716. *Aspects of Stereochemistry. Part XVII.** *Oxetans from Monoarenesulphonyl Esters of* 1,3-*Diols.*

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A study of the formation of oxetans in reactions of mono-*p*-bromobenzenesulphonates of 1,3-diols with potassium t-butoxide in t-butyl alcohol at 26° or 30° is reported.

Earlier work [1] showed that potassium t-butoxide in t-butyl alcohol was an effective base for decomposition of monoarenesulphonates of cyclohexane-1,3-diols. Fission (fragmentation [2]) into an unsaturated carbonyl compound was the usual reaction, but in one case a 1,3-epoxycyclohexane was also formed (3α,5α-epoxycholestane from 5α-hydroxycholestan-3β-yl toluene-*p*-sulphonate in 55% yield). In this exceptional reaction, a decrease in steric compression helps to overcome the usual reluctance for the strained endo-1,3-epoxide structure to be formed. The present paper describes the reactions of potassium t-butoxide with monosulphonyl esters of 1,3-diols that could lead to less strained 1,3-epoxides. Both the formation of epoxides by intramolecular substitution and the fragmentation [2] have fairly precise geometrical requirements, and thus the choice of reaction may be expected to depend on the preferred conformation of the anion of the initial hydroxy-ester. Mono-*p*-bromobenzenesulphonates, that could be purified by crystallisation, were used as starting materials.

Results and Discussion.—Solutions of the mono-*p*-bromobenzenesulphonates in t-butyl alcohol were treated with potassium t-butoxide in the same alcohol at 26° or 30°. The reactions were followed by titration, and rates were compared by comparing the half-lives.

The *cis*-hydroxy-ester (I) gave, on reaction at 26°, a 67% yield of the epoxide (II), presumably the same as that obtained in low yield by Rupe and Klemm [3] on treatment of mixed *cis*- and *trans*-2-hydroxymethylcyclohexanols with acid. The half-life of the *trans*-hydroxy-ester (III) was about forty times that of its *cis*-isomer (I), the main products being polymers showing infrared vinyl absorption but weak carbonyl absorption; these were clearly derived from the alkali-sensitive hept-6-enal (IV), the initial product of fragmentation (cf. the condensation of hex-5-enal under similar conditions [1]). Fragmentation occurs therefore in place of the formation of the very strained *trans*-epoxide. A small amount (14%) of the t-butyl ether (V) was also obtained, a type of side-reaction also observed in the steroid series.[1]

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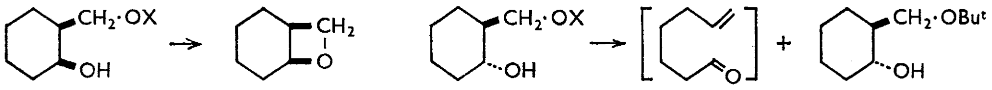
* Part XVI, preceding paper.

[1] Clayton, Henbest, and Smith, *J.*, 1957, 1982.

[2] Cf. Grob in " Theoretical Organic Chemistry (Kekulé Symposium)," Butterworths, 1959, p. 114.

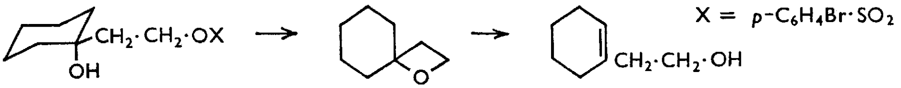
[3] Rupe and Klemm, *Helv. Chim. Acta*, 1938, **21**, 1538.

In various studies[4] of the action of alkaline reagents on 1,3-halogenohydrins it has been observed that higher yields of epoxides are often obtained when R is changed from hydrogen to alkyl in molecules of the type $HO\cdot CR_2\cdot CH_2\cdot CH_2\cdot Hal$ or $HO\cdot CH_2\cdot CR_2\cdot CH_2\cdot Hal$. It seems that substituents cause such effects largely by increasing the likelihood of the molecule's adopting the conformation necessary for cyclisation. The mono-*p*-bromobenzenesulphonates of 1-2′-hydroxyethylcyclohexanol (VI) and the diol (IX; X = H) are related to these types of dialkyl-substituted molecule. In the preferred conformation (VI) of the former compound the anion of the hydroxyl group and the sulphonate group are likely to be correctly placed for an intramolecular displacement. In agreement, the epoxide (VII) (62% isolated) was formed in a relatively rapid reaction [half-life of (VI) about one-twentieth that of the *cis*-compound (I)]. It was helpful to have calcium hydroxide present during the initial distillation of this epoxide as traces of acid (formed, for example, by decomposition of small amounts of unchanged sulphonic ester) isomerised the epoxide to the unsaturated alcohol (VIII). This isomerisation was also carried out separately with boron trifluoride as catalyst (cf. the similar reaction of 3α,5α-epoxycholestane[1]).



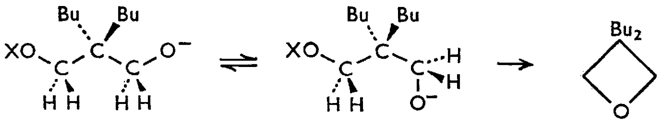
(I) (II) (III) (IV) (V)

$X = p\text{-}C_6H_4Br\cdot SO_2$



(VI) (VII) (VIII)

The compound (IX) also reacted rapidly with potassium t-butoxide, the epoxide (X) being obtained in 72% yield. This shows that the starting material can readily adopt the conformation (IXB) instead of the usual, more extended conformation (IXA). An alternative argument that the epoxide is formed because fragmentation is inhibited appears less likely as recent work[2] on the stereochemistry of fragmentation suggests that either of the conformations (IXA or IXB) could be suitable for the cleavage (to formaldehyde and 2-butylhex-1-ene).



(IXA) (IXB) (X)

Finally, the reactions of the mono-*p*-bromobenzenesulphonates of *meso*- and *rac*.-pentane-2,4-diol (XI) and (XII) were compared. Both compounds underwent fragmentation, propene being obtained in 55—60% yields (as the osmium tetroxide–pyridine adduct). The half-life of the ester (XI) from the *meso*-diol was about twice that of its isomer (XII), a consequence of the more favourable conformation (two methyl groups apart) of the latter compound for fragmentation.

The esters (XI) and (XII) were obtained starting from acetylacetone. Hydrogenation[5]

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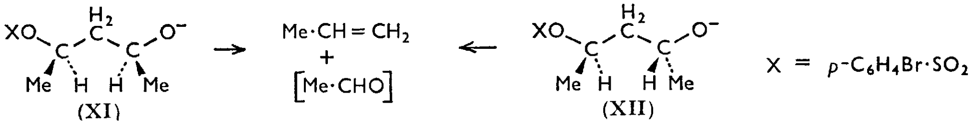
[4] Searles and Gortatowski, *J. Amer. Chem. Soc.*, 1953, **75**, 3030; Farthing, *J.*, 1955, 3648; Forsberg, *Acta Chem. Scand.*, 1954, **8**, 135.

[5] Cf. Reynolds and Kenyon, *J. Amer. Chem. Soc.*, 1950, **72**, 1593.

of this gave pentane-2,4-diol which was converted by reaction with *p*-bromobenzenesulphonyl chloride (1·03 mol.) into a product separated by chromatography into two monoesters, m. p.s 57° and 77—79°, and a diester, m. p. 145—146°, shown to be derived from the lower-melting monoester. The isolation of two monoesters shows that the *meso*- and *rac*-diols are both formed in the hydrogenation of acetylacetone. The configurations of the pentane-2,4-diols have recently been established.[6,7] The diester, m. p. 145—146°, was prepared from a pure sample of the *meso*-diol kindly provided by Dr. J. Dale; the lower-melting monoester is therefore derived from the *meso*-diol. The pentane-2,4-diol mixture from our hydrogenation contained about 75% of the *meso*-compound, as estimated by the isolation of a crystalline borate ester (Dr. J. Dale, personal communication).



(XI) (XII)

The 1,3-diols were prepared by methods given in the literature. The diols used for preparing the esters (VI) and (IX) were obtained crystalline for the first time.

Infrared Spectra.—A general trend in the infrared absorption of cyclic ethers is a movement of the principal C–O–C stretching band to a lower frequency as the size of the ring diminishes and the strain increases. *cis*-Substituted oxirans generally absorb in the 830—890 cm.$^{-1}$ range, and the related oxetans in the 970—980 cm.$^{-1}$ region.[8] The absorption bands for the oxetans prepared in this and earlier work are given below. The frequency values for the last three compounds are lower than normal (approaching the values for oxirans), clearly because of increasing strain in the oxide ring arising from fusion to carbocyclic systems. Similarly, in 1,4-epoxides, the more rigid 5α,8α-epoxy-steroids[9] absorb at a lower frequency (960 cm.$^{-1}$) than less strained compounds, *e.g.*, 1,4-cineole (990 cm.$^{-1}$) and tetrahydrofuran (1076 cm.$^{-1}$).

Principal absorption bands (cm.$^{-1}$) of oxetans (in CS_2) in the 10—12 μ range (strongest band in italic type).

Oxetan	*990*, 905
2-Methyloxetan	*985*, 955 (also 853)
3,3-Dibutyloxetan (X)	*982* (also 830w)
1-Oxaspiro[5,3]nonane (VII)	996, *970*, 992 (also 850w)
cis-7-Oxabicyclo[4,2,0]octane (II)	985, *958*, 930 (also 825w)
3α,5α-Epoxybicyclo[2,2,1]heptan-2β-ol[10]	968, *938* (also 822)
3α,5α-Epoxycholestane[1]	890

EXPERIMENTAL

M. p.s were determined on a Kofler block. P. Spence's alumina (Grade H) was neutralised by treatment with 5% of its weight of 10% aqueous acetic acid before use in chromatography. Light petroleum had b. p. 60—80°. t-Butyl alcohol was treated with sodium and distilled. Other solvents were also dried. Ultraviolet absorptions were determined for cyclohexane solutions. The minimum times of reaction for destruction of the hydroxy-sulphonates were determined beforehand by small-scale experiments in which the consumption of alkali was determined by titration.

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[6] Nagai, Kuribayashi, Shiraki, and Ukita, *J. Polymer Sci.*, 1959, **35**, 295.
[7] Dr. J. Dale (European Research Associates), unpublished work.
[8] Bellamy, " The Infra-red Spectra of Complex Molecules," Methuen & Co. Ltd., 1958, p. 119.
[9] Clayton, Crawshaw, Henbest, Jones, Lovell, and Wood, *J.*, 1953, 2009, 2015.
[10] Henbest and Nicholls, *J.*, 1959, 221.

Preparation of cis- *and* trans-2-*Hydroxymethylcyclohexanols.*—Methyl salicylate (118 g.) in dry methanol (250 c.c.) was hydrogenated over Raney nickel (5 g.) at 150°/125 atm. for 14 hr. The product was carefully fractionated to give almost pure methyl *cis*-2-hydroxycyclohexanecarboxylate (77 g., 63%), b. p. 100·5—102°/12 mm., n_D^{21} 1·467 (lit.,[11] b. p. 105°/14 mm., n_D^{25} 1·4645). The higher-boiling fractions (27·4 g., 22%) consisted mainly of the *trans*-hydroxy-ester, but this compound was better prepared by an adaption of a published method [12] as follows. *trans*-2-Chlorocyclohexanol (24·1 g.) in ethanol (125 c.c.), and potassium cyanide (25 g.) in water (100 c.c.), were mixed and heated under reflux for 4 hr. Potassium hydroxide (35 g.) was added and the heating continued for 13 hr. Ethanol was removed by steam-distillation, and the aqueous solution was washed with ether and then acidified with concentrated sulphuric acid (60 c.c.). Continuous extraction with ether gave material which crystallised from isopropyl ether as prisms (12·4 g., 48%), m. p. 105—110°. Recrystallisation gave *trans*-2-hydroxycyclohexanecarboxylic acid, m. p. 109—111° (lit.,[11] 111°). Methylation with a distilled solution of diazomethane in ether gave the *trans*-hydroxy-ester, m. p. 34—36° (from light petroleum) (lit.,[11] 35—36°).

Reduction of the *cis*-hydroxy-ester (28 g.) with an excess of lithium aluminium hydride in boiling ether followed by isolation of the product by continuous ether-extraction gave the *cis*-diol (6·4 g.), m. p. 45—53° (from isopropyl ether). Further crystallisation gave diol with m. p. 49—51°. The di-*p*-nitrobenzoate had m. p. 133—135° (from aqueous ethanol), and the *bis*-3,5-*dinitrobenzoate* had m. p. 158—159° (from ethanol–benzene) (Found: C, 48·8; H, 3·3. $C_{21}H_{18}N_4O_{12}$ requires C, 48·7; H, 3·5%) (lit., diol,[13] m. p. 49—50°; di-*p*-nitrobenzoate,[12] m. p. 132—134°).

Similar reduction of the *trans*-hydroxy-ester afforded the *trans*-diol in 75% yield, b. p. 100—101°/0·3 mm., n_D^{22} 1·484 (lit.,[13] b. p. 112—130°/0·07 mm.).

Reactions starting from cis-2-*Hydroxymethylcyclohexanol.*—*p*-Bromobenzenesulphonyl chloride (9·7 g., 0·038 mol.) was added to a solution of the diol (5 g., 0·038 mole) in pyridine (45 c.c.) at −30°. After being kept at −30° overnight the mixture was treated with ice (120 g.) and hydrochloric acid (55 c.c.) and extracted with benzene. The material from this extract was chromatographed on deactivated alumina (600 g.). Benzene eluted the *monoester* (I) (9·6 g., 72%), m. p. 47—49° (from isopropyl ether) (Found: C, 44·9; H, 4·8. $C_{13}H_{17}O_4BrS$ requires C, 44·7; H, 4·9%), $\lambda_{max.}$ 2345 Å (ε 18,000).

N-Potassium t-butoxide (20 c.c., 0·02 mole) was added to a solution of the ester (I) (4·4 g., 0·013 mole) in t-butyl alcohol (25 c.c.) at 26°. After 30 min. the mixture was diluted with pentane (150 c.c.) and cooled to −80°, and the mixture of inorganic material and t-butyl alcohol was removed by rapid filtration at 0°. The filtrate was washed with a little water and, after careful removal of pentane, distillation afforded *cis*-7-oxabicyclo[4,2,0]octane (II) (0·96 g., 67%), b. p. 35·5°/7 mm., n_D^{20} 1·465 (Found: C, 75·2; H, 10·6. Calc. for $C_7H_{12}O$: C, 75·0; H, 10·8%) (lit.,[3] b. p. 54°/11 mm.). The infrared spectrum of the distillation residue (40 mg.) indicated the presence of unchanged starting material, and a compound containing vinyl groups (bands at 910 and 3060 cm.$^{-1}$); this is probably a condensation product of hept-5-enal (cf. next experiment).

Reactions starting from trans-2-*Hydroxymethylcyclohexanol.*—*p*-Bromobenzenesulphonyl chloride (22·2 g., 0·087 mole) was allowed to react with the diol (11·3 g., 0·087 mole) in pyridine (100 c.c.) at −30° overnight. Ice (200 g.) and hydrochloric acid (100 c.c.) were added and the product was isolated with ether. Crystallisation from light petroleum–isopropyl ether gave material (22·1 g., 73%), m. p. 70—80°. The pure *monoester* (III) had m. p. 82—84° (Found: C, 44·7; H, 4·9. $C_{13}H_{17}O_4BrS$ requires C, 44·7; H, 4·9%), $\lambda_{max.}$ 2350 Å (ε 17,000).

Treatment of the ester (III) (5·5 g., 0·016 mole) in t-butyl alcohol (20 c.c.) at 30° with N-potassium t-butoxide (30 c.c.) under nitrogen gave a yellow suspension which after 19 hr. was diluted with water and pentane. The latter extract gave a yellow oil (2·2 g.) which was distilled at 0·25 mm. from a little calcium hydroxide. Redistillation gave trans-2-*t-butoxymethylcyclohexanol* (V) (0·38 g., 14%), b. p. 66—67°/0·15 mm., n_D^{23} 1·454 (Found: C, 70·8; H, 11·9. $C_{11}H_{22}O_2$ requires C, 70·9; H, 11·9%). The 3,5-*dinitrobenzoate* had m. p. 80—81·5° (from aqueous ethanol) (Found: C, 56·8; H, 6·2. $C_{18}H_{24}O_7N_2$ requires C, 56·8; H, 6·4%). The next fraction (0·2 g.), b. p. 96—108°/0·15 mm., n_D^{22} 1·476, showed weak carbonyl bands at

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[11] Pascual, Sistare, and Regas, *J.*, 1949, 1943.
[12] Mousseron, Julien, and Winternitz, *Compt. rend.*, 1948, **226**, 1909.
[13] Siegel, *J. Amer. Chem. Soc.*, 1953, **75**, 1317.

1635, 1670, and 1700 cm.$^{-1}$ and vinyl bands at 910 and 3050 cm.$^{-1}$ consistent with a condensation product from hept-5-enal (IV). The orange higher-boiling residue (0·5 g.) showed similar infrared absorption.

Reactions starting from 1-2′-*Hydroxyethylcyclohexanol*.—The diol was prepared by reducing methyl 1-hydroxycyclohexylacetate[14] with an excess of lithium aluminium hydride in boiling ether. Isolation by continuous ether-extraction gave the diol (53%), b. p. 96—97°/0·2 mm., n_D^{21} 1·487, m. p. 21—25° (lit.,[15,16] b. p. 140—145°/10 mm., n_D^{25} 1·4870). The 3,5-*dinitrobenzoate* had m. p. 105—107° (from aqueous ethanol) (Found: C, 53·2; H, 5·4. $C_{15}H_{18}O_7N_2$ requires C, 53·5; H, 5·4%).

The diol (10·2 g., 0·071 mole) was esterified with *p*-bromobenzenesulphonyl chloride (18·1 g., 0·071 mole) in pyridine (80 c.c.), as in the previous experiment. The product precipitated by water was dried and crystallised from isopropyl ether to give the ester (VI) (23·2 g., 90%), m. p. 73—78°. Pure 2-1′-*hydroxycyclohexylethyl* p-*bromobenzenesulphonate* had m. p. 77—78° (Found: C, 46·3; H, 5·0. $C_{14}H_{19}O_4BrS$ requires C, 46·3; H, 5·3%), $\lambda_{max.}$ 2350 Å (ε 17,000).

Solutions of the ester (VI) (2·6 g.) in t-butyl alcohol (20 c.c.) and N-potassium t-butoxide (20 c.c.) were mixed and kept at 26° under nitrogen for 23 hr. Water was added, followed by dilute hydrochloric acid until the solution reached pH 8. The product was isolated with benzene and distilled from a little calcium hydroxide to give 1-*oxaspiro*[5,3]*nonane* (VII) (0·56 g., 62%), b. p. 57°/16 mm., n_D^{23} 1·458 (Found: C, 76·4; H, 11·1. $C_8H_{14}O$ requires C, 76·1; H, 11·2%). From another experiment, when an excess of hydrochloric acid was added to the reaction mixture and the product was distilled without the addition of calcium hydroxide, 1-2′-hydroxyethylcyclohexene (VIII) was obtained, b. p. 101°/17 mm., n_D^{22} 1·487 (Found: C, 76·4; H, 11·5. Calc. for $C_8H_{14}O$: C, 76·1; H, 11·2%), ε_{2040} 4000. The 3,5-dinitrobenzoate had m. p. 85—87° (from aqueous ethanol) (lit.,[17] b. p. 74—75°/2 mm., n_D^{20} 1·4878, for the alcohol; m. p. 78—80° for the 3,5-dinitrobenzoate). Hydrogenation of the unsaturated alcohol in ethanol in the presence of Adams catalyst afforded 2-cyclohexylethanol isolated as its 3,5-dinitrobenzoate, m. p. 71·5—72° (lit.,[18] m. p. 70·5°).

Reactions starting from 2-*Butyl*-2-*hydroxymethylhexan*-1-*ol*.—The diol was prepared in 77% yield by reduction of diethyl di-n-butylmalonate with an excess of lithium aluminium hydride in ether. It had m. p. 113—115°/0·2 mm., n_D^{27} 1·457. Low-temperature crystallisation from pentane gave the *diol*, m. p. 37—40·5° (Found: C, 70·2; H, 13·0. $C_{11}H_{24}O_2$ requires C, 70·2; H, 12·9%). This compound had been obtained [19] earlier as a liquid, b. p. 125—130°/1 mm.

p-Bromobenzenesulphonyl chloride (5·5 g., 0·022 mole) was added in portions to a solution of the diol (3·7 g., 0·02 mole) in pyridine (25 c.c.) at 0°. The solution was kept at 0° overnight, then an excess of hydrochloric acid and ice was added and the product was extracted with carbon tetrachloride and chromatographed on deactivated alumina (200 g.). Elution with light petroleum–benzene (1 : 4) gave the *diester* (1·6 g., 13%), m. p. 111—112° (from benzene) (Found: C, 44·2; H, 4·95. $C_{23}H_{30}O_6Br_2S_2$ requires C, 44·1; H, 4·8%), $\lambda_{max.}$ 2360 Å (ε 35,000). Elution with benzene afforded the *monoester* (IX) (4·1 g., 51%), m. p. 59—61° (from light petroleum) (Found: C, 50·4; H, 6·35. $C_{17}H_{27}O_4BrS$ requires C, 50·1; H, 6·7%), $\lambda_{max.}$ 2360 Å (ε 16,000). A mixture of N-potassium t-butoxide (25 c.c., 0·025 mole) and a solution of the monoester (3·7 g., 0·0091 mole) in t-butyl alcohol (60 c.c.) was kept at 30° for 2 hr. The product was isolated with ether and distilled, to give 3,3-*dibutyloxetan* (X) (1·1 g., 72%), b. 105°/15 mm., n_D^{20} 1·439 (Found: C, 77·9; H, 13·1. $C_{11}H_{22}O$ requires C, 77·6; H, 13·0%).

Reactions starting from Pentane-2,4-*diol*.—Redistilled acetylacetone (68·4 g.) in ethanol (400 c.c.), hydrogenated over Raney nickel (5 g.) at 100°/150 atm., gave pentane-2,4-diol (60·5 g.), b. p. 100°/14 mm., n_D^{20} 1·436. The diol (28·9 g., 0·28 mole) was esterified with *p*-bromobenzenesulphonyl chloride (73 g., 0·29 mole) in pyridine (300 c.c.) at −30° overnight. The product was isolated with benzene and chromatographed on deactivated alumina (3 kg.). Benzene–light petroleum eluted the meso-*diester*, (13·1 g., 9%), m. p. 145—146° (decomp.) (from benzene) (Found: C, 38·0; H, 3·2. $C_{17}H_{18}O_6Br_2S_2$ requires C, 37·7; H, 3·4%). Benzene eluted the *monoester* (XII) of the *rac.*-diol; this (4·5 g., 5%) had m. p. 69—77° (from isopropyl

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[14] Wallach, *Annalen*, 1906, **347**, 328.

[15] Billimoria and Maclagan, *J.*, 1952, 3067.

[16] Mousseron, Jacquier, Mousseron-Canet, and Zagdown, *Bull. Soc. chim. France*, 1952, 1042.

[17] Benkeser, Arnold, Lambert, and Thomas, *J. Amer. Chem. Soc.*, 1955, **77**, 6042.

[18] Huston and Agett, *J. Org. Chem.*, 1941, **6**, 123.

[19] Yale, Pribyl, Braker, Bernstein, and Lott, *J. Amer. Chem. Soc.*, 1950, **72**, 3716.

ether), raised to 77—79° by crystallisation from the same solvent (Found: C, 41·1; H, 4·5. $C_{11}H_{15}O_4BrS$ requires C, 40·9; H, 4·7%). Benzene next eluted the *monoester* (XI) of the *meso*-diol; this (5·6 g., 6%) had m. p. 48—56° (from isopropyl ether) raised to 57° by crystallisation from the same solvent (Found: C, 41·2; H, 4·7%).

Samples of each monoester (0·25 g.) in t-butyl alcohol (5 c.c.) were treated with N-potassium t-butoxide (1·5 c.c.) at 26°. A slow stream of nitrogen was bubbled through the mixture for 4 hr. and into a 1% solution of osmium tetroxide in ether (20 c.c.) and pyridine (1·5 c.c.) at 0°. Brown crystals of the complex, $C_3H_6OsO_4,2C_5H_5N$, separated; these were collected on a sintered-glass filter, washed with ether, and dried *in vacuo*. The yields of propene (as complex) were 59% from the ester (XII) and 56% from the ester (XI). The infrared spectra (in $CHCl_3$) of the complexes were identical with that of an authentic sample of the propene complex.

The t-butyl alcohol solutions became brown during the reactions. On dilution with water and neutralisation with hydrochloric acid, brown resins, largely insoluble in benzene, were precipitated. Similar material was obtained from the self-condensation of acetaldehyde catalysed by potassium t-butoxide.

Samples of each of the monoesters (50 mg.) in t-butyl alcohol (0·5 c.c.) were treated with N-potassium t-butoxide (0·25 c.c.) in a sealed tube at 26° for 1 hr. Vapour-phase chromatography gave a peak with the same retention time as propene. No other peak (apart from that of t-butyl alcohol) was present but this does not rule out the presence of 2,4-dimethyloxetan as this compound may not be easily resolved from the large excess of t-butyl alcohol.

The monoester (0·323 g.) of m. p. 57° was added to *p*-bromobenzenesulphonyl chloride (0·26 g.) in pyridine (2·5 c.c.). After 30 hr. the product was isolated with methylene chloride and crystallised from isopropyl alcohol, to give the *meso*-diester (0·22 g.), m. p. and mixed m. p. 144—145°.

Half-lives of p-*bromobenzenesulphonates* (*solutions in t-butyl alcohol added to solutions of potassium t-butoxide in t-butyl alcohol*).

Ester	Initial concn. of ester (10^{-4} mole/l.)	Initial concn. of $KOBu^t$ (10^{-4} mole/l.)	Temp.	Half-life (±5%) (min.)
(III)	112	396	30°	60
(I)	118	397	30	1·5
(I)	7·73	12·8	26	60
(VI)	7·45	12·5	26	3
(IX)	105	349	30	0·5
(XI)	6·95	12·8	26	6
(XII)	7·0	12·8	26	3

The *meso*-diol (0·104 g., kindly supplied by Dr. J. Dale [7]) in pyridine (2 c.c.) was added to *p*-bromobenzenesulphonyl chloride (0·562 g.) in pyridine (5 c.c.), at 20°. Isolation as before gave the *meso*-diester (1·21 g.), m. p. and mixed m. p. 144—146°.

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