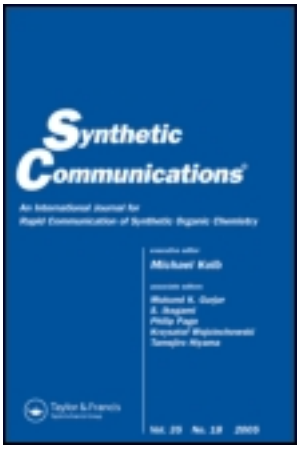

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DBU-CH_3I, a Potential Substitute for CH_2N_2 in the Preparation of Methyl Esters and Methyl Aryl Ethers: Studies with Assorted Acids

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DBU-CH_3I, a Potential Substitute for CH_2N_2 in the Preparation of Methyl Esters and Methyl Aryl Ethers: Studies with Assorted Acids

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Department of Chemistry, Indian Institute of Technology, Kharagpur, India

Abstract: DBU-CH_3I has been poised to be a substitute for diazomethane in the preparation of methyl esters from carboxylic acids. The reactions can be carried out in commercial untreated acetone and acetonitrile, which have been exemplified with several methyl esters, otherwise it is difficult to prepare. Bis-esterification using diiodomethane can also be achieved in a similar fashion. Sufficiently acidic phenols are also conveniently *O*-methylated by the method.

Keywords: DBU, diiodomethane, iodomethane, phenols, phthalaldehydic acids, pyridine carboxylic acids

Methyl esterification of carboxylic acids is an important process in the purification, characterization, and selective transformation of carboxylic acids.[1a] It is generally achieved by the use of preprepared ethereal solution of diazomethane. Despite the toxicity and direct inaccessibility, it enjoys wide acceptance because of its selectivity and mildness of reaction conditions. Shioiri's method, based on commercially available trimethylsilyldiazomethane, appears to be the best alternative to diazomethane.[1b,1c]

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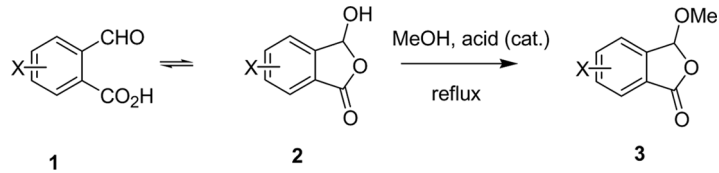
Quite some time ago, we reported a facile chemoselective *O*-methylation of carboxylic acids with 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU)-CH_3I, following the original report[2a] of Ono et al., and introduced the notion that such a methylation is faster for the stronger OH acids.[2b] The method has since found a good number of applications in organic chemistry.[3,4] To widen the scope of the method to solving some of the commonly encountered literature problems, we studied *O*-methylation of assorted carboxylic acids and phenols using readily available iodomethane. It is now amply demonstrated that DBU-CH_3I could be a good substitute for toxic diazomethane, at least for *O*-methylation of carboxylic acids and sufficiently acidic phenols. The method can be conveniently conducted on a large scale in commercial acetone as a solvent. It can also be extended to methylenation with diiodomethane.

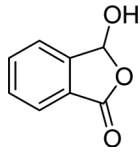
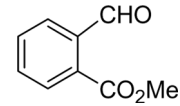
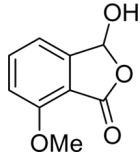
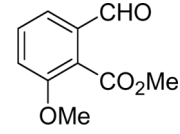
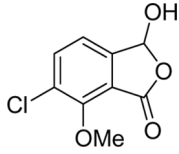
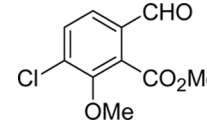
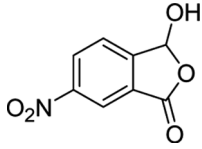
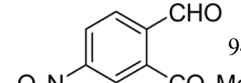
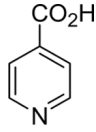
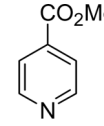
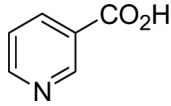
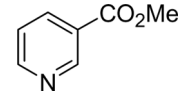
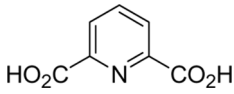
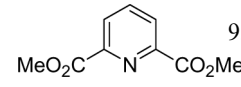
This work was initiated to solve the commonly encountered problems of methyl esterification of *ortho* formylbenzoic acids **1**, which exit in the form of phthalaldehydic acids **2**. The common method of methyl esterification involving methanol and a catalytic amount of mineral acids almost always yields 3-methoxyphthalides **3** as the sole products (Scheme 1).[5a] Attempted methyl esterification by treatment with $SOCl_2$, followed by methanol, resulted in a 57:43 mixture of methyl 2-formylbenzoate and 3-methoxyphthalide.[5b] With K_2CO_3 and a three-fold excess of iodomethane in acetonitrile, phthalaldehydic acid (**4**) was methylated in 70% yield after 24 h of reflux.[5c,d] Methyl opianate, a dimethoxyphthalaldehydic acid methyl ester, was recently prepared from the acid by treatment with ethereal diazomethane.[5e]

In the present work, a number of phthalaldehydic acids were *O*-methylated more conveniently at room temperature with DBU-CH_3I. When commercially available phthalaldehydic acid (**4**) was treated with 1 equivalent of DBU in untreated acetonitrile followed by iodomethane (3 equiv) (Table 1), the reaction provided methyl *o*-formylbenzoate **5**[6] in 87% yield. The corresponding methoxyphthalide was not formed. Likewise, substituted phthalaldehydic acids **6**, **8**, and **10** underwent smooth *O*-methylation with DBU-CH_3I to give the



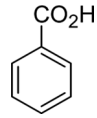
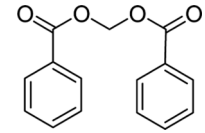
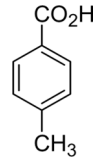
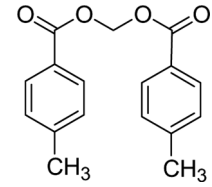
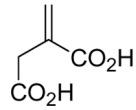
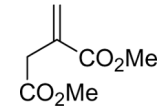
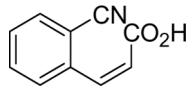
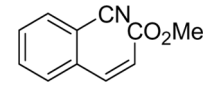
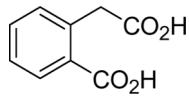
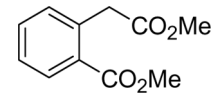
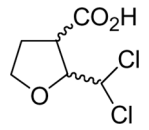
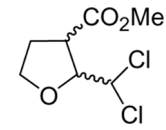
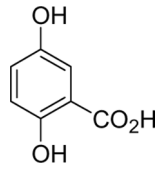
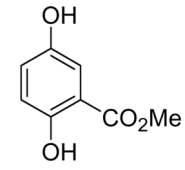
Scheme 1. Reaction of phthalaldehydic acids with methanol.

Table 1. Esterification of carboxylic acids with DBU and an alkyl halide

Acid no.	Acid	Solvent (DBU:CH_3I)	Product no.	Product	Yield[a] (%)
4		Acetonitrile (1:3)	**5**		87
6		Acetone (2:5)	**7**		82
8		Acetone (1:3)	**9**		94
10		Acetone (1:3)	**11**		94
12		Acetone (1:1.2)	**13**		62
14		Acetonitrile (1:3)	**15**		72
16		Acetonitrile (2:5)	**17**		93

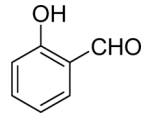
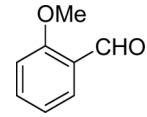
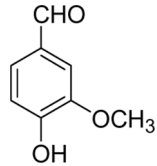
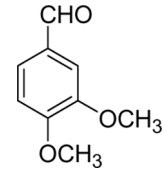
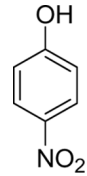
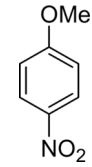
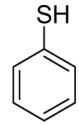
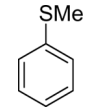
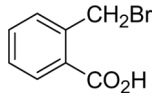
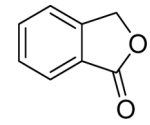
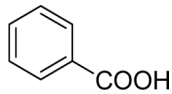
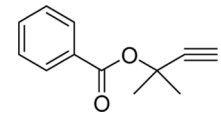
(*Continued*)

Table 1. Continued

Acid no.	Acid	Solvent (DBU:CH_3I)	Product no.	Product	Yield[a] (%)
18		Acetonitrile (2:1)[b]	**19**		69
20		Acetonitrile (2:1)[b]	**21**		66
22		Acetonitrile (2:5)	**23**		85
24		Acetonitrile (1.1:3)	**25**		92
26		Acetone (2:5)	**27**		
28[c]		Acetone (1:3)	**29**		91
30		Acetone (1:3)	**31**		92

(*Continued*)

Table 1. Continued

Acid no.	Acid	Solvent (DBU:CH_3I)	Product no.	Product	Yield[a] (%)
32		Acetone (1:3)	**33**		80
34		Acetonitrile (1:3)	**35**		84
36		Acetone (1:3)	**37**		98
38		Acetone (1:3)	**39**		89
40		Acetone (1:0)	**41**		96
42		Acetone (2:1.2)[d]	**43**		68

[a]Yields refer to the isolated yields.
[b]Diiodomethane.
[c]Mixture of cis and trans isomers.
[d]3,3-Dimethyl-3-chloropropyne.

corresponding esters **7**[6], **9**, and **11** in excellent yields. No side products were found. It is worthy of note that untreated commercial acetone works well as the reaction medium. The products were conveniently purified by quick silica-
gel filtration. Most often, they were obtained in sufficiently pure (>90%) form for further use without resorting to chromatographic purification.

For pyridine carboxylic acids **12**, **14**, and **16**, the acid-catalyzed methyl esterification with methanol is conventionally followed, which often requires long reaction time ranging between 1 and 2 days at reflux temperature.[7] Occasionally, the method based on dimethyl acetal of acetone in the presence of conc. HCl is used.[7c] The present method, however, worked well with pyridine carboxylic acids and gave esters **13**, **15**, and **17**, respectively, with the best yield for pyridine-2,6-dicarboxylic acid **16**. Analysis of NMR spectra of the crude reaction mixtures revealed that the monocarboxylic acids **13** and **15** formed some amount of *N*-methylated esters.

Considering the importance of the methylene esters in the development of organic materials,[8] we briefly examined the present method of preparing the esters using DBU. Indeed, the reactions of benzoic acid **18** and *p*-toluic acid **20** with DBU-CH_2I_2 at room temperature gave the corresponding esters **19** and **21** in good yields.

Mildness of the reaction conditions of the present method was demonstrated by the acids **22**, **24**, **26**, and **28**. Dimethyl ester **23** obtained from itaconic acid (**22**) was free from contamination with its double-bond-isomerized product.[9] However, the attempted monomethylation of the acid **22** by limiting the amount of iodomethane resulted in a mixture of monoesters and diester. For the *cis* cinnamic acid **24**, the reaction was clean. There was no trace of the more stable *trans* isomer. For homophthalic acid (**26**), which could be considered a vinylogous malonic acid, no complication arose during the methylation, although there was a possibility of *C*-alkylation at the benzylic position. With the furan acid **28**, the dichloromethyl group did not suffer any loss of HCl, and the desired product **29** was obtained in good yield.

In our earlier publication,[2b] we reported that *O*-methylation of the carboxyl groups can be performed neatly while phenolic hydroxyl groups remain intact. To test the viability of the reaction for large-scale preparation, we chose methyl 1,4-dihydroxy-2-naphthoate (**31**), which is prepared from the acid by diazomethane treatment[10a] or via the reaction of preformed sodium salt of the acid with methyl iodide in hexamethylphosphoramide (HMPA).[10b] In view of the toxicity of diazomethane and HMPA, we submitted the acid **30** to the present method of using DBU-CH_3I in acetone on a 5-g scale. The reaction provided the ester **31** in 92% yield.

During the course of our work[3c] on angucycline, we noted that with longer time and an excess of iodomethane, phenolic OH groups are converted to the methyl derivative to an appreciable extent. To find the scope of the reactivity, we submitted salicylaldehyde (**32**) to DBU-CH_3I. The ether **33** was formed in good yield. Similarly, vanillin (**34**), nitrophenol (**36**), and thiophenol (**38**) were methylated to give **35**,[11] **37**, and **39** respectively. For the phenols, the reactions were carried out with 1 equiv. of DBU and 1 equiv. of MeI in a stoppered flask. Because they were incomplete (by TLC) after 2 h, an additional molar equivalent of DBU and MeI was added. The reactions soon became complete. Workup by removal of acetone and dilution with ethyl acetate followed by washing with brine yielded the products in practically pure form.

Intramolecular cyclization[12] of *o*-bromomethylbenzoic acids is a useful methodology for the preparation of the phthalides. To improve upon the process, the acid **38** was treated with 1 equivalent of DBU in acetone. Phthalide (**41**) was formed in excellent yield at room temperature.

In conjunction with the present work, we came across the problem of preparing[13] dimethylallyl esters via the corresponding propargyl esters. To overcome the problem, we applied the present method to benzoic acid and 3,3-dimethyl-3-chloropropyne. The ester **43** was obtained in good yield.

In conclusion, the DBU-CH_3I method is ideally suited for room temperature *O*-methylation of phthalaldehydic acids, pyridine carboxylic acids, and phenols, and thus the use of hazardous diazomethane can be avoided. Short reaction time is also a characteristic of the method.

EXPERIMENTAL

Typical Experimental Procedure

DBU was added to a stirred solution of an acid or a phenol (1 mmol) in acetonitrile or acetone (5 mL) (see Table 1 for the molar ratio), and the reaction was stirred for 10 min. Iodomethane (see Table 1 for the molar ratios) was added to the mixture over a period of 10–15 min, and stirring continued for 4 h at room temperature. The reaction mixture was then concentrated and diluted with ether/ethyl acetate (50 mL). The resulting solution was washed successively with hydrochloric acid (5 mL, 2 M), water (10 mL), saturated aqueous solution of sodium thiosulfate (5 mL), and brine (10 mL). The organic layer was dried (anhydrous Na_2SO_4) and concentrated. The residue was purified by silica-gel filtration or column/preparative thin-layer chromatography (TLC) to afford pure methyl ester or methyl ether. In most cases during workup, washing the organic layer with brine only leads to a NMR-pure product.

Physical Data

The characterization data that are not reported or readily accessible are provided.

Ester **9**: mp: 40–42 °C; ν_{max} (KBr): cm^{-1}: 1729, 1567, 1454, 1270, 1110, 950, 811; ^1H NMR (200 MHz, $CDCl_3$): δ 9.92 (s, 1H), 7.60 (ABq, 2H, $J = 8.4$ Hz), 4.02 (s, 3H), 3.94 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 188.7, 165.8, 153.5, 134.7, 132.8, 131.8, 129.9, 127.3, 62.2, 52.8.

Ester **11**: mp: 64–66 °C; ν_{max} (KBr): cm^{-1}: 1727, 1533, 1444, 1286, 1135, 977, 815; ^1H NMR (200 MHz, $CDCl_3$): δ 10.7 (s, 1H), 8.85 (d, 1H, $J = 1.8$ Hz), 8.48 (dd, 1H, $J = 1.8$, 8.4 Hz), 8.09 (d, 1H, $J = 8.4$ Hz), 4.06 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): δ 190.3, 164.5, 149.8, 141.3, 132.8, 129.9, 127.0, 125.6, 53.4.

Ester **15**: ^1H NMR (400 MHz, $CDCl_3$): δ 9.22 (t, 1H, $J = 1.6$ Hz), 8.77 (dd, 1H, $J = 3.2$, 9.7 Hz), 8.35–8.20 (m, 1H), 7.45–7.30 (m, 1H), 3.95 (s, 3H).

Ester **21**: ^1H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, 4H, $J = 8$ Hz), 7.24 (d, 4H, $J = 8.4$ Hz), 6.22 (s, 2H), 2.41(s, 6H).

Ester **23**: ^1H NMR (200 MHz, $CDCl_3$): δ 6.31 (s, 1H), 5.70 (s, 1H), 3.75 (s, 3H), 3.68 (s, 3H), 3.33 (s, 2H); ^{13}C NMR (400 MHz, $CDCl_3$): δ 171.1, 166.5, 133.5, 128.5, 52.09, 52.01, 37.4.

Ester **25**: mp: 61–62 °C; 7.70–7.62 (m, 2H), 7.57 (dt, 1H, $J = 7.7$, 1.4 Hz), 7.41 (dt, 1H, $J = 7.7$, 1.4 Hz), 7.18 (d, 1H, $J = 12.3$ Hz), 6.21 (d, 1H, $J = 12.3$ Hz), 3.68 (s, 3H).

Ester **27**: ^1H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, 1H, $J = 8$ Hz), 7.49 (t, 1H, $J = 8$ Hz), 7.37 (t, 1H, $J = 7.6$ Hz), 7.27 (d, 1H, $J = 7.4$ Hz), 4.01 (s, 2H), 3.87 (s, 3H), 3.70 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$): δ 171.9, 167.4, 135.8, 132.3, 132.2, 130.9, 129.5, 127.3, 51.9, 51.8, 40.4.

Ester **29**: ^1H NMR (400 MHz, $CDCl_3$), mixture of isomers: δ 5.99 (d, 1H, $J = 8$ Hz), 5.83 (d, 1H, $J = 3.6$ Hz), 4.53–4.7 (m, 1H), 4.34–4.42 (m, 1H), 4.10–4.25 (m, 1H), 3.82–4.12 (m, 4H), 3.75 (s, 3H), 3.73 (s, 3H), 3.15–3.35 (m, 2H), 2.16–2.41 (m, 3H).

Ether **33**: ^1H NMR (400 MHz, $CDCl_3$): δ 10.39 (s, 1H), 7.73 (d, 1H, $J = 7.6$ Hz), 7.43–7.36 (m, 1H), 6.92–6.80 (m, 2H), 3.82 (s, 3H).

Ether **35**: ^1H NMR (400 MHz, $CDCl_3$): δ 9.84 (s, 1H), 7.46 (dd, 1H, $J = 8.2$, 1.6 Hz), 7.41 (d, 1H, $J = 1.6$ Hz), 6.98 (d, 1H, $J = 8.2$ Hz), 3.96 (s, 3H), 3.94 (s, 3H).

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