

Lineken.⁶ Qualitative tests for conducting power were made in some instances with small volumes of hydrogen sulfide in test tubes using unstandardized dip type electrodes.

Conductance Data.—The results of conductance measurements on solutions of tetraethylammonium sulfamate in liquid hydrogen sulfide are shown in Table II and in Fig. 1.

TABLE II
CONDUCTANCE OF TETRAETHYLAMMONIUM SULFAMATE IN LIQUID HYDROGEN SULFIDE^a

C	$k \times 10^6$	Λ
0.00016	0.4833	3.0
.000789	1.107	1.40
.001421	1.593	1.121
.003749	3.217	0.8581
.005949	4.558	0.7662

^a C is concentration in moles per liter. k is the specific conductance of the solute. (The specific conductance of the solvent was 11×10^{-10} ohms⁻¹ cm.⁻¹.) Λ is the molar conductance.

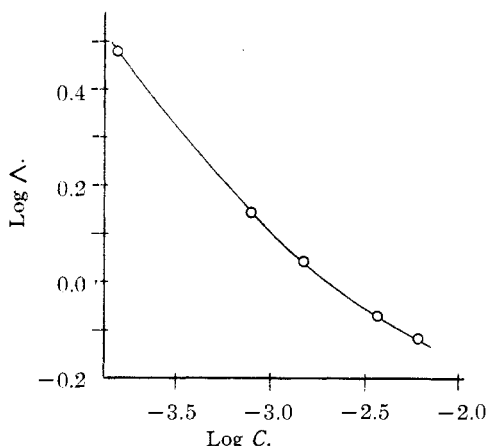


Fig. 1.

The specific conductance of the solute in a solution containing excess solid and presumably saturated, because the resistance of the solution was not changed by further agitation, was 4.79×10^{-6} ohm⁻¹ cm.⁻¹.

The resistances were measured at about 1000 cycles per second. The resistance of the most dilute solution varied considerably less than 0.1% with changes in frequency (470 to 2640 cycles per second). It was not considered necessary to examine each solution for possible frequency effects because the specific conductance range involved was small.

(6) Lineken, *THIS JOURNAL*, **68**, 1966 (1946).

SOMERVILLE, N. J.

RECEIVED OCTOBER 9, 1946

The Interaction of 6-Chloro-2-methoxy-9-phenoxyacridine and Ethylene Diamine

BY R. L. MCKEE¹ AND R. W. BOST

Recently it has been reported² that the reaction between 9-chloroacridine and an excess of ethylenediamine forms N,N'-bis-(9-acridyl)-ethylenediamine rather than the expected 9-(β-aminoethylamino)-acridine.

In confirmation of this report, we wish to re-

(1) Wm. S. Merrell Co., Postdoctoral Fellow.

(2) Albert and Gledhill, *J. Soc. Chem. Ind.*, **64**, 169 (1945).

cord the similar formation of N,N'-bis-(6-chloro-2-methoxy-9-acridyl)-ethylenediamine.

Experimental

A mixture of 5.5 g. (0.016 mole) of 6-chloro-2-methoxy-9-phenoxyacridine,³ 10 g. of phenol and 2.0 g. (0.033 mole) of ethylenediamine (Eastman Kodak Co., 95–100% dried over potassium hydroxide and distilled) was warmed on a steam-bath for two hours. Concentrated hydrochloric acid (10 cc.) was added, followed by 50 cc. of ether. The precipitate was filtered and washed with ether. The solid was ground thoroughly under concentrated ammonium hydroxide, filtered, washed with water and dried. The product was rather difficultly soluble in alcohol or dioxane and was recrystallized from absolute alcohol containing 20% of pyridine. The yield was 3.5 g. (81.5% of the theoretical) of fine yellow crystals melting with decomposition at 134–136°.

Anal. Calcd. for C₃₀H₂₄Cl₂N₂O₂: N, 10.34; Cl, 10.35. Found: N, 10.35, 10.39; Cl, 13.19.

(3) Drozdov, *J. Gen. Chem., U. S. S. R.*, **7**, 1668 (1937); through *C. A.*, **32**, 160 (1938).

THE VENABLE CHEMICAL LABORATORY
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RECEIVED SEPTEMBER 23, 1946

The Reaction between 4-Methoxybenzaldehyde and 2,3-Dimethylquinoxaline

BY R. L. MCKEE,¹ MARTHA K. MCKEE AND R. W. BOST

The reaction between 2,3-dimethylquinoxaline and a number of aromatic aldehydes has been studied by Bennett and Willis.² However, these authors carried out the condensations according to a standardized procedure to determine the effect of structure on reactivity, thereby frequently leaving the fate of the major portions of the reactants unascertained; 2,3-dimethylquinoxaline and *p*-methoxybenzaldehyde thus reacted to form 2-*p*-methoxystyrylquinoxaline (10%) and 2,3-di-*p*-methoxystyrylquinoxaline (10%).

A somewhat more exhaustive examination of this reaction is here reported. It is of interest that no 2'-*p*-methoxyphenyl-2,3-trimethylquinoxaline could be found.

Experimental

2,3-Dimethylquinoxaline (15.8 g., 0.10 mole) and 27.2 g. (0.20 mole) of *p*-anisaldehyde were refluxed together in 43 cc. of acetic anhydride for thirteen hours, after which 35 cc. of solvent was removed by distillation. Water (20 cc.) was added to the residue, followed by a dropwise addition of acetone until the solution was homogeneous at its boiling point. Crystallization was allowed to proceed for thirty-six hours in the icebox, after which the solid was filtered and washed with a little ether. The brilliant yellow product weighed 8.5 g. (22%) and consisted of 2,3-di-*p*-methoxystyrylquinoxaline (m. p. 167–169°).

The filtrate from this product was steam distilled while still acidic, resulting in the recovery (by ether extraction of the distillate) of 11.0 g. of anisaldehyde. The residue in the still was made slightly alkaline (sodium hydroxide) and again steam distilled, thus yielding 1.0 g. of 2,3-dimethylquinoxaline (m. p. 102–104°). The still residue was extracted with ether, and the ether layer was dried over potassium carbonate and allowed to concentrate slowly. No crystallization occurred, and the oil was sub-

(1) Wm. S. Merrell Co., Postdoctoral Fellow.

(2) Bennett and Willis, *J. Chem. Soc.*, 1960 (1928).

jected to vacuum distillation. At 3 mm. pressure a few drops of liquid distilled below 200°, the main product distilling from 200–225° as a red-brown viscous liquid which soon crystallized. The residue (9.0 g.) could not be distilled or crystallized. After recrystallization from acetone and petroleum ether, the volatile portion weighed 9.3 g. (34%) and was found to be 2-*p*-methoxystyryl-3-methylquinoxaline (m. p. 124–125°).

Of the starting materials 0.041 mole of anisaldehyde and 0.038 mole of dimethylquinoxaline are unaccounted for; thus the 9.0 g. of undistillable residue probably consists of a polymeric 1:1 condensation product.

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RECEIVED NOVEMBER 25, 1946

The Critical Concentration of Dodecanesulfonic Acid

By H. F. WALTON

The writer recently measured the activity of 1-*n*-dodecanesulfonic acid in aqueous solution at two temperatures by an electromotive force method, and concluded from the results that the critical concentration, above which the association of negative ions to form micelles begins, is 0.0064 *M* at 25°, and 0.003 *M* at 0°. These values, especially that at 0°, are at variance with those found by other methods, as in Table I.

TABLE I

Method	Critical concn., molar		Ref.
	0°	25°	
E. m. f.	0.003	0.0064	1
Conductivity	.010	.0072	2, 3
Freezing point	.010	3
Indicator	.0095 (4°)	.0075	This work

This note describes measurements of the light absorption by a dissolved indicator dye, thymol blue. This indicator normally changes from red to yellow in the *pH* range 1.2–2.8, but in 0.01 *M* dodecanesulfonic acid it is a darker red than in 2 *M* hydrochloric acid. The position of the absorption bands of the indicator is not changed appreciably by the colloidal electrolyte, but their intensity is increased, and the red form appears to be solubilized or stabilized in some way. Solubilization is a property of micelles rather than simple molecules, as Hartley showed,⁴ and the concentration where solubilization first occurs is approximately equal to the critical concentration of the colloidal electrolyte.^{4,5,6}

Experimental

Solutions of the yellow form of thymol blue in water and in 0.02 *M* dodecanesulfonic acid were prepared, the indicator concentration being the same in both. The optical density of the sulfonic acid solution was measured at 550 μ , using a Beckman spectrophotometer with 1-

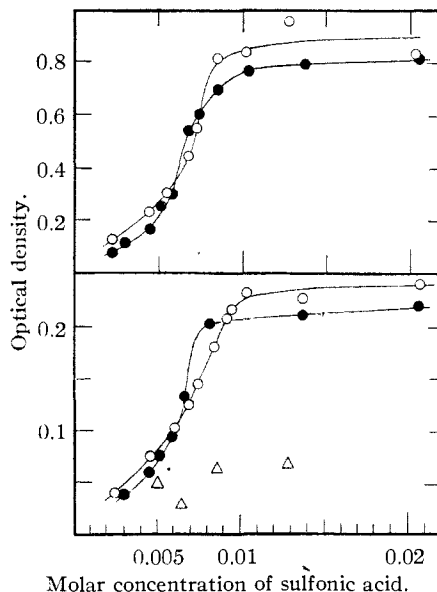


Fig. 1.—Optical density versus acid concentration: upper curves, 10 p.p.m. thymol blue; lower curves, 2.5 p.p.m. thymol blue; open circles, 4°; filled circles, 25°; triangles, hydrochloric acid plus 2.8 p.p.m. thymol blue at 25°.

cm. cell. Five hundred and fifty μ is the peak of the main absorption band of the red form; the yellow form absorbs hardly at all at this wave length. The sulfonic acid concentration was progressively reduced by diluting a measured volume of solution with the aqueous indicator solution added from a buret; the indicator concentration thus remained constant. Runs were made with two different indicator concentrations, 10 p. p. m. (2.1×10^{-6} *M*) and 2.5 p. p. m. (5.2×10^{-6} *M*), and at two temperatures, 25 and 4°; the 4° measurements were made in the cold room. The results are shown in the graph, with comparative data for the indicator in hydrochloric acid. The curves for the lower indicator concentration show the sharper inflections. Since the concentration of indicator is so much less than that of the acid, the effect of the indicator in promoting micelle formation must be very small, and these inflections, coming at 0.0095 *M* at 4° and 0.0075 *M* at 25°, probably represent the critical concentrations of sulfonic acid for micelle formation. If so, they agree well with conductivity and freezing point measurements, as is shown in Table I. Why the electromotive force method should give different results is still not apparent.

A comparison of the optical densities in hydrochloric acid and in dodecanesulfonic acid shows that the red indicator color is considerably enhanced by the sulfonic acid even below the critical concentration. At 25°, the optical density rises approximately with the square of the sulfonic acid concentration, suggesting that a binary complex, such as $(C_{12}H_{25}SO_3)_2^-$, is formed which associates with the indicator.

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RECEIVED SEPTEMBER 13, 1946

Note on the Darzens-Claisen Reaction

By MELVIN S. NEWMAN AND BARNEY J. MAGERLEIN

On attempting to alkylate cyclohexanone by treating its sodium enolate with the *p*-toluenesulfonic acid ester of ethyl glycolate, the main prod-

(1) Walton, *THIS JOURNAL*, **68**, 1180 (1946).

(2) Brady, Thesis, Stanford University, 1944.

(3) McBain, Dye and Johnston, *THIS JOURNAL*, **61**, 3210 (1939).

(4) Hartley, *J. Chem. Soc.*, 1968 (1938).

(5) Sheppard and Geddes, *J. Chem. Phys.*, **13**, 63 (1945).

(6) Corrin, Klevens and Harkins, *ibid.*, **14**, 216 (1946).