of sodium azide (20 g, 0.31 mol), ammonium chloride (0.5 g), water (50 mL), and ethanol (150 mL), heated, and stirred under reflux for 16 h. The resulting mixture was evaporated to a residue keeping the heating bath below 40 °C. The residue was suspended in chloroform (500 mL) and washed with saturated brine (2 × 50 mL). The organic layer was evaporated to an oil yielding 4.2 g of crude **16**.

(1β,2α,3β)-3-Amino-1,2-cyclopentanediol Hydrochloride (17). The crude **16** (4.2 g) was dissolved in methanol (150 mL), 10% Pd/C (0.5 g) was added, and the mixture was hydrogenated at 50 psi on a Parr hydrogenator for 2 h. The catalyst was removed by filtration, and the solution was evaporated to an oil. The oil was stirred with 5 M ethanolic HCl for 16 h and then evaporated to a residue. Crystallization from propan-1-ol yielded **17** (2.8 g, 38%), mp 34–37 °C: IR (Nujol) 3300 cm^{-1}; ^1H NMR (Me_2SO-d_6) δ 8.23 (3 H, br s), 5.47 (1 H, d), 5.16 (1 H, br s), 3.81 (1 H, t, $J_{1,2}$ = 5.4), 3.74 (1 H, q, $J_{1,2}$ = 5.4, $J_{2,3}$ = 5.9), 3.15 (1 H, br q, $J_{2,3}$ = 5.9), 1.55–1.9 (4 H, m); MS (CI), *m/e* 118 (M + 1), 100 (base), 82.

Anal. Calcd for $C_5H_{11}NO_2$·HCl; C, 39.09; H, 7.87; N, 9.11. Found, C, 38.95; H, 7.69; N, 9.18.

Synthesis of Mono and Unsymmetrical Bis Ortho Esters of *scyllo*-Inositol

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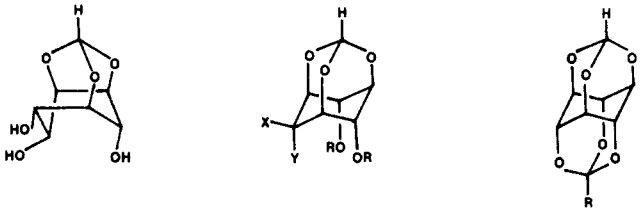
Related to an ongoing program, we needed a cyclohexane derivative bearing three axially disposed hydroxyl groups at the 1, 3, and 5 positions. An adamantane system obviously meets with this requirement because of its conformational rigidity. In particular, we have felt that mono ortho esters of *scyllo*-inositol ideally satisfies our requirement. In this paper, we would like to report a synthesis of mono and unsymmetrical bis ortho esters of *scyllo*-inositol.

Direct mono ortho ester preparation of *scyllo*-inositol did not seem promising since the rate of bis ortho ester formation was anticipated to be faster than that of mono ortho ester formation.[1] For this reason, we were interested in *myo*-inositol, which should form a mono ortho ester but not a bis ortho ester. Indeed, in 1966, Luk'yanov and Tolkachev disclosed the synthesis of monoorthoformate of *myo*-inositol by treatment with triethyl orthoformate in toluene containing *p*-toluenesulfonic acid and assigned the structure **1** to this product.[2] We were curious about the assigned structure and decided to reexamine this reaction. With modifications of the reported method,[3] we were able to isolate the monoorthoformate (mp 300–302 °C (sealed tube)) in 76% yield. The ^1H NMR spectra of both this product and its triacetate (mp 173–174 °C) clearly demonstrate that the monoorthoformate isolated must have a structure with a symmetry element, allowing assignment of the adamantane structure **2**. Unfortunately, however, there are no physical data available to conclude that this product is identical with the monoorthoformate previously reported.[2]

Selective protection of the equatorial hydroxyl group of **2** was readily achieved by treatment with *tert*-butyldimethylsilyl chloride in DMF in the presence of imidazole,[4] to yield **3** (mp 179–181 °C) in 48% yield.[5] The spectroscopic data show that **3** has only one *tert*-butyldimethylsilyl group, which was further confirmed from the fact that **3** yielded a diacetate (mp 64–65 °C) on treatment with acetic anhydride and pyridine. The ^1H NMR spectra of both **3** and its diacetate clearly establish that **3** was symmetrical, establishing the structure of this product as **3**. Benzylation of **3** under standard conditions gave the dibenzyl ether **4** (mp 124–125 °C; 93% yield), of which the desilylation furnished the dibenzyl ether alcohol **5** (viscous oil). Spectroscopic data of **4** and **5** are fully consistent with the assigned structures.



1

2: X=OH, Y=H, R=H
3: X=OSi(t-Bu)(Me)$_2$, Y=H, R=H
4: X=OSi(t-Bu)(Me)$_2$, Y=H, R=CH_2Ph.
5: X=OH, Y=H, R=CH_2Ph
6: X=Y=O(ketone), R=CH_2Ph.
7: X=H, Y=OH, R=CH_2Ph
8: X=H, Y=OH, R=H

9: R=H
10: R=Me

Swern oxidation[6] of **5** yielded the ketone **6**, which was, without purification, reduced with sodium borohydride in a mixture of tetrahydrofuran and methanol, to give exclusively the axial alcohol **7** (mp 98–99 °C), the epimeric alcohol of **5**, in 87% overall yield from **4**. The structures of **6** and **7** are established by their spectroscopic data. Debenzylation of **7** under standard hydrogenolysis conditions (5% or 10% Pd on C in methanol) or under catalytic transfer hydrogenation conditions[7] failed. However, in the presence of Pearlman's catalyst (20% $Pd(OH)_2$ on C)[8] hydrogenolysis did take place smoothly to yield the desired monoorthoformate **8** (mp 330 °C (sealed tube)) of *scyllo*-inositol in 94% yield. The ^1H NMR spectra of both **8** and its triacetate (mp 124–126 °C) establish the assigned structure. The overall yield of **8** from *myo*-inositol was about 28%.

Treatment of monoorthoformate **8** with triethyl orthoformate in THF containing a small amount of *p*-toluenesulfonic acid at room temperature smoothly gave quantitatively the known bis(orthoformate) **9** of *scyllo*-inositol.[9] Similarly, **8** yielded quantitatively unsymmetrical bis ortho ester **10** (mp 177–178 °C) of *scyllo*-inositol on treatment with triethyl orthoacetate. There was no product from

(1) Partial hydrolysis of the bis(orthoformate) of *scyllo*-inositol (for the preparation of this substance, see ref 9) was attempted under acidic conditions, but the starting material was either unchanged (aqueous AcOH/dioxane/90 °C) or only *scyllo*-inositol was, as expected, obtained (6 N HCl/dioxane/90 °C).

(2) Luk'yanov, A. V.; Tolkachev, O. N. USSR Patent 184 841, 1966; *Chem. Abstr.* **1967**, *66*, 95365.

(3) We attempted the orthoformate preparation in toluene containing *p*-toluenesulfonic acid but were unable to reproduce the reported results. The near insolubility of *myo*-inositol in toluene was obviously a problem in this reaction. For this reason, Me_2SO was chosen for the solvent.

(4) Corey, E. J.; Venkateswarlu, A. *J. Am. Chem. Soc.* **1972**, *94*, 6190.

(5) Attempted selective oxidation of **2** (O_2/Pd–C or Swern oxidation[6]) did not give encouraging results.

(6) Mancuso, A. J.; Huang, S.-L.; Swern, D. *J. Org. Chem.* **1978**, *43*, 2480.

(7) Anantharamaiah, G. M.; Sivanandaiah, K. M. *J. Chem. Soc., Perkin Trans. 1* **1977**, 490.

(8) Rylander, P. N. "Catalytic Hydrogenation of Platinum Metals": Academic Press: New York, 1967; p 464.

(9) Vogl, O.; Anderson, B. C.; Simons, D. M. *J. Org. Chem.* **1969**, *34*, 204.

0022-3263/85/1950-4402$01.50/0

transesterification detected in this reaction.

Experimental Section

Reagents and solvents were commercial grades and were used as supplied with the following exceptions: ether and tetrahydrofuran (THF) were distilled from sodium benzophenone ketyl; toluene was distilled from sodium metal; pyridine was dried over KOH; Me_2SO was stored over 4A molecular sieves. Moisture- and/or air-sensitive reactions were conducted under nitrogen atmosphere.

Analytical TLC was performed on 0.25-mm precoated silica gel plates purchased from E. Merck. Preparative TLC separations were performed on plates (20 × 20 cm) prepared with a 2-mm layer of silica gel PF_{254} from E. Merck. Column chromatography was performed on E. Merck kieselgel 60.

Melting points (mp) were determined on a Koffler hot stage apparatus and are uncorrected. Elemental analyses were performed at Analytical Laboratories, Meijo University, Nagoya, Japan.

^1H NMR spectra were measured on a 300-MHz Bruker AM-300 instrument in the Fourier transform mode. Chemical shifts are reported in ppm downfield from Me_4Si (δ) as internal standard unless otherwise stated. The following abbreviations are used for spin multiplicity: s, singlet; d, doublet; t, triplet; m, multiplet. IR spectra were recorded on a Perkin-Elmer Model 727 spectrometer and reported in wavenumbers (cm^{-1}). MS spectra were determined in Kratos MS-50 double focusing instrument in chemical ionization mode using isobutane as the reagent gas.

***myo*-Inositol Monoorthoformate (2).** A solution of *myo*-inositol (10.80 g, 60 mmol) and triethyl orthoformate (18 mL) in 150 mL of dimethyl sulfoxide in the presence of *p*-toluenesulfonic acid monohydrate (*p*-TsOH·H_2O) (3.0 g) was heated for 18 h at 100 °C under N_2. After cooling, 20 mL of 10% aqueous $NaHCO_3$ solution was added, and stirring was continued for 30 min. The color of the solution changed from dark brown to purple. The reaction mixture was evaporated to dryness in vacuo, dissolved in the minimum amount of water, and chromatographed on silica gel column by eluting with CH_3CN. The resulting syrup was triturated with chloroform to yield the monoorthoformate **2** (8.68 g, 76% yield). A sample for elemental analysis was recrystallized from hot methanol: mp 300–302 °C (sealed tube); ^1H NMR (D_2O, DSS) δ 4.23 (2 H, m), 4.26 (1 H, m), 4.33 (1 H, m), 4.57 (2 H, t, J = 3.8 Hz), 5.59 (1 H, d, J = 0.8 Hz): IR (KBr disk) 3350, 2930, 1450, 1162, 1050, 996; MS, m/e (relative intensity) 191 (M^+ + H, 100). Anal. Calcd for $C_7H_{10}O_6$: C, 44.21; H, 5.30. Found: C, 43.98; H, 5.46.

The triacetate of **2** was prepared under standard conditions (Ac_2O/py/room temperature). A sample for elemental analysis was obtained by recrystallization from hexane–ethyl acetate: mp 173–174 °C; ^1H NMR ($CDCl_3$) δ 2.10 (6 H, s), 2.22 (3 H, s), 4.34 (2 H, m), 4.60 (1 H, m), 5.20 (1 H, m), 5.51 (2 H, t, J = 3.8 Hz), 5.61 (1 H, s). Anal. Calcd for $C_{13}H_{16}O_9$: C, 49.37; H, 5.10. Found C, 49.33; H, 5.22.

***tert*-Butyldimethylsilyl Ether 3.** To a stirred solution of the triol **2** (1.90 g, 10 mmol) and imidazole (1.70 g, 25 mmol) in DMF (20 mL) at 0 °C was added *tert*-butyldimethylsilyl chloride (1.81 g, 12 mmol) under N_2. After 4 h the reaction mixture was slowly warmed to room temperature, and stirring was continued for 20 h. Then the reaction mixture was evaporated in vacuo to dryness, and the diol **3** (1.44 g, 48% yield) was isolated by silica gel column chromatography with CH_2Cl_2:MeOH = 10:1. A sample for elemental analysis was prepared by recrystallization with hot methanol: mp 179–181 °C; ^1H NMR ($CDCl_3$) δ 0.95 (6 H, s), 1.25 (9 H, s), 3.23 (2 H, d, J = 7.6 Hz), 4.16 (2 H, m), 4.28 (2 H, s), 4.59 (2 H, m), 5.51 (1 H, s); IR (KBr disk) 3445, 2929, 1166, 980; MS, m/e (relative intensity) 305 (M^+ + H, 77), 131 (100). Anal. Calcd for $C_{13}H_{24}O_6Si$: C, 51.29; H, 7.95. Found C, 51.06; H, 8.25.

Diacetate of **3** was prepared under standard conditions (Ac_2O/py/room temperature). A sample for elemental analysis was obtained by recrystallization from hexane–ethyl acetate: mp 64–65 °C; ^1H NMR ($CDCl_3$) δ 4.14 (1 H, s), 4.17 (2 H, m), 4.55 (1 H, m), 5.47 (2 H, t, J = 3.8 Hz), 5.58 (1 H, s). Anal. Calcd for $C_{17}H_{28}O_8Si$: C, 52.56; H, 7.27. Found: C, 52.36; H, 7.52.

***tert*-Butyldimethylsilyl Ether Dibenzyl Ether 4.** To a stirred solution of the diol **3** (456 mg, 1.5 mmol) in 0.5 mL of DMF was added NaH (108 mg, 4.5 mmol) under N_2. After 30 min benzyl bromide (535 μL, 4.5 mmol) was added dropwise, and stirring was continued for 24 h. Several drops of water were added to quench the reaction. Following evaporation of the solvent in vacuo, the reaction mixture was passed through a silica gel column by eluting with 10:1 CH_2Cl_2–MeOH and was separated on a silica gel column by eluting with 1:6 ethyl acetate:hexane to give 678 mg of the desired product **4** (viscous oil; 678 mg, 93% yield): ^1H NMR ($CDCl_3$) δ 0.13 (6 H, s), 0.93 (9 H, s), 4.15 (2 H, m), 4.33 (2 H, t, J = 3.8 Hz), 4.41 (2 H, m), 4.62 (4 H, AB, J = 11.8 Hz), 7.27 (10 H, s); IR (film) 2950, 2850, 1460, 1260, 1170. MS, m/e (relative intensity) 485 (M^+ + H, 12), 91 (100).

Dibenzyl Ether Alcohol 7. To a stirred solution of **4** (678 mg, 1.40 mmol) in 10 mL of THF was added tetrabutylammonium fluoride (4.2 mL of 1.0 M solution in THF) dropwise. After 30 min the reaction mixture was diluted with ether until it became turbid, and then the ether solution was passed through a silica gel column by eluting with ether to obtain 420 mg of syrup, which was used for the next step without further purification. A sample for elemental analysis was prepared by recrystallization from hot toluene: mp 124–125 °C; ^1H NMR ($CDCl_3$) δ 3.05 (1 H, d, J = 11.5 Hz), 4.23 (3 H, m), 4.37 (2 H, t, J = 3.6 Hz), 4.46 (1 H, m), 4.62 (4 H, AB, J = 11.5 Hz), 7.28 (10 H, m); IR (film) 3460, 2960, 2870, 1460; MS, m/e (relative intensity) 371 (M^+ + H, 100), 91 (71). Anal. Calcd for $C_{21}H_{22}O_6$: C, 68.09; H, 5.99. Found C, 68.32; H, 6.06. Monoacetate of this alcohol was prepared under standard conditions (Ac_2O/py/room temperature). A sample for elemental analysis was obtained by recrystallization from hexane–ethyl acetate: mp 116–118 °C; ^1H NMR ($CDCl_3$) δ 2.20 (3 H, s), 4.39 (4 H, m), 4.50 (1 H, m), 4.63 (4 H, s), 5.40 (1 H, m), 5.54 (1 H, d, J = 1.3 Hz). Anal. Calcd for $C_{23}H_{24}O_7$: C, 66.98; H, 5.87. Found: C, 66.85; H, 6.03.

Oxalyl chloride (244 μL, 2.8 mmol) was added to dichloromethane (20 mL) at room temperature under N_2 and then was cooled to −78 °C. Dimethyl sulfoxide (391 μL, 5.6 mmol) was added rapidly via syringe. After stirring for 5 min, the crude alcohol in 50 mL of dichloromethane was added dropwise and stirring was continued for 1 h. Triethylamine (1.95 mL, 14 mmol) was added dropwise, and the reaction mixture was allowed to come gradually to room temperature. About 70 mL of ether was added to the reaction mixture, and the resulting slurry was filtered through a short silica gel column by eluting with ether to obtain about 440 mg of the crude crystalline ketone.

The crude ketone was dissolved in a mixture of methanol (20 mL) and THF (5 mL), and $NaBH_4$ (132.4 mg, 3.5 mmol) was added in one portion. After being stirred for 20 min, the reaction was quenched with water, and the reaction mixture was diluted with 200 mL of dichloromethane and filtered through a short silica gel column (eluent; 10:1 CH_2Cl_2–MeOH) to obtain crude alcohol, which was purified by PTLC (silica gel, 2:1 hexane–ethyl acetate) to afford the dibenzyl ether alcohol **7** (449 mg, 87% overall yield from **4**). A sample for elemental analysis was prepared by recrystallization from hot methanol: mp 98–99 °C; ^1H NMR ($CDCl_3$) δ 4.12 (1 H, d, J = 12 Hz), 4.40 (3 H, m), 4.49 (2 H, m), 4.62 (1 H, m), 4.65 (4 H, s), 5.50 (1 H, s), 7.29 (10 H, m); IR (film) 3500, 2950, 2870, 2458, 1160; MS, m/e (relative intensity) 371 (M^+ + H, 73), 91 (100). Anal. Calcd $C_{21}H_{22}O_6$: C, 68.09, H, 5.99. Found: C, 67.88; H, 5.88. The monoacetate of **7** was prepared under standard conditions (Ac_2O/py/room temperature). A sample of elemental analysis was obtained by recrystallization from hexane–ethyl acetate: mp 102–104 °C; ^1H NMR ($CDCl_3$) δ 1.77 (3 H, s), 4.36 (2 H, m), 4.64 (3 H, m), 4.62 (4 H, AB, J = 11.2 Hz), 5.35 (1 H, m), 5.56 (1 H, s), 7.25 (10 H, m). Anal. Calcd for $C_{23}H_{24}O_7$: C, 66.98; H, 5.87. Found: C, 66.70; H, 5.82.

***scyllo*-Inositol Monoorthoformate (8).** The recrystallized dibenzyl ether alcohol **7** (380 mg, 1 mmol) was dissolved in 5 mL of THF and Pearlman's catalyst[8] (190 mg of 20% $Pd(OH)_2$ on C (Aldrich)) was added in a pressure bottle. After flushing the bottle three times with H_2 (30 psi), hydrogenolysis was performed over 3 h on a Parr apparatus at 50 psi of H_2. The resulting reaction mixture was filtered through a Celite pad, which was subsequently washed with THF. The combined filtrate was concentrated to give the pure triol **8** (174 mg, 94% yield). A sample for elemental analysis was prepared by recrystallization from hot ethyl acetate: mp 330 °C (sealed tube); ^1H NMR (D_2O, DSS) δ 4.42 (3 H, dd, J = 4.6, 3.0 Hz), 4.53 (3 H, dd, J = 4.6, 3.0 Hz), 5.64 (1 H, s); IR (KBr disk) 3280, 3180, 2950, 1480, 1440, 1165; MS, m/e (relative

intensity) 191 (M^+ + H, 100). Anal. Calcd for $C_7H_{10}O_6$: C, 44.21; H, 5.30. Found: C, 43.99; H, 5.37.

The triacetate of **8** was prepared under standard conditions (Ac_2O/py/room temperature). A sample for elemental analysis was obtained by recrystallization from hexane–ethyl acetate: mp 124–126 °C; ^1H NMR ($CDCl_3$) δ 2.07 (9 H, s), 4.60 (3 H, dd, J = 4.4, 3.0 Hz), 5.37 (3 H, dd, J = 4.4, 3.0 Hz), 5.57 (1 H, s). Anal. Calcd for $C_{13}H_{16}O_9$: C, 49.37; H, 5.10. Found: C, 49.26; H, 5.21.

Bis Ortho Ester Preparation from Monoorthoformate 8. The monoorthoformate **8** (38 mg, 2 mmol) and triethyl orthoacetate (73 L, 4 mmol) were dissolved in 2 mL of THF, and a catalytic amount of *p*-TSH·H_2O was added. The reaction mixture was stirred at room temperature for 5 h, and then 1 mL of 10% aqueous $NaHCO_3$ solution was added. The reaction mixture was evaporated to dryness in vacuo, and the residue was partitioned between CH_2Cl_2 and water. The organic layer was washed with 10% aqueous $NaHCO_3$ solution, water, and brine. The organic layer was dried over Na_2SO_4 and evaporated to dryness, to give the desired bis ortho ester **10** (42.1 mg, 98% yield). A sample for elemental analysis was prepared by recrystallization from hexane–ethyl acetate: mp 177–178 °C; ^1H NMR ($CDCl_3$) δ 1.47 (3 H, s), 4.54 (6 H, s), 5.51 (1 H, s). IR (KBr disk) 2960, 1420, 1400, 1310, 1280, 1180, 1138, 1098, 1045, 958, 870; MS, *m/e* (relative intensity) 215 (M^+ + H, 100). Anal. Calcd for $C_9H_{10}O_6$: C, 50.47; H, 4.71. Found: C, 50.49; H, 4.76.

Bis ortho ester **9**[9] was similarly obtained by treatment with triethyl orthoformate.

Acknowledgment. Financial support from the National Institutes of Health (Grant No. CA22215) is gratefully acknowledged. The Bruker AM300 NMR instrument used in this research was purchased through the NIH instrument Grant No. S10 RR01748 to Harvard University.

Registry No. **2**, 98510-20-4; **2** (triacetate), 98510-21-5; **3**, 98510-22-6; **3** (diacetate), 98510-23-7; **4**, 98510-24-8; **5**, 98510-25-9; **5** (monoacetate), 98510-26-0; **6**, 98510-27-1; **7**, 98575-46-3; **7** (monoacetate), 98575-47-4; **8**, 98575-48-5; **8** (triacetate), 98575-49-6; **9**, 4922-14-9; **10**, 98510-28-2; *myo*-inositol, 87-89-8; triethyl orthoformate, 122-51-0; triethyl orthoacetate, 78-39-7.

Photolysis of Methyl 3-Diazo-2-oxopropionate. Wolff Migration of the Carbomethoxy Group[1]

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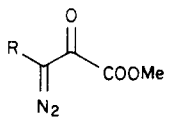
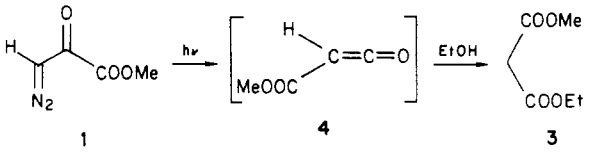
In 1977 Bien and Segal[2] reported an unusual palladium-catalyzed Wolff rearrangement of ethyl 3-diazo-2-oxopropionate. Here we describe our results on the photolysis of the related methyl ester **1**[3] and of other diazo-2-oxopropionates (**2a**, **2b**).



1, R = H
2a, R = COMe
2b, R = COOMe

Photolysis of a dilute solution of **1** in benzene and a fourfold molar excess of ethanol (relative to **1**) led to a single major product in 37% yield, ethyl methyl malonate (**3**). The clear inference is that Wolff rearrangement to

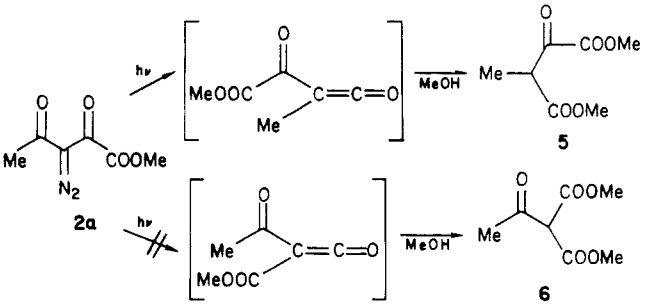


1 **4** **3**

4 was followed by capture of the ketene by alcohol. When the photolysis was carried out in benzene alone, and the excess ethanol added immediately afterwards, the yield of **3** declined to 14%. Thus **3** is not produced by any *direct* reaction of **1** with ethanol. When no alcohol was added, an 8% yield of dimethylmalonate was isolated. The precise source of the required methyl alcohol is obscure, but it must be derived from the carboalkoxy group of **1**.

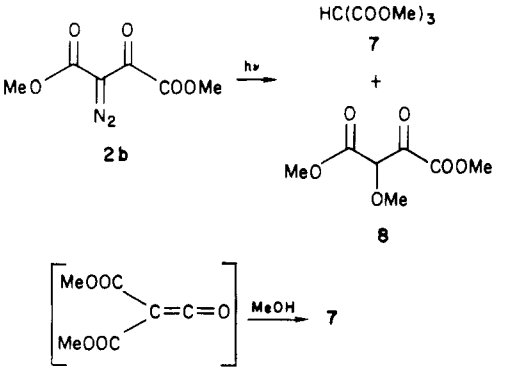
Although an example of ester migration in the Wolff rearrangement was described as early as 1917,[4] the migration remains an unusual one, and the ester group is not well placed on the scale of migratory aptitudes.

We have synthesized diazo esters **2a** and **2b** in order to compare the migratory aptitude of carboalkoxy with a relatively efficient migrating group, methyl, and an inefficient group, methoxy.[5] Irradiation of **2a** in a dilute benzene solution containing a fourfold excess of methanol led to the isolation of a single major product **5**, the product of methyl migration. No trace of **6**, the product of ester migration, was found. Thus methyl migrates to the exclusion of carbomethoxy.



2a **5** **6**

Irradiation of **2b** in a benzene/methanol solution led to three products. The major product, trimethyl methanetricarboxylate (**7**) (83% relative yield), was accompanied by 9% of **8** and 8% of an unidentified product.



2b **8**

(1) Support for this work through grants from the Van't Hoff Fund and the National Science Foundation (MPS 74-05690, CHE-8318345) is gratefully acknowledged.

(2) Bien, S.; Segal, Y. *J. Org. Chem.* **1977**, *42*, 1685.

(3) A copper-catalyzed addition reaction of **1** has been previously described by us: Gallucci, R. R.; Jones, M., Jr. *J. Am. Chem. Soc.* **1976**, *98*, 7704.

(4) Staudinger, H.; Hirzel, H. *Chem. Ber.* **1916**, *49*, 2522.

(5) For a comparison of various groups, see: Meier, H.; Zeller, K.-P. *Angew. Chem., Int. Ed. Engl.* **1975**, *14*, 32. Roedig, A.; Fahr, E.; Aman, H. *Chem. Ber.* **1964**, *97*, 77. Zeller, K.-P.; Meier, H.; Müller, E. *Tetrahedron* **1972**, *28*, 5831. Heyes, G.; Holt, G. *J. Chem. Soc., Perkin Trans. 1* **1973**, 1206. Buu, N. T.; Edward, J. T. *Can. J. Chem.* **1972**, *50*, 3719, 3730.

0022-3263/85/1950-4404$01.50/0