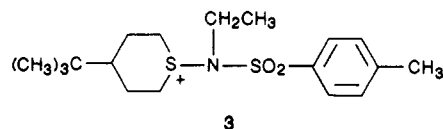


it hydrolyzed to the sulfoxide. Therefore **1** was treated with *N*-chlorobenzotriazole⁸ in acetonitrile, and the chloride salt was metathesized using AgPF₆. Swern and co-workers^{13a,b} have isolated and characterized acyclic *N*-acetylaza sulfonium salts and Gassman and co-workers^{13c} have generated acyclic *N*-acetyl-*N*-arylaaza sulfonium intermediates at 0 °C in dichloromethane and used these in situ. We could find no literature reports on any crystal structures of *N*-acylaza sulfonium salts. The X-ray structure of the acylazasulfonium salt of **2**, as shown in Figure 2, indicates that it has a bicyclic [4.3.0] ring system with an N-S bond distance of 1.76 (2) Å. The N-S bond is a single bond since the sum of the covalent radii of S and N is 1.74 Å.¹⁴ The dihedral angle between C(3)-N-C(4) and N-C(4)-O is 164°, showing a distortion of the amide planarity by the formation of the bicyclic system. The sum of the angles about the nitrogen is 359°, which indicates that it is sp² hybridized. We can compare these

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parameters to those of *trans*-4-*tert*-butyl-1-[ethyl(*p*-tolylsulfonyl)amino]-1-thioniacyclohexane ion, **3**.¹⁵ The



nitrogen atom in **3** is also sp² hybridized, but the S-N bond distance of 1.644 Å is considerably shorter than the predicted single-bond distance of 1.74 Å. In ion **3** the shortened S-N bond has been attributed to p(N)-d(S) π bonding.¹⁵

The preparation and characterization of **2**·PF₆ strengthens our proposal that the reaction of **1** proceeds with neighboring amide participation via an acylaza sulfonium intermediate.

Supplementary Material Available: Tables of atomic coordinates and anisotropic thermal parameters, bond distances and angles for **2**·PF₆ (4 pages). Ordering information is given on any current masthead page.

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Notes

Reaction of Ethyl Azidoformate with Ketene Silyl Acetals¹

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The thermolysis of ethyl azidoformate (EtOCON₃) in enol trimethylsilyl ethers gave *N*-(ethoxycarbonyl)-α-amino ketones.² Our interest has been directed, in general, to the reactivity of other electron-rich alkenes toward ethyl azidoformate and particularly toward (ethoxycarbonyl)-nitrene (EtOCON).^{3,4} In this paper we describe the results obtained in the reactions of ketene silyl acetals⁵ with ethyl azidoformate.

Results and Discussion

The ketene silyl acetals **1**⁶ were reacted with ethyl azidoformate either at room temperature or at 110 °C. GC

Table I. Reaction of EtOCON₃ with Ketene Alkyl Silyl Acetals 1

entry ^a	condts	reactn time, h	products, ^b %		
			2	3	4
a	110 °C	0.5	12 (1)	65 (42)	18 (4)
	rt ^c	24	14	53	32
	hν, 0 °C	5	44 (21)	1	20 (4)
b	110 °C	0.5	6 (4)	68 (42)	14 (13)
	rt	24	10	79	10
	hν, 0 °C	5	70 (38)	2 (2)	8 (4)
c	110 °C	0.5	19 (7)	16 (9)	61 (35)
	rt	120	34	11	53
	hν, 25 °C	5	48 (30)	2 (1)	8 (7)

^a See Scheme I. ^b GC percentages; absolute yields are given in parentheses. ^c rt = room temperature.

analysis of the crude reaction mixtures showed only insignificant differences in product ratio (see Table I) and silica gel chromatography allowed isolation of the three main products detected by GC analysis.

Reaction of EtOCON₃ at 110 °C with the monosubstituted ketene silyl acetal **1a** (see Scheme I) gave a compound retaining the trimethylsilyl group as the main product (42%), which was identified by analytical and spectral data (IR, ¹H NMR, ¹³C NMR, MS) as the carbonimidate **3a**. Two other minor products were separated by column chromatography: the first (4%) was identified as the alkoxyacylurethane **4a** and the second (1%) as the *N*-(ethoxycarbonyl)-α-amino ester **2a**. The other monosubstituted ketene silyl acetal **1b** gave similar results: the silylated carbonimidate **3b** was the main product (42%) accompanied by **4b** (13%) and **2b** (4%) as minor products.

The ratio of products from the disubstituted ketene silyl acetal **1c** was different. In this case the alkoxyacylurethane **4c** was isolated as the principal compound (35%) with **3c** (9%) and **2c** (7%) as minor products. The structure of

(1) Taken in part from the thesis of R. Maestro, 1985. Part of this paper has been presented at the First Belgian Organic Synthesis Symposium (BOSS-1), Namur, May 19-23, 1986, Abstract P1B14.

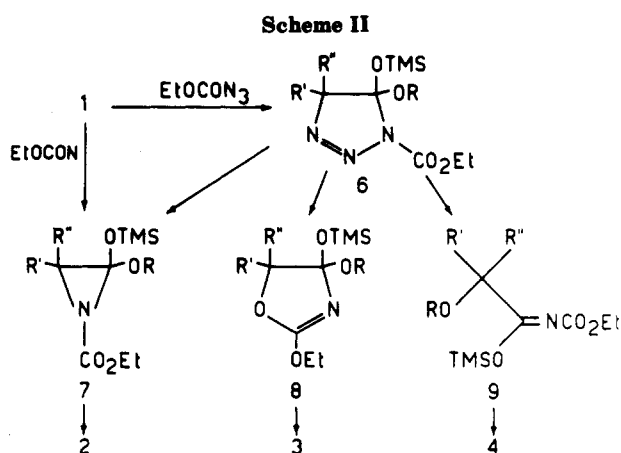
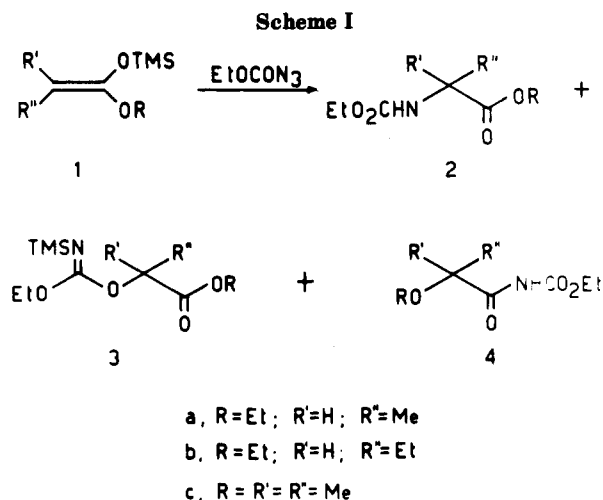
(2) Lociuro, S.; Pellacani, L.; Tardella, P. A. *Tetrahedron Lett.* 1983, 24, 593.

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(5) Among very recent examples of ketene silyl acetal reactions, see: (a) Sakakura, T.; Tanaka, M. *J. Chem. Soc., Chem. Commun.* 1985, 1309. (b) Colvin, E. W.; McGarry, D. G. *J. Chem. Soc., Chem. Commun.* 1985, 539. (c) Slougui, N.; Rousseau, G. *Tetrahedron* 1985, 41, 2643. (d) Gennari, C.; Bernardi, A.; Scolastico, C.; Potenza, D. *Tetrahedron Lett.* 1985, 26, 4129. (e) Oppolzer, W.; Pedrosa, R.; Moretti, R. *Tetrahedron Lett.* 1986, 27, 831.

(6) Slougui, N.; Rousseau, G.; Conia, J. M. *Synthesis* 1982, 58.

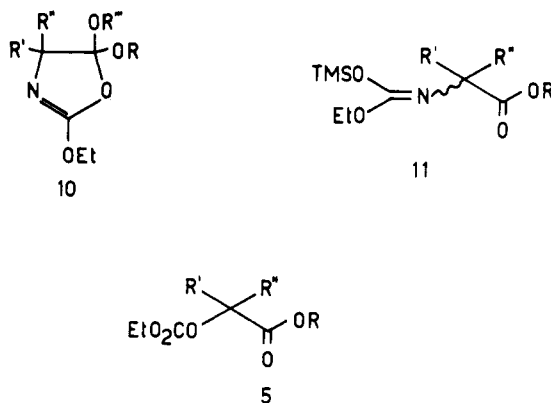


products 3 was fully authenticated by controlled quantitative hydrolysis (AcOH, THF, H₂O, room temperature) to the corresponding *O*-(ethoxycarbonyl)- α -hydroxy esters 5. The reactivity of the substrate 1c was lower than that of the monosubstituted ketene silyl acetals: at room temperature the azide was completely consumed only after 120 h vs. 24 h for the monosubstituted substrates.

The above results deserve comment. Comparison of the reactivity of ketene silyl acetals and of enol silyl ethers⁷ toward ethyl azidoformate shows that at room temperature enol silyl ethers do not react,² while ketene silyl acetals give products resulting from 1,3-dipolar cycloaddition of the azide to a good dipolarophile.⁸ Such reactions are considered to be LUMO controlled and should be accelerated by substituents which raise the dipolarophile HOMO.⁹

The intermediacy of the compounds indicated in Scheme II may be considered on the basis of the results reported by Scarpati¹⁰ in the reactions of non-silylated ketene acetals. In that case a regioisomeric oxazoline 10 (R''' = alkyl) was also postulated as an alternative precursor of the *N*-(ethoxycarbonyl)- α -amino ester.

In our case reaction mixtures obtained at room temperature showed ¹H NMR signals probably attributable



to the triazolines 6, but those signals rapidly disappeared, giving final ¹H NMR spectra quite close to those of the mixtures from the reactions at 110 °C. With the monoalkylated ketene silyl acetals 1a and 1b formation of the main products 3a and 3b may be attributed to facile migration of the trimethylsilyl group¹¹ from the oxygen to the nitrogen atom in the oxazolines 8.

The stability of carbonimidates 3 might be useful for further transformations. Their hydrolysis, under mild conditions, as mentioned above, gave ethoxycarbonyl derivatives of α -hydroxy esters 5.¹²

The products obtained in the reaction at 110 °C are not likely to involve (ethoxycarbonyl)nitrene. However, when EtOCON₃ was photolyzed in the ketene silyl acetals 1 (0 °C, 5 h), the reaction mixtures contained higher amounts (up to 38%, Table I) of the *N*-(ethoxycarbonyl)- α -amino esters 2 as main products. These are the expected products on the basis of the analogy with the results obtained in the thermolysis of EtOCON₃ in enol silyl ethers.² The products 2 probably derive from the silatropic rearrangement of the EtOCON addition product, the aziridine 7, through the isomeric *O*-silylated carbonimide 11 followed by an easy hydrolytic cleavage of the Si-O bond.² However, the photolysis does not necessarily involve a nitrene, but the reaction may proceed by addition of electronically excited ethyl azidoformate and subsequent loss of nitrogen from the resulting triazoline.

On the other hand, it is well-known that ketene silyl acetals undergo addition with chloromethylcarbene⁶ (and other carbenes)^{5c} and that adducts thermally rearrange with loss of Me₃SiCl.⁶

Experimental Section

GC analyses were performed on a Carlo Erba 4100 gas chromatograph with a column of 3% SP 2250 (2 m \times 2 mm) on 100/120 Supelcoport. Mass spectra (MS) and high-resolution mass spectra (HRMS) were obtained on a Kratos MS 80 spectrometer. ¹H NMR spectra (in CDCl₃) and ¹³C NMR spectra (in CDCl₃) were obtained on a Bruker WP-80 SY spectrometer and on a Varian XL-300 spectrometer with CHCl₃ as an internal standard at δ 7.27 and with CDCl₃ as an internal standard at δ 77.00, respectively. IR spectra (in CCl₄) were obtained on a Perkin-Elmer 298 infrared instrument. Boiling points are referenced to an external bath. Ethyl azidoformate was prepared from ethyl chloroformate and sodium azide.¹³ Ketene alkyl silyl acetals were synthesized from the corresponding esters according to the reported procedure.⁶

Reaction of Ethyl Azidoformate with Ketene Alkyl Silyl Acetals at 110 °C. General Procedure. Ethyl azidoformate

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(12) Other more direct transformations of ketene silyl acetals into α -hydroxy esters has been recently reported: Oppolzer, W.; Dudfield, P. *Helv. Chim. Acta* 1985, 68, 216 and references therein.

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(7) A recent example of different reactivity is reported: Sakakura, T.; Hara, M.; Tanaka, M. *J. Chem. Soc., Chem. Commun.* 1985, 1545.

(8) For a recent review, see: Lwowski, W. In *1,3-Dipolar Cycloaddition Chemistry*; Padwa, A., Ed.; Wiley: New York, 1984; Vol. 1, Chapter 5.

(9) A difference of ca. 0.8 eV between the values of the ionization potential of enol ethers and ketene acetals has been reported and probably a similar difference may be expected in the silylated analogues: Bock, H.; Wagner, G.; Wittel, K.; Sauer, J.; Seebach, D. *Chem. Ber.* 1974, 107, 1869.

(10) Scarpati, R.; Graziano, M. L. *J. Heterocycl. Chem.* 1976, 13, 205.

(0.4 mL) and the ketene alkyl silyl acetal (4 mL) in an atmosphere of N_2 were heated at 110 °C. After 0.5 h the excess of the ketene alkyl silyl acetal was distilled in vacuo; the residue was analyzed by GC (the percentages are shown in Table I) and then chromatographed on silica gel (hexane/ethyl acetate, 8:2), giving the products 2,¹⁴⁻¹⁶ 3, and 4 in the yields reported in Table I.

3a: bp 80 °C (10 mmHg); IR 1730, 1710 cm^{-1} ; 1H NMR δ 4.37 (q, 1 H, CH), 4.15 (q, 2 H, CH_2O), 4.13 (q, 2 H, CH_2O), 1.42 (d, 3 H, CH_3CH), 1.29 (t, 3 H, CH_3CH_2O), 1.27 (t, 3 H, CH_3CH_2O), 0.11 (s, 9 H, $(CH_3)_3Si$); ^{13}C NMR δ 166.85 (CO), 160.51 (CN), 68.12 (CH), 62.97 (CH_2O), 61.74 (CH_2O), 22.17 (CH_3C), 14.19 (CH_3C-H_2O), 13.81 (CH_3CH_2O), -0.53 ($(CH_3)_3Si$); mass spectrum, m/z (relative intensity) 261 (M^+ , 23), 246 (7), 216 (23), 188 (34), 144 (46), 117 (100), 103 (19), 100 (23), 75 (15), 73 (61); HRMS, M^+ , 261.1395, calcd for $C_{11}H_{23}NO_4Si$, 261.1396.

3b: bp 83 °C (10 mmHg); IR 1730, 1700 cm^{-1} ; 1H NMR δ 4.30-4.00 (m, 5 H, CH + 2 CH_2O), 1.86-1.66 (m, 2 H, CCH_2CH_3), 1.29 (t, 3 H, CH_3CH_2O), 1.27 (t, 3 H, CH_3CH_2O), 0.92 (t, 3 H, CH_3CH_2C), 0.11 (s, 9 H, $(CH_3)_3Si$); ^{13}C NMR δ 166.46 (CO), 160.46 (CN), 73.15 (CH), 62.89 (CH_2O), 61.73 (CH_2O), 28.83 (CCH_2CH_3), 14.21 (CH_3CH_2O), 13.87 (CH_3CH_2O), 9.76 (CH_3CH_2C), -0.57 ($(CH_3)_3Si$); mass spectrum, m/z (relative intensity) 275 (M^+ , 26), 260 (11), 230 (23), 188 (43), 161 (10), 158 (37), 157 (15), 132 (15), 131 (100), 116 (15), 100 (24), 73 (37); HRMS, M^+ , 275.1554, calcd for $C_{12}H_{25}NO_4Si$, 275.1552.

3c: bp 73 °C (10 mmHg); IR 1730 (br) cm^{-1} ; 1H NMR δ 4.16 (q, 2 H, CH_2O), 3.70 (s, 3 H, CH_3O), 1.50 (s, 6 H, $(CH_3)_2C$), 1.30 (t, 3 H, CH_3CH_2), 0.12 (s, 9 H, $(CH_3)_3Si$); ^{13}C NMR δ 166.96 (CO), 160.01 (CN), 76.35 ($C(CH_3)_2$), 61.61 (CH_2O), 54.46 (CH_3O), 28.49 ($(CH_3)_2C$), 14.13 (CH_3CH_2O), 2.12 ($(CH_3)_3Si$); mass spectrum, m/z (relative intensity) 246 (M^+ - 15, 16), 216 (16), 188 (23), 131 (94), 101 (20), 89 (13), 73 (100); HRMS, M^+ - 15, 246.1159, calcd for $C_{10}H_{20}NO_4Si$, 246.1161.

4a:¹⁷ IR 3410, 1800, 1730 cm^{-1} ; 1H NMR δ 8.57 (br s, 1 H, NH), 4.28 (q, 2 H, CH_2O), 3.90 (q, 1 H, CH), 3.57 (q, 2 H, CH_2O), 1.46-1.10 (m, 8 H); ^{13}C NMR δ 171.52 (CCON), 150.33 (NCOO), 76.09 (CH), 65.42 (CH_2OCH), 62.00 (CH_2OCO), 17.73 (CH_3CH), 14.94 (CH_3CH_2OCH), 13.89 (CH_3CH_2OCO); mass spectrum, m/z (relative intensity) 189 (M^+ , 2), 145 (11), 73 (100), 45 (67).

4b: bp 110 °C (3 mmHg); IR 3410, 1805, 1735 cm^{-1} ; 1H NMR δ 8.50 (s, 1 H, NH), 4.08 (q, 2 H, CH_2OCO), 3.61 (t, 1 H, CH), 3.41 (q, 2 H, CH_2O), 1.59 (m, 2 H, CH_2CH), 1.15 (t, 3 H, CH_3CH_2O), 1.09 (t, 3 H, CH_3CH_2O), 0.78 (t, 3 H, CH_3CH_2C); ^{13}C NMR δ 171.08 (CCON), 150.07 (NCOO), 80.84 (CH), 65.84 ($C-H_2OCH$), 61.65 (CH_2OCO), 24.98 (CH_3CH_2C), 14.69 (CH_3CH_2O-CH), 13.67 (CH_3CH_2OCO), 8.50 (CH_3CH_2C); mass spectrum, m/z (relative intensity) 203 (M^+ , 1), 87 (65), 85 (66), 83 (100), 58 (49), 45 (24); HRMS, M^+ 203.1146, calcd for $C_9H_{17}NO_4$, 203.1157.

4c:¹⁶ IR 3410, 1800, 1730 cm^{-1} ; 1H NMR δ 8.60 (br s, 1 H, NH), 4.25 (q, 2 H, CH_2OCO), 3.25 (s, 3 H, CH_3O), 1.40 (s, 6 H, $(CH_3)_2C$), 1.30 (t, 3 H, CH_3CH_2); ^{13}C NMR δ 173.39 (CCON), 150.83 (NCOO), 79.04 ($C(CH_3)_2$), 62.14 (CH_2OCO), 51.01 (CH_3O), 22.61 ($(CF_3)_2C$), 14.20 (CH_3CH_2); mass spectrum (50 eV), m/z (relative intensity) 189 (M^+ , 2), 130 (8), 86 (9), 84 (15), 74 (32), 73 (100), 43 (55), 41 (33).

Reaction of Ethyl Azidoformate with Ketene Alkyl Silyl Acetals at Room Temperature. General Procedure. Ethyl azidoformate (0.2 mL) and the ketene silyl acetal (2 mL) in an atmosphere of N_2 were allowed to react at room temperature. When the azide band disappeared in the IR spectrum, the excess of the ketene silyl acetal was distilled in vacuo at room temperature. The residue was immediately analyzed by 1H NMR. Spectra changed on standing and after 24 h the spectra of the mixtures were very similar to those of the reactions at 110 °C. The probable triazoline **6b** coming from **1b** was detected by 1H NMR spectrum at 300 MHz: δ 4.35 (m, 2 H, CH_2OC), 3.91 (dd, 1 H, CH), 3.60-3.38 (m, 2 H, CH_2OC), 1.85-1.56 (m, 2 H, CH_2CH), 1.36 (t, 3 H, CH_3CH_2O), 1.21 (t, 3 H, CH_3CH_2O), 1.12 (t, 3 H,

CH_3CH_2C). The percentages of the final mixtures are reported in Table I.

Photolysis of Ethyl Azidoformate with Ketene Alkyl Silyl Acetals. Ethyl azidoformate (0.2 mL) and the ketene silyl acetal (2 mL), in an atmosphere of N_2 , were photolyzed in a quartz vessel using a medium pressure Hanovia PCR lamp (100 W). When the azide band disappeared in the IR spectrum the excess of the ketene silyl acetal was distilled in vacuo at room temperature. The residue was worked up as above, giving the products 2, 3, and 4 in the yields reported in Table I.

Hydrolysis of 3. **3b** (150 mg) was stirred with 5.4 mL of THF, 2.16 mL of acetic acid, and 0.54 mL of water at room temperature for 16 h. The mixture was neutralized by aqueous sodium bicarbonate solution and extracted with ethyl ether. The organic layer was washed with water and brine, dried on molecular sieves (4 Å), and then evaporated giving **5b**¹⁸ (105 mg; 95%): IR 1750 cm^{-1} ; 1H NMR δ 4.85 (dd, 1 H, CH), 4.22 (2 q, 4 H, 2 CH_2O), 1.99-1.81 (m, 2 H, CH_2CH), 1.35-1.21 (m, 6 H, 2 CH_3CH_2O), 1.01 (t, 3 H, CH_3CH_2C); ^{13}C NMR δ 169.91 (CCO), 154.70 (OCO), 72.26 (CH), 64.41 (CH_2O), 61.33 (CH_2O), 24.55 (CH_2CH), 14.14 ($C-H_3CH_2O$), 14.10 (CH_3CH_2O), 9.35 (CH_3CH_2C); mass spectrum, m/z (relative intensity) 205 (M^+ + 1, 27), 204 (M^+ , 9), 176 (29), 159 (86), 131 (21), 130 (86), 115 (21), 114 (37), 102 (17), 87 (33), 73 (10), 59 (100).

The above procedure was followed for **3a**, giving **5a**¹⁹ (70%): IR 1750 cm^{-1} ; 1H NMR δ 5.00 (q, 1 H, CH), 4.22 (q, 4 H, 2 CH_2O), 1.52 (d, 3 H, CH_3CH), 1.33 (t, 3 H, CH_3CH_2O), 1.29 (t, 3 H, CH_3CH_2O); ^{13}C NMR δ 170.43 (CCO), 154.38 (OCO), 71.48 (CH), 64.35 (CH_2O), 61.40 (CH_2O), 16.90 (CH_3CH), 14.12 (2 CH_3CH_2O); mass spectrum, m/z (relative intensity) 191 (M^+ + 1, 7), 190 (M^+ , 12), 145 (65), 118 (47), 117 (58), 102 (31), 101 (22), 75 (10), 74 (11), 73 (33), 45 (100), 43 (16), 29 (67).

The hydrolysis of **3c** required more drastic conditions: 150 mg of **3c** was stirred with 6.0 mL of THF, 6.0 mL of acetic acid, and 1.5 mL of water at room temperature for 120 h, giving **5c**¹⁹ (70%). A specimen of **5c** was prepared according to the reported procedure¹⁸ starting from 2-hydroxybutanoic acid: IR 1750 cm^{-1} ; 1H NMR δ 4.18 (q, 2 H, CH_2O), 3.76 (s, 3 H, CH_3O), 1.61 (s, 6 H, $(CH_3)_2C$), 1.31 (t, 3 H, CH_3CH_2); ^{13}C NMR δ 172.80 (CCO), 153.58 (OCO), 80.06 ($(CH_3)_2C$), 64.11 (CH_2O), 52.54 (CH_3O), 24.57 ($C-H_3C$), 14.18 (CH_3CH_2O); mass spectrum, m/z (relative intensity) 190 (M^+ , 5), 159 (17), 131 (78), 101 (33), 86 (10), 73 (10), 59 (100).

Acknowledgment. This work has been financially supported by the Consiglio Nazionale delle Ricerche, Progetto Finalizzato "Chimica Fine e Secondaria".

Registry No. **1a**, 80675-53-2; **1b**, 65946-52-3; **1c**, 31469-15-5; **2a**, 90243-91-7; **2b**, 73689-70-0; **2c**, 30087-75-3; **3a**, 107010-02-6; **3b**, 107010-03-7; **3c**, 107010-05-9; **4a**, 33094-08-5; **4b**, 107010-04-8; **4c**, 30087-74-2; **5a**, 609-08-5; **5b**, 133-13-1; **5c**, 6065-55-0; **6b**, 107010-06-0.

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Preparation of Desmosterol from (20S,22R,S)-3 β -Acetoxychola-5,23-dien-22-ol^{1a}

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Desmosterol (cholesta-5,24-dien-3 β -ol) was originally synthesized by dehydration of 25-hydroxycholesterol but was considered to be 25-dehydrocholesterol at that time.²

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