ELSEVIER

Journal of Photochemistry and Photobiology A: Chemistry 150 (2002) 77–84

Journal of Photochemistry and Photobiology A:Chemistry

www.elsevier.com/locate/jphotochem

# Comparison of photomacrocyclization reactions of trimethylsilyl- and tributylstannyl-terminated phthalimido- and maleimido-polyethers

Ung Chan Yoon [a,*], Ying Xue Jin [a], Sun Wha Oh [a], Dae Won Cho [a], Ki Hyun Park [a], Patrick S. Mariano [b]

[a] *Department of Chemistry and Chemistry, Institute for Functional Materials, Pusan National University, Pusan 609-735, South Korea*
[b] *Department of Chemistry, University of New Mexico, Albuquerque, NM 87131, USA*

Received 13 August 2001; received in revised form 17 December 2001; accepted 28 January 2002

**Abstract**

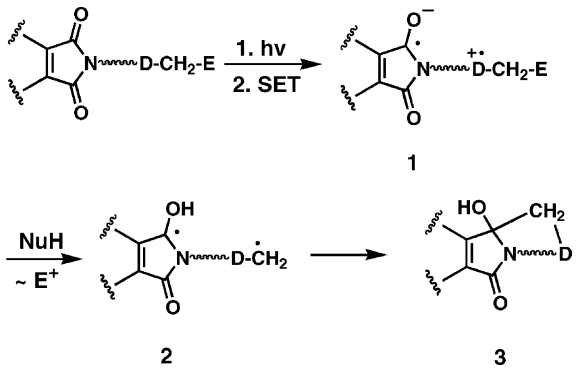
SET-promoted photomacrocyclization reactions of trimethylsilyl- and tributylstannyl-terminated phthalimido- and maleimido-polyethers were investigated. The results indicate that the excited state cyclization processes, which take place via sequential SET-destannation pathways, produce macrocyclic polyethers more efficiently than those involving sequential SET-desilylation routes do. In addition, differences in product distributions obtained from photoreactions of trimethylsilyl- and tributylstannyl-terminated maleimido-polylethers suggest that more than one mechanistic pathway is followed in excited-state reactions of the tin-containing substrates. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Single electron transfer; Photomacrocyclizations; Tributylstannyl-terminated; Trimethylsilyl-terminated; Phthalimido-polyethers; Maleimido-polyethers

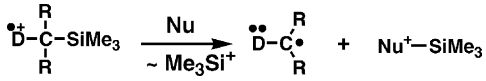
## 1. Introduction

The combined results of earlier investigations in the area of conjugated imide photochemistry [1] have demonstrated that SET(single electron transfer)-promoted photocyclization reactions of these substrates serve as highly efficient methods to generate a variety of N-heterocyclic products. Numerous examples documenting this conclusion are found in the photochemistry of phthalimides and maleimides which contain N-linked ether, thioether, silane, arene and carboxylate moieties. In these excited-state processes, SET pathways (Scheme 1) proceed more rapidly than those initiated by H-atom transfer [2]. The intermediate zwitterionic biradicals **1** formed by intramolecular SET, typically undergo secondary α-fragmentation at the cationic center to produce biradical precursors **2** of the cyclic products **3**. The most widely studied cation radical α-fragmentation processes that serve as the driving force for these photocyclization reactions include base-promoted deprotonation and retro-aldol cleavage, unimolecular decarboxylation, and silophile-induced desilylation.

Product distribution and laser flash photolysis investigations carried out in our laboratories [3,4] have shown that cation radical desilylation (Scheme 2) is a fast process when




Scheme 1.

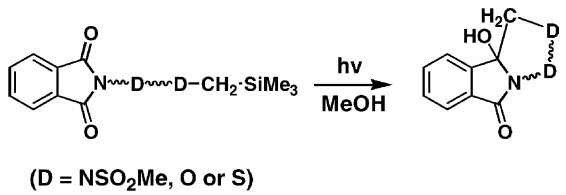



Scheme 2.

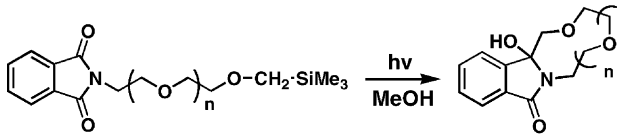
it is promoted by silophiles such as water and methanol. Consequently, SET-initiated photocyclization reactions of substrates possessing α-silyl-amine, -ether and -thioether electron donor sites proceed efficiently and with high levels of regiochemical control [5–8]. Recently, we demonstrated how this chemistry can be employed in the design of phthalimide excited-state processes that form macrocyclic poly-sulfonamides, -ethers and -thioethers (Scheme 3) [9].

* Corresponding author.

1010-6030/02/$ – see front matter 
PII: S1010-6030(02)00032-1




Scheme 3.

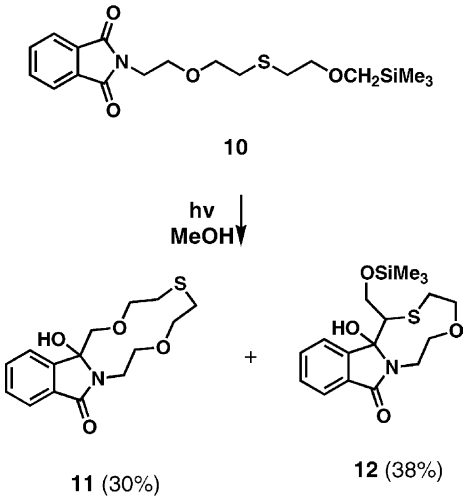



**4** (n=0, 98%)
**5** (n=1, 99%)
**6** (n=2, 60%)
**7** (n=3, 64%)
**8** (n=4, 50%)
**9** (n=5, 53%)

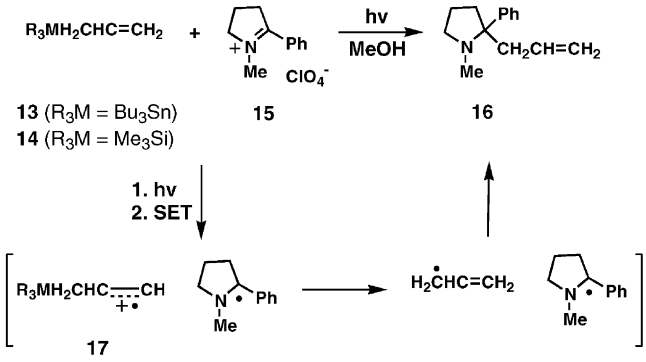
Scheme 4.

However, in the course of these studies we noted some limitations of processes driven by cation radical α-desilylation. For example, the yields of photocyclization reactions of the silicon-terminated phthalimido-polyethers **4**–**9** (Scheme 4) decrease rather dramatically as the length of the polyether tether increases [6]. In addition, photocyclizations of silicon-terminated mixed poly-ether–thioether substrates are complicated by the competitive operation of sequential SET-deprotonation and SET-desilylation pathways (e.g., transformation of ***10*** → ***11*** + ***12***, Scheme 5) [9].

Although less well studied, SET-promoted ground and excited-state reactions driven by α-destannylation of cation radical intermediates are known to proceed with high chemical and quantum efficiencies. An example of this is found in the photoaddition of allylstannane **13** to the pyrrolinium **15** in MeOH, which furnishes adduct **16** (Scheme 6) [10]. The quantum efficiency of this process is ca. 10-fold greater than



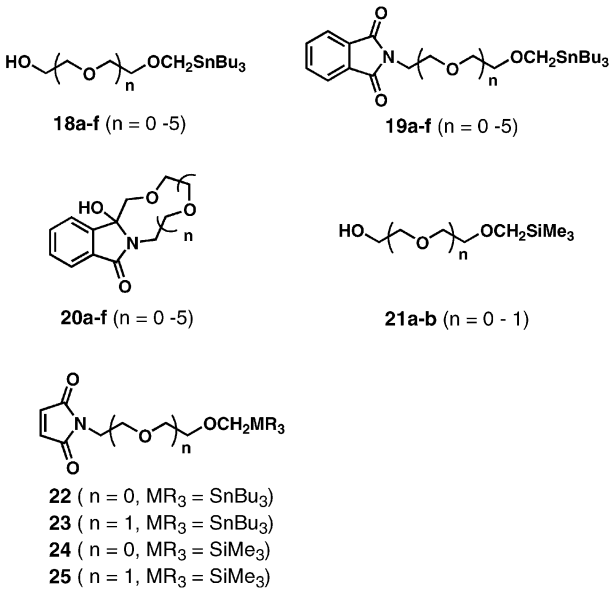

Scheme 5.




Scheme 6.

that for photoaddition of the analogous allylsilane **14**–**15**. This result suggests that the rate of fragmentation of the cation radical **17** ($R_3M$ = $Bu_3Sn$) by transfer of a tributylstannyl group to MeOH is ca. one order of magnitude greater than MeOH-induced cleavage of the silicon substituted cation radical **17** ($R_3M$ = $Me_3Si$). In addition, anodic oxidation of α-stannyl ethers efficiently generates oxonium ions via a sequence involving SET followed by destannylation of cation radical intermediates [11,12].

These observations lead to the proposal that replacement of the $Me_3Si$-group in the phthalimido polyethers **4**–**9** and related maleimide derivatives by a $Bu_3Sn$ group would lead to an enhancement of the efficiencies of their SET-promoted photomacrocylization reactions. To test this suggestion, we have prepared and subjected to photochemical investigation a series of tributylstannyl terminated phthalimido-polyethers **19a**–**f**, tributylstannyl- and trimethylsilyl-terminated maleimido-polyethers **22**–**25**. Comparisons of the photochemical properties of these compounds show that the $Bu_3Sn$-containing substrates undergo more efficient and chemically selective SET-promoted photomacrocyclization reactions than do their $Me_3Si$-terminated analogs (**4**–**9** and **24**–**25**).



## 2. Experimental

### 2.1. General procedures

All NMR spectra were recorded by using $CDCl_3$ solutions. All compounds were isolated as oils unless otherwise specified.

### 2.2. Tri-n-butylstannylmethyl-substituted polyethyleneglycol-alcohols *18a–f*

To independent THF (100 ml) solutions of the mono- and polyethyleneglycol-diols $HOCH_2CH_2OH$ (12.0 ml), $HOCH_2CH_2OCH_2CH_2OH$ (20.9 ml), $HOCH_2(CH_2OCH_2CH_2)_2OH$ (29.5 ml), $HOCH_2(CH_2OCH_2CH_2)_3OH$ (28.0 ml), $HOCH_2(CH_2OCH_2CH_2)_4OH$ (46.6 ml), and $HOCH_2(CH_2OCH_2CH_2)_5OH$ (55 ml) was added Na metal (1.00 g, 43.0 g atm) in portions over a 4 h period. To each solution was then added tributylstannylmethyl iodide (18.5 g, 4.30 mmol) dropwise and the resulting mixtures were stirred at 80 °C for 2 days, cooled to 25 °C and extracted with *n*-pentane. The pentane solutions were washed with water, dried ($Na_2SO_4$) and concentrated in vacuo to afford residues which were subjected to column chromatography (silica gel, 1:5 EtOAc-hex) to yield 10.7 g (68%) of **18a**, 11.4 g (65%) of **18b**, 9.8 g (50%) of **18c**, 8.6 g (40%) of **18d**, 9.3 g (40%) of **18e** and 8.3 g (33%) of **18f**.

**18a**: $^1$H NMR 0.84–0.93 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.24–1.42 (m, 6H, $CH_2CH_3$), 1.46–1.58 (m, 6H, $SnCH_2CH_2$), 3.43 (t, 2H, $J$ = 4.6 Hz, $HOCH_2CH_2$), 3.66–3.70 (m, 3H, $HOCH_2$), 3.76 (s, 2H, $CH_2SnBu_3$); $^{13}$C NMR 8.9 ($CH_3$), 13.6 ($CH_2CH_3$), 27.2 ($CH_2CH_2CH_2CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.6 ($OCH_2SnBu_3$), 62.4 ($HOCH_2CH_2$), 76.3 ($HOCH_2$); MS(EI), *m/z* (rel. intensity) 365 (1), 309 (100), 292 (22), 195 (48), 175 (65); HRMS(EI), *m/z* 309.0876 (M–$C_4H_9$, $C_{11}H_{25}O_2{}^{120}$Sn requires 309.0876).

**18b**: $^1$H NMR 0.83–0.92 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.24–1.37 (m, 6H, $CH_2CH_3$), 1.40–1.56 (m, 6H, $SnCH_2CH_2$), 3.46–3.73 (m, 9H, $HO(CH_2CH_2O)_2$), 3.75 (s, 2H, $CH_2SnBu_3$); $^{13}$C NMR 9.0 ($CH_3$), 13.6 ($CH_2CH_3$), 27.2 ($CH_2CH_2CH_2CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.8 ($OCH_2SnBu_3$), 62.5 ($CH_2OCH_2CH_2$), 70.3 ($CH_2OCH_2SnBu_3$), 72.4 ($HOCH_2CH_2O$), 74.8 ($HOCH_2$); MS(FAB), *m/z* (rel. intensity) 409 (2), 353 (100), 309 (4), 235 (28), 176 (20); HRMS(FAB), *m/z* 353.1163 (M–$C_4H_9$, $C_{13}H_{29}O_3{}^{120}$Sn requires 353.1190).

**18c**: $^1$H NMR 0.82–0.92 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.20–1.36 (m, 6H, $CH_2CH_3$), 1.39–1.52 (m, 6H, $SnCH_2CH_2$), 3.44–3.56 (m, 2H, $CH_2OCH_2SnBu_3$), 3.58–3.72 (m, 1$^1$H, $HO(CH_2CH_2O)_2CH_2$), 3.79 (s, 2H, $CH_2SnBu_3$); $^{13}$C NMR 9.0 ($CH_3$), 13.6 ($CH_2CH_3$), 27.2 ($CH_2CH_2CH_2CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.7 ($CH_2SnBu_3$), 62.5, 70.4, 70.5, 70.6, 72.5 and 74.7); MS(EI), *m/z* (rel. intensity) 397 (M–$C_4H_9$, 98), 291 (22), 235 (29); HRMS(EI), *m/z* 397.1378 (M–$C_4H_9$, $C_{15}H_{33}O_4{}^{120}$Sn requires 397.1401).

**18d**: $^1$H NMR 0.82–0.92 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.26–1.36 (m, 6H, $CH_2CH_3$), 1.39–1.52 (m, 6H, $SnCH_2CH_2CH_2CH_3$), 3.58–3.72 (m, 15H, $HO(CH_2CH_2O)_3CH_2$), 3.76 (s, 2H, $OCH_2SnBu_3$); $^{13}$C NMR 9.0 ($CH_3$), 13.6 ($CH_2CH_3$), 27.2 ($CH_2CH_2CH_2CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.6 ($CH_2SnBu_3$), 62.4 ($CH_2OCH_2SnBu_3$), 70.2, 70.3, 70.5, 72.5 and 74.6; MS(EI), *m/z* (rel. intensity) 441 (100), 439 (88), 383 (12), 235 (36), 179 (48); HRMS(EI), *m/z* 441.1673 (M–$C_4H_9$, $C_{17}H_{37}O_5{}^{120}$Sn requires 441.1663).

**18e**: $^1$H NMR 0.83–0.90 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.18–1.33 (m, 6H, $CH_2CH_3$), 1.41–1.52 (m, 6H, $SnCH_2CH_2$), 3.44–3.71 (m, 2$^1$H, $HOCH_2(CH_2OCH_2)_4CH_2$), 3.75 (s, 2H, $OCH_2SnBu_3$); $^{13}$C NMR 9.0 ($CH_3$), 13.7 ($CH_2CH_3$), 27.3 ($CH_2CH_2CH_2CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.7 ($CH_2SnBu_3$), 62.5 ($CH_2OCH_2SnBu_3$), 70.2, 70.3, 70.4, 70.5, 70.6, 70.7 and 70.9; 72.5 ($HOCH_2CH_2$), 74.6 ($HOCH_2$); MS(EI), *m/z* (rel. intensity) 485 (M–$C_4H_9$, 100), 291 (21), 235 (33); HRMS(EI), *m/z* 485.1949 (M–$C_4H_9$, $C_{19}H_{41}O_6{}^{120}$Sn requires 485.1925).

**18f**: $^1$H NMR 0.83–0.91 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.21–1.36 (m, 6H, $CH_2CH_3$), 1.40–1.52 (m, 6H, $SnCH_2CH_2$), 3.57–3.71 (m, 25H, $HOCH_2(CH_2OCH_2)_5CH_2$), 3.75 (s, 2H, $CH_2SnBu_3$); $^{13}$C NMR 9.0 ($CH_3$), 13.7 ($CH_2CH_3$), 27.3 ($CH_2CH_2CH_2$ $CH_3$), 29.1 ($CH_2CH_2CH_3$), 61.6 ($CH_2SnBu_3$), 62.4 ($CH_2OCH_2SnBu_3$), 70.1, 70.3, 70.4, 70.5, 70.6, 70.7, 70.8, 70.9 and 71.1, 72.6 ($HOCH_2CH_2$), 74.5 ($HOCH_2$); MS(EI), *m/z* (rel. intensity) 529 (M–$C_4H_9$, 100), 291 (23), 235 (35); HRMS(EI), *m/z* 529.2175 (M–$C_4H_9$, $C_{21}H_{45}O_7{}^{120}$Sn requires 529.2188).

### 2.3. Tri-n-butylstannylmethyl-substituted phthalimido-polyethers *19a–f*

To independent THF (100 ml) solutions of 2.00 mmol of the polyethyleneglycol-alcohols **18a** (0.73 g), **18b** (0.82 g), **18c** (0.91 g), **18d** (1.00 g), **18e** (1.10 g) and **18f** (1.20 g) and triphenylphosphine (0.52 g, 2.00 mmol) was added THF (10 ml) solutions of diisopropyl azodicarboxylate (0.40 g, 2.00 mmol) over 1 h periods. The resulting solutions were stirred for 3 days at 25 °C and concentrated in vacuo to afford residues which were subjected to column chromatography (silica gel, 1:5 EtOAc-hex) yielding 0.64 g (65%) of **19a**, 0.48 g (45%) of **19b**, 0.46 g (40%) of **19c**, 0.41 g (33%) of **19d**, 1.10 g (83%) of **19e** and 1.20 g (80%) of **19f**.

**19a**: $^1$H NMR 0.76–0.87 (m, 15H, $SnCH_2CH_2CH_2CH_3$), 1.11–1.33 (m, 6H, $CH_2CH_3$), 1.36–1.45 (m, 6H, $CH_2CH_2CH_3$), 3.56 (t, 2H, $J$ = 10.8 Hz, $NCH_2CH_2$), 3.87 (t, 2H, $J$ = 11.4 Hz, $NCH_2$), 3.70 (s, 2H, $OCH_2SnBu_3$), 7.69–7.85 (m, 4H, aromatic); $^{13}$C NMR 8.7 ($CH_3$), 13.5 ($CH_2CH_3$), 27.1 ($CH_2CH_2CH_2CH_3$), 28.9 ($CH_2CH_2CH_3$), 37.5 ($NCH_2$), 61.8 ($CH_2SnBu_3$), 71.7 ($NCH_2CH_2$), 123 and 133.7 (CH, aromatic), 132.1 (C, aromatic), 168.0 (C=O); MS(FAB), *m/z* (rel. intensity) 494 (2), 438 (100), 437 (41), 324 (15), 174 (91), 120 (66); HRMS(FAB), *m/z* 438.1095 (M–$C_4H_9$, $C_{19}H_{28}O_3{}^{120}$SnN, requires 438.1091).

**19b**: $^1$H NMR 0.81–0.89 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.23–1.39 (m, 6H, C$H_2$C$H_3$), 1.41–1.51 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.40–3.47 (m, 2H, C$H_2$OC$H_2$SnBu$_3$), 3.57–3.60 (m, 2H, NC$H_2$C$H_2$OC$H_2$), 3.67–3.74 (m, 2H, NC$H_2$C$H_2$), 3.71 (s, 2H, OC$H_2$SnBu$_3$), 3.84–3.87 (m, 2H, NC$H_2$), 7.67–7.83 (m, 4H, aromatic); $^{13}$C NMR 8.9 (C$H_3$), 13.7 (C$H_2$C$H_3$), 27.3 (C$H_2$C$H_2$C$H_2$C$H_3$), 29.0 (C$H_2$C$H_2$C$H_3$), 37.2 (NC$H_2$), 62.5 (C$H_2$SnBu$_3$), 67.9 (C$H_2$OC$H_2$SnBu$_3$), 69.8 (C$H_2$C$H_2$OC$H_2$SnBu$_3$), 74.6 (NC$H_2$C$H_2$O), 123.2 and 133.8 (CH, aromatic), 132.1 (C, aromatic), 168.2 (C=O); MS(EI), *m/z* (rel. intensity) 489 (8), 432 (100), 291 (28), 233 (56), 124 (70); HRMS(EI), *m/z* 489.1899 ($C_{21}H_{39}O_4{}^{120}$SnN requires 489.1901).

**19c**: $^1$H NMR 0.82–0.90 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.21–1.32 (m, 6H, C$H_2$C$H_3$), 1.39–1.51 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.38–3.43 (t, 2H, $J$ = 10.0 Hz, C$H_2$OC$H_2$SnBu$_3$), 3.51–3.56 (t, 2H, $J$ = 10.0 Hz, C$H_2$C$H_2$OC$H_2$SnBu$_3$), 3.57–3.64 (m, 4H, NHC$H_2$C$H_2$OC$H_2$C$H_2$), 3.69–3.75 (t, 2H, $J$ = 11.0 Hz, NC$H_2$C$H_2$), 3.86–3.92 (m, 2H, $J$ = 11.0 Hz, NC$H_2$), 3.73 (s, 2H, C$H_2$SnBu$_3$), 7.67–7.72 (m, 2H, aromatic), 7.81–7.83 (m, 2H, aromatic); $^{13}$C NMR 9.0 (C$H_3$), 13.7 (C$H_2$C$H_3$), 27.3 (C$H_2$C$H_2$C$H_2$C$H_3$), 29.1 (C$H_2$C$H_2$C$H_3$), 37.2 (NC$H_2$), 62.4 (C$H_2$SnBu$_3$), 67.9 (C$H_2$OC$H_2$SnBu$_3$), 70.0 (C$H_2$C$H_2$OC$H_2$SnBu$_3$), 70.4 (NC$H_2$C$H_2$OC$H_2$C$H_2$), 70.6 (NC$H_2$C$H_2$OC$H_2$), 74.5 (NC$H_2$C$H_2$O), 123.2 and 133.9 (CH, aromatic), 132.1 (C, aromatic), 168.0 (C=O); MS(EI), *m/z* (rel. intensity) 583 (3), 526 (100), 467 (8), 411 (12), 354 (6), 230 (14), 124 (48); HRMS(EI), *m/z* 526.1591 (M–C$_4$H$_9$, $C_{25}H_{40}O_6{}^{120}$SnN requires 526.1614).

**19d**: $^1$H NMR 0.82–0.90 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.17–1.32 (m, 6H, C$H_2$C$H_3$), 1.35–1.47 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.41–3.46 (m, 2H, C$H_2$OC$H_2$SnBu$_3$), 3.51–3.64 (m, 10H, NC$H_2$C$H_2$(OC$H_2$C$H_2$)$_2$OC$H_2$), 3.71 (t, 2H, $J$ = 10.0 Hz, NC$H_2$C$H_2$), 3.73 (s, 2H, C$H_2$SnBu$_3$), 3.87 (t, 2H, $J$ = 10.0 Hz, NC$H_2$), 7.67–7.71 (m, 2H, aromatic), 7.80–7.84 (m, 2H, aromatic); $^{13}$C NMR 8.9 (C$H_3$), 13.7 (C$H_2$C$H_3$), 27.2 (C$H_2$C$H_2$C$H_2$C$H_3$), 29.0 (C$H_2$C$H_2$C$H_3$), 37.1 (NC$H_2$), 62.4 (C$H_2$SnBu$_3$), 67.8 (C$H_2$OC$H_2$SnBu$_3$), 69.9, 70.3, 70.4, 70.5, 70.6 and 74.5 (CH$_2$), 123.1 and 133.8 (CH, aromatic), 132.0 (C, aromatic), 168.2 (C=O); MS(EI), *m/z* (rel. intensity) 570 (71), 566 (29), 456 (4), 235 (23), 174 (100); HRMS(EI), *m/z* 570.1887 (M–C$_4$H$_9$, $C_{25}H_{40}O_6{}^{120}$SnN requires 570.1877).

**19e**: $^1$H NMR 0.82–0.89 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.20–1.35 (m, 6H, C$H_2$C$H_3$), 1.39–1.54 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.43–3.47 (m, 2H, C$H_2$OC$H_2$SnBu$_3$), 3.56–3.71 (m, 16H, NC$H_2$(CH$_2$OC$H_2$)$_4$), 3.74 (s, 2H, C$H_2$SnBu$_3$), 7.69 (t, 2H, $J$ = 5.7 Hz, NC$H_2$), 7.67–7.71 (m, 2H, aromatic), 7.80–7.84 (m, 2H, aromatic); $^{13}$C NMR 8.9 (C$H_3$), 13.7 (C$H_2$C$H_3$), 27.2 (C$H_2$C$H_2$C$H_2$C$H_3$), 29.0 (C$H_2$C$H_2$C$H_3$), 37.1 (NC$H_2$), 62.4 (C$H_2$SnBu$_3$), 67.8 (C$H_2$OC$H_2$SnBu$_3$), 69.9, 70.1, 70.3, 70.4, 70.5, 70.6 and 70.9 (CH$_2$), 74.5 (NC$H_2$C$H_2$), 123.2 and 133.9 (CH, aromatic), 132.0 (C, aromatic), 168.2 (C=O); MS(EI), *m/z* (rel. intensity) 614 (M–C$_4$H$_9$, 68), 291 (14), 235 (23), 174 (100); HRMS(EI), *m/z* 614.2158 (M–C$_4$H$_9$, $C_{27}H_{44}O_7{}^{120}$SnN requires 614.2140).

**19f**: $^1$H NMR 0.82–0.90 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.21–1.35 (m, 6H, C$H_2$C$H_3$), 1.39–1.55 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.45–3.47 (m, 2H, NC$H_2$), 3.56–3.71 (m, 20H, NC$H_2$(CH$_2$OC$H_2$)$_5$), 3.74 (s, 2H, C$H_2$SnBu$_3$), 3.87 (t, 2H, $J$ = 5.6 Hz, NC$H_2$), 7.67–7.71 (m, 2H, aromatic), 7.80–7.84 (m, 2H, aromatic); $^{13}$C NMR 8.9 (C$H_3$), 13.7 (C$H_2$C$H_3$), 27.2 (C$H_2$C$H_2$C$H_2$C$H_3$), 29.0 (C$H_2$C$H_2$C$H_3$), 37.1 (NC$H_2$), 62.4 (C$H_2$SnBu$_3$), 67.8 (C$H_2$OC$H_2$SnBu$_3$), 70.1, 70.2, 70.3, 70.4, 70.6, 70.7, 70.8, 70.9 and 71.1 (CH$_2$), 74.6 (NC$H_2$C$H_2$), 123.2 and 133.9 (CH, aromatic), 132.1 (C, aromatic), 168.2 (C=O); MS(EI), *m/z* (rel. intensity) 658 (M–C$_4$H$_9$, 100), 600 (20), 291 (4),174 (7); HRMS(EI), *m/z* 658.2390 (M–C$_4$H$_9$, $C_{29}H_{48}O_8{}^{120}$SnN requires 658.2402).

### *2.4. Tri-n-butylstannylmethyl-substituted maleimido-polyethers **22** and **23***

To independent THF (100 ml) solutions of 3.60 mmol of polyethyleneglycol-alcohols **18a** (1.32 g) and **18b** (1.47 g) and triphenylphosphine (3.60 mol, 0.95 g) was added THF (10 ml) solutions of diisopropyl azodicarboxylate (3.60 mmol, 0.73 g) over a 2 h period. The resulting solutions were stirred for 2 days at 25 °C and concentrated in vacuo to afford residues which were subjected to column chromatography (silica gel, 1:5 EtOAc-hex) yielding 0.64 g (40%) of **22** and 0.53 g (30%) of **23**.

**22**: $^1$H NMR 0.79–0.89 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.19–1.30 (m, 6H, C$H_2$C$H_3$), 1.39–1.44 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.45 (t, 2H, $J$ = 11.4 Hz, NC$H_2$), 3.65 (t, 2H, $J$ = 11.6 Hz, NC$H_2$C$H_2$), 3.66 (s, 2H, C$H_2$SnBu$_3$), 6.66 (s, 2H, HC=CH); $^{13}$C NMR 9.7 (C$H_3$), 13.5 (C$H_2$C$H_3$), 27.1 (C$H_2$C$H_2$C$H_2$C$H_3$), 28.9 (C$H_2$C$H_2$C$H_3$), 36.9 (NC$H_2$), 62.2 (C$H_2$SnBu$_3$), 72.1 (NC$H_2$C$H_2$), 133.9 (CH), 170.3 (C=O); MS(EI), *m/z* (rel. intensity) 388 (16), 291 (13), 235 (34), 176 (100), 120 (30); HRMS(EI), *m/z* 388.0938 (M–C$_4$H$_9$, $C_{15}H_{26}O_3{}^{120}$SnN requires 388.0935).

**23**: $^1$H NMR 0.75–0.91 (m, 15H, SnC$H_2$C$H_2$C$H_2$C$H_3$), 1.19–1.37 (m, 6H, C$H_2$C$H_3$), 1.41–1.53 (m, 6H, C$H_2$C$H_2$-C$H_3$), 3.42 (t, 2H, $J$ = 4.4 Hz, NC$H_2$), 3.54–3.72 (m, 6H, NC$H_2$C$H_2$OC$H_2$C$H_2$), 3.73 (s, 2H, C$H_2$SnBu$_3$), 6.69 (s, 2H, HC=CH); $^{13}$C NMR 8.7 (C$H_3$), 13.5 (C$H_2$C$H_3$), 27.1 (C$H_2$C$H_2$C$H_2$C$H_3$), 28.9 (C$H_2$C$H_2$C$H_3$), 36.9 (NC$H_2$), 62.2 (C$H_2$SnBu$_3$), 67.2 (C$H_2$C$H_2$SnBu$_3$), 69.6 (NC$H_2$C$H_2$OC$H_2$), 74.5 (NC$H_2$C$H_2$), 133.9 (CH), 170.3 (C=O); MS(EI), *m/z* (rel. intensity) 432 (98), 374 (8), 343 (7), 291 (28), 235 (58), 179 (100); HRMS(EI), *m/z* 489.0899 ($C_{21}H_{39}O_4{}^{120}$Sn requires 489.1901).

### *2.5. Trimethylsilylmethyl-substituted maleimido-polyethers **24** and **25***

To independent THF (100 ml) solutions of the known [6] polyethyleneglycol-alcohols **21a** (1.45 g, 10.2 mmol) and **21b** (2.00 g, 10.4 mmol) and triphenylphosphine (2.70 g,

10.4 mmol) was added solutions of diisopropyl azodicarboxylate (2.10 g, 10.4 mmol) in 10 ml of THF over a 2 h period. The solutions were stirred for 3 days at 25 °C and concentrated in vacuo to afford residues which were subjected to column chromatography (silica gel, 1:5 EtOAc-hex) yielding 1.61 g (68%) of **24** and 1.97 g (70%) of **25**.

**24**: m.p. 39–40 °C; $^1$H NMR −0.04 (s, 9H, $SiMe_3$), 3.08 (s, 2H, $CH_2SiMe_3$), 3.52 (t, 2H, $J$ = 5.6 Hz, $NCH_2CH_2$), 3.69 (t, 2H, $J$ = 5.6 Hz, $NCH_2$), 6.67 (s, 2H, CH=CH); $^{13}$C NMR −2.7 ($SiMe_3$), 37.4 ($CH_2SiMe_3$), 65.2 ($NCH_2CH_2$), 71.9 ($NCH_2$), 134.6 (CH=CH), 171.0 (C=O); IR (KBr) 1710 (C=O); MS(EI), *m/z* (rel. intensity) 227 (2), 124 (19), 103 (39), 73 (100), 59 (18); HRMS(EI), *m/z* 227.0976 ($C_{10}H_{17}NO_3Si$ requires 227.0998).

**25**: $^1$H NMR 0.01 (s, 9H, $SiMe_3$), 3.11 (s, 2H, $CH_2SiMe_3$), 3.48–3.71 (m, 8H, $NCH_2CH_2OCH_2CH_2$), 6.68 (s, 2H, CH=CH); $^{13}$C NMR 3.1 ($SiMe_3$), 37.1 ($NCH_2$), 65.3 ($CH_2SiMe_3$), 67.7 ($CH_2OCH_2SiMe_3$), 69.8 ($NCH_2CH_2OCH_2$), 74.5 ($NCH_2CH_2$), 134.0 (CH=CH), 170.5 (C=O); MS(EI), *m/z* (rel. intensity) 271 (1), 212 (4), 183 (11), 124 (86), 73 (100); HRMS(EI), *m/z* 271.1234 ($C_{12}H_{21}O_4NSi$ requires 271.1240).

### 2.6. General photochemical procedures

Nitrogen purged solutions of the substrates in the indicated solvents were irradiated by using Pyrex glass filtered light in an water cooled (17 °C) immersion reactor for time periods required to give >90% conversion (determined by UV spectroscopy). Concentration of the photolysates in each case was followed by column chromatography (silica gel, EtOAc-hex) to yield the photoproducts listed below.

### 2.7. Irradiation of tri-n-butylstannylmethyl-substituted phthalimido-polyethers *19a–f*

Solutions of the tri-*n*-butylstannylmethyl-substituted phthalimido-polyethers (**19a**, 0.30 g, 5.87 mmol; **19b**, 0.30 g, 5.58 mmol; **19c**, 0.27 g, 4.59 mmol; **19d**, 0.27 g, 4.30 mmol; **19e**, 0.36 g, 0.50 mmol; **19f**, 0.33 g, 0.46 mmol) in 100 ml of methanol were independently irradiated. The general work-up and purification procedure (see above) gave the previously characterized [6] cyclized products **20a–f** (Table 1).

### 2.8. Irradiations of tri-n-butylstannylmethyl-substituted maleimido-polyethers *22* and *23*

Solutions of tri-*n*-butylstannylmethyl-substituted maleimido-polyethers **22** and **23** in 100 ml of the indicated solvent (Table 1) were independently irradiated. The general work-up and purification procedure (see above) gave the cyclized products listed in Table 1.

**26**: m.p. 144–146 °C; $^1$H NMR 3.19–3.27 (m, 2H, $NCH_2CH_2$), 3.30–3.41 (m, 2H, $NCH_2$), 3.95 (s, $^1$H, OH), 4.02 and 4.20 (2d, 2H, $J$ = 11.6 Hz, $C(OH)CH_2$), 6.26 and 6.96 (2d, 2H, $J$ = 5.9 Hz, CH=CH); $^1$H NMR −38.0 ($NCH_2$), 67.3 ($NCH_2CH_2$), 75.0 ($C(OH)CH_2$), 87.1 (COH), 130.0 and 146.6 (CH=CH), 168.2 (C=O); IR(KBr) 3200–3500 (br, OH), 1660 (C=O); MS(EI), *m/z* (rel. intensity) 155 (56), 125 (38), 110 (100), 58 (23), 110 (100), 58 (23); HRMS(EI), *m/z* 155.0587 ($C_7H_9NO_3$ requires 155.0582).

**28**: m.p. 167–169 °C; $^1$H NMR 1.83 (s, $^1$H, OH), 3.46–4.06 (m, 8H, $NCH_2CH_2OCH_2CH_2$), 3.90 and 4.11 (2d, 2H, $J$ = 4.4 Hz, $C(OH)CH_2O$), 6.11 and 6.83 (2d, 2H, $J$ = 2.4 Hz, CH=CH); $^{13}$C NMR 40.1 ($NCH_2$), 70.9 ($NCH_2CH_2$), 71.3 ($NCH_2CH_2OCH_2$), 74.9 ($NCH_2CH_2OCH_2CH_2OCH_2$), 91.2 (COH), 128.3 and 147.8 (CH=CH), 170.4 (C=O); MS(EI), *m/z* (rel. intensity) 199 (3), 182 (1), 181 (2), 156 (100), 125 (42); HRMS(EI), *m/z* 199.0851 ($C_9H_{13}NO_4$ requires 199.0845).

**29**: m.p. 108–109 °C; $^1$H NMR 2.67 (dd, $^1$H, $J$ = 6.4 and 0.6 Hz, $H_2C$–CON), 2.79 and 2.83 (2d, $^1$H, $J$ = 2.4 Hz, $H_2C$–CON), 2.88 (dd, $^1$H, $J$ = 0.6 Hz and $J$ = 2.4 Hz, $CH_2CHCON$), 3.38–4.05 (m, 10H, $NCH_2(CH_2OCH_2)_2$); $^{13}$C NMR 32.7 ($CH_2CON$), 39.3 ($NCH_2$), 42.9 (HCCON), 67.7 ($NCH_2CH_2O$), 72.7 ($NCH_2CH_2OCH_2$), 73.5 ($NCH_2CH_2OCH_2CH_2$), 73.9 ($NCH_2CH_2OCH_2$ $CH_2OCH_2$), 177.5 and 180.0 (C=O); MS(EI), *m/z* (rel. intensity) 199 (8), 169 (84), 139 (62), 97 (25), 54 (100); HRMS(EI), *m/z* 199.0836 ($C_9H_{13}NO_4$ requires 199.0845).

### 2.9. Irradiation of trimethylsilylmethyl-substituted maleimido-polyethers *24* and *25*

Solutions of trimethylsilylmethyl-substituted maleimido-polyethers **24** and **25** (0.27 g, 4.59 mmol) in 100 ml of the indicated solvent (Table 1) were independently. The general work-up and purification procedure (see above) gave the cyclized products listed in Table 1.

**27**: m.p. 173–175 °C; $^1$H NMR −0.10 (s, 9H, $SiMe_3$), 3.10–3.28 (m, 4H, $NCH_2CH_2$), 3.93–4.02 (2d, 2H, $J$ = 9.8 Hz, $C(OSiMe_3)CH_2$), 6.28 and 6.94 (d, 2H, $J$ = 5.9 Hz, CH=CH); $^{13}$C NMR −1.5 ($SiMe_3$), 38.4 ($NCH_2$), 69.2 ($NCH_2CH_2$), 81.1 ($C(OSiMe_3)CH_2$), 90.2 ($COSiMe_3$), 129.6 and 148.0 (CH=CH), 167.9 (C=O); IR(KBr) 1670 (C=O); MS(EI), *m/z* (rel. intensity) 227 (13), 218 (43), 125 (84), 110 (100), 73 (90); HRMS(EI), *m/z* 227.0972 ($C_{10}H_{17}NO_3Si$ requires 227.0978).

**30** (asymmetric): m.p. 85–87 °C; $^1$H NMR 0.10 and 0.16 (s, 18H, $SiMe_3$), 2.56–2.58 (m, 2H, $NCOCH_2$), 2.60–2.62 (m, 2H, $NCOCH_2$), 2.81–2.84 (m, 2H, $NCOCH_2CH$), 3.02 (s, 2H, $CH_2SiMe_3$), 3.18 (s, 2H, $CH_2SiMe_3$), 3.34–3.89 (m, 14H, $NCH_2CH_2OCH_2CH$); $^{13}$C NMR −2.7 and −2.9 ($SiMe_3$), 39.2 and 39.3 ($CH_2SiMe_3$), 31.4 and 39.7 ($NCOCH_2$), 43.5 and 43.7 (NOCH), 66.8, 68.3, 71.7, 72.6, 74.7 and 75.2 ($CH_2$), 79.5 and 80.3 ($CH_2OCH_2SiMe_3$), 177.1, 177.8, 177.9 and 182.3 (C=O); MS(FAB), *m/z* (rel. intensity) 543 (M + 1, 2), 272 (36), 256 (14), 138

(10), 73 (100); HRMS(FAB), *m/z* 272.1309 (1/2M + 1, $C_{12}H_{22}O_4NSi$ requires 272.1318).

**31** (symmetric): m.p. 108–110 °C; $^1$H NMR 0.14 (s, 18H, $SiMe_3$), 3.10 (s, 4H, $CH_2SiMe_3$), 3.5–3.9 (m, 24H); $^{13}$C NMR –3.0 ($SiMe_3$), 39.2 ($CH_2SiMe_3$), 39.6 ($NCOCH_2$), 43.4 (OCH); 68.2, 71.7, 74.9, 80.2 ($OCH_2SiMe_3$), 177.0, 177.7; MS(FAB), *m/z* (rel. intensity) 543 (M + 1, 5), 272 (100), 256 (12), 138 (46); HRMS(FAB), *m/z* 543.2598 (M+ 1, $C_{24}H_{43}O_8N_2Si_2$ requires 272.1318).

**32**: m.p. 149–151 °C; $^1$H NMR −0.02 (s, 18H, $2SiMe_3$), 3.11 (s, 4H, $CH_2SiMe_3$), 3.36 (s, 4H, 4CH), 3.61 (t, 4H, $J$ = 5.4 Hz, $2NCH_2CH_2$), 3.80 (t, 4H, $J$ = 5.4 Hz, $2NCH_2$); $^{13}$C NMR −3.2 ($SiMe_3$), 38.4 ($CH_2SiMe_3$), 41.3 (CH), 64.7 ($NCH_2CH_2$), 70.4 ($NCH_2$), 174.6 (C=O); IR(KBr) 1700 (C=O); MS(EI), *m/z* 454 (0.2), 439 (28), 351 (38), 197 (20), 103 (24), 73 (100); HRMS(EI), *m/z* 454.1955 ($C_{20}H_{34}N_2O_6Si$ requires 454.1956).

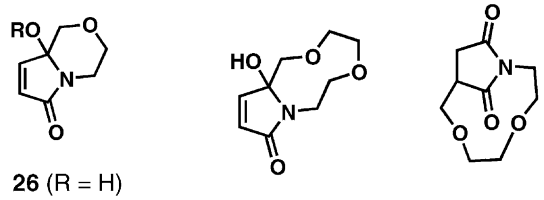
## 3. Results and discussion

The phthalimido-polyethers **19a–f** and maleimido-polyethers **22–25** used in this study are prepared by using Mitsunobo coupling of the corresponding polyether-alcohols **18a–f** and **21a–b** with phthalimide or maleimide (Section 2). Photocyclization reactions of these substrates are performed by using the same conditions employed for analogous reactions of the $Me_3Si$-terminated phthalimido-polyethers. Accordingly, irradiation of $N_2$-purged solutions (ca. 0.05 mM) of the substrates with Pyrex glass filtered light ($\lambda > 290$ nm) were conducted for time periods leading to >90% conversion of the starting materials. Concentration of each photolysate followed by chromatographic separation leads to isolation of the photoproducts in the yields recorded in Table 1. The macrocyclic amidols **20a–f**, arising by photocyclization of the corresponding phthalimido-polyethers **19a–f**, have been characterized previously [6]. The structures of the photoproducts obtained from maleimides **22–25** are assigned based on their spectroscopic properties and, when appropriate, by comparisons of their spectroscopic data with those of related phthalimide derived photoproducts [6].

As can be seen by viewing the results of the phthalimido-polyether photocyclization reactions (Scheme 4 and Table 1), replacement of the $Me_3Si$ group by the $Bu_3Sn$ moiety leads to a significant yield enhancement, especially for reactions of the longer chain ($n > 1$) substrates. These findings are likely a consequence of the larger rates for destannylation of intermediate zwitterionic biradicals **33** (Scheme 7). The enhanced rates of conversion of **33** to 1,ω-biradicals **35** results in increases in the quantum efficiencies of formation of the macrocyclic products. Consequently, the chemical yields of products produced by the sequential SET-destannation pathway become larger than those arising by other yield diminishing, excited-state routes (e.g., H-atom abstraction to form **34**).

Table 1
Products and yields from photoreactions of the tributylstannane-substituted phthalimido-polyethers **19a–f** and maleimido-polyethers **22–25**

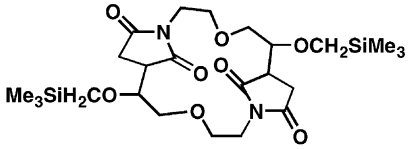
| Polyether substrate | Solvent | Products (yields, %) |
|---|---|---|
| **19a** | MeOH | **20a** (98) |
| **19b** | MeOH | **20b** (98) |
| **19c** | MeOH | **20c** (95) |
| **19d** | MeOH | **20d** (68) |
| **19e** | MeOH | **20e** (88) |
| **19f** | MeOH | **20f** (72) |
| **22** | MeCN | **26** (22) |
| **22** | MeOH | **26** (58) |
| **22** | 35% $H_2O$–MeCN | **26** (77) |
| **22** | 35% $H_2O$–MeOH | **26** (86) |
| **22** | Acetone | **26** (82) |
| **23** | MeOH | **29** (22) |
| **23** | 35% $H_2O$–MeCN | **28** (7), **29** (21) |
| **23** | 35% $H_2O$–MeOH | **28** (7), **29** (34) |
| **23** | Acetone | **28** (7) |
| **24** | MeCN | **26** (18), **27** (13), **32** (28) |
| **24** | 35% $H_2O$–MeCN | **26** (64), **27** (34) |
| **24** | 35% $H_2O$–MeCN | **26** (80), **27** (8) |
| **24** | Acetone | **26** (6), **27** (7) **32** (54) |
| **25** | MeOH | **28** (2), **30** (34) |
| **25** | 35% $H_2O$–MeCN | **28** (11), **30** (25), **31** (11) |
| **25** | 35% $H_2O$–MeON | **28** (12), **30** (28) |
| **25** | Acetone | **31** (99) |



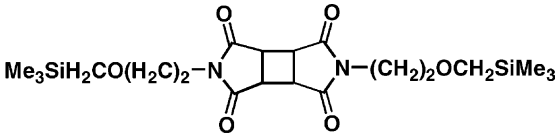

**26** (R = H)
**27** (R = $SiMe_3$)

**28**

**29**

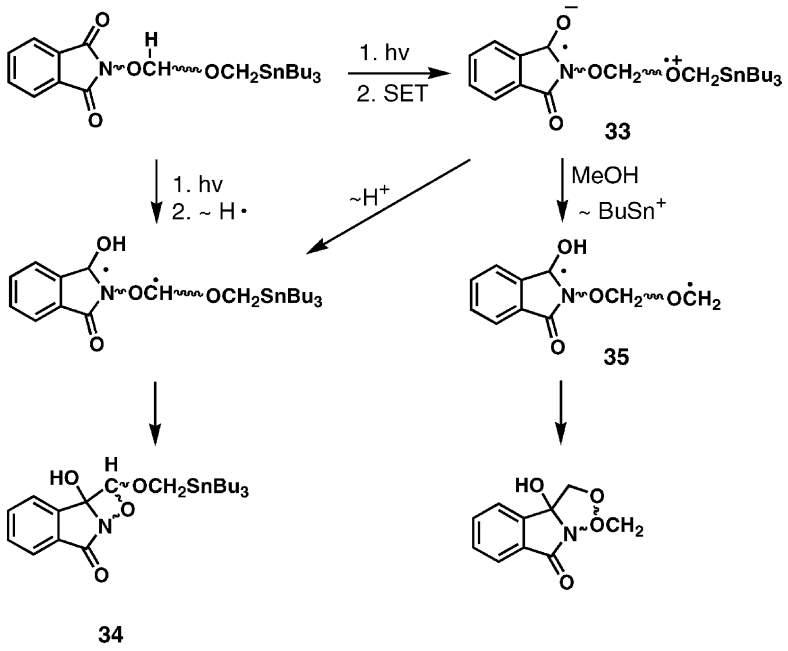



**30** (asymmetric)
**31** (symmetric)




**32**

Similar conclusions can be drawn from a comparison of the results of photocyclization reactions of the $Me_3Si$- and $Bu_3Sn$-substituted maleimido-polyethers **22–25**. In sol-

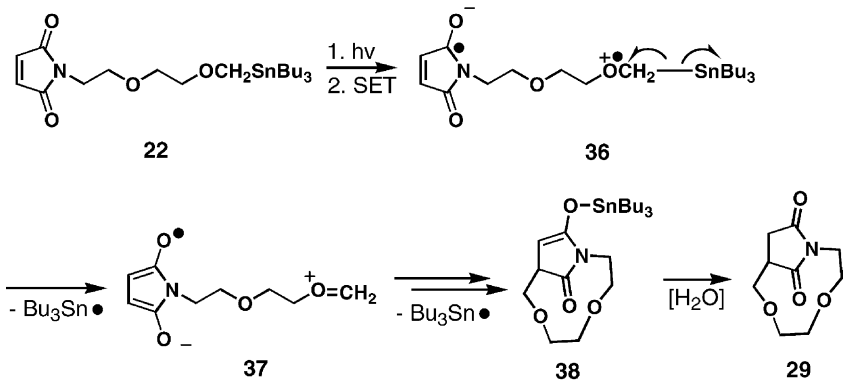



Scheme 7.

vents of lower silophilicity (e.g., MeCN), photoreactions of the $Me_3Si$-terminated substrates, **24** and **25**, generally lead to mixtures of products arising by competitive sequential SET desilylation (→**26**–**28**), H-atom abstraction (→**30**–**31**) and 2 + 2-dimerization (→**32**) routes. In contrast, the $Bu_3Sn$-containing substrates, **22** and **23**, undergo more selective photocyclizations. The major products formed in these processes result from sequential SET-destannation pathways. In both cases, an increase in solvent nucleophilicity, which enhances the rates of cation radical desilylation and destannation, results in an increase in the yields of products coming from sequential SET demetallation routes. As an informed referee of this manuscript has pointed out, it is not clear why replacement of a $Me_3Si$ by a $Bu_3Sn$ group has a greater effect on the yields of photoreactions of the longer chain rather than shorter chain substrates. If no interaction between the anion radical and cation radical centers occurs when the $Me_3Si$ or $Bu_3Sn$ groups are transferred to an external silophile, why should the greater rate of destannation more influence reactions of the long chain substrates. Thus, another source of the rate enhancement by the $Bu_3Sn$-analogs must exist. This source might be found in the greater driving force for SET from the more easily oxidized Sn–C bond to the excited phthalimide chromophore. In the tin-containing long chain substrates, the enhanced driving force could translate into a greater rate of long range SET, thus, making SET occur more rapidly than other reactive pathways for excited state decay. This would translate into higher photocyclization yields for the tin- vs. silicon-containing phthalimides.

Acetone triplet sensitization appears to have opposite effects on photoreactions of the $Me_3Si$- and $Bu_3Sn$-terminated maleimides. For example, triplet reaction of **24** is dominated by photodimerization, which yields the 2 + 2 adduct **32**. However, the bicyclic-amidol **26** is formed cleanly by acetone sensitized irradiation of the $Bu_3Sn$-substrate **22**. This phenomenon might be a consequence of the lower oxidation potentials of α-stannyl vs. α-silyl ethers [13]. Generally, triplet excited-state reduction potentials are smaller than those of the corresponding singlets owing to the normal energetic ordering which places singlet excited states at higher energies than the corresponding triplets. Thus, in the case of $Me_3Si$ substrates, SET from the terminal ether moiety to the triplet excited maleimide might be energetically unfavorable and, therefore, less competitive with 2 + 2-cycloaddition. In contrast, rapid SET from the more easily oxidized $OCH_2SnBu_3$ moiety in **22** would initiate reaction leading to the amidol **26**.

Another interesting observation made in this investigation points to the possible involvement of a second mechanism in SET-induced photoreactions of the $Bu_3Sn$-containing maleimides. This proposal stems from difference seen in the nature of the cyclization products formed upon irradiation of the $Me_3Si$- vs. $Bu_3Sn$-substituted maleimido-polyethers, **23** and **25**. The major product of photocyclization of the $Bu_3Sn$-derivative **23** is succinimide **29** while the sole product of SET-promoted cyclization of the silicon analog **25** is the amidol **28**. The differences in the positions of bond formation between the terminal carbon and maleimide ring in these processes can be explained by invocation of a dipolar cyclization mechanism as competitive pathway in the photoreaction of the $Bu_3Sn$ maleimide **23**. In this sequence (Scheme 8), SET from the terminal $OCH_2SnBu_3$ moiety to the excited maleimide is followed by homolytic (rather




Scheme 8.

than heterolytic) cleavage α to the cation radical site in the intermediate zwitterionic biradical **36** [14]. The lower BDE of C–Sn vs. C–Si bonds [15] and greater stability of the tributylstannyl vs. trimethylsilyl radical may account for the facility of the homolytic vs. heterolytic cleavage pathways in the tin-containing intermediate. The dipolar transient **37**, generated in this manner, can undergo cyclization by addition of the α-carbon of the enolate anion to the oxonium ion followed by coupling to the $Bu_3Sn$ radical (→**38**). Hydrolysis of the stannyl ether moiety in **38** on work-up would then furnish the succinimide derivative **29**.

## 4. Summary

The observations reported above demonstrate that replacement of $Me_3Si$ with $Bu_3Sn$ as the terminal electrofugal group in phthalimido- and maleimido-polyethers leads to an enhancement in the yields and product selectivity of their macrocyclic ring forming photocyclization reactions. In addition, the differences in the nature of the major cyclization products formed by irradiation of the $Me_3Si$- vs. $Bu_3Sn$-terminated maleimido-polyethers suggests that an alternative, perhaps polar cyclization mechanism is operable in photoreactions of the $Bu_3Sn$-containing substrates.

## Acknowledgements

This investigation was supported by grants from the Basic Research Program of the Korea Science and Engineering Foundation (2000-1-123-006-3, UCY) and National Science Foundation International Program (INT-9796064, PSM) and National Institutes of Health (GM-27251, PSM).

## References

[1] U.C. Yoon, P.S. Mariano, Acc. Chem. Res. 34 (2001) 523.
[2] J.D. Coyle, in: W.M. Horspool (Ed.), Synthetic Organic Photochemistry, Plenum Press, New York, 1984, pp. 259.
[3] X.M. Zhang, S.P. Yeh, S. Hong, M. Freccero, A. Albini, D.E. Falvey, P.S. Mariano, J. Am. Chem. Soc. 116 (1994) 4211.
[4] Z. Su, D.E. Falvey, U.C. Yoon, S.W. Oh, P.S. Mariano, J. Am. Chem. Soc. 120 (1998) 10676.
[5] U.C. Yoon, J.W. Kim, J.Y. Ryu, S.J. Cho, S.W. Oh, P.S. Mariano, J. Photochem. Photobiol. A 106 (1997) 145.
[6] U.C. Yoon, S.W. Oh, C.W. Lee, Heterocycles 41 (1995) 2665.
[7] U.C. Yoon, S.J. Lee, K.J. Lee, S.J. Cho, C.W. Lee, P.S. Mariano, Bull. Korean Chem. Soc. 15 (1994) 154.
[8] U.C. Yoon, J.H. Oh, S.J. Lee, D.U. Kim, J.G. Lee, K.T. Kang, P.S. Mariano, Bull. Korean Chem. Soc. 13 (1992) 166.
[9] U.C. Yoon, S.W. Oh, J.H. Lee, J.H. Park, K.T. Kang, P.S. Mariano, J. Org. Chem. 66 (2001) 939.
[10] R.M. Borg, P.S. Mariano, Tetrahedron Lett. 27 (1986) 2821.
[11] J.-I. Yoshida, K. Takada, Y. Ishichi, S. Isoe, J. Chem. Soc., Chem. Commun. (1994) 2361.
[12] J.-I. Yoshida, Y. Ishichi, S. Isoe, J. Am. Chem. Soc. 114 (1992) 7594.
[13] J.-I. Yoshida, S. Isoe, Tetrahedron Lett. 28 (1987) 6621; J.-I. Yoshida, M. Itoh, S. Isoe, J. Chem. Soc., Chem. Commun. 547 (1993).
[14] J.-I. Yoshida, Y. Ishichi, K. Nishikawa, S. Shiogawa, S. Isoe, Tetrahedron Lett. 33 (1992) 2599.
[15] R.A. Jackson, J. Organometal. Chem. 166 (1979) 17.