gene/ml of benzene) was added to 1.0 g of solasodine acetate (Ia) in 55 ml of benzene. Following the addition of 9 ml of pyridine, the reaction mixture was heated under reflux for 1 hr. An aqueous solution (25 ml) of dimethylamine (25%) was then added to the mixture with stirring and the reaction was continued for 15 min. The organic phase was washed with water, 2 *N* HCl, and again water. After removal of the solvent, the residue, twice crystallized from absolute EtOH, yielded needles of V (415 mg): mp 264–267°; $[\alpha]^{19}D$ −285° (*c* 1.0, $CHCl_3$); ir (CCl_4) 1732, 1240 (OAc), 1700, 1653 (-OCON<), 1335, 1316 cm^{-1}; nmr ($CDCl_3$) δ 4.06 (m, 1), 4.59 (m, 1), 4.72 (m, 1), 4.83 (t, 1, H-23), 5.37 (d, 1, H-6); mass spectrum *m/e* 481 (M^+).

Anal. Calcd for $C_{30}H_{43}O_4N$: C, 74.81; H, 9.00; N, 2.91. Found: C, 74.87; H, 8.81; N, 2.78.

The mother liquors were combined and evaporated to dryness. The residue was dissolved in pyridine (2.5 ml) and treated with acetic anhydride (1.0 ml). The mixture was then allowed to stand at room temperature for 1 day. After the usual work-up, the resinous residue was chromatographed on alumina (neutral, grade III, 30 g). Elution with toluene afforded a further crop of V (141 mg) and a subsequent fraction eluted with toluene and ethyl acetate (10%) yielded plates of the pseudoformamido compound VI (87 mg) from ethyl acetate: mp 154–159°; $[\alpha]^{18}D$ −30.2° (*c* 0.8, $CHCl_3$); ir (CCl_4) 3488 (NH), 1737, 1248 (OAc), 1694 (CCO), 1518, 1669 cm^{-1} (NHCO-); nmr ($CDCl_3$) δ 0.70 (s, 3), 0.92 (d, 3, *J* = 5.8 Hz), 1.04 (s, 3), 1.60 (s, 3), 2.02 (s, 3), 2.89 (s, 6), 3.12 (t, 2, *J* = 5.8 Hz), ~4.1 (m, 2), 5.38 (d, 1, *J* = 3.5 Hz); mass spectrum *m/e* 526 (M^+).

Anal. Calcd for $C_{32}H_{50}O_4N_2$: N, 5.32. Found: N, 5.45.

Reaction of 5,6-Dihydrosolasodine Acetate with Phosgene–Pyridine.—To 45 ml of a benzene solution of 5,6-dihydrosolasodine acetate (0.84 g) was added 4 ml of a benzene solution of phosgene (0.34 g of phosgene/ml of benzene) while the reaction flask was cooled in ice–water. After the reaction mixture had stood for 5 min at room temperature, 3 ml of pyridine was added and the mixture was refluxed for 1 hr. Then 20 ml of 25% aqueous dimethylamine was added to the cold reaction mixture with stirring and agitation for another hour. Following the addition of water, the reaction mixture was extracted with benzene. The benzene extract, after successively being washed with water, 2 *N* HCl solution, and water, yielded needles of Va (243 mg) from absolute EtOH: mp 288.5–291°; $[\alpha[^{18}D$ −225° (*c* 0.4, $CHCl_3$); ir 1735, 1245 (OAc), 1704, 1657 (NCOO-), 1339, 1321, 1189 cm^{-1}; nmr ($CDCl_3$) δ 4.06 (m, 1), 4.68 (m, 2), 4.82 (t, 1, H-23).

Anal. Calcd for $C_{30}H_{45}O_4N$: C, 74.49; H, 9.38; N, 2.90. Found: C, 74.62; H, 9.28; N, 2.86.

Reaction of Solasodine Acetate (Ia) with Phosgene–Triethylamine.—To 3 g of solasodine acetate (Ia) dissolved in 100 ml of benzene and 30 ml of triethylamine was added with stirring 20 ml of 12.5% phosgene in benzene during the course of 3 min while cooling the reaction flask in ice–water. After 1 hr at room temperature, 60 ml (25%) of aqueous dimethylamine was added with stirring and cooling of the reaction mixture. Vigorous stirring was continued for another hour. The benzene layer was successively washed with water, 2 *N* HCl, and water. The residue, after removal of the solvent, yielded a crude crystalline material [tlc, $CHCl_3$(2):EtOAc(1)] which was chromatographed on neutral alumina (95 g, grade III). Fractions eluted with benzene gave impure crystals of II: mp 160–170° (2.14 g); ir (CCl_4) 1735, 1245 (AOc), 1667 (-CON-), 979, 911 cm^{-1} (spiro amino ketal linkage). The compound is very unstable and attempts at purification by crystallization in acetone seemed to lead to diverse products. Therefore II was treated with 10 ml of boiling HOAc containing 0.1 ml of Ac_2O for 8 min, and the product after removal of the acid *in vacuo* was crystallized from EtOAc and then from aqueous CH_3OH to form plates (1.469 g), mp 158–161°. It was identical with VI (mixture melting point, ir, tlc).

Subsequent fractions eluted with 10% EtOAc in benzene gave rhombic crystals of III from ether: mp 132–140° (0.58 g); ir (CCl_4) 3445 (NH), 1736, 1243 (OAc), 1688 (C=CO), 1662, 1519 cm^{-1} (-NHCO-). Attempts to recrystallize the compound from hot CH_3OH isomerized it partially to VI. The same compound was obtained by treating 0.3 g of crude III with 1.5 ml HOAc containing 2 drops of Ac_2O under reflux for 10 min. The residue crystallized from aqueous CH_3OH to yield 0.17 g of VI melting at 151–156°. The ir spectrum was superposable with that of an authentic specimen. In another run of the same reaction, a 22ξ-hydroxy compound (IV) was obtained in poor yields from fractions eluted with 30% EtOAc in benzene. It melted at 163–172° and possessed the following spectral bands: ir 3590 (OH), 3478 (NH), 1728, 1250 (OAc), 1643, 1526 cm^{-1} (-NHCO-); mass spectrum *m/e* 526 ($M^+ - H_2O$). IV, like III and II, was converted into VI with HOAc.

Upon closer examination of the reaction products with tlc ($CHCl_3$:EtOAc, 20:1), small amounts of compounds V and VI were also detected. Although attempts were made to run the experiments under identical conditions, the production of the products, II, III, and IV, was always variable.

Reduction of Va.—Va (185 mg) was dissolved in 30 ml of AcOH and with 180 mg of Pd/C (180 mg) and hydrogenated for 2 days under atmospheric pressure at 25° when uptake ceased. The compounds were separated by preparative tlc with the solvent systems $CHCl_3$:EtOAc (20:1 and 10:1). Two compounds (IX and IXa) along with the starting material (Va) were obtained. Compound IX (18 mg), needles (CH_3OH), melted at 323.5–324.5°: ir (CCl_4) 1735, 1247 (OAc), 1697 cm^{-1} (OCON<). It was identical (melting points, mixture melting point, tlc, and ir) with the synthetic specimen. The isomeric IXa melted at 276–278° (8 mg): ir (CCl_4) 1736, 1248 (OAc), 1700 cm^{-1} (-OCON); mass spectrum *m/e* 485 (M^+, $C_{30}H_{47}NO_4$).

Hydrolysis of V.—V (36 mg) was dissolved in CH_3OH (4 ml) and a drop of water and 105 mg KOH were added. The mixture was refluxed for 2 hr. The free alcohol crystallized from MeOH as needles: mp <330°; $[\alpha]^{18}D$ −305° (*c* 0.4, $CHCl_3$); ir ($CHCl_3$) 3590 (OH), 1678, 1652 cm^{-1} (-OCON).

Anal. Calcd for $C_{28}H_{41}O_3N$: C, 76.49; H, 9.04; N, 3.19. Found: C, 76.57; H, 9.20; N, 3.10.

Sodium Borohydride Reduction of V.—V (150 mg) was dissolved in 2 ml of CH_2Cl_2 and treated with a solution of 300 mg of $NaBH_4$ in 8 ml of EtOH containing a few drops of water. After 4 hr of refluxing, the reaction mixture was acidified with 2 *N* HCl and extracted with $CHCl_3$. The residue, when crystallized, proved to be the C-3 alcohol as in the above hydrolysis.

Oxidation of VI to VII.—To 30 mg of VI dissolved in 2 ml of HOAc was added dropwise 1.8 ml of a solution (80% HOAc) of CrO_3 (11.4 mg, 2 molar equiv) with stirring while the reaction flask was being cooled with ice–water. The mixture was stirred at room temperature for 1 hr and then quenched with water followed by addition of a pinch of Na_2SO_3 to decompose the excess CrO_3. The reaction mixture was saturated with NaCl and extracted with ether. After removal of the ether the residue was dissolved in HOAc (5 ml) and refluxed for 2 hr. The HOAc was removed *in vacuo* and the dry residue was chromatographed on 2 g of alumina (neutral, grade I). Fractions eluted with benzene (40 ml) and benzene–ethyl acetate (23:2; 25 ml) were combined and twice crystallized from aqueous CH_3OH to yield needles, mp 172–175°, which agreed in properties (melting point, mixture melting point, and ir) with an authentic specimen of VII.

Registry No.—II, 34608-94-1; III, 34638-80-7; IV, 34638-81-8; V, 34638-82-9; Va, 34638-83-0; V free alcohol, 34638-84-1; VI, 34638-85-2; IX, 24694-77-7; IXa, 34638-87-4.

Studies on the Oxidation of Homosemibullvalene (Tricyclo[6.1.0.0^{4,9}]nona-2,6-diene). Photosensitized Oxygenation

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Received January 3, 1972

The dye-sensitized photooxygenation of organic compounds has been studied extensively by many workers and represents a very smooth method for introducing

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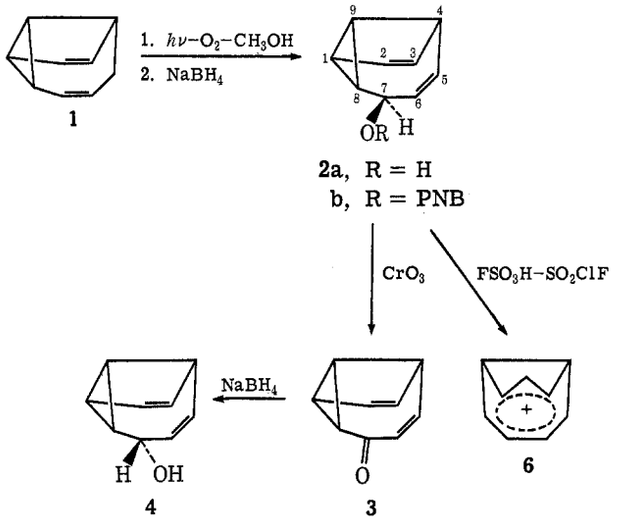
oxygen in a highly specific fashion into organic compounds.[3] Typically reactions of singlet oxygen with olefins have been studied with olefins possessing allylic hydrogen atoms.[4] It was suggested that the resulting allylic hydroperoxides arose through an "ene"-type mechanism.[5]

Now we wish to report the successful preparation of *exo*-tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-ol (homosemibullvalenol) (**2a**),[6] an important intermediate from which 1,4-bishomotropylium ion (**6**) is formed, by singlet oxygen oxidation of tricyclo[6.1.0.0^{4,9}]nona-2,6-diene (homosemibullvalene) (**1**).[7] In our laboratory, we have recently prepared **6** by extraction of **2a** from a CD_2Cl_2 solution into a mixture of FSO_3H–SO_2ClF at $-135°$ and observed by nmr at $-125°$.[8]

When the photooxidation of **1** was conducted in methanol solution, **2a**, mp 88.5–89.5°, was obtained in 35% yield after $NaBH_4$ reduction of the hydroperoxide mixture (Scheme I). All photooxidations were performed at room temperature, using a 200-ml Pyrex immersion well apparatus fitted with an oxygen bubbler and a Sylvania DWY projection bulb. Rose bengal was used as dye sensitizer, and reagent grade anhydrous methanol was used as the solvent. The above-described reaction did not occur when oxygen, dye, or irradiation was omitted. Irradiation times varied from 55 min to 15 hr depending upon the concentration of **1**.

SCHEME I



The structure of the allylic alcohol **2a** was determined by its nmr spectrum, which consisted of a multiplet at τ 4.14 (2 H, olefinic), a doublet at τ 4.40 (1 H, olefinic), a doublet at τ 5.17 (1 H, olefinic), a multiplet at τ 6.52 (2 H, α-H and bisallylic), a sharp singlet at τ 7.55 (1 H, hydroxyl), and a multiplet at τ 7.92–8.88 (3 H, cyclopropyl), and by its mass spectrum, which showed a molecular ion peak at *m/e* 134 with isotope peak at *m/e* 135 (P + 1, 9.90) and 136 (P + 2, 0.65) of the appropriate intensities for the formula $C_9H_{10}O$.

Oxidation of **2a**, R_f[9] 0.31, ν_{max} 3601 cm^{-1} (CCl_4), with chromium trioxide in moist ether gave tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-one (**3**) (R_f 0.81). Reduction of the ketone **3** with $NaBH_4$ in methanol provided only the new alcohol **4**, *endo*-tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-ol, R_f 0.54, ν_{max} 3550 cm^{-1} (sharp, strong) (CCl_4), nmr τ 5.5 (α-H, $CDCl_3$). The endo configuration was assigned to **4** because of the very hindered hydroxyl group, as indicated by rapid elution from alumina, the strong 3550 cm^{-1} infrared absorption, and the deshielding of the α H from τ 6.5 in **2a** to 5.5 in **4**.

exo-Tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-yl *p*-nitrobenzoate (**2b**), mp 138.5–139.5°, was prepared by the usual procedure and its structural assignment has been made from a 100-MHz nmr spectrum and a decoupling experiment. **2b** was decoupled by irradiating at the H_7 resonance, the H_4 resonance, and the H_1 resonance. The coupling constants shown in Table I were obtained from these experiments.

TABLE I
CHEMICAL SHIFTS (τ) AND COUPLING CONSTANTS (J) FOR **2b** ($CDCl_3$)

		J, Hz							
	τ	2	3	4	5	6	7	8	9
1	7.86	2.1	Small	0	0	0	0	8.0	7.5
2	4.20		5.0	1.5	0	0	0	0	0
3	5.00			1.0	0	0	0	0	0
4	6.26				7.0	0	0	0	7.0
5	3.83					9.2	2.5	0	0
6	4.09						1.0	1.0	0
7	5.16							1.7	0
8	8.53								8.0
9	8.16								
Aromatic	2.73								

The assignment of stereochemistry at C_7 is based on the vicinal coupling constants. **2b** has J_{76} = 1.0 and J_{75} = 2.5 Hz. The observed coupling constants are most consistent with the structure shown. The H_7–H_6 dihedral angle is *ca.* 90°, which should give a small coupling constant, whereas the H_7–H_5 angle is also *ca.* 90°, which should give a larger coupling constant because of allylic coupling.[10] This assignment corroborates the previous assignment by R_f, ν_{max} (OH), and τ (α H) values.

A number of other unsuccessful attempts (A–F) to prepare the allylic alcohol are outlined in Scheme II. No other products were detected by tlc or vpc in any of these systems.

The *p*-nitrobenzoate **2b** also might be a precursor for **6** and a full discussion of the solvolysis will be forthcoming.

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(6) **2a**, **2b** and **4** are racemic and attempts to separate these into optically active components were not examined.

(7) **1** was prepared by Wolff–Kishner reduction of bicyclo[4.2.1]nona-2,4,7-trien-9-one: M. Sakai, D. L. Harris, and S. Winstein, *Chem. Commun.*, in press.

(8) P. Ahlberg, D. L. Harris, and S. Winstein, *J. Amer. Chem. Soc.*, **92**, 4454 (1970).

(9) See Experimental Section.

(10) S. Sternhell, *Rev. Pure Appl. Chem.*, **14**, 15 (1964).

SCHEME II

(A)[a] 1 + SeO_2 ⟶ Hydrocarbons and Polymers

(B)[b] 1 + t-BuO_2COPh ⟶ (OH⁻) ⟶ Seven Alcohols

(C) 1 + $Pd(OAc)_2$ ⟶ (LiAlH₄) ⟶ Hydrocarbons

(D)[c] 1 + NBS ⟶ (AgOAc) ⟶ (OH⁻) ⟶ Polymers

(E) 1 + $Hg(OAc)_2$ ⟶ Recovered 1

(F)[d] 1 + CrO_3–Py_2 ⟶ Recovered 1

[a] R. B. Woodward and T. J. Katz, *Tetrahedron*, **5**, 70 (1959). [b] H. L. Goering and U. Mayer, *J. Amer. Chem. Soc.*, **86**, 3753 (1964). [c] A. C. Cope, M. Brown, and H.-H. Lee, *ibid.*, **80**, 2855 (1958). [d] W. G. Dauben, personal communication.

Experimental Section

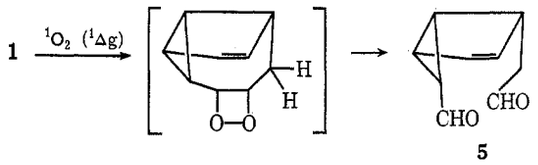
Melting points were taken in capillaries and are uncorrected.

exo-**Tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-ol (2a).**—A solution of 2.0 g of **1** and 15 mg of rose bengal in 100 ml of anhydrous methanol was bubbled with oxygen and irradiated with a tungsten–iodine lamp until the absorption of oxygen ceased (calcd 200 ml, obsd 350 ml). The reaction mixture was reduced with 10 g of $NaBH_4$ at room temperature, quenched with 100 ml of 20% KOH solution, and then extracted with ether. The ether layer was dried over K_2CO_3 and evaporation of ether gave a viscous oil. This oil was chromatographed on a column of 100 ml of Silica AR with 10% ether–pentane, and divided into 20 fractions of 150 ml each. After evaporation of the solvent, fractions 6, 7, and 8 gradually crystallized on standing at room temperature to give white needles, mp 87.0–88.0°. These were recrystallized from pentane to give 800 mg (35%) of white needles: mp 88.5–89.5°; nmr, see text; ir (CCl_4) 3601 (m), 3360 (s), 3050 (s), 2915 (m), 2840 (w), 1586 (w), 1376 (m), 1341 (m), 1265 (m), 1038 (s), 997 (s), 970 (m), 956 (m), 942 (w), 919 (m), 910 (m), 892 (m), 853 (w), 727 (m) and 693 cm^{-1} (s); mass spectrum, parent peak at m/e 134 ($C_9H_{10}O^+$) and a base peak at m/e 43; R_f 0.31 (5 × 20 alumina coated plate eluted with 25% ether–pentane).

Anal. Calcd for $C_9H_{10}O$: C, 80.56; H, 7.51. Found: C, 80.45; H, 7.56.

Fractions 1 and 2 consisted of a mixture of hydrocarbons (0.1 g). Combination of fractions 3, 4, and 5 yielded a viscous oil (0.5 g). The ir spectrum of this oil showed a characteristic aldehyde absorption at 2710 and 1730 cm^{-1}, a double bond absorption at 1650 cm^{-1}, and a cyclopropyl absorption at 3050 cm^{-1} (neat). We believe that this component is dialdehyde **5**. A possible explanation for the unusual formation of this aldehyde is presented in Scheme III. $^1\Delta g$ oxygen reacts with **1** to form a

SCHEME III



5

1,2-dioxetane intermediate[11] which cleaves to **5**. A more detailed report on further chemistry of **5** is forthcoming.

Fractions 9–15 consisted of viscous polymers (1.0 g). Fractions 16–20 consisted of small amounts of polymers and inorganic materials (0.1 g).

exo-**Tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-yl *p*-Nitrobenzoate (2b).**—To a solution of 100 mg of **2a** in 3 ml of dry pyridine was added 270 mg of *p*-nitrobenzoyl chloride at 0°. The solution was stirred for 0.5 hr at this temperature and then allowed to stand in the freezer for 6 hr. The mixture was decomposed with five drops of water at 0° and extracted with ether. The ether layer was washed with cold dilute HCl and saturated $NaHCO_3$, and then saturated NaCl. After drying over K_2CO_3, evaporation of the solvent gave a yellow solid, which was recrystallized from ether–pentane to give 190 mg of pale yellow leaflets, mp 138.5–139.5°, 91% yield.

Anal. Calcd for $C_{16}H_{13}NO_4$: C, 67.84; H, 4.63. Found: C, 67.69; H, 4.67.

Tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-one (3).—To a solution of 50 mg of **2a** in 10 ml of ether was added 0.5 g of CrO_3 in 6 ml of water. The mixture was stirred at room temperature for 3.5 hr. After usual work-up, the ketone **3** was collected by preparative tlc (Silica AR) with 25% ether–pentane (detected with uv lamp), ir (CCl_4) 1715 cm^{-1}, R_f 0.81.

Anal. Calcd for C_9H_8O: C, 81.79; H, 6.10. Found: C, 81.85; H, 6.01.

endo-**Tricyclo[6.1.0.0^{4,9}]nona-2,5-dien-7-ol (4).**—A solution of 28 mg of **3** in 2 ml of methanol was added to stirred $NaBH_4$ (80 mg) in 2 ml of methanol at room temperature. After the mixture had been stirred for 2 hr, the excess hydride was destroyed with 1 ml of 20% KOH solution. The aqueous layer was extracted with ether and the ether solution was washed with water and dried over K_2CO_3. Evaporation of the ether yielded a residue which was purified by preparative tlc (Silica AR) with 25% ether–pentane (detected with uv lamp): mp 111–112°; ir (CCl_4) 3550 cm^{-1} (OH); nmr ($CDCl_3$) τ 4.0–5.0 (4 H, multiplet, olefinic), 5.5 (1 H, narrow multiplet, α H), 6.70 (1 H, narrow multiplet, bisallylic), and 8.0–9.0 (3 H, multiplet, cyclopropyl); mass spectrum m/e 134 ($C_9H_{10}O^+$); R_f 0.54.

Anal. Calcd for $C_9H_{10}O$: C, 80.56; H, 7.51. Found: C, 80.41; H, 7.52.

Registry No.—**1**, 30767-78-3; **2a**, 34886-41-4; **2b**, 34886-42-5; **3**, 34886-43-6; **4**, 34886-44-7.

Acknowledgment.—The authors gratefully acknowledge the help of Professor C. S. Foote, of the University of California at Los Angeles, with whom many fruitful discussions of this work were held.

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Twofold Redox Addition of Carbon Tetrachloride to Olefins

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Received December 20, 1971

The addition of haloalkanes to olefins, first described by Kharasch, has been known for a long time.[1] One of the drawbacks has been that with less reactive carbon halogen compounds, telomerization is observed rather than simple 1:1 addition. This difficulty has been overcome by a technique utilizing copper or iron salts as catalysts.[2–4] Even under these conditions some by-products of telomeric 2:1 structures have been reported.

We now show that under proper conditions products of "twofold" addition (not telomeric) can be obtained.

$$CCl_4 + 2CH_2{=}CHR \longrightarrow \underset{\text{twofold addition}}{RCHClCH_2CCl_2CH_2CHClR}$$
$$\searrow$$
$$\underset{\text{2:1 telomer}}{CCl_3CH_2CHRCH_2CHClR}$$

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