[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF NEW HAMPSHIRE]

Heterocycles of Bivalent and Quadrivalent Tin

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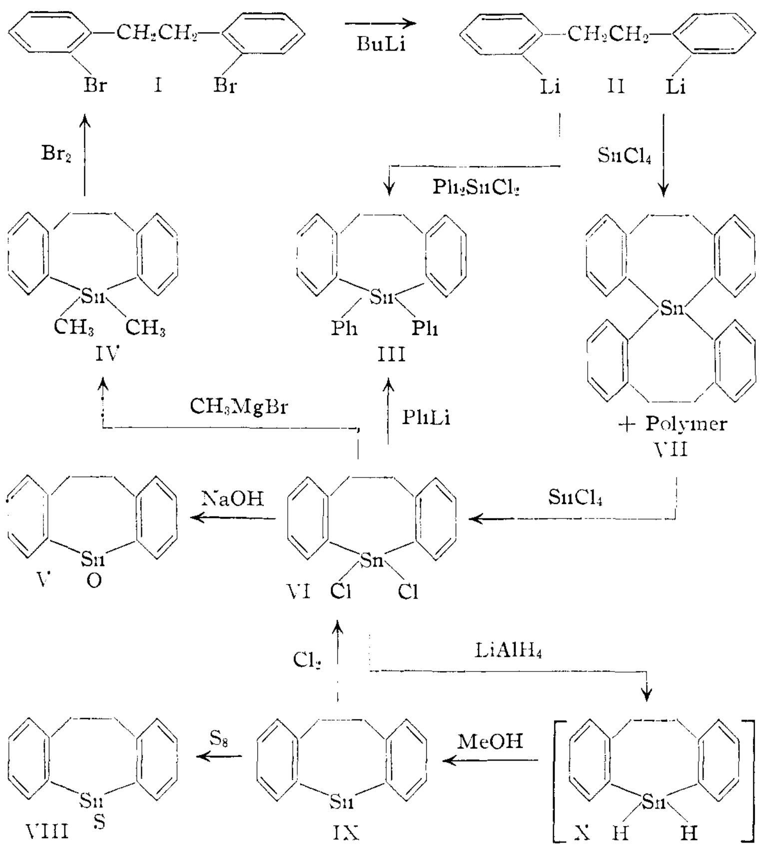
Several heterocycles in the dibenzocycloheptadiene series containing tin in the seven-membered ring have been prepared. Ring closure was effected through the reaction of *o,o'*-dilithiobibenzyl with diphenyltin dichloride or with stannic chloride. The first heterocycle containing bivalent tin in the ring is among the compounds described.

Although the literature of organometallic compounds of group IV-A is quite extensive, that dealing with heterocycles of these elements is limited. In the case of tin, for example, only two heterocyles have been reported. These are the pentamethylenedimethyltin and pentamethylenediethyltin prepared by Krause and his co-workers.[1] The method of synthesis involved the reaction of pentamethylenedimagnesium bromide with the appropriate dialkyltin dibromide.



This paper describes our first efforts to extend this area in organotin chemistry. We set out to prepare some members of the dibenzocycloheptadiene system for two main reasons. First, no tin heterocycles with rings of this size had been prepared. Second, it seemed reasonable to expect that both quadrivalent and bivalent tin could be introduced into the ring if the tin were attached to the two aromatic rings (see structures VI and IX in the chart, for example). The presence of the aromatic rings was desirable because it was known that diphenyltin hydride decomposes with hydrogen evolution and production of diphenyltin.[2,3] It was thought that compound X would behave in similar fashion. The analogous reaction for dialkyltin hydrides has not been reported.

The common starting material in our syntheses of tin heterocycles was *o,o'*-dilithiobibenzyl (II). In the first attempts at ring closure, this compound was allowed to react with diphenyltin dichloride. The main product was a polymeric material which was insoluble and very high melting; it was not examined in any detail. In addition, a monomeric product, 5,5-diphenyl-10,11-dihydrodibenzo[b,f]stanniepin (III) was obtained in yields amounting to only 1–2%.[4] It is dimorphic. One modification, m.p. 136–137°, is obtained upon crystallization from 60–90° ligroin. The other, m.p. 146–147°, is obtained upon crystallization from ethanol or by heating the first modification at its melting point for a few minutes.

Because of the low yields obtained in this direct ring closure, another approach was attempted. The *o,o'*-dilithiobibenzyl was allowed to react with a half-molar equivalent of stannic chloride. This reaction was expected to yield a mixture of the spiro compound VII and polymeric products, in which the chlorine atoms on the tin are replaced by the *o*-phenyl groups of bibenzyl. This mixture was isolated, but not purified, and heated with a molar equivalent of stannic chloride. Now, it is well known that stannic chloride reacts with compounds of the type R_4Sn to produce mixtures of R_3SnCl and R_2SnCl_2.[5] In the present case it seemed reasonable to suppose that 5,5-dichloro-10,11-dihydrodibenzo[b,f]stanniepin (VI) would be the lowest boiling of any of the products of the reaction. This appears to be borne out by the fact that 35% yields of the dichloro compound

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[1] G. Grüttner, E. Krause and M. Wiernik, *Ber.*, **50**, 1549 (1917).

[2] R. F. Chambers and P. G. Scherer, THIS JOURNAL, **48**, 1060 (1926).

[3] It should be possible to prepare heterocycles of bivalent tin by reaction of a polymethylenedimagnesium halide with stannous chloride, but this was not attempted in the present investigation.

[4] Since both bicovalent and quadricovalent tin heterocycles are described in this paper, the nomenclature used must distinguish between these two kinds of tin. We have chosen to indicate this distinction with the classical *ic* and *-ous* suffixes. A *stanniepin* will contain quadrivalent tin in the ring, and a *stannoepin* will contain bivalent tin in the ring.

[5] K. A. Kocheshkov, *Ber.*, **62**, 996 (1929); K. A. Kocheshkov and M. M. Nad, *ibid.*, **67**, 717 (1934); K. A. Kocheshkov, M. M. Nad and A. P. Alexandrov, *ibid.*, **64**, 628 (1931).

were obtained when the reaction mixture was distilled slowly. The observations that the molecular weight and elemental analysis of this compound agree with those for structure VI are taken as evidence that it, and each of the other compounds described below, is cyclic.

The dichloride reacted readily with phenyllithium, yielding the diphenyl compound III in 83% yield. Thus, the over-all yield of this compound from *o,o'*-dibromobibenzyl is improved to 29%.

Reaction of the dichloride with methylmagnesium bromide produced 5,5-dimethyl-10,11-dihydrodibenzo[b,f]stanniepin (IV), a liquid at room temperature, in 73% yield. Upon treatment of this compound with two moles of bromine, rapid reaction ensued. A high yield of the expected *o,o'*-dibromobibenzyl (I) was isolated from the reaction mixture. The other expected product, dimethyltin dibromide, was not isolated.

An essentially quantitative yield of 5-oxo-10,11-dihydrodibenzo[b,f]stanniepin (V) was obtained by treating the dichloride with sodium hydroxide. This compound, like dialkyl- and diaryltin oxides, is infusible and insoluble.

The bicovalent tin heterocycle 10,11-dihydrodibenzo[b,f]stannoepin (IX) has been made using a recently developed reaction sequence.[6] The dichloride reacted readily with lithium aluminum hydride to produce, presumably, 10,11-dihydrodibenzo[b,f]stanniepin (X).[7] Dilution of the ether solution of this compound with methanol resulted in the slow evolution of hydrogen and precipitation of the analytically pure stannous heterocycle in 70–90% yields. It varies in color from colorless to yellow. Generally the first-formed material is colorless and melts around 60°. Those fractions which appear later are yellow and have higher and higher melting points, ranging up to 160°. They are also less soluble in methylene chloride, indicating varying degrees of polymerization. Diphenyltin is known to exist in several modifications which differ in molecular weight, solubility, color and crystalline form, as well as reactivity.[2,8] The structure of the stannoepin is confirmed by the fact that it was converted, by reaction with chlorine, to the dichloride from which it was prepared. It also reacted with sulfur to produce 5-thio-10,11-dihydrodibenzo[b,f]stanniepin (VIII), in 85% yield. However, in spite of repeated attempts at purification, this compound was not obtained analytically pure.

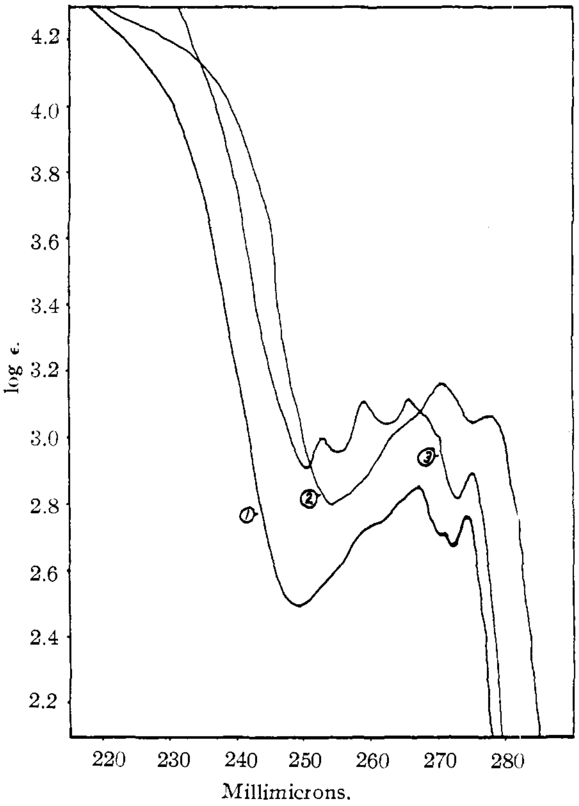

Fig. 1.—Ultraviolet absorption spectra of: 1, 5,5-dimethyl-10,11-dihydrodibenzo[b,f]stanniepin (IV); 2, 5,5-dichloro-10,11-dihydrodibenzo[b,f]stanniepin (VI); 3, 5,5-diphenyl-10,11-dihydrodibenzo[b,*f*]stanniepin.

The ultraviolet absorption spectra of compounds III, IV and VI are shown in Fig. 1. It can be seen that replacement of the two methyl groups in IV by the two chlorines in VI does not alter the shape of the curve appreciably. However, the position of the longest wave length maximum is shifted from 273.5 to 278.0 mμ; and the extinction coefficients at the maxima are increased by a factor of about two. These changes may reasonably be attributed to a greater electron deficiency on the tin when attached to chlorine. This would be expected to lower the energy of the polar excited state, thus leading to the bathochromic shift. An increase in intensity of absorption suggests that this excited state is relatively more polar than that in the dimethyl derivative.

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Experimental

Tin analyses were carried out by the method of Gilman and Rosenberg.[9] Other analyses were performed by the Galbraith Laboratories, Knoxville, Tenn. Melting points are uncorrected.

***o,o'*-Dibromobibenzyl (I).**—*o*-Bromobenzyl bromide was prepared according to the procedure of Letsinger and Skoog[10] with the exception that a 150 watt incandescent bulb was used for illumination instead of a mercury arc lamp. A 74% yield of *o*-bromobenzyl bromide, b.p. 127–133° (15 mm.) was obtained.

o,o'-Dibromobibenzyl was prepared by coupling *o*-bromobenzyl bromide according to the method of Letsinger and Skoog.[10]

5,5-Diphenyl-10,11-dihydrodibenzo[b,f]stanniepin (III). A.—To 200 ml. of 0.345 M butyllithium in ether, cooled in an ice–water-bath, was added 78 g. (0.031 mole) of *o,o'*-dibromobibenzyl in 100 ml. of ether over about an hour.

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[6] In these laboratories by A. K. Sawyer.

[7] G. J. M. van der Kerk, J. G. Noltes and J. G. A. Luijten, *J. Appl. Chem.*, **7**, 366 (1957).

[8] E. Krause and R. Becker, *Ber.*, **53**, 173 (1920); K. A. Jensen and N. Clauson-Kaas, *Z. anorg. allgem. Chem.*, **250**, 277 (1943). Unpublished observations in these laboratories.

[9] H. Gilman and S. D. Rosenberg, THIS JOURNAL, **75**, 3592 (1953).

[10] R. L. Letsinger and I. H. Skoog, *ibid.*, **77**, 5176 (1955).

(A nitrogen atmosphere was maintained throughout the reaction.) The ice-bath was then removed and the mixture was stirred for 1.5 hours, and 11.6 g. (0.039 mole) of diphenyltin dichloride dissolved in 100 ml. of ether was then added dropwise over 0.5 hour. After stirring for an hour at room temperature, the reaction mixture was heated under reflux for two hours and then allowed to stand overnight.

The white solid present in the reaction mixture was removed by filtration and digested with boiling water. There remained 3 g. of a colorless, insoluble, and infusible solid which was discarded. The ethereal filtrate was concentrated by passing a stream of air over it. A precipitate gradually appeared as concentration progressed. When the liquid volume reached 10 ml., 4.4 g. of this material had been obtained. This solid was recrystallized repeatedly from ethanol and ligroin. If ethanol was the last solvent used the product had m.p. 146–147°. When this was recrystallized from ligroin it melted at 136–137°. When this lower melting product was heated above its melting point for a few minutes it resolidified and then melted at 146–147°. The yield of pure product amounted to about 2%.

Anal. Calcd. for $C_{26}H_{22}Sn$: C, 68.91; H, 4.91; Sn, 26.19. Found: C, 68.48; H, 5.03; Sn, 25.90.

B.—5,5-Dichloro-10,11-dihydrodibenzo[b,f]stanniepin (VI) (1 g., 0.0027 mole) was dissolved in 25 ml. of ether and added dropwise to 0.0068 mole of phenyllithium in 25 ml. of ether. The reaction mixture was heated under reflux for two hours, then hydrolyzed with 30 ml. of water. The ether layer was evaporated to dryness and taken up in 10 ml. of 60–90° ligroin, treated with Norit, filtered and cooled. The yield was 0.94 g. (82.5%) of crystals, m.p. 145–146°, undepressed upon admixture with those described above. Furthermore the two samples showed identical infrared spectra.

5,5-Dichloro-10,11-dihydrodibenzo[b,f]stanniepin (VI).—*o,o'*-Dibromobibenzyl (25.2 g., 0.074 mole) dissolved in 200 ml. of diethyl ether was added dropwise to 0.50 mole of butyllithium in 200 ml. of ether cooled in an ice-bath. After the addition was complete, the flask was heated under reflux for an hour. It was then cooled in an ice-bath, and 9.6 g. (0.037 mole) of anhydrous stannic chloride in 10 ml. of benzene was added dropwise. Again the solution was heated under reflux for two hours. After cooling, the solution was hydrolyzed with distilled water. The ether layer was dried over calcium chloride and then evaporated until nothing could be removed at steam-bath temperature and aspirator vacuum.

Stannic chloride (9.6 g., 0.037 mole) was again added, the mixture heated on a steam-bath for a half-hour and then placed in a bath at 150° for three hours.

The contents of the flask were then distilled under vacuum. The fraction boiling from 175–190° (0.4 mm.) was collected; it was light yellow and crystallized in the receiver. This product was washed with 5 ml. of petroleum ether (40–60°) which removed the color. It was then dried and dissolved in 50 ml. of ether and filtered through a bed of decolorizing charcoal. The product was recrystallized by allowing the ether to evaporate at room temperature to near dryness. After two recrystallizations 9.8 g. (35.7%) of crystalline solid was obtained, m.p. 106–106.5°.

Anal. Calcd. for $C_{14}H_{12}Cl_2Sn$: C, 45.46; H, 3.27; Cl, 19.17; Sn, 32.09; mol. wt., 369.9. Found: C, 45.39; H, 3.39; Cl, 19.1; Sn, 31.39; mol. wt., 380 (isopiestic in CH_2Cl_2).

5,5-Dimethyl-10,11-dihydrodibenzo[b,f]stanniepin (IV).—Methylmagnesium bromide was prepared from 0.786 g. (0.0324 mole) of magnesium in 200 ml. of ether. To this was added a solution of 3 g. (0.0081 mole) of 5,5-dichloro-10,11-dihydrodibenzo[b,f]stanniepin in 50 ml. of dry ether. The mixture was stirred at room temperature for two hours. It was then hydrolyzed with 50 ml. of 1% hydrochloric acid solution, the two layers were separated and the ether layer was dried over magnesium sulfate. The mixture was filtered and the ether distilled off on a steam-bath, leaving a colorless oily residue. The oil obtained was distilled under vacuum and the fraction boiling 130–135° (0.2 mm.), n^{25}_{D} 1.6130, was collected. The yield was 73%, 1.95 g.

Anal. Calcd. for $C_{16}H_{18}Sn$: C, 58.36; H, 5.47; Sn, 36.08. Found: C, 58.26; H, 5.51; Sn, 35.63.

Cleavage of 5,5-Dimethyl-10,11-dihydrodibenzo[b,f]-stanniepin (IV) with Bromine.—5,5-Dimethyl-10,11-dihydrodibenzo[b,f]stanniepin (1.016 g., 0.00309 mole) was dissolved in 10 ml. of CCl_4. Bromine (0.989 g., 0.00618 mole) dissolved in 10 ml. of CCl_4 was added dropwise. The solution was decolorized immediately.

Removal of the solvent left a colorless crystalline residue which was dissolved in 25 ml. of ether and extracted with four 10-ml. samples of distilled water. The ether layer was treated with decolorizing charcoal and filtered. The filtrate was evaporated and 0.978 g. (93% yield) of *o,o'*-dibromobibenzyl (I), m.p. 77–81°, was obtained. Recrystallization from 60–90° ligroin gave 0.79 g. (75%) of product melting 81–83°. Mixture melting point with an authentic sample gave no depression; reported melting point 84°.

10,11-Dihydrodibenzo[b,f]stannoepin (IX).—Finely divided lithium aluminum hydride (0.394 g., 0.01 mole, 100% excess), was suspended in 20 ml. of dry ether and cooled in an ice-bath. Then solid 5,5-dichloro-10,11-dihydrodibenzo[b,f]stanniepin (3.70 g., 0.01 mole), was added in five approximately equal portions at 5 minute intervals. After the addition was complete the mixture was stirred for a half-hour.

A mixture of 1 ml. of methanol and 1 ml. of ether was added dropwise very slowly, followed by the addition of 10 ml. of distilled water. It is important that this hydrolysis be done slowly so that the temperature does not rise to room temperature. Any warming will cause excessive polymerization of the product as revealed by a yellow color and low solubility.

The mixture was stirred for an additional 10 minutes. The ether layer was washed three times with 10-ml. portions of ice-cold water, and then filtered into 50 ml. of methanol and allowed to stand at room temperature in the dark, under nitrogen.

After standing for from one to three hours a precipitate began to form and gas began to evolve. At first the precipitate was a sticky white semi-solid and later, a pale yellow solid. It was collected in several fractions and dried in a vacuum desiccator. All fractions turned solid and the fractions which precipitated first were more soluble in organic solvents than the later, light yellow fractions. The yield was 2.69 g. or 90%.

Melting points were taken of one typical series of products. The following results were obtained; the temperatures listed are the lowest temperatures which would give complete melting on immersion in a pre-heated bath.

Fraction	Weight, g.	M.p., °C.
1	1.222	60
2	0.717	71
3	.344	82
4	.104	86
5	.230	160

Anal. Calcd. for $C_{14}H_{12}Sn$: C, 56.26; H, 4.02; Sn, 39.72. Found: C, 56.39; H, 4.12; Sn, 39.50.

5,5-Dichloro-10,11-dihydrodibenzo(b,f)stanniepin (VI).—10,11-Dihydrodibenzo(b,f)stannoepin (2.2 g., 0.0074 mole) was dissolved in 15 ml. of methylene chloride. Chlorine gas was bubbled in slowly until the white precipitate which formed at first completely disappeared. Ligroin (60–90°, 5 ml.) was added and the methylene chloride was allowed to evaporate; yield 2.2 g. (81%), m.p. 106–107°. Mixture melting point with a sample made by the previously described method gave no depression.

5-Thio-10,11-dihydrodibenzo[b,f]stanniepin (VIII).—Flowers of sulfur (0.18 g., 0.057 mole), was placed in 50 ml. of toluene and heated under reflux until the sulfur dissolved. The solution was filtered and 1.5 g. (0.0445 mole) of 10,11-dihydrodibenzo(b,f)stannoepin was added as a finely divided solid. The flask was stoppered tightly and allowed to stand overnight.

The following day there were colorless crystals and a small amount of brown powdery material in the reaction mixture. It was then evaporated to dryness and the sulfide recrystallized repeatedly from methylene chloride; yield 1.15 g. (69%), m.p. 274–275°.

Anal. Calcd. for $C_{14}H_{12}SSn$: C, 50.81; H, 3.63; S, 9.69; Sn, 35.86. Found: Preparation 1: C, 48.80; H, 3.99; S, 9.02. Preparation 2: C, 48.58, 48.45; H, 3.74, 3.65; S, 9.60; Sn, 34.13, 34.12.

5-Oxo-10,11-dihydrodibenzo[b,f]stanniepin (V).—5,5-Dichloro-10,11-dihydrodibenzo[b,f]stanniepin (1.5 g., 0.0405 mole) was dissolved in 10 ml. of 1,4-dioxane and 0.0648 (0.0162 mole) of NaOH dissolved in 10 ml. of 50% ethanol–water mixture was added. A precipitate formed immediately. The mixture was placed on a steam-bath for an hour. It was then filtered and washed repeatedly with distilled water. The solid was then dried and digested in boiling water for an hour. Again it was filtered and washed, then dried overnight in a drying pistol; yield 1.1 g. (86.5%).

Anal. Calcd. for $C_{14}H_{12}OSn$: C, 53.41; H, 3.81; Sn, 37.70. Found: C, 53.35; H, 3.88; Sn, 37.66.

Spectra.—Ultraviolet spectra were measured with a Beckman model DU spectrophotometer; isoöctane was the solvent. Infrared spectra of all compounds but X were measured and will appear in the doctoral dissertation of O. F. Beumel, Jr.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE UNIVERSITY OF WISCONSIN]

The Effect of Solvent on Spectra. I. A New Empirical Measure of Solvent Polarity: Z-Values

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The position of the charge-transfer (c.-t.) absorption band of 1-alkylpyridinium iodide complexes is remarkably sensitive to the nature of the solvent in which it is measured. The transition energies derived from the band positions are linear in the Winstein–Grunwald **Y**-value (a kinetic measure of solvent ionizing power) over a wide range of methanol–water, ethanol–water and acetone–water mixtures. The c.-t. bands can be measured in solvents for which it is not possible or convenient to seek a **Y**-value. It is proposed that the transition energies, in kcal./mole, be adopted as empirical measures of solvent polarity, and that they be called **Z**-values. The complex formed from 1-ethyl-4-carbomethoxypyridinium iodide was selected as a standard, and pyridine-1-oxide was used to extend the **Z**-value scale to isoöctane, in which the salt is insoluble. As an initial check on the validity of the **Z**-values, the transition energies for the c.-t. bands of the 1-ethyl-4-cyanopyridinium iodide complex in six solvents have been found to be linear in **Z**. A theoretical analysis of the data based on a reasonable model for the 1-alkylpyridinium iodide complex provides the free energy of solution of the complex dipole. The difference in free energy of solution of the 1-ethyl-4-carbomethoxypyridinium iodide complex dipole between isoöctane and water is 17.3 kcal./mole. Using the apparent absorption coefficient as a measure of the effect of the solvent on the association of the pyridinium and iodide ions into complex, it is found that certain solvents (dimethylformamide, dimethyl sulfoxide) are *dissociating*, and others are *associating* (acetic acid), in comparison with "normal" solvents (chloroform, alcohols, etc.).

In the course of an investigation of 1-alkylpyridinium iodide complexes[1], it was noted that the absorption attributable to the complex appeared to move to longer wave lengths in less polar solvents.[1b,2] 1-Methyl-4-carbomethoxypyridinium iodide was chosen for further study because the salt had a deep orange color, in contrast with the colorless appearance of 1-methylpyridinium iodide, suggesting that the absorption band of interest might be more accessible than were those of the polymethylpyridinium iodides.[3]

In fact, a new absorption band did appear in many solvents and was clearly distinct from the absorption of the pyridinium ion. The position of the band was remarkably sensitive to the nature of the solvent, the maximum varying in a manner parallel with the ionizing power of the solvents used. In an effort to discover how precisely the charge-transfer absorption reflected the ionizing power of the medium, a series of mixtures of methanol, ethanol and acetone with water were used as solvents. An independent measure of solvent ionizing power, the **Y**-value of Winstein and Grunwald,[4] is available for these media.

Experimental

Pyridinium Salts.—1-Methyl-4-carbomethoxy iodide, orange crystals from methanol, m.p. 190–191° dec. (in bath at 184°) (reported[5] 176–179°); 1-ethyl-4-carbomethoxy iodide, bright orange-yellow crystals, crystallized by cooling a solution in acetone prepared at room temperature, m.p. 111–112°, equiv. wt. (by titration of the iodide ion) calcd. 293.1, found 293.2; 1-methyl-4-cyano iodide, bright orange-yellow crystals from cold water by addition of acetone, m.p. 197–198.5° dec. (in bath 180°), equiv. wt. (Ag I) calcd. 246.1, found 247.4; 1-ethyl-4-cyano iodide, bright yellow crystals from cold water by addition of acetone, m.p. 144.5–146° (evacuated sealed tube), equiv. wt. (titration) calcd. 260.1, found 260.0.

Pyridine-1-oxide was redistilled twice under reduced pressure, colorless, hygroscopic crystals, b.p. 146–147° (13 mm.).

Solvents.—Methanol, A.C.S. grade, was either used directly or dried with magnesium and redistilled; both solvents gave the same results. Ethanol, commercial absolute, was dried with magnesium and redistilled, or, 95% alcohol was dried with calcium oxide, then with magnesium. 1-Propanol, Eastman Kodak Co. white label was dried with calcium oxide and fractionated, b.p. 95–96°. 1-Butanol, C.P. was redistilled, b.p. 115–116°; isopropyl alcohol, C.P., was dried with magnesium and fractionated, b.p. 81–82°; *t*-butyl alcohol was dried with lithium aluminum hydride, and fractionated, b.p. 82–82.8°; dioxane was purified according to Fieser[6] and distilled from sodium, b.p. 100–101°; dimethylformamide, Eastman Kodak Co. white label was dried and redistilled, b.p. 80° (150 mm.); pyridine, Eastman Kodak Co. spectro grade was used directly; acetonitrile, Eastman Kodak Co. spectro grade was used directly; 2,2,3,3-tetrafluoropropanol was fractionated, b.p. 106–106.6°; 2,2,3,3,4,4,5,5-octafluoropentanol, as supplied; chloroform (0.13 *M* ethanol), A.C.S. grade, or purified according to Fieser[6]; dimethyl sulfoxide (Stepan Chemical Co.), was redistilled, b.p. 84° (16 mm.);

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[1] (a) E. M. Kosower and P. E. Klinedinst, Jr., THIS JOURNAL, **78**, 3493 (1956); (b) E. M. Kosower and J. C. Burbach, *ibid.*, **78**, 5838 (1956).

[2] A preliminary communication of a portion of this work has appeared, *ibid.*, **78**, 5700 (1956). Certain conclusions derived from this paper and the two succeeding articles have been presented at a symposium "Solvent Effects and Reaction Mechanism," Queen Mary College, University of London, July 8-9, 1957, and at the XVIth International Congress of Pure and Applied Chemistry at Paris, July 18-24, 1957.

[3] *Cf.* F. Kröhnke, *et al.*, *Chem. Ber.*, **83**, 35 (1950); **86**, 1132 (1953); **87**, 1126 (1954); **88**, 851, 863 (1955).

[4] (a) E. Grunwald and S. Winstein, THIS JOURNAL, **70**, 846 (1948); (b) S. Winstein, E. Grunwald and H. W. Jones, *ibid.*, **73**, 2700 (1951); (c) A. H. Fainberg and S. Winstein, *ibid.*, **78**, 2770 (1956); **79**, 1597, 1602, 1608 (1957).

[5] C. A. Grob and E. Renk, *Helv. Chim. Acta*, **37**, 1672 (1954).

[6] L. F. Fieser, "Experiments in Organic Chemistry," D. C. Heath and Co., Boston, Mass., 1955.