7.35 (dd, 4 H, arom, $J_{aa'xx'}$ = 6.70 Hz); mass spectrum *m/e* 197 $(M + 2)^+$, 195 (M^+), 166, 152, 127, 111, 83, 75, 42. Anal. Calcd for $C_{10}H_{10}ClNO$: C, 61.39; H, 5.15; N, 7.16. Found: C, 61.22; H, 5.30; N, 7.09; UV λ_{max} 298 nm (ϵ 20 670).

(*E*)-α-Ethenyl-α-methyl-*N*-(2-naphthyl)nitrone (8f): oil; yield 71%; IR (neat, cm^{-1}) 1505 (C=N), 1050 (N—O); ^1H NMR ($CDCl_3$) δ 2.45 (s, 3 H, N=CCH_3), 5.23 (d, 1 H, C=CH_2, J_{ax} = 11.20 Hz), 5.55 (d, 1 H, C=CH_2, J_{bx} = 17.10 Hz), 6.41 (dd, 1 H, N=CCH=C, J_{ax} = 11.20 Hz, J_{bx} = 17.10 Hz), 7.40–7.80 (m, 7 H, arom); mass spectrum, *m/e* 211 (M^+), 182, 166, 128, 115, 77, 63, 39. Anal. Calcd for $C_{14}H_{13}NO$: C, 79.59; H, 6.20; N, 6.63. Found: C, 79.30; H, 6.44; N, 6.50.

(*Z*)-α-Phenyl-*N*-(1-buten-3-yl)nitrone (10a): oil; yield 77%; IR (neat, cm^{-1}) 1450 (C=N), 1140 (N—O); ^1H NMR ($CDCl_3$) δ 2.65 (d, 3 H, CH_3, *J* = 6.70 Hz), 4.55 (quint, 1 H, NCH, *J* = 6.70 Hz), 5.30–5.43 (m, 2 H, =CH_2), 6.10–6.25 (m, 1 H, NCH=C), 7.35–7.45 (m, 3 H, arom + 1 H, ArCH=), 8.20–8.25 (m, 2 H, arom ortho); mass spectrum, *m/e* 175 (M^+), 145, 121, 104, 89, 77, 55, 39. Anal. Calcd for $C_{11}H_{13}NO$: C, 75.39; H, 7.47; N, 7.99. Found: C, 75.14; H, 7.32; N, 7.87.

(*Z*)-α-*n*-Pentyl-*N*-(1-buten-3-yl)nitrone (10b): oil; yield 90%; IR (neat, cm^{-1}) 1590 (C=N), 1165 (N—O); ^1H NMR ($CDCl_3$) δ 0.85 (t, 3 H, CH_3CH$_2$, *J* = 7.35 Hz), 1.10–1.25 (m, 6 H, $CH_2CH_2CH_2$), 1.45 (d, 3 H, NCHCH_3, *J* = 6.75 Hz), 2.35–2.45 (m, 2 H, CH_2CH=N), 4.30 (quint, 1 H, NCH, *J* = 6.75 Hz), 5.15–5.30 (m, 2 H, =CH_2), 5.95–6.10 (m, 1 H, CH=C), 6.65 (t, 1 H, CH=N, *J* = 4.65 Hz); mass spectrum, *m/e* 169 (M^+), 152, 126, 98, 57, 55, 43, 41, 39. Anal. Calcd for $C_{10}H_{19}NO$: C, 70.96; H, 11.31; N, 8.27. Found: C, 70.78; H, 11.21; N, 8.12.

(*Z*)-α-*n*-Propyl-*N*-(1-buten-3-yl)nitrone (10c): oil; yield 58%; IR (neat, cm^{-1}) 1560 (C=N), 1165 (N—O); H NMR ($CDCl_3$) δ 0.95 (t, 3 H, CH_3CH$_2$, *J* = 7.35 Hz, 1.20–1.30 (m, 2 H, CH_3CH_2), 155 (d, 3 H, NCHCH_3, *J* = 6.75 Hz), 2.40–2.50 (m, 2 H, $C_2H_5CH_2$), 4.40 (quint, 1 H, NCH, *J* = 6.75 Hz), 5.25–5.30 (m, 2 H, =CH_2), 6.00–6.15 (m, 1 H, C*H*=CH_2), 6.62 (t, 1 H, CH=N, *J* = 4.65 Hz); mass spectrum, *m/e* 141 (M^+), 124, 98, 82, 72, 55, 41, 39. Anal. Calcd for $C_8H_{15}NO$: C, 68.04; H, 10.70; N, 9.91. Found: C, 67.85; H, 10.89; N, 9.75.

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Registry No. **1a**, 88-72-2; **1b**, 88-73-3; **1c**, 81-20-9; **1d**, 577-19-5; **1e**, 100-00-5; **1f**, 581-89-5; **8a**, 127279-63-4; **8b**, 127279-64-5; **8c**, 127309-72-2; **8d**, 127279-65-6; **8e**, 127279-66-7; **8f**, 127279-67-8; **9** (R = Ph), 622-42-4; **9** (R = *n*-C_5H_{11}), 646-14-0; **9** (R = *n*-C_3H_7), 627-05-4; **10a**, 127279-68-9; **10b**, 127279-69-0; **10c**, 127279-70-3; **12**, 127279-71-4; $H_3CCH=CHCH_2MgCl$, 6088-88-6.

Synthesis of 2,2,4,4-Tetranitroadamantane

Paritosh R. Dave* and Mark Ferraro

GEO-CENTERS, INC. at ARDEC, 762 Route 15 South, Lake Hopatcong, New Jersey 07849

Herman L. Ammon

Department of Chemistry and Biochemistry, University of Maryland, College Park, Maryland 20742

C. S. Choi

Energetics and Warhead Division, ARDEC, Picatinny Arsenal, New Jersey 07806

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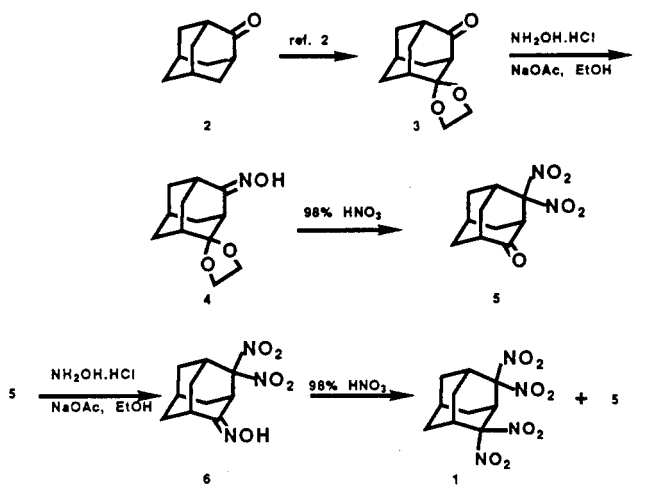
There is considerable current interest in the synthesis and chemistry of polynitropolycycles.[1] The compounds are primarily of interest because of their energetic properties, that is, they can function as explosives, propellants, and/or fuels. Since high density is an important property for these materials to possess, the incorporation of nitro group substituents in compact cage molecules can result in high energy-density materials. Polynitroadamantanes have received little attention. 1,3,5,7-Tetranitroadamantane was synthesized by oxidation of the corresponding amine.[1a] Recently, 2,2-dinitro- and 2,2,6,6-tetranitroadamantane have been synthesized[1j] from the corresponding oximes. Similar attempts aimed at the synthesis of 2,2,4,4-tetranitroadamantane (**1**) failed, apparently due to proximity effects. In order to synthesize higher polynitroadamantanes bearing geminal nitro groups, it is essential to overcome problems associated with steric crowding. A similar difficulty was encountered in the synthesis of 8,8,11,11-tetranitropentacyclo-[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane.[1h] It was shown that treating the carbonyls one at a time provided an easy solution to this problem. We now report a similar strategy that resulted in the synthesis of the title compound.

Scheme I



The starting material, 4,4-(ethylenedioxy)adamantan-2-one (**3**), was prepared from adamantan-2-one by known procedures[2] (Scheme I). Conversion of **3** to the corresponding oxime was achieved by using the conditions developed by Corey et al.[3] Treatment of **4** with 98% nitric acid in refluxing methylene chloride[4] gave 4,4-dinitroadamantan-2-one (**5**) in 35% yield. A transient blue-green color was observed initially, apparently due to formation of the corresponding nitroso compound. This color gradually faded as more nitric acid was added. Compound **5** was converted into the corresponding oxime **6** in 79%

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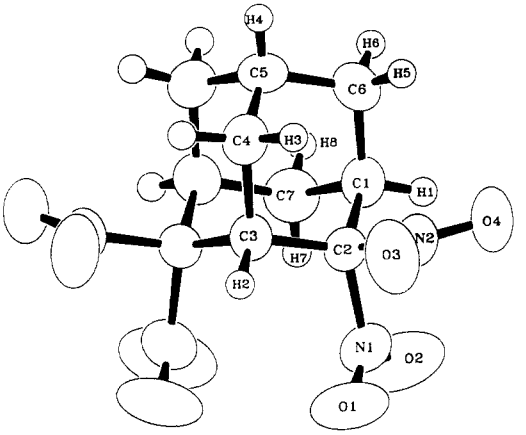

Figure 1. ORTEP drawing for 1. The C, N, and O atoms are shown as 50% ellipses and the H's as B = 1.5 Å spheres. The diagram was prepared by the TEXRAY graphics subroutine as a Hewlett-Packard 7550A pen plotter file; this file was read by the PLOTMD program,[10] which displayed the drawing on a VaxStation monitor, allowed label changes to be made, and prepared an HP laser-jet printer file.

yield. Nitration of **6** with 98% nitric acid afforded 2,2,4,4-tetranitroadamantane (**1**) in 29% yield, along with recovered **5**, which could be recycled.

The structure of **1** was deduced on the basis of its proton and ^{13}C spectra and was confirmed via single-crystal X-ray diffraction techniques.

Thermal stability of **1** was investigated by using differential scanning calorimetry (DSC). When **1** was heated at 10 °C/min, a strong exotherm occurred beginning at 240 °C and reaching a maximum at 255 °C, indicating its high thermal stability.

X-ray Crystallographic Study of 1. The molecule has mirror symmetry with the C3–C4–C5–C7 unit on the mirror plane (Figure 1). The various bond lengths and angles are normal with the exception of the parameters associated with the dinitromethylene moiety. Here the C–N lengths are stretched to 1.547 and 1.551 Å and the N–C–N angle has diminished to 98.2°. Similar values were found in 2,2-dinitroadamantane,[5] with C–N distances of 1.555 and 1.560 Å and a N–C–N angle of 98.7°. The typical $C(sp^3)$–NO_2 distance is in the 1.46–1.50 Å range. These distortions observed in the adamantanes may be attributed to the geminal nitro groups that, because of the strong electron demand placed on C2, require an enrichment of the amount of p character in the exocyclic orbitals at C. The crystal density was found to be 1.65 g/mL.

Experimental Section

Melting points are uncorrected. NMR spectra were run at 300 MHz for ^1H and at 75 MHz for ^{13}C. Chemical shifts are reported in ppm downfield from internal tetramethylsilane. Elemental microanalyses were performed by Galbraith Laboratories, Knoxville, TN.

4,4-(Ethylenedioxy)-2-oximidoadamantane (4). To a suspension of **3** (14.0 g, 67 mmol) in absolute ethanol (300 mL) were added hydroxylamine hydrochloride (9.30 g, 135 mmol) and sodium acetate trihydrate (36.7 g, 270 mmol). The resulting mixture was stirred overnight at room temperature. The reaction mixture then was concentrated in vacuo. The residue was partitioned between water (200 mL) and methylene chloride (200 mL), and the layers were separated. The organic phase was dried ($MgSO_4$) and filtered, and the filtrate was concentrated under reduced pressure. The residue was recrystallized from ethanol to give **4** as a colorless microcrystalline solid (12.6 g, 84%), mp 116–120 °C: IR (KBr) 3200 (br s), 1675 cm^{-1} (m).

4,4-Dinitroadamantan-2-one (5). To a refluxing solution of **4** (5 g, 22.4 mmol) in dry methylene chloride under nitrogen was added dropwise a solution of 98% nitric acid (20 mL), urea (150 mg, 250 mmol), and ammonium nitrate (150 mg, 200 mmol) in methylene chloride (20 mL). (*CAUTION*: 98% nitric acid should be handled carefully. Urea and ammonium nitrate should be carefully added in small portions to the nitric acid/methylene chloride solution since a slight exotherm occurs and nitrogen oxide fumes are evolved.) A dark green color developed initially, which faded as more nitric acid solution was added. After the addition had been completed, the mixture was refluxed for a further 15 min. The reaction mixture then was allowed to cool to room temperature. The mixture was washed with ice–water (2 × 50 mL). The organic layer was dried ($MgSO_4$) and filtered and the filtrate was concentrated in vacuo. The resulting oil was purified by column chromatography on silica gel, eluting with a 2:3 mixture of methylene chloride/hexane. The first fraction thereby eluted gave pure **5** (1.9 g, 35.3%). An analytical sample, mp 246–247 °C, was prepared by recrystallization from methylene chloride–hexane mixed solvent: IR (KBr) 1730 (s), 1580 (s), 1315 cm^{-1} (m); ^1H NMR ($CDCl_3$) δ 1.75–2.35 (m, 9 H), 2.7 (s, 1 H), 3.3 (m, 1 H), 3.82 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 25.57 (d), 30.47 (d), 33.82 (d), 34.20 (t), 37.99 (t), 40.03 (t), 43.82 (d), 51.84 (d), 122.6 (s), 206.66 (s). Anal. Calcd for $C_{10}H_{12}N_2O_5$: C, 50.02; H, 5.03. Found: C, 50.11; H, 5.06.

2,2-Dinitro-4-oximidoadamantane (6). To a suspension of **5** (1.8 g, 7.50 mmol) in absolute ethanol (100 mL) were added hydroxylamine hydrochloride (1 g, 15.0 mmol) and sodium acetate trihydrate (4 g, 30.0 mmol). The resulting mixture was stirred overnight at room temperature. The mixture then was concentrated in vacuo. The residue was partitioned between methylene chloride (100 mL) and water (100 mL), and the layers were separated. The organic phase was dried ($MgSO_4$) and filtered, and the filtrate was concentrated in vacuo to give an oil, which solidified on standing to afford a pale yellow solid. This solid was recrystallized from ethanol, thereby affording **6** as a colorless microcrystalline solid (1.5 g, 79%), mp 182–185 °C: IR (KBr) 3300 (br s), 1680 (w), 1580 (s), 1305 cm^{-1} (m).

2,2,4,4-Tetranitroadamantane (1). To a refluxing solution of **6** (1.4 g, 5.50 mmol) in dry methylene chloride (100 mL) under nitrogen was added dropwise a solution of 98% nitric acid (15 mL), urea (150 mg, 250 mmol), and ammonium nitrate (150 mg, 200 mmol) in methylene chloride (15 mL). A transient green color appeared initially, which faded as the reaction progressed. After all the reactants had been added the mixture was refluxed for an additional 15 min and then allowed to cool to room temperature. The resulting mixture was washed with ice-cold water (2 × 50 mL), dried ($MgSO_4$), and filtered, and the filtrate was concentrated in vacuo. The residue was purified by column chromatography on silica gel, using a methylene chloride/hexane mixed solvent gradient elution scheme (starting with 10% methylene chloride to 50% methylene chloride in hexane). The first fraction afforded **1** (0.50 g, 29%) as a colorless microcrystalline solid. An analytical sample was obtained by recrystallization from ethyl acetate, mp 138–40 °C: IR (KBr) 1580 (s), 1310 cm^{-1} (m); ^1H NMR ($CDCl_3$) δ 1.75–2.10 (m, 5 H), 2.30–2.39 (m, 3 H), 2.75–2.82 (m, 1 H), 3.4 (m, 1 H), 1.7 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 23.27 (d), 30.68 (t), 31.99 (d), 34.42 (t), 34.56 (t), 39.06 (d), 122.2 (s). Anal. Calcd for $C_{10}H_{12}N_4O_8$: C, 37.98; H, 3.82. Found: C, 38.06; H, 3.73. Further elution gave recovered **5** (0.40 g).

X-ray crystallographic analysis of 1: $C_{10}H_{12}N_4O_8$, molecular weight = 316.23, 0.16 × 0.163 × 0.23 mm colorless crystal, Enraf-Nonius CAD4 diffractometer, λ(Cu Kα) = 1.5418 Å (incident beam graphite monochromator), monoclinic space group $P2_1/m$, a = 7.074 (1), b = 13.395 (2), and c = 7.304 (1) Å, β = 112.79 (1)°, V = 638.1 (4) Å^3, Z = 2, ρ = 1.65 g cm^{-3}, μ = 12.8 cm^{-1}, $F(000)$ = 328, T = 293 K. Lattice parameters from 25 reflection in the range of $9.9 \le \theta \le 23.8°$, data collection range of $-7 \le h \le k \le 15$, $0 \le l \le 8$, $2 \le \theta \le 60°$, 2θ–θ scan with θ of 8.14° min^{-1}, θ scan range = 1.5 (0.5 + 0.14 tan θ)°, intensity profile sampled at ca. 0.01° intervals and subjected to on-line profile analysis,[6] five standard intensities monitored every one hour of crystal X-ray exposure, maximum and average change in standard intensities

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of 4.7 and 2.6%, 1096 total reflections, 1072 without standards, 888 with $I_o \geq 3\sigma(I_o)$, R_{sym} for equivalent reflections = 0.019. The diffractometer was controlled by a MicroVax II computer with the NRCCAD system of programs.[7] The crystallographic calculations done with the TEXRAY[8] package on MicroVax II and VaxStation II computers, structure solved with the direct methods link MITHRIL,[9] refinement by full matrix least squares, $\sum_w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0.0025F_o^2]$, carbon, nitrogen, and oxygen parameters refined with anisotropic parameters, hydrogen atoms with isotropic terms, secondary isotropic extinction parameter = 0.00002 (4), final R, R_w and error-of-fit values of 0.047, 0.069, 1.96, minimum and maximum in final difference map of −0.13 and 0.21 e Å^{-3}.

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Supplementary Material Available: Fractional coordinates, U values, bond lengths, and bond angles for 1 (4 pages); tables of observed and calculated structure factors for 1 (6 pages). Ordering information is given on any current masthead page.

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Preparation of New Acetal Type Cleavable Surfactants from Epichlorohydrin

Daisuke Ono, Araki Masuyama, and Mitsuo Okahara*

Department of Applied Chemistry, Faculty of Engineering, Osaka University, Yamadaoka, Suita, Osaka 565, Japan

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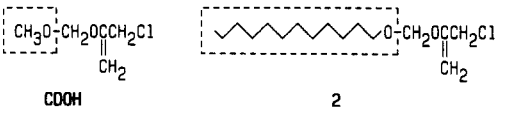
Recently, cleavable surfactants have become a focus of great interest in the field of surfactant chemistry.[1] Such compounds are designed so as to decompose into non surface active species on exposure to acid, alkali, light, or heat after fulfilling their original functions, which might include emulsification, solubilization, micellar catalytic activity, and so on. Among the various types of known cleavable surfactants, compounds in which the decomposition property can be controlled through adjustment of the solution pH seem to be the most common. In particular, there are many reports concerning acetal[2] and 1,3-dioxolane[3] types of amphiphilic compounds.

We previously found that a series of 2-substituted 1-(chloromethyl)ethyl ethers can be synthesized regioselectively in high yields through the reaction of epoxides with organic chlorides in the presence of dodecyltrimethylammonium chloride.[4] We have also reported 2-(chloromethyl)-3,5-dioxahex-1-ene (CDOH; Scheme I) which is prepared from epichlorohydrin according to this method. This compound is stable under ambient conditions and can be applied as an effective acetonylating reagent to active proton-containing compounds under the appropriate conditions.[5] We now report that we have synthesized the allyl chloride derivative **2** (Scheme I) with a long-chain alkyloxy group in place of the methoxy group in CDOH from epichlorohydrin. We have also easily obtained the new acetal type cleavable surfactants **3**, **4**, and **5** with any one of the desired hydrophilic groups (anionic, cationic, and nonionic) through substitution reactions with **2**. In this paper, we present the preparation methods for a series of surfactants, their basic surface active properties, and their decomposition profiles in an aqueous medium through addition of acid, as determined by ^{1}H NMR measurements.

Scheme I



Results and Discussion

The synthetic route to acetal type surfactants **3**, **4**, and **5** is shown in Scheme II. First, chloromethylation of dodecanol was carried out in methylene chloride according to the usual procedure.[6] A solution of crude chloromethyl dodecyl ether in methylene chloride was added dropwise into a mixture of epichlorohydrin and dodecyltrimethylammonium chloride at 0 °C. The reaction mixture was stirred at 30 °C. Dichloride (**1**) was isolated by Kugelrohr distillation. The key intermediate, 2-(chloromethyl)-3,5-dioxaheptadec-1-ene (**2**), was prepared by the dehydrochlorination of **1** under phase-transfer (PT) catalytic conditions, and **2** was also isolated by Kugelrohr distillation.

The sulfonate salt type anionic surfactant **3** was prepared through a modification of the Strecker reaction.[7] In this case, both a stoichiometric amount of sodium iodide and a catalytic amount of tetrabutylammonium bisulfate

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