It is noteworthy that photolysis of *tert*-butyl disulfide under similar conditions also gave a weak spectrum of the perthiyl radical, which was substantially enhanced when benzophenone (ca. 0.05 M) was present in the mixture. Clearly, the matrix is capable of preserving the perthiyl radicals even though they are formed with a low quantum yield.

Finally, thermolysis of $t\text{-BuS}_4\text{Bu-}t$ [250 °C (10^{-3} torr)] in an apparatus consisting of a quartz furnace, an electron monochromator, and quadrupole mass spectrometer[22] gave a good yield of $t\text{-BuSS}\cdot$. The ionization potential of the radical was found to be 8.25 eV.

Acknowledgment. We thank Dr. J. C. Scaiano for the use of the laser flash photolysis equipment.

Registry No. $t\text{-BuS}_4\text{Bu-}t$, 5943-35-1; t-BuSSCl, 6009-05-8; t-BuSSBu-t, 110-06-9; $t\text{-BuSS}\cdot$, 71256-14-9.

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[22] Lossing, F. P.; Traeger, J. C. *Int. J. Mass. Spectrom. Ion Phys.* **1976**, *19*, 9.

Electrooxidative Transformation of Aldehydes to Esters Using Mediators[1]

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Oxidative transformation of aldehydes **1** to esters **2** under mild conditions is often requisite in organic synthesis, and hence much effort has been devoted to find new methods or new reagents effective for this type of oxidation.[2] In our continuing study on anodic oxidation using mediators,[3] we have found that this electrooxidation is a convenient tool for the transformation of **1** to **2**.

$$\underset{\mathbf{1}}{\text{RCHO}} \xrightarrow[\text{R'OH}]{-2e} \underset{\mathbf{2}}{\text{RCO}_2\text{R'}} \tag{1}$$

The electrooxidation of **1** to the corresponding methyl ester is remarkably simple as briefly described below. Namely, methyl esters **5a–10a** were easily prepared by passing a constant current at room temperature through a solution of aldehydes **5–10** in methanol containing KI or KBr.[4] The results are shown in Table I.

On the other hand, butyl esters **7b**, **9b**, and **11b–15b** were successfully prepared by carrying out the electrooxidation of aldehydes **7**, **9**, and **11–15** under the conditions of a two-phase system consisting of an aqueous layer containing KI or KBr and an organic one containing **1** and butanol, since the low electric conductivity of butanol did not allow its use as a sole solvent. Yields of butyl esters are summarized in Table II. An ester of *n*-hexyl alcohol was also prepared by a similar method (run 10 in Table II).

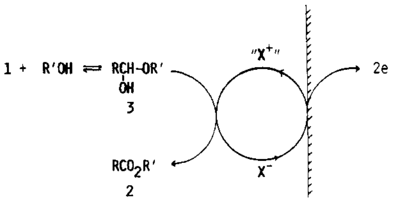
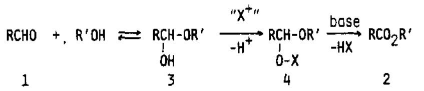


Figure 1. Electrooxidative transformation of **1** to **2**.

The reaction may proceed through the pathway described in eq 2 and 3.[3a,e] A positively charged active species "X^+" generated by the anodic oxidation of a halide ion X^- attacks on aldehyde hemiacetals **3** to yield **4**.[5] Since a base may be formed as the cathodic reaction,[8] it abstracts hydrogen halide from **4** to afford **2** while regenerating X^-. Accordingly, the overall process can schematically be represented by Figure 1 in which the conversion of **1** to **2** proceeds with a catalytic amount of halide ion. In fact, even 0.1 mol equiv of KI was enough to complete the transformation of **1** to **2** (run 5 in Table I).

$$X^- \xrightarrow{-2e} \text{"}X^+\text{"} \tag{2}$$



$$\underset{\mathbf{1}}{\text{RCHO}} + \text{R'OH} \rightleftharpoons \underset{\mathbf{3}}{\underset{\text{OH}}{\text{RCH-OR'}}} \xrightarrow[-\text{H}^+]{\text{"X}^+\text{"}} \underset{\mathbf{4}}{\underset{\text{O-X}}{\text{RCH-OR'}}} \xrightarrow[-\text{HX}]{\text{base}} \underset{\mathbf{2}}{\text{RCO}_2\text{R'}} \tag{3}$$

This electrochemical method for the oxidation of **1** to **2** possesses high potentiality in organic synthesis, since (1) no special oxidizing reagent is required, (2) the procedures of the electrooxidation and subsequent workup are simple and easy, and (3) the reaction conditions are so mild that aldehydes having a carbon–carbon double bond (runs 8 and 9 in Table II) or an epoxy group (run 11 in Table II) can selectively be oxidized to the corresponding esters with these functional groups intact.

Experimental Section

IR spectra were taken with a Hitachi 215 spectrometer. ^{1}H NMR spectra were recorded on a Varian Associates EM-390 spectrometer using tetramethylsilane as an internal standard. Elemental analyses were determined by the Center for Instrumental Analysis of Kyoto University. Boiling points were uncorrected. Electrochemical oxidation was carried out using DC Power Supply (GP0502) of Takasago Seisakusho, Ltd.

Materials. Aldehydes **5–14**, **5a–8a**, and **12b** and potassium halides were commercially available. Epoxy aldehyde **15** was prepared by the oxidation of **14** with *m*-chloroperbenzoic acid and isolated by column chromatography (silica gel, AcOEt, hexane) in 68% yield: IR (film) 2950, 2930, 2730, 1715, 1380, 1120 cm^{-1}; NMR (CCl_4) δ 1.0 (d, J = 4 Hz, 3 H), 1.23 (s, 3 H), 1.26 (s, 3 H),

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[1] Electroorganic Chemistry. 94. A part of this work has been preliminarily presented at the Autumn Meeting of the Electrochemical Society of Japan, Tsukuba, November 30, 1983; p 140.

[2] For examples: (a) Corey, E. J.; Gilman, N. W.; Ganem, B. E. *J. Am. Chem. Soc.* **1968**, *90*, 5616. (b) Sakuragi, H.; Tokumaru, K. *Chem. Lett.* **1974**, 475. (c) Ogawa, T.; Matsui, M. *J. Am. Chem. Soc.* **1976**, 1629. (d) Sundararaman, P.; Walker, E. C.; Djerassi, C. *Tetrahedron Lett.* **1978**, 1627. (e) Chiba, T.; Okimoto, M.; Nagai, H.; Takata, Y. *Bull. Chem. Soc. Jpn.* **1982**, *55*, 335. (f) Wilson, S. R.; Tofigh, S.; Misra, R. N. *J. Org. Chem.* **1982**, *47*, 1360.

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[4] The electrooxidation of **1** in methanol containing Et_4NOTs did not give methyl esters but rather gave the corresponding acetals in good yields.

[5] It has been known that aldehyde is generally equilibrated with its hydrated form in an aqueous solution,[6] and also the latter is the actual substrate in the chromic acid oxidation of aldehyde in an aqueous solution.[7]

[6] Zuman, P. *J. Electroanal. Chem. Interfacial Electrochem.* **1977**, *75*, 523.

[7] Roček, J.; Ng, C.-S. *J. Org. Chem.* **1973**, *38*, 3348.

[8] (a) Shono, T. "Electroorganic Chemistry as a New Tool in Organic Synthesis"; Springer-Verlag: Berlin and Heidelberg, 1984. (b) Shono, T.; Kashimura, S.; Ishizaki, K. *Electrochim. Acta* **1984**, *29*, 603. (c) Shono, T.; Kashimura, S.; Ishizaki, K.; Ishige, O. *Chem. Lett.* **1983**, 1311. (d) Shono, T.; Kashimura, S.; Nogusa, H. *J. Org. Chem.* **1984**, *49*, 2043.

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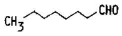
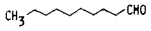
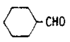
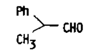
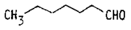
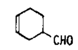
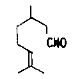
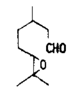
Table I. Electrooxidative Transformation of Aldehydes (RCHO) to Methyl Esters (RCO_2CH_3)[a,b]

run	RCHO[g]	mediator[h] (KX)	electricity passed, F/mol	yield of RCO_2CH_3 compd	yield of RCO_2CH_3 %
1	 5	KI	5	**5a**	72[c]
2		KBr	4.9	**5a**	78 (88)[d]
3	 6	KBr	4.9	**6a**	85
4	 7	KI	5	**7a**	~100[d]
5		KI[e]	5	**7a**	~100[d]
6		KBr	5	**7a**	99[d]
7	 8	KBr	5.1	**8a**	71
8	$PhCH_2CH_2CHO$ 9	KBr	3.6	**9a**	85
9	 10	KBr	5.5	**10a**	69[f]

[a] Solvent, CH_3OH (30 mL); anode, platinum plate (20 mm × 20 mm); cathode, carbon rod (8 mm i.d.). [b] Diaphragm was not used. [c] A byproduct, 2-hydroxyoctanal dimethyl acetal (7%) was also formed. [d] Yields were determined by GLC method. [e] The amount of KI was 0.4 mmol. [f] A byproduct, 2-hydroxy-2-phenylpropionaldehyde dimethyl acetal (10%) was also formed. [g] 4 mmol. [h] 2 mmol.

Table II. Electrooxidative Transformation of Aldehydes (RCHO) to *n*-Butyl Esters (RCO_2-*n*-C_4H_9)[a]

run	RCHO[g]	mediator[h] (KX)	electricity passed, F/mol	isolated yield[b] (RCO_2-*n*-C_4H_9)	isolated yield[b] %[b]
1	 11	KI	3.9	**11b**	56
2		KI	12.0	**11b**	96[c]
3		KI	3.2	**7b**	72 (144)[d]
4		KI[e]	3.2	**7b**	57 (228)[d]
5		KBr	3.2	**7b**	69[c]
6		KI	4.6	**9b**	74
7	PhCHO 12	KI	3.7	**12b**	61
8	 13	KI	5.6	**13b**	75
9	 14	KI	5.6	**14b**	86
10		KI	3.2	**14c**	65[f]
11	 15	KI	6.5	**15b**	38

[a] *n*-Butyl alcohol (60 mmol) was used. [b] Yields were calculated on the basis of aldehyde. [c] Yields were determined on GLC method. [d] Yields in parenthesis were calculated on the basis of used KI. [e] The amount of KI was 5 mmol. [f] *n*-Hexyl alcohol (60 mmol) was used. The product was *n*-hexyl ester. [g] 20 mmol. [h] 10 mmol.

1.36–1.65 (m, 4 H), 1.90–2.43 (m, 3 H), 2.43–2.66 (m, 1 H), 9.78 (t, J = 1 Hz, 1 H). Anal. Calcd for $C_{10}H_{18}O_2$: C, 70.54; H, 10.66. Found: C, 70.15; H, 10.32.

Electrooxidative Transformation of Aldehydes to Methyl Esters. General procedure for the anodic oxidation of aldehydes to methyl esters is exemplified by the oxidation of octyl aldehyde (**5**) using KBr as a supporting electrolyte (run 2 in Table I). In a cell equipped with a platinum plate anode (20 mm × 20 mm) and a carbon rod cathode (8 mm i.d.) was put a solution of **5** (4 mmol) in methanol (30 mL) containing KBr (2 mmol). After a constant current of 0.3 A (terminal voltage, ~15 V) was passed through the solution for 1.8 h (4.9 F/mol of electricity) with external cooling with an ice–water bath, the solvent was evaporated under reduced pressure and water was added to the residue. The resulting solution was extracted with CH_2Cl_2 (3 × 50 mL), and the extracts were dried with $MgSO_4$. Evaporation of CH_2Cl_2 afforded crude methyl octanoate (**5a**). The yield of **5a** determined by GLC method was 88%, and purification was achieved by Kugelrohr distillation (78%). Esters **6a**–**10a** were also isolated by Kugelrohr distillation, and identification of the structures of **5a**–**10a** was carried out by comparison of their spectroscopic data with those of authentic samples.[9]

Synthesis of Butyl Esters. To a cell (32 mm i.d. × 70 mm) equipped with a platinum anode (20 mm × 20 mm), a carbon rod cathode (8 mm i.d.), and a dropping funnel were added butanol (4.44 g, 60 mmol) and a solution of potassium iodide (1.66 g, 10 mmol) in water (10 mL). The cell was cooled with a water jacket while a constant current (0.5 A) was passed through the two-phase solution with vigorous stirring. A solution of aldehyde (20 mmol) in cyclohexane (5 mL) was added dropwise to the two-phase solution at a rate so that 2 F/mol of electricity was passed during the addition of aldehyde (2.16 h). The terminal voltage was 5–10 V. After 3.2–5.6 F/mol of electricity was passed, the organic layer was separated, and the aqueous layer was extracted with three portions of ether. The combined organic and ethereal layers were washed with aqueous sodium thiosulfate. The products isolated by distillation were identified by spectroscopic and elemental analysis or by comparison with authentic samples. Samples for elemental analysis were collected by VPC. Esters bearing a carbon–carbon double bond were isolated by column chromatography. Yields and the electricity passed are shown in Table I.

***n*-Butyl *n*-heptanoate (11b)**: bp 94–97 °C (12–13 mm); IR (film) 2950, 1720, 1170 cm^{-1}; NMR (CCl_4) δ 0.75–1.10 (m, 6 H), 1.10–1.85 (m, 12 H), 2.23 (t, J = 4.5 Hz, 2 H), 4.00 (t, J = 4 Hz, 2 H). Anal. Calcd for $C_{11}H_{22}O_2$: C, 70.92; H, 11.90. Found: C, 71.08; H, 11.88.

***n*-Butyl cyclohexanecarboxylate (7b)**: bp 115–125 °C (42 mm); IR (film) 2920, 1720, 1165 cm^{-1}; NMR (CCl_4) δ 0.96 (t, J = 4.5 Hz, 3 H), 1.00–2.40 (m, 15 H), 4.00 (t, J = 4.5 Hz, 2 H). Anal. Calcd for $C_{11}H_{20}O_2$: C, 71.69; H, 10.94. Found: C, 71.55; H, 11.04.

***n*-Butyl β-phenylpropionate (9b)**: bp 135–138 °C (22–23 mm); IR (film) 3030, 2960, 1730, 1165, 750, 700 cm^{-1}; NMR (CCl_4) δ 0.91 (t, J = 4.5 Hz, 2 H), 1.10–1.80 (m, 4 H), 2.53 (t, J = 4.5 Hz, 2 H), 2.90 (t, J = 4.5 Hz, 2 H), 4.00 (t, J = 4 Hz, 2 H), 6.90–7.50 (m, 5 H). Anal. Calcd for $C_{13}H_{18}O_2$: C, 75.69; H, 8.80. Found: C, 75.82; H, 9.03.

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(9) Data of **9a** and **10a**; Brink, M.; Larsson, E. *Org. Magn. Reson.* **1973**, *5*, 327.

***n*-Butyl 4-cyclohexenecarboxylate (13b)** was isolated by column chromatography (silica gel, 1:10 AcOEt–hexane): IR (film) 2930, 1710, 1165 cm^{-1}; NMR (CCl_4) δ 0.96 (t, J = 4.5 Hz, 3 H), 1.15–2.70 (m, 1 H), 4.03 (t, J = 4 Hz, 2 H), 5.63 (s, 2 H). Anal. Calcd for $C_{11}H_{18}O_2$: C, 72.49; H, 9.96. Found: C, 72.18; H, 9.92.

***n*-Butyl 3,7-dimethyl-6-octenoate (14b)** was isolated by column chromatography (silica gel, 1:10 AcOEt–hexane): IR (film) 2960, 2930, 1735, 1190, 1155 cm^{-1}; NMR (CCl_4) δ 0.97 (br t, J = 4 Hz, 6 H), 1.10–1.40 (m, 1 H), 1.60 (s, 3 H), 1.67 (s, 3 H), 4.00 (t, J = 4 Hz, 2 H), 5.06 (t, J = 4.5 Hz, 1 H). Anal. Calcd for $C_{14}H_{26}O_2$: C, 74.28; H, 11.58. Found: C, 74.14; H, 11.71.

***n*-Hexyl 3,7-dimethyl-6-octenoate (14c)** was isolated by column chromatography (silica gel, 1:10 AcOEt–hexane): IR (film) 2920, 1730, 1190, 1160 cm^{-1}; NMR (CCl_4) δ 0.92 (br t, J = 4.5 Hz, 6 H), 1.10–2.30 (m, 15 H), 1.60 (s, 3 H), 1.67 (s, 3 H), 4.00 (t, J = 4 Hz, 2 H), 5.06 (t, J = 4.5 Hz, 1 H). Anal. Calcd for $C_{16}H_{30}O_2$: C, 75.53; H, 11.89. Found: C, 75.19; H, 11.73.

***n*-Butyl 3,7-dimethyl-6,7-epoxyoctanoate (15b)** was isolated by column chromatography (silica gel, 1:3 AcOEt–hexane): IR (film) 2955, 2870, 1730, 1165 cm^{-1}; NMR (CCl_4) δ 0.83–1.10 (m, 6 H), 1.23 (s, 3 H), 1.26 (s, 3 H), 1.30–1.73 (m, 8 H), 1.80–2.33 (m, 3 H), 2.40–2.63 (m, 1 H), 4.03 (t, J = 4 Hz, 2 H). Anal. Calcd for $C_{14}H_{26}O_3$: C, 69.38; H, 10.81. Found: C, 68.95; H, 11.09.

Acknowledgment. T.S. thanks the Ministry of Education, Science, and Culture, Japan, for a Grant-in-Aid for Special Project Research (1) (57118003, 58110003, and 59104003).

Registry No. **5**, 124-13-0; **5a**, 111-11-5; **6**, 112-31-2; **6a**, 110-42-9; **7**, 2043-61-0; **7a**, 4630-82-4; **7b**, 6553-81-7; **8**, 6654-36-0; **8a**, 627-93-0; **9**, 104-53-0; **9a**, 103-25-3; **9b**, 20627-49-0; **10**, 93-53-8; **10a**, 31508-44-8; **11**, 111-71-7; **11b**, 5454-28-4; **12**, 100-52-7; **12b**, 136-60-7; **13**, 100-50-5; **13b**, 37981-14-9; **14**, 106-23-0; **14b**, 98652-74-5; **14c**, 98652-75-6; **15**, 25825-48-3; **15b**, 98652-76-7; KI, 7681-11-0; KBr, 7758-02-3.

Group 14 Metal Assisted Carbon–Sulfur Bond Formation[1]

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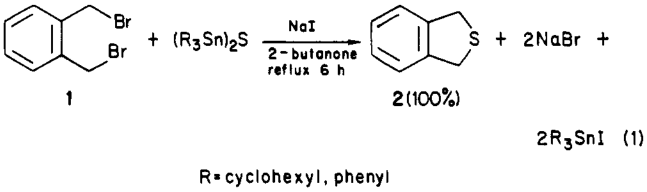
Protein-bound macrocyclic thiolactones, owing to their diverse and important biochemical properties,[2a] are a special class of the macrolide family of compounds that is currently under intensive investigation.[2] In contrast to the many new macrolide-forming methods that have recently been developed,[1] very few procedures for the preparation of macrocyclic thiolactones are reported in the chemical literature.[3] In conjunction with our ongoing studies directed toward the synthesis of synthetic and naturally occurring sulfur-bridged cyclic systems,[1,4] we require efficient methodology for the facile construction of large- and medium-sized thiacycloalkanes. We herein describe a group 14[11] metal assisted method for carbon–sulfur bond formation that is applicable to the synthesis of cyclic organosulfides and macrocyclic thiolactones.

Although carbon–sulfur bond formation enjoys many well-established synthetic avenues,[5] negotiating sulfur into a medium-sized cyclic hydrocarbon skeleton, on the other hand, is limited to but a few useful routes.[6] To help reduce unwanted polymerization, these methods rely on the use of techniques of high dilution and special functional group activation. Since the mercaptide ion is one of the more powerful nucleophiles known, an S_N2-type displacement reaction is usually invoked to attach sulfur onto a carbon atom.[5,6]

In previous work relating to thiocarbonyl synthesis,[4b] we demonstrated the utility of group 14 metal sulfides as effective transporters of the sulfur atom. These readily available reagents can also be used, in a simple way, to efficiently form carbon–sulfur bonds. Thus, when benzylic bromides are treated with alkyl-/aryltin sulfides in refluxing 2-butanone with stoichiometric amounts of sodium iodide (eq 1), a quantitative yield of the benzylic sulfide is readily obtained. Unfortunately however, alkyl bromides, even under considerably more vigorous (refluxing mesitylene) reaction conditions, are completely resistant to this exchange and sulfuration of these substrates requires an alternate approach.



1

2 (100%)

(1)

We have found that addition of 2 equiv of MeLi to an anhydrous THF solution of hexamethyldisilathiane (HMDST)[7] at room temperature, followed by refluxing for 2 h, produces a clear solution of "Li_2S" and Me_4Si (eq 2).

$$Me_3SiSSiMe_3 + 2MeLi \xrightarrow[25\ ^\circ C]{THF} \text{“}Li_2S\text{”} + 2Me_4Si \quad (2)$$

If left to stand for a few hours, the reaction mixture becomes turbid and after 24 h a high yield of Li_2S is precipitated. The chemical and physical properties of this precipitated form of Li_2S are entirely consistent with those recorded in the literature for this compound.[5c] In sharp contrast however, and in accordance to similar observations by Gladysz,[5c] the soluble form of the Li_2S thus produced, was found to be considerably more reactive than the precipitated form with the extent of chemical reactivity dependent on the "age" of its formation. For example, addition of dibromide 1 to a freshly prepared solution of Li_2S with continued refluxing for an additional 6 h affords 1,3-dihydroisothianaphthene (2) in greater than 95% *yield*; whereas the precipitated form of Li_2S results (after 24 h

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(1) Reagents for Organic Synthesis. 4. For the previous paper in this series, see: Steliou, K.; Poupart, M.-A. *J. Am. Chem. Soc.* **1983**, *105*, 7130.

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(3) (a) Malherbe, R.; Rist, G.; Belluš, D. *J. Org. Chem.* **1983**, *48*, 860. (b) Mahajan, J. R.; de Araújo, H. C. *Synthesis* **1980**, 64. (c) Shanzer, A.; Libman, J. *Synthesis* **1984**, 140.

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(7) (a) Hexamethyldisilathiane[7b] is used instead of the analogous tin reagents because the latter react with organolithium reagents to form stable "ate" complexes (Still, W. C. *J. Am. Chem. Soc.* **1978**, *100*, 1481. Meyor, N.; Seebach, D. *Chem. Ber.* **1980**, *113*, 1290) that limit their utility in this reaction. (b) Harpp, D. N.; Steliou, K. *Synthesis* **1978**, 721. This reagent is commercially available from Petrarch Systems Inc. Levittown, PA 19059.