mixture was concentrated in vacuo. The residue was digested with hot hexane. The hexane was evaporated, leaving the crude urethane which was recrystallized from ethyl acetate–hexane mixed solvent to afford pure **10**: 0.32 g, 57%; mp 133–134 °C; ^{1}H NMR ($CDCl_3$) δ 1.3 (s, 9 H), 1.51 (*AB*, J_{AB} = 11 Hz, 1 H), 2.26 (*AB*, J_{AB} = 11 Hz, 1 H), 2.9–3.7 (m, 7 H), 5.1 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 28.34 (q), 35.29 (d), 37.67 (t), 42.22 (d), 43.57 (d), 45.30 (d), 45.96 (d), 47.31 (d) 58.14 (d), 67.14 (s), 80.20 (s), 128.36 (s), 154.14 (s); IR (KBr) 3330 (s), 2980 (m), 1660 (vs), 1550 (vs), 1360 (m), 1165 (s), 785 (m) cm^{-1}; mass spectrum (70 eV), *m/e* (relative intensity) (no molecular ion), 235.15 (3.8), 189.05 (10.0), 115.05 (9.1), 66.05 (21.6), 64.95 (10.0), 59.05 (32.9), 57.05 (100.0), 56.05 (15.9), 55.05 (11.1), 53.05 (12.0), 50.95 (15.4), 43.95 (31.2), 43.05 (11.5), 41.05 (59.3).

Anal. Calcd for $C_{15}H_{19}N_3O_6$: C, 53.41; H, 5.68. Found: C, 53.67; H, 5.55.

3,5,5-Trinitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decane (1). Urethane **10** (100 mg, 0.297 mmol) was dissolved in dry methanol (10 mL). This solution was cooled via external application of a dry ice–acetone bath, and dry hydrogen chloride gas was bubbled through the solution for 3 h. The reaction mixture was then allowed to warm to ambient temperature and was stirred overnight. The reaction mixture was then concentrated in vacuo, and the residue was washed with hexane. A colorless microcrystalline solid, 5,5-dinitropentacyclo[5.3.0.0^{2,6}.0^{3,10}.0^{4,8}]decyl-3-amine hydrochloride, (**11**, 73 mg, 90%), mp >200 °C, was thereby obtained.

Amine salt **11** (250 mg, 0.91 mmol) was dissolved in water (10 mL), and the resulting aqueous solution was extracted with ether to remove organic contaminants. The aqueous solution was rendered basic by careful addition of excess saturated aqueous sodium bicarbonate solution. The resulting turbid mixture was extracted with ether (20 mL). The ether extract was washed successively with water and with brine, dried (anhydrous sodium sulfate), and filtered, and the filtrate was concentrated in vacuo. The free amine, **12** (210 mg, 82%), was thereby obtained.

A solution of amine **12** (210 mg, 0.886 mmol) in dichloroethane (2 mL) was added dropwise to a refluxing solution of *m*-chloroperbenzoic acid (611 mg, 3.54 mmol) in dichloroethane (10 mL). After all of the amine had been added, refluxing was continued for an additional 3 h. The reaction mixture was then cooled to room temperature, and methylene chloride was added. The resulting mixture was washed sequentially with dilute aqueous sodium bicarbonate solution, with water, and with brine, dried (anhydrous sodium sulfate), and filtered, and the filtrate was concentrated in vacuo. The residue was recrystallized from ethyl acetate–hexane mixed solvent, furnishing **1** (150 mg, 61.3%) as a colorless microcrystalline solid, mp 191–192 °C: ^{1}H NMR ($CDCl_3$) δ 1.70 (*AB*, J_{AB} = 9.5 Hz, 1 H), 2.29 (*AB*, J_{AB} = 9.5 Hz, 1 H), 2.9–3.4 (m, 3 H), 3.45–3.68 (m, 1 H), 3.7–3.9 (m, 2 H), 4.0–4.2 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 35.23 (d), 37.99 (t), 42.43 (d), 43.19 (d), 44.93 (d), 46.66 (d), 50.72 (d), 58.79 (d), 92.11 (s), 126.24 (s); IR (KBr) 3000 (m), 1550 (vs), 1510 (vs), 1350 (vs), 1320 (s) cm^{-1}; mass spectrum (70 eV), *m/e* (relative intensity) (no molecular ion), 175.05 (38.1), 144.05 (20.2), 129.05 (26.4), 128.05 (63.8), 127.05 (32.7), 117.05 (40.6), 116.05 (53.1), 115.05 (100.0), 105.05 (31.9), 103.05 (31.1), 102.05 (27.5), 91.05 (61.9), 79.05 (33.2), 78.05 (24.8), 77.05 (59.7), 66.05 (52.3), 65.05 (41.7), 54.95 (42.2), 51.05 (63.8).

Anal. Calcd for $C_{10}H_9N_3O_6$: C, 44.95; H, 3.40. Found: C, 45.08; H, 3.59.

Acknowledgment. Financial[13] support of our study by the United States Army Armament, Munitions, and Chemical Command, the Air Force Office of Scientific Research, The Robert A. Welch Foundation (Grant B-963), and the North Texas State University Faculty Research Committee is gratefully acknowledged.

Registry No. 1, 89773-39-7; **3**, 89773-40-0; **4**, 69580-37-6; **5**, 69580-38-7; **6** (isomer 1), 89773-41-1; **6** (isomer 2), 89773-42-2; **7** (isomer 1), 89773-43-3; **7** (isomer 2), 89826-71-1; **8** (isomer 1), 89773-44-4; **8** (isomer 2), 89826-72-2; **9**, 89773-45-5; **10**, 89773-46-6; **11**, 89773-47-7; **12**, 89773-48-8.

(13) Research sponsored by Air Force Office of Scientific Research, Air Force Systems Command, USAF, under Grant No. AFOSR-84-0085.

A Novel Electrogenerated Base. Alkylation of Methyl Arylacetates at the α-Methylene Group[1]

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The formation of basic species from probasic compounds (PB) under the conditions of electroreduction has attracted much attention from mechanistic and synthetic points of view, though only limited types of PBs such as azobenzenes,[2-5] α,β-unsaturated compounds,[6-8] and oxygen[9,10] have been reported in the latter case so far. We have previously reported that a novel electrogenerated base (EGB, **2**) is formed by the cathodic reduction of 2-pyrrolidone (**1**) in DMF (Scheme I), and **2** is effective in promoting the aldol condensation of aldehydes,[11] the Stevens rearrangement,[12] and the condensation of chloroform with aliphatic aldehydes.[13]

In this paper, we report that **2** is also effective as a catalyst to promote α-alkylation of methyl arylacetates leading to the synthesis of some α-alkylarylacetic acids which possess high antiinflammatory and analgesic activities.[14,15]

Although one of the most convenient methods for the synthesis of α-alkylarylacetic acids seems to be the direct alkylation at the α-position of alkyl arylacetates **3**, the exclusive α-monoalkylation of **3** is not easily achievable under the reaction conditions with the usual bases. For example, α-monoethylation of ethyl phenylacetate in the presence of potassium[16] gave the desired product only in 35% yield, and α-methylation of alkyl phenylacetate using sodium amide[17] or sodium hydride as a base gave a mixture of alkyl 2-phenylpropanoate and alkyl 2-methyl-2-phenylpropanoate in 69–90% yield.[18] Because of the

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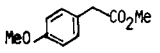
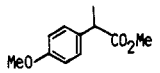
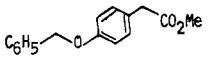
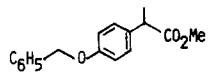
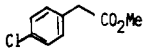
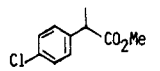
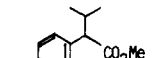
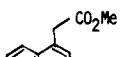
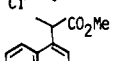
(18) The separation of these products is not always easy.

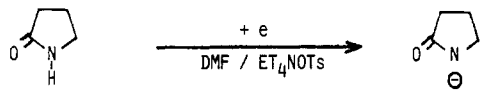
0022-3263/84/1949-2043$01.50/0

Table I. Alkylation of Methyl Acrylacetates

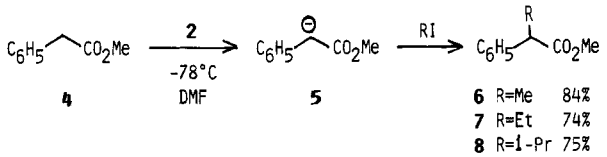
run	RCO_2Me		alkyl iodide	product[a]		yield,[b] %
1		9	MeI		13	82
2		10	MeI		14	88
3		11	MeI		15	86
4	11		*i*-PrI		16	81
5		12	MeI		17	99

[a] All the products gave satisfactory spectroscopic values for the assigned structures. [b] Isolated.

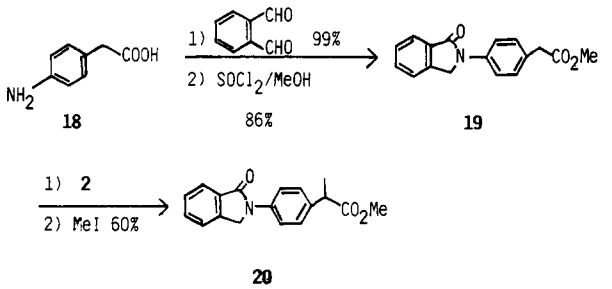
Scheme I



Scheme II



Scheme III



difficulty in α-monoalkylating **3**, most of the recent syntheses of α-alkylarylacetic acids use aryl alkyl ketones[14,19-21] or diethyl arylmalonate[22] as the starting materials, though these methods involve many steps and troublesome operations. On the other hand, the basicity of **2** seems to be adequate as a base to achieve the selective α-monoalkylation of **3**, and it has indeed been confirmed as shown in Scheme II.

Thus, the addition of a solution of methyl phenylacetate (**4**) in DMF to a DMF solution of **2** followed by the trapping of the resulting anionic species **5** with alkyl iodides gave the corresponding methyl α-alkylphenylacetates **6**–**8** in high yield; dialkylation was not observed. Similar anion to **2** may be generated by the reaction of **1** with sodium hydride. The α-methylation of **4** using this anion, however, gave a mixture of **6** (85%) and dialkylated product (4%).

This method using **2** as a base is also applicable to the α-alkylation of a variety of methyl arylacetates **9**–**12**; examples are summarized in Table I. In addition, it is noteworthy that methyl 2-(4-chlorophenyl)-3-methylbutanoate (**16**) (run 4), which is one of the important components of a synthetic pyrethroid,[23] is easily synthesized in high yield. One of the significant applications of this reaction is shown by the transformation of commercially available *p*-aminophenylacetic acid (**18**) to the methyl ester of indoprofen (**20**), which shows high antiinflammatory activity.[25] As shown in Scheme III, the addition of a solution of **19** in DMF to a DMF solution of **2** followed by the trapping of the corresponding anion with methyl iodide gave **20** in 60% yield.

Because of the simplicity of operation and satisfactory yields, the present method is highly useful in the synthesis of a variety of α-alkylarylacetic acid esters.

Experimental Section

Alkylation of Methyl Arylacetates 4 and 9–12. The electrochemical reduction of a solution of 1 (10 mmol) in 20 mL of DMF containing Et_4NOTs (10 mmol) as a supporting electrolyte was carried out in a divided cell equipped with a glass filter diaphragm and platinum anode and cathode. After 2 F/mol of electricity was passed through the cell under the conditions of constant current (0.2 A, cathode potential was more negative than −2.4 V vs. SCE), methyl acrylacetate (2.5 mmol) was added to the catholyte at −78 °C under an atmosphere of nitrogen. To this solution was added 3.5 mmol of alkyl iodide and the reaction mixture was stirred for 15 min at −78 °C. All the products were isolated through silica gel column using a mixed solvent of hexane and ethyl acetate (10:1) and identified by spectroscopic comparison with the authentic samples (**6**,[19] **7**,[26] **8**,[19] **13**,[26] and **15**[26]) and elemental and spectroscopic analyses (**14**, **16**, and **17**).

6: IR (neat) 3050, 3000, 2970, 1730, 1180 cm^{-1}; NMR (CCl_4) δ 1.42 (d, 3 H, J = 7.0 Hz), 3.54 (s, 3 H), 3.60 (q, 1 H, J = 7.0 Hz), 7.23 (s, 5 H).

7: IR (neat) 2970, 1740, 1170 cm^{-1}; NMR (CCl_4) δ 0.88 (t, 3 H, J = 7.5 Hz), 1.50–2.30 (m, 2 H), 3.47 (t, 1 H, J = 7.5 Hz), 3.67 (s, 3 H), 7.33 (s, 5 H).

8: IR (neat) 2970, 1735, 1160 cm^{-1}; NMR (CCl_4) δ 0.69 (d, 3 H, J = 6.0 Hz), 1.00 (d, 3 H, J = 6.0 Hz), 2.10–2.50 (m, 1 H), 3.06

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(d, 1 H, J = 10.5 Hz), 3.59 (s, 3 H), 7.28 (s, 5 H).

13: IR (neat) 2950, 1740, 1240 cm^{-1}; NMR (CCl_4) δ 1.42 (d, 3 H, J = 7.0 Hz), 3.54 (q, 2 H, J = 7.0 Hz), 3.60 (s, 3 H), 3.74 (s, 3 H), 6.76 (d, 2 H, J = 7.0 Hz), 7.14 (d, 2 H, J = 7.0 Hz).

14: IR (neat) 1735, 1240, 790 cm^{-1}; NMR (CCl_4) δ 1.42 (d, 3 H, J = 7.0 Hz), 3.57 (s, 3 H), 3.57 (q, 1 H, J = 7.0 Hz), 5.00 (s, 2 H), 6.73–7.50 (m, 9 H). Anal. Calcd for $C_{17}H_{18}O_3$: C, 75.53; H, 6.71. Found: C, 75.66; H, 6.67.

15: IR (neat) 2990, 1740 cm^{-1}; NMR (CCl_4) δ 1.43 (d, 3 H, J = 8.0 Hz), 3.60 (s, 3 H), 3.61 (q, 1 H, J = 8.0 Hz), 7.23 (s, 4 H).

16: IR (neat) 1950, 1740 cm^{-1}; NMR (CCl_4) δ 0.70 (d, 3 H, J = 6.0 Hz), 1.00 (d, 3 H, J = 6.0 Hz), 2.05–2.65 (m, 1 H), 3.04 (d, 1 H, J = 10.5 Hz), 3.45 (s, 3 H), 7.26 (s, 4 H). Anal. Calcd for $C_{12}H_{15}ClO_2$: C, 63.58; H, 6.69. Found: C, 63.34; H, 6.63.

17: IR (neat) 3050, 3000, 1740 cm^{-1}; NMR (CCl_4) δ 1.60 (d, 3 H, J = 7.0 Hz), 3.60 (s, 3 H), 4.41 (q, 1 H, J = 7.0 Hz), 7.30–8.10 (m, 1 H). Anal. Calcd for $C_{14}H_{14}O_2$: C, 78.48; H, 6.59. Found: C, 78.40; H, 6.68.

Synthesis of 20. Synthesis of **19** was carried out according to the known method.[27] Into a solution of **2** (10 mmol) in 10 mL of DMF was added 2.5 mmol of **19** in 5 mL of DMF at −78 °C. After the reaction mixture was stirred for 15 min at the same temperature, the solution was added dropwise to a solution of methyl iodide (10 mmol) in 10 mL of DMF at −78 °C. The product **20** was isolated by using a silica gel column (CH_2Cl_2).

20: IR (KBr) 1730, 1670, 1610, 740 cm^{-1}; NMR ($CDCl_3$) δ 1.51 (d, 3 H, J = 7.5 Hz), 3.65 (s, 3 H), 3.37 (q, 1 H, J = 7.5 Hz), 4.74 (s, 2 H), 7.30–8.00 (m, 8 H). Anal. Calcd for $C_{18}H_{17}NO_3$: C, 73.20; H, 5.80. Found: C, 73.15; H, 5.73.

Registry No. **1**, 616-45-5; **2**, 45373-29-3; **4**, 101-41-7; **6**, 31508-44-8; **7**, 2294-71-5; **8**, 72615-27-1; **9**, 23786-14-3; **10**, 68641-16-7; **11**, 52449-43-1; **12**, 2876-78-0; **13**, 50415-73-1; **14**, 89618-33-7; **15**, 50415-70-8; **16**, 86618-06-6; **17**, 72221-62-6; MeI, 74-88-4; *i*-PrI, 75-30-9; EtI, 75-03-6.

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New Preparations of Lanthanide Alkoxides and Their Catalytical Activity in Meerwein–Ponndorf–Verley–Oppenauer Reactions

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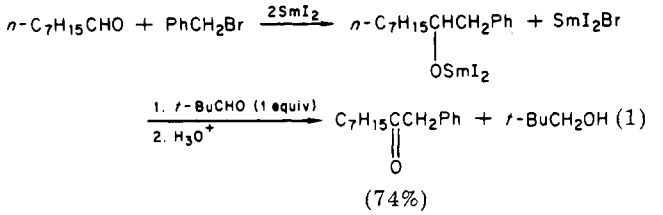
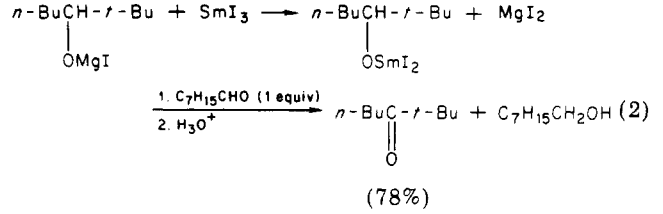
Meerwein–Ponndorf–Verley reductions of aldehydes and ketones[1] and Oppenauer oxidations of alcohols[2] have been known for a long time.[3] These reactions are useful tools for the synthesis of vitamins and steroids[4-6] but otherwise have restricted synthetic applications. Extensive studies of the experimental conditions of the reaction have been performed, dealing with the nature of oxidizing and reducing agents[1,2,7] or of catalysts. The most widely used catalysts are aluminum alkoxides,[3,8] but potassium,[9] sodium,[10] and zirconium[11] alkoxides, alumina,[12] and some transition-metal complexes have also been reported as catalysts.[13]

Oppenauer oxidations can be achieved under mild conditions as compared with many other oxidation methods. Moreover, oxidants (ketones or aldehydes) are cheap. However, a drastic limitation to its use for a wide range of organic substrates is the requirement of great amounts of metal alkoxides. Usually, stoichiometric quantities (or an excess) are used for these reactions. Recently, Seebach reported a system using a catalytic amount of zirconium *tert*-butoxide, but few examples using aluminum alkoxides in catalytic amounts have appeared in the literature.[7,14]

In 1977,[15] we mentioned the occurrence of a competitive Meerwein–Ponndorf reaction to explain the product distribution obtained in the "pseudo-Barbier" alkylation of aldehydes by organic halides using diiodosamarium. Recently, we prepared secondary samarium alkoxides in a similar way using a benzylic halide as the alkylating agent.[16] We directly verified their reducing activity in the Meerwein–Ponndorf reduction of pivalaldehyde. Reaction 1 has been performed in stoichiometric conditions, in THF, at room temperature.



When samarium triiodide is added to a magnesium alkoxide (reaction 2), the mixture reduces octanal, whereas no reaction occurs with the magnesium alkoxide alone. So



we assume an exchange reaction and formation of samarium alkoxide. Although this reaction needs a stoichiometric amount of samarium triiodide, it is interesting to point out that it can be considered as a quite general preparation of dissymmetrical ketones. Encouraged by these preliminary results, we explored the potential of samarium alkoxides in oxidoreduction reactions. We now report *new methods of synthesis* of samarium alkoxides and their *catalytic activity* in Meerwein–Ponndorf–Verley reductions and Oppenauer oxidations.

Catalysts

Several routes have been described for the synthesis of alkoxy lanthanide compounds:[8,17] (i) reaction of an alcohol

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