

parts of potassium cyanide, placed in a ten gram clay crucible and covered with salt. The fusion is made in a muffle at a bright red heat, allowing it to remain there thirteen minutes. When removed it is thoroughly shaken to collect the globules into one metallic button and allowed to cool. The crucible is broken and the resulting button carefully broken from the slag and weighed.

In conclusion the writer desires to express his obligation both to Mr. M. Elsasser and to Mr. Ferdinand McCann for their kind assistance in furnishing him with suggestions, data, etc., without which this paper would not have been written.

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## ELECTROLYTIC DETERMINATION OF ZINC IN THE PRESENCE OF MANGANESE.

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**I**N determining zinc quantitatively by means of the electric current, it is essential to have the zinc in the form of sulphate and there should be neither nitrates nor chlorides present.

It was found best to use a platinum dish, on which silver had been deposited, for a cathode. In the first experiments, I tried copper deposits on platinum, but discarded this owing to the fact that copper is too easily oxidized, and consequently causes variation in weight. During the deposition of zinc it is found best to stir the solution (using a mechanical stirrer) continually, so as to obtain an even and uniform deposit; and to keep the temperature under 26° Celsius.

The current used for the deposition of zinc was four volts, giving a current of three and five-tenths volts and 0.20 to 0.26 amperes for 100 sq. cm. of cathode area. The current may vary from 0.20 to 0.26 amperes and the temperature from 15° to 26° Celsius.

Different proportions of zinc and manganese were tried so as to determine the action of the manganese, but in no case could any traces of manganese be found in the zinc which was deposited on the cathode.

The following reagents were used for the electrolysis:

*a.* A solution of lactic acid ( $\text{CH}_3\text{.CHOH.COOH}$ , each

cubic centimeter of which contained 0.937 gram of lactic acid.

b. A solution of ammonium lactate ( $\text{CH}_3\text{CHOH.COOH.NH}_4$ ) each cubic centimeter of which contained 0.50 gram of ammonium lactate.

c. Ammonium sulphate,  $(\text{NH}_4)_2\text{SO}_4$ , crystalline.

The volume of the solution in all cases was 230 cc.; the cathode surface 150 sq. cm., and the anode one and one-half cm. distant from the cathode.

The zinc and manganese salts used were the sulphates  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  and  $\text{MnSO}_4 \cdot 7\text{H}_2\text{O}$ .

#### ANALYSIS NO. 1.

Zinc sulphate .....	0.5074 gram
Manganese sulphate .....	0.1634 "
Ammonium lactate .....	5.0000 "
Lactic acid .....	0.7500 "
Ammonium sulphate .....	2.0000 "
Ampere, varying from .....	0.24-0.26 N.D. <sub>100</sub>
Voltage, " " .....	3.7-3.9
Temperature varying from .....	20°-25° C
Percentage of zinc found .....	22.786
" " " theory .....	22.78
Time required to deposit zinc completely .....	4 hours, 10 minutes

No traces of manganese were found in the zinc.

#### ANALYSIS NO. 2.

Zinc sulphate .....	0.5000 gram
Manganese sulphate .....	0.4466 "
Ammonium lactate .....	5.0000 "
Lactic acid .....	0.7500 "
Ammonium sulphate .....	2.0000 "
Ampere varying from .....	0.2-0.22 N.D. <sub>100</sub>
Voltage " " .....	3.5-3.8
Temperature varying from .....	15°-22° C.
Percentage of zinc found .....	22.82
" " " theory .....	22.78
Time required to deposit zinc completely .....	4 hours, 10 minutes.

#### ANALYSIS NO. 3.

Zinc sulphate .....	0.5000 gram
Manganese sulphate .....	2.0000 "
Ammonium lactate .....	5.0000 "
Lactic acid .....	0.3800 "
Ammonium sulphate .....	2.0000 "
Ampere varying from .....	0.2-0.23 N.D. <sub>100</sub>
Voltage " " .....	3.0-3.6
Temperature varying from .....	18°-24° Celsius
Percentage of zinc found .....	22.72
" " " theory .....	22.78
Time required to deposit zinc completely .....	5½ hours

In all I have made about sixteen separations of zinc from manganese in the manner described, but in only two cases did the electrolysis require more than five and one-half hours, or did the result vary more than 0.10 per cent. from the theoretical value. Constant stirring was found to give a more rapid and uniform silver-like deposit of zinc.

In several cases manganese hydroxide was found on the anode, in small amounts during the electrolysis, but in no case could any traces of manganese be found in the deposited zinc.

During every electrolysis containing manganese, it was noticed that the solution turned from colorless to amethystine, the more manganese present the darker the color. On allowing this solution to stand for a short while, after having removed it from the platinum dish, and disconnected the current, it was noticed that the solution again became colorless.

It was found that the best results were obtained between the temperatures of 15° and 28°C. If below 15° C. the electrolysis requires longer, if above 28° C. the zinc may deposit in such a manner (either large crystals or even spongy) as might cause loss in washing. The amount of zinc deposited in each electrolysis should not be more than 0.1700 gram of pure zinc.

After the electrolysis is completed the solution is poured off, the zinc deposit washed with distilled water, alcohol, and finally with pure ether to remove alcohol; then place a in the hot water oven to dry perfectly, then placed in a desiccator until ready for weighing. This washing and drying should not require more than five minutes.

When an electrolysis is started the current should be regulated to 0.25 ampere N.D.<sub>100</sub>, and if the temperature is between 15° and 28° C. it will be found that during the entire process the current will vary but slightly.

If the current is increased to more than 0.30 ampere N.D.<sub>100</sub> (normal density of current for 100 sq. cm. of cathode area) it is found that the zinc will not be deposited evenly, and more manganese hydroxide will be found at the anode; also that the zinc being deposited so quickly, and in larger crystals, will mechanically enclose some manganese or solution. If the current is less than 0.20 ampere N.D.<sub>100</sub>, the time required for complete deposition will be longer.

The solution from which zinc is to be deposited should always be neutral or slightly acid.

These experiments were made during 1897-1898 in the laboratory of the Koenigliches Polytechnikum in Munich, Germany.

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## THE INFLUENCE OF SUBSTITUENTS ON THE ELECTRICAL CONDUCTIVITY OF BENZOIC ACID.<sup>1</sup>

BY ALFRED TINGLE.

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### INTRODUCTORY.

VICTOR MEYER and his pupils have shown that in a substituted aromatic acid, the rate of esterification is affected by the nature and position of the substituting atoms or groups. Kellas<sup>2</sup> proved that for monochlor-, monobrom-, and moniodobenzoic acids, the rate of esterification varies with the molecular weight of the acid, or in other words, with the atomic weight of the substituting halogen. Thus, graphically represented, when the molecular weight of the acid is taken as the abscissa, and the percentage of ester formed in a given time, as the ordinate, a straight line is obtained.

No attempt appears to have been made, even in view of this work, to examine the effect of the position and nature of a substituent on the electrical conductivity of the aromatic acids. The conductivities of substituted benzoic acids have been examined by many investigators, but usually with the view of determining their affinity coefficients, and with little regard for the nature of the substituting group.

The conductivities of salts of organic acids have also been investigated, notably by Ostwald, but never, as it would appear, with the view of finding any relation between the conductivities in question and the atomic weights of the metals forming these salts.

The present investigation was undertaken with the object of examining into the changes introduced in the conductivity of benzoic acid, by the substitution of various metals for the

<sup>1</sup> From author's thesis for the Ph.D. degree.

<sup>2</sup> Kellas: Inaugural Dissertation, Heidelberg, 1897.