silver triflate in 3 ml of carbon tetrachloride at ambient temperature. Yields were determined after 2 hr by both proton and fluorine nmr using benzotrifluoride as a quantitative standard. A 97% yield of triflates was obtained consisting of 34% propyl triflate and 66% isopropyl triflate. The yields of the components varied ±10% but the total was always nearly quantitative. The same results were obtained using 1,1,2-trichlorotrifluoroethane or pentane as solvent. Methylene chloride gave a 95% yield consisting of 59% propyl triflate and 41% ispropyl triflate. Using benzene as solvent (18 hr) gave a 92% yield of propyl triflate with no isopropyl triflate. A solvent consisting of 33% benzene and 67% 1,1,2-trichlorotrifluoroethane gave a 98% yield containing 43% propyl triflate and 57% isopropyl triflate; 50% benzene and 50% 1,1,2-trichlorotrifluoroethane gave a 98% yield with 51% propyl triflate and 49% isopropyl triflate; 67% benzene and 33% 1,1,2-trichlorofluoroethane gave a 94% yield with 77% propyl triflate and 23% isopropyl triflate.

Propyl Triflate. A solution of 0.30 g (5 mmol) of propanol and 0.395 g (2 mmol) of pyridine in 5 ml of carbon tetrachloride was added dropwise with stirring to a solution of 1.41 g (5 mmol) of triflic anhydride in 10 ml of carbon tetrachloride at 0°. In 15 min the solution was filtered, washed with water, and dried over magnesium sulfate. Nmr analysis using chlorobenzene as a quantitative reference, showed an 86% yield of propyl triflate: proton nmr (CCl_4) δ 4.45 (t, 2 H, J = 6 Hz, CH_2O-), 1.83 (m, 2 H, CH_2CH_2O-), and 1.08 ppm (t, 3 H, J = 6 Hz, CH_3); fluorine nmr (CCl_4) ϕ 75.80 ppm (s); ir (CCl_4) 2990 (m), 1460 (w), 1420 (vs), 1250 (s), 1220 (vs), 1155 (vs), and 950 cm^{-1} (vs).

Preparation of Alkyl Triflate Solutions. By the procedure used above for propyl iodide, equivalent amounts of silver triflate were reacted with methyl iodide, ethyl iodide, isopropyl iodide, allyl iodide, pentyl iodide, hexyl iodide, and decyl iodide to give the corresponding triflates. The respective solvents and yields are shown in Table II.

Pentyl Triflate. Pentyl iodide (0.91 g, 4.6 mmol) was added dropwise with stirring to a partial suspension of 2.40 g (9.2 mmol) of silver triflate in 25 ml of benzene. The mixture was stirred 18 hr, filtered, washed with water, dried over magnesium sulfate, and distilled to give 0.785 g (82%) of pentyl triflate, bp 55–57 (1.5 mm), with spectra identical with those reported.[7]

Hexyl Triflate. Hexyl iodide (2.12 g, 10 mmol) was reacted with 2.57 g (10 mmol) of silver triflate in 50 ml of benzene as above to give 2.13 g (91%) of hexyl triflate, bp 26–28° (0.1 mm): proton nmr (CCl_4) δ 4.43 (t, 2 H, J = 6 Hz, CH_2O), 1.80 (m, 2 H, CH_2CH_2O), 1.26 (m, 6 H, CH_2), and 0.90 ppm (m, 3 H, CH_3); fluorine nmr (CCl_4) ϕ 75.8 ppm (s); ir (CCl_4) 1420, 1225, 1155, and 940 cm^{-1} (SO_3CF_3).

Anal. Calcd for $C_7H_{13}F_3SO_3$: C, 35.90; H, 5.59. Found: C, 35.81; H, 5.72.

Decyl Triflate. Decyl iodide (4.02 g, 15 mmol) was reacted by the above procedure with 5.14 g (20 mmol) of silver triflate in 100 ml of benzene. The washed and dried benzene solution was filtered through silicic acid and stripped of solvent to give 4.05 g (93%) of decyl triflate, a colorless oil: proton nmr ($CDCl_3$) δ 4.42 (t, 2 H, J = 6 Hz, CH_2O-), 1.82 (m, 2 H, CH_2CH_2O-), 1.27 (m, 14 H, CH_2), and 0.83 ppm (m, 3 H, CH_3); fluorine nmr ϕ 75.4 (s); ir (CCl_4) 1420, 1220, 1160, and 950 cm^{-1} (SO_3CF_3).

Anal. Calcd for $C_{11}H_{21}F_3SO_3$: C, 45.50; H, 7.29; S, 11.05. Found: C, 45.44; H, 7.09; S, 11.40.

Registry No.—Silver perchlorate, 7783-93-9; silver triflate, 2923-28-6; propyl triflate, 29702-90-7; propanol, 71-23-8; triflic anhydride, 358-23-6; pentyl triflate, 41029-43-0; hexyl triflate, 53059-88-4; decyl triflate, 53059-89-5; $CH_3(CH_2)_8CH_2I$, 2050-77-3.

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New Carbonyl Compounds from the Alkaline Ferricyanide Dehydrogenation of *p*-Cresol

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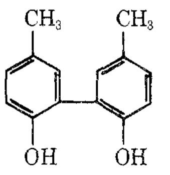
Pummerer's ketone type trimeric ketone **8** and tetrameric ketone **9** have been obtained from the dehydrogenation of *p*-cresol with alkaline ferricyanide in addition to **1**, **2**, **3**, and **4**. The tetrameric ketone **9** is apparently formed through dehydrogenation and subsequent intramolecular radical substitution of **10** which was produced by Pummerer's ketone type oxidative coupling of the diphenyl **1**. Trimeric hemiketals **6** and **7** obtained previously from the ferric chloride dehydrogenation of *p*-cresol and/or their derivatives were not found in the alkaline ferricyanide or peroxide–peroxidase dehydrogenation of *p*-cresol. It had been concluded that in the ferric chloride dehydrogenation hemiketals **6** and **7** were formed through acid-catalyzed hydration of **12**, which was produced by dehydrogenation and subsequent intramolecular radical substitution of **10**, rather than by subsequent acid-catalyzed reactions of **11**. Dehydration of **6** by general acid catalysis results in rearrangement of the molecule involving an intramolecular ether interchange by O-5 participation of the benzofuran oxygen, followed by dienol–benzene rearrangement to give **13**.

We previously reported that the dehydrogenation of *p*-cresol by the one electron transfer agent ferric chloride in acidic solution yielded three new ketonic products **5–7** and a dimeric ether **3** in addition to **1**, **2**, and Pummerer's ketone **4**.[2] In that communication we reported that **5** was isolated from the peroxide–peroxidase dehydrogenation of *p*-cresol but not hemiketals **6** and **7**. Compounds **1**, **2**, and **4** were isolated earlier from the ferric chloride,[3-5] alkaline ferricyanide[6] and peroxide–peroxidase[7] dehydrogenation of *p*-cresol.



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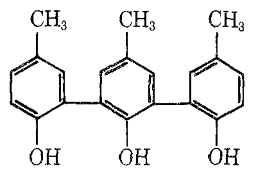

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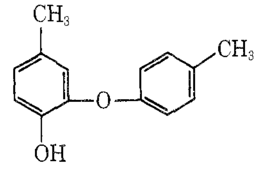

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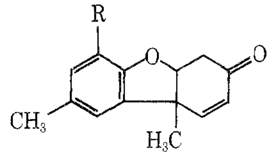
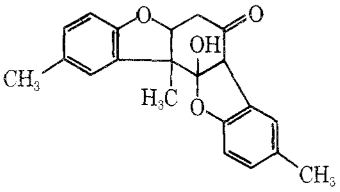


4, R = H
5, R = 2-hydroxy-5-methylphenyl



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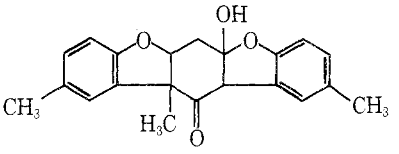

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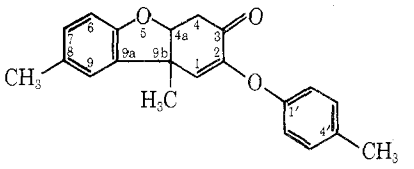

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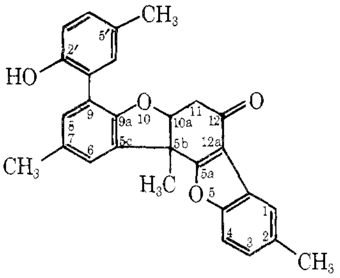
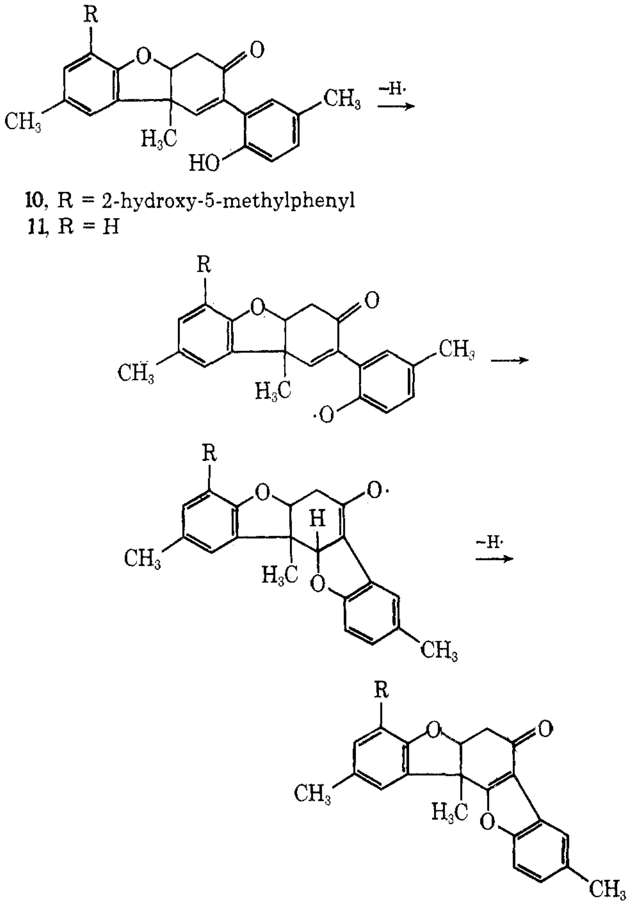


9

We have now reinvestigated the alkaline ferricyanide dehydrogenation of *p*-cresol which was previously reported by Haynes, *et al.*[6] In that communication it was reported that **1**, **2**, and **4** were isolated and that 32% nonketonic polymers of unknown structure were formed. In our present work, *p*-cresol in 0.4 *N* sodium carbonate solution was dehydrogenated with 1.5 equiv of potassium ferricyanide. During the reaction *ca.* 1.3 equiv of potassium ferricyanide was consumed. Two new ketonic products, 2-(4'-methylphenoxy)-4a,9b-dihydro-8,9b-dimethyl-3(4*H*)-dibenzofuranone (**8**) and 9-(2'-hydroxy-5'-methylphenyl)-5b,10a-dihydro-2,5b,7-trimethyl-12(11*H*)-benzo[1,2-*b*:3,4-*b*']bisbenzofuranone (**9**) were isolated in addition to **1**, **2**, **3**, and **4**.

The ir spectrum of the trimeric ketone **8**, $C_{21}H_{20}O_3$ (M^+, *m/e* 320), indicated the presence of an α,β-enone (1690 and 1635 cm^{-1}), cyclic trisubstituted double bond (863 and 851 cm^{-1}), isolated and two adjacent aromatic hydrogens (880 and 805 cm^{-1}). The nmr spectrum showed that two geminal protons H-4, H-4a with H-1 constituted the characteristic ABMX system of Pummerer's ketone derivatives[2] with J_{AB} = 17.8, J_{AM} = 2.7, J_{BM} = 3.9, and J_{MX} = 1.8 Hz. The absence of H-2 signal indicated that **8** was a derivative of **4** with a substitutional group corresponding either to 4-methylphenoxyl or 2-hydroxy-5-methylphenyl group on C-2. The ir and uv spectra showed the absence of phenolic hydroxyl group in **8**. The mass spectrum exhibited ion peaks corresponding to M^+, M − 15, and M − 107 ions. Therefore, structure **8** for the trimeric ketone was apparent.

Chart I



10, R = 2-hydroxy-5-methylphenyl
11, R = H

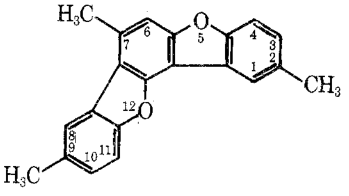
9, R = 2-hydroxy-5-methylphenyl
12, R = H

The tetrameric ketone **9**, $C_{28}H_{24}O_4$ (M^+, *m/e* 424), had two hydrogen atoms less than the expected tetrameric compound of *p*-cresol. The phenolic nature of the compound was indicated by the bathochromic shift observed in the uv spectrum when base was added. The ir spectrum showed the presence of a hydroxyl group (3320 cm^{-1}), a conjugated carbonyl (1652 cm^{-1}), isolated and two adjacent aromatic hydrogens (857 and 807 cm^{-1}). The nmr spectrum was consistent with the structure proposed for this compound. Two geminal protons H-11 and H-10a constituted an ABX system with J_{AB} = 17.8, J_{AX} = 2.9, and J_{BX} = 4.3 Hz. The absence of the α,β-enone olefinic H-5a and H-12a signals indicated that the benzofuran moiety was fused to **5** at C-5a and C-12a. The mass spectrum exhibited ion peaks corresponding to M^+ and M − 15 which is characteristic of Pummerer's ketone derivatives.[2]

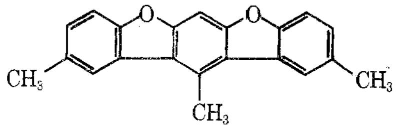
The trimeric ketone **8** is formed through the Pummerer's ketone type ortho–para dehydrogenative coupling of *p*-cresol and **3**. Tetrameric ketone **9** is formed through intramolecular dehydrogenation–radical substitution–dehydrogenation[8] of intermediate **10** which was formed through ortho–para dehydrogenative coupling of biphenyl **1**.

We previously postulated[2] that in the acidic ferric chloride dehydrogenation of *p*-cresol hemiketals **6** and **7** could be formed through addition of water to the α,β-enone portion of **11**, followed by oxidation of the resulting 1,3 ketol to the 1,3 dione and subsequent tautomerization and acid-catalyzed cyclization. However, this mechanism is not tenable in view of our present investigation. These hemiketals resulted from intermediate **12** which was formed by the intramolecular dehydrogenation–radical substitution–dehydrogenation of **11** in a manner analogous to the formation of **9** from **10**. Acid-catalyzed addition of water on the α,β-enone of **12** afforded the hemiketal **6** which underwent acid-catalyzed hydrolysis, tautomerization, and cyclization to give the isomeric hemiketal **7**.[9] This accounts for the absence of these hemiketals and/or their derivatives in the alkaline ferricyanide and peroxide–peroxidase dehydrogenation products of *p*-cresol (Chart I).

Dehydration of **6** with *p*-toluenesulfonic acid in toluene unexpectedly resulted in elimination of 2 mol of water to give the product **13**, $C_{21}H_{16}O_2$ (M^+, *m/e* 300). The ir spectrum of the product indicated the presence of 1,2,4-trisubstituted benzenes and an isolated aromatic hydrogen but the absence of hydroxyl and carbonyl groups. The uv spectrum showed very intense adsorption bands at λ_{max} 267 and 294 nm corresponding to the 250- and 280-nm bands of dibenzofuran.[10] The intensities of these bands are approximately twice those of the corresponding bands of dibenzofuran. The near constancy of $\epsilon/(n - 1)$ for both bands observed in Table I is analogous to that of *m*-polyphenyls.[11] This indicates that the product has a *m*-terphenyl skeleton consisting of two dibenzofuran units with a common benzene ring. The nmr spectrum showed the presence of three aromatic methyl groups with one of them being deshielded, and seven aromatic hydrogens which constituted two separated ABX systems with J_{AB} = 8.0 and J_{BX} = 2.2 Hz and a singlet. The mass spectrum exhibited ion peaks corresponding to M^+ and $M - 1$ ions but not the characteristic $M - 15$ ion. There are two possible structures for the product. Structure **14** belongs to symmetry species point group C_{2v} with the C_2 axis bisecting the molecule into two equivalent parts and should give a nmr spectrum with a single ABX system in the aromatic region. This is not in agreement with the nmr spectrum obtained. The structure **13**, 2,7,9-trimethylbenzo[1,2-*b*:3,4-*b*′]bisbenzofuran is consistent with the spectral data discussed above (Chart II).



13



14

It is apparent that the dehydration of **6** by general acid catalysis results in rearrangement of the molecule involving an intramolecular ether interchange followed by dienol–benzene rearrangement to give **13**. Protonation and subsequent dehydration of **6** produces the carbonium ion **15** which would give **12** by deprotonation. However, the mesomeric effect of the benzofuran oxygen to the carbonium ion

Chart II

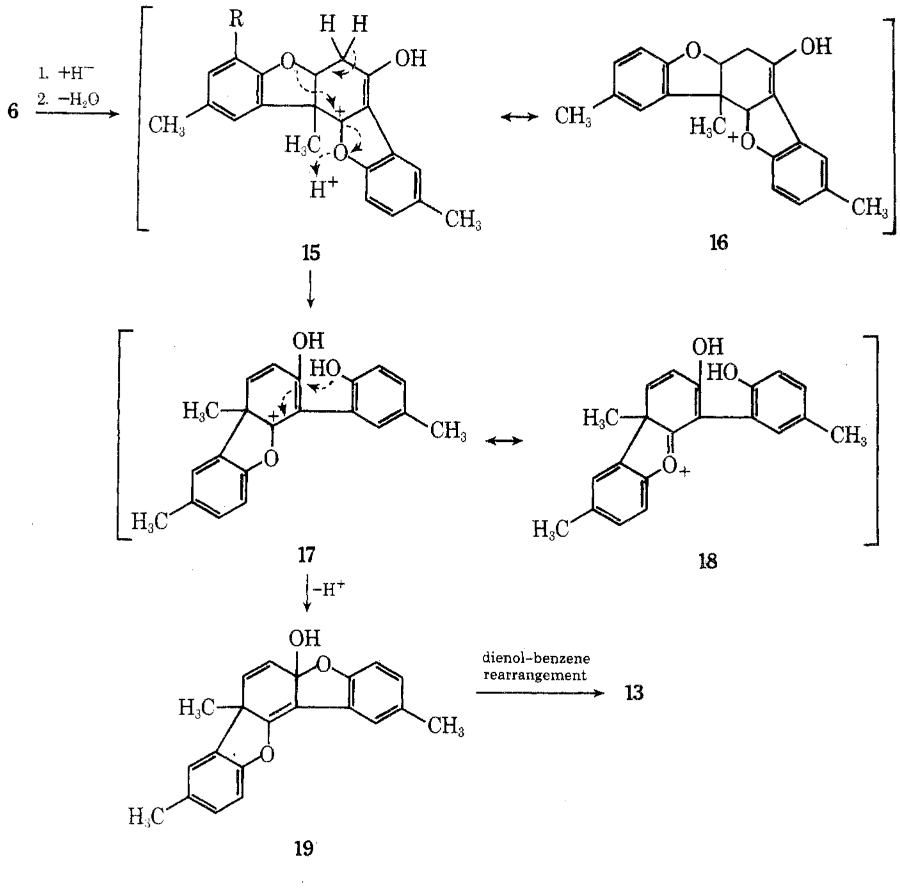


Table I

Compd	n^a	λ, nm	ε	ε/(n − 1)	λ, nm	ε	ε/(n − 1)
Dibenzofuran[b]	2	249	17,400	17,400	280	14,200	14,200
4-Methyldibenzofuran[b]	2	252	15,700	15,700	282	12,400	12,400
13	3	267	49,840	24,920	294	28,640	14,320

[a] *n* is the number of benzene rings. [b] Reference 10.

center in form **16** results in stabilization of the carbonium ion. Consequently, the equilibrium is in favor of the carbonium ion rather than the deprotonation under the reaction condition. This also prevents **15** from undergoing an alternative Wagner-Meerwein rearrangement which would lead to formation of **14** through subsequent acid-catalyzed retroaddition of α,β-enone, cyclization to hemiketal, and further dehydration. The carbonium ion **15** undergoes intramolecular ether interchange to give the carbonium ion **17** by O-5 participation of the second benzofuran oxygen.[12] Acid-catalyzed cyclization of **17** affords the hemiketal dienol **19** which undergoes dienol-benzene rearrangement to give **13**.

Experimental Section

Nmr spectra were obtained with a Varian HA 100 spectrometer, mass spectrum with an AEI MS-1201, ir with a Beckman 12 spectrophotometer, and uv with a Cary 15 uv spectrophotometer. Melting points were uncorrected.

Dehydrogenation of *p*-Cresol with Alkaline Ferricyanide. A solution of potassium ferricyanide (49.4 g) in 500 ml of water was added dropwise during an hour to a stirred solution of *p*-cresol (10.8 g) in 1 l. of 0.4 *N* sodium carbonate at room temperature. After 4 hr the reaction mixture was extracted with ether. Titration of the solution indicated that 1.32 equiv of ferricyanide had been consumed. The ether solutions was shaken with 1 *N* sodium hydroxide solution and was divided into alkali-soluble and -insoluble parts. A total of 6.2 g of alkaline-insoluble material was obtained.

The alkaline-insoluble material (5.8 g) was chromatographed on a silica gel column with chloroform–cyclohexane (4:1) as solvent to isolate **3**, **5**, **8**, and **9**.

2-Hydroxy-4′,5-dimethyl Diphenyl Ether 3. This compound was isolated from the first fraction of the column chromatography and purified by preparative tlc on silica gel as an oil (56 mg). The identification of the compound was carried out by comparison of ir and nmr with authentic sample.[2]

Pummerer's Ketone 4. This compound was obtained from the second fraction and was recrystallized from methanol; colorless plates (2.1 g), mp 124–125° (lit.[2] 124–125°).

2-(4′-methylphenoxy)-4a,9b-dihydro-8,9b-dimethyl-3(4*H*)-dibenzofuranone (8). This compound was isolated and purified from the third fraction of the column chromatography in the same manner as **3**. The compound was recrystallized from methanol as colorless plates (36 mg): mp 102–103°; uv λ_{max} (methanol) 283 (sh), 296 nm; ir (KBr) 3020 (ArH), 2963, 2920, 2902, 2860 (CH_3 and CH_2), 1690, 1634 (α,β-enone), 1612, 1506, 1490 (phenyl), 1247, 1220 ($ArOCH_3$), 880, 863, 851, 824, 811, and 805 cm^{-1}; nmr ($CDCl_3$) τ 8.44 (s, 3, Anu–CH_3), 7.72 (s, 6, Ar–CH_3), 7.06 (m, 1, J = 17.8, 3.9 Hz, $\mathrm{OCH_MCH_A}\mathbf{H}_\mathrm{B}\mathrm{CO}$), 6.83 (m, 1, J = 17.8, 2.7 Hz, $\mathrm{OCH_MC}\mathbf{H}_\mathrm{A}\mathrm{H_BCO}$), 5.38 (m, 1, $\mathrm{ArOC}\mathbf{H}_\mathrm{M}\mathrm{CH_AH_B}$), 4.26 (d, 1, J = 1.8 Hz, $-\mathrm{CH_X{=}C{-}CO}$), 3.40–2.90 (m, 7, ArH); ms *m/e* (rel int) 321 (26), 320 (M^+, 100), 306 (22), 305 (92), 290.7 (m*), 278 (5), 277 (17), 251.6 (m*), 213 (11), 198 (6), 186 (5), 185 (19), 160 (7), 159 (23), 146 (6), 145 (16), 129 (7), 128 (8), 115 (14), 91 (27), 77 (12), 65 (17).

9-(2′-Hydroxy-5′-methylphenyl)-5b,10a-dihydro-2,5b,7-trimethyl-*12(11H)-benzo[1,2-b:3,4-b′]bisbenzofuranone* (9). This compound was isolated and purified from the fourth fraction of the column chromatography in the same manner as **3**. The compound was recrystallized from methanol as colorless plate (18 mg): mp 219–220°; uv λ_{max} (methanol) 297 nm; λ_{max} (0.05 *N* CH_3ONa in methanol) 294 and 335 nm; ir (KBr) 3320 (OH), 3016 (ArH), 2974, 2958, 2910, 2893, 2860 (CH_3 and CH_2), 1652 (conjugated CO), 1614, 1583, 1510, 1488 (phenyl), 866 (sh), 857, 845, 821, 807 cm^{-1}; nmr ($CDCl_3$) τ 8.19 (s, 3, Anu–CH_3), 7.66 (s, 3, Ar–CH_3), 7.65 (s, 3, Ar–CH_3), 7.55 (s, 3, Ar–CH_3), 7.00 (m, 1, J = 17.8 and 4.3 Hz, $\mathrm{OCH_XCH_A}\mathbf{H}_\mathrm{B}\mathrm{CO}$), 6.81 (m, 1, J = 17.8 and 2.9 Hz, $\mathrm{OCH_XC}\mathbf{H}_\mathrm{A}\mathrm{H_BCO}$), 5.22 (m, 1, J = 2.9 and 4.3 Hz, $\mathrm{OC}\mathbf{H}_\mathrm{X}\mathrm{CH_AH_BCO}$), 4.94 (s, 1, eliminated by D_2O exchange, OH), 3.35 (d, 1, J = 8.0, Ar–H), 3.04 (m, 1, J = 8.0 and 2.2 Hz, Ar–H), 3.03 (d, 1, J = 8.0, Ar–H), 2.85 (m, 3, Ar–H), 2.73 (m, 1, J = 2.2 and 0.6 Hz, Ar–H), 2.13 (m, 1, J = 2.2 and 0.6 Hz, Ar–H); ms *m/e* (rel int) 425 (31), 424 (M^+, 100), 410 (20), 409 (56), 394.5 (m*), 382 (8), 381 (17).

2,7,9-Trimethylbenzo[1,2-*b*:3,4-*b′*]bisbenzofuran (13). A mixture of 5a-hydroxy-5a,5b,10a,12a-tetrahydro-2,5b,7-trimethyl-12(11*H*)-benzo[1,2-*b*:3,4-*b′*]bisbenzofuranone (**6**) (80 mg), *p*-toluenesulfonic acid (40 mg), and toluene (40 ml) was heated at reflux temperature with continuous slow removal of the solvent for 6 hr. After cooling, pyridine was added to the reaction mixture to *neutralize the acid. The mixture in chloroform* (100 ml) was washed with water, 0.5 *N* HCl, and again with water, dried, and evaporated *in vacuo*. The residue was recrystallized from chloroform–methanol to give colorless needles (36 mg), mp 164–166°; uv λ_{max} in chloroform 258, 267, 294, 309 (sh), and 323 nm (ε 32,000, 49840, 28640, 3910, and 7000); ir (KBr) 3052, 3020 (Ar–H), 2972, 2943, 2917, 2858 (CH_3), 1868, 1860, 1801, 1794, 1735, 1730 (sh), (1,2,4-trisubstituted benzene) 1653 (m, condensed aromatic ring), 1612, 1589, 1480, 1469, 1458, 1440 (phenyl and benzofuran), 869, 858 (isolated Ar–H), 820, 795 (two adjacent Ar–H) cm^{-1}; nmr ($CDCl_3$) τ 7.64 (s, 6, Ar–CH_3), 7.13 (s, 3, Ar–CH_3), 2.76 (m, 2, J = 8.0 and 2.2 Hz, Ar–H), 2.68 (s, 1, Ar–H), 2.54 (d, 1, J = 8.0 Hz, Ar–H), 2.43 (d, 1, J = 8.0 Hz, Ar–H), 2.21 (d, 1, J = 2.2 Hz, Ar–H), 1.97 (d, 1, J = 2.2 Hz, Ar–H); ms *m/e* (rel int) 301 (23), 300 (M^+, 100), 299 (31), 288 (m*).

Acknowledgments. The authors are indebted to Messrs. M. W. Wesolowski and L. C. Zank of the Forest Products Laboratory for recording nmr and ir spectra.

Registry No.—**1**, 15519-73-0; **3**, 10568-14-6; **6**, 53042-29-8; **8**, 53042-30-1; **9**, 53042-31-2; **13**, 53042-32-3; *p*-cresol, 106-44-5; potassium ferricyanide, 13746-66-2; dibenzofuran, 132-64-9; 4-methyldibenzofuran, 7320-53-8.

References and Notes

(1) (a) Part of this work was carried out at Ruhr-University Bochum, 463 Bochum-Querenburg, West Germany, (b) North Carolina State University, (c) Forest Service, U.S. Department of Agriculture. (d) Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

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