at reflux for 2 h. Solvent was evaporated, and water (30 mL) was added to the residue, which was extracted with $CHCl_3$ (3 × 25 mL). The organic phase was washed with water (15 mL), dried, and concentrated to yield 66 mg of product. The aqueous layers were further basified with ice-cold 5% NaOH (10 mL) and extracted with $CHCl_3$ (4 × 40 mL). After a water wash (25 mL), solvent was removed to give 0.149 g (combined) **10a** for a 95% yield: NMR δ 1.35–1.95 (m, 25 H), 2.25–2.85 (m, 6 H), 2.95–3.38 (m, 6 H), 3.50 (s, 2 H), 5.32 (br s, 1 H), 7.33 (m, 5 H). Anal. Calcd for $C_{27}H_{45}Cl_3N_4O_4$: C, 54.41; H, 7.61; N, 9.40. Found: C, 54.28; H, 7.64; N, 9.31.

Acylation of 10a (10b). *p*-Toluoyl chloride (36.9 mg, 0.239 mmol) in CH_2Cl_2 (6 mL) was added to **10a** (0.130 g, 0.218 mmol) and triethylamine (27.6 mg, 0.273 mmol) and the solution stirred for 1 day. The reaction was worked up by the method of **9b** to give 0.17 g of the crude product. Preparative-layer chromatography (3% EtOH/$CHCl_3$) furnished 0.137 g of **10b** for an 88% yield: NMR δ 1.37–2.05 (m, 23 H), 2.3–2.62 (m, 7 H), 3.02–3.6 (m, 10 H), 5.3 (br s, 1 H), 7.15–7.92 (m, 10 H). Anal. Calcd for $C_{35}H_{51}Cl_3N_4O_5 \cdot H_2O$: C, 57.41; H, 7.30; N, 7.65. Found: C, 57.70; H, 7.01; N, 7.67.

Removal of TCBOC from 7 (6). Freshly activated[21] zinc dust (0.98 g, 15.0 mmol) was added to **7** (0.181 g, 0.262 mmol) in distilled THF (5 mL) with stirring. Potassium dihydrogen phosphate (1.0 M, 1.0 mL) was added and the mixture stirred for 21 h. The solids were filtered and washed with THF and the filtrate concentrated. Ice-cold 5% NaOH (20 mL) was added to the residue, followed by extraction with $CHCl_3$ (3 × 25 mL). The organic phase was washed with water (20 mL), dried, and concentrated to give 0.135 g of the crude product. Column chromatography (8.0 g SiO_2, CH_3OH) produced 98 mg of **6** (77% yield): NMR δ 1.35–1.85 (m, 18 H), 2.3–2.67 (m, 6 H), 2.7–2.9 (m, 2 H), 3.17 (q, 2 H, J = 7), 3.36–3.6 (m, 4 H), 5.3–5.4 (br s, 1 H), 7.3–7.4 (m, 5 H).

Acylation of 6 (11). *p*-Toluoyl chloride (46.3 mg, 0.300 mmol) in CH_2Cl_2 (5 mL) was added to **6** (from **7**, 0.135 g, 0.276 mmol) and triethylamine (29.5 mg, 0.292 mmol) in CH_2Cl_2 (5 mL), and the solution was stirred for 1 day. The reaction was diluted with CH_2Cl_2 (20 mL) and washed with 5% $NaHCO_3$ (20 mL). After further extraction with CH_2Cl_2 (2 × 20 mL), the organic phase was washed with H_2O (20 mL), dried, and evaporated to give 0.18 g of oil. Purification by preparative-layer chromatography (4% EtOH/$CHCl_3$) gave 0.149 g of **11** for an 89% yield: NMR δ 1.2–2.0 (m, 17 H), 2.2–2.55 (m, 7 H), 2.95–3.65 (m, 10 H), 5.15 (br s, 1 H), 7.2–7.4 (m, 9 H), 8.5 (br s, 1 H). Anal. Calcd for $C_{32}H_{45}F_3N_4O_4$: C, 63.35; H, 7.48; N, 9.23. Found: C, 63.19; H, 7.55; N, 9.15.

Benzylamine and *N*-Benzylmethylamine with 5. Reagent **5** in benzene (0.34 M, 6.0 mL, 2.04 mmol) was added by syringe over 4 min to a rapidly stirred solution of benzylamine (0.23 g, 2.15 mmol) and *N*-benzylmethylamine (0.26 g, 2.15 mmol) in dry CH_2Cl_2 (50 mL) at 0 °C under N_2. The reaction was stirred for 21 h (0 °C to room temperature). After solvent removal, 1 N HCl (20 mL) was added and the mixture extracted with ether (3 × 20 mL). The combined organic phase was washed with brine (20 mL), dried, and concentrated to give 0.336 g of the product. Column chromatography (10 g SiO_2, 30% *n*-hexane/$CHCl_3$) combining all eluant beginning with column loading through the UV-active band gave 0.285 g of *N*-benzyltrifluoroacetamide (70% yield). Note: *N*-benzyl-*N*-methyltrifluoroacetamide elutes faster than *N*-benzyltrifluoroacetamide. NMR δ 4.53 (d, 2 H, J = 6), 6.6–7.53 (m, 6 H). A multiplet at δ 3.0 indicated less than 5% *N*-benzyl-*N*-methyltrifluoroacetamide.

Aniline and *N*-Methylaniline with 5. Reagent **5** in benzene (0.34 M, 6.0 mL, 2.04 mmol) was added by syringe over 3 min to a rapidly stirred solution of aniline (0.21 g, 2.25 mmol) and *N*-methylaniline (0.23 g, 2.15 mmol) in dry CH_2Cl_2 (25 mL) at 0 °C under N_2. The reaction was stirred for 15 h (0 °C to room temperature). After solvent removal, 1 N HCl (25 mL) was added and the mixture extracted with ether (3 × 25 mL). The organic phase was washed with brine (25 mL), dried, and concentrated to give 0.37 g of solid. Column chromatography (30.6 g SiO_2, 30% *n*-hexane/$CHCl_3$) combining all eluant beginning with column loading through the UV-active band furnished 0.328 g of *N*-phenyltrifluoroacetamide (85% yield). Note: *N*-methyl-*N*-phenyltrifluoroacetamide elutes faster than *N*-phenyltrifluoroacetamide. NMR δ 7.1–8.2 (m).

Aniline and *N*-Methylaniline with Trifluoroacetic Anhydride. Trifluoroacetic anhydride (0.40 g, 1.90 mmol) in dry CH_2Cl_2 (15 mL) was added dropwise over 15 min to a stirred solution of aniline (0.21 g, 2.25 mmol), *N*-methylaniline (0.23 g, 2.15 mmol), and triethylamine (0.3 mL, 2.15 mmol) in CH_2Cl_2 (15 mL). After being stirred for 12 h, the reaction was worked up following the prior procedure to give 0.22 g of product. Column chromatography (10.6 g SiO_2, 30% *n*-hexane/$CHCl_3$) gave 0.155 g of product, which contained a 3:1 mixture of *N*-phenyltrifluoroacetamide and *N*-methyl-*N*-phenyltrifluoroacetamide. NMR δ 3.37 (s, *N*-methyl).

Acknowledgment. Financial support was provided by the National Institutes of Health, Grant CA-37606.

Registry No. **2**, 90914-14-0; **3**, 114491-93-9; **4**, 114491-94-0; **5**, 5672-89-9; **6**, 114491-95-1; **7**, 114491-96-2; **8a**, 114491-97-3; **8b**, 114504-97-1; **9a**, 114491-98-4; **9b**, 114504-98-2; **10a**, 114491-99-5; **10b**, 114492-00-1; **11**, 114492-01-2; spermine, 71-44-3; acrylonitrile, 107-13-1; benzylamine, 100-46-9; *N*-benzylmethylamine, 103-67-3; *N*-benzyltrifluoroacetamide, 7387-69-1; *N*-benzyl-*N*-methyltrifluoroacetamide, 68464-36-8; aniline, 62-53-3; *N*-methylaniline, 100-61-8; *N*-phenyltrifluoroacetamide, 404-24-0; *N*-methyl-*N*-phenyltrifluoroacetamide, 345-81-3.

(21) *Organic Syntheses*; Wiley: New York, 1955; Collect. Vol. III, pp 410–412.

Direct Conversion of Silyl Ethers into Alkyl Bromides with Boron Tribromide

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Direct conversion of silyl ethers such as *tert*-butyldimethylsilyl (TBDMS)[1] and *tert*-butyldiphenylsilyl (TBDPS)[2] ethers into synthetically useful functional groups without deprotection seems to be very important for further manipulation in the synthesis of complex molecules. It has been reported that silyl ethers have been directly converted into the corresponding acetates with acetic anhydride/ferric chloride[3] and an acid chloride/zinc chloride.[4] Furthermore, direct conversion of silyl ethers into alkyl bromides with triphenylphosphine dibromide[5] and triphenylphosphine/carbon tetrachloride[6] has been recently reported during our studies on the same subject.

We have found that boron tribromide in methylene chloride is very effective for the conversion of TBDMS and TBDPS ethers into the corresponding bromides in high yields (eq 1). It has been known that TBDMS ethers are

$$ROR' + BBr_3 \xrightarrow[CH_2Cl_2]{\text{room temperature}} RBr \qquad (1)$$

R′ = TBDMS, TBDPS

deprotected to the alcohols with boron trifluoride etherate[7] and dimethylboron bromide.[8] Furthermore, it has been

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(4) Kim, S.; Lee, W. *J. Synth. Commun.* **1986**, *16*, 659.
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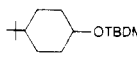
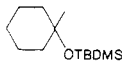
Table I. Conversion of Silyl Ethers into Alkyl Bromides with Boron Tribromide

silyl ether	time	yield, %[a]	bp, °C (mmHg)[b,c]	lit. bp, °C (mmHg)
$C_6H_5CH_2CH_2OTBDMS$	4 h	89	72–75 (5)	92 (11)[f]
$C_6H_5CH_2CH_2OTBDPS$	7 h	88		
$CH_3(CH_2)_8OTBDMS$	4 h	92	63–67 (1)	88 (4)[f]
$CH_3(CH_2)_5CH(OTBDMS)CH_3$	10 min	93	69–71 (14)	72 (14)[f]
$CH_3(CH_2)_5CH(OTBDPS)CH_3$	10 min	91		
$CH_3(CH_2)_2CH{=}CHCH_2OTBDMS$[d]	10 min	85[d]	49–54 (20)	67–72 (44)[g]
$CH_3(CH_2)_2CH{=}CHCH_2OTBDPS$[d]	10 min	81[d]		
$CH_2{=}CHCH(OTBDMS)(CH_2)_4CH_3$	10 min	84[e]	60–63 (6.1)	80–85 (14)[h]
$C_6H_5CH_2OTBDMS$	10 min	90	55–58 (3)	201 (760)[f]
$C_6H_5CH(OTBDMS)CH_3$	10 min	93	57–60 (3)	85 (13)[f]
$C_6H_5CH(OTBDPS)CH_3$	10 min	94		
	10 min	90[j]	71–75 (1.5)	104–110 (14)[j]
	10 min	85	49–53 (20)	156–160 (760)[f]

[a] Isolated yields. [b] Reported boiling points are those obtained during distillation with Kugelrohr apparatus. [c] Satisfactory NMR data were obtained for all compounds. [d] Trans double bond. [e] 1-Bromo-*trans*-2-octene. [f] *Handbook of Data on Organic Compounds*; Weast, R. C.; Astle, M. J., Eds.; CRC: Boca Raton, FL 1985. [g] Yoshioka, T. *Chem. Abstr.* **1958**, *52*, 11740h. [h] Myaghova, G. I.; Novozhilov, A. V.; Bainova, M. S.; Bazilevskaya, G. I.; Yakimenko, S. M.; Preobrazhenskii, N. A. *Chem. Abstr.* **1969**, *71*, 123453y. [i] 93:7 (cis:trans) by GLC analysis. [j] 65:35 (cis:trans) by ^1H NMR analysis. Eliel, E. L.; Martin, R. J. L. *J. Am. Chem. Soc.* **1968**, *90*, 689.

found that the reaction of phenethyl *tert*-butyldimethylsilyl ether with an equimolar amount of boron trichloride in methylene chloride gives a 68:21 mixture of phenethyl chloride and phenethyl alcohol at room temperature in 24 h, while the reaction with boron tribromide gave 89% of phenethyl bromide without the formation of phenethyl alcohol in 4 h under the same conditions.

Table I summarizes some experimental results and illustrates the efficiency and the applicability of the present method. The reaction of TBDMS ethers with an equimolar amount of boron tribromide in methylene chloride proceeded smoothly at room temperature, yielding the corresponding bromides in high yields after simple aqueous workup. The reaction rate depends very much on the nature of TBDMS ethers. Tertiary, secondary, benzylic, and allylic TBDMS ethers reacted much more rapidly than primary alkyl TBDMS ethers. Furthermore, it is noteworthy that primary and secondary allylic TBDMS ethers were converted into the primary allylic bromides, in which isomerization of a double bond in a secondary allylic TBDMS ether occurred exclusively. Similar results were generally obtained with TBDPS ethers under the similar conditions.

The mechanism and stereochemistry of the present method were briefly studied with optically active (−)-2-octyl *tert*-butyldimethylsilyl ether. The reaction with boron tribromide in methylene chloride at room temperature for 10 min afforded 2-bromooctane in 90% yield with 71% retention of configuration. Thus, in the case of secondary alkyl TBDMS ethers, this data supports a dual mechanism involving complex formation from TBDMS ether and boron tribromide followed by internal nucleophilic displacement (S_Ni) along with S_N1.[9] Finally, it is of interest to note that conversion of (−)-2-octyl *tert*-butyldimethylsilyl ether into 2-bromooctane with triphenylphosphine dibromide proceeded with 97% inversion of configuration.

Experimental Section

NMR spectra were recorded with Varian T-60A and FT-80A spectrometers and reported boiling points are those observed during distillation with a Kugelrohr apparatus. GLC analysis was performed on a Varian 3700 chromatograph using a 10% Carbowax 20M column and optical rotations were measured with an automatic polarimeter Autopol III. Analytical thin-layer chromatography was performed on precoated silica gel plates (0.25 mm, 60F-254, E. Merck), and silica gel (0.063–0.020 mm, E. Merck) was used for column chromatography.

All the reagents purchased from Aldrich were used without further purification. TBDMS and TBDPS ethers were generally prepared by the known procedures,[1,2] and tertiary alkyl TBDMS ethers were prepared by using TBDMS triflate.[10]

General Procedure for the Conversion of TBDMS and TBDPS Ethers into Alkyl Bromides with Boron Tribromide. To a solution of phenethyl *tert*-butyldimethylsilyl ether (473 mg, 2.0 mmol) in methylene chloride (5 mL) in an ice bath was added a solution of boron tribromide (1.0 M, 2.2 mL) in methylene chloride, and then the ice bath was removed. The reaction mixture was stirred at room temperature, and the progress of the reaction was followed by TLC analysis. After being stirred at room temperature for 4 h, saturated $NaHCO_3$ solution (5 mL) and ether (30 mL) were added. The organic layer was washed with saturated $NaHCO_3$ solution (30 mL) and brine (30 mL), dried over anhydrous $MgSO_4$, and evaporated to dryness under reduced pressure. The crude product was purified by distillation with a Kugelrohr apparatus to afford phenethyl bromide (329 mg, 89%).

Conversion of (−)-2-Octyl *tert*-Butyldimethylsilyl Ether into (−)-2-Bromooctane with Boron Tribromide. (−)-2-Octyl *tert*-butyldimethylsilyl ether ($[\alpha]^{20}_D$ −13.27° (neat), 91% optical purity) was prepared from (−)-2-octanol ($[\alpha]^{20}_D$ −9.0 (neat), 91% optical purity)[11] by the known procedure.[3] To a solution of (−)-2-octyl *tert*-butyldimethylsilyl ether (615 mg, 2.0 mmol) in methylene chloride (5 mL) in an ice bath was added a solution of boron tribromide (1.0 M, 2.2 mL) in methylene chloride, and the ice bath was removed. The reaction mixture was stirred at room temperature for 10 min and then treated with saturated $NaHCO_3$ solution (5 mL) and ether (30 mL). The organic layer was washed with saturated $NaHCO_3$ (30 mL), dried over anhydrous $MgSO_4$, and evaporated to dryness. Kugelrohr distillation gave 2-bromooctane (348 mg, 90%) having $[\alpha]^{20}_D$ −17.01° (neat) which corresponds to 71% retention of configuration.[12] Furthermore, the reaction of (−)-2-octyl *tert*-butyldimethylsilyl ether with triphenylphosphine dibromide was carried out in methylene

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(9) Although the reaction may proceed to some extent via S_N2 attack by bromide, it is believed that S_N1 is much more favored than S_N2 because secondary alkyl TBDMS ethers reacted much more rapidly than primary alkyl TBDMS ethers.

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(11) (*S*)-(−)-2-Octanol was purchased from Aldrich ($[\alpha]^{20}_D$ −9.0° (neat)). The maximum reported rotation is $[\alpha]^{20}_D$ −9.9° (neat) (*The Merck Index*; Merck & Co. Inc.: Rahway, NJ, 1976).

(12) The maximum reported rotation for (−)-2-bromooctane is $[\alpha]^{20}_D$ −44.91° (neat) (*The Merck Index*; Merck & Co. Inc.: Rahway, NJ, 1976). The corrected value based on 91% ee for (−)-2-octyl *tert*-butyldimethylsilyl ether is $[\alpha]^{20}_D$ −18.69°.

chloride by the known procedure,[5] and 2-bromooctane having $[\alpha]^{20}_D$ +38.71° (neat) was obtained in 92% yield. This represents 97% inversion.[12]

Acknowledgment. We thank the Korea Advanced Institute of Science and Technology for financial support.

Registry No. $C_6H_5(CH_2)_2OTBDMS$, 78926-09-7; $C_6H_5(CH_2)_2OTBDPS$, 105966-41-4; $CH_3(CH_2)_8OTBDMS$, 71733-81-8; (–)-$CH_3(CH_2)_5CH(OTBDMS)CH_3$, 114127-43-4; $CH_3(CH_2)_5CH(OTBDPS)CH_3$, 105966-42-5; (*E*)-$CH_3(CH_2)_2CH{=}CHCH_2OTBDMS$, 113997-32-3; (*E*)-$CH_3(CH_2)_2CH{=}CHCH_2OTBDPS$, 11997-33-4; $CH_2{=}CHCH(OTBDMS)(CH_2)_4CH_3$, 107220-03-1; $C_6H_5CH_2OTBDMS$, 53172-91-1; $C_6H_5CH(OTBDMS)CH_3$, 92976-56-2; $C_6H_5CH(OTBDPS)CH_3$, 105966-44-7; $C_6H_5(CH_2)_2Br$, 103-63-9; $CH_3(CH_2)_8Br$, 693-58-3; (*E*)-$CH_3(CH_2)_2CH{=}CHCH_2Br$, 73881-10-4; $C_6H_5CH_2Br$, 100-39-0; $C_6H_5CHBrCH_3$, 585-71-7; (–)-2-bromooctane, 5978-55-2; (+)-2-bromooctane, 1191-24-8; *cis*-1-*tert*-butyl-4-[(*tert*-butyldimethylsilyl)oxy]cyclohexane, 71009-12-6; *trans*-1-*tert*-butyl-4-[(*tert*-butyldimethylsilyl)oxy]cyclohexane, 71009-16-0; 1-methyl-1-[(*tert*-butyldimethylsilyl)oxy]cyclohexane, 76358-83-3; 1-bromo-*trans*-2-octene, 56318-83-3; *cis*-1-bromo-4-*tert*-butylcyclohexane, 5009-36-9; *trans*-1-bromo-4-*tert*-butylcyclohexane, 5009-37-0; 1-bromo-1-methylcyclohexane, 931-77-1.

Synthesis of Monocyclic β-Lactams by the Photolytic Reaction of Chromium Carbene Complexes with *s*-1,3,5-Triazines

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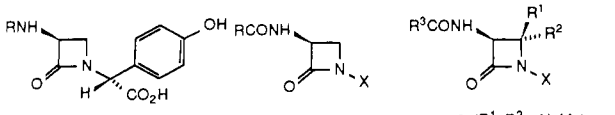
Introduction

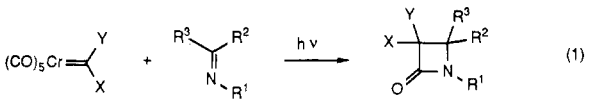
Following the discovery of naturally occurring monocyclic β-lactams such as the nocardicins **1** and the monobactams **2**, many synthetic analogues having more desirable biological properties have been developed.[1] These include monobactams alkylated at the 4–position (**3**).[2]



1 **2** (X = SO_3^-) **3** (R^1, R^2 = H, Me) (X = SO_3^-, OSO_3^-)

Nocardicins have been synthesized by the reaction of acid chlorides with a Schiff base,[3] by Ugi four-component condensation chemistry,[4] by β-halopropionamide ring closure, ring expansion, or azetidine carboxylate oxidative decarboxylation,[5] by Pd(0)-catalyzed carbonylation of α-bromoallylamine,[6] and by cyclization of the hydroxamic acid of *N*-boc-L-serine.[7] Monobactams have been synthesized from 6-APA,[8] and by cyclization of β-hydroxyacyl sulfamates[9] or of β-hydroxy hydroxamic acid derivatives.[10]

A new approach to β-lactams involving the photolytic reaction of chromium carbene complexes with imines has recently been developed in these laboratories (eq 1).[11] The application of this approach to the synthesis of monocyclic and bicyclic β-lactams is described below.



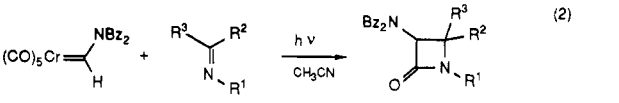
(1)

X = R; Y = OR', NR_2'

X = H; Y = NR_2'

Results and Discussion

The process described in eq 1 is very general, in that a wide variety of substituted imines are cleanly converted to β-lactams by this chemistry. A potential problem with its application to monobactam syntheses results from existence of the requisite aldehyde imines predominantly as cyclic trimers—hexahydro-1,3,5-triazines—rather than as imine monomers. One of the few formaldehyde imines that is monomeric is *N*-methylidene *tert*-butylamine, and this substrate converted cleanly to β-lactam **2a** upon irradiation in the presence of carbene complex **1** (eq 2). The monomeric *N*-benzyl imine of acetone also was converted to the corresponding β-lactam **2b**, in modest yield (eq 2).



(2)

1

2a R^1 = t-Bu; R^2, R^3 = H 72%

2b R^1 = Bz; R^2, R^3 = Me 56%

(Bz = $PhCH_2$)

Cyclic trimers of formaldehyde imines (eq 3) and carbocyclic imines (eq 4) also underwent efficient reaction with carbene complex **1**, producing azetidinone **3a,b**, carbapenam **4a**, and carbacepham **4b** derivatives in good yield.

Nocardicin precursors have recently been synthesized by the reactions of ketenes with the chiral imine produced in situ from the BF_3·etherate assisted cleavage of chiral, optically active trimer **5**. These reactions went in good yield and gave a 3:1 mixture of diastereoisomers, thus showing modest asymmetric induction.[12] In an attempt to use a similar process, chiral, optically active trimer **5** was photolyzed with carbene complex **1** (eq 5). Although

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