freeze" for most of its known existence, is proving to be a very versatile reagent with a broad spectrum of uses in both fluorine and general organic chemistry.

Experimental Section

^1H NMR spectra were recorded with Bruker WH-360 and AC200E spectrometers at 360 and 200 MHz, respectively, with $CDCl_3$ as solvent and Me_4Si as an internal standard. The ^{19}F NMR spectra were measured at 338.8 MHz and are reported in parts per million upfield from $CFCl_3$, which served as both solvent and internal standard. The proton broad-band-decoupled ^{13}C NMR spectra were recorded on the Bruker AC200E spectrometer at 50.30 MHz; $CDCl_3$ served as solvent and TMS as internal standard. Mass spectra were measured with a Du Pont 21-491B spectrometer. IR spectra were recorded as neat films, in $CHCl_3$ solution or as KBr pellets on a Perkin-Elmer 177 spectrophotometer.

General Procedure for Working with Fluorine. Fluorine is a strong oxidizer and a very corrosive material. An appropriate vacuum line made from copper or monel in a well-ventilated area should be constructed for working with this element. For more experimental details, see for example ref 28. For the occasional user, however, various premixed mixtures of F_2 in inert gases are commercially available, simplifying the whole process. The reactions themselves can be carried out in glass vessels. If elementary precautions are taken, work with fluorine is relatively simple and we have had no bad experiences working with this element.

General Procedure for Producing the Oxidizing Reagent. Mixtures of 10% F_2 with nitrogen were used in this work. The gas mixture was prepared in a secondary container before the reaction was started. This mixture was then passed at a rate of about 400 mL/min through a cold (−10 °C) and vigorously stirred mixture of 400 mL of CH_3CN and 40 mL of H_2O. The formation of the oxidizing power was monitored by reacting aliquots with an acidic aqueous solution of KI. The liberated iodine was then titrated with thiosulfate. We have thus achieved concentrations of more then 1 mol/L of the oxidizing reagent.

General Epoxidation Procedure. An appropriate amount of olefin (see discussion) was dissolved in about 50 mL of CH_2Cl_2, and the resultant mixture was cooled to 0 °C and added in one portion to the reaction vessel in which the oxidizing agent had been prepared. With the exception of the more resistant olefins mentioned earlier, the reaction was stopped after 1 min by neutralizing with saturated sodium bicarbonate solution. The mixture was then poured into 1500 mL of water, extracted with CH_2Cl_2, and washed with $NaHCO_3$ and water until neutral. The organic layer was dried over $MgSO_4$ and the solvent evaporated, preferably at room temperature. The crude product was usually purified by vacuum flash chromatography using silica gel 60-H (Merck) and if needed also by HPLC (Waters) on Merck's LiChrosorb Si-100. The purity was also confirmed by GC on 20% SE-30 or 10% OV-17 columns. The spectral and physical properties of the known products thus obtained were compared either with those of authentic samples or with the properties reported in the literature. In every case excellent agreement was obtained. All new compounds were properly identified by ^1H NMR, ^{13}C NMR, IR, MS, and microanalysis. Some of the spectral properties of these compounds can be found in the appropriate footnotes.

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Notes

A Tandem Denitration–Deoxygenation of α-Nitro Ketones via (*p*-Tolylsulfonyl)hydrazones with Lithium Aluminum Hydride: A Practical Synthesis of (*Z*)-9-Tricosene, the Sex Pheromone of the Housefly (*Musca domestica*)

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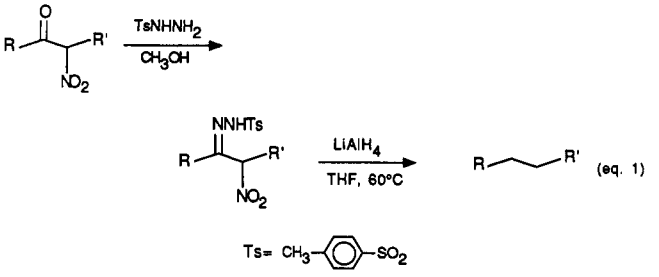
The utilization of α-nitro ketones in organic synthesis has been increased by the discovery of efficient procedures to affect the substitution of the nitro group with hydrogen or deuterium. Procedures and examples of the utilization of functionalized nitroalkanes as reagents for alkyl anion synthons to build structures in a convenient and predictable way[1,2] have been reviewed.

Now, we report a tandem denitration–deoxygenation sequence performed by reduction of (*p*-tolylsulfonyl)-hydrazones of α-nitro ketones 1 with lithium aluminum hydride in tetrahydrofuran (THF) at 60 °C (eq 1). On the basis of our indirect method to effect the denitrohydrogenation of α-nitro ketones[1,3] and on the Caglioti



reaction[4,5] for the carbonyl to methylene conversion, this procedure provides the corresponding alkanes in good to high yields (Table I).

Linear α-nitro ketones can be easily prepared by classical chain-lengthening sequences such as acylation of primary nitroalkanes[6,7] and nitro aldol reaction (Henry reaction)

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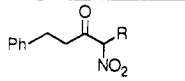
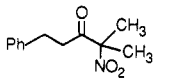
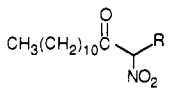
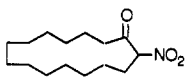
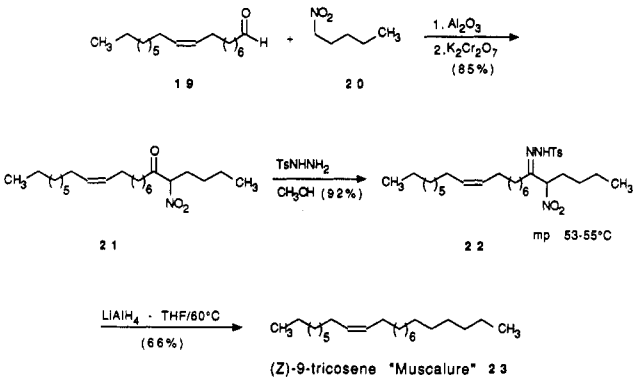
Table I. Reductive Denitration–Deoxygenation of α-Nitro Ketones via Their (p-Tolylsulfonyl)hydrazones

α-nitro ketone			tosylhydrazone		alkane	
structure	R	no.	no.	yield (%)	no.	yield (%)
	CH_3	1	2	94	3	70
	nC_8H_{17}	4	5	96	6	61
		7	8	65	9	67
	CH_3	10	11	93	12	65
	nC_6H_{11}	13	14	95	15	68
		16	17	98	18	71

Scheme I. Synthesis of (Z)-9-Tricosene ("Muscalure"), Sex Pheromone Component of Female Housefly (*Musca domestica*)

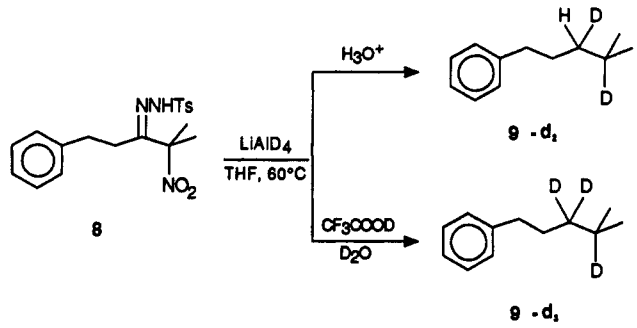


between aldehydes and aliphatic nitroalkanes, followed by oxidation of 2-nitroalkanol intermediates.[1,8-11]

The preparation of (Z)-9-tricosene, a sex pheromone component of the mature female housefly[12,13] (*Musca domestica*) is reported as an application of this reaction (Scheme I). Nitro aldol reaction of oleic aldehyde (**19**) with 1-nitropentane (**20**) on basic alumina in the absence of a solvent followed by in situ oxidation of the resulting nitro alcohol with potassium dichromate in the presence of tetra-*n*-butylammonium hydrogen sulfate as phase-transfer catalyst[14] affords the α-nitro ketone **21** in 85% yield. The conversion of compound **21** into the corresponding (*p*-tolylsulfonyl)hydrazone **22** was performed in 92% yield after recrystallization. Reduction of compound **22** with lithium aluminum hydride at 60 °C produced (Z)-9-tricosene (**23**) in 66% yield. The purity of the product, analyzed by GC, is 98%. None of the corresponding *E* isomer was detected by ^{13}C NMR spectroscopy.

The denitration–deoxygenation reaction should be useful for specific deuterium labeling[15] (Scheme II). When α-nitro ketone tosylhydrazone **8** was reduced with lithium aluminum deuteride at 60 °C for 10 h, quenching with 2 N aqueous hydrochloric acid gave compound **9**-d_2 (at least 98% isotopically pure dideuterated alkane) in 55% yield, while treating the reaction mixture with trifluoroacetic acid-*d*/deuterium oxide (1/9) allowed us to obtain compound **9**-d_3 (at least 98% isotopically pure trideuterated alkane) in 52% yield.

Scheme II. Deuterium Labeling by Denitration–Deoxygenation of α-Nitro Ketones



Experimental Section

General. Tetrahydrofuran (THF) was distilled from sodium/benzophenone immediately prior to use. Gas chromatographic analyses were performed on a capillary column of Duran glass (0.32 mm × 25 mt), stationary phase OV1 (film thickness 0.4–0.45 nm). All ^{13}C and ^1H NMR spectra were recorded, in $CDCl_3$ as solvent, at 300 MHz. Melting point (Pyrex capillary) and boiling points are uncorrected. (*p*-Tolylsulfonyl)hydrazine, trifluoroacetic acid-*d*, lithium aluminum hydride, lithium aluminum deuteride, and deuterium oxide were purchased from Aldrich Chimica. The α-nitro ketones **1**, **4**, **7**, **10**, and **13** were prepared from aldehyde and nitroalkanes as previously reported.[9] 2-Nitrocyclopentadecanone (**16**) was obtained by nitration of enol acetate of cyclopentadecanone.[16] (*Z*)-9-Octadecenal (oleic aldehyde) (**19**) was prepared by oxidation of commercial oleic alcohol with pyridinium chlorochromate in the presence of sodium acetate.[17]

Preparation of α-Nitro Ketone (*p*-Tolylsulfonyl)hydrazones. General Procedure. A solution of (*p*-tolylsulfonyl)hydrazine (1.65 g, 8.9 mmol) in methanol (10 mL) was added to a solution of the α-nitro ketone (8.53 mmol) in methanol (5 mL), and the mixture was stirred for 10 h; then water was added to obtain the corresponding (*p*-tolylsulfonyl)hydrazone, which was recrystallized from methanol/water.

2-Nitro-5-phenyl-3-pentanone (*p*-tolylsulfonyl)hydrazone (2): yield 90%; mp 104–105 °C; analytical data are in agreement with those previously reported.[3]

1-Phenyl-4-nitro-3-dodecanone (*p*-tolylsulfonyl)hydrazone (5): yield 96%; mp 94–95 °C; IR (KBr) ν 3210 (NH), 1600 (C=C), 1550 cm^{-1} (NO_2); ^1H NMR δ 0.88 (t, 3 H, *J* = 7 Hz), 1.05–2.27 (m, 18 H), 2.43 (s, 3 H), 4.85 (m, 1 H), 7.00–7.90 (m, 9 H). Anal. Calcd for $C_{25}H_{35}N_3O_4S$: C, 63.40; H, 7.45; N, 8.87; S, 6.75. Found: C, 63.54; H, 7.59; N, 8.98; S, 6.61.

2-Methyl-2-nitro-5-phenyl-3-pentanone (*p*-tolylsulfonyl)hydrazone (8): yield 65%; mp 142–143 °C; analytical data are in agreement with those previously reported.[3]

2-Nitro-3-tetradecanone (*p*-tolylsulfonyl)hydrazone (11): yield 93%; mp 103–104 °C; IR (KBr) ν 3220 (NH), 1600 (C=C), 1550 cm^{-1} (NO_2); ^1H NMR δ 0.85 (t, 3 H, *J* = 6.5 Hz), 1.15–1.5 (m, 18 H), 2.65 (d, 3 H, *J* = 6.8 Hz), 2.1–2.25 (m, 2 H), 2.43 (s, 3 H), 5.12 (q, 1 H, *J* = 6.8 Hz), 7.57 (dd, AA'BB' pattern, 4 H, *J* = 8 Hz), 8.1 (s, 1 H). Anal. Calcd for $C_{21}H_{35}N_3O_4S$: C, 59.27; H, 8.29; N, 9.88; S, 7.52. Found: C, 59.50; H, 8.42; N, 9.72; S, 7.43.

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6-Nitro-7-octadecanone (*p*-tolylsulfonyl)hydrazone (14): yield 95%; mp 63–65 °C; IR (KBr) ν 3260 (NH), 1600 (C=C), 1555 cm^{-1} (NO_2); ^1H NMR δ 0.72–0.92 (m, 6 H), 0.95–2.6 (m, 28 H), 2.42 (s, 3 H), 5.0 (m, 1 H), 7.57 (dd, AA'BB' pattern, 4 H, J = 8 Hz), 8.2 (s, 1 H). Anal. Calcd for $C_{25}H_{43}N_3O_4S$: C, 62.34; H, 9.00; N, 8.73; S, 6.64. Found: C, 62.45; H, 9.12; N, 8.59; S, 6.50.

2-Nitrocyclopentadecanone (*p*-tolylsulfonyl)hydrazone (17): yield 98%; mp 120–121 °C; IR (KBr) ν 3240 (NH), 1600 (C=C), 1552 cm^{-1} (NO_2); ^1H NMR δ 1.1–1.55 (m, 22 H), 2.05–2.4 (m, 4 H), 2.42 (s, 3 H), 5.0 (t, 1 H, J = 7.5 Hz), 7.57 (dd, AA'BB' pattern, 4 H, J = 8 Hz), 8.2 (s, 1 H). Anal. Calcd for $C_{22}H_{35}N_3O_4S$: C, 60.39; H, 8,06; N, 9.61; S, 7.31. Found: C, 60.50; H, 8.15; N, 9.48; S, 7.47.

Reductive Denitration–Deoxygenation of α-Nitro Ketone (*p*-Tolylsulfonyl)hydrazones to Alkanes. General Procedure. To a solution of lithium aluminum hydride (0.3 g, 7.92 mmol) was added dry THF (30 mL), the mixture was stirred and cooled to 0 °C, and, under N_2 a solution of (*p*-tolylsulfonyl)-hydrazine (2.64 mmol) in dry THF (20 mL) was added dropwise. The mixture was stirred for 10 h at 60 °C, then treated carefully with cold water (25 mL), acidified with 2 N HCl, and extacted with *n*-pentane (3 × 30 mL). The organic layer was dried with Na_2SO_4, the solvent was removed by distillation, and the crude product was purified by short chromatography, over silica gel, using *n*-pentane as eluent.

Pentylbenzene (3): yield 70%; bp_{60} 115 °C (lit.[18] bp 185–186 °C).

Dodecylbenzene (6): yield 61%; $bp_{0.2}$ 120 °C (lit.[22] bp_1 148 °C).

(4-Methylpentyl)benzene (9): yield 67%; bp_{10} 90 °C (lit.[23] bp_{767} 219 °C).

***n*-Tetradecane (12)**: yield 65%; bp_{20} 137 °C (lit.[20] $bp_{0.003}$ 60–64 °C).

***n*-Octadecane (15).** yield 68%; bp_8 168 °C (lit.[19] $bp_{0.15}$ 99–104 °C); ^1H NMR δ 0.89 (t, 6 H, J = 7 Hz), 1.1–1.35 (m, 32 H); ^{13}C NMR δ 14.107, 22.721, 29.408, 29.704, 29.746, 31.968; MS (*m/e*, relative intensity) 254 (M^+, 3), 141 (5), 127 (7), 113 (12), 112 (4), 99 (23), 98 (8), 97 (100), 85 (86), 84 (11), 83 (12).

Cyclopentadecane (18): yield 71%; mp 60–61 °C (lit.[21] mp 62–61 °C).

(*Z*)-19-Nitro-9-tricosen-18-one (21). A mixture of **19** (6.65 g, 25 mmol) and 1-nitropentane (**20**) (2.92 g, 25 mmol) was mechanically stirred for 5 min and then cooled in an ice bath. After the addition of chromatographic alumina (activity I, 5 g) and stirring at room temperature for 24 h, CH_2Cl_2 (50 mL) and tetra-*n*-butylammonium hydrogen sulfate (0.85 g, 2.5 mmol) were added. Under stirring and cooling to −10 °C, 30% H_2SO_4 (65 mL) and potassium dichromate (9.55 g, 32.5 mmol) were simultanously added. After stirring for 2 h at −10 °C, aqueous $FeSO_4$ (40 mL) was added, and the organic layers were separated. The organic phase was dried (Na_2SO_4) and passed through a bed of Florisil. The solvent was removed under reduced pressure to afford the crude α-nitro ketone **21** (8.57 g), which does not need purification. However, after chromatography (cyclohexane–ethyl acetate, 8:2) 8.1 g (85%) of the pure (*Z*)-19-nitro-9-tricosen-18-one (**21**) was obtained as an oil: IR (neat) ν 1730 (CO), 1552 (NO_2); ^1H NMR δ 0.9 (2t, 6 H, J = 6.8 Hz), 1.1–2.12 (m, 32 H), 2.55 (m, 2 H), 5.08–5.15 (m, 1 H, J = 4.7 Hz), 5.28–5.42 (m, 2 H). Anal. Calcd for $C_{23}H_{43}NO_3$: C, 72.39; H, 11.36; N, 3,67. Found: C, 72.55; H, 11.49; N, 3.50.

(*Z*)-19-Nitro-9-tricosen-18-one (*p*-Tolylsulfonyl)hydrazone (22). To a solution of compound **21** (3 g, 7.874 mmol) in methanol was added (*p*-tolylsulphonyl)hydrazine (1.54 g, 8.26 mmol). The mixture was stirred for 10 h at room temperature and then, after workup as in the general procedure, 3.96 g (92%) of the pure compound **22** was obtained: mp 53–55 °C; IR (KBr) ν 3223 (NH), 1600 (C=C), 1550 (NO_2), 1340, 1170 (SO_2); ^1H NMR δ 0.88 (2t, 6 H, J = 6.8 Hz), 1.05–1.50 (m, 26 H), 1.85–2.05 (m, 4 H), 2.05–2.22 (m, 4 H), 2.42 (s, 3 H), 4.95–5.02 (m, 1 H, J = 4.6 Hz), 5.25–5.42 (m, 2 H), 7.57 (dd, AA'BB' pattern, 4 H, J = 8 Hz), 7.9 (s, 1 H). Anal. Calcd for $C_{30}H_{51}N_3O_4S$: C, 65.54; H, 9.35; N, 7.64; S, 5.82. Found: C, 65.71; H, 9.60; N, 7.45; S, 6.01.

(*Z*)-9-Tricosene (23). A suspension of $LiAlH_4$ (0.49 g, 10.5 mmol), in dry THF (40 mL) was stirred under nitrogen and cooled to 0 °C. The tosylhydrazone **25** (1 g, 1.82 mmol) was dissolved in dry THF (20 mL) and added dropwise. The mixture was stirred for 0.5 h at 0 °C and for 10 h at 60°C; then, after workup as in the general procedure, the crude product obtained was purified by short chromatography using *n*-pentane as eluent. Kugelrohr distillation at 170 °C (0.1 min) gave 0.38 g (66%) of pure (*Z*)-9-tricosene (Muscalure) (**23**) (lit.[13a] $bp_{0.01}$ 140–142 °C): ^1H NMR δ 0.88 (2t, 6 H, J = 6.8 Hz), 1.11–1.42 (m, 34 H), 1.9–2.11 (m, 4 H), 5.28–5.41 (m, 2 H); ^{13}C NMR δ 14.11, 22.72, 27.225, 29.36, 29.41, 29.57, 29.61, 29.71, 29.74, 29.81, 31.96, 129.87; MS (*m/e*, relative intensity) 322 (M^+, 4), 125 (20), 112 (11), 111 (45), 98 (19), 97 (94), 85 (31), 84 (32), 83 (100), 82 (26), 71 (46), 70 (47), 69 (88).

Deuterium Labeling by Denitration–Deoxygenation of α-Nitro Ketone. Reduction of 8 to (4-Methyl-3,4-dideuteriopentyl)benzene (9-d_2). A suspension of $LiAlD_4$ (0.158 g, 3.96 mmol) in dry THF (15 mL) was stirred under nitrogen in a 100-mL flask fitted with a septum inlet and cooled to 0 °C. The tosylhydrazone **20** (0.513 g, 1.32 mmol) was dissolved in dry THF (10 mL) and added dropwise. The mixture was refluxed at 60 °C for 10 h, then cooled, treated carefully with cold water (10 mL), acidified with 2 N HCl, and extracted with *n*-pentane (3 × 10 mL). The organic layer was dried ($MgSO_4$), the solvent was removed by distillation, and the crude product **9**-d_2 was purified by short chromatography using *n*-pentane as eluent: yield 0.12 g (55%); ^1H NMR δ 0.88 (s, 6 H), 1.2–1.35 (m, 1 H), 1.55–1.7 (m, 2 H), 2.58–2.63 (m, 2 H), 7.15–7.35 (m, 5 H); ^{13}C NMR δ 22.45, 27.317, 29.25, 36.24, 37.87, 38.12, 38.37, 125.55, 128.23, 128.32, 128.38, 128.46, 142.98; MS (*m/e*, relative intensity) 164 (M^+, 20), 105 (5), 94 (12), 93 (37), 92 (47), 91 (100).

(4-Methyl-3,3,4-trideuteriopentyl)benzene (9-d_3). The procedure was the same as in case of the compound **9**-d_2 except that trifluoroacetic acid-*d*/deuterium oxide (1:9) was used for the quenching of the reaction in place of cold water and 2 N HCl: yield 52%; ^1H NMR δ 0.88 (s, 6 H), 1.52–1.68 (m, 2 H), 2.55–2.63 (m, 2 H), 7.13–7.25 (m, 5 H); ^{13}C NMR δ 22.425, 29.15, 36.21, 125.56, 128.22, 128.38, 142.983; MS (*m/e*, relative intensity) 165 (M^+, 82), 107 (28), 95 (31), 94 (77), 93 (90), 91 (100), 79 = 78 (32), 77 (37), 65 (64).

Acknowledgment. We thank the Ministero della Pubblica Istruzione of Italy for financial support of this work.

Supplementary Material Available: Characterization data (^1H NMR, ^{13}C NMR, and MS) of compounds **3**, **6**, **9**, **12**, **15**, and **18** (1 page). Ordering information is given on any current masthead page.

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NMR Spectroscopy of Malondialdehyde[1]

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β-Dicarbonyl compounds tend to exist as stable enols,[2] which creates the possibility of cis–trans isomerism about

(1) Part 8 in the series Chemistry under Physiological Conditions. Part 7 is ref 7 below.

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