proceeds from the start, there precipitates out of the conductance vessel a white, crystalline solid with a composition corresponding to $Al_2Cl_6{\cdot}S_2Cl_2$.[9] After the molar ratio ($S_2Cl_2/AlCl_3$) of 0.5 is reached, this compound ceases to precipitate and no further change in the solution is apparent. A brief summary of the data corresponding to Fig. 2 is given in Table II.

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[9] Found for $Al_2Cl_6S_2Cl_2$: 13.3% Al, 70.4% Cl. Calcd.: 13.48% Al, 70.89% Cl.

SCHOOL OF CHEMISTRY, UNIVERSITY OF MINNESOTA
MINNEAPOLIS, MINNESOTA, AND
DEPARTMENT OF CHEMISTRY, SYRACUSE UNIVERSITY
SYRACUSE, N. Y.

The Aluminum Chloride-Catalyzed Condensation of γ-Butyrolactone with Benzene

BY WILLIAM E. TRUCE AND CECIL E. OLSON

RECEIVED APRIL 14, 1952

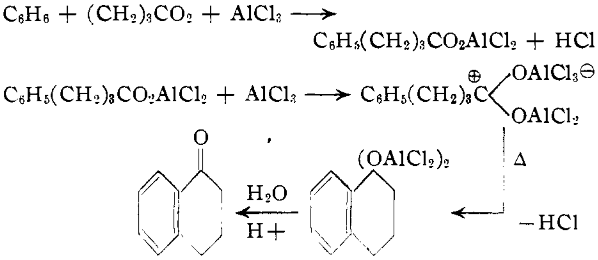
The condensation of γ-butyrolactone with benzene in the presence of aluminum chloride has been reported by Christian.[1] We have independently studied this reaction and observed several differences. A higher yield of γ-phenylbutyric acid was realized and α-tetralone was obtained as a product. The relative amounts of the two products can be greatly altered by varying the amount of aluminum chloride as shown in Table I.

TABLE I

Molar ratio $AlCl_3$/lactone	Yield of α-tetralone, %	Yield of γ-phenylbutyric acid %
1.25	11	73
1.67	32	43
2.50	66	..

The formation of a ketone by the condensation of a lactone with benzene does not appear to have been reported previously. It is submitted as a convenient preparation of γ-tetralone and possibly it could be extended to the preparation of related cyclic ketones.

The following steps and intermediates for the reaction are suggested.



$$C_6H_6 + (CH_2)_3CO_2 + AlCl_3 \longrightarrow C_6H_5(CH_2)_3CO_2AlCl_2 + HCl$$

$$C_6H_5(CH_2)_3CO_2AlCl_2 + AlCl_3 \longrightarrow C_6H_5(CH_2)_3\overset{\oplus}{C}\begin{matrix}OAlCl_3^{\ominus}\\OAlCl_2\end{matrix}$$

Recent work by Birch and co-workers,[2] and later by Snyder and Werber,[3] in which γ-phenylbutyric acid was condensed to α-tetralone with various strong acids, lends support to the above proposal.

Experimental

The three reactions described below were carried out in a 500-ml., three-neck, round-bottom flask equipped with a reflux condenser (capped with a drying tube), mechanically-driven, sealed stirrer and a 125-ml. erlenmeyer flask connected to the reaction flask by flexible tubing.

Reaction No. 1.—To a well-stirred solution of benzene (200 ml., 176 g., dried over CaH_2) and γ-butyrolactone (20.8 g., 0.24 mole), C.P. anhydrous aluminum chloride (40.2 g., 0.3 mole) was added at a rate that kept the solution refluxing (about one hour). The solution was heated at reflux temperature until the evolution of hydrogen chloride had nearly ceased (about four hours).

The mixture was cooled to room temperature and poured over 200 g. of ice drenched in concentrated hydrochloric acid. The organic layer was separated and washed twice with water. The aqueous part was combined with the washings, washed twice with ether and discarded. The ether washings and the organic layer were combined, dried and distilled under reduced pressure. The first product was a colorless liquid, α-tetralone; b.p. 120–124° (10 mm.) (lit.[4] 128° (12 mm.)); $n^{20}D$ 1.5691, (lit.[3] 1.5688); semicarbazone, m.p. 214–216° (lit.[3] 216°), and yield 4.0 g. (11%). The second product was a waxy crystalline solid, γ-phenylbutyric acid, b.p. 148–155° (10 mm.) (lit.[1] 120–125° (1 mm.)), m.p. 47–48° (lit.[1] 48–49°), and yield 28.5 g. (73%).

Reaction No. 2.—The same amounts of benzene and γ-butyrolactone were used as in Reaction No. 1 with 53.6 g. (0.4 mole) of aluminum chloride and under the same conditions. The yield of α-tetralone was 11.1 g. (32%) and the yield of γ-phenylbutyric acid was 17.0 g. (43.3%).

Reaction No. 3.—Following the same procedure but using 80 g. (0.6 mole) of aluminum chloride, the yield of α-tetralone was 23.2 g. (66%). No phenylbutyric acid was isolated from the brown residue in the distilling flask.

Acknowledgment.—The authors are grateful to the Procter and Gamble Co. for financial support in this work.

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(1) R. V. Christian, Jr., THIS JOURNAL, **74**, 1591 (1952).

(2) A. J. Birch, R. Jaeger and R. Robinson, *J. Chem. Soc.*, 582 (1945).

(3) H. R. Snyder and F. X. Werber, THIS JOURNAL, **72**, 2965 (1950).

(4) H. Luther and C. Wächter, *Chem. Ber.*, **82**, 161 (1949).

DEPARTMENT OF CHEMISTRY
PURDUE UNIVERSITY
LAFAYETTE, INDIANA

Ionophoresis in Non-aqueous Solvent Systems

BY M. H. PAUL AND E. L. DURRUM

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Recently there has been renewed interest in what Tiselius[1] has termed "Zone Electrophoresis." Electrophoresis on paper, particularly, has become a convenient and useful method for the separation of an ever increasing number of charged substances. Although considerable data had been amassed about the properties of non-aqueous solvent systems by Walden,[2] a search of the recent literature uncovered no references to electrophoretic separations in non-aqueous solvents with the exception of a paper[3] on the mobility of carbon black particles suspended in kerosene.

A preliminary study on the application of non-aqueous systems to filter paper ionophoresis was undertaken in an attempt to effect resolution of mixtures of certain biological compounds which are insoluble in aqueous electrolytes, such as cholesterol, higher fatty acids and steroid hormones.

The movement of dyes on filter paper was studied first because their migration is conveniently followed. Paper strips (30 × 1.5 cm.) cut from "Whatman 3 MM" filter paper were suspended in a glass and bakelite electrophoresis cell (apex height—13 cm.) similar to that previously described.[4]

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(1) A. Tiselius, Abstracts, XII International Congress of Pure and Applied Chemistry, New York, N. Y., Sept., 1951, p. 67.

(2) P. Walden, "Electrochemie Nichtwässriger Lösungen," Barth, Leipzig, 1924.

(3) M. Hayek, *J. Phys. Colloid Chem.*, **55**, 1527 (1951).

(4) E. L. Durrum, THIS JOURNAL, **72**, 2943 (1950).

A small drop of the dye mixture (dissolved in the solvent system used) was placed at the apex of the strips and after the ascending solvent fronts had coalesced at the apex, a potential was applied across the strips by means of platinum electrodes immersed in the electrode vessels.

Figure 1 was prepared from a tracing of paper strips and illustrates the separation of an electropositive dye (crystal violet) and an electronegative dye (Oil Red O). In this separation a mixture of nitromethane (9 vol.) and glacial acetic acid (1 vol). was used. Similar separations have been effected in absolute ethyl alcohol, methanol and in pyridine–glacial acetic acid mixtures.[5] Included in Fig. 1 is an ascending chromatogram[6] in the same nitromethane–glacial acetic acid solvent where it will be noted no separation was achieved.

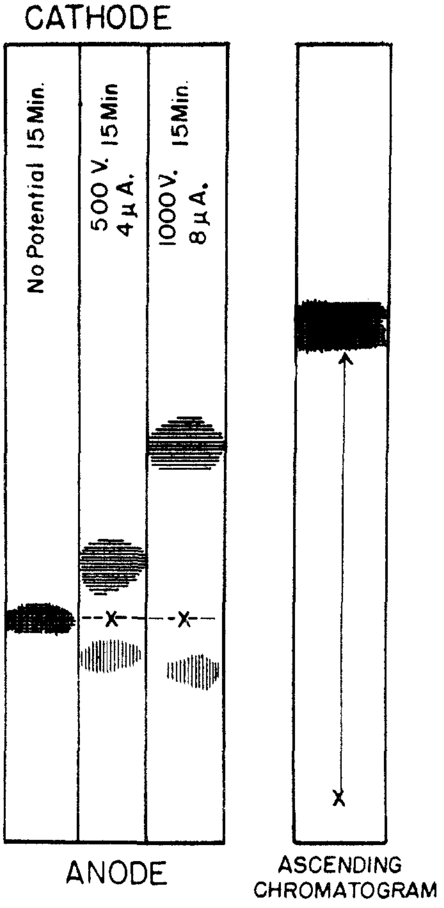

Fig. 1.—Separation of oil red O (vertical lines) and crystal violet (horizontal lines) in nitromethane–glacial acetic acid mixture.

In Fig. 2 the separation of a mixture of four dyes in absolute ethyl alcohol is illustrated.

Cholesterol, palmitic acid, 17-hydroxy-11-dehydrocorticosterone and testosterone were found to migrate toward the anode in the ethyl alcohol or nitromethane–glacial acetic acid systems; however, no separation of these mixtures of compounds was apparent over the short distances traversed. It is perhaps significant that separations were achieved only with ions containing strongly polar groups (the dyes). The migration of the biological substances mentioned may be a passive electroendosmotic effect; however it is possible that these less polar materials could be separated in experiments of longer duration.

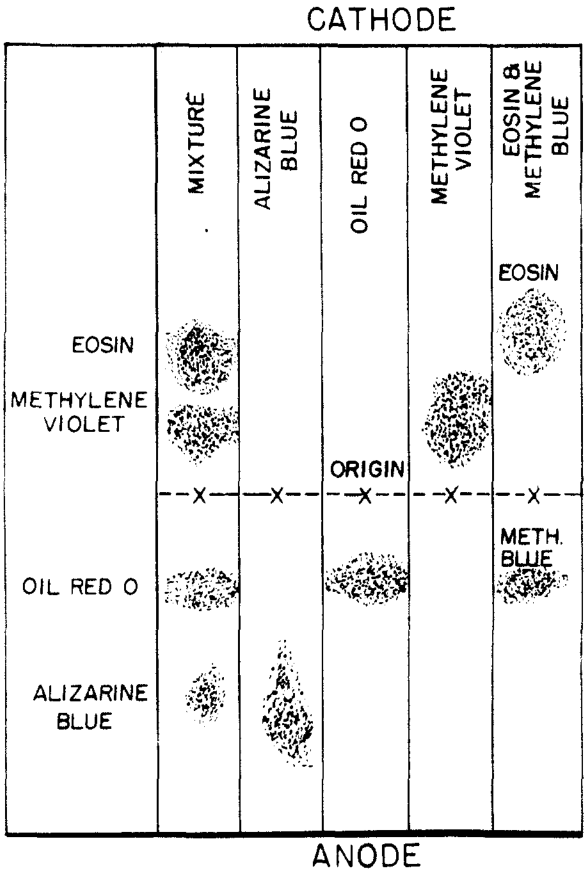

Fig. 2.—Separation of dye mixture in ethyl alcohol; 1000 volts 60 microamps, 50 min.

The separation of the dye mixtures on the filter paper establishes the feasibility of electrophoretic separations in non-aqueous solvent systems. It is possible that other solvent systems and experimental conditions may provide extension of this technique to a wider range of substances.

Army Medical Service Graduate School
Walter Reed Army Medical Center
Washington 12, D. C.

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[5] The most highly purified commercially available solvents were employed; however, no effort was made to remove traces of moisture with which the solvents were in equilibrium, since the cell design did not completely exclude atmospheric moisture.

[6] R. J. Williams and H. Kirby, *Science*, 107, 481 (1948).

The Dissociation Energies of the First and Second Bond in H_2S and Some Comments on a Recent Paper by Franklin and Lumpkin

By A. H. Sehon[1]

Received April 21, 1952

Franklin and Lumpkin[2] have recently derived a value of 38.4(±5) kcal./mole for ΔH_f(SH) from relevant thermochemical data and their measurements of appearance potentials of carbonium ions

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[1] Post-doctoral fellow, 1951–1952.

[2] J. L. Franklin and H. E. Lumpkin, This Journal, 74, 1023 (1952).