2 H), 2.05 (s, 3 H), 1.50–2.35 (m, 2 H), 1.10 (t, J = 7 Hz, 3 H), 0.70–1.50 (18 H).

Bicyclo[10.3.0]-1(15)-pentadecen-14-one (4). In a 300-mL three-necked flask, equipped with a reflux condenser and a magnetic stirring bar, was placed a mixture of the diketone **7** (10.0 g, 31.5 mmol), KOH (17.6 g, 315 mmol), and EtOH (150 mL). The mixture was refluxed for 1 h and then poured into ice-cold 3 N HCl and extracted with CH_2Cl_2. The extract was washed with $NaHCO_3$ solution and brine and dried over $MgSO_4$. After removal of the solvent, the residual solid was recrystallized from EtOH to give bicyclo[10.3.0]-1(15)-pentadecen-14-one (**4**, 5.16 g, 74%): mp 95–97 °C (lit.[6] mp 95–96 °C); IR (KBr) 1720, 1610 cm^{-1}; NMR (CCl_4) δ 5.90 (s, 1 H), 2.00–3.20 (m, 5 H), 0.7–2.00 (18 H).

14,14-(Ethylenedioxy)bicyclo[10.3.0]-1(12)-pentadecene (8). A benzene solution of the enone **4** (1.00 g, 4.55 mmol), ethylene glycol, and a catalytic amount of p-TsOH was refluxed with continuous removal of water by azeotropic distillation overnight. After the mixture was cooled to room temperature, aqueous $NaHCO_3$ solution was added. The organic layer was separated, and the aqueous layer was extracted with benzene. The combined benzene solution was washed with brine and dried over $MgSO_4$. After evaporation of the solvent, the residue was purified by column chromatography on SiO_2 with ether–n-hexane (1:5) to give 14,14-(ethylenedioxy)bicyclo[10.3.0]-1(12)-pentadecene (**8**, 1.18 g, 98%): IR (neat) 2940 cm^{-1}; NMR (CCl_4) δ 3.80 (s, 4 H), 2.40 (s, 4 H), 1.95–2.30 (m, 4 H), 0.80–1.80 (16 H).

3,14-Dihydroxycyclopentadecanone (9). A solution of the dioxolane **8** (1.18 g, 4.47 mmol) in CH_2Cl_2 (60 mL), placed in a 100-mL three-necked flask, was treated with ozone at −60 °C. After nitrogen purge, a suspension of $LiAlH_4$ (306 mg, 8.1 mmol) in dry THF (60 mL) was added to the ozonide carefully at −20 to ~−40 °C, and the resulting mixture was allowed to warm to 0 °C with stirring. To the mixture were added EtOAc and water carefully at 0 °C with stirring, and the resulting mixture was filtered through Celite. The filtrate was poured into $NaHCO_3$ solution and extracted with CH_2Cl_2. The extract was washed with brine, dried over $MgSO_4$, and concentrated to give crude 1,1-(ethylenedioxy)-3,14-dihydroxycyclopentadecane (1.36 g) as a solid which was used in the next step without purification.

A solution of this crude material (1.36 g) and a catalytic amount of p-TsOH in aqueous THF (30 mL of THF and 30 mL of water) was refluxed for 3 h and then cooled to room temperature. After most of the THF was evaporated in vacuo, the mixture was extracted with CH_2Cl_2. The extract was washed with $NaHCO_3$ solution and brine and dried over $MgSO_4$. After evaporation of the solvent, recrystallization from ether–n-hexane (1:4) gave 3,14-dihydroxycyclopentadecanone (**9**, 983 mg, 86% from **8**) as white needles:[7] mp 103–104 °C; IR (KBr) 3300, 2920, 1700 cm^{-1}; NMR (CCl_4) δ 3.75–4.25 (2 H), 2.60–3.10 (2 H), 2.55–2.80 (4 H), 1.10–1.80 (20 H).

Muscone (1). In a 300-mL three-necked flask, equipped with a reflux condenser and a stirring bar, was placed a solution of the keto diol **9** (983 mg, 3.84 mmol) and a catalytic amount of p-TsOH in dry toluene (150 mL). The mixture was refluxed for 10 min and cooled to 0 °C. A saturated $NaHCO_3$ solution was added, and the resulting mixture was extracted with ether. The extract was washed with brine and dried over $MgSO_4$. After removal of the solvent, the residue was purified by column chromatography on SiO_2 with ether–n-hexane (1:5) to give an isomeric mixture of the α,β:α',β'-dienone **10a** and the α,β:β',γ'-dienone **10b** (717 mg, 85%). The mixture of **10a** and **10b** was used in the next step without separation. Pure samples were obtained from another experiment by column chromatography on SiO_2 with ether–n-hexane (1:20). (*E,E*)-2,14-Cyclopentadecadien-1-one (**10a**): TLC R_f 0.615 (Merck Kieselgel 60F_{254}, EtOAc–n-hexane, 1:3); IR (neat) 2930, 1660, 1620 cm^{-1}; NMR (CCl_4) δ 6.60 (dt, J = 7, 7 Hz, 2 H), 6.10 (d, J = 17 Hz, 2 H), 2.00–2.50 (m, 4 H), 1.00–1.80 (16 H). (*E,E*)-2,13-Cyclopentadecadien-1-one (**10b**): TLC R_f 0.692 (Merck Kieselgel 60F_{254}, EtOAc–n-hexane, 1:3); IR (neat) 2930, 1695, 1625 cm^{-1}; NMR (CCl_4) δ 6.70 (dt, J = 15, 7 Hz, 1 H), 6.05 (d, J = 15 Hz, 1 H), 5.30–5.60 (m, 2 H), 2.90–3.10 (m, 2 H), 1.80–2.45 (4 H), 0.85–1.75 (14 H).

In a 50-mL two-necked flask, equipped with a stirring bar and rubber septum, was placed completely dried CuI (2.04 g, 10.8 mmol). Dry ether (15 mL) was added to the flask, and the mixture was cooled to −25 to ~−30 °C. Then to the suspension was added MeLi (16.6 mL of a 1.18 M ethereal solution, 19.6 mmol) with stirring. The yellow solution became colorless. After the mixture was stirred for 20 min, a solution of the dienones **10** (717 mg, prepared above) in ether (10 mL) and THF (3 mL) was added over a period of 20 min to give an orange solution. The mixture was stirred for 1 h at −25 °C, and then water and 3 N HCl were added. The resulting mixture was extracted with ether, and the extract was washed with $NaHCO_3$ solution, $Na_2S_2O_3$ solution, and brine and dried over $MgSO_4$. Removal of the solvent gave a crude material which was purified by column chromatography on SiO_2 with ether–n-hexane (1:10) to afford (*E*)-3-methyl-14-cyclopentadecanone (**11a**) and (*E*)-3-methyl-13-cyclopentadecanone (**11b**) (737 mg, 96%). The IR and NMR data of **11a** and **11b** were obtained from another experiment. **11a**: IR (neat) 2930, 1690, 1620 cm^{-1}; NMR (CCl_4) δ 6.70 (dt, J = 15, 7 Hz, 1 H), 6.05 (d, J = 15 Hz, 1 H), 1.75–2.60 (m, 4 H), 0.80–1.75 (19 H), 1.00 (t, J = 6 Hz, 3 H). **11b**: IR (neat) 2930, 1710 cm^{-1}; NMR (CCl_4) δ 5.35–5.60 (m, 2 H), 2.80–3.05 (m, 2 H), 1.75–2.60 (6 H), 0.75–1.70 (17 H), 0.90 (t, J = 7 Hz, 3 H).

The mixture of **11** (737 mg) in AcOH (7 mL) was stirred in the presence of a catalytic amount of 5% Pd on C under atmospheric pressure of H_2 at room temperature. After absorption of H_2 ceased, water was added to the mixture, and the resulting mixture was extracted with CH_2Cl_2. The extract was washed with $NaHCO_3$ solution and brine and dried over $MgSO_4$. After evaporation of the solvent, the residue was chromatographed on SiO_2 with ether–n-hexane (1:10) to give muscone (**1**, 708 mg, 77% from **9**): semicarbazone, mp 129.5–132.0 °C (lit.[8] mp 131–132 °C); IR (neat) 2920, 1710 cm^{-1}; NMR (CCl_4) δ 1.85–2.45 (4 H), 1.10–1.80 (23 H), 0.90 (d, J = 6 Hz, 3 H).

Cyclopentadecanone (Exaltone, 2). The cyclopentadecadienones **10** were prepared and hydrogenated by a method similar to that in the case of muscone to give cyclopentadecanone (**2**) in a quantitative yield: semicarbazone, mp 185.5–187.5 °C (lit.[9] mp 187–188 °C); IR (KBr) 2940, 1717 cm^{-1}; NMR (CCl_4) δ 2.42 (t, J = 7 Hz, 4 H), 1.45–1.90 (4 H), 1.10–1.45 (20 H).

Acknowledgment. This work was supported financially by the Ministry of Education of the Japanese Government and The Asahi Glass Foundation.

Registry No. **1**, 956-82-1; **1** semicarbazone, 898-26-0; **2**, 502-72-7; **2** semicarbazone, 13756-56-4; **4**, 56975-50-9; **5**, 75232-70-1; **6**, 75232-71-2; **7**, 75232-72-3; **8**, 75232-73-4; **9**, 75232-74-5; **10a**, 73125-66-3; **10b**, 73125-53-8; **11a**, 75232-75-6; **11b**, 75232-76-7; diethyl carbonate, 105-58-8; cyclododecanone, 830-13-7; allyl bromide, 106-95-6; ethylene glycol, 107-21-1; 1,1-(ethylenedioxy)-3,14-dihydroxycyclopentadecane, 75232-77-8.

(6) B. A. McAndrew and S. W. Russell, *J. Chem. Soc., Perkin Trans. 1*, 1172 (1975).
(7) The stereochemistry of the diol **9** was not confirmed.
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Temperature Effects on the Bromination of 2-Bromobicyclo[2.2.1]hept-2-ene. Synthesis of 2,3-Dibromobicyclo[2.2.1]hept-2-ene

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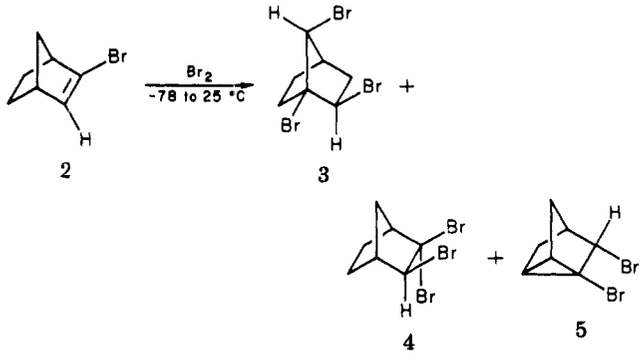
As part of our studies on the generation and reactions of bicyclo[2.2.1]hept-2-yne (norbornyne),[2] we had need of

2,3-dibromobicyclo[2.2.1]hept-2-ene (1). An attractive starting material for the synthesis of 1 was 2-bromobicyclo[2.2.1]hept-2-ene (2) which was readily prepared from bicyclo[2.2.1]hept-2-ene via free-radical bromination with dibromotetrachloroethane[3] followed by dehydrobromination with potassium *tert*-butoxide.[4] The bromination of 2 with known free-radical transfer agents, such as dibromotetrachloroethane[5a] and *N*-bromosuccinimide[5b] gave only low yields (10–20%) of a mixture of brominated products (mixed with 2) after prolonged reaction times.

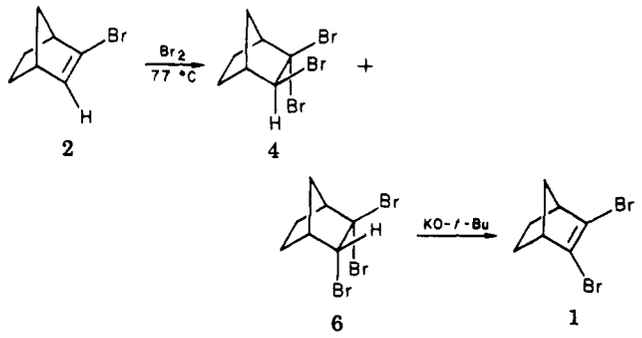
Direct bromination of 2 with bromine in diethyl ether over a temperature range of −78 to 25 °C gave a 58% yield of a mixture of 3, 4, and 5 in the ratio of 13:4:1, respectively. Similar results were obtained in carbon tetrachloride at 0 °C. Separation of this mixture by a combination of column chromatography and distillation afforded pure samples of each of the three components. Structural assignments were made on the basis of NMR spectral data and through spectral comparison with the analogous chlorinated compounds.



The unfavorable yield of 4 from the direct bromination of 2 prompted us to seek an alternate method. It had been previously noted in the literature[6] that the bromination of cyclohexene at high temperature and at high dilution resulted in allylic bromination rather than bromine addition to the double bond. Clearly, these conditions promoted free-radical bromination over ionic bromination. Since allylic bromination should be unfavorable for 2, bromine free radicals would be expected to add to the double bond.

Slow addition of gaseous bromine in a stream of nitrogen into a refluxing solution of 2 in carbon tetrachloride gave a 92% yield of a 2.5:1 mixture of 4 and 6.[7] The ratio of the isomers was determined by NMR spectroscopy since the epimers were not conveniently separated. Treatment of the crude mixture of 4 and 6 with 1.2 equiv of potassium *tert*-butoxide in *tert*-butyl alcohol gave a 51% yield of 1, bp 58–60 °C (1.5 mm). the structure of 1 was clearly established by its ^{13}C NMR spectrum which showed only four peaks; ^{13}C NMR ($CDCl_3$) δ 125.37 (s), 51.42 (d), 46.80 (t), 25.53 (t).

In summary, the change from room temperature to 77 °C resulted in a dramatic change in the mechanism of bromination of 2.[7]



Experimental Section

Bromination of 2-Bromobicyclo[2.2.1]hept-2-ene (2) below 25 °C. A solution of 60.4 g of 2 in 400 mL of anhydrous ether was cooled to −78 °C and treated with 18 mL (1 equiv) of bromine over a 2-h period. The reaction mixture was allowed to warm to 25 °C and washed with saturated sodium thiosulfate solution. The organic phase was separated, dried over anhydrous magnesium sulfate, and filtered, and the filtrate was concentrated in vacuo to yield 77.5 g (81%) of a pale yellow oil. Chromatography on neutral alumina using pentane as eluant gave 18.5 g of a 4:1 mixture of 4 and 5 (NMR analysis) as an early fraction. Elution of the column with ether afforded 48.6 g (42%) of 3 as a white solid, mp 75–80 °C.

The fraction containing the mixture of 4 and 5 was fractionally distilled to yield 2.9 g (3%) of 2,3-dibromotricyclo[2.2.1.$0^{2,6}$]-heptane (5): bp 46–50 °C (0.3 mm); ^1H NMR ($CDCl_3$) δ 4.0 (1 H, s), 2.4–1.2 (7 H, m); ^{13}C NMR ($CDCl_3$) 61.51 (d), 39.09 (d), 35.13 (s), 31.81 (t), 31.57 (t), 23.39 (d), 22.01 (d) ppm; exact mass calcd for $C_7H_8{}^{79}Br_2$ 249.8983, found 249.8987.[8]

The pot residue from the distillation crystallized on standing to give 15.0 g (13%) of *exo*-2,2,3-tribromobicyclo[2.2.1]heptane (4):[9] mp 46–50 °C; ^1H NMR ($CDCl_3$) δ 4.48 (1 H, d, J = 3 Hz), 3.12 (1 H, br s), 2.50 (1 H, br s), 2.4–1.2 (6 H, m); ^{13}C NMR ($CDCl_3$) 75.21 (s), 68.13 (d), 58.76 (d), 49.63 (d), 34.50 (t), 28.11 (t), 27.12 (t) ppm. An analytical sample was obtained by recrystallization from methanol, mp 53–55 °C.

Anal. Calcd for $C_7H_9Br_3$: C, 25.27; H, 2.72. Found: C, 25.43; H, 2.84.

The fraction eluted from the column with ether was identified as 1,2,7-tribromobicyclo[2.2.1]heptane (3): ^1H NMR ($CDCl_3$) δ 4.18 (2 H, m), 3.1–1.3 (7 H, m); ^{13}C NMR ($CDCl_3$) 65.50 (s), 60.24 (d), 53.17 (d), 42.60 (t), 42.21 (d), 38.63 (t), 27.20 (t) ppm. An analytical sample was obtained by recrystallization from 95% ethanol, mp 92–94 °C.

Anal. Calcd for $C_7H_9Br_3$: C, 25.27; H, 2.72. Found: C, 25.24; H, 2.74. A similar mixture of products was obtained when the bromination was carried out at 25 °C.

2,3-Dibromobicyclo[2.2.1]hept-2-ene (1). To a refluxing solution of 16.3 g of 2 in 100 mL of carbon tetrachloride was added slowly 15 g (1 equiv) of gaseous bromine in a stream of nitrogen over a 7-h period. After the addition was complete, the reaction mixture was cooled to 25 °C and washed with two 100-mL portions of saturated thiosulfate solution. These washes were back extracted with methylene chloride and the organic phases were combined, washed with brine, dried over anhydrous magnesium sulfate, and filtered, and the filtrate was concentrated to give 28.8 g (92%) of a yellow liquid which became semisolid on standing. NMR analysis of this material showed it to be a 2.5:1 mixture

(1) National Science Foundation Predoctoral Fellow, 1975–1978; Louise T. Dosdall Graduate Fellow, 1978–1979; Lubrizol Foundation Fellow, Summer 1979.

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(9) The structure of 4 was assigned on the basis of the close similarity of its ^1H NMR spectrum to that of *exo*-2,2,3-trichlorobicyclo[2.2.1]heptane; δ 4.11 (1 H, d, J = 3 Hz), 2.88 (1 H, m), 2.42 (1 H, m), 2.3–1.3 (6 H, m). T. J. Atkins, Ph.D. Thesis, The Ohio State University, 1972.

of 4 and 6; ^{1}H NMR ($CDCl_3$) δ 5.16 (0.29 H, d, J = 4 Hz), 4.48 (0.71 H, d, J = 3 Hz), 3.04 (1 H, br s), 2.5–1.2 (7 H, m). Since these isomers could not be readily separated, the mixture was used in the dehydrobromination step.

A solution of 52.9 g of the mixture of 4 and 6 in 100 mL of *tert*-butyl alcohol was added dropwise over a 4-h period to a solution of 22 g (1.2 equiv) of potassium *tert*-butoxide in 150 mL of *tert*-butyl alcohol. The reaction mixture was refluxed for 8 h, cooled to 25 °C, and poured into 300 mL of water. The aqueous solution was extracted with three 100-mL portions of ether. The combined extracts were washed with brine, dried over anhydrous magnesium sulfate, and filtered, and the filtrate was concentrated. Fractional distillation gave 20.4 g (51%) of 2,3-dibromobicyclo-[2.2.1]hept-2-ene (1): bp 58–60 °C (1.5 mm); ^{1}H NMR ($CDCl_3$) δ 2.92 (2 H, t, J = 2 Hz), 1.8–1.0 (6 H, m); ^{13}C NMR ($CDCl_3$) 125.37 (s), 51.42 (d), 46.80 (t), 25.53 (t) ppm; exact mass calcd for $C_7H_8{}^{79}Br_2$ 249.8993, found 249.9004. An analytical sample was prepared by preparative VPC on 10 ft × 0.25 in. 10% SE-30 on 60/80 Chromosorb W at 170 °C.

Anal. Calcd for $C_7H_8Br_2$: C, 33.37; H, 3.20; Br, 63.43. Found: C, 33.46; H, 3.24; Br, 63.53.

Acknowledgment. We are indebted to the National Science Foundation for Grant CHE78-10231 which supported this investigation.

Registry No. 1, 75267-72-0; 2, 694-90-6; 3, 75267-73-1; 4, 75267-74-2; 5, 75267-75-3; 6, 75267-76-4.

A Convenient and Unambiguous Synthesis of 1-Bromoindene

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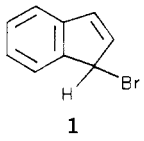
The literature on 1-bromoindene (1) is sparse and inconclusive. It was first reported in 1944[1] as the product (24% yield) of the reaction of indene and *N*-bromosuccinimide. Later, Crofts and Williamson found this method gave them only low and variable yields.[2] However, they did describe a new route to 1 involving dehydrobromination of *trans*-1,2-dibromoindane (3). This reaction



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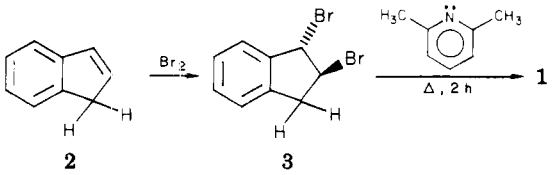
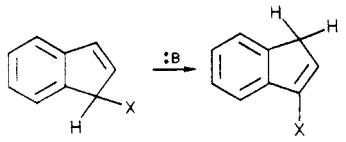


2 3

gave a single product (24% yield) that had a boiling point and infrared spectrum similar to that reported earlier.[1] It was further argued[2] that 1 should be the exclusive product because the reaction conditions favored the E2 mechanism, i.e., strong base, high temperature, and nonpolar solvent.

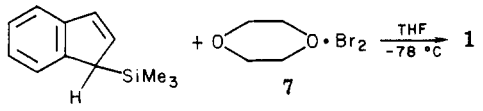
In light of a recent study by Freidrich and Taggart,[3] however, in which it was shown that 1-substituted indenes (4) isomerize rapidly and quantitatively to derivatives of structure 5 in the presence of even weakly basic amines,



4 5

the earlier assignments became questionable. Since other work in our laboratories required ready access to 1-bromoindene and the published methods[1,2] gave us low yields of complex mixtures, we looked for a simple and unambiguous synthetic procedure.

The successful cleavage of silicon–carbon bonds by electrophilic reagents[4] led us to consider that approach as a route to 1. We found that 1-(trimethylsilyl)indene (6), when treated with a 10% excess of dioxane dibromide (7) in tetrahydrofuran at −78 °C, gave 1 in 66% yield. All



6 7

of silane 6 was consumed and 1-bromoindene (1) was detected as the sole product. The yield corresponded to pure product after column chromatography. The ^{1}H NMR spectrum permitted an unambiguous structural assignment, eliminating the possibility that the isomer 5, X = Br, was the product. The complexation of bromine by dioxane is essential to the success of this preparation. When bromine was substituted for 7, 1 could be detected by ^{1}H NMR but not isolated.

Experimental Section

1-(Trimethylsilyl)indene (6) was prepared by the method of Rakita and Davison.[5] Dioxane dibromide (7) was prepared by the method of Billimoria and Maclagen.[6] Tetrahydrofuran was purified by distillation from sodium benzophenone ketyl immediately before use. Pentane was stirred over H_2SO_4 and distilled. NMR spectra were recorded on a Varian EM-390 spectrometer, using tetramethylsilane as the internal standard.

1-Bromoindene (1). A 100-mL three-neck flask fitted with an addition funnel and a nitrogen inlet was dried and purged with nitrogen. The entire apparatus was covered with aluminum foil to exclude light. The flask was charged with (trimethylsilyl)indene (2.00 g, 10.7 mmol) and THF (25 mL). Dioxane dibromide (2.92 g, 11.8 mmol) was placed in the addition funnel to which 10 mL of THF was added to form a homogeneous solution. The flask was cooled in a bath of acetone–dry ice and the contents of the addition funnel were slowly added. Upon completion of this addition, the flask was allowed to warm to room temperature and the solvent was removed by rotary evaporation. The remaining oil was placed on top of 14 × 3.5 cm column of Florisil, 60–100 mesh, and eluted with 200 mL of pentane. Removal of solvent left 1.38 g (7.1 mmol, 66%) of pure 1 as a pale yellow liquid: NMR ($CDCl_3$) δ 5.48 (br s, 1 H, CHBr), 6.6 (d of d, J = 2, 6 Hz, 1 H, C=CHCHBr), 6.82 (d, J = 6 Hz, 1 H, HC=CHCHBr), 7.67–7.13 (m, 4 H, aromatic).

Anal. Calcd for C_9H_7Br: C, 55.38; H, 3.59. Found: C, 55.16; H, 3.74.

Acknowledgment. This work was supported in part with funds from the U.S. Department of the Interior, Office of Water Research and Technology, and the Research Corporation.

Registry No. 1, 61083-09-8; 6, 18053-75-3; 7, 15481-39-7.

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