was washed four times with portions of 25 ml of warm tetrahydrofuran. The combined organic solutions were dried and evaporated under reduced pressure. The residue was analyzed by glc and shown to contain the glycols **2** and **3** in a ratio of 64:36. This ratio did not change when a similar reaction mixture was left at room temperature for 80 days and then worked up as above. When the reaction was conducted in a more dilute solution (1.13 mmol of **1** and 1.24 mmol of benzoic acid in 15 ml of $CHCl_3$ for 20 days at room temperature) the ratio of **2** to **3** was 71:29. In all cases glc revealed the presence of some 2-indanol (5–10%), which could derive either from some unreacted epoxide or from some 2-indanone in the primary reaction products.

Reactions of 1 with Trichloroacetic Acid. These reactions were carried out in carefully dried vessels and solvents in the following way. A solution of **1** (0.53 mmol) in the appropriate solvent (7 ml) was treated with trichloroacetic acid (0.58 mmol) as a *ca.* 1 *M* solution in the same solvent, left at room temperature for 24 hr, and then evaporated *in vacuo*.[14] The residue was taken up in dry tetrahydrofuran (15 ml), treated with lithium aluminum hydride (0.300 g), and refluxed for 30 min. Work-up was carried out as in the case of the benzoic acid reaction. The ratios of **2** to **3** obtained by glc are shown in Table I. Amounts of 2-indanone (revealed as 2-indanol), ranging from 13 to 20%, were also found.

Hydrolysis of 1. A suspension of **1** (0.100 g) in 0.1 or 1 *N* aqueous H_2SO_4 (10 ml) was stirred for 24 hr at room temperature and then made alkaline with $NaHCO_3$, saturated with NaCl, and extracted with five 25-ml portions of ether. The residue obtained after evaporation of the dried extract was analyzed by glc; see Table I. The diols **2** and **3** were found to be stable under the reaction conditions. Small amounts of 2-indanone (1 and 4% in the reactions carried out in 0.1 and 1 *N* H_2SO_4, respectively) were found.

Equilibration and Rearrangement of 2 and 3. Solutions of each of the diols (50 mg) in 1 *N* H_2SO_4 in 75:25 dioxane–water (v/v, 5 ml) were refluxed for 1 hr and then worked up as above. Glc analysis gave the following results: from the trans diol **2**, 7% 2-indanone, 93% **2** + **3** (ratio 67:33); from the cis diol **3**, 18% 2-indanone, 82% **2** + **3** (ratio 69:31).

Comparison between the Reactions of Indene with Peroxyformic Acid and of 1 with Formic Acid. A. A mixture of 90% formic acid (7 ml), water (0.36 ml), and 35% hydrogen peroxide (1.2 ml) was heated at 35° for 15 min. Indene (1.16 g, 10 mmol) was then slowly added under stirring, while the temperature was kept at 35–40°. Stirring was continued for 1 hr at 35°, then at room temperature for 1 night. NaOH (6 *N*, 25 ml) was added; the mixture was heated at 90° for 3 hr, cooled, saturated with NaCl, and extracted with six 30-ml portions of ether. The dried extract was evaporated; glc analysis of the residue revealed the presence of **2** and **3** in a ratio of 15:85.

In a second run, with the same amounts of reagents and reaction conditions, the reaction product was not treated with base, but instead diluted with water (10 ml), saturated with NaCl, and extracted with five 30-ml portions of ether. The ether extracts were washed with water (2 × 25 ml), saturated $NaHCO_3$ (6 × 10 ml), and water (5 ml). The combined washings were extracted again with three 20-ml portions of ether, all the ether solutions were combined and evaporated, and the residue was examined by glc. The results are discussed in the introductory part.

B. The reactions were repeated under exactly the same conditions as in A, except for the reagents, that were 90% formic acid (7 ml), water (1.33 ml), 35% hydrogen peroxide (0.22 ml), and 1,2-epoxyindane (1.32 g, 10 mmol). In the hydrolyzed crude product **2** and **3** were present in a ratio of 25:75.

Acknowledgment. This work was supported in part by a grant from the Consiglio Nazionale delle ricerche.

Registry No.—**1**, 768-22-9; **2**, 4647-43-2; **3**, 4647-42-1; **6**, 19597-99-0; *trans*-2-bromo-1-indanol, 10368-44-2; 2-indanone, 615-13-4; 2-indanol, 4254-29-9.

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(14) Quenching with aqueous base was avoided in order to minimize loss of water-soluble products, after it was established that direct evaporation of the reaction solutions did not produce any appreciable changes in product compositions.

Ring Closure Reactions. III.[1] Synthesis of Some Medium-Sized Cyclic Aromatic Ethers from *o*-(ω-Bromoalkyl)phenols

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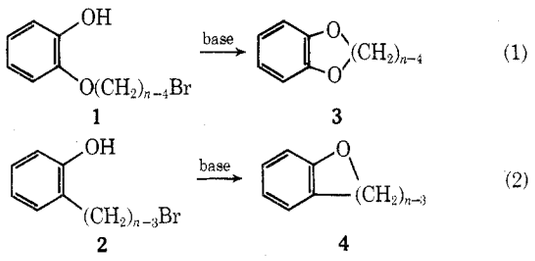
A synthesis of cyclic ethers of ring size n = 8, 9, and 10 as an alternative, convenient route to Ziegler's high-dilution technique is described. It is based on the highly favorable cyclization of *o*-(ω-bromoalkyl)phenate ions (**2**) in DMSO solution to yield 3,4,5,6-tetrahydro-2*H*-1-benzoxocin (**4**, n = 8), 2,3,4,5,6,7-hexahydro-2*H*-1-benzoxonin (**4**, n = 9), and 3,4,5,6,7,8-hexahydro-2*H*-1-benzoxecin (**4**, n = 10). The formation of varying amounts of isomeric alkenylphenols as by-products is recorded and discussed. Two alternative routes to the open-chain precursors from ω-X-alkyl *o*-anisyl ketones (**6** and **12**) are compared. In one of them the interesting competitive cyclization of 5-bromopentyl *o*-hydroxyphenyl ketone to cyclopentyl *o*-hydroxyphenyl ketone (**9**) is observed.

In the course of our investigation on the kinetics of ring closure of the anions derived from ω-bromoalkoxy- and ω-bromoalkylphenols, **1** and **2**, to the cyclic diethers[1] and monoethers,[2] **3** and **4**, respectively, cyclization on a preparative scale of compounds **2**, n = 8, 9, and 10, to the corresponding new macrocyclic monoethers[3] **4** was re-



quired. This could be accomplished by the high-dilution method given by Ziegler, Lüttringhaus, and Wohlgemuth[4] for the cyclization of 1, n = 7–14, in boiling amyl alcohol in the presence of an excess solid K_2CO_3. However, the high-dilution technique suffers from long reaction times, large volumes of solvent, and an awkward set-up for the slow addition of the reagent to the reaction medium,[5] so that a more expeditious, less cumbersome procedure was highly desirable. Preliminary kinetic data showed that intramolecular alkylation of phenoxides occurred in DMSO some 10^4 times as fast as in aqueous ethanol, the $t_{1/2}$ for the cyclization of compounds 2, n = 8, 9, and 10, being about 0.3, 3, and 2 min at 30°, respectively. This finding led us to an alternative method for the preparation of the three cyclic ethers 4, n = 8, 9, and 10, involving the cyclization of the corresponding ω-bromoalkylphenols in DMSO by conventional procedures and apparatus and under mild conditions. Thus, since the reactants could be mixed in a relatively concentrated state and the addition times were as short as 1 hr, the high-dilution technique was avoided.

The results are reported in Table I. The yields are satisfactory in two out of the three reactions. With n = 8 and 9 the cyclic ether is accompanied by an open-chain isomer, the alkenylphenol **14**, to a different extent. Since the eight-membered ring is formed significantly faster than the nine- and ten-membered homologs and identical experimental conditions were adopted in all cases, a second-order β-elimination reaction of the E2 type with OH^- as a base may be ruled out, because it should compete the more favorably the slower the cyclization reaction, yielding the greatest amount of **14** for n = 9. The fact that just the opposite is observed indicates that an intramolecular β-elimination reaction is likely to be responsible for olefin formation, the phenoxide oxygen acting as a base. It should be noted that olefins were also found to accompany eight- and nine-membered ring formation in the closely related reaction 1.[1] The high intramolecular elimination/intramolecular substitution ratio observed is probably due to a steric congestion in the transition states leading to the eight- and nine-membered rings of both series, as strongly supported by our kinetic data.[1,2]

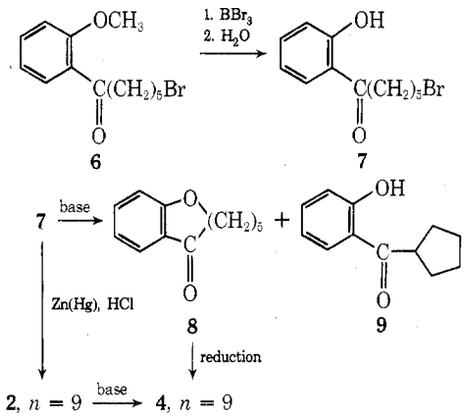
For the syntheses of the bifunctional precursors the alternative routes reported in Scheme I were first attempted in the case of n = 9. Compound **6** was readily prepared by standard methods. However, Clemmensen reduction of **7**

Table I
Products Obtained from Reaction of o-(ω-Bromoalkyl)phenols 2 with Sodium Hydroxide in DMSO at 55°

o-(ω-Bromoalkyl)phenol	n[b]	Products, %[a] Cyclic ether 4	Alkenylphenol 14
o-(5-Bromopentyl)phenol	8	30	56.5
o-(6-Bromohexyl)phenol	9	62	11.5
o-(7-Bromoheptyl)phenol	10	82	None

[a] Per cent of actually isolated products as based on the starting phenol. [b] Size of ring formed.

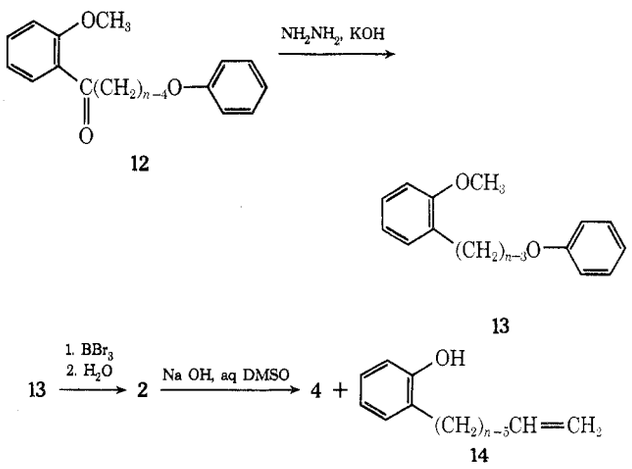
Scheme I



to **2**, n = 9, was unsuccessful, as the reduced phenolic material did not contain any bromine. Moreover, treatment of **7** with base under varying experimental conditions gave only low yields of the expected macrocyclic keto ether, 3,4,5,6-tetrahydro-2*H*-1-benzoxonin-7-one (**8**), the major product being cyclopentyl o-hydroxyphenyl ketone (**9**), presumably formed *via* a carbanion intermediate. Thus, it appears that the lower acidity of the α-methylene group as compared to that of the phenolic hydroxyl is more than offset by the extremely greater ease of cyclization to a five-membered ring rather than to a nine-membered one. An analogous phenomenon was recently reported by Greco and Warchol,[6] in connection with the unexpected formation of a five-membered ring instead of a larger one.

Protection of the $-CH_2Br$ function by phenoxylation together with the proper modifications of Scheme I resulted in a substantial improvement, the above complications being thus avoided (Scheme II, n = 8, 9, and 10). Huang-Minlon reduction of ketones **12** gave the diethers **13** in fairly good yields. A simultaneous cleavage of both ethereal functions of the latter compounds was achieved by treatment with 2 equiv of BBr_3 and afforded the desired o-(ω-bromoalkyl)phenols.

Scheme II



Experimental Section

Infrared spectra were obtained on a Perkin-Elmer 257 spectrophotometer, from 2% solutions in CCl_4. Proton magnetic resonance spectra were obtained in CCl_4 solutions either on a Varian A-60 or Jeol JNM-C60HL spectrometer, using TMS as the internal standard. Mass spectra were performed on a AEI MS 12 spectrometer. The preliminary rate experiments for the cyclization of compounds **2** were performed by monitoring the disappearance of

the phenoxide ions absorption at either 263 or 312 nm on a Beckman DB GT spectrophotometer.

1,4-Dibromobutane (Fluka), 1,5-dibromopentane (Fluka), 1,6-dibromohexane (Merck), *o*-bromoanisole (Fluka), boron tribromide (Merck), and phenol (Erba RP) were all commercial samples and used as such.

The purity of synthesized compounds was thoroughly checked by tlc using several eluents.

***o*-Hydroxyphenyl 5-Bromopentyl Ketone (7).** Treatment of 1,5-dibromopentane with KCN in water–ethanol[7] gave 6-bromohexanenitrile (**5**) in 31% yield, bp 80° (2 mm), $n^{19}D$ 1.4777. Interaction of **5** with the Grignard reagent derived from *o*-bromoanisole, followed by acid hydrolysis,[8] afforded 5-bromopentyl *o*-anisyl ketone (**6**) in 52% yield, ir 1675 cm^{-1} (C=O). Demethylation of **6** to **7** was effected in 73% yield by treatment with slightly more than the equimolecular amount of BBr_3 in dry CH_2Cl_2 at −20°.[9] Compound **7** was crystallized from methanol: mp 44.5–45°; ir 1640 (C=O), 3000–3500 cm^{-1} (OH, very broad); pmr δ 12.2 (s, 1 H, OH), 6.6–7.8 (m, 4 H, nuclear protons), 3.35 (t, 2 H, $COCH_2$ protons), 2.95 (t, 2 H, CH_2Br protons), 1.4–2.1 (m, 6 H, "central" methylene protons).

Cyclization Experiments with 7. These were effected by treatment with base under various experimental conditions, *i.e.*, (i) anhydrous K_2CO_3 in boiling dry amyl alcohol under high-dilution conditions;[4] (ii) NaOH in refluxing 1-butanol, 20 hr; (iii) NaOH in boiling 75% ethanol, 7 hr; (iv) NaOH in 95% aqueous DMSO at 70°, 5 hr. The crude reaction products showed in all cases in the ir spectra a very broad hydroxyl absorption at 3000–3500 cm^{-1} together with two carbonyl absorptions at 1640 and 1675 cm^{-1}. The lower frequency band was the more intense one. Chromatography on silica gel (eluent $CHCl_3$) of the crude product coming from the reaction carried out in 75% ethanol allowed the elution of the following components as separate, pure (tlc) fractions in the given order. Cyclopentyl *o*-hydroxyphenyl ketone (**9**), 47.5% yield, and 3,4,5,6-tetrahydro-2*H*-1-benzoxonin-7-one (**8**), 23% yield. Structure assignments were effected on the basis of spectral data. Compound **9**: ir 3000–3500 (OH), 1640 cm^{-1} (C=O); pmr δ 12.4 (s, 1 H, OH), 6.6–7.8 (m, 4 H, nuclear protons), 3.2–4.0 (m, 1 H, α carbonyl proton), 1.3–2.3 (m, 8 H, cyclopentyl methylene protons). Compound **8**: ir 1675 cm^{-1} (C=O); pmr δ 6.7–7.7 (m, 4 H, nuclear protons), 4.1 (m, 2 H, CH_2O protons), 3.0 (m, 2 H, $COCH_2$ protons), 1.7 (m, 6 H, "central" methylene protons). In the mass spectrum both isomers showed a molecular peak at *m/e* 190 (calcd, 190), together with a base peak at *m/e* 121, probably due to the fragment *o*-$HOC_6H_4CO^+$.

***o*-(ω-Bromoalkyl)phenols (2, *n* = 8, 9, and 10).** ω-Phenoxy bromides **10** were obtained by the reaction of sodium phenoxide with the appropriate α,ω-dibromoalkane with a procedure derived from that of Marvel and Tanenbaum,[10] the major difference being that longer reaction times were used, namely 5, 7.5, and 20 hr for *n* = 8, 9, and 10, respectively. The compounds were purified by fractional distillation: compound **10**, *n* = 8, 65% yield, bp 110–123° (2.5 mm), mp 37.5–39.5°; compound **10**, *n* = 9, 52% yield, bp 126–130° (2 mm), $n^{27}D$ 1.5446; compound **10**, *n* = 10, 73% yield, bp 107–112° (0.2 mm), $n^{27}D$ 1.5291. Treatment of such compounds with KCN gave nearly quantitative yields of the phenoxy nitriles **11**, ir 2250 cm^{-1} (C≡N). The crude nitriles **11** were converted as above (see formation of **6** from **5**) to the corresponding ketones **12** in 90% yield, ir 1680 cm^{-1} (C=O). The crude ketones were reduced to the diethers **13** in about 55% yield by the Huang-Minlon modification of the Wolff–Kischner reduction. The diethers were purified by chromatography on silica gel using $CHCl_3$–light petroleum (2:1) as eluent: *n* = 8, $n^{28}D$ 1.5497; *n* = 9, $n^{28}D$ 1.5470; *n* = 10, $n^{28}D$ 1.5384. Treatment with 2 equiv of BBr_3[9] gave **2**, *n* = 8, 9, and 10, in 53, 60, and 75% yield (16, 16, and 28% overall yield), respectively. After work-up of the reaction mixtures the crude reaction products were dissolved in light petroleum and repeatedly washed with water in order to remove phenol, until a negative test with $FeCl_3$ was obtained in the aqueous washings. After drying over anhydrous sodium sulfate and removal of the solvent, the product was purified by fractional distillation: compound **2**, *n* = 8, bp 127–133° (1 mm), $n^{24.5}D$ 1.5549; compound **2**, *n* = 9, bp 153–157° (1 mm), $n^{24.5}D$ 1.5471; compound **2**, *n* = 10, bp 137–140° (0.2 mm), $n^{24.5}D$ 1.5420. All three compounds showed the expected ir and pmr spectra: ir 3605 cm^{-1} (OH); pmr δ 6.5–7.2 (m, 4 H, nuclear protons), 4.9 (s, 1 H, OH), 3.35 (t, 2 H, $-CH_2Br$), 2.4–2.8 (distorted t, 2 H, benzylic CH_2), 1.2–2.1 (m, "central" methylene protons).

Anal. Calcd for $C_{11}H_{15}BrO$, *n* = 8: C, 54.34; H, 6.22; Br, 32.86. Found: C, 54.49; H, 6.41; Br, 32.72. Calcd for $C_{12}H_{17}BrO$, *n* = 9: C, 56.04; H, 6.63; Br, 31.07. Found: C, 56.00; H, 6.60; Br, 31.11. Calcd for $C_{13}H_{19}BrO$, *n* = 10: C, 57.58; H, 7.06; Br, 29.46. Found: C, 57.55; H, 7.16; Br, 29.41.

Cyclization of Compounds 2, *n* = 8, 9, and 10. To 100 ml of DMSO 2.5 ml of 40% (w/w sodium hydroxide was added with stirring. To the vigorously stirred suspension heated to 55° about 8 mmol of **2** in DMSO (50 ml) was added dropwise over 1 hr. Heating and stirring was continued for additional 30 min. When cold, the yellow-brown mixture was diluted with water, made strongly alkaline with sodium hydroxide, then repeatedly extracted with hexane; the combined extracts were washed with water and dried over anhydrous sodium sulfate and the solvent was removed by distillation. Chromatography of the residue on silica gel, eluent $CHCl_3$–light petroleum (1:1), afforded the cyclic ethers **4**, *n* = 8, 9, and 10. The yields are reported in Table I. Analytical samples were obtained after one distillation: 3,4,5,6-tetrahydro-2*H*-1-benzoxocin (**4**, *n* = 8), bp 82° (1.8 mm), $n^{19}D$ 1.5353; 2,3,4,5,6,7-hexahydro-1-benzoxonin (**4**, *n* = 9), bp 101° (1.8 mm), mp 29–31°, $n^{19}D$ 1.5394 (supercooled liquid); 3,4,5,6,7,8-hexahydro-2*H*-1-benzoxecin (**4**, *n* = 10), bp 111° (1.6 mm), $n^{19}D$ 1.5410.

Any phenolic material present in the above alkaline solution was recovered by acidification with concentrated hydrochloric acid, followed by ether extraction. After the usual work-up, elution with chloroform on silica gel yielded the pure alkenylphenols **14**, *n* = 8 and 9. No definite product was isolated for *n* = 10. Yields are reported in Table I. For analytical purposes, the compounds were further purified by distillation: *o*-(4-pentenyl)phenol (**14**, *n* = 8), bp 104° (1.8 mm); *o*-(5-hexenyl)phenol (**14**, *n* = 9), bp 115° (1.5 mm).

Structure assignments for both the cyclic ethers and alkenylphenols were made on the basis of spectral data and elemental analyses: cyclic ethers **4**, *n* = 8, 9, and 10, pmr δ 6.6–7.2 (m, 4 H, nuclear protons), 4.15 for *n* = 9 and 10, 3.95 for *n* = 8 (m, 2 H, $-CH_2O$), 2.7 (m, 2 H, benzylic protons), 1.2–2.0 for *n* = 8 and 9 and 0.8–2.0 for *n* = 10 (m, "central" protons); alkenylphenols **14**, *n* = 8 and 9, ir 3605 (OH), 1640, 990, 915 cm^{-1} ($-CH=CH_2$); pmr δ 6.4–7.1 (m, 4 H, nuclear protons), 5.2–6.1 (m, 1 H, vinylic proton), 4.6–5.1 (m, 2 H, terminal vinylic protons), 5.0 for *n* = 8 and 4.7 for *n* = 9 (1 H, OH, superimposed to terminal olefinic protons), 2.25 (distorted t, 2 H, benzylic protons), 1.2–2.3 (m, allylic and "central" protons).

Anal. Calcd for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found for **4**, *n* = 8: C, 81.31; H, 8.63. Found for **14**, *n* = 8: C, 81.28; H, 8.71. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found for **4**, *n* = 9: C, 81.71; H, 9.14. Calcd for $C_{13}H_{18}O$: C, 82.06; H, 9.53. Found for **4**, *n* = 10: C, 81.87; H, 9.67.

Registry No.—**2** (*n* = 8), 51795-89-2; **2** (*n* = 9), 51795-90-5; **2** (*n* = 10), 51795-91-6; **4** (*n* = 8), 51060-43-6; **4** (*n* = 9), 51795-92-7; **4** (*n* = 10), 51795-93-8; **5**, 6621-59-6; **6**, 51795-94-9; **7**, 51821-14-8; **8**, 51795-95-0; **9**, 51795-96-1; **10** (*n* = 8), 22921-72-8; **10** (*n* = 9), 51795-97-2; **10** (*n* = 10), 51795-98-3; **13** (*n* = 8), 51795-99-4; **13** (*n* = 9), 51796-00-0; **13** (*n* = 10), 51796-01-1; **14** (*n* = 8), 51796-02-2; **14** (*n* = 9), 51796-03-3.

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