obtain **6** or **7** by this route, a displacement on tertiary carbon at a highly hindered site would be involved, and this process seems unlikely.

Experimental Section[10]

1-Pyrrolidinobicyclo[4.1.0]heptane (1).—Into a 200-ml. flask was charged zinc–copper couple[11] (13.46 g., 0.2 g.-atom), diethyl ether (62 ml.), and methylene iodide (53.6 g., 0.2 mole). A crystal of iodine was added, and the mixture was heated at reflux for 1 hr., whereupon the rate of gas evolution equalled 17 cc./min. The flask was chilled in ice–water under a positive nitrogen pressure and then filtered through a dry, sintered disk. One-half of this filtrate was charged into a 100-ml. flask and pyrrolodino-1-cyclohexene (14.7 g., 0.097 mole) (Aldrich) was added. An immediate reaction occurred, resulting in a gelatinous precipitate. The mixture was heated at reflux for 2 hr., cooled, and treated with two portions of hot ammonium chloride solution. The two-phase system was separated, and the aqueous phase was extracted with two 100-ml. portions of ether and then made alkaline with NaOH. The alkaline solution was extracted with two 100-ml. portions of ether, this extract was dried over magnesium sulfate and filtered, and the ether was evaporated. The residue was combined with the product from an identical reaction and distilled through a semimicro spinning-band column to give 2.57 g. (8%) of product, b.p. 70–74.5° (4 mm.). Purification by gas chromatography afforded pure product having $n^{25}\mathrm{D}$ 1.4876.

Anal. Calcd. for $C_{11}H_{19}N$: C, 80.00; H, 11.50; N, 8.50. Found: C, 79.67; H, 11.85; N, 8.68.

1-Dimethylaminobicyclo[4.1.0]heptane (2).—In a 500-ml. flask was charged chloroiodomethane (53 g., 0.3 mole), zinc–copper couple (20.2 g., 0.3 g.-atom), and ether (63 ml.). The mixture was heated at reflux with stirring for 1 hr. and 20 min. The flask was chilled in an ice–water bath, and dimethylamino-1-cyclohexene[12] (18.75 g., 0.15 mole) was added with stirring. A two-phase system resulted. Tetrahydrofuran (THF) (35 ml.) was added slowly to aid solution, and the mixture was stirred at reflux for 24 hr. The slurry was cooled in an ice bath, and aqueous ammonium chloride (100 ml.) was added slowly. The ether phase was discarded, and the aqueous phase was filtered, extracted twice with 50 ml. of ether, and then made alkaline with a solution of 40 g. of NaOH in 150 ml. of water. The alkaline solution was extracted with three 100-ml. portions of ether. This ether phase was dried over $MgSO_4$ and filtered, and the ether was distilled through a spinning-band column. Distillation of the residue afforded 1.7 g. (8.15%) of product, b.p. 76.5–77° (36 mm.). A sample purified *via* its picrate salt had $n^{25}\mathrm{D}$ 1.4596.

Anal. Calcd. for $C_9H_{17}N$: C, 77.70; H, 12.22; N, 10.08. Found: C, 77.74; H, 12.55; N, 9.97.

1-Dimethylaminobicyclo[3.1.0]hexane (3).—In the manner described for the preparation of 1-dimethylaminobicyclo[4.1.0]-heptane, a reaction between zinc–copper couple (33 g., 0.49 g.-atom), chloroiodomethane (86.5 g., 0.49 mole), and dimethylamino-1-cyclopentene[12] (27.8 g., 0.25 mole) in diethyl ether (200 ml.) was carried out. Fractionation of the product through a semimicro spinning-band column gave 11.19 g. (35.8%) of product which had b.p. 78–81° (100 mm.), $n^{25}\mathrm{D}$ 1.4487–1.4495.

Anal. Calcd. for $C_8H_{15}N$: C, 76.80; H, 12.00; N, 11.20. Found: C, 76.70; H, 12.27; N, 11.04.

1-Bicyclo[3.1.0]hexyltrimethylammonium Bromide (5).—To a cold (−20°) solution of methyl bromide (9.5 g., 0.1 mole) in nitromethane (30 ml.) was added dropwise with stirring 1-dimethylaminobicyclo[3.1.0]hexane (4.91 g., 0.039 mole). The mixture was stirred at −20° for 2 hr. and then allowed to warm to 25° for 40 hr. Ether (250 ml.) was added, and the crystals were collected on a filter and dried under nitrogen to leave 8.0 g. (89%) of **5**. A sample recrystallized from acetonitrile was obtained as white needles, m.p. 252° dec.

Anal. Calcd. for $C_9H_{18}BrN$: Br, 36.30. Found: Br, 36.25.

Reaction of 5 with Methyllithium.—To a stirred slurry of 1-bicyclo[3.1.0]hexyltrimethylammonium bromide (4.27 g., 0.019 mole) in ether (20 ml.) cooled to 0° was added a 2 *N* solution of methyllithium in ether (10 ml., 0.02 mole). The mixture was allowed to warm to 25° and to stir for 5 days. Water (25 ml.) was added, and the ether phase was separated. The aqueous phase was extracted with ether (25 ml.), the combined ether phase was dried over anhydrous magnesium sulfate and filtered, and the ether was removed by fractionation. The residue was flash distilled under vacuum to give 0.5 g. (30%)[13] of hydrocarbon product. Gas chromatography (g.c.) (triscyanoethylated glycerol) revealed one major and two very minor products. The major product was purified by g.c. and shown to be 1-methylbicyclo[3.1.0]hexane (**6**) by comparison of its n.m.r., infrared, and mass spectra.

1-Methylbicyclo[3.1.0]hexane (6).—This hydrocarbon was prepared in 61% yield from Zn–Cu couple (13 g., 0.2 g.-atom), methylene iodide (50 g., 0.19 mole), and 1-methylcyclopentene (8.2 g., 0.1 mole) in ether (60 ml.) according to the general procedure of Simmons and Smith[1–4]: b.p. 90°, $n^{25}\mathrm{D}$ 1.4310.

Anal. Calcd. for C_7H_{12}: C, 87.50; H, 12.50. Found: C, 87.54; H, 12.60.

Reaction of 5 with Phenyllithium.—To a stirred slurry of 1-bicyclo[3.1.0]hexyltrimethylammonium bromide (39.6 g., 0.18 mole) in ether (250 ml.) at 0° was added over 30 min. 1.84 *N* phenyllithium in benzene–ether (250 ml., 0.46 mole). The mixture was stirred at 0° for 3 hr. and then at 25° for 15 hr. The resulting red solution was treated with methanol (10 ml.) and water (250 ml.) whereupon the color disappeared. The organic phase was separated and extracted twice with water (250 ml.). After drying over anhydrous magnesium sulfate, the organic phase was distilled to give 2.16 g. of 1-dimethylaminobicyclo[3.1.0]hexane and 15.08 g. of hydrocarbon product, b.p. 45–55° (0.5 mm.). G.c. (15% squalane on Chromosorb W) revealed three components with areas of 78, 10, and 12%).

The major component was shown to be 1-phenylbicyclo[3.1.0]-hexane by comparison of its infrared and proton n.m.r. spectra with those of a synthetic sample.

The second two components were collected together and then separated by chromatography on silica gel using pentane as solvent. The first component eluted is considered to be 1-benzylbicyclo[3.1.0]hexane. Thus, the proton n.m.r. spectrum has resonances at τ 9.6–9.75 (2H) (cyclopropyl methylene group), 7.28 (2H) (benzylic methylene group), 8.0–9.1 (7H) (broad multiplet), and 2.94 (5H) (aromatic protons). The mass spectrum reveals a parent peak at m/e 172 with large m/e at 91 (benzyl$^+$) and 81 (bicyclo[3.1.0]hexyl$^+$ or more probably cyclohexenyl$^+$).

The third component was identified as biphenyl, present in the phenyllithium used.

1-Phenylbicyclo[3.1.0]hexane (7).—This hydrocarbon was prepared from Zn–Cu couple (27.3 g., 0.435 g.-atom), methylene iodide (83.8 g., 0.315 mole), and 1-phenylcyclopentene (28.8 g., 0.182 mole) in ether (115 ml.) according to the general procedure of Simmons and Smith.[1–4] Flash distillation under vacuum gave 21.7 g. of product consisting of 66% of **7** and 34% of starting olefin. The product, **7**, was separated by g.c. and had b.p. 236° (micro), $n^{25}\mathrm{D}$ 1.5458.

Anal. Calcd. for $C_{12}H_{14}$: C, 91.15; H, 8.85. Found: C, 91.29; H, 8.96.

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[10] Melting and boiling points are uncorrected.

[11] R. S. Shank and H. Shechter, *J. Org. Chem.*, **24**, 1825 (1959).

[12] E. P. Blanchard, Jr., *ibid.*, **28**, 1397 (1963).

(13) It should be noted that the cyclopropene mechanism requires 2 moles of alkyllithium reagent. This yield would then actually be 60% based on alkyllithium reagent.

Perfluoroalkanesulfonate Esters as Alkylating Agents

ROBERT L. HANSEN

Contribution No. 345 from Central Research Laboratories,
Minnesota Mining and Manufacturing Company,
St. Paul, Minnesota 55119

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The recent paper of Burdon and McLoughlin[1] concerning trifluoromethanesulfonate esters prompts us to report our results concerning these esters. Alkyl

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(1) J. Burdon and V. C. R. McLoughlin, *Tetrahedron*, [1] **21**, 1 (1965).

TABLE I

SUMMARY OF PROPERTIES OF PERFLUOROALKANESULFONATE ESTERS

Compd.	B.p., °C. (mm.)	$n^{25}D$	Yield, %	Calcd.,% C	Calcd.,% F	Found, % C	Found, % F
$CF_3SO_2OCH_2CF_3$	89–91 (740)	1.3037	74	15.5	49.1	15.6	49.4
$CF_3SO_2OCH_2CF_2CF_3$	102–105 (740)	1.3012	77	17.0	53.9	16.9	54.3
$CF_3SO_2OCH_2(CF_2)_2CF_3$	118–120 (730)	1.3020	81	18.1	57.2	18.2	57.3
$CF_3SO_2OCH_2CF_2CF_2H$	122–125 (737)	1.3203	71	18.2	50.4	18.3	50.5
$CF_3CF_2SO_2OCH_2CF_3$	105–106 (740)	1.3028	70	17.0	53.9	16.8	54.6

esters of perfluoroalkanesulfonic acids, first described by Brown[2] and Brice and Trott[3] are very reactive alkylating agents. For example, methyl or ethyl trifluoromethanesulfonate will alkylate benzene if a mixture of the two is warmed. These esters will also alkylate ether to give an ethereal solution containing trialkyloxonium trifluoromethanesulfonate.[2,4]

A measure of the great reactivity of the trifluoromethanesulfonate group (TFMS) and higher homologs is found in the acetolysis rates of their methyl esters. Methyl trifluoromethanesulfonate (I) and methyl nonafluorobutanesulfonate (II) have first-order solvolytic rate constants in acetic acid of $(7.13 \pm 0.05) \times 10^{-5}$ and $(1.49 \pm 0.03) \times 10^{-4}$ sec.$^{-1}$, respectively. Extrapolation of the rate data to 25° for acetolysis of methyl *p*-toluenesulfonate at 75 and 100°[5] gives $k = 3.1 \times 10^{-9}$ sec.$^{-1}$. Thus, the perfluoroalkanesulfonate groups are 10^4 times as reactive as the toluenesulfonate group in acetolysis.

$$\underset{\text{I}}{CF_3SO_2OCH_3} \qquad \underset{\text{II}}{C_4F_9SO_2OCH_3}$$

Fluorocarbon sulfonate leaving groups can enhance reactivity in alkylating systems which are quite unreactive with arenesulfonate or halide leaving groups. For example, the 1,1-dihydroperfluoroalkyl halides and arenesulfonates are quite unreactive to nucleophilic substitution.[6-8] The 1,1-dihydroperfluoroalkyl trifluoromethanesulfonates should react readily.

A series of 1,1-dihydroperfluoroalkyl trifluoromethanesulfonates (III) were prepared by treating trifluoromethanesulfonyl fluoride and the appropriate alcohol in the presence of triethylamine in methylene chloride solution at −30°. Ester formation was rapid and

$$R_FCH_2OH + CF_3SO_2F \longrightarrow \underset{\text{III}}{CF_3SO_2OCH_2R_F}$$

$$R_F = \text{perfluoroalkyl}$$

yields were good. Table I lists properties of some of these esters.

When 2,2,2-trifluoroethyl trifluoromethanesulfonate reacts with excess diethylamine in refluxing benzene, a good yield of the tertiary amine IV is obtained. Brown and Tiers[9] report that the corresponding *p*-toluenesulfonate (CF_3CH_2OTs) reacts only with great difficulty or not at all in amine alkylations.

$$CF_3SO_2OCH_2CF_3 + 2Et_2NH \longrightarrow \underset{\text{IV}}{CF_3CH_2NEt_2} + CF_3SO_3^{-}H_2\overset{+}{N}Et_2$$

High temperatures are required for replacement of the toluenesulfonate group by halide ion.[6-8] Trifluoroethyl trifluoromethanesulfonate is found to react more than 10^5 times as fast as CF_3CH_2Br with I^- in acetone. McBee and co-workers[8] studied the Finkelstein reaction of several polyfluoroalkyl bromides with KI in acetone. Extrapolation of their data for CF_3CH_2Br at 115 and 160° to 25° gives $k_2 = 2.1 \times 10^{-7}$ l. mole^{-1} sec.$^{-1}$. $CF_3SO_2OCH_2CF_3$ is found to react with 0.10 *N* NaI in acetone at 25.0° with $k_2 = (1.49 \pm 0.03) \times 10^{-2}$ l. mole^{-1} sec.$^{-1}$.

Table II lists first-order rate constants for methanolysis of several TFMS esters as an indication of their general reactivity. This reactivity is great enough to permit a wide variety of nucleophilic substitutions on these esters. The toluenesulfonate group, an anion of low nucleophilicity,[10] will displace CF_3SO_3 readily to form $C_3F_7CH_2OTs$ when LiOTs reacts with $C_3F_7CH_2OSO_2CF_3$ in DMF at 120°. In general we find that numerous other anions, with nucleophilicities greater than that of toluenesulfonate, easily displace CF_3SO_3 from these esters.

TABLE II

RATE OF METHANOLYSIS AT 100.0° FOR $CF_3SO_2OCH_2R_F$

R_F	$k_1 \times 10^5$, sec.$^{-1}$
CF_3	4.32 ± 0.04
CF_3CF_2	1.01 ± 0.01
$CF_3CF_2CF_2$	0.631 ± 0.006
HCF_2CF_2	6.46 ± 0.04
CF_3[a]	6.76 ± 0.1

[a] Rate of methanolysis of $C_2F_5SO_2OCH_2CF_3$.

Experimental Section

Preparation of $CF_3SO_2OCH_2CF_3$.—Into a tared 1-l. round-bottom flask fitted with mechanical stirrer, alcohol thermometer, Dry Ice cold-finger condenser, acetone–Dry Ice bath, gas-inlet tube, and drying tube was passed dry N_2, and then CF_3SO_2F was passed in and condensed. The flask was removed and weighed periodically to ascertain the charge of sulfonyl fluoride. When 84.5 g. (0.555 mole) of CF_3SO_2F had been condensed, 100 ml. of CH_2Cl_2 at −80° was added. The gas inlet tube was replaced by a dropping funnel containing 55.5 g. (0.555 mole) of CF_3CH_2OH and 56 g. (0.555 mole) of Et_3N. The mixture was added slowly over a 20-min. period at such a rate as to maintain the reaction at −30 to −40°. After addition the reaction was warmed to 0°, then the clear CH_2Cl_2 solution was washed with 100 ml. of 5% HCl, 100 ml. of 5% NaOH, and twice with 50 ml. of water, dried over $MgSO_4$, and filtered. The CH_2Cl_2 solution was concentrated carefully on the steam bath by distillation through a short column. The residue upon distillation through a 10-cm. fractionating column yielded 95.5 g. (74.5%) of $CF_3SO_2OCH_2CF_3$, b.p. 89–91° (740 mm.), $n^{25}D$ 1.3037.

The compounds listed in Table I were prepared using a similar procedure.

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(2) H. A. Brown, 128th National Meeting of the American Chemical Society, Minneapolis, Minn., April 1955; Abstracts, p. 29M.

(3) T. J. Brice and P. W. Trott, U. S. Patent 2,732,398 (Jan. 1956).

(4) T. Gramstad and R. N. Haszeldine, *J. Chem. Soc.*, 4069 (1957).

(5) S. Winstein and H. Marshall, *J. Am. Chem. Soc.*, **74**, 1120 (1952).

(6) G. V. D. Tiers, H. A. Brown, and T. S. Reid, *ibid.*, **75**, 5978 (1953).

(7) W. V. Cohen, *J. Org. Chem.*, **26**, 4021 (1961).

(8) E. T. McBee, R. D. Battershell, and H. P. Braendlin, *J. Am. Chem. Soc.*, **84**, 3157 (1962).

(9) H. A. Brown and G. V. D. Tiers, *J. Org. Chem.*, **22**, 454 (1957).

(10) C. G. Swain and C. B. Scott, *J. Am. Chem. Soc.*, **75**, 141 (1953).

Alternatively, the 1,1-dihydroperfluoroalkyl alcohol and a strong tertiary base such as triethylamine can be placed in the apparatus and maintained at 0°. Then CF_3SO_2F is introduced until the solution remains saturated with CF_3SO_2F as indicated by a persistent reflux. Excess sulfonyl fluoride is removed by applying a gentle vacuum to the reaction. The rest of the work-up is as described.

Alkylation of Diethylamine with $CF_3SO_2OCH_2CF_3$.—A 500-ml. round-bottom flask magnetically stirred and fitted with a reflux condenser was charged with 46.4 g. (0.20 mole) of $CF_3SO_2OCH_2CF_3$ and 32.2 g. (0.44 mole) of diethylamine in 100 ml. of benzene. The solution was refluxed and stirred 1 hr. during which time a light brown oil separated. Upon cooling to 0°, the oil crystallized. It was separated by filtration, slurried with cold benzene, and dried to give 44 g. (98%) of $Et_2NH \cdot CF_3SO_3H$. The combined benzene solution was extracted with 100 ml. of 10% HCl. The HCl extract was washed with 20 ml. of ether, then neutralized with concentrated NaOH. The clear oil which separated was extracted with three 50-ml. aliquots of ether, dried over $MgSO_4$, and concentrated carefully through a short column. The residue was distilled at atmospheric pressure through a 10-cm. column to yield 26.1 g. (84%) of $CF_3CH_2N(CH_2CH_3)_2$, b.p. 89–91° (740 mm.), n^{25}D 1.3508. The hydrochloride was prepared in ether solution with HCl gas, m.p. 164–165°.

Anal. Calcd. for $C_6H_{13}ClF_3N$: C, 37.6; F, 29.7. Found: C, 37.6; F, 29.8.

Preparation of 1,1-Dihydroperfluorobutyl *p*-Toluenesulfonate.—Lithium *p*-toluenesulfonate (2.0 g.) and 2.0 g. of 1,1-dihydroperfluorobutyl trifluoromethanesulfonate were dissolved in 8 ml. of DMF and heated to 120° whereupon a vigorous reaction occurred. The mixture was then refluxed 0.5 hr. longer, cooled, poured into 30 ml. of cold water, and extracted into 20 ml. of ether. The ether phase was washed with 10 ml. of water, dried over $MgSO_4$, filtered, and concentrated to an oil which was then distilled to yield 1.5 g. of colorless liquid, b.p. 65–75° (1 mm.), which eventually solidified, m.p. 28–29°, identical with an authentic sample prepared earlier.[6]

Kinetic Determinations. A. Methanolysis.—Rates of methanolysis were determined using essentially the same kinetic procedure as Winstein, *et al.*[11] Methanol was dried as described by Fieser.[12]

B. **Iodide Ion.**—The reaction rate of iodide ion with $CF_3CH_2OSO_2CF_3$ in acetone was determined as follows. Reagent grade acetone, dried over molecular sieves, was distilled. A solution of NaI in acetone was made by dissolving 1.499 g. (0.0100 mole) of NaI (A.R.) in dry acetone at 25.0° in a 100-ml. volumetric flask. In a 50-ml. volumetric flask was weighed 1.160 g. (0.00500 mole) of $CF_3CH_2OSO_2CF_3$. This was made up to the mark with the NaI in acetone solution and thermostated at 25.0°. At various intervals, a 5.00-ml. aliquot of solution was withdrawn and pipetted into a mixture of 10 ml. of distilled water and 5 ml. of ether in a separatory funnel. The aqueous layer was separated, and the ether was thoroughly washed with water four times. The combined aqueous phase was added to 5.00 ml. of 0.100 *N* $AgNO_3$ and titrated potentiometrically with 0.1162 *N* NH_4SCN. As the initial iodide concentration equals the ester concentration, the reaction was treated as a homogeneous second-order rate and integrated rate constants were calculated for each point using the equation $k = (1/A - 1/A_0)/t$, where A is ester and iodide concentration at time t and A_0 is initial ester and iodide concentration (see Table III).

C. **Acetolysis of $CF_3SO_2OCH_3$.**—The equipment consisted of a wide-mouth test tube which was inserted in a glass jacket. Water from a thermostat at 25.0° was circulated rapidly through the jacket while a small magnetic stirring bar in the test tube provided mixing. A 5-ml. buret was inserted into the test tube through a rubber stopper and a drying tube was attached. The buret was filled with glacial acetic acid, 0.0200 *N* in NaOAc and 0.01 *N* in Ac_2O.

Approximately 16 mg. (0.1 mmole) of $CF_3SO_2OCH_3$ was placed in the bottom of the test tube, 2 ml. of dry acetic acid, 0.01 *M* in Ac_2O, and 2 drops of saturated brom thymol blue indicator in acetic acid were added. The acetic acid solution was continuously titrated to neutrality. Time *vs.* titer was recorded at appropriate intervals. The final titer was determined after 10 "half-lives." Calculated integrated rate constants between 10 and 70% reaction were constant within the error limits reported here. The acetolysis rate of $CF_3CF_2CF_2CF_2SO_2OCH_3$ was similarly determined.

TABLE III

Point	Time,[a] min.	Titer,[b] ml. of SCN	$k_2 \times 10^2$, moles l.$^{-1}$ sec.$^{-1}$
1	0.00	0.575	...
2	5.00	1.445	1.46
3	14.00	2.245	1.48
4	21.00	2.570	1.46
5	26.00	2.940	1.49
6	33.00	2.965	1.54
7	∞[c]	3.955	...

$k = (1.49 \pm 0.03) \times 10^{-2}$ l. mole^{-1} sec.$^{-1}$

[a] $t_{1/2} \cong 22$ min. [b] Blank of $AgNO_3$ solution requires 4.062 ml. of NH_4SCN solution. [c] At 35° for 1.5 hr.

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[11] S. Winstein, B. Morse, E. Grunwald, K. C. Schreiber, and J. Corse, *J. Am. Chem. Soc.*, **74**, 1113 (1952).

[12] L. F. Fieser, "Experiments in Organic Chemistry," 3rd Ed. (revised), D. C. Heath and Co., Boston, Mass., p. 289.

The Pyrolysis of Tetracyclone[1]

EDWARD McNELIS

Research and Development,
Sun Oil Company, Marcus Hook, Pennsylvania

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During an investigation of the formation of benzyne intermediates at elevated temperatures,[2] tetraphenylcyclopentadienone (tetracyclone) was used as a trapping agent at 300–325°. The ketone was shown to be stable at these temperatures, but at 375° a decomposition was evident. When tetracyclone (I) was pyrolyzed at 410–425° in a nitrogen atmosphere, none of the ketone was recovered. At present, several products have been isolated from the reaction mixture and three have been characterized—1,2,4-triphenylnaphthalene, tetraphenylcyclopentenone, and 5,6-diphenyl-11(H)-benzo[*a*]fluoren-11-one. Their formation poses interesting mechanistic questions and represents potentially convenient entries into otherwise difficultly accessible types of compounds.

The principal product of the pyrolysis of I is 1,2,4-triphenylnaphthalene (II). Structural identification was made by comparison with authentic material of infrared spectrum, ultraviolet spectrum,[3] and melting point. The authentic sample of II was prepared by the method of Blum[4]—2,2-diphenylvinyl bromide and sodium in ether followed by methanol and subsequent dehydrogenation. The yields of purified II range from 10 to 25%; the crude values range from 25 to 42%.

The second characterized product is tetraphenylcyclopentenone (III), which is formed in about 13% yield. It is identical in all respects with authentic material.[5] This compound was reported as a reaction product of the treatment of I with selenium at 380°.[6]

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[1] Presented in part at the 147th National Meeting of the American Chemical Society, Philadelphia, Pa., April 1964; Organic Division Abstracts, p. 33N.

[2] E. McNelis, *J. Org. Chem.*, **28**, 3188 (1963).

[3] R. Breslow and M. Battiste, *J. Am. Chem. Soc.*, **82**, 3626 (1960).

[4] O. Blum, *Chem. Ber.*, **62**, 881 (1939).

[5] N. Sonntag, S. Linder, E. Becker, and P. Spoerri, *J. Am. Chem. Soc.*, **75**, 2283 (1953).

[6] L. Fortina and G. Montaudo, *Ann. chim.* (Rome), **50**, 1401 (1960).