allowed to cool and the solid triturated with anhydrous ethyl ether, filtered, and washed with ethyl ether. The residue was dissolved in the minimum amount of water and the salt precipitated by the addition of a copious quantity of acetone. This suspension was filtered and dried to yield crude light-brown V (8.25 g, 50%). Crude V was decolorized with activated carbon and recrystallized from isopropyl alcohol to give colorless needles: mp >330; NMR (F_3CCO_2H) δ 8.24 (AA′BB′ system, 4 H), 2.70 (oct, 12 H, J = 12.8 Hz), 1.36 (hex, 18 H, J = 19 Hz); IR (KBr) 1450, 1115 cm^{-1}, characteristic of a phosphonium salt. Anal. ($C_{18}H_{34}P_2Br_2$) P, Br.

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Registry No.—V dibromide, 63216-62-6; $V^{+\cdot}$, 63216-63-7; triethylphosphine, 554-70-1; *p*-dibromobenzene, 106-37-6.

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Quantitative Dealkylation of Alkyl Ethers via Treatment with Trimethylsilyl Iodide. A New Method for Ether Hydrolysis

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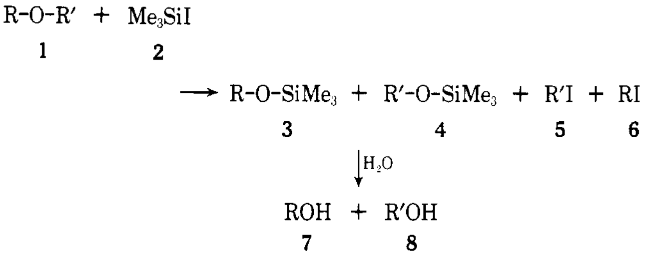
Many alkyl ethers have found extensive use in organic chemistry as protecting groups for alcohols.[1] Foremost among these are *tert*-butyl, triphenylmethyl (trityl), and benzyl ethers, all of which can be removed under relatively mild conditions, i.e., nonaqueous acid, mild aqueous acid, and hydrogenation, respectively.[2] Certain other alkyl ethers have also been employed to advantage on occasion, e.g., allyl,[3] tritylone,[4] *p*-halophenyl,[5] and similar systems.[6] However, simple methyl ethers have found little use in the protection of aliphatic hydroxyl groups (although they are quite extensively utilized for the protection of phenols) due to the difficulties associated with their removal. This fact has promoted the following statement which appeared in a recent review[1a] on protecting groups for aliphatic alcohols: "the methyl group is too stable to be used for the routine protection of alcohols". We report now observations which are contrary to the above statement, namely, the simple and efficient dealkylation of methyl and other alkyl ethers by trimethylsilyl iodide in both aliphatic and aromatic systems, the overall process affording ether hydrolysis in high yields.

Recently, several new modifications of long-standing procedures have been published which increase somewhat the usefulness of aliphatic alkyl ethers as protecting groups. These include the generation of HI in situ[7] and the use of acetic anhydride–Lewis acid media.[8] Boron trihalides have been used occasionally to cleave aliphatic methyl ethers,[9] although the yields in these cases are somewhat lower than with aromatic methyl ethers.[10] However, these methods do not solve the problem of clean and efficient demethylation of alkyl methyl ethers, since they often result in mixtures of dealkylated products.

We have very recently reported the simple and quantitative dealkylation of alkyl carboxylic esters by treatment with trimethylsilyl iodide to afford trimethylsilyl carboxylates, which upon addition of water furnish carboxylic acids.[11] We find that alkyl methyl ethers **1** (R′ = Me) also react with trimethylsilyl iodide **2** to afford mixtures of dealkylated products, the trimethylsilyl ethers **3** and **4** and the alkyl iodides **5** and **6**, in which, in general, the demethylation products **3** and **5** (methyl iodide) greatly predominate.[12] For example, cyclohexyl methyl ether **1a** when treated with 1.1 equiv of trimethylsilyl iodide **2** at 25 °C for 6 h affords 95% of the demethylation products, cyclohexyl trimethylsilyl ether **3a** and methyl iodide **5**, with only 5% of cyclohexyl iodide **6a** and methyl trimethylsilyl ether **4** being formed. It is interesting that at a somewhat higher temperature (50 °C) the reaction is complete in only 2 h, but now affords a 90:10 mixture of cyclohexyl silyl ether and methyl silyl ether. Aryl alkyl ethers (**1q–z**) are all cleaved unidirectionally when treated with trimethylsilyl iodide to afford only the aromatic trimethylsilyl ethers (**3q–z**) and the alkyl iodides (**5q–z**) in very high yields. In all cases, the silyl ethers **3** and **4** can be easily converted into the alcohols or phenols **7** and **8** in high yield upon simple hydrolysis.



$$\underset{1}{\mathrm{R{-}O{-}R'}} + \underset{2}{\mathrm{Me_3SiI}} \longrightarrow \underset{3}{\mathrm{R{-}O{-}SiMe_3}} + \underset{4}{\mathrm{R'{-}O{-}SiMe_3}} + \underset{5}{\mathrm{R'I}} + \underset{6}{\mathrm{RI}}$$

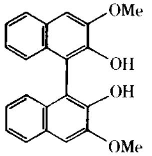
$$\downarrow \mathrm{H_2O}$$

$$\underset{7}{\mathrm{ROH}} + \underset{8}{\mathrm{R'OH}}$$

The results with a series of methyl ethers and other alkyl ethers **1a–z** are listed in Table I. Several conclusions can be made from this data: (1) trityl, benzyl, and *tert*-butyl ethers are cleaved at a very much faster rate than the other alkyl ethers (methyl, ethyl, isopropyl, cyclohexyl, etc.), thus permitting the former to be selectively hydrolyzed in the presence of the latter; (2) aryl alkyl ethers (e.g., anisole) react with trimethylsilyl iodide at a significantly slower rate than dialkyl ethers (e.g., cyclohexyl methyl ether), so that dialkyl ethers can, in general, be cleaved completely under conditions which cause only 5–10% clevage of phenolic ethers; (3) the rates of dealkylation are such that alkyl methyl ethers can be cleaved cleanly in the presence of methyl esters[11] by conducting the reaction with just slightly more than 1 equiv of the silyl iodide at room temperature or below; (4) as was the case with alkyl esters,[11] many functional groups are stable to the conditions for ether hydrolysis, including acetylenes, olefins, ketones, amines, aromatic halides; (5) by careful variation of solvent, one can cleanly demethylate methyl ethers of straight-chain secondary alcohols, e.g., methyl 2-octyl ether **1g**, since the use of a propene–acetonitrile solution permitted the production of predominately 2-octanol.

As in the case of dealkylation of esters,[11] the possibility that the observed reactions are due entirely to catalytic amounts of HI present in the trimethylsilyl iodide cannot be totally discounted. However, we believe that trimethylsilyl iodide itself is causing dealkylation because, in the presence of 15 mol % pyridine or 2,6-di-*tert*-butyl-4-methylpyridine,[15] ether

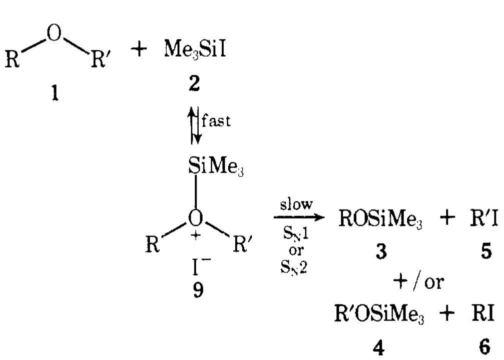
Table I. Dealkylation of Ethers by Trimethylsilyl Iodide:[a] $\underset{1}{ROR'} + \underset{2}{Me_3SiI} \rightarrow \underset{3}{ROSiMe_3} + \underset{4}{R'OSiMe_3} + \underset{5}{R'I} + \underset{6}{RI}$

Ethers							Products and Yields[c]							
Registry no.	Compd 1	R	R'	Temp (°C)	Solvent	Time (h)[b]	3	Registry no.	4	Registry no.	5	Registry no.	6	Registry no.
931-56-6	a	C_6H_{11}	Me	25	$CDCl_3$	6	95[d]	13871-89-1	5	1825-61-2	95	74-88-4	5	626-62-0
	a	C_6H_{11}	Me	50	CCl_4	2	90		10		90		10	
932-92-3	b	C_6H_{11}	Et	25	$CDCl_3$	12	48.7[e]		22.5	1865-62-3	77.5	75-03-6	51.3	
	b	C_6H_{11}	Et	25	$CDCl_3$	16	0		0		100		100	
1860-29-3	c	C_6H_{11}	*i*-Pr	25	CH_2Cl_2	48	0[e]		0		100	75-30-9	100	
25246-83-7	d	C_6H_{11}	*t*-Bu	25	CCl_4	<0.1	100		0		100	558-17-8	0	
16224-09-2	e	C_6H_{11}	CH_2Ph	25	$CDCl_3$	<0.1	100		0		100		0	
20705-40-2	f	C_6H_{11}	CPh_3	25	$CDCl_3$	<0.1	100		0		100	2206-53-3	0	
1541-09-9	g	$CH_3(CH_2)_5CHCH_3$	Me	25	CD_3CN	8.5	86[f]	18023-52-4	0		100		7	557-36-8
1981-90-4	h	3β-cholestanyl	Me	25	$CHCl_3$	12	100[g]	18880-51-8	0		100		0	
13482-23-0	i	4-oxocyclohexyl	Me	25	$CDCl_3$	2.5	100	23510-94-3	0		100		0	
110-71-4	j	CH_2CH_2 (glyme)	2Me	25	$CDCl_3$	9	67[h]	7381-30-8	33[h]	26305-99-7	100[h]		0	
63250-84-0	k	$HC{\equiv}CC(Me)_2$-$C{\equiv}CCH_2$	Me	25	$CDCl_3$	1.3	0		0		100		100	63250-91-9
61478-24-8	l	$PrCOCHEtCH_2$[i]	*t*-Bu	25	$CDCl_3$	<0.1	100	63250-87-3	0		100		0	
61478-23-7	m	*i*-$PrCOC(Me)_2CH_2$[i]	*t*-Bu	25	$CDCl_3$	<0.1	100	63250-88-4	0		100		0	
63250-85-1	n	Ph_3CCH_2CO-$(CH_2)_5$	CPh_3	25	$CDCl_3$	<0.1	100	63250-89-5	0		100		0	
60-29-7	o	C_2H_5	Et	25	$CDCl_3$	70	0		0		100		100	
109-99-9	p	$-CH_2(CH_2)_2CH_2-$		25	$CDCl_3$	<0.1	100	18001-91-7	0		100		0	
	p	$-CH_2(CH_2)_2CH_2-$		25	$CDCl_3$	75	0		0		100		100	628-21-7
100-66-3	q	C_6H_5	Me	25	$CDCl_3$	48	100	1529-17-5	0		100		0	
	q	C_6H_5	Me	50	$CDCl_3$	21	100		0		100		0	
104-93-8	r	*p*-C_6H_4	2Me	25	$CDCl_3$	30	100	17902-32-8	0		100		0	
578-57-4	s	*o*-BrC_6H_4	Me	25	$CDCl_3$	125	100	36601-47-5	0		100		0	
2398-37-0	t	*m*-BrC_6H_4	Me	25	$CDCl_3$	120	30[j]	36971-28-5	0		30		0	
104-92-7	u	*p*-BrC_6H_4	Me	25	$CDCl_3$	120	100	17871-44-3	0		100		0	
90-04-0	v	*o*-$NH_2C_6H_4$	Me	50	$(CH_2)_4SO_2$	12	100	36309-44-1	0		100		0	
536-90-3	w	*m*-$NH_2C_6H_4$	Me	50	$(CH_2)_4SO_2$	22	100	36309-43-0	0		100		0	
104-94-9	x	*p*-$NH_2C_6H_4$	Me	50	$(CH_2)_4SO_2$	22	100	36309-42-9	0		100		0	
621-32-9	y	*m*-$CH_3C_6H_4$	Et	50	CCl_4	140	100	17902-31-7	0		100		0	
63250-86-2	z			60	$CDCl_3$	26	100[k]	63250-90-8	0		100		0	63250-92-0

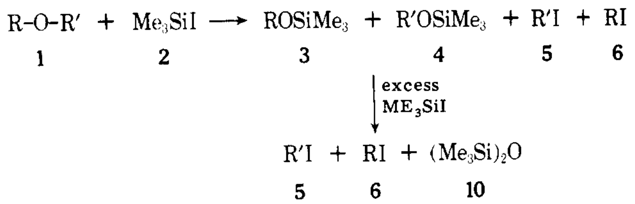
[a] Approximately 1.3 equiv of trimethylsilyl iodide was used in all simple dealkylation experiments. When converting the ethers completely into the two alkyl iodides, approximately 2.5 equiv was employed. [b] The times required for the transformations listed could all be decreased by utilizing a large excess of trimethylsilyl iodide or by using higher temperatures. [c] The yields listed were determined by NMR integration of the pertinent peaks. [d] Starting with 1.5 g of the ether, 1.00 g of cyclohexanol was isolated (column chromatography on silica gel), implying a 76% yield. [e] Examination of the NMR spectra at intermediate times indicates that the ethyl and isopropyl groups are dealkylated at a faster rate than the cyclohexyl group. [f] In this experiment there was still starting material present (7%), due to the insufficient amount of trimethylsilyl iodide employed. Starting with 216 mg of the ether, 119 mg of 2-octanol was isolated (column chromatography on silica gel), implying a 61% yield. See Experimental Section. [g] Starting with 300 mg of the ether, 232 mg of 3β-cholestanol was isolated (column chromatography on silica gel) and indentified as pure by comparison to an authentic sample, implying a yield of 80%. [h] The bis(trimethylsilyl ether) of ethylene glycol is designated 3j, the trimethylsilyl ether of 2-iodoethanol 4j, and methyl iodide 5j. [i] Prepared from the corresponding bis(*tert*-butyl) or bis(trityl) ether by trityl salt oxidation.[18] [j] In this experiment after 5 days there was still a large amount of starting material left (70%), even though a slight excess of trimethylsilyl iodide was still present. [k] The product of dealkylation of the optically active (*R*)-ether was the optically active tetrol, isolated in 95% yield with no loss in optical purity. See Experimental Section.

dealkylation does occur albeit at a slower rate. Furthermore, dealkylation occurs facilely in a propene-saturated carbon tetrachloride solution, in which even trace amounts of HI are trapped out as isopropyl iodide.

We suggest a rather straightforward mechanism for this process: the ether **1** reacts with trimethylsilyl iodide **2** in a fast and reversible step to produce the silylated oxonium iodide **9** which can then go on to products in a slow, irreversible process by either an S_N2 mechanism or an S_N1 (S_Ni) mechanism.



If the alkyl trimethylsilyl ethers, **3** and **4**, are allowed to react with excess trimethylsilyl iodide at 50 °C for longer periods of time, they are efficiently converted into the corresponding alkyl iodides, **5** and **6**, respectively, and hexamethyldisiloxane (**10**). Thus, if desired one can easily convert both alkyl residues in a dialkyl ether into the two corresponding alkyl iodides. For example, compounds **1b**, **1c**, **1k**, **1o**, and **1p** in Table I have been converted totally into the two alkyl iodides in good yields as shown. This then provides a convenient synthesis of iodides directly from ethers.



$$\underset{1}{\text{R-O-R}'} + \underset{2}{\text{Me}_3\text{SiI}} \longrightarrow \underset{3}{\text{ROSiMe}_3} + \underset{4}{\text{R}'\text{OSiMe}_3} + \underset{5}{\text{R}'\text{I}} + \underset{6}{\text{RI}}$$

$$\xrightarrow{\text{excess ME}_3\text{SiI}} \underset{5}{\text{R}'\text{I}} + \underset{6}{\text{RI}} + \underset{10}{(\text{Me}_3\text{Si})_2\text{O}}$$

Trimethylsilyl iodide **2** can be conveniently synthesized in two steps from the readily available trimethylsilyl chloride.[11] This procedure is readily applicable for the preparation of fairly large amounts of this compound[16] (see Experimental Section). We believe that the simplicity and high yields of this method will cause it to be quite useful to chemists interested in synthesis.[17]

Experimental Section

Trimethylsilyl Iodide. A variation of Vornokov's original procedure was used.[19] Aluminum powder (5.6 g, 0.21 mol) and hexamethyldisiloxane[20] (16.2 g, 0.1 mol) are placed in a 250-mL round-bottom flask equipped with a solid addition funnel, reflux condenser, magnetic stirrer, and nitrogen inlet. The flask is warmed in an oil bath to 60 °C. Iodine (50.8 g, 0.2 mol) is added piecewise over a period of 55 min via the solid addition funnel. The mixture is refluxed for 90 min (bath temperature 140 °C). The reflux condenser is then replaced by a distilling head. Distillation at atmospheric pressure (bath temperature 140–210 °C) with collection at 0 °C gives 35.0 g (87.5%) of trimethylsilyl iodide (**2**), a clear colorless liquid, bp 106 °C: NMR ($CDCl_3$) δ 0.8 (s, 9 H).

General Experimental Procedure. Reactions were normally conducted on a 1 mmol scale in an NMR tube with a tightly sealed cap or on a 20–30 mmol scale in a round-bottom flask under nitrogen as follows. To a 2 M solution containing 1 equiv of the ether **1** in the indicated solvent (see Table I) was added 1.3 equiv of neat trimethylsilyl iodide **2** via a dry syringe. The reaction was maintained at the indicated temperature (see Table I) and monitored by NMR analysis for the given period of time (see Table I). Yields were calculated by NMR integration of the pertinent peaks. For isolation of the alcohols, at the completion of the reaction, the excess trimethylsilyl iodide was destroyed and the intermediate trimethylsilyl ethers **3** formed during the reaction were hydrolyzed to the alcohols **7** by pouring the reaction mixture into 4 equiv of methanol. The volatile components were removed at reduced pressure and the residue was taken up in diethyl ether, washed with aqueous sodium bisulfite, aqueous sodium bicarbonate, and brine, and dried. The residue left after evaporation of solvent was further purified (if necessary) by column chromatography on silica gel. In the case of the alkyl cyclohexyl ethers, the extraction and washings were omitted and the residue after evaporation of the methanol was chromatographed directly.

The following are specific experimental procedures.

1,1′-Binaphthalene-2,2′,3,3′-tetrol. To 52.0 mg (0.15 mmol) of (+)-(*R*)-diol **1z** in an NMR tube was added 0.5 mL of $CDCl_3$ to give a clear solution. Then 0.1 mL (0.75 mmol) of trimethylsilyl iodide was added. The solution was maintained at 60 °C by an oil bath, and the reaction was monitored by NMR periodically. The reaction was apparently complete after 26 h as indicated by the absence of a signal for the OCH_3 protons in the NMR spectrum and the presence of a signal at 2.15 for protons of CH_3I. The brown-purple solution was filtered and $\simeq$0.25 mL of MeOH was added to the filtrate. After 1 day, the solvent was removed by rotary evaporation to give 45.4 mg (95%) of a pink solid, which was identified as (+)-(*R*)-tetrol by its NMR spectrum and its specific rotations, which were identical to those of an authentic sample.

2-Octanol. Methyl 2-octyl ether **1g** (0.216 g, 1.5 mmol) was placed in an NMR tube and $\simeq$0.2 mL of propene was condensed into the tube at −78 °C. To this at −78 °C was added the supernatant liquid from the centrifugation of 1.0 mL CD_3CN and 0.65 mL (5.0 mmol) of trimethylsilyl iodide (all transfers performed with clean dried syringes). After addition of the Me_3SiI/CD_3CN solution, the NMR tube was allowed to warm to room temperature (allowing excess propene to evaporate). The reaction was monitored by NMR. After 8.5 h, the reaction was quenched by pouring the contents of the NMR tube into 1 mL of MeOH through which gaseous HCl had been passed for 5 s. The volatiles were removed on the rotary evaporator. NMR analysis of the residue indicated 86% 2-octanol, 7% 2-octyl iodide, and 7% methyl 2-octyl ether. Chromatography on 30 g of silica gel, elution with anhydrous diethyl ether, and evaporation of the solvent from the combined alcohol-containing fractions afforded 0.119 g (61%) of 2-octanol, identical with an authentic sample.

Acknowledgment. We thank Mr. Paul Ornstein for performing several preparative and dealkylative experiments and Dr. Thomas Tarnowski for performing the reaction involving the binaphthol **1z**.[21]

Registry No.—Trimethylsilyl iodide, 16029-98-4; cyclohexanol, 108-93-0; 2-octanol, 123-96-6; 3β-cholesterol, 80-97-7; hexamethyldisiloxane, 63250-9301; (+)-(*R*)-1,1′-dinaphthalone-2,2′,3,3′-tetrol, 63323-58-0.

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(20) Hexamethyldisiloxane is prepared by hydrolysis of trimethylsilyl chloride in 86% yield [R. O. Sauer, *J. Am. Chem. Soc.*, **66,** 1707 (1944)]. It is also commercially available from Petrarch Systems and Silar Laboratories, Inc.

(21) This work was presented in part at the 173rd National Meeting of the American Chemical Society, New Orleans, La., March 1977, abstract ORGN89.

Monohalogenation of Primary Nitroparaffins

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Treatment of the salts of primary nitroparaffins with halogens generally gives the monohalonitro compound contaminated with greater or lesser amounts of the dihalonitro compound and the starting nitroparaffin. In 1960 Trippett and Walker[1] reported that pure 1-bromo-1-nitroalkanes could be prepared by addition of the dry, finely powdered, sodium or ammonium salt of the nitro compound to bromine at 0 °C; they obtained 65–70% yields of pure products. Several years later, Levering[2] devised a procedure for chlorinating the sodium salt of nitroethane, which gives excellent yields of pure 1-chloronitroethane. Unfortunately, his procedure depends on density differences between the monochloronitro compound and the solution of nitroparaffin salt; these differences quickly become relatively small as the molecular weight increases, so that even the preparation of pure 1-chloro-1-nitrobutane is no longer a simple matter. And still more recently, Novikov et al.[3] proposed the use of *N*-halo-*N*-nitroamines or *N*-halosuccinimides as reagents for monohalogenating the salts of primary nitroparaffins, but the purity of their products cannot be regarded as established.

We now report a simple, convenient procedure for chlorinating, brominating, and iodinating primary nitroparaffins; the yields of pure products range from 82 to 94%. All that is required is addition of a methylene chloride solution of the halogen, precooled to −78 °C, to a slurry of the nitroparaffin salt and ice. The reaction is complete in 1–2 min, the methylene chloride phase is dried, the solvent is removed, and the residue is subjected to a simple distillation. This gives analytically pure monohalonitro compound in the yields shown in Table I.

Experimental Section

Chlorination is exemplified by the preparation of 1-chloro-1-nitropropane.

1-Chloro-1-nitropropane. A solution of 22.2 g (337 mmol) of 85% potassium hydroxide in 120 mL of water is prepared under nitrogen in a 500-mL flask fitted with an efficient stirrer.[4] When this solution comes to room temperature 30.0 g (337 mmol) of 1-nitropropane is added all at once and the mixture is stirred under nitrogen until the nitro compound dissolves (ca. 15 min). Following this, the solution is cooled until it just starts to freeze (ca. −15°C), at which point ca. 300 mL of crushed ice is added, and then a solution of 20 mL (ca. 60 g) of liquid chlorine in 100 mL of methylene chloride, which has been cooled to −78 °C, is added all at once with vigorous stirring.[4] In about 1 min the reaction is over. After separation of the two layers, the aqueous phase is extracted with 50 mL of methylene chloride; the combined methylene chloride solutions are washed once with 50 mL of a saturated sodium chloride solution, and then dried over anhydrous magnesium sulfate. The methylene chloride and excess chlorine are removed by distillation through a short column, the column is removed, and the residue is distilled in vacuo. This gives 37.8 g (93% yield) of 1-chloro-1-nitropropane, bp 55 °C (26 mm). On VPC analysis only a single peak is obtained and elemental analysis (Table I) confirms the purity of this material.

Bromination may be carried out as for chlorination. Alternatively, a slightly modified procedure also works well, especially when small batches are being prepared. Instead of adding ice, it suffices to cool a larger volume of methylene chloride than would otherwise be used. The following procedure is illustrative.

1-Bromo-1-nitrooctane. In a 100-mL flask fitted with an efficient stirrer[4] a solution of 2.08 g (31.4 mmol) of 85% potassium hydroxide in 40 mL of 25% aqueous methanol[5] is prepared under nitrogen. At room temperature 5 g (31.4 mmol) of 1-nitrooctane is added, and the mixture is stirred under nitrogen until the nitro compound dissolves (ca. 40 min). The resulting solution is cooled until it just starts to freeze, and then 4.93 g (30.8 mmol) of bromine in 50 mL of methylene chloride (precooled to −78 °C) is added, all at once, with vigorous stirring.[4] After about 1 min the reaction is over; the layers are separated and the aqueous phase is extracted with 20 mL of methylene chloride. The combined methylene chloride solutions are washed with 20 mL of H_2O and dried over anhydrous magnesium sulfate, and then the solvent is removed. Distillation of the residue gives 6.64 g (89% yield) of analytically pure 1-bromo-1-nitrooctane; bp 75 °C (0.4 mm).

1-Iodo-1-nitroethane. To a solution of 4.40 g (66.7 mmol) of 85% KOH in 40 mL of water at 20–25 °C is added 5 g (66.7 mmol) of nitroethane all at once; the mixture is stirred under nitrogen until the nitro compound dissolves (ca. 10 min). With minimal exposure to light, the aqueous solution is cooled until freezing begins, and then 50 mL of CH_2Cl_2 (cooled to −78 °C) is added followed by 16.6 g (65.3 mmol) of powdered iodine (nitrogen atmosphere; vigorous stirring). In about 3 min the reaction is complete. After separating the layers, the aqueous phase is extracted with 20 mL of CH_2Cl_2, the combined methylene chloride solutions are dried (anhydrous $MgSO_4$), and then the solvent is removed by distillation through a short Vigreux column, first at atmospheric pressure and, finally, at 50 mm. The column is removed and the residue is distilled at 0.4 mm, whereupon 10.95 g (82% yield) of analytically pure 1-iodo-1-nitroethane is obtained (bp 42 °C). This compound soon develops a pink color.

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Table I. Monohalonitroparaffins

Compd	Registry no.	Yield, %	Bp, °C (mm)	Anal. Calcd	Anal. Found
1-Chloro-1-nitroethane	598-92-5	92	52 (45)	C, 21.94; H, 3.66; Cl, 32.36	C, 21.86; H, 3.73; Cl, 32.42
1-Chloro-1-nitropropane	600-25-9	94	55 (26)	C, 29.17; H, 4.86; Cl, 28.69	C, 29.38; H, 4.89; Cl, 29.00
1-Chloro-1-nitrooctane	63599-45-1	92	60 (0.4)	C, 49.74; H, 8.29; Cl, 18.34	C, 50.00; H, 8.25; Cl, 18.19
1-Bromo-1-nitroethane	563-97-3	89	48 (11)	C, 15.59; H, 2.60; Br, 51.92	C, 15.63; H, 2.68; Br, 51.79
1-Bromo-1-nitropropane	5447-96-1	92	50 (4)	C, 21.44; H, 3.57: Br, 47.59	C, 21.61; H, 3.65; Br, 47.39
1-Bromo-1-nitrooctane	63569-74-4	89	75 (0.4)	C, 40.35; H, 6.72; Br, 33.59	C, 40.48; H, 6.86; Br, 33.80
1-Iodo-1-nitroethane	51771-09-6	82	42 (0.4)	C, 11.94; H, 1.99; I, 63.18	C, 11.87; H, 1.87; I, 63.03