Structure Analysis and Refinement. The crystal structure was solved by direct methods using the MULTAN-76 program. The structure was refined by the full-matrix least-squares method for non-H atoms with anisotropic temperature factors and with isotropic temperature factors for H atoms, which were located in a difference map in the expected positions. The final R value was 0.051, R_w = 0.061. Final atomic positional and thermal parameters are listed in Tables II and III (supplementary material).

Acknowledgment. This work was financially supported by the Polish Academy of Sciences and Ministry of Education within the project CPBP-01-13 and RP-II-10.

Registry No. 5, 20570-22-3; (5,5-dimethyl-1,3-dioxan-2-yl)-trimethylammonium, 62999-89-7; isopropyl diphenylphosphinite, 1706-91-8.

Supplementary Material Available: Final atomic coordinates and temperature factors (Tables II and III), bond lengths and angles (Tables IV and V), torsion angles (Table VI), displacements of atoms from selected least-squares planes and angles between normals to planes (Table VII) for compound **5** (6 pages); a list of observed and calculated structure factors (Table VIII) for compound **5** (20 pages). Ordering information is given on any current masthead page.

Easy One-Step General Synthesis of Acylsilanes

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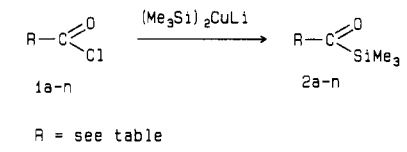
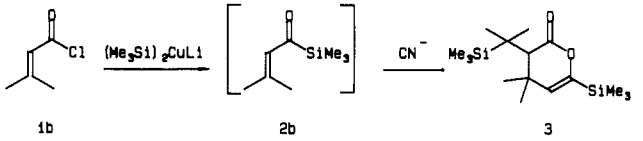
Acylsilane chemistry has seen an increasing interest in the recent years,[1] due to the emerging potentialities of these compounds as valuable building blocks for the synthesis of more complex molecules.[2] Acylsilanes in fact may undergo a number of synthetically useful transformations, such as Brook reactions,[3] oxidation to carboxylic acids,[4] fluoride-promoted conversion to aldehydes,[5] and catalyzed nucleophilic acylation.[6]

Scheme I



Scheme II



Since the first discovery of acylsilanes by A. G. Brook, several synthetic methods have been developed, but most of them suffer of lengthy procedures[7] or involve the use of expensive or not easily available starting materials.[8] Moreover, none of the present synthetic methods seem to be of a general nature, often being applied to the preparation of a specific series of acylsilanes. We report here a simple one-pot synthesis of acyltrimethylsilanes, through the reaction of (trimethylsilyl)cuprate with a variety of acyl chlorides, as depicted in Scheme I.

All the reactions were performed with (trimethylsilyl)-cuprate generated from (trimethylsilyl)lithium and CuCN, following Fleming's procedure.[9] The results are summarized in Table I.

The use of (trimethylsilyl)cuprate proved to be essential, in order to achieve a clean reaction, since treatment of (trimethylsilyl)lithium itself with several acyl chlorides led to complex reaction mixtures where only traces of the wanted compounds could be detected.

Reactions proceed smoothly and under mild conditions, usually affording, after the workup, the required acylsilane with a high degree of purity.

An outstanding feature of this new preparation of acylsilanes is its generality: as shown in Table I, this synthetic approach may be used with a wide variety of acyl halides, leading to a simple, direct synthesis of either aliphatic, aromatic, or heteroaromatic acylsilanes from commercially available starting materials.

Items 5 and 9–12 in Table I point out an even more interesting feature of this reaction, which affords the preparation of several novel mono- and bis(acylsilanes), otherwise not easily obtainable by known common routes. Thus for instance, whereas the literature methods failed when applied to the synthesis of the sterically hindered acylsilane **2e**, this compound could be obtained with good yields by following the silylcuprate procedure. In the particular case of item 2, the silylcuprate was prepared by using CuI instead of CuCN. The high reactivity of the expected 3-methyl-1-(trimethylsilyl)-2-buten-1-one in the presence of catalytic amounts of cyanide ion, as already pointed out in a previous paper,[6c] led to the formation of compound **3**, as shown in Scheme II.

The use of CuI turned out to minimize, even if it did not completely inhibit, the formation of **3**, allowing the isolation of **2b** in reasonable yield.

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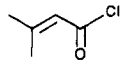
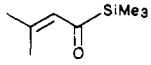
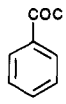
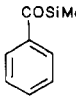
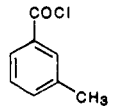
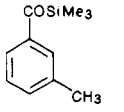
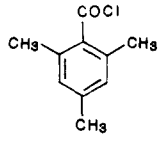
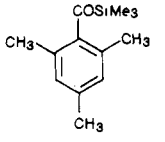
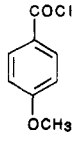
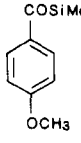
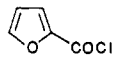
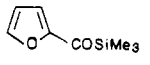
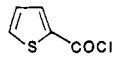
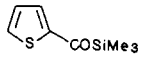
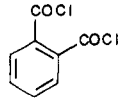
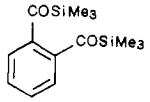
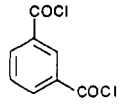
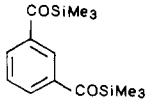
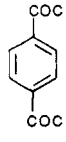
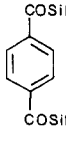
Table I. Synthesis of Acylsilanes through Reaction of RCOCl with $(Me_3Si)_2CuLi$

entry	acyl chloride	copper salt	product	yield,[a] %	ref
1	CH_3COCl **1a**	CuCN	$CH_3COSiMe_3$ **2a**	47	7c
2	 **1b**	CuI	 **2b**	41[b]	10
3	 **1c**	CuCN	 **2c**	87[c]	11
4	 **1d**	CuCN	 **2d**	79	12
5	 **1e**	CuCN	 **2e**	70[c]	
6	 **1f**	CuCN	 **2f**	54	8a
7	 **1g**	CuCN	 **2g**	83	6b
8	 **1h**	CuCN	 **2h**	79	6b
9	$ClCO(CH_2)_4$-COCl **1i**	CuCN	$Me_3SiCO(CH_2)_4$-$COSiMe_3$ **2i**	45	
10	 **1l**	CuCN	 **2l**	43	
11	 **1m**	CuCN	 **2m**	53	
12	 **1n**	CuCN	 **2n**	39[c]	

[a] Isolated yields. [b] Compound **3** was isolated as a byproduct in 34% yield. [c] Scaling up of these reactions from 2 mmol to 30 mmol afforded, after purification, compounds **2c**, **2e**, and **2n** in 73%, 67%, and 39% yields, respectively.

Finally (see footnote *c* in Table I), the reaction is not affected by scaling up, thus presenting the possibility of obtaining acylsilanes at least on a several-grams scale.

In conclusion, the new synthesis of trimethylacylsilanes reported here gives a direct and easy access with high yield to acylsilanes. This should aid the development of the chemistry of these compounds, whose applications are playing an increasingly important role in organic synthesis.

Experimental Section

^1H, ^{13}C, and ^{29}Si NMR spectra were recorded on Perkin-Elmer R-32, Varian FT-80A, and Varian VXR-300 spectrometers. IR spectra were obtained on a Perkin-Elmer 283 spectrophotometer, and mass spectra on a HP 5970A-HP 5790 GC-selective ion detector equipped with a high-performance dimethylsilicone fluid capillary 25-m column, or on a Varian MAT 112 apparatus.

NMR spectra were measured in CCl_4 or $CDCl_3$ solutions. IR spectra were recorded as neat liquids or in $CDCl_3$ solutions. THF was freshly distilled from $LiAlH_4$, and HMPA was dried by distillation over CaH_2 and stored under nitrogen over molecular sieves. GC analysis were performed on a 5% OV 101 column on Chromosorb. Column chromatography was carried out with the flash chromatography technique on Merck Kieselgel 60 (230–400 mesh ASTM). Preparative TLC was performed by using Merck Kieselgel 60 plates. All the reactions were performed under a dry nitrogen atmosphere. CuCN was purchased from Janssen Chimica (Beerse, Belgium) and used as received.

General Procedure. To a solution of Me_3SiLi (2 mmol) [prepared by following the procedure of Hudrlik[13] from hexamethyldisilane (0.5 mL, 2.5 mmol), HMPA (1 mL), and MeLi (2 mmol, 1.25 mL, 1.6 M solution in hexane) in 3 mL of anhydrous THF] cooled to −23 °C was added 90 mg (1 mmol) of CuCN in one portion, the mixture was stirred for 30 min, and then an equimolar amount of the appropriate acyl chloride, dissolved in 1.5 mL of THF, was added dropwise via a syringe. The mixture was stirred for 5 min at −23 °C and then allowed to rise to room temperature over 1 h. The reaction mixture was quenched with NH_4Cl and extracted with ether and the organic layer washed several times with water. Evaporation of the solvent afforded the crude product, which was purified by preparative TLC or column chromatography.

Compound 2e: IR 3070–3030, 2960–2900, 1640, 1250, 840 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.20 (s, 9 H), 2.06 (s, 6 H), 2.22 (s, 3 H), 6.73 (s, 2 H); MS, *m/e* (relative intensity) 220 (25.5, M^+), 205 (49.9), 177 (17.5), 147 (98.8), 119 (4.7), 73 (100).

Compound 2f: IR 3070–3020, 2990–2910, 1610, 1250, 840 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.14 (s, 9 H), 3.88 (s, 3 H), 6.99 (d, 2 H), 7.83 (d, 2 H); MS, *m/e* (relative intensity) 208 (15.3, M^+), 207 (43.7), 177 (55.3), 165 (70.5), 135 (71.3), 73 (100).

Compound 2i: IR 2960, 1640, 1250, 840 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.15 (s, 18 H) 1.40–1.73 (m, 4 H), 2.13–2.43 (m, 4 H); MS, *m/e* (relative intensity) 258 (2.0), 157 (6.2), 147 (16.8), 133 (2.3), 73 (100), 55 (10.3).

Compound 2l: IR 3030, 1610, 1250, 840 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.07 (s, 18 H), 7.26–7.70 (m, 2 H), 7.66–8.06 (m, 2 H); MS, *m/e* (relative intensity) 278 (2.6), 277 (5.3), 262 (13.3), 205 (70.4), 147 (65.2), 73 (100).

Compound 2m: IR 3100–2860, 1620, 1250, 840 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.32 (s, 18 H), 7.42–7.6 (m, 1 H), 7.83–7.97 (m, 2 H), 8.18–8.25 (s, 1 H); MS, *m/e* (relative intensity) 278 (3.2, M^+), 263 (4.8), 207 (7.1), 177 (12.5), 73 (100).

Compound 2n: IR ($CDCl_3$) 3050, 2960, 1620, 1250, 840 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 7.90 (s, 4 H), 0.40 (s, 18 H); ^{13}C NMR (20 MHz, $CDCl_3$) 236.00, 127.56, −1.64 ppm; MS, *m/e* (relative intensity) 278 (4, M^+), 207 (14), 177 (24), 73 (100), 45 (25).

Compound 3: When the procedure described above was followed, 297 mg of crude material could be obtained from 2 mmol of $(Me_3Si)_2CuLi$, prepared by using CuCN, and 237 mg of 3-methyl-2-butenoic acid chloride. Purification by TLC (petroleum ether/ether, 10:1) afforded 225 mg (69%) of compound **3**: IR ($CDCl_3$) 2960, 1730, 1250, 840 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.01 (s, 9 H), 0.02 (s, 9 H), 1.05 (s, 6 H), 1.12 (s, 6 H), 2.24 (s, 1 H), 5.10 (s, 1 H); MS, *m/e* (relative intensity) 231 (4), 147 (26), 140 (13), 125 (29), 73 (100), 45 (22).

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Registry No. **1a**, 75-36-5; **1b**, 3350-78-5; **1c**, 98-88-4; **1d**, 1711-06-4; **1e**, 938-18-1; **1f**, 100-07-2; **1g**, 527-69-5; **1h**, 5271-67-0; **1i**, 111-50-2; **1l**, 88-95-9; **1m**, 99-63-8; **1n**, 100-20-9; **2a**, 13411-48-8; **2b**, 73452-05-8; **2c**, 5908-41-8; **2d**, 68185-95-5; **2e**, 114885-74-4; **2f**, 75748-09-3; **2g**, 80671-28-9; **2h**, 88372-95-6; **2i**, 114885-75-5; **2l**, 114907-34-5; **2m**, 107325-83-7; **2n**, 107325-82-6; **3**, 114885-76-6; Me_3SiLi, 18000-27-6; $(Me_3Si)_2CuLi$, 94140-66-6.

Studies on the Synthesis of (-)-Neplanocin A. Homochiral Preparation of a Key Cyclopentanoid Intermediate†

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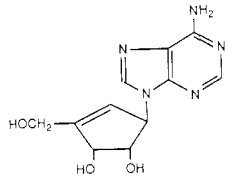
Certain carbocyclic analogues of purine and pyrimidine nucleosides are known to exhibit a variety of therapeutically promising properties[1] which include antitumor, antibacterial, and antiviral activities. Their presumed mechanism of action is that of biological mimicry. However, unlike furanosyl-derived nucleosides which are rapidly disabled by phosphorylase and hydrolase enzymes, carbocyclic analogues appear resistant to most nucleoside metabolases.[2] Appealing traits such as these have made carbocyclic nucleosides attractive synthetic targets.[3,4]

Our ongoing investigation into the total synthesis[3c,4] of neplanocin A (**1**), a novel cyclopentene-derived nucleoside,[5]

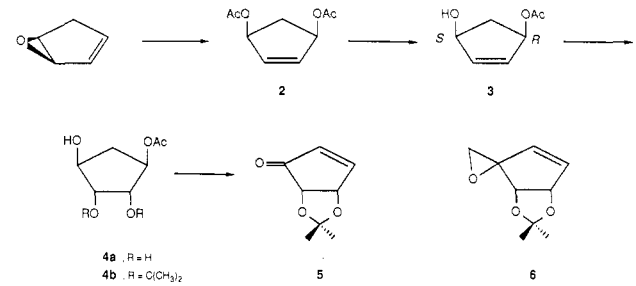


1

prompted our sojourn into cyclopentanoid chemistry.[6,7] Retrosynthetic analysis on neplanocin A had suggested to us that cyclopentenone **5** would make the ideal synthetic precursor since it could be easily homologated to epoxide **6**—a compound we perceive to be an effective 1,4-addition substrate. Herein, we describe the homochiral preparation[8,9] of enone **5** from optically inactive starting material. Interest in this elaborated cyclopentanoid appears to be widespread as evidenced by the fact that it has been the centerpiece of other synthetic ventures.[10]

The synthesis of optically pure **5** is diagrammed in Scheme I. The desired antipodal form of our retrosynthetic starting material, 3(*R*)-acetoxy-5(*S*)-hydroxycyclopent-1-ene (**3**),[7] is accessible via a stereoselective hydrolysis of its parent diester (**2**).[6] Compound **3** is prepared[7] in high optical (>99% ee; $[\alpha]^{26}_D$ +69.6°) and chemical (94%) yields by using the commercially available acetyl cholinesterase (from electric eel) in buffered media. Treatment of **3** with *N*-methylmorpholine *N*-oxide (NMO)[11] and catalytic OsO_4 gave triol **4a** as predicted from guidelines developed by Kishi.[12] Spectral evidence indicated the presence of only this isomer. Conversion of **4a** to hydroxy acetonide **4b** was

Scheme I



effected under standard conditions (acetone and *p*-TSA) in a 74% overall yield based on **3**. Pyridinium chlorochromate oxidation of **4b** proceeded with the fortuitous β-elimination of acetic acid to afford the conjugated enone **5** in 80% yield. Recrystallization of **5** from pentane/ether produced colorless crystals of high optical purity ($[\alpha]^{26}_D$ +70.0°).[13]

Experimental Section

^{1}H NMR spectra were obtained on a Varian EM 360A (60 MHz) or IBM AF 200 (200 MHz) spectrometer with $CDCl_3$ as solvent

† Dedicated to Professor E. J. Corey on the occasion of his 60th birthday.

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(13) Reference 10c reports $[\alpha]^{25}_D$ +71.8° (*c* 0.91, $CHCl_3$); ref 10d reports $[\alpha]_D$ -33° (*c* 0.5, MeOH).

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