3,5-Me_2, bulb-to-bulb distillation;[37] 3-OH, 190–191; 3-MeO, 100–102;[25] 3-NHAc, 209–214;[8a,25,30,38] 3-NH_2, 120–121.[38,39]

The ^{15}N INEPT measurements[40,41] were made on a Bruker AM360 NMR spectrometer at 36.50 MHz at ambient temperature with a 10 mm i.d. tube for solutions of ca. 10–20 w/v % in a mixture of DMSO (2.5 mL) and DMSO-d_6 (0.5 mL for internal deuterium lock). ^{15}N SCS values were calculated from absolute frequencies. Typical experimental conditions were 31-μs pulse width (90° flip angle), acquisition time of 0.56 s, spectral width of 14.7 kHz, 16K data points, and $\Delta_t = 0.25/(J_{NH}) = 0.050$ s. Digital resolution was 0.05 ppm (1.8 Hz), and reproducibility was ±0.1 ppm.

Acknowledgment. We are grateful to Hitoshi Yamada, Fusako Fukuda, and Takanori Tanaka for the measurements of mass and IR spectra and elemental microanalyses in Material Analysis Center, ISIR, Osaka University.

Registry No. ^{15}N, 14390-96-6; ^{17}O, 13968-48-4; 4-NO_2-PYNO, 1124-33-0; 4-CN-PYNO, 14906-59-3; 4-CO_2Me-PYNO, 3783-38-8; 4-Ac-PYNO, 2402-96-2; 4-PHCO-PYNO, 14178-29-1; 4-Cl-PYNO, 1121-76-2; 4-Ph-PYNO, 1131-61-9; 4-Me-PYNO, 1003-67-4; 4-OMe-PYNO, 1122-96-9; 4-AcNH-PYNO, 14906-56-0; 4-NH_2-PYNO, 3535-75-9; PYNO, 694-59-7; 3-CN-PYNO, 14906-64-0; 3-CO_2Me-PYNO, 15905-18-7; 3-Ac-PYNO, 14188-94-4; 3-F-PYNO, 695-37-4; 3-Cl-PYNO, 1851-22-5; 3,5-Cl_2-PYNO, 15177-57-8; 3-Br-PYNO, 2402-97-3; 3-Ph-PYNO, 1131-48-2; 3-Me-PYNO, 1003-73-2; 3,5-Me_2-PYNO, 3718-65-8; 3-OH-PYNO, 6602-28-4; 3-OMe-PYNO, 14906-61-7; 3-AcNH-PYNO, 15010-23-8; 3-NH_2-PYNO, 1657-32-5.

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Ylide Rearrangement of Benzyltrialkylammonium Salts: The Improved Sommelet–Hauser Rearrangement. 2

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When a tetraalkylammonium salt is treated with a base, a hydrogen atom α to the nitrogen atom is removed to give an ammonium ylide intermediate.[1] Since an alkylbenzyldimethylammonium salt contains different kinds of α-hydrogen atoms, different ylides may be formed simultaneously, and they are isomerized to the tertiary amines via the Sommelet–Hauser rearrangement and/or the Stevens rearrangement, respectively. If the salt contains a β-hydrogen to the nitrogen, elimination of the alkyl group is usually important (Hofmann elimination). Thus the base-promoted reaction of alkylbenzyldimethylammonium salts leads to usually complex mixtures of tertiary amines.[2]

Table I. Benzyldialkyl[(trimethylsilyl)methyl]ammonium Halides 3

entry		R^1	R^2	X^-	condition temp[c]/time, h	% yield from 1
1	**a**	Me	Et	I	reflux/18	94
2	**b**	*i*-Pr	Me	I	rt/20	62
3	**c**	*t*-Bu	Me	I	rt/90[a]	78
4	**d**	c-Hx	Me	I	rt/20	74
5	**e**	Et	Et	I	reflux/44	62
6	**f**	$(CH_2)_4$		Br	reflux/2	87[b]
7	**g**	$(CH_2)_5$		Br	reflux/2	95[b]

[a] *N,N*-Dimethylformamide was used as the solvent. [b] Yield from **2**. [c] rt = room temperature.

Table II. Reaction of Benzyldialkyl[(trimethylsilyl)methyl]ammonium Halides 3 with Cesium Fluoride

				products 5 and 6		
entry		reactn time, h	bp,[a] °C (mmHg)	yield,[b] %	ratio[c] 5/6	ref of 5
1	**a**	23	155 (160)	86	97:3	2, 8
2	**b**	27	165 (110)	86	93:7	2, 8
3	**c**	24	140 (120)	83	*d*	2
4	**d**	27	190 (40)	88	89:11	2
5	**e**	24	100 (140)	82	91:9	9
6	**f**	43	165 (65)	69	98:2	10
7	**g**	43	180 (90)	74	97:3	11

[a] Oven temperature of a Buchi Kugelrohr distillation apparatus. [b] Total yield of **5** and **6**. [c] The ratio was determined by GLC analysis (10% PEG 20M). [d] **5c**/**7**/**8** = 65:33:2.

Table III. *N*-Alkyl-*N*-[(trimethylsilyl)methyl]benzylamine 1[a]

		^1H NMR ($CDCl_3$), δ		
	bp °C (mmHg)	$SiCH_3$	$SiCH_2N$	NCH_2Ph
1a	111–114 (16)[b]			
1b	140 (20)[c]	0.00	1.86	3.44
1c	140 (25)[c]	−0.11	2.08	3.64
1d	180–195 (20)[c]	0.00	1.97	3.44
1e	79–83 (1.5)	0.00	1.89	3.44

[a] Satisfactory analytical data or high-resolution mass spectra were submitted for reviews. [b] Reference 5; bp 132–134 °C (14 mmHg). [c] Oven temperature of a Buchi Kugelrohr distillation apparatus.

Previously we reported that fluoride anion induced desilylation of substituted benzyldimethyl[(trimethylsilyl)methyl]ammonium halides gives the methyl ylide intermediates exclusively, and leads to high yields of the Sommelet–Hauser rearrangement products, *N,N*-dimethyl-2-methylbenzylamines.[3] This paper describes the desilylation reaction of benzyldialkyl[(trimethylsilyl)methyl]ammonium salts having hydrogens β to the nitrogen.

Seven benzyldialkyl[(trimethylsilyl)methyl]ammonium halides (**3**) were synthesized by quaternization of *N*-alkyl-*N*-[(trimethylsilyl)methyl]benzylamines (**1**) or *N,N*-dialkyl[(trimethylsilyl)methyl]amines (**2**) with alkyl halides (Tables I and III).

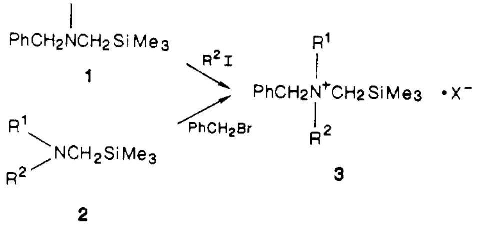

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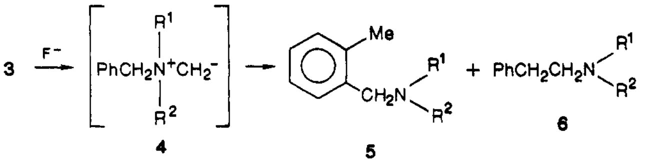
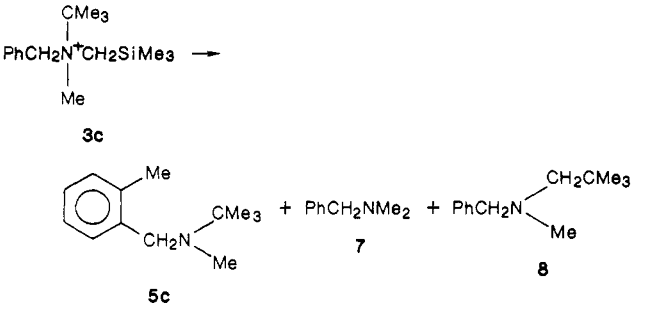
Table IV. Benzyldialkyl[(trimethylsilyl)methyl]ammonium Halides 3[a]

		^{1}H NMR ($CDCl_3$), δ				
	mp, °C	$SiCH_3$	NCH_3	$SiCH_2N$	NCH_2Ph	others
3a	162–163	0.35	3.20	3.23, 3.41 (AB q, *J* = 14 Hz)	4.92 (s)	1.55 (t, *J* = 7 Hz, 3 H), 3.60 (m, 2 H)
3b	169.5–170	0.33	3.07	2.91, 3.39 (AB q, *J* = 15 Hz)	4.49, 4.75 (AB q, *J* = 13 Hz)	1.58, 1.69 (d × 2, *J* = 7 Hz, 3 H × 2), 2.75 (m, 1 H)
3c	164–165	−0.07	3.17	3.17 (s)	3.52, 3.62 (AB q, *J* = 12 Hz)	1.72 (s, 9 H)
3d	169–169.5	0.32	3.10	2.90, 3.43 (AB q, *J* = 15 Hz)	4.51, 4.77 (AB q, *J* = 13 Hz)	1.00–2.75 (m, 10 H), 3.28 (m, 1 H)
3e	133–133.5	0.38		3.23 (s)	4.75 (s)	1.52 (t, *J* = 7 Hz, 6 H), 3.47 (q, *J* = 7 Hz, 4 H)
3f	174–176	0.39		3.34 (s)	4.89 (s)	2.10–2.35 (m, 4 H), 3.50–4.10 (m, 4 H)
3g	169–171	0.40		3.39 (s)	5.00 (s)	1.50–2.20 (m, 6 H), 3.45–3.70 (m, 4 H)

[a] Satisfactory analytical data (±0.4% for C, H, and N) were submitted for review.

Salts **3** were treated with cesium fluoride in hexamethylphosphoramide (HMPA) at room temperature. High yields of the Sommelet–Hauser rearrangement products *N*,*N*-dialkyl-2-methylbenzylamines **5** were obtained, accompanied by small amounts of the Stevens rearrangement products *N*,*N*-dialkyl-2-phenylethylamines **6**, except for benzyl-*tert*-butyl[(trimethylsilyl)methyl]ammonium iodide (**3c**) (Table II).





The formation of ylide carbanion from benzyltrialkylammonium halides by strong bases occurs mainly on the benzyl carbon. A β-proton of the *N*-alkyl substituent is easily transferred to the carbanion followed by elimination of an alkene.[2] The desilylation of **3** gives the methyl ylide intermediates **4** regioselectively, **4** is isomerized selectively to form the Sommelet–Hauser products **5**, and the elimination of the *N*-alkyl group does not occur.

In the case of **3c**, however, formation of an elimination product, *N*,*N*-dimethylbenzylamine (**7**) was formed in competition with the Sommelet–Hauser rearrangement by loss of isobutylene. A small amount of *N*-(2,2-dimethylpropyl)-*N*-methylbenzylamine (**8**), a new type of Stevens rearrangement product, was also formed. That is, transfer of a β-proton of the *tert*-butyl group to the methyl ylide anion resulting in **7** and the migration of the *tert*-butyl group to give **8** compete with the Sommelet–Hauser rearrangement, presumably because the bulky *tert*-butyl group blocks nucleophilic attack of the methyl ylide anion at the ortho position of the phenyl group. The ratio of products was **5c**/**7**/**8** = 65:33:2, and (phenylethyl)amine **6c** was not detected in the product mixture.

Experimental Section

Hexamethylphosphoramide (HMPA) was dried by distillation under reduced pressure from sodium prior to use.[4] Cesium fluoride was dried over P_2O_5 at 180 °C under reduced pressure. NMR spectra were recorded on a JEOL JNM-MH-100 or FX-100 spectrometer using Me_4Si as internal standard. Mass spectra were measured on a JEOL JMS-DX 300 GC–MS system. GLC analyses were carried out on a Hitachi Model 263-30 gas chromatograph with FID detector using a 1 m, 10% PEG 20M on Chromosorb AW column. All melting points and boiling points are uncorrected.

***N*-Alkyl-*N*-[(trimethylsilyl)methyl]benzylamines 1a,b,d,e.** A solution of *N*-methyl-, *N*-ethyl-, *N*-isopropyl-, or *N*-cyclohexylbenzylamine (21 mmol) and (chloromethyl)trimethylsilane (1.227 g, 10 mmol) in dimethyl sulfoxide (DMSO; 10 mL) was heated at 140 °C for 3–17 h. The mixtures were poured into water (100 mL) and extracted with ether. The extracts were washed with 1% Na_2CO_3, dried ($MgSO_4$), and concentrated. Acetic anhydride (3 mL) was added to the residue at 0 °C, in order to remove unreacted secondary amines, and the mixture was stirred for 3 h at room temperature. Then the mixture was added to 10% HCl and extracted with ether. The acidic solution was neutralized with NaOH and extracted with ether. The ethereal extract was dried ($MgSO_4$) and concentrated. Distillation of the residue gave **1a,b,d,e.** The boiling points and NMR data of the new compounds are summarized in Table III.

***N*-*tert*-Butyl-*N*-[(trimethylsilyl)methyl]benzylamine (1c).** A solution of *tert*-butylamine (12.3 g, 168 mmol), (chloromethyl)trimethylsilane (9.1 g, 74 mmol), and DMSO (40 mL) was heated at 90 °C for 3 h and worked up in a similar manner described above to give 9.75 g (81%) of *N*-*tert*-butyl[(trimethylsilyl)methyl]amine: bp 142–144 °C; ^{1}H NMR ($CDCl_3$) δ 0.00 (s, 9 H, $SiCH_3$), 1.00 (s, 9 H, CCH_3), 1.90 (s, 2 H, NCH_2Si); exact mass calcd for $C_8H_{21}NSi$ 159.1442, obsd 159.1453.

A mixture of *N*-*tert*-butyl[(trimethylsilyl)methyl]amine (3.018 g, 18.9 mmol) and benzyl bromide (1.546 g, 9.0 mmol) was stirred at 140 °C for 1 h. After the addition of water (40 mL), the mixture was extracted with ether. The extract was washed with water, dried ($MgSO_4$), concentrated, and distilled to give 1.882 g (83%) of **1c**. The boiling point and spectral data are shown in Table III.

Benzyldialkyl[(trimethylsilyl)methyl]ammonium Halides 3. **(A)** A mixture of *N*-alkyl-*N*-[(trimethylsilyl)methyl]benzylamine **1a–e** (10 mmol) and alkyl iodide (30 mmol) in acetone or dimethylformamide (10 mL) was treated under the conditions described in Table I. Ether was added with the reaction mixtures, and the precipitated crystals were filtered, washed with ether, and dried. The yields and characteristic data are summarized in Tables I and IV.

(B) A mixture of 7 mmol of 1-[(trimethylsilyl)methyl]pyrrolidine[5] (**2f**) or -piperidine[6] (**2g**) and benzyl bromide (1.44 g, 8.4 mmol) in acetone (10 mL) was heated at reflux for 2 h. The precipitated crystals were filtered, washed with acetone, and dried.

Reaction of Benzyldialkyl[(trimethylsilyl)methyl]ammonium Halides 3 with CsF. In a 20-mL flask equipped with a magnetic stirrer and a dropping funnel was placed the ammonium halides (2 mmol) and CsF (1.5 g, 10 mmol). The flask was dried under reduced pressure and was flushed with high-purity nitrogen. HMPA (10 mL) was added into the flask, and the mixture was stirred at room temperature (the reaction time are

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shown in Table II). The mixture was mixed with 2% Na_2CO_3 (200 mL) and was extracted with ether (100 mL × 4). The extract was washed with 1% Na_2CO_3 (100 mL × 2), dried with $MgSO_4$, and concentrated. Kugelrohr distillation of the residual oil gave a mixture of isomers **5** and **6**. Their structures were confirmed by ^1H NMR, GC-MS, and IR spectra. The ratios obtained from peak areas by GLC were corrected according to response factors obtained from mixtures of known concentrations of **5** and **6** (see Table II).

***N*-Methyl-*N*-(2,2-dimethylpropyl)benzylamine (8).** To a mixture of *N*-methylbenzylamine (2.45 g, 20 mmol) and 30% NaOH (7 g) was added pivaloyl chloride (2.60 g, 22 mmol) at 0 °C with vigorous stirring. After the mixture was stirred for 1 h at room temperature, water was added to the mixture. The ethereal extract of the reaction mixture was washed with water, dried, and evaporated to give 3.88 g (94%) of *N*-benzyl-*N*-methylpivalamide: bp 160 °C (12 mmHg, Kugelrohr); ^1H NMR ($CDCl_3$) δ 1.34 (s, 9 H, CH_3), 2.96 (s, 3 H, NCH_3), 4.63 (s, 2 H, NCH_2C), 7.2 (m, 5 H); IR (Nujol) 1635 cm^{-1}; exact mass calcd for $C_{13}H_{19}NO$ 205.1466, obsd 205.1476.

Then a solution of *N*-benzyl-*N*-methylpivalamide (1.527 g, 7.4 mmol) in ether (5 mL) was added slowly to a suspension of $LiAlH_4$ (440 mg, 12 mmol) in ether (5 mL). After the mixture was stirred for 1.5 h at room temperature, the reaction was quenched with a saturated aqueous solution of potassium sodium tartrate, and extracted with ether. The ethereal extract was dried ($MgSO_4$), concentrated, and distilled to give 1.343 g (95%) of **8**: bp 140 °C (120 mmHg, Kugelrohr) [lit. bp 38-41 °C (0.7 mmHg)[7]].

Registry No. 1a, 51951-99-6; **1b**, 111267-94-8; **1c**, 111267-95-9; **1d**, 111267-96-0; **1e**, 111267-97-1; **2f**, 54600-29-2; **2g**, 17877-17-7; **3a**, 111267-98-2; **3b**, 111267-99-3; **3c**, 111268-00-9; **3d**, 111268-01-0; **3e**, 111268-02-1; **3f**, 111290-73-4; **3g**, 111268-03-2; **5a**, 91338-96-4; **5b**, 70160-72-4; **5c**, 70161-02-3; **5d**, 70160-91-7; **5e**, 6565-69-1; **5f**, 91562-61-7; **5g**, 51180-63-3; **6a**, 73676-26-3; **6b**, 70160-93-9; **6d**, 70160-97-3; **6e**, 5300-21-0; **6f**, 6908-75-4; **6g**, 332-14-9; **7**, 103-83-3; **8**, 68723-29-5; PhCH$_2$NHMe, 103-67-3; PhCH$_2$NHPr-*i*, 102-97-6; *t*-BuNH$_2$, 75-64-9; PhCH$_2$NHEt, 14321-27-8; ClCH$_2$TMS, 2344-80-1; TMSCH$_2$NHBu-*t*, 79250-80-9; PhCH$_2$Br, 100-39-0; C(Me$_3$)COCl, 3282-30-2; C(Me$_3$)CON(Me)CH$_2$Ph, 73551-24-3; *N*-cyclohexylbenzylamine, 4383-25-9.

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Photosensitized Cope Rearrangement of *syn*-7-(1,2-Butadienyl)bicyclo[2.2.1]hept-2-ene [*syn*-7-(3-Methylallenyl)norbornene]†

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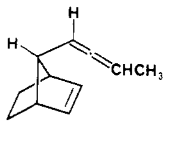
Earlier[1] we reported that *syn*-7-(1,2-butadienyl)bicyclo[2.2.1]hept-2-ene (**1**) undergoes a thermal rearrangement above 160 °C to afford racemic trienes **2**, as an approximate 50:50 mixture of *E* and *Z* diastereomers, whereas the anti epimer **3** was found to be thermally stable. Our results were contrasted with those obtained with corresponding vinyl systems.[2] For example,[2b] at 250 °C, *syn*-7-ethenyl-*anti*-7-methoxynorbornene (**4**) and its anti,syn epimer **5** were found to give the same formal Cope product, 1-methoxy-3a,6,7,7a-tetrahydroindene; 2-methoxybicyclo[3.2.2]nona-2,6-diene, a formal [1,3] sigmatropic shift product, was also formed in each case. These results were interpreted in terms of biradical processes initiated by cleavage of the 1,7-bond in **4** or **5**. Furthermore, Berson[3] has found that the parent vinyl hydrocarbon *syn*-7-ethenylnorbornene is stable at 250 °C, and at 320 °C it decomposes without rearrangement. Thus, the allenyl group affords an improved pathway for rearrangement relative to a vinyl group in these systems, and we have interpreted[1b] the **1** → **2** thermal rearrangement in terms of an orbital symmetry allowed[4] [$_\sigma 2_s + {}_\pi 2_s + ({}_\pi 2_s + {}_\pi 2_a)$] augmented eight-electron pericyclic Cope process.



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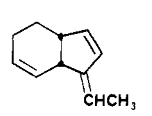

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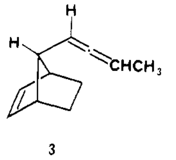

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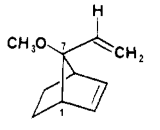

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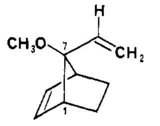

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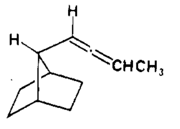

6

We now report on a comparative study of the separate triplet-sensitized photochemistry of bicyclic allenes **1** and **3**. In the past[1b] we separated small amounts (<2 mg) of **1** and **3** by reverse-phase HPLC, through a rather tedious procedure and with a low recovery. We have since found that up to 30 μL of a 1/3 mixture can be successfully separated by rotating disk chromatography on a single 1-mm silica gel–silver nitrate rotor by using a hexane–ethyl acetate solvent gradient.

Equal molar amounts of epimers **1** and **3** were separately photolyzed (254 nm) with an equivalent of *p*-xylene in methanol solution. Kropp[5] has reported that irradiation of bicyclo[2.2.1]hept-2-ene (norbornene) in xylene–methanol gave a complex mixture of products including bicyclo[2.2.1]heptane (norbornane), the product of photoreduction. Photolysis of anti allene **3** ($C_{11}H_{14}$) was shown by capillary GC-MS to give a reduced $C_{11}H_{16}$ photoproduct as the only nonpolymeric product. This product was determined to be 7-(1,2-butadienyl)bicyclo[2.2.1]heptane (**6**) by independent synthesis (cf. Experimental Section). As shown in the plot of reaction mixture composition vs irradiation time for **3** → **6** (Figure 1A), the maximum yield of **6** (24%) occurs after about 6 h. Allenylnorbornane (**6**) is remarkably photostable vis a vis **3** (and **1**, see below). When the photolysis was prolonged for a total of 50 h, the yield of **6** only dropped from 24% to 16% in a nearly linear fashion.

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† Taken in part from the 1987 Undergraduate Thesis of K.S.K.

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