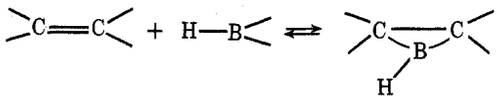
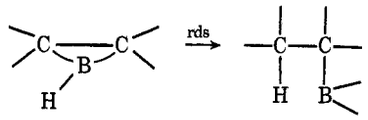
on the least hindered side of the olefin. The direction of addition depends on two factors: the orientation of the remaining hydrogens on boron in the complex, and the electronic effect of other substituents on the olefin. With bulky groups on the olefin or boron one would expect the hydrogen on boron to be juxtaposed with the internal lobe of the Cn orbital of the π complex. The conversion of this intermediate to products would result in boron substitution on the least hindered carbon of the olefin. With strongly electron-withdrawing substituents on the olefin, the collapse of the complex to product should be influenced by the tendency of the electron pair in the preformed three-center bond to move toward the more electron-deficient carbon, giving boron substitution α to the electronegative group when the steric factors of the reaction permit.

In summary, we consider the hydroboration reaction as a two-step process, the first step an equilibrium resulting in the production of three-center, two-electron π complex intermediate (eq 1); the second step a rate-determining *concerted* conversion of the intermediate to products (eq 2). This mechanism satisfies the requirements of the hydroboration reaction while not involving the buildup of any significant hydridic character on the boron hydrogens. More important, the fact that olefin π complexes can convert to products by a concerted, symmetry-allowed process not involving significant charge separation in the transition state should be useful in the consideration of other reactions which involve such intermediates.



(1)



(2)

Acknowledgment.—The author expresses his thanks to the donors of the Petroleum Research Fund administered by the American Chemical Society and to the North Texas State University Faculty Research Fund for financial support of this work.

A New Synthesis of Coenzyme Q_1

KIKUMASA SATO,* SEIICHI INOUE, AND RYOHEI YAMAGUCHI

Department of Applied Chemistry, Faculty of Engineering, Yokohama National University, Minami-ku, Yokohama, Japan

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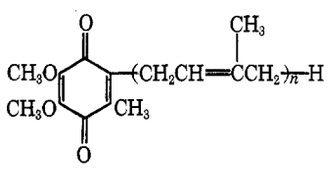
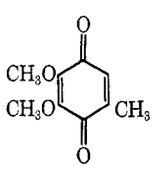
A new synthesis of coenzyme Q_1 is reported. 2,3-Dimethoxy-5-methylbenzoquinone (**2**) is converted to 6-bromo-2,3-dimethoxy-5-methylhydroquinone bis(methoxymethyl) ether (**18**), which is condensed with 1,1-dimethyl-π-allylnickel bromide (**9**) in hexamethylphosphoramide to afford 2,3-dimethoxy-5-methyl-6-(3-methyl-2-butenyl)hydroquinone bis(methoxymethyl) ether (**19**) in good yield. The hydrolysis of the condensation product **19** followed by oxidation gives coenzyme Q_1. The reaction of **9** with several other aryl halides is also reported.

Coenzyme Q_n (**1**), ubiquinone $5n$, functions in electron transfer and oxidative phosphorylation. The ten known ubiquinones, coenzyme Q_1–Q_{10}, are named according to the number of isoprene units in the side chain. Coenzymes Q_6–Q_{10} were isolated by Lester[1,2] and their structures were determined as **1**[3–5] (n = 6–10). These compounds were synthesized by Folkers, *et al.*,[3] and also by Isler and coworkers.[4,5]

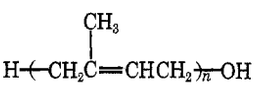


1, n = 1–10

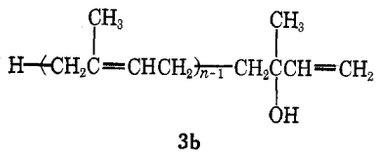


2

In the synthesis of coenzyme Q_n there are three key steps, which include (i) a synthesis of 2,3-dimethoxy-5-methylbenzoquinone (**2**), (ii) a stereospecific synthesis of the polyprenyl alcohols **3a** or **3b**, (iii) a condensation of the aromatic nucleus **2** with the alcohols **3a** or **3b**.



3a



3b

All of the coenzyme Q_n synthesis reported involved the condensation of 2,3-dimethoxy-5-methylhydroquinone (**4**) with **3a** or **3b** using acid catalysts. Such condensation reactions suffer from the disadvantage that cyclization to chromanol and a cyclization of the unsaturated isoprenoid side chain often results. In order to minimize these side reactions, many kinds of catalysts ($SnCl_2$, K_2SO_4, oxalic acid, BF_3, etc.) have been used.[6] Unfortunately, these methods generally give low yields and, in addition, much labor is needed to isolate the condensation product from the complex reaction mixture. Reported here is a new method for the

(1) F. L. Crane, Y. Hatefi, R. L. Lester, and C. Widmer, *Biochim. Biophys. Acta*, **25**, 220 (1957).

(2) R. L. Lester, F. L. Crane, and Y. Hatefi, *J. Amer. Chem. Soc.*, **80**, 4751 (1958).

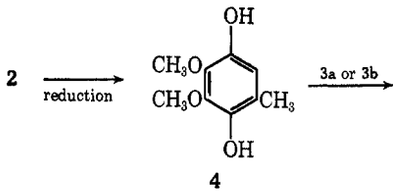
(3) D. E. Wolf, C. H. Hoffman, N. R. Trenner, B. H. Arison, C. H. Shunk, B. O. Linn, J. F. McPherson, and K. Folkers, *ibid.*, **80**, 4752 (1958).

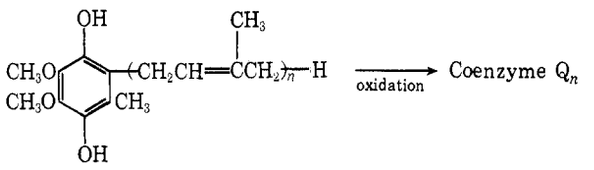
(4) R. A. Morton, U. Gloor, O. Schindler, W. M. Wilson, L. H. Chopard-dit-Jean, F. W. Hemming, O. Isler, W. M. F. Leat, J. F. Pennock, R. Rüegg, U. Schwieter, and O. Wiss, *Helv. Chim. Acta*, **41**, 2343 (1958).

(5) U. Gloor, O. Isler, R. A. Morton, R. Rüegg, and O. Wiss, *ibid.*, **41**, 2357 (1958).

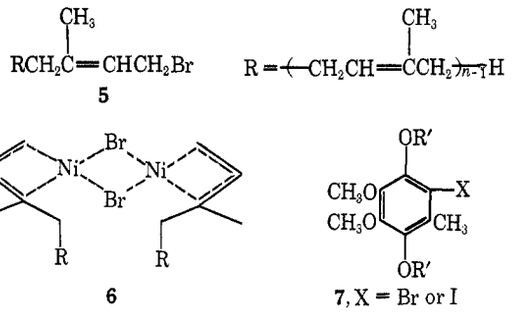
(6) Merck Co., British Patent 921,538, 928,161 (1963); *Chem. Abstr.*, **59**, 6316, 14032 (1963).

synthesis of coenzyme Q which does not suffer from the above drawbacks.



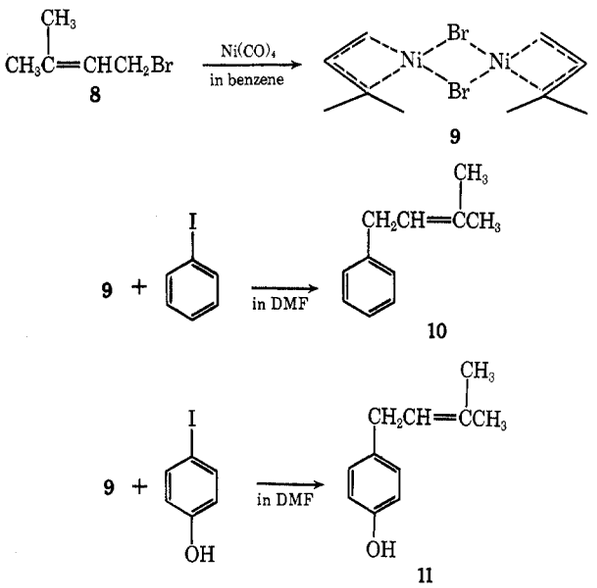


Recently a number of organometallic complexes have been used for syntheses which proceed under mild conditions and in good yields. Corey and Semmelhack reported that π-allylic nickel complexes react with aliphatic or aromatic halides under a mild condition to afford the corresponding allylic derivatives in good yields and in high selectivity.[7] We, therefore, thought that the reaction of the π-allylic nickel complexes **6** prepared from polyprenyl bromide (**5**) with the halogenated derivative **7** of 2,3-dimethoxy-5-methylbenzoquinone (**2**) would give coenzyme Q_1. Indeed, such a transformation did take place.

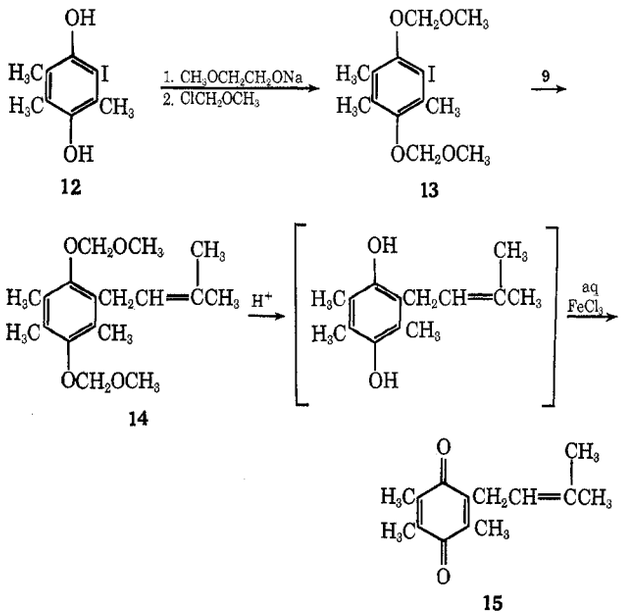


The reaction of 1-bromo-3-methyl-2-butene (**8**) with nickel carbonyl in benzene at 50° under a stream of nitrogen gave 1,1-dimethyl-π-allylnickel bromide (**9**), which corresponds to the side chain of coenzyme Q_1. After removal of benzene under vacuum this compound was reacted with iodobenzene at 20° in dimethylformamide (DMF) to give (3-methyl-2-butenyl)benzene (**10**) in good yield, showing high reactivity and high selectivity to the aromatic halide. Since there has been no report on the reaction of π-allylic nickel complexes with aromatic halides having a hydroxyl substituent,[7] the influence of a phenolic hydroxyl substituent was investigated using *p*-iodophenol. *p*-Iodophenol reacted with 1,1-dimethyl-π-allylnickel bromide (**9**) in DMF at 20° to give *p*-(3-methyl-2-butenyl)phenol (**11**) in 38% yield. This result shows that a hydroxyl group does not prevent the condensation reaction, although the yield is lower.

Reaction of a π-allylic nickel complex with halogenated hydroquinones was attempted next. Treatment of 1,1-dimethyl-π-allylnickel bromide (**9**) with iodotrimethylhydroquinone (**12**) gave trimethylhydroquinone in 25% yield, while reaction with iodotrimethylbenzoquinone gave **12** in 60% yield. It is concluded from the above results that the oxidizable and reducible functional groups must be protected.



Since it has been reported that the methoxymethyl group is easily removed under very mild conditions,[8] this was thought to be a suitable protecting group for hydroquinone derivatives which are very sensitive to an acid and base. Iodotrimethylhydroquinone bis(methoxymethyl) ether (**13**) was prepared from **12** and chloromethyl methyl ether. The reaction of **13** with **9** in DMF at 50° for 10 hr gave the condensation product **14** in good yield. The structure of **14** was assigned on the basis of the nmr spectrum (see Experimental Section). Removal of the methoxymethyl groups in **14** by methanolic HCl, followed by oxidation with aqueous ferric chloride, afforded (3-methyl-2-butenyl)trimethylbenzoquinone (**15**).

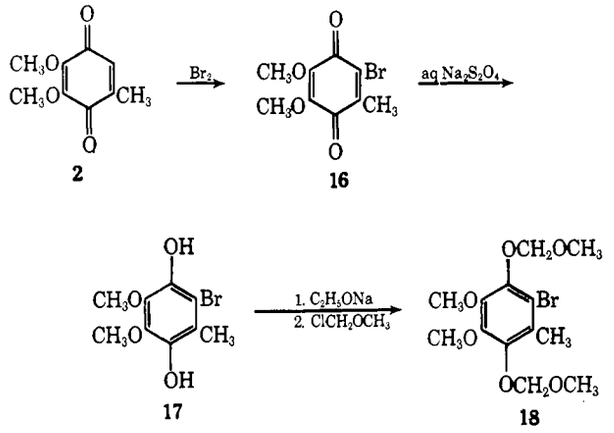


The above result suggested a new synthesis of coenzyme Q_1 by application of the same reaction sequence to the halogenated derivative of 2,3-dimethoxy-5-methylbenzoquinone (**2**). Bromination of **2** gave

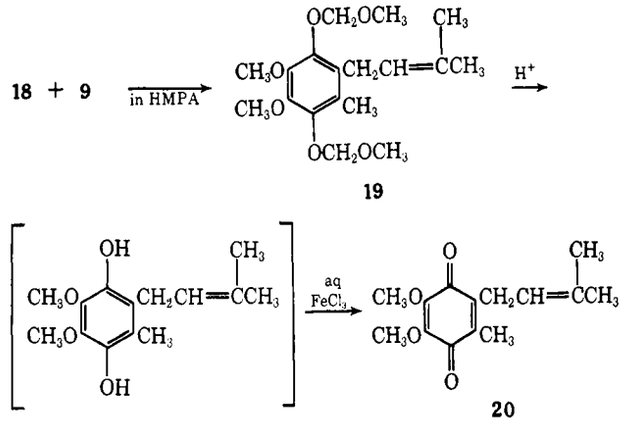
(7) E. J. Corey and M. F. Semmelhack, *J. Amer. Chem. Soc.*, **89**, 2755 (1967).

(8) R. Stern, J. English, Jr., and H. G. Cassidy, *ibid.*, **79**, 5792 (1957).

6-bromo-2,3-dimethoxy-5-methylbenzoquinone (**16**), which was reduced and methoxymethylated to give 6-bromo-2,3-dimethoxy-5-methylhydroquinone bis-(methoxymethyl) ether (**18**).



The reaction of **18** with **9** in DMF at 50° did not proceed. Even at 75° only a trace of the condensation product **19** was detected. Since it has been thought that a coordinating solvent, *e.g.*, DMF, coordinates with π-allylic nickel complexes and activates them,[7] hexamethylphosphoramide (HMPA) was substituted as solvent because it coordinates with metal complexes more strongly than DMF.[9] When **18** was treated with **9** in HMPA at 60°, **19** was obtained in 57% yield. Hydrolysis and oxidation of **19** was followed by purification by column chromatography to give 2,3-dimethoxy-5-methyl-6-(3-methyl-2-butenyl)benzoquinone, coenzyme Q_1 (**20**).



This new synthesis of coenzyme Q_1 should provide a general method for synthesizing higher homologs. Further work in this area is in progress.

Experimental Section

General.—Boiling points and melting points are uncorrected. Infrared (ir) spectra were recorded on a Hitachi Model 215 spectrophotometer. Ultraviolet (uv) spectra were recorded on a Hitachi Model EPS-3T spectrophotometer. Nuclear magnetic resonance (nmr) spectra were obtained on JEOL Model C-60 spectrometer. Reactions involving π-allylnickel complexes were carried out under a stream of nitrogen.

(3-Methyl-2-butenyl)benzene (10).—To a stirred solution of 10.3 g (0.06 mol) of nickel carbonyl in 58 g of dry benzene was added dropwise 6.0 g (0.04 mol) of 1-bromo-3-methyl-2-butene[10] (**8**) in 36 g of dry benzene at 50° for 1.5 hr under a stream of nitrogen. The reaction mixture was allowed to stand at 50° for 2 hr and cooled. Benzene was removed under reduced pressure and 42 ml of DMF was added to the dark red residue. To this solution was added 6.4 g (0.03 mol) of iodobenzene[11] in 20 ml of DMF at 20° for 1.5 hr. The reaction mixture was stirred for 3 hr and, after treatment with water containing a small quantity of hydrochloric acid, extracted with petroleum ether (bp 30–50°). The extract was washed with water, dried with magnesium sulfate, and freed of solvent. The residual liquid was distilled to give 3.3 g (75%) of **19**: bp 74–77° (8 mm); n^{20}D 1.5165 [lit.[12] bp 81.2–81.6° (11 mm); n^{20}D 1.5136]; ir (neat) 2925, 1670, 1600, 1450, 735, 695 cm^{-1}; nmr (CCl_4) δ 1.71 (s, 6, 2 CH_3), 3.29 (d, 2, J = 7 Hz, CH_2), 5.29 (t, 1, J = 7.5 Hz, =CH), 7.10 (s, 5, C_6H_5).

***p*-(3-Methyl-2-butenyl)phenol (11).**—1,1-Dimethyl-π-allylnickel bromide (**9**) was prepared from 6.0 g (0.04 mol) of **8** and 10.3 g (0.06 mol) of nickel carbonyl by the same method described above. To the solution of **9** in 40 ml of DMF was added dropwise 5.0 g (0.023 mol) of *p*-iodophenol[13] in 15 ml of DMF at 20°. The reaction mixture was stirred for 3 hr and, after treatment with water containing a small quantity of hydrochloric acid, extracted with petroleum ether. The extract was washed with water, dried with magnesium sulfate, and freed of solvent, and the residual oil was chromatographed on silica gel. Elution with 50% benzene in petroleum ether gave 1.4 g (38%) of **11**: n^{20}D 1.5429 (lit.[14] n^{20}D 1.5400); ir (neat) 3320, 2920, 1614, 1514, 1234, 818 cm^{-1}; nmr (CCl_4) δ 1.67 (s, 6, 2 CH_3), 3.17 (d, 2, J = 7.5 Hz, CH_2), 5.21 (t, 1, J = 7.5 Hz, =CH), 5.52 (s, 1, OH), 6.78 (q, 4, C_6H_4).

Iodotrimethylhydroquinone Bis(methoxymethyl) Ether (13).—To a stirred solution of 11.2 g (0.04 mol) of iodotrimethylhydroquinone (**12**)[15] in 80 ml of ethylene glycol monomethyl ether were added dropwise a quater portion of the solution prepared by dissolving 3.7 g (0.16 mol) of sodium in 48 ml of ethylene glycol monomethyl ether and then 3.2 g (0.04 mol) of chloromethyl methyl ether, with the temperature of the reaction mixture being maintained at −10 to 0° under dry nitrogen. The remaining alcoholate solution was dropped in three equal portions, each addition being followed by dropwise addition of 3.2-g portions of chloromethyl methyl ether. After all addition were complete, the reaction mixture was stirred for 1 hr at −10 to 0° and treated with water. Collection of the precipitates by suction and recrystallization from petroleum ether gave 12.1 g (82.9%) of **13**: mp 85–86°; ir (KBr) 2900, 2830, 1460, 1380, 1165, 1065, 970, 930 cm^{-1}; nmr ($CDCl_3$) δ 2.18, 2.26, and 2.42 (3 s, 9, 3CH_3), 3.61 and 3.69 (2 s, 6, 2 OCH_3), 4.89 and 4.99 (2 s, 4, 2 OCH_2O).

Anal. Calcd for $C_{13}H_{19}O_4I$: C, 42.64; H, 5.23. Found: C, 42.44; H, 5.18.

(3-Methyl-2-butenyl)trimethylbenzoquinone (15).—**9** was prepared from 6.4 g (0.04 mol) of **8** and 10.3 g (0.06 mol) of nickel carbonyl by the same method described above. To the solution of **9** in 40 ml of DMF was added dropwise 5.5 g (0.015 mol) of **13** in 45 ml of DMF at 50° over 1 hr. The reaction mixture was stirred at 50° for 9 hr and, after treatment with water containing a small quantity of ammonia and ammonium chloride and filtration, extracted with chloroform. The extract was washed with water, dried with magnesium sulfate, and freed of solvent. The residual liquid (4.2 g) consisted of **13** (10%) and **14** (90%) by nmr assay,[16] but **14** could not be isolated by column chromatography on silica gel. A portion of the residue (2.0 g) was dissolved in 35 ml of methanol containing a drop of hydrochloric acid. The solution was refluxed for 1 hr, cooled, neutralized with alcoholic potassium hydroxide, and freed of solvent. The residue (1.3 g) was dissolved in 30 ml of ether, oxidized with aqueous ferric chloride, and extracted with ether. The extract was washed with water, dried with magnesium sulfate, and freed of solvent to give a reddish oil, which was chromatographed on silica gel. Elution with 5% isopropyl

(9) H. Normant, *Angew. Chem., Int. Ed. Engl.*, **6**, 1046 (1967).

(10) J. Tanaka, T. Katagiri, and S. Yamada, *Nippon Kagaku Zasshi*, **87**, 877 (1966).

(11) H. J. Lucas and E. R. Kennedt, "Organic Syntheses," Collect. Vol. II, Wiley, New York, N. Y., 1943, p 351.

(12) L. Bateman and J. I. Cunneen, *J Chem. Soc.*, 2283 (1956).

(13) F. B. Dains and F. Eberly, "Organic Syntheses," Collect. Vol. II, Wiley, New York, N. Y., 1943, p 355.

(14) E. A. Vdovtsova, *Zh. Org. Khim.*, **1** (12), 2192 (1965).

(15) H. W. J. Cressman and J. R. Thirtle, *J. Org. Chem.*, **31**, 1279 (1966).

(16) As a result of comparison with the nmr spectrum of **13**, chemical shifts of protons in **14** are as follows: δ ($CDCl_3$) 1.72 and 1.80 [2 s, =C-$(CH_3)_2$], 2.21 (s, 3 CH_3), 3.43 (d, CH_2), 3.63 (s, 2 OCH_3), 4.92 (s, 2 OCH_2O), 5.10 (m, =CH).

ether in *n*-hexane afforded 1.1 g of **15**. The estimated yield of **15** from **13** was 67%: ir (neat) 2970, 2930, 1640, 1440, 1375, 1300, 1260, 840, 710 cm^{-1}; uv max (95% EtOH) 260 mμ (ε 18,900) and 267 (19,100) [lit.[17] uv max (95% EtOH) 259 mμ (ε 17,200) and 266 (17,200)]; nmr (CCl_4) δ 1.65 and 1.71 [2 s, 6, $=C(CH_3)_2$], 1.93 (s, 9, 3 CH_3), 3.10 (d, 2, J = 7.5 Hz, CH_2), 4.89 (t, 1, J = 7.5 Hz, CH=).

Anal. Calcd for $C_{14}H_{18}O$: C, 77.03; H, 8.31. Found: C, 76.83; H, 8.51.

6-Bromo-2,3-dimethoxy-5-methylbenzoquinone (16).—To a stirred solution of 10.6 g (0.058 mol) of **2**[18] in 120 ml of carbon tetrachloride was added dropwise 10.5 g (0.068 mol) of bromine at room temperature. The reaction mixture was stirred for 2 hr, treated with water, dried with magnesium sulfate, and evaporated. The dark residue was washed with a very small quantity of ethanol until the color of crystals turned to red and recrystallized from petroleum ether to afford 11.2 g (74%) of **16**: mp 73–74°; ir (KBr) 2850, 1650, 1600, 1280 cm^{-1}.

Anal. Calcd for $C_9H_9O_4Br$: C, 41.41; H, 3.47. Found: C, 41.26; H, 3.62.

6-Bromo-2,3-dimethoxy-5-methylhydroquinone (17).—The quinone **16** (5.0 g) was dissolved in warm methanol and to this solution was added warm aqueous sodium hydrosulfite until the red color of the solution disappeared. Removal of methanol under reduced pressure in a stream of nitrogen afforded 4.3 g (83%) of **17**: mp 73–74°; ir (KBr) 3300, 2880, 1450, 1420, 1280, 1105, 1070, 1000, 910 cm^{-1}; nmr (CCl_4) δ 2.21 (s, 3, CH_3), 3.84 and 3.88 (2 s, 6, 2 OCH_3), 5.14 and 5.27 (2 s, 2, 2 OH).

Anal. Calcd for $C_9H_{11}O_4Br$: C, 41.09; H, 4.21. Found: C, 40.81; H, 4.43.

6-Bromo-2,3-dimethoxy-5-methylhydroquinone Bis(methoxymethyl) Ether (18).—To a stirred solution of 6.0 g (0.023 mol) of **17** in 150 ml of absolute ethanol was added dropwise 0.6 g (0.025 mol) of sodium in 13 ml of absolute ethanol and then 2.0 g (0.025 mol) of chloromethyl methyl ether at −10 to 0° under a stream of nitrogen, and then 1.8 g of sodium in 39 ml of absolute ethanol and 6.0 g of chloromethyl methyl ether were added by the same method described in the preparation of **13**. The reaction mixture was stirred for 3 hr at −10 to 0°, filtered, and concentrated under reduced pressure in a stream of nitrogen. The concentrated solution was washed with dilute aqueous potassium hydroxide and water and extracted with ether. The extract was dried with magnesium sulfate, freed of solvent, and chromatographed on silica gel. Elution with 30% isopropyl ether in *n*-hexane afforded 6.7 g (83.2%) of **18**: $n^{20}D$ 1.5282; ir (neat) 2800, 1460, 1405, 1380, 1160, 1000, 965 cm^{-1}; nmr (CCl_4) δ 2.29 (s, 3, CH_3), 3.48 and 3.56 (2 s, 6, 2 OCH_2OCH_3), 3.79 (s, 6, 2 OCH_3), 4.93 and 4.98 (2 s, 4, 2 OCH_2O).

Anal. Calcd for $C_{13}H_{19}O_6Br$: C, 44.33; H, 5.72. Found: C, 44.52; H, 5.53.

2,3-Dimethoxy-5-methyl-6-(3-methyl-2-butenyl)benzoquinone (Coenzyme Q_1) (20).—**9** was prepared from 4.5 g (0.03 mol) of **8** and 8.7 g (0.05 mol) of nickel carbonyl by the same method described above. To the solution of **9** in 36 ml of HMPA was added 5.4 g (0.015 mol) of **18** in 20 ml of HMPA at room temperature. The reaction mixture was warmed to 60° and stirred for 12 hr. The solution was treated with water containing a small quantity of ammonia and ammonium chloride, filtered, and extracted with petroleum ether. The extract was washed with water, dried with magnesium sulfate, and freed of solvent, affording a residual liquid (4.9 g) which consisted of **18** (43%) and **19** (57%) by nmr assay.[19] This liquid was chromatographed, but **19** could not be isolated. A 3-g portion of the liquid was dissolved in 50 ml of methanol containing a drop of hydrochloric acid. The solution was refluxed for 1 hr, cooled, neutralized with alcoholic potassium hydroxide, and freed of solvent. The residue was dissolved in 15 ml of ether, oxidized with aqueous ferric chloride, and extracted with ether. The extract was washed with water, dried with magnesium sulfate, and freed of solvent to afford a reddish oil, which was chromatographed on silica gel. Elution with 20% isopropyl ether in *n*-hexane afforded 0.89 g of **20**. The estimated yield of **20** from **18** was 40%: ir (neat) 2950, 1650, 1460, 1270, 1100, 1020 cm^{-1}; uv max (*n*-hexane) 270 mμ (ε 15,100); nmr (CCl_4) δ 1.65 and 1.73 [2 s, 6, $=C(CH_3)_2$], 1.94 (s, 3, CH_3), 3.08 (d, 2, J = 7.5 Hz, CH_2), 3.89 (s, 6, 2 OCH_3), 4.82 (t, 1, J = 7.5 Hz, CH=).

Anal. Calcd for $C_{14}H_{18}O_4$: C, 67.18; H, 7.25. Found: C, 67.28; H, 7.39.

Registry No.—**10**, 4489-84-3; **11**, 1200-09-5; **13**, 34417-76-0; **15**, 2134-78-3; **16**, 30685-17-7; **17**, 34417-79-3; **18**, 34407-31-3; **20**, 727-81-1.

(17) P. Mamont, P. Cohen, and R. Azerad, *Bull. Soc. Chim. Fr.*, 1485 (1967).

(18) W. K. Anslow, J. N. Ashley, and H. Raistrick, *J. Chem. Soc.*, 439 (1938).

(19) As a result of comparison with the nmr spectrum of **18**, chemical shifts of protons in **19** are as follows: δ (CCl_4) 1.67 and 1.73 [2 s, $=C(CH_3)_2$], 2.09 (s, CH_3), 3.28 (d, CH_2), 3.47 (s, 2 OCH_2OCH_3), 3.67 (s, 2 OCH_3), 4.90 (s, 2 OCH_2O), 5.09 (m, =CH).

Synthesis of Steroidal Aziridines

R. IKAN,* A. MARKUS, AND Z. GOLDSCHMIDT

Department of Organic Chemistry, Hebrew University, Jerusalem, Israel

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Desmostanyl 3α-acetate (**5**) was synthesized from litocholic acid and converted into the aziridine **8**, the essential steps being addition of iodine isocyanate to **5** which was converted to the corresponding carbamate **7**. Treatment of **7** with alcoholic base formed the aziridine **8**. Analogous sequence of reactions led to the formation of aziridine **9** from stigmasteryl acetate.

It has been reported recently that the aziridine functional grouping shows favorable carcinostatic activity in a number of tumor systems.[1] Most of the compounds reported to date have had the nitrogen mustard group attached to certain positions on the nucleus of the steroid. In connection with our work on the utilization of natural sterols and their derivatives by insects,[2] it was of interest to synthesize some steroidal aziridines having the nitrogen function in the side chain of the steroid.[3]

In the present communication we report the synthesis of 5,6-dihydro-24,25-iminodesmostanyl acetate (**8**) and 22,23-iminostigmasteryl acetate (**9**). Desmostanyl 3α-acetate (**5**) was readily obtained by the photochemical Wolff rearrangement in a THF–methanol solution of diazo ketone **2**[4] to give the methyl ester **3**. Grignard reaction of **3** with methylmagnesium iodide and subsequent dehydration[5] yielded **5**. Addition of iodine isocyanate[6] to **5** gave the adduct **6**,[7]

(1) S. A. Dyogtera, *Angew. Chem., Int. Ed. Engl.*, **1**, 600 (1962).

(2) R. Ikan, A. Markus, P. Klein, Z. Levinson, and E. D. Bergmann, *J. Insect Physiol.*, submitted for publication.

(3) Preliminary tests have indicated that the new aziridines caused total mortality of the larvae of *Dermestes maculatus*.

(4) A. S. Kende and Z. Goldschmidt, *Org. Photochem. Syn.*, **1**, 92 (1971).

(5) G. Habermehl and G. Volkwein, *ibid.*, **742**, 145 (1970).

(6) A. Hassner, M. E. Lorber, and C. Heathcock, *J. Org. Chem.*, **32**, 540 (1967).

(7) Structure **6** was tentatively assigned on the basis of the previous studies of INCO addition to olefins.[6]