2-Phenylcyclobutane-1-carboxylic acid (VI). (a) To a stirred and refluxing slurry of 5 g. of sodium hydride in 200 ml. of dry xylene was added, over a period of 10 hr., a solution of 6 g. of ethyl 5-bromo-3-phenylvalerate (IV) in 100 ml. of xylene. After refluxing overnight, excess sodium hydride was destroyed with ethanol and then with water. The solvents were concentrated to a small volume, and the residual oil was refluxed with a solution of 2 g. of potassium hydroxide in 50 ml. of 75% ethanol for 4 hr. The solution was concentrated, diluted with 50 ml. of water, acidified, and extracted with ether. The oily acidic residue from the dried ether extracts weighed 1 g. It dissolved in bicarbonate solution and yielded, with benzylthiourea, an *S*-benzylsothiuronium salt, m.p. 150–151°, after recrystallization from ethanol.

Anal. Calcd. for $C_{19}H_{22}N_2O_2S$: C, 66.62; H, 6.48. Found: C, 66.82; H, 6.58.

(b) A small amount of 2-phenylcyclobutane-1,1-dicarboxylic acid was heated in a molecular still at 160–180° (1 mm.) until no further gas evolution was visible, and the oily residue was distilled onto a cold-finger. It gave an *S*-benzylisothiuronium salt, m.p. 144–145°. A mixture of melting point with the salt from method (a) was 147–148°.

The *p-toluidide* was prepared by allowing 0.2 g. of the oily acid to stand in 10 ml. of benzene containing 2 ml. of thionyl chloride at 26° for 3 days, removing the solvent, and mixing the residue with a solution of 0.3 g. of *p*-toluidine in 10 ml. of benzene. Precipitated toluidine hydrochloride was filtered, the benzene solution was washed with 2*N* hydrochloric acid and bicarbonate solution, dried, and evaporated. The crystalline amide (75 mg.) melted at 159–161°. After several passages through aluminum oxide columns (10 cm. × 18 mm.) in benzene-petroleum ether solutions, the melting point became constant at 166–167°.

Anal. Calcd. for $C_{18}H_{19}NO$: C, 81.47; H, 7.22. Found: C, 81.66; H, 7.36.

3-Phenylvaleric acid. This material was prepared from diethyl cinnamamide and ethylmagnesium bromide followed by acid hydrolysis as described by Maxim.[6] Its *S*-benzylisothiuronium salt melted at 161.5–162° after recrystallization from ethanol.

Anal. Calcd. for $C_{19}H_{24}N_2O_2S$: C, 66.24; H, 7.03. Found: C, 66.42; H, 6.86.

A mixture melting point with *S*-benzylisothiuronium 2-phenylcyclobutanecarboxylate obtained by method (a) above (m.p. 150–151°) was 146.5–147°.

Acknowledgment. Valuable discussions concerning the synthetic procedure were held with Dr. C. L. Zirkle of Smith Kline and French Laboratories. Dr. Raymond Bennett carried out the final purification of 2-phenylcyclobutane-1,1-dicarboxylic acid.

CHARLOTTESVILLE, VA.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, COLLEGE OF PHARMACY, UNIVERSITY OF ILLINOIS]

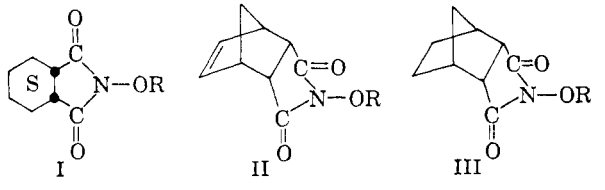
Stereospecific Lossen Rearrangements

LUDWIG BAUER AND STANLEY V. MIARKA

Received March 23, 1959

The Lossen rearrangement of several *cis*-*N*-phenylsulfonyloxy-1,2-cyclohexanedicarboximides yielded corresponding 2-aminocyclohexanecarboxylic acids whose stereochemistry remained *cis*. This method presents a facile route to 2-aminocyclohexanecarboxylic acids of known stereochemistry.

It had been shown previously that the Hofmann, Curtius, and Lossen and Wolff rearrangements at an optically active carbon atom proceeded without inversion.[1] This study was undertaken to test the stereospecificity of the Lossen rearrangement in *cis*-*N*-hydroxycyclohexanedicarboximides. For this purpose, *N*- hydroxy imides of type I, II, and III [R = H] were chosen as their stereochemistry was known.



I II III

These hydroxamic acids were synthesized from the corresponding known anhydrides and aqueous hydroxylamine as described for the preparation of *N*-hydroxyphthalimide.[2] If the hydroxamic acid was not completely precipitated, it was isolated by chloroform extraction and thus excellent yields were obtained. The hydroxamic acids were colorless and afforded colorless anions—in striking contrast to the highly colored anions derived from aromatic *N*-hydroxy imides.[3]

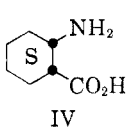
Acylation of the above hydroxamic acids with benzenesulfonyl chloride either in cold aqueous sodium carbonate solution or in chloroform solution in the presence of triethylamine gave the corresponding derivatives [I, II, and III; R = $SO_2C_6H_5$] which were used for this study. It was found that the benzenesulfonyl derivatives readily dis-

(1) (a) L. W. Jones and E. S. Wallis, *J. Am. Chem. Soc.*, **48**, 169 (1926). (b) E. S. Wallis and S. C. Nagel, *J. Am. Chem. Soc.*, **53**, 2787 (1931). (c) E. S. Wallis and R. D. Dripps, *J. Am. Chem. Soc.*, **55**, 1701 (1933). (d) E. S. Wallis and W. W. Moyer, *J. Am. Chem. Soc.*, **55**, 2787 (1933). (e) C. L. Arcus and J. Kenyon, *J. Chem. Soc.*, 916 (1939). (f) J. Kenyon and D. P. Young, *J. Chem. Soc.*, 263 (1941). (g) J. F. Lane and E. S. Wallis, *J. Am. Chem. Soc.*, **63**, 1674 (1941).

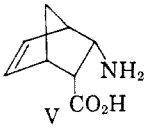
(2) W. R. Orndorff and D. S. Pratt, *Am. Chem. J.*, **47**, 89 (1912).

(3) L. Bauer and S. V. Miarka, *J. Am. Chem. Soc.*, **79**, 1983 (1957).

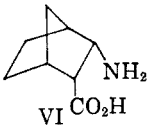
solved in aqueous sodium hydroxide solution with concomitant rearrangement to the benzenesulfonates of the corresponding amino acids (IV, V, and VI). The salts were isolated in good yields:



IV

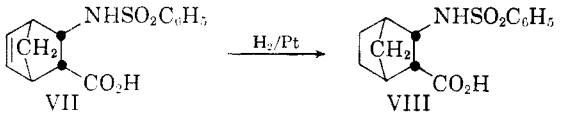


V



VI

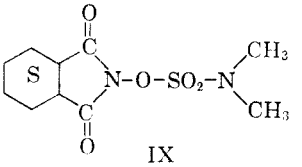
Cis - and *trans* - 2 - aminocyclohexanecarboxylic acids have been described in the literature.[4] The 2-carboxycyclohexylammonium benzenesulfonate (salt of IV) obtained in the Lossen rearrangement of I [R = $SO_2C_6H_5$] was passed through an ion exchange resin of the amine type (as its acetate) and the amino acid isolated from the eluate was the known *cis* amino acid and was further characterized by its known benzoyl derivative.[4] This demonstrated the stereospecificity observed during this rearrangement. In a similar manner, rearrangements of II and III [R = $SO_2C_6H_5$] afforded two new amino acid as their benzenesulfonates. The amino acid (VI) was isolated and characterized by a benzenesulfonyl derivative (VIII):



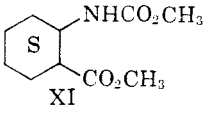
VII VIII

When II was rearranged, the benzenesulfonate of V was obtained and was converted to the benzenesulfonyl derivative (VII). To exclude skeletal changes during the rearrangement of II, the benzenesulfonyl derivative (VII) was hydrogenated over a platinum catalyst and the product found to be identical to VIII.

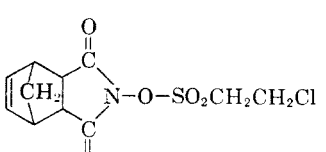
While this work was in progress, the synthesis of I and II [R = H] was reported.[5,6] Kühle and Wegler also reported the Lossen rearrangement in methanol of a number sulfonic esters, specifically, IX and X to the corresponding urethans XI and XII.



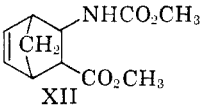
IX



XI



X



XII

(4) S. Hünig and H. Kahanek, *Chem. Ber.*, **86**, 518 (1953).

(5) M. A. Stolberg, W. A. Mosher, and T. Wagner-Jauregg, *J. Am. Chem. Soc.*, **79**, 2615 (1957).

(6) E. Kühle and R. Wegler, *Ann.*, **616**, 183 (1958).

We have found that II [R = $SO_2C_6H_5$] rearranges smoothly in methanol containing triethylamine. This rearrangement in alcohols has been studied by Kühle and Wegler, but they did not establish the stereochemistry of rearrangement in this medium.

EXPERIMENTAL[7]

cis-N-Hydroxyhexahydrophthalimide (I; R = H). This acid was prepared from hexahydrophthalic anhydride and hydroxylamine in aqueous solution as described for the preparation of the other *N*-hydroxy imides.[2] Due to its solubility, the product was isolated by chloroform extraction. It crystallized from ethyl acetate, m.p. 112–113°. Stolberg *et al.*,[5] report m.p. 110–112°. The infrared spectrum showed the OH band at 3100 cm.$^{-1}$ and carbonyl absorption as two strong bands at 1695, 1712, and a medium band at 1790 cm.$^{-1}$

The *benzenesulfonyl derivative* could not be prepared in chloroform solution in the presence of triethylamine. Schotten-Baumann acylation at 25° of the *N*-hydroxy imide (13.5 g.) in 100 ml. of 10% sodium carbonate solution with benzenesulfonyl chloride (10 ml.) afforded the derivative, 14.5 g. (58%). The compound crystallized from methanol (on cooling to −10°) and melted at 77–78°.

Anal. Calcd. for $C_{14}H_{15}NO_5S$: C, 54.33; H, 4.90; N, 4.53. Found: C, 54.40; H, 5.06; N, 4.38.

The carbonyl absorption bands in the infrared spectrum (in Nujol) consisted of a strong band at 1735 and of a medium band at 1795 cm.$^{-1}$

cis-2-Aminocyclohexanecarboxylic acid (IV). *cis-N*-phenylsulfonyloxyhexahydrophthalimide (9.3 g.) was heated with 60 ml. of 10% sodium hydroxide solution on the steam bath for 0.5 hr. The clear solution was acidified with hydrochloric acid and evaporated to dryness *in vacuo*. The solid was extracted with hot ethanol and the extract concentrated to small bulk. Addition of dry ether yielded the crude amino acid as the benzenesulfonate, (7.0 g.) m.p. 165–170° (dec.). Recrystallization from 2-propanol purified the salt and its melting point rose to 183–184°.

Anal. Calcd. for $C_{13}H_{19}NO_5S$: C, 51.80; H, 6.35; N, 4.65. Found: C, 51.76; H, 6.14; N, 4.33.

The infrared spectrum of this salt (in Nujol) showed NH, OH absorption at 3170 and the C=O absorption at 1700 cm.$^{-1}$

When the salt was passed through ion exchange resin IRA-400[8] (in the acetate form), the amino acid was isolated which crystallized from ethanol ether, m.p. 234°. Hünig[4] reported a m.p. of 235°.

The infrared spectrum (in Nujol) showed a weak band at 2120, a broad absorption band between 2600 and 2700 cm.$^{-1}$ typical of amino acids, and a broad band at 1640 cm.$^{-1}$ characteristic of the carboxylate anion.

The *benzoyl derivative* of the amino acid was made by Schotten-Baumann benzoylation. It was purified from ethanol m.p. 175–176°. Hünig[4] quotes the m.p. as 175.5–177°.

Anal. Calcd. for $C_{14}H_{17}NO_3$: C, 67.98; H, 6.92; N, 5.67. Found: C, 68.13; H, 7.06; N, 5.70.

The infrared spectrum showed a sharp medium band at 3380 and two strong carbonyl bands at 1620 (for the amide) and 1695 cm.$^{-1}$ (for the acid).

The *benzenesulfonyl derivative* was prepared and recrystallized from acetic acid, m.p. 160–162°.

Anal. Calcd. for $C_{13}H_{17}NO_4S$: C, 55.09; H, 6.05; N, 4.95. Found: C, 55.52; H, 5.96; N, 5.37.

(7) Analyses quoted in the paper were determined by Drs. Weiler and Strauss, Oxford, England, and Dr. K. Eder, Geneva, Switzerland.

(8) The ion exchange resin was kindly supplied by Rohm & Haas Co., Philadelphia 5, Pa.

Isomerization of the *cis*- to the *trans*-amino acid was accomplished by heating its benzenesulfonate (0.5 g.) with concentrated hydrochloric acid (5 ml.) in a sealed tube at 200° for 6 hr. as directed.[4] The product was converted to the benzoyl derivative, which crystallized from 70% ethanol, m.p. 225–226°. The lit. m.p. is 226–227°.[4]

Anal. Calcd. for $C_{14}H_{17}NO_3$: C, 67.98; H, 6.92; N, 5.67. Found: C, 68.23; H, 7.04; N, 5.54.

The infrared spectrum had similar bands to those in the *cis* compound for the functional groups, but the fingerprint region was decidedly different to that of the *cis* benzoyl derivative.

N-Hydroxy-5-norbornene-endo-2,3-dicarboximide (II: R = H). A solution of hydroxylamine was prepared by adding sodium carbonate (20.5 g.) to an aqueous solution of hydroxylamine hydrochloride (26.3 g. in 60 ml.). 5-Norbornene-*endo*-2,3-dicarboxylic anhydride (50.0 g.) was added to the aqueous solution and the mixture heated between 60–70° for 1 hr. The clear solution was cooled overnight in a refrigerator. The crystals (32.5 g.) were collected and washed twice with 10-ml. portions of ice cold 5*N* hydrochloric acid. The dry product melted at 160–164°. The acidified mother liquor was extracted repeatedly with chloroform (fifteen 30-ml. portions). Evaporation of the chloroform solution afforded another 17.0 g. of II, m.p. 163–164°. The total yield 49.5 g. represented a 91% yield based on the anhydride.

For analysis, II was recrystallized for ethyl acetate. It formed colorless plates, m.p. 165–166°. Lit. m.p. is 165–166°.[5]

Anal. Calcd. for $C_9H_9NO_3$: C, 60.32; H, 5.06; N, 7.82. Found: C, 60.70; H, 5.13; N, 7.70.

The infrared spectrum (in Nujol) showed the C=O absorption with strong bands at 1695, 1710, and 1770 while the OH band appeared at 3100 cm.$^{-1}$ Similar bands were reported by Stolberg *et al.*[5]

Acetyl derivative. The *N*-hydroxy imide (5.0 g.) was refluxed wth acetic anhydride (25 ml.) for 10 min. The solvent was removed *in vacuo*. The residue was recrystallized from ethanol and the product melted at 112–113°. It weighed 5.2 g. (84%). Further recrystallization from ethanol raised the m.p. to 113–114°.

Anal. Calcd. for $C_{11}H_{11}NO_4$: C, 59.71; H, 5.02; N, 6.33. Found: C, 60.04; H, 5.28; N, 6.36.

The carbonyl absorption in the infrared spectrum was in the form of a triplet at 1730, 1770, and 1815 cm.$^{-1}$

Benzoyl derivative. The *N*-hydroxy imide was benzoylated in ice cold aqueous sodium hydroxide solution at *p*H 8. The derivative was crystallized from 2-propanol or benzene and formed colorless cubes, m.p. 143–144°.

Anal. Calcd. for $C_{16}H_{13}NO_4$: C, 67.82; H, 4.62; N, 4.94. Found: C, 68.36; H, 4.63; N, 5.25.

The infrared spectrum (in Nujol) exhibited two strong bands in the carbonyl region, at 1738, 1770, and a weak band at 1793 cm.$^{-1}$

Benzenesulfonyl derivative. (a) *Preparation by the Schotten-Baumann method.* The *N*-hydroxy imide (17.9 g.) was dissolved in 10% sodium carbonate solution (80 ml.) at 25°. The solution was stirred while benzenesulfonyl chloride (13.0 ml.) was added dropwise. The derivative crystallized out almost immediately and was filtered off after 30 min. The material weighed 31.5 g. (almost quantitative yield) and melted at 126–127°. Further crystallization from ethanol raised the melting to 129–130°.

Anal. Calcd. for $C_{15}H_{13}NO_5S$: C, 56.44; H, 4.10; N, 4.39. Found: C, 56.85; H, 4.06; N, 4.36.

The infrared spectrum (in Nujol) had two bands in the carbonyl region, a strong one at 1745 and a medium one at 1800 cm.$^{-1}$

(b) *Preparation in chloroform.* To a cooled solution of the *N*-hydroxy imide (18.2 g.) and triethylamine (48 ml.) in chloroform (150 ml.) was added benzenesulfonyl chloride (15 ml.). The mixture was allowed to stand for 0.5 hr. Solvents were distilled *in vacuo* and the residue recrystallized from ethanol. The derivative was recrystallized from ethanol. It weighed 30 g. and melted at 126°.

Rearrangement of N-phenylsulfonyloxy-5-norbornene-endo-2,3-dicarboximide. The benzenesulfonyl derivative (3.2 g.) was heated with aqueous 10% sodium hydroxide (20 ml.) on the steam bath until a clear solution was obtained (5 min.) The solution was cooled and acidified with hydrochloric acid and then evaporated to dryness *in vacuo*. The dry residue was extracted with boiling ethanol (three 25-ml. portions). The ethanol extract was concentrated to a small bulk *in vacuo* and diluted with dry ether. The salt (IV) (2.3 g. or 73% based on III) was collected. When 9.6 g. were rearranged with 60 ml. of 10% sodium hydroxide solution, 6.1 g. of the salt was obtained. It melted at 195–200° (dec.). Recrystallization from a very small volume of ethanol raised the m.p. to 200–205° (dec.). Repeated recrystallization from ethanol ether did not raise the melting point, but did not remove a small quantity of inorganic salts which persisted through a number of crystallizations. Other attempts to free the salt from that trace of inorganic salts failed. The amino acid was hence characterized by two derivatives.

The infrared spectrum of the salt (in Nujol) showed NH absorption at 3130 and the carbonyl band of the COOH group as a strong band at 1705 cm.$^{-1}$

The *benzenesulfonyl derivative* of the amino acid was made by the Schotten-Baumann technique. It crystallized from ethyl acetate and melted at 168°.

Anal. Calcd. for $C_{14}H_{15}NO_4S$: C, 57.31; H, 5.16; N, 4.77; S, 10.6. Found: C, 57.76; H, 4.90; N, 4.85; S, 10.9.

The infrared spectrum of the derivative showed a band at 3280 cm.$^{-1}$ (NH and OH absorption) and at 1680 cm.$^{-1}$ (C=O of the CO_2H group).

The *benzoyl derivative* of the amino acid was prepared by the Schotten-Baumann method. It crystallized from ethanol and melted at 198–199° (dec.).

Anal. Calcd. for $C_{15}H_{15}NO_3$: C, 70.03; H, 5.88; N, 5.45. Found: C, 69.90; H, 5.95; N, 5.14.

The infrared spectrum had bands at 3280 (due to NH and OH), 1695 (C=O of the acid) and 1625 cm.$^{-1}$ (C=O of the amide).

N-Hydroxynorbornane-endo-2,3-dicarboximide (III, R = H). An aqueous solution of hydroxylamine hydrochloride (5.3 g. in 100 ml.) was treated with sodium carbonate (4.5 g.) and then norbornane-*endo*-2,3-dicarboxylic anhydride (10.0 g.) was added. The mixture was heated at 100° for 1 hr., and acidified with hydrochloric acid and cooled. The hydroxamic acid was filtered off and dried. It weighed 9.0 g. (82%), m.p. 146–147°. Recrystallization from benzene or chloroform raised the m.p. to 148–149°.

Anal. Calcd. for $C_9H_{11}NO_3$: C, 59.60; H, 6.12; N, 7.73. Found: C, 59.66; H, 6.21; N, 7.70.

The infrared spectrum showed the OH band at 3170 and two strong bands in the carbonyl region at 1690 and 1760 cm.$^{-1}$

The *benzenesulfonyl derivative* was prepared (95% yield) in chloroform solution as described above. It was crystallized from ethanol and melted at 133–134°.

Anal. Calcd. for $C_{15}H_{15}NO_5S$: C, 56.10; H, 4.71; N, 4.36. Found: C, 56.32; H, 4.80; N, 4.44.

The infrared spectrum (in Nujol) showed a strong band at 1745 and a weak one at 1800 cm.$^{-1}$

endo-2-Amino-3-norbornanecarboxylic acid (VI). The benzenesulfonyl derivative prepared above (3.2 g.) was rearranged in 20 ml. of 10% aqueous sodium hydroxide solution at 100° for 1 hr. The solution was acidified and evaporated to dryness and the residue extracted with hot ethanol. The alcohol solution was concentrated and the benzenesulfonate of the amino acid crystallized. Recrystallization was carried out from ethanol, and the m.p. of the salt was 232° (dec.).

Anal. Calcd. for $C_{14}H_{19}NO_5S$: C, 53.65; H, 6.12; N, 4.47. Found: C, 53.29; H, 6.21; N, 4.56.

The infrared spectrum of the amino acid salt (in Nujol) showed a strong band at 3190 (OH and NH bands), three weak bands at 2520, 2600, and 2750 (ammonium ion) and a sharp strong band at 1715 cm.$^{-1}$ (C=O of CO_2H group).

An aqueous solution of the benzenesulfonic acid salt (0.52 g. in 100 ml. water) was passed through a column of ion exchange resin (IRA 400 in the acetate form). Evaporation of the eluate afforded the amino acid (0.2 g.), m.p. 265°. Recrystallization from ethanol ether raised the m.p. to 266°.

Anal. Calcd. for $C_8H_{13}NO_2$: C, 61.90; H, 8.44; N, 9.03. Found: C, 61.98; H, 8.43; N, 8.99.

The *benzenesulfonyl derivative* of the amino acid was prepared by the Schotten-Baumann method and after crystallization from ethanol melted at 173–174°.

Anal. Calcd. for $C_{14}H_{17}NO_4S$: C, 56.94; H, 5.81; N, 4.74. Found: C, 56.74; H, 5.75; N, 5.18.

The infrared spectrum (in Nujol) had a sharp band at 3320 (NH, OH absorption) and at 1682 cm.$^{-1}$ (C=O of CO_2H).

This derivative was also formed when *endo*-2-benzenesulfonamido-3-carboxy-5-norbornene (VII) was hydrogenated over platinum in ethanol. The derivative of the reduced product melted at 173–174°, did not show any melting point depression, and had an identical infrared spectrum as the compound prepared from III above.

Acknowledgment. The authors thank the Research Corp. for a Frederick Gardner Cottrell grant which made this work possible. We would also like to express our appreciation to Mr. Ronald Wilbois for technical help, to Messrs. James Brader and Paul McMahon for determining the infrared spectra. The National Aniline Division of the Allied Chemical and Dye Corp. is also thanked for their generous supply of the anhydrides used.

833 S. Wood St.
Chicago 12, Ill.

[Contribution from the Department of Chemistry of the University of Michigan]

Reactions of the Mono-*p*-toluenesulfonic Acid Ester of Yohimbyl Alcohol[1,2]

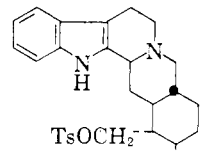
Robert C. Elderfield, Allan E. Hydorn, Erhard Schenker, and Kenneth K. Wyckoff

Received March 24, 1959

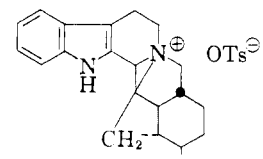
Yohimbyl alcohol mono-*p*-toluenesulfonate alkylates representative alcohols and amines with the formation of ethers of yohimbyl alcohol and 16-aminomethyl derivatives of yohimbol, respectively. Acid hydrolysis of yohimbyl alcohol mono-*p*-toluenesulfonate regenerates yohimbyl alcohol. On the other hand, attempted basic hydrolysis of the ester resulted in intramolecular alkylation of the C-17 hydroxyl group with the formation of an oxetane.

The mono-*p*-toluenesulfonic acid ester (tosyl ester) of yohimbyl alcohol (I) was first prepared by one of us[3] by unimolecular tosylation of yohimbyl alcohol for use as a model compound in connection with projected degradation studies on the alkaloid alstonine. Subsequent investigations required the preparation of fairly large amounts of the ester during the purification of which an interesting, if not entirely unpredictable behavior, was noted. In the present communication, we wish to present certain observations on the behavior of I under a variety of conditions.

At the outset it was noted that, whereas small amounts of I could be recrystallized without difficulty from ethanol, when similar recrystallization of larger amounts of the ester (m.p. 147°) was attempted, a high melting substance (279°) resulted. The infrared spectrum of this high melting compound showed absorption bands at 8.56, 8.95, 9.71, and 9.94μ characteristic of those ascribed to the *p*-toluenesulfonate anion and reminiscent of the absorption displayed by the product of the action of tosyl chloride on reserpinol in pyridine.[4,5]



I



II

Subsequent investigation showed that a substance of similar high melting point was more readily obtained by refluxing I in the higher boiling isoamyl alcohol. After recrystallization from ethanol, material thus prepared furnished analytical data in approximate agreement with those demanded by an internal alkylation product of the type of II solvated by one molecule of ethanol. Subsequent experiments were done with material prepared in this manner. In order to avoid solvation, subsequent batches were recrystallized from acetone.

However, there are obvious difficulties in such a simple interpretation of the formation of the high

(1) This work was supported in part by a Research Grant (H-1733) from the National Heart Institute and in part by a Research Grant (CY-2961) from the National Cancer Institute.

(2) Portions of the work here presented are taken from a dissertation submitted by Kenneth K. Wyckoff in partial fulfillment of requirements for the degree of Doctor of Philosophy in the University of Michigan.

(3) R. C. Elderfield and A. P. Gray, *J. Org. Chem.*, **16**, 506 (1951).

(4) P. A. Diassi, F. L. Weisenborn, C. M. Dylion, and O. Wintersteiner, *J. Am. Chem. Soc.*, **77**, 4687 (1955).

(5) E. E. van Tamelen and P. D. Hance, *J. Am. Chem. Soc.*, **77**, 4692 (1955).