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.

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

BY WILLIAM E. TRUCE AND CECIL E. OLSON

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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.

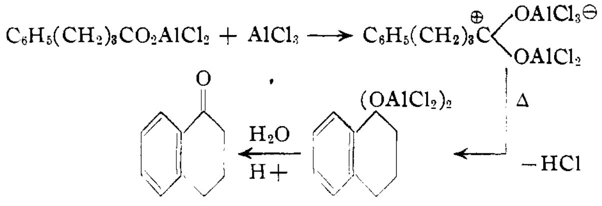
TABLE I

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.

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



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.

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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).

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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).