accomplished in this way. The mole percentages of these materials were determined from the corrected areas of the peaks. The *meta* isomer could not be resolved despite the use of various conditions and column packings. Analysis for the *meta* isomer was carried out by a previously reported[2] standard base-line technique on a Beckman IR-9 infrared spectrophotometer at 12.90 μ. The mean of several trials was subtracted from the *meta* + *para* value obtained by vpc to give the isomer distributions. A summary of several runs is given in Table V.

TABLE V

PRODUCT ISOMER DISTRIBUTIONS IN THE CHLORINATION OF ETHYLBENZENE IN ACETIC ACID

Run	*ortho*	*meta*	*para*
1	52.1	0.53	47.4
2	53.5	0.48	46.0
3	51.2	0.58	48.3
Mean	52.3	0.53	47.2

A similar procedure was used on the products from the chlorination of toluene in acetic acid at 25°. The results, shown in Table II, are in agreement with reported values.[5] The infrared analysis for *m*-chlorotoluene was made at 12.96 μ.

Registry No.—I, 100-41-4; acetic acid, 64-19-7; *o*-chloroethylbenzene, 89-96-3; *m*-chloroethylbenzene, 620-16-6; *p*-chloroethylbenzene, 622-98-0.

Regeneration of Carbonyl Compounds from Oximes Using Iron Pentacarbonyl and Boron Trifluoride

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The parent carbonyl compound may be regenerated from an oxime by treatment of the latter in aqueous solution with sodium bisulphite,[1] levulinic or pyruvic acids,[2] or formaldehyde and concentrated hydrochloric acid.[3] However, there may be instances when it is desirable to effect this transformation under anhydrous conditions. We have found that treatment of an oxime with an equimolar quantity of iron pentacarbonyl and a catalytic amount of boron trifluoride in refluxing butyl ether results in the formation of the carbonyl compound in 55–81% yield. The examples listed in Table I indicate that the reaction is applicable to the oximes of aldehydes and ketones of widely differing character; the limitations, if any, of the reaction are not yet apparent.

The mechanism of the reaction, and hence the function of the boron trifluoride, are not yet known. Reactive of santonin oxime ($C_{15}H_{19}NO_3$) with iron pentacarbonyl alone gave a complex $C_{15}H_{19}NO_3 \cdot Fe(CO)_3$;[4] the other oximes failed to react. No reaction took place when the oximes were treated with a catalytic amount of boron trifluoride in butyl ether. No carbonyl compound was regenerated by treatment of fluorenone phenylhydrazone or of N-2,6-trichloro-*p*-benzoquinoneimine with iron pentacarbonyl and boron trifluoride. Hence it seems likely that the oxygen atom of the regenerated carbonyl group comes from the oxygen atom of the oxime.

(1) S. H. Pines, J. M. Chemerda, and M. A. Kozlowski, *J. Org. Chem.*, **31**, 3446 (1966).
(2) E. B. Hershberg, *ibid.*, **13**, 542 (1948).
(3) W. H. Perkin, W. M. Roberts, and R. Robinson, *J. Chem. Soc.*, **101**, 232 (1912).
(4) H. Alper and J. T. Edward, unpublished results.

TABLE I

YIELDS OF CARBONYL COMPOUNDS FROM OXIMES

Parent carbonyl compd	Reflux time, hr	Isolation procedure	Yield, %
Cyclohexanone	20	...	81
4-Methyl-4-trichloromethyl-cyclohexadien-1-one	10	b	72
Fluorenone	16	a	69
Cholest-4-en-3-one[a]	16	d	67
Santonin	18	c	67
O-Methylpodocarpinal	17	c	55

[a] Oxime mp 152–152.5, prepared according to C. W. Shoppee, G. Kreiger, and R. N. Mirrington, *J. Chem. Soc.*, 1050 (1962).

Experimental Section

A mixture of the oxime (2–35 mmoles) and iron pentacarbonyl (1.1 mole/mole of oxime) in dry butyl ether (50–100 ml) containing boron trifluoride etherate (about 5% w/w of oxime) was refluxed with stirring under nitrogen. The solution was cooled and filtered and the solvent removed at 30 mm (except when the volatile cyclohexanone was formed; this was isolated as the 2,4-dinitrophenylhydrazone by treating the filtrate with 2,4-dinitrophenylhydrazine in the usual manner). The residue of crude carbonyl compound was purified by trituration with petroleum ether (bp 30–60°), hexane, or methylene chloride (procedures a, b, or c, respectively) or by chromatography on Florisil using acetone as eluent (procedure d). The purity of the products recorded in Table I was indicated by melting points agreeing with values in the literature and by the absence of more than one spot on thin layer chromatograms.

Registry No.—Boron trifluoride, 7637-07-2.

Acknowledgments.—We thank Mr. M. J. Davis and Mr. W. Zehetner for the gift of oximes and the National Research Council for financial support.

The Preparation of Esters of 4-Alkyl-2,4-pentadienoic Acids by the Phosphonate Modification of the Wittig Reaction

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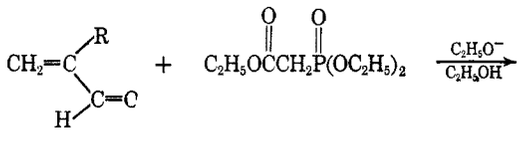
This note describes conditions for the rapid and convenient preparation of the ethyl esters of several 4-alkyl-2,4-pentadienoic acids from 2-alkylacroleins (**1**) and triethyl phosphonoacetate (**2**). Subsequent saponification of the esters gives the corresponding carboxylic acids and this route has been found to be useful for the preparation of several 4-alkyl-2,4-pentadienoic acids (**5**).

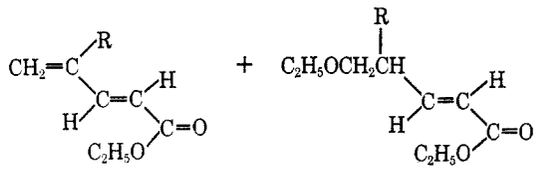
TABLE I
PRODUCT DATA

Compd	R	Yield, %	Bp (mm), °C	Calcd, % C	Calcd, % H	Found, % C	Found, % H
3a	H	4	52–55 (15)[a]	...	...	...	...
3b	CH_3	52	81–83 (17)	68.5	8.6	68.5	8.7
3c	CH_3CH_2	66	50 (0.7)[c]	70.1	9.2	70.2	9.3
3d	$CH_3(CH_2)_4$	57	72–74 (0.1)	73.4	10.3	73.4	10.3
4a	H	7	102 (15)[b]	62.8	9.4	62.5	9.4
4b	CH_3	4	120–122 (17)	64.5	9.7	64.2	9.9
4c	CH_3CH_2	3	84 (0.9)	66.0	10.0	66.0	10.1
4d	$CH_3(CH_2)_4$	0	...	...	...	...	...

[a] Lit.[2] bp 53–55° (19 mm). [b] Lit.[12] bp 73–76° (7 mm). [c] Lit.[13] bp 73–77° (11 mm).



1 **2**



3 **4**

Derivatives of vinylacrylic acid have a number of synthetic uses. An interesting recent example is their conversion to carbocyclic rings on reaction with enamines.[1,2] The preparation of vinylacrylic acid and its alkyl derivatives often involves amine-catalyzed decarboxylative condensation of an α,β-unsaturated aldehyde with malonic acid. The preparation of sorbic acid[3] and vinylacrylic acid[4] are familiar examples. Attempts to use similar procedures to prepare 4-ethyl-2,4-pentadienoic acid gave only low yields of the crude acid contaminated with impurities which were difficult to remove. The use of the phosphonate modification of the Wittig reaction for the conversion of α,β-unsaturated aldehydes to ethyl pentadienoates was therefore investigated.

There are a number of reports of the reaction of the triethyl phosphonoacetate carbanion with unsaturated carbonyl compounds.[5–9] The normal Wittig reaction using alkylidenephosphoranes has, of course, been applied to a number of unsaturated carbonyl compounds[10] including acrolein.[11] The possibility that conjugate addition of the phosphonate carbanion to the unsubstituted β-carbon atom of the 2-alkylacroleins might seriously compete with reaction at the carbonyl group caused some concern. The only application of the phosphonate olefin synthesis to an acrolein lacking a β substituent of which we are aware is the reaction of acrolein itself, which gives only a 14% yield of ethyl 2,4-pentadienoate.[9]

The 2-alkylacroleins examined were converted to 4-alkyl-2,4-pentadienoates in 50–65% yield. The product yields and physical and nmr spectral properties are shown in Tables I[12,13] and II. In the case of **1b** and **1c**, low yields of ethyl 5-ethoxy-4-methyl-2-pentenoate (**4b**) and ethyl 5-ethoxy-4-ethyl-2-pentenoate (**4c**) accompanied the major product. The structures of these by-products follow from the elemental analyses and from the nmr spectral data in Table II. Acrolein itself was not converted to ethyl vinylacrylate (**3a**) in satisfactory yield. Under the standard conditions (see the Experimental Section), **3a** was isolated in only 4% yield, accompanied by a 7.5% yield of ethyl 5-ethoxy-2-pentenoate (**4a**). At Dry Ice–acetone temperature, only **4a** was formed.

TABLE II
NMR SPECTRA OF COMPOUNDS **3**, **4**, AND **5**

Compd	Peaks[a] δ (multiplicity)
3b	1.3 (t), 1.9 (d, J = 1 cps), 4.2 (q), 5.4 (s), 5.8 (d, J = 16 cps), 7.3 (d, J = 16 cps)
3c	1.1 (t), 1.3 (t), 2.3 (q), 4.1 (q), 5.3 (s), 5.8 (d, J = 16 cps), 7.2 (d, J = 16 cps)
3d	0.9 (t),[b] 1.3 (t), 0.9–1.8 (m), 2.2 (m), 4.2 (q), 5.25 (s), 5.35 (s), 5.8 (d, J = 16 cps), 7.3 (d, J = 16 cps)
4a	1.2 (t), 1.3 (t), 2.5 (q), 3.3–3.7 (m),[c] 4.2 (q), 5.8 (d, J = 15 cps),[d] 7.0 (d, J = 15 cps)[e]
4b	1.0–1.5 (m),[f] 2.6 (m), 3.3–3.7 (m),[g] 4.2 (q), 5.8 (d, J = 16 cps), 6.9 (d, J = 16 cps)[h]
4c	0.7–1.6 (m),[i] 2.3 (m), 3.3 (d), 3.4 (q), 4.1 (q), 5.7 (d, J = 15 cps), 6.7 (d, J = 16 cps)[h]
5b[j]	2.0 (s), 5.4 (s), 6.0 (d, J = 16 cps), 7.5 (d, J = 16 cps), 12.6 (s)
5c	1.2 (t), 2.3 (q), 5.4 (s), 5.9 (d, J = 16 cps), 7.4 (d, J = 16 cps)
5d	0.9 (t),[b] 1.2–1.8 (m), 2.4 (t),[b] 5.4 (s), 5.9 (d, J = 15 cps), 7.4 (d, J = 15 cps), 12.6 (s)

[a] Chemical shift from tetramethylsilane in approximately 20% by weight solution in carbon tetrachloride. [b] Extremely distorted triplet. [c] Triplet overlapping a quartet. [d] Further split to triplets, J = 1 cps. [e] Further split to triplets, J = 7 cps. [f] Overlapping methyls: 1.1 (d), 1.2 (t), and 1.3 (t). [g] Overlapping methylenes: 3.3 (d), 3.4 (q). [h] Further split to doublets, J = 7 cps. [i] Overlapping methyls: 0.9 (t), 1.2 (t), 1.3 (t). [j] Chemical shift values are approximate.

(1) G. A. Berchtold, J. Ciabattoni, and A. A. Tunick, *J. Org. Chem.*, **30**, 3679 (1965).
(2) S. Danishefsky and R. Cunningham, *ibid.*, **30**, 3676 (1965).
(3) C. F. H. Allen and J. Van Allan in "Organic Syntheses," Coll. Vol. III, E. C. Horning, Ed., John Wiley and Sons, Inc., New York, N. Y., 1955, p 783.
(4) E. Adlerová, L. Bláha, M. Borovička, I. Ernest, J. O. Jílek, B. Kakáč, L. Novak, M. Rajšner, and M. Protiva, *Collection Czech. Chem. Commun.*, **25**, 226 (1960).
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(7) Y. Ishikawa, *Bull. Chem. Soc. Japan*, **36**, 1527 (1963).
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(11) H. O. House and G. H. Rasmusson, *J. Org. Chem.*, **26**, 4278 (1961).
(12) V. F. Kucherov, B. G. Kovalev, I. I. Nazarova, and L. A. Yanovskaya, *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk*, 1512 (1960); *Chem. Abstr.*, **55**, 1420b (1961).
(13) F. Zymalkowski and A. W. Frahm, *Arch. Pharm.*, **297**, 219 (1964).

The esters **3b–3d** were saponified in good yield to the corresponding dienoic acids. The product yields and physical and nmr spectral properties are recorded in Tables II and III.[14] The esters **3a–3d** appear to be the

TABLE III

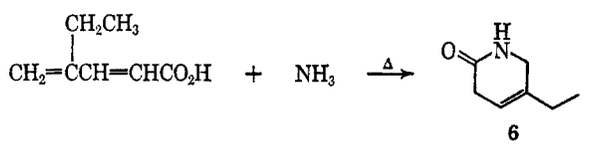
4-ALKYL-2,4-PENTADIENOIC ACIDS

Compd	R	Yield, %	Mp, °C	Calcd, % C	Calcd, % H	Found, % C	Found, % H
5b	CH_3	72	64–65[a]	64.3	7.2	64.1	7.3
5c	CH_3CH_2	80	80–82	66.6	8.0	66.6	8.0
5d	$CH_3(CH_2)_4$	67	35–37	71.4	9.6	71.4	9.4

[a] Lit.[14] mp 56.5°.

pure *trans* isomers. The nmr spectra show coupling constants of 15–16 cps for the 2 and 3 protons and reveal no extraneous peaks which could be attributed to the *cis* isomers. Esters **3b** and **3c** gave a single peak on gas chromatographic analysis on an SE-30 column. This stereochemical course of the reaction is a further example of preferred formation of the *trans* isomer in reactions involving phosphonate carbanions.[15]

The original purpose for the synthesis of 4-ethyl-2,4-pentadienoic acid was to convert it to 5-ethyl-5,6-dihydro-2(1H)-pyridone by reaction with ammonia using the procedure developed for sorbic acid by Shamma and Rosenstock.[16] Under these conditions the only lactam which could be isolated was 5-ethyl-3,6-dihydro-2(1H)-pyridone (**6**) in which the double bond is found in the unconjugated but more highly substituted 4,5 position. The structural assignment



6

followed from the absence of any maxima in the ultraviolet region from 220 to 300 mμ and was confirmed by the nmr spectrum of **6**, which shows a single vinyl proton as a broad multiplet at δ 5.45 and other features in accord with structure **6**. Vinylacrylic acid was also heated with ammonia under pressure. The lactam which was isolated was 5,6-dihydro-2(1H)-pyridone (**7**). Vinylacrylic acid thus reacts similarly to sorbic and 2-styrylacrylic acid under these conditions to give the 5,6-dihydropyridone. The vinyl proton region of the nmr spectrum of **7** differed markedly from that of **6**, showing two vinyl protons, a doublet (J = 10 cps) at δ 5.9 which showed additional splitting of ~2 cps, and a doublet of triplets, J = ~10 and 4 cps. The ultraviolet spectrum of **7** showed maxima at 206 and 240 mμ in agreement with reports in the literature for other 5,6-dihydro-2(1H)-pyridones.[17–19]

The isolation of nonconjugated 3,6-dihydropyridones along with the 5,6-dihydro compounds from the reaction of sorbic acid with various amines has been noted.[19] The stabilizing influence of the 5-alkyl substituent on the 4,5 double bond may make the 3,6-dihydro isomer the more stable in the 5-alkyldihydro-2(1H)-pyridone system, but more systematic investigation would be required to allow any firm conclusion to be drawn.

(14) T. Lennartz, *Ber.*, **76**, 1006 (1943).

(15) D. H. Wadsworth, O. E. Schupp, III, E. J. Seus, and J. A. Ford, Jr., *J. Org. Chem.*, **30**, 680 (1965); L. Horner, H. Hoffmann, H. G. Wippel, and G. Klahre, *Chem. Ber.*, **92**, 2499 (1959).

(16) M. Shamma and P. D. Rosenstock, *J. Org. Chem.*, **26**, 718 (1961).

(17) G. Di Maio and P. A. Tardella, *Gazz. Chim. Ital.*, **94**, 584 (1964); *Chem. Abstr.*, **61**, 11967d (1964), have reported the isolation of **7** in noncrystalline form from the reaction of 1-hydroxy-2-piperidone with polyphosphoric acid.

(18) O. E. Edwards and T. Singh, *Can. J. Chem.*, **32**, 683 (1954).

(19) A. J. Verbiscar and K. N. Campbell, *J. Org. Chem.*, **29**, 2472 (1964).

Experimental Section

2-Substituted Acroleins.—The acroleins **1b**, **1c**, and **1d** were prepared as described by Marvel, Myers, and Saunders.[20]

Ethyl 4-Alkyl-3,4-pentadienoates.—The procedure for **3b**, given in detail below, is typical. Sodium (4.6 g, 0.2 g-atom) was added to absolute ethanol (100 ml). After the metal had dissolved, the solution was cooled in an ice bath and triethyl phosphonoacetate (44.8 g, 0.20 mole) was added. After the resulting solution had been stirred at 0° for about 10 min, a solution of 2-methylacrolein (14.0 g, 0.20 mole) in ethanol (20 ml) was added slowly. The reaction was strongly exothermic and the rate of addition was controlled so that the solution temperature did not exceed 20°. When addition of the aldehyde was complete, the ice bath was removed and the yellow solution was stirred at room temperature for 15–30 min. The reaction mixture was then poured into brine and the mixture was extracted with hexane. The hexane was dried over magnesium sulfate, concentrated at reduced pressure, and distilled to give **3b** and **4b**. A few crystals of hydroquinone were added to the product prior to distillation and to the distilled ester.

4-Alkyl-2,4-pentadienoic Acids.—The procedure for **5b** is typical. Compound **3b** (4.0 g, 0.029 mole) was dissolved in methanol (40 ml), and sodium hydroxide (2.5 g) and water (5 ml) were added. The solution was refluxed for 2 hr and then diluted with water and washed with ether. The alkaline solution was acidified with cold, dilute sulfuric acid and extracted with chloroform. Evaporation of the chloroform and recrystallization of the residue from hexane gave **5b** (2.4 g, 0.021 mole, 72%), mp 58–59°. The analytical sample was prepared by recrystallization from hexane. A small amount of hydroquinone was usually added to the recrystallization solvent.

5-Ethyl-3,6-dihydro-2(1H)-pyridone (**6**).—A solution of the acid **5c** (17.8 g, 0.14 mole) in cold concentrated ammonium hydroxide (300 ml) was saturated with ammonia and sealed in a high-pressure bomb. The bomb was maintained at 200 ± 20° for 13 hr and then allowed to cool overnight. The reaction solution was filtered and then concentrated to a viscous oil on a rotary evaporator. The oil was dissolved in methanol, transferred to a distillation apparatus, and heated at 180° for 5 hr during which time nitrogen was passed through the solution. After this heating period, the residue was distilled, giving an oil, bp 109–120° (0.8 mm), which partially crystallized. Recrystallization from ether–hexane gave 1.55 g (0.012 mole, 9%) of **6** as white needles: mp 79–80°; ν_{NH} 3200 cm^{-1}; $\nu_{C=O}$ 1675 cm^{-1} in CCl_4; $\lambda_{max}^{95\%\ EtOH}$ no maxima 220–300 mμ; nmr peaks ($CDCl_3$) at δ 1.1 (triplet, 3 H), 2.1 (quartet, 2 H), 2.7–3.2 (multiplet, 2 H), 3.7–4.0 (multiplet, 2 H), 5.4 (broad singlet, 1 H), and 7.8 (broad singlet, 1 H).

Anal. Calcd for $C_7H_{11}NO$: C, 67.17; H, 8.85. Found: C, 67.27; H, 8.74.

5,6-Dihydro-2(1H)-pyridone (**7**).—Vinylacrylic acid (17.0 g, 0.17 mole), hydroquinone (0.5 g), and concentrated ammonium hydroxide (400 ml) were sealed in a high-pressure bomb. The mixture was heated to 180 ± 15° for 14 hr with shaking and then allowed to cool overnight. The reaction solution was filtered, concentrated on a rotary evaporator, and then dissolved in methanol and transferred to a 100-ml distilling flask. The contents were carefully heated to 150–170° at ~4 mm and the distillate, bp 130–180°, was collected. Redistillation through a small Vigreux column gave a liquid which solidified (4.9 g), bp 101–105° (0.5 mm). Recrystallization from ether–hexane gave colorless prisms: mp 65.5–67°; ν_{NH} 3180 cm^{-1}; $\nu_{C=O}$ 1670 cm^{-1}; $\nu_{C=C}$ 1610 cm^{-1}; $\lambda_{max}^{95\%\ EtOH}$ 206 (log ε 4.07) 240 (log ε 3.15); nmr peaks at δ 2.4 (multiplet, 2 H), 3.4 (triplet of doublets, 2 H), 5.9 (doublet with further fine splitting, 1 H), 6.7 (doublet, J = 10 cps, further split into triplets, 1 H), and 7.3 (broad singlet, 1 H).

(20) C. S. Marvel, R. L. Myers, and J. H. Saunders, *J. Am. Chem. Soc.*, **70**, 1694 (1948).

Anal. Calcd for C_8H_7NO: C, 61.83; H, 7.27. Found: C, 62.02; H, 7.38.

Registry No.—**3a**, 13369-23-8; **3b**, 13369-24-9; **3c**, 13369-25-0; **3d**, 13369-26-1; **4a**, 13369-27-2; **4b**, 13369-28-3; **4c**, 13369-29-4; **5b**, 4941-92-8; **5c**, 13369-31-8; **5d**, 13369-32-9; **6**, 13369-33-0; **7**, 6052-73-9.

Acknowledgment.—This work was supported in part by National Science Foundation Grant GP-5292.

4-Cycloheptatrienylidene-2,6-dibromocyclohexa-2,5-dienone. A Stable Quinocycloheptatriene

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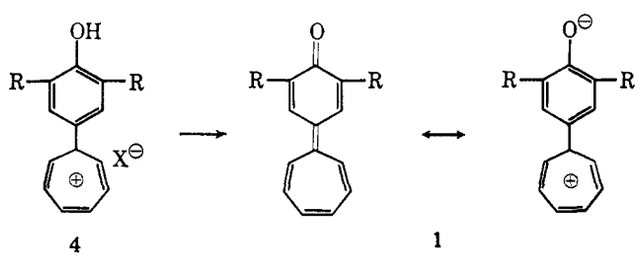
Received January 19, 1967

Interest in quinone methides of type **1** has centered around the question as to whether the charge-separated form contributes significantly to the structure.[1-3] This can presumably be determined by examination of the infrared carbonyl absorption. Several attempts have been made, therefore, to prepare compounds of type **1** by removal of HX from the appropriate tropylium ion **4** with base or thermally.[1-3] Compounds **1a** and **1b** have been reported to be air-sensitive, unstable, purple solids for which no satisfactory elemental analyses could be obtained. Their structural assignment[2] was supported[3] by the reaction of **1a** with acid to give the tropylium ion **4a** or with sodium borohydride to give the cycloheptatriene **3a**. Lack of absorption between 1600 and 2850 cm^{-1} was offered as evidence for the charge-separated form.[3] A recent patent[4] also reports several quinone methides of type **1**.



4 **1**

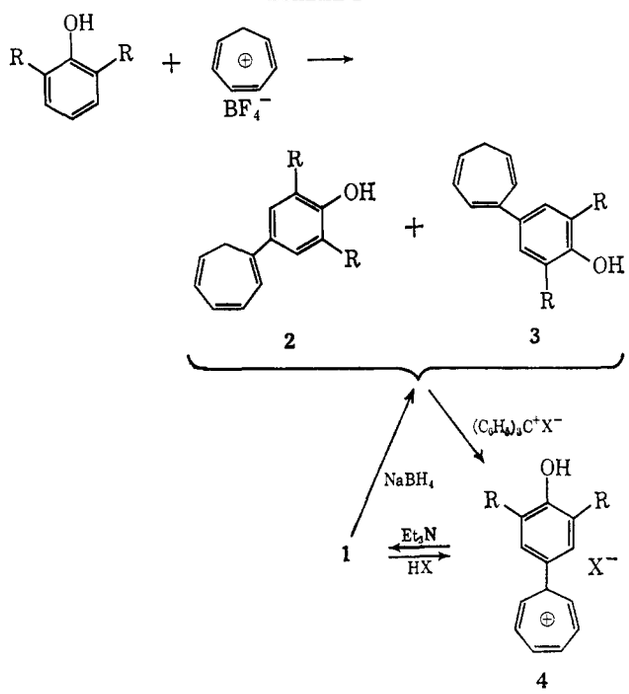
a, R = H; **b**, R = CH_3; **c**, R = Br

We wish to report the preparation and some reactions of a stable quinone methide, **1c**. The presence of bromine atoms has been found to increase the stability of analogous quinone methides in the cyclopropene series,[5,6] and it was hoped that the effect would be operative in the cycloheptatriene series.

(1) R. VanHelden, A. P. terBorg, and A. F. Bickel, *Rec. Trav. Chim.*, **81**, 599 (1962).
(2) C. Jutz and F. Voithenleitner, *Chem. Ber.*, **97**, 29 (1964).
(3) P. Bladon, P. L. Pauson, G. R. Proctor, and W. J. Rodger, *J. Chem. Soc., Sect. C*, 926 (1966).
(4) T. Nozoe, Japanese Patent 17674 (1964); see *Chem. Abstr.*, **62**, 5234 (1965).
(5) B. Föhlisch and P. Burgle, *Tetrahedron Letters*, 2661 (1965).
(6) A. S. Kende, *J. Am. Chem. Soc.*, **85**, 1882 (1963).

Alkylation of 2,6-dibromophenol with tropylium fluoroborate in pyridine gave, upon distillation, a 46% yield of a mixture of **2c** and **3c** from which the 3 isomer **3c** was separated by fractional crystallization. The mixture was converted to the tropylium salt **4c** with the appropriate triphenylmethyl salt. When the tropylium salt **4c** was treated with triethylamine in acetonitrile, the crystalline quinone methide **1c** precipitated. It could also be obtained by treating an aqueous solution of **4c** with base (see Scheme I).

SCHEME I



2 **3**

a, R = H; **b**, R = CH_3; **c**, R = Br

The quinone methide **1c** is stable at room temperature in air and in acetonitrile solution. Addition of hydroxylic solvents or triethylamine to the solution destroys the characteristic purple color. The proposed structure is supported by chemical evidence. The compound is converted to the tropylium ion **4c** by perchloric or fluoroboric acid, and is reduced to a mixture of **2c** (45%) and **3c** (55%) by sodium borohydride.

A satisfactory nmr spectrum was not obtained for lack of a suitable solvent. The material is not sufficiently soluble in deuteriochloroform, deuteriobenzene, dueteriopyridine, acetone, acetonitrile, or methylene chloride; it reacts with dimethyl sulfoxide. Visible absorption occurs at 552 mμ. The infrared spectrum (KBr) shows no bands between 1580 and 3000 cm^{-1}, which suggests that the compound exists as the charge-separated form.

Since the dibromoquinone methide **1c** had been isolated as a stable solid from triethylamine in acetonitrile, the preparation of the dimethyl compound **1b** was reinvestigated under these conditions. Tropylation of 2,6-dimethylphenol in pyridine, followed by distillation, gave a 70% yield of a mixture of **2b** and **3b**. This yield compares favorably to the one previously reported from the alkylation in acetic acid[1] (9.1%). The 3 isomer **3b** could be separated by crystallization. The mixture was readily converted to the desired tropylium