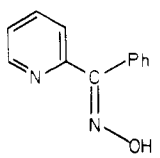
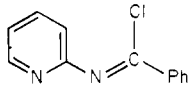
provided by the unequivocal synthesis of *N*-(2-pyridyl)-benzimidoyl cyanide. Treatment of *syn*-phenyl 2-pyridyl ketoxime (8) with thionyl chloride gave the hydrochloride



8



9

of the imidoyl chloride[5] **9**. Initial attempts to convert the hydrochloride of **9** to **3** with triethylamine and sodium cyanide under a variety of conditions failed. Zinc cyanide which has been recently used to synthesize α-cyano enamines from imidoyl chlorides[6] was also ineffective. However, when the hydrochloride of **9** was refluxed in dioxane with triethylamine and cuprous cyanide, the required nitrile **3** was obtained. This imidoyl cyanide was identical with the product obtained by the reaction of 3-nitroso-2-phenylimidazo[1,2-*a*]pyridine with triethyl phosphite.

Experimental Section

All melting points are uncorrected. IR spectra were recorded on a Perkin-Elmer 197 spectrophotometer. Proton magnetic resonance spectra were obtained on a Varian EM390 instrument or in the case of the decoupling experiments on a Perkin-Elmer R34 instrument. Mass spectral data was taken on an EMI MS9-02 spectrometer. All flash chromatography was done by using Merck 60 silica (230–400 mesh).

Reaction of 3-Nitroso-2-phenylimidazo[1,2-*a*]pyridine with Triethyl Phosphite. The reaction was carried out as described by Adhikary et al.[1] The crude product was flash chromatographed with EtOAc/toluene (3:20) as the solvent. Recrystallization from hexane gave a 63% yield of **3** as yellow prisms: mp 77–78 °C; mass spectrum, *m/e* 207 (M^+), 206 (M – H), 181 (M– CN), 179 (206 – HCN); ^1H NMR ($CDCl_3$; see structure **3** for numbering) δ 8.55 (dd, H_1), 8.25 (m, H_5 or H_9), 8.20 (m, H_5 or H_9), 7.80 (td, H_3), 7.50 (m, H_6, H_7 and H_8), 7.20 (m, H_2 and H_4). Irradiation at the H_1 proton caused the H_3 proton resonance to collapse to a triplet and the H_2 and H_4 multiplet to simplify whereas irradiation at the H_3 proton prompted the H_1 proton resonance to collapse to a doublet and the H_2 and H_4 multiplet to simplify.

1-[*N*-(2-Pyridyl)benzimidoyl]morpholine (5). A solution of **3** (1.035 g, 0.005 mol) and morpholine (0.5 mL, 0.00625 mol) in toluene (10 mL) was stirred at room temperature for 16 h. The solvent was evaporated under vacuum, and the residue was flash chromatographed with $CHCl_3$/MeOH (20:1) as the solvent. The resultant oil slowly solidified and was recrystallized from hexane/anhydrous Et_2O to afford 0.98 g (75%) of **5**: colorless prisms; mp 81–82 °C; ^1H NMR ($CDCl_3$) δ 8.15 (dd, 1 H, pyridine C(6) proton), 7.20 (m, 6 H, aryl protons and pyridine C(4) proton), 6.60 (m, 1 H, pyridine C(5) proton), 6.30 (d, 1 H, pyridine C(3) proton), 3.75 (m, 4 H, O–CH_2), 3.4 (m, 4 H, CH_2–N). Anal. Calcd for $C_{16}H_{17}N_3O$: C, 71.9; H, 6.4; N, 15.7. Found: C, 71.5; H, 6.1; N, 15.5.

Reaction of the Triethyl Phosphite Product 3 with Sodium Hydroxide in Methanol. The triethyl phosphite reaction product **3** (1.035 g, 0.005 mol) was added to a solution of sodium hydroxide (0.2 g, 0.005 mol) in 20 mL of H_2O/MeOH (1:1), and the resultant solution was stirred for 1 h at room temperature. The methanol was removed under vacuum and the residue extracted with toluene. The organic extract was dried and on evaporation under vacuum gave a sticky solid, which was flash chromatographed with toluene/EtOAc (4:1) as the solvent. The chromatography gave, in the order of elution, 2-(benzoylamino)pyridine [0.42 g; mp 82 °C (lit.[7] mp 82–83 °C)] and the imino ether **6**: 0.30 g; colorless oil; ^1H NMR ($CDCl_3$) δ 8.35 (dd, 1 H, pyridine C(6) proton), 7.35 (m, 6 H, aryl protons and pyridine C(4) proton), 6.90 (m, 1 H, pyridine C(5) proton), 6.55 (d, 1 H, pyridine C(3) proton), 4.05 (s, 3 H, OMe); mass spectrum, *m/e* 211 (M^+).

3-Amino-2-phenylimidazo[1,2-*a*]pyridine (7). Sodium borohydride (0.095 g, 0.005 mol) was added gradually to an ice-cooled, well-stirred solution of the triethyl phosphite reaction product **3** (1.035 g, 0.005 mol) in absolute ethanol (10 mL). After the addition the reaction mixture was stirred for 1 h at room temperature. The solvent was evaporated under vacuum and the residue extracted with 1 N hydrochloric acid (30 mL). The acid extract was made alkaline by utilizing 2 N sodium hydroxide solution, and the amine was extracted with ethyl acetate. The organic phase was separated and dried over $MgSO_4$, and the solvent was removed under vacuum to give crude **7**. Recrystallization from absolute EtOH/hexane furnished **7**: 0.57 g (55%); colorless prisms; mp 211 °C (lit.[1] mp 212–214 °C).

***N*-(2-Pyridyl)benzimidoyl Cyanide (3).** Triethylamine (0.88 g, 0.0088 mol) was added to a solution of the hydrochloride of **9**[5] (2.0 g, 0.0008 mol) in dry dioxane (15 mL), and the solution was stirred at room temperature for 30 min. Cuprous cyanide (2.2 g, 0.025 mol) was added, and the reaction mixture was refluxed for 2 h. The solvent was evaporated under vacuum and the residue extracted with three 50-mL portions of ether. The combined extracts were dried over $MgSO_4$ and evaporated. The residual gum was flash chromatographed with toluene/EtOAc (4:1) as the solvent to give, after recrystallization from hexane, 0.48 g (30%) of **3**, mp 77–78 °C. Anal. Calcd for $C_{13}H_9N_3$: C, 75.4; H, 4.3; N, 20.3. Found: C, 75.3; H, 4.2; N, 19.9.

Registry No. **2**, 3672-37-5; **3**, 82093-41-2; **5**, 82093-42-3; **6**, 82093-43-4; **7**, 3999-29-9; **8**, 14178-31-5; **9**·HCl, 82093-44-5; $P(OEt)_3$, 122-52-1; morpholine, 110-91-8; 2-(benzoylamino)pyridine, 4589-12-2.

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(6) Toye, J.; Ghosez, L. *J. Am. Chem. Soc.* **1975**, *97*, 2276.
(7) Huntress, E. H.; Walter, H. C. *J. Org. Chem.* **1948**, *13*, 735.

Preparation of 3-*C*-Methylene Sugars by Peterson Olefination

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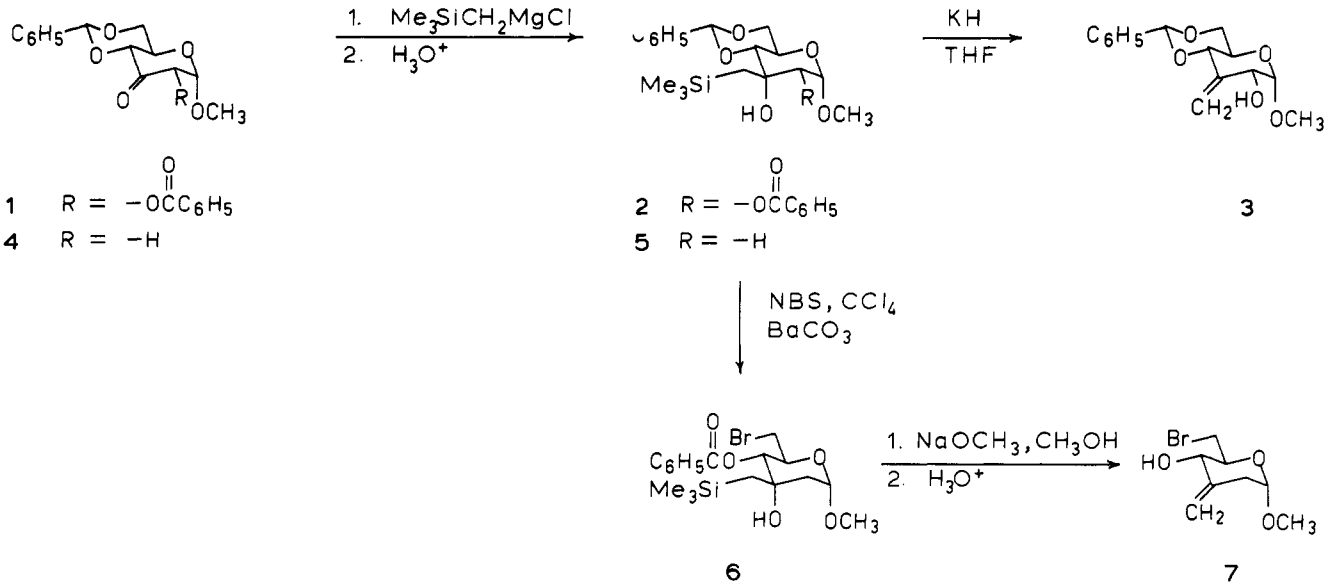
Olefination of glycosides of 3-keto sugars is complicated by the ease with which these materials undergo elimination of the anomeric alkoxy group under the usual conditions of the Wittig reaction with unstabilized ylides. A careful study of the conversion of methyl 2-*O*-benzoyl-4,6-*O*-benzylidene-α-D-*ribo*-hexopyranosid-3-ulose (**1**) to methyl 4,6-*O*-benzylidene-3-deoxy-3-*C*-methylene-α-D-*ribo*-hexopyranoside (**3**) has been described[1] which testifies to the difficulty of effecting this seemingly straightforward transformation. An interest in branched-chain amino sugars required unsaturated carbohydrates as their potential synthetic precursors, and our experience with [(trimethylsilyl)methyl]magnesium reagents[2] encouraged us to believe that the Peterson olefination sequence[3] might be a superior method for the preparation of exocyclic methylene sugars. The present work, summarized in Scheme I, presents evidence that this is indeed the case.

Addition of the Grignard reagent from (chloromethyl)trimethylsilane to **1** gave methyl 2-*O*-benzoyl-4,6-*O*-benzylidene-3-[(trimethylsilyl)methyl]-α-D-allopyranoside (**2**, 90%).[4] Elimination of trimethylsiloxide

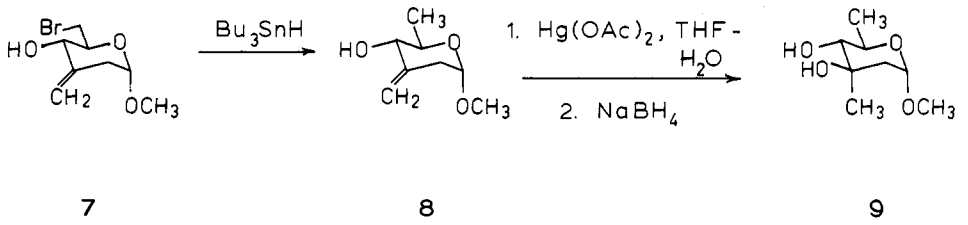
(1) Iley, D. E.; Tam, S. Y.; Fraser-Reid, B. *Carbohydr. Res.* **1977**, *55*, 193–204.
(2) Carey, F. A.; Toler, J. R. *J. Org. Chem.* **1976**, *41*, 1966–1971.
(3) (a) Peterson, D. J. *J. Org. Chem.* **1968**, *33*, 780–784. (b) Chan, T. H.; Chang, E.; Vinokur, E. *Tetrahedron Lett.* **1970**, 1137–1140.

0022-3263/82/1947-3548$01.25/0

Scheme I



Scheme II



and debenzoylation occurred on treatment with potassium hydride in refluxing tetrahydrofuran to give the desired **3** in 58% yield. For its efficiency and simplicity, the Peterson olefination is recommended as the method of choice for this and analogous conversions.

Additionally, we have found that the [(trimethylsilyl)methyl]carbinol function can serve as a protected methylene group allowing transformations to be carried out elsewhere in the molecule. When methyl 4,6-*O*-benzylidene-2-deoxy-α-D-*erythro*-hexopyranosid-3-ulose (**4**) was treated with [(trimethylsilyl)methyl]magnesium chloride, the crystalline β-hydroxy silane **5** was isolated in 87% yield. Photochemical bromination of **5** with *N*-bromosuccinimide afforded bromomethyl benzoate ester **6** (75%) with the [(trimethylsilyl)methyl]carbinol function remaining intact.[5] Base-catalyzed debenzoylation accompanied elimination of Me_3SiO^- from **6** and produced methyl 6-bromo-3-*C*-methylene-2,3,6-trideoxy-α-D-*erythro*-hexopyranoside (**7**, 95%).

One use of the highly functionalized unsaturated sugar **7** is shown in Scheme II and serves to illustrate the value of these transformations. Reduction of **7** with tri-*n*-butyltin hydride produced the 6-deoxy derivative **8** which on oxymercuration–demercuration gave methyl α-D-evermicoside (**9**).[6,7]

(4) Equatorial addition of organometallic reagents to **1** is usually highly stereoselective. For a summary, see: Yoshimura, J.; Sato, K.; Kobayashi, K.; Shin, C. *Bull. Chem. Soc. Jpn.* **1973**, *46*, 1515–1519.

(5) For the conversion of 4,6-*O*-benzylidene derivatives of carbohydrates to bromomethyl benzoate esters, see: Hannessian, S.; Plessas, N. R. *J. Org. Chem.* **1969**, *34*, 1035–1044.

(6) For stereoselective oxymercuration in a related system, see: Williams, E. H.; Szarek, W. A.; Jones, J. K. N. *Can. J. Chem.* **1969**, *47*, 4467–4471.

(7) For earlier syntheses of D-evermicose, see: (a) Funabashi, M.; Hong, D.; Kodama, H.; Yoshimura, J. *Carbohydr. Res.* **1978**, *67*, 139–145. (b) Dyong, I.; Glittenberg, D. *Chem. Ber.* **1977**, *110*, 2721–2728.

Experimental Section

Nuclear magnetic resonance (NMR) spectra were recorded on Varian EM-390 and Nicolet Magnetics Corporation NT-360/Oxford instruments in $CDCl_3$ and chemical shifts are reported in parts per million (δ) from internal tetramethylsilane. Melting points were measured on a Thomas-Hoover apparatus and are uncorrected. Elemental analyses were performed by Atlantic Microlabs, Atlanta, GA. Optical rotations were measured in chloroform solution, using a Perkin-Elmer 141 polarimeter.

Methyl 4,6-*O*-Benzylidene-3-deoxy-3-*C*-methylene-α-D-*ribo*-hexopyranoside (3). Magnesium turnings (2.57 g, 106 mmol) and a magnetic stir bar were placed in a 1-L, three-necked, round-bottom flask equipped with condenser, dry ice condenser, and equilibrating side arm addition funnel. Rubber septums were attached, and the system was flushed with argon and flamed dry. Argon was passed through the condenser, exiting to a bubbler via dry ice condenser, for the duration of the experiment. Anhydrous ether (75 mL) and (bromomethyl)trimethylsilane (0.841 g, 5.0 mmol) were introduced. (Chloromethyl)trimethylsilane (14.2 g, 116 mmol) in ether (50 mL) was added dropwise at a rate sufficient to maintain reflux and stirred at reflux for an additional hour. The apparatus was cooled to room temperature and a solution of 6.33 g (16.5 mmol) of keto sugar **1**[8] in 400 mL of warm toluene was added dropwise. The solution was stirred for 3 h, quenched with saturated ammonium chloride solution and extracted with 1 L of ether. After drying ($MgSO_4$), the ether was evaporated to leave 8.85 g (90%) of crude **2** as a syrup: ^1H NMR ($CDCl_3$) δ 8.3–8.0 (m, 2 H, arom), 7.1–7.6 (m, 8 H, arom), 7.58 (s, 1 H, C_6H_5CH), 4.88 and 5.10 (AB q, 2 H, J = 6 Hz, H-1, H-2), 3.5–4.5 (m, 4 H, H-4, H-5, H-6, H-6'), 3.40 (s, 4 H, OCH_3, OH), 1.20 and 1.37 (AB q, 2 H, J = 15 Hz, $SiCH_2$), 0.10 (s, 9 H, $SiCH_3$).

The crude **2** was dissolved in 250 mL of anhydrous tetrahydrofuran and carefully added to a suspension of 8.5 g (205 mmol) of potassium hydride in 225 mL of tetrahydrofuran. A reflux condenser was attached, and the reaction mixture was heated to reflux for 4 h under argon. The opaque brown liquid was poured slowly into a saturated ammonium chloride solution

(8) Carey, F. A.; Hodgson, K. O. *Carbohydr. Res.* **1970**, *12*, 463–465.

(300 mL) overlaid with ether (500 mL), and the layers were separated. The aqueous layer was extracted twice with ether, and the combined extracts were evaporated to leave 3.9 g of crude **3**. Recrystallization from dichloromethane–hexane (100 mL/250 mL) gave 2.71 g (58%) of **3** in two crops: mp 194.5–195 °C and mp 188–189 °C; $[\alpha]^{20}_D$ +145° (lit.[1] mp 192.5–194 °C and $[\alpha]_D^{23}$ +159°). The ^{1}H NMR spectrum was identical with that reported.

Methyl 4,6-*O*-Benzylidene-2-deoxy-3-*C*-[(trimethylsilyl)methyl]-α-D-*ribo*-hexopyranoside (5). In the same manner as above, 8.2 g (31 mmol) of ulose **4**[9] was allowed to react with the Grignard reagent derived from 92 mmol of (chloromethyl)trimethylsilane. Recrystallization of the crude product from pentane gave 9.48 g (87%) of **5**: mp 85–86 °C; $[\alpha]_D^{19}$ +68.6°; ^{1}H NMR ($CDCl_3$) δ 7.26–7.60 (m, 5 H, arom), 5.53 (s, 1 H, C_6H_5CH), 4.73 (d, J_{ax-eq} = 4 Hz, 1 H, $CHOCH_3$), 3.50–4.43 (m, 4 H, H-4, H-5, H-6, H-6′), 3.38 (s, 3 H, OCH_3), 3.26 (m, 1 H, OH), 2.20 (dd, J_{gem} = 13 Hz, $J_{1,2} \leq$ 1 Hz, 1 H, H-2_{eq}), 1.83 (dd, J_{gem} = 13 Hz, $J_{1,2}$ = 4 Hz, H-2_{ax}), 0.80 and 1.36 (AB q, J_{gem} = 15 Hz, 1 H, $SiCH_2$), 0.10 (s, 9 H, CH_3Si). Anal. Calcd for $C_{18}H_{18}O_5Si$: C, 61.33; H, 8.01. Found: C, 61.17; H, 8.07.

Methyl 4-*O*-Benzoyl-6-bromo-6-deoxy-3-*C*-[(trimethylsilyl)methyl]-α-D-*ribo*-hexopyranoside (6). *N*-Bromosuccinimide (3.5 g, 15.6 mmol), barium carbonate (0.91 g), **5** (5.44 g, 15.4 mmol), and 380 mL of carbon tetrachloride were placed in a 500-mL, round-bottom flask equipped with a magnetic stirrer and a condenser. This mixture was heated (oil bath, 95 °C) and simultaneously irradiated with a 200-W flood lamp. The combination provided a very gentle reflux. When the solution began to turn red, the lamp was turned off. The color intensified and then began to disappear after 0.5 h. The lamp was again turned on to complete the reaction. After the solution became yellow, it was filtered (while still hot) and the succinimide was washed well with hot carbon tetrachloride. The solution was extracted with half-saturated sodium bisulfite (100 mL), saturated sodium bicarbonate (2 × 100 mL), and brine (100 mL) and dried (Na_2SO_4). Thin-layer chromatography (silica/chloroform) showed one spot (R_f 0.55). Concentration appeared to give rise to some decomposition overnight, but after column chromatography (230–400-mesh silica, 3:1 chloroform/pentane), **6** (4.81 g, 74%) was isolated as a syrup: ^{1}H NMR ($CDCl_3$) δ 8.06–8.20 (m, 2 H, arom), 7.33–7.73 (m, 3 H, arom), 4.83–5.00 (m, 2 H, H-4 and $CHOCH_3$), 4.06–4.40 (m, 1 H, H-5), 3.86 (br s, 1 H, OH), 3.46 (s, 3 H, OCH_3), 3.42 (2 H, CH_2Br), 2.26 (dd, J_{gem} = 14 Hz, $J_{eq-eq} \leq$ 1 Hz, 1 H, H-2_{eq}), 1.87 (dd, J_{gem} = 14 Hz, J_{ax-eq} = 4 Hz, 1 H, H-2_{ax}), 0.73 and 1.13 (AB q, J_{gem} = 15 Hz, 2 H, $SiCH_2$), 0.07 (s, 9 H, $SiCH_3$). Anal. Calcd for $C_{18}H_{27}BrO_5Si$: C, 50.11; H, 6.31. Found: C, 49.02; H, 6.19.

Methyl 6-Bromo-3-*C*-methylene-2,3,6-trideoxy-α-D-*erythro*-hexopyranoside (7). The product just described **6** (2.98 g, 6.9 mmol) and freshly distilled anhydrous methanol (120 mL) were placed in a 200-mL, round-bottom flask under argon. Sodium (200 mg) in methanol (35 mL) was added portionwise, and the mixture was stirred for 14.5 h at room temperature. The loss of benzoate was monitored by TLC (silica, 2% methanol in chloroform). When starting material had disappeared, a large excess of analytical grade Dowex 50W-X8 cation-exchange resin that had been washed several times with methanol was added to the reaction mixture and the mixture stirred for 10 h at room temperature. The reaction was monitored by TLC (silica, chloroform). The resin was removed by filtration, and the methanol solution was concentrated to a small volume (20 mL). This was transferred to a separatory funnel with dichloromethane (75 mL) and extracted with saturated sodium bicarbonate (2 × 50 mL), water (50 mL), and brine (50 mL). It was dried ($MgSO_4/K_2CO_3$), and evaporated. Flash column chromatography (silica, 1% methanol in chloroform) gave **7** as a syrup (1.56 g, 95%): ^{1}H NMR ($CDCl_3$) δ 5.13 (s, 1 H, vinyl), 4.95 (s, 1 H, vinyl), 4.80 (t, J = 3 Hz, 1 H, $CHOCH_3$), 3.90–4.20 (m, 1 H, H-4), 3.40–3.90 (m, 3 H, H-5, CH_2Br), 3.37 (s, 3 H, OCH_3), 2.51 (br s, 2 H, H-2_{ax}, H-2_{eq}), 2.00 (br d, 1 H, OH). Anal. Calcd for $C_8H_{13}BrO_3$: C, 40.53; H, 5.53. Found: C, 40.38; H, 5.60.

Methyl 3-*C*-Methylene-2,3,6-trideoxy-α-D-*erythro*-hexopyranoside (8). A solution of dry benzene (6.7 mL) containing 500 mg (2.11 mmol) of **7** and 1.0 g (4.2 mmol) of tri-*n*-butyltin hydride was refluxed for 3 h. A mercury vapor 200-W flood lamp was used as both a heat and light source. By the Jacobus workup,[10] aqueous potassium fluoride was added, the mixture was stirred for 3 h at room temperature, and insoluble material was removed by filtration. The layers were separated, the aqueous layer was extracted with ether (5 mL), and the organic layers were combined, dried ($MgSO_4$), filtered, and evaporated to yield crude **8**. Flash column chromatography (silica), using 1% methanol/chloroform, gave 326 mg (98%) of **8** as a colorless syrup: ^{1}H NMR ($CDCl_3$) δ 5.09 and 4.91 (2 br s, 2 H, vinyl H), 4.70 (br t, 1 H, H-1), 3.90–3.40 (m, 2 H, H-4, H-5), 3.31 (s, 3 H, OCH_3), 2.50 (br s, 2 H, H-2), 1.70 (br s, 1 H, OH), 1.32 (d, 3 H, J = 6 Hz, H-6); CI accurate mass measurement ($C_8H_{14}O_3 \cdot NH_4^+$) calcd m/e 176.128, found m/e 176.126.

Preparation of Methyl α-D-Evermicoside (9) by Oxymercuration of 8. A solution of **8** (100 mg, 0.63 mmol), mercuric acetate (203 mg, 0.70 mmol), tetrahydrofuran (1.5 mL), and water (1.5 mL) was stirred at room temperature for 1 h. The flask was cooled to 0 °C, and 2 N sodium hydroxide was added until the solution was strongly basic, maintaining the temperature at 0–5 °C. A solution of 4 N sodium borohydride in 2 N sodium hydroxide was added dropwise maintaining the temperature at 0–10 °C until the reaction was no longer exothermic. Ether was added, and the flask was placed in a refrigerator overnight. The solution was decanted, leaving the coagulated mercury behind. Transfer was completed with ether, and the layers were separated. The aqueous layer was extracted with ether, and the organic layers were combined, washed with brine, dried ($MgSO_4$), filtered through a Celite pad, and concentrated to give a colorless oil (27.3 mg, 25%) identified as methyl evermicoside (methyl 2,6-dideoxy-3-*C*-methyl-α-D-*arabino*-hexopyranoside, **9**) by comparison of its ^{1}H NMR spectrum at 360 MHz with that reported for its enantiomer at 270 MHz.[11] The two spectra were identical.

Registry No. **1**, 28642-65-1; **2**, 82198-67-2; **3**, 50827-20-8; **4**, 6752-49-4; **5**, 82190-42-9; **6**, 82190-43-0; **7**, 82190-44-1; **8**, 82190-45-2; **9**, 73712-09-1; (chloromethyl)trimethylsilane, 2344-80-1.

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Strain-Assisted α-Cleavage Reactions of Thioketones: Diphenylcyclopropenethione

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Photochemical α cleavage is characteristic of most alkanones[1] but is infrequently observed in thiocarbonyl systems.[2] Perhaps, because of the rarity of its occurrence, the photochemical Norrish type I α-cleavage process is one of the least studied excited-state transformations of thiocarbonyls. Nevertheless, it has been shown to occur in cyclobutanethiones.[3] Recently, we have studied the effect of strain on the α-cleavage process and, of course, cyclopropene derivatives were suitable substrates. Prompted by the desire to explore the possibility of elimination of carbon monosulfide upon α cleavage and intrigued by the

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