) ^b
1	1.00	1.00	0.13	40	110	85	8.9	12
2	1.50	1.00	0.13	40	110	93	26	9.3
3	2.00	1.00	0.13	40	110	94	43	12
4	2.50	1.00	0.13	40	110	97	38	6.5
5	1.00	1.00	0	40	110	0.9	81	94
6	1.00	1.00	0.001	40	110	2.2	79	93
7	1.00	1.00	0.01	40	110	20	86	75
8	1.00	1.00	0.05	40	110	54	21	29
9	1.00	1.00	0.10	40	110	82	9.0	17
10	1.00	1.00	0.20	40	110	92	5.8	17
11	1.00	1.00	0.30	40	110	88	5.8	11

^a Recovery of **12** was calculated from starting material of **12**.

^b Recovery of **13** was calculated from starting material of **13**.

III. Conclusion

We found the reaction conditions of synthesis **15** with short reaction time, solvent free, high yield, and high recovery of starting materials. In these conditions, satisfactory yield was obtained with shorter reaction time and milder conditions than those in the previous literatures. The optimization and determination of the product's yield from the reaction mixture were successful by using ERETIC2. Further research is underway to convert the results to an experiment that can actually be done in Cambodia.

IV. Experimental section

1. General

All reagents and solvents were of reagents grade quality and purchased commercially. Structure of synthetic compounds were confirmed by ^1H and ^{13}C NMR spectroscopy. ^1H and ^{13}C NMR spectra were recorded with a Bruker AVANCE III instrument operating at 400.13 and 100.62 MHz, respectively. Chemical shifts were referenced to TMS in CDCl_3 as internal standard. Benzoic acid (16.6 mg, 0.136 mmol) was used for the reference substance of ERETIC2. Thin layer chromatography (TLC) was performed on precoated silica gel 60 F254 plates. Column chromatography was performed on silica gel 60N (spherical neutral) purchased from Kanto Chemical Company, Japan.

2. Typical synthesis of dodecyl benzoate

Benzoic acid (1.19–2.98 g, 9.75–24.4 mmol, 1.00–2.50 equiv) was added to dodecan-1-ol (2.20 mL, 1.81 g, 9.75 mmol, 1.00 equiv). 4-Methylbenzene-1-sulfonic acid hydrate (0–0.53 g, 0–2.8 mmol, 0–0.29 equiv) was added to the mixture. The mixture was stirred for 5–180 min at 30–130 °C. Then, the mixture was cooled with ice-water bath and distilled with chloroform (90 mL). The mixture was washed with saturated aqueous solution of sodium hydrogen carbonate (30 mL \times 3) and saturated aqueous solution of sodium chloride (30 mL \times 3), dried over anhydrous sodium sulfate, and concentrated under reduced pressure to give **15** (37.1 μmol –9.49 mmol, 0.4–97%) as a liquid. $R_f = 0.30$ (hexane)

In the case of isolation of **15**, the residue was separated by silica gel column chromatography eluting with hexane to give pure **15**.

3. The determination of concentration by ERETIC2

The reaction mixture (16.4 mg–16.7 mg) before or after washing with saturated aqueous solution of sodium hydrogen carbonate was transferred to a 1 mL volumetric

flask. The mixture was diluted with chloroform- d_1 until the solution became 1 mL. The mixture was transferred to NMR tube. The concentration of **12**, **13**, **14**, and **15** in the mixture were determined from integral value of ^1H NMR spectra by ERETIC2 with using the reference substance.

4. Synthesis of **15** from benzoyl chloride

Dodecan-1-ol (2.41 mL, 1.99 g, 10.7 mmol, 1.00 equiv) was dissolved with dry dichloromethane (39.8 mL, 0.268 M). Then, the solution was cooled by ice-water bath. Benzoyl chloride (1.86 mL, 2.27 g, 16.1 mmol, 1.51 equiv) was added to the solution. Then, pyridine (2.60 mL, 2.55 g, 32.2 mmol, 3.02 equiv) was added to the solution. The solution was stirred for 8.5 h at room temperature and for 13.5 h at 40 °C. The solution was cooled by ice-water bath. Then aqueous solution of sodium hydrogen carbonate and powder of sodium hydrogen carbonate was added to the solution and stirred for 7 h in order to decompose the remaining benzoyl chloride. The organic layer was washed with 5% w/w hydrochloric acid (40 mL \times 2), saturated aqueous solution of sodium hydrogen carbonate (40 mL \times 2), and saturated aqueous solution of sodium chloride (40 mL \times 2), dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was separated by silica gel column chromatography eluting with 3% v/v ethyl acetate-hexane to give **15** (2.38 g, 8.19 mmol, 77%) as a colorless liquid. $R_f = 0.43$ (5% v/v ethyl acetate-hexane)

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