

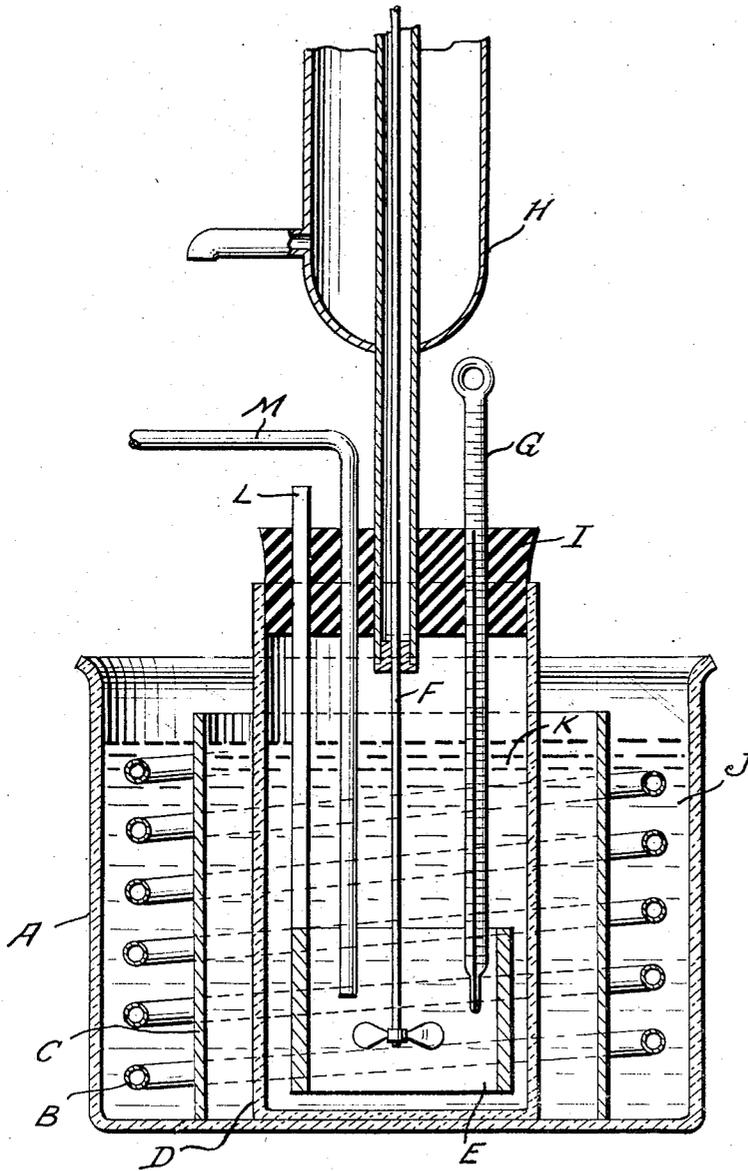
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ELECTROLYTIC PRODUCTION OF AMINOALCOHOLS

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ELECTROLYTIC PRODUCTION OF AMINOALCOHOLS

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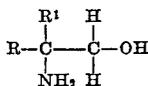
Application March 13, 1944, Serial No. 526,328

2 Claims. (Cl. 204-74)

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This invention relates to the preparation of reduction products of aliphatic nitro hydroxy compounds and more particularly to a process for preparing such compounds by the electrolytic treatment of aliphatic nitro hydroxy compounds at the cathode pole of an electrolytic cell.

A primary object of the invention is to provide a process for the electrolytic preparation of aliphatic amino hydroxy compounds of the following structural formula



wherein R represents a substituent selected from a class consisting of hydroxymethyl, methyl and ethyl groups and R¹ represents a substituent selected from a class consisting of hydrogen, methyl, ethyl and hydroxymethyl groups.

A further object is to prepare such compounds in high yields and a high state of purity.

Another object is to afford a process for completely reducing aliphatic nitro hydroxy compounds to aliphatic amino hydroxy compounds.

A still further object is to carry out the reductive electrolysis under such controlled conditions that undesired side reactions are suppressed, and the main reactions involved in the reduction are favored.

Further objects will be apparent from the description and claims.

The figure of the drawing illustrates a vertical sectional view of an apparatus that may be utilized to attain the above objects.

Amino hydroxy compounds have been prepared in the past from nitro hydroxy compounds by catalytic hydrogenation, and by the action of reducing agents such as iron and the like. Electrolytic methods have been used with some success in reducing aromatic nitro derivatives to the corresponding amino derivatives. However, attempts to apply the electrolytic method of reduction to aliphatic nitro compounds has met with little or no success due not only to the fact that the aliphatic nitro compounds are somewhat more difficult to reduce than the aromatic compounds, but also because they are extremely reactive, so that under the conditions usually obtaining in the reducing atmosphere of an electrolytic cell, so many competing side reactions take place, that many contaminating products are formed along with minimum amounts of the desired reduction product. In the case of the nitro hydroxy compounds of my invention, the

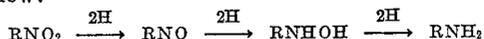
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presence of hydroxy groups in the molecule of the aliphatic nitro compound renders it even more difficult to reduce electrolytically than the simple aliphatic nitro compounds because of its added activity attributable to the hydroxy group which gives rise to an even great number of side reactions, which further lessen the possible yields of desired reduction products.

I have found, nevertheless, that I can electrolytically reduce aliphatic nitro hydroxy compounds completely to the amino hydroxy compounds according to my invention in which side reactions are suppressed and held in check while the main reactions of reduction are fostered.

Electrolytic reduction processes are based on the fact that the cathode, or negative pole of an electrolytic cell establishes a reducing atmosphere in which reactions involving a transfer of electrons from electrode to components of the solution are promoted. Thus, under such conditions, a nitro group will tend to be reduced to an amino group with the gain of electrons from the cathode pole. Once the reduction is initiated however, the intermediate products of the reduction, particularly of the aliphatic nitro hydroxy compounds of my invention are themselves so reactive that side reactions of these intermediate compounds either with themselves, or with the original nitro compound take place, often irreversibly to the virtual exclusion of the desired reductions. It is these, and all reactions other than those contributing to the eventual formation of the amino compounds that must be substantially inhibited or suppressed if high yields of the amino compounds are to be obtained.

In the acid reduction of nitro hydroxy compounds to the amino compound the mechanism of reduction proceeds first to the nitroso compound, next to a hydroxylamino compound and only then to the amino compound as represented below:



Thus, the reaction is a stepwise one and the reaction therefor, can be no faster than the slowest or most limiting step, nor can it be complete unless the intermediate products are preserved from side reactions until they can be made to undergo the desired reduction reactions. I have found that the limiting step in the preparation of the completely reduced aminohydroxy compound is usually the last one in which reduction of the hydroxylamino compound to the amine takes place. Under a wide variety of conditions the nitro and nitroso compounds react swiftly

and completely to form the hydroxylamino compound, but on the other hand, very little amine compound forms until after nearly all the nitro compound has been transformed to the hydroxyl amine compound and then only under carefully controlled conditions which not only tend to prevent side reactions of the auxiliary materials present, but also which protect the amino hydroxy compound from undesired reactions with these other materials present in the cell, including dehydration, deamination, reactions of the nitro compound with the hydroxylamine compound, and the like.

I have found that among the most important factors influencing the progress of the reactions described are the nature and concentration of the electrolytes, particularly the catholyte, the concentration of the nitro hydroxy compound, the temperatures and the current densities employed, and that by carefully controlling these major factors as described hereinafter, high yields of the order of 80-95% or higher of amino hydroxy compounds can be obtained with purities of 90% or more, usually between about 95-99%.

Best results are obtained when the electrolytes, and particularly the catholyte, are compatible with the solutions surrounding them, that is, non-reactive therewith. The catholyte must also be compatible with the nitro hydroxy compound, and with the amino hydroxy compound, and in fact with all the reacting products and products of reaction present in the cell as well as with electrolytic hydrogen, and the material of the electrode. In other words, I select a catholyte which performs its primary purpose of carrying an electric current, without in any substantial way entering into or affecting the reactions which take place in its presence. I have found that the catholyte should be acidic in nature, as free OH ions appear to catalyze certain decomposition reactions of the products present in the cathode compartment, particularly of the nitro compound, and the hydroxyl amino compound. For a catholyte therefore, I have found that acid solutions capable of carrying an electric current, and at the same time which are substantially unreactive and of suitable weakness or dilution as described, are suitable, such as dilute hydrochloric and sulfuric acids, and the like. In order to increase the conductivity and to obtain the highest current efficiency in the cell, it would be presumed that a relatively high concentration of catholyte would be desirable. I have found, however, that under the critical conditions of temperature and the like to be hereinafter described, the acid, if present in high concentrations, results in loss of product and consequent lowering of yields. Therefore, I have found that the acid employed as catholyte should not be present in concentrations much greater than about 25% by weight of the catholyte. The acid may be added in one portion or in several portions during the course of the run. The latter procedure insures lower total acid consumption. If acid is added in several portions, it still must total less than about 25%. The optimum concentration value varies slightly with the compound being reduced, and to some extent with particular acid catholyte but in no case greatly exceeds the concentrations named.

It is, of course, desirable to use the least possible amount of acid consistent with good operation, for reasons of economy. The more acid used above the minimum required, the higher the recovery cost and the greater the likelihood

of undesired side reactions. The less acid used, the greater the cell resistance and the lower the current efficiency. For each acid there is an optimum value of the catholyte concentration at which cell resistance is low, side reactions will be at a minimum, and the difficulty and expense of product recovery will be minimized. For hydrochloric acid, this value is about 10% by weight of the catholyte solution and for sulfuric acid the value is in the neighborhood of 25%.

In the anolyte likewise, I have found that the requirement for maximum conductivity which is desirable for high current efficiency must be compromised somewhat to secure optimum conditions for my process. If the concentration of the anolyte is high as compared to that of the catholyte, the pressure exerted across the diaphragm of the electrolytic cell will be high, causing a tendency for adjustment and interaction, i. e., electroendosmosis will ensue, resulting in mechanical loss of electrolyte as well as in interfering reactions. Accordingly, the concentration of the anolyte must be maintained at a low value, and here, too, an acid, for example, phosphoric, or preferably sulfuric acid, having a concentration of approximately between 10-20% based on the weight of the anolyte must be used. Hydrochloric acid is suitable for use at the anode as far as its electrolytic operability is concerned but since at this pole it is oxidized to form chlorine, and thus is soon lost, it is not desirable to use this acid as the anolyte material. Somewhat higher anolyte concentrations than described above can be used, but result in no substantial increase in conductivity. Lower concentrations on the other hand, markedly reduce the conductivity of the cell, for example, in a 5% sulfuric acid solution, the potential drop is about one-quarter larger than in a 10% solution. Accordingly, as brought out above, it is desirable to maintain the concentration of anolyte fairly close to the 10%-20% figure for maximum yields under the critical conditions employed.

The nitro hydroxy compound which is to be reduced in the reducing atmosphere produced by the cathode pole of the cell should be maintained at a low concentration. This is totally unexpected as it would be assumed from the laws of mass action that high concentrations would produce highest yields. However, I have found that high concentrations of this depolarizer not only produce wasteful side reactions between acid and nitro hydroxy compounds, and between nitro hydroxy compound and certain of the intermediate reduction compounds, notably the RNHOH or hydroxyl amine compound described, but also a lower current efficiency, lowering the yields of amino compounds substantially. These side reactions can be suppressed by conducting the reductions on solutions whose concentrations are not substantially greater than 10% by weight of depolarizer (nitro hydroxy compound) based on total weight of solution in the cathode compartment when hydrochloric acid is used, or about 25% when sulfuric acid is used in cathode solutions, and, as mentioned above, which contain in the neighborhood of 10%-25% or less of acid catholyte.

Both the intensity and total amount of current passed through the cell are also of importance in obtaining maximum yields of amino hydroxy compounds. The intensity factor is conveniently described in terms of current density which may be defined as the amperes of current passed per unit (square centimeter) of active electrode sur-

face. By "active" electrode surface, I mean that portion of the total surface area of the electrode which is immersed in the electrolyte and which is facing the opposite electrode. I have found that the cathode current density as defined should be high and should be maintained at least as high as about 0.1 ampere per square centimeter during the initial stages of the reduction while the concentration of depolarizer is high, conveniently until the input of the amount of current theoretically necessary to completely reduce the amount of nitro compound present to the corresponding amino compound. At low current densities the nitro compound will tend to be reduced only so far as the hydroxylamino derivative. At high current densities the evolution of hydrogen will be a preferred reaction compared with reduction to the amine. Somewhere between these extremes of current density falls the range in which reduction to the amine is the preferred reaction. The lower limit will be more or less inflexible and will fall at about 0.05 ampere/cm.². The upper limit must be determined for each new cell setup—as it will be regulated in some degree by the cell efficiency. In each case, the optimum current density will be a balance of two factors (1) the cell capacity which will be greater at greater effective current densities, (2) the current efficiency which is inversely proportional to the current density. The amount of current is usually measured in faradays which designate the gram equivalent of current for any element, and may be defined as the amount of current necessary to liberate 1 gram atom of hydrogen. Thus the theoretical amount of current necessary to reduce any aliphatic mono-nitro hydroxy compound of my invention to its amino derivative is the same amount per mole of nitro compound, namely six faradays. I have discovered, however, that the complete reduction of the nitro hydroxy compounds to amino hydroxy compounds according to my process requires somewhat more current than that theoretically required for complete reduction. In fact, after the theoretical amount of current has been applied, the nitro hydroxy compound appears to be only partially reduced, lacking perhaps as much as 25% of complete reduction. Therefore, in order to complete the reduction and to obtain an end product in a high state of purity easy to recover, I find that additional current must be added, usually several additional faradays. At this stage, however, it is no longer necessary to maintain the current density at the high figure essential for the initial conversion. Accordingly, I have found that best results are obtained when the nitro compound is subjected to a preliminary electrolysis at a high current density, for example, at least about 0.2 ampere per square centimeter until the concentration of the depolarizer is somewhat reduced, conveniently until the theoretical amount of current has been passed, and then subjected to further electrolysis at a somewhat lower current density, for example, between 0.10 and 0.13 ampere per square centimeter until complete reduction has been effected, although in many cases the initial reduction can be carried out at a current density as low as about 0.10, and continued at this rate until complete reduction has taken place, or several successive reductions in current density may be made during the course of the reduction as the concentration of depolarizer falls off. Complete reduction can be determined by any of the well known tests such as the Fehling's solution test

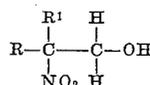
for the presence of reducing agents, or other known tests for indicating such materials. When Fehling's solution, mixed with a portion of the catholyte solution fails to produce the characteristic red or red-brown precipitate, i. e., when this test is negative, it can be concluded that no RNHOH is present, and hence that the reaction has gone to completion.

The optimum temperature for carrying out this reaction varies with the compound to be reduced, but the range of temperature within which effective reduction will occur is quite wide. Optimum temperatures lie generally between 65 and 85° C., but reactions may be carried out at temperatures through at least as wide a range as 50° to 100° C., at atmospheric pressures with useful results as to yields and purity.

The electrodes employed in my process should be selected with regard to good electrolytic practice and accordingly should preferably be inert to the electrolyte solutions and such as efficiently to produce the desired currents and the like. The cathode pole, of course, is of prime importance, and I have found that lead cathodes, especially when highly polished, are particularly effective in their high activity and their contribution to the high yields of my invention. During use, the lead cathodes may gradually lose their activity particularly in catholytes of hydrochloric acid solution but they can readily be reactivated by restoring their highly polished condition, for example, by buffing or polishing the cathode material at intervals. With this procedure, a cathode can be used for an almost indefinite number of runs without loss of activity. Mercury cathodes or copper cathodes also may be used. Stainless steel cathodes are suitable electrolytically but result in somewhat discolored solutions.

Within the limits described, considerable flexibility is inherent in my process, and each of the limiting conditions may be varied with corresponding compensating variations in one or several of the other limiting conditions. For example, when higher temperatures, within the operable ranges are used, either the current density can be lowered, or the acid concentration can be decreased or both, though in case the acid concentration is decreased appreciably, subsequent additions of acid should be made to maintain the cathode solution always sufficiently acid, for efficient reaction. Likewise, if the concentration of depolarizer is decreased, the current density may be decreased, and efficient operation and high yields still be obtained. Other similar variations which will be obvious to one skilled in the art can be made.

My invention is applicable to the reduction of aliphatic nitrohydroxy compounds of the type having the following general structure



wherein R represents a substituent selected from a class consisting of hydroxymethyl, methyl and ethyl groups and and R¹ represents a substituent selected from a class consisting of hydrogen, methyl, ethyl and hydromethyl groups.

This includes such compounds as 2-nitro-2-methyl-1-propanol; 2-nitro-2-methyl-1,3-propanediol; 2-nitro-1-butanol, 2-nitro-2-ethyl-1,3-propanediol; tris(hydroxymethyl)nitromethane; 2-nitro-2-methyl-1-butanol; 2-nitro-1-propanol; and the like.

That is, the invention is generally applicable

to the reduction of all the aliphatic nitro hydroxy compounds of the class described and comprises a general method of synthesis for the corresponding amino hydroxy compounds, although higher efficiencies and smoother reactions are obtained with compounds having fewest hydrogen atoms on the carbon alpha to the nitro group. For example, tertiary nitro compounds respond somewhat less efficiently than secondary nitro compounds but both respond with high yields within the range of commercial utility.

In accordance with my invention, I first prepare an electrolytic cell consisting of anode and cathode compartments filled with anolyte and catholyte solutions, respectively, as described, the anolyte consisting preferably of about a 10-20% solution of sulfuric acid, the catholyte solution preferably of a solution of hydrochloric acid of less than about 10%, or sulfuric acid of about 25% or somewhat less. Anode and cathode poles of suitable conducting material are placed in their respective chambers filled with anolyte and catholyte respectively. The nitro hydroxy compound to be reduced is placed in the cathode compartment of the cell in the concentrations described, and an electric current is passed through the cell at a current density at least as high as about 0.1 to 0.2 ampere per square centimeter, probably in most cases at the higher figure. The temperature of the cell is maintained between about 50°-100° C., depending somewhat on the compound being reduced, throughout the electrolysis. After about six faradays of current per mol of nitro compound have been passed, the amount of current theoretically required to completely reduce the nitro compounds of my invention to the corresponding amino compound, the electrolysis is continued but the current density may be lowered, for example, to about .10-.13 ampere per square centimeter and continued at this intensity until samples of catholyte solution are negative to the Fehling's solution test or other tests indicating absence of oxidizing agents, that is, completion of the reduction of nitro compound to amino compound. Or, successive reductions in current density may be made as the reaction nears completion.

The recovery of the amino alcohol can be effected either as an acid salt or as the free amine. The salt is readily obtained simply by evaporating the catholyte solution to dryness. The amine may then be obtained from the acid salt by any desired known method, for recovering amines from aqueous solutions of their salts, for example, any of the processes involving neutralization and distillation or neutralization and filtration depending on the nature of the amine and of the particular acid salt involved.

The word "purity" as used to describe the products of my invention, is employed in the usual dictionary sense of freedom from foreign admixture or deleterious matter.

One type of electrolytic cell which is suitable for use in my invention, and the one used in the specific examples described below, is illustrated in the figure of the drawing, and comprises a glass vessel A containing a cylindrical lead anode C, and a cylindrical unglazed porcelain diaphragm D. The annular space between the diaphragm cup and the glass vessel holds, in addition to the anode C, a glass cooling-coil B through which hot or cold water can be passed as required to control the temperature. The diaphragm cup serves as a container for the catholyte solution and depolarizer K and for a

cylindrical lead cathode E whose active area is 150 square centimeters. The diaphragm cup is closed off by a rubber stopper I through which are passed a reflux condenser H, stirrer F, thermometer G, cathode connector L and a glass sampling tube M into which samples of the catholyte can be drawn periodically for analysis. Direct current is used. Best results are obtained when the lead cathode is highly polished.

Illustrative of my invention are the following specific examples employing in each case the type of electrolytic cell described above.

EXAMPLE I

Reduction of 2-nitro-2-methyl-1,3-propanediol

In the cell described above, 150 ml. of distilled water, 42.5 ml. of concentrated hydrochloric acid and 25 g. of 2-nitro-2-methyl-1,3-propanediol are mixed to form a catholyte solution containing 11.1% by weight of 2-nitro-2-methyl-1,3-propanediol and 7.8% by weight of hydrogen chloride. The mixture is placed in the cathode section of the cell described above in contact with a polished lead cathode.

Six hundred milliliters of a 10% (by weight) aqueous solution of sulfuric acid are placed in the anode section of the cell.

An electric current is passed through the cell at a current density equal to 0.2 ampere per square centimeter of immersed cathode surface until the quantity of current theoretically required to reduce the nitro compound to amino compound, i. e., six faradays per mole of nitro compound, has been passed. The current density is then reduced to 0.13 ampere and the electrolysis continued until a negative Fehling's solution test is obtained. Throughout the run the catholyte is agitated and maintained at about 65° C. Seventeen grams of 2-amino-2-methyl-1,3-propanediol are recovered, amounting to a material yield of 88% and a current yield of 65%. The purity of this compound was 98.5%, as determined from its neutral equivalent of 106.5, theoretical 105.

EXAMPLE II

Reduction of tris(hydroxymethyl)nitromethane

In the same type of cell described above, 200 ml. of distilled water, 20 ml. of concentrated hydrochloric acid and 25 g. of tris(hydroxymethyl)nitromethane are mixed, and introduced into the cathode section of the cell in contact with a polished lead cathode. The resulting solution in the cathode compartment contains 3.4% by weight of hydrogen chloride and 10% by weight of nitro hydroxy compound. Six hundred milliliters of 10% (by weight) aqueous solution of sulfuric acid are placed in the anode section of the cell. Current is passed through the cell at a current density of 0.2 ampere per square centimeter, as defined, until a theoretical amount—6 faradays per mol—have been passed. The current density is then adjusted to 0.13, and the electrolysis continued until the catholyte gives no positive test with Fehling's solution. The temperature is maintained at about 80-85° C. and the catholyte is agitated throughout the run. Periodically concentrated hydrochloric acid is added to keep the solution acidic as the relatively low initial acidity (3.4%) tends to become reduced as the reaction proceeds. A total of 40 ml. of concentrated hydrochloric acid is used for the entire experiment amounting to 6.1% by weight of the original catholyte solution. The product, tris(hydroxymethyl)aminomethane is

obtained from the catholyte solution as 24.5 g. of the hydrochloride. The material yield is 94%, the current yield 51%, purity, 96%.

EXAMPLE III

Reduction of 2-nitro-2-methyl-1-propanol

In the same cell as in the previous examples, 200 ml. of distilled water, 40 ml. of concentrated hydrochloric acid and 25 g. of 2-nitro-2-methyl-1-propanol are mixed and placed in the cell in contact with a polished lead cathode. The catholyte solution was 9.2% by weight of 2-nitro-2-methyl-1-propanol and 6.2% by weight of hydrogen chloride. The anolyte comprises 600 ml. of a 10% (by weight) aqueous sulfuric acid solution. Current is passed through the cell of a current density of 0.2 ampere per square centimeter until 6 faradays, the theoretical amount, has been passed. The current density is then adjusted to 0.13 and the electrolysis continued until the catholyte solution gives no positive test with Fehling's solution. Throughout the electrolysis the temperature is maintained at approximately 80-85° C. and the solution is agitated.

The product, 2-amino-2-methyl-1-propanol, is obtained in 24.5 g., as the hydrochloride, corresponding to a material yield of 94% and a current yield of 63%. Purity, 97%.

EXAMPLE IV

Reduction of 2-nitro-2-ethyl-1,3-propanediol

In the cell described above, 200 ml. of distilled water, 40 ml. of concentrated hydrochloric acid and 25 g. of 2-nitro-2-ethyl-1,3-propanediol are mixed to form a catholyte solution containing 9.2% by weight of 2-nitro-2-ethyl-1,3-propanediol and 6.2% by weight of hydrogen chloride. The mixture is placed in the cathode section of the cell in contact with a polished lead cathode. Six hundred milliliters of a 10% (by weight) aqueous solution of sulfuric acid are placed in the anode section of the cell. An electric current is passed through the cell at a current density of 0.2 ampere per square centimeter until the theoretical amount, 6 faradays, has been passed. The current density is then reduced to 0.13 ampere, and electrolysis is continued until the solution in the cathode compartment gives no positive test with Fehling's solution. Throughout the electrolysis, the catholyte solution is agitated and the temperature is maintained at approximately 65° C. The product, 2-amino-2-ethyl-1,3-propanediol, is obtained as 21 g. of the hydrochloride. The material yield is 81%, the current yield 44%. Purity, 96%.

EXAMPLE V

Reduction of 2-nitro-1-butanol

In the cell described above, 190 ml. of distilled water are mixed with 30 ml. of concentrated hydrochloric acid and 25 g. of 2-nitro-1-butanol, resulting in a catholyte solution containing 10% by weight of nitro compound and 5% by weight of hydrogen chloride. The mixture is placed in the cathode section of the cell in contact with a polished lead cathode. Six hundred milliliters of a 10% (by weight) aqueous solution of sulfuric acid are placed in the anode section of the cell. An electric current is passed through the cell at a current density of 0.13 ampere per square centimeter, and electrolysis continued at this level throughout the run until the catholyte solution gave no positive test with Fehling's solution. The catholyte solution was agitated throughout the

run, and the temperature maintained at approximately 65° C. The product, 2-amino-1-butanol, is obtained as 22 g. of hydrochloride, representing a material yield of 84%, a current yield of 41%. Purity, 97%.

EXAMPLE VI

Reduction of 2-nitro-2-methyl-1,3-propanediol

In the cell described above, 205 g. of distilled water were mixed with 104.3 g. of sulfuric acid (100% basis) and 100 g. of 2-nitro-2-methyl-1,3-propanediol, resulting in a catholyte solution containing 25.5% by weight of sulfuric acid and 24.4% by weight of nitro compound. The mixture was placed in the cathode section of the cell in contact with a polished lead cathode. Six hundred grams of a 10% aqueous solution of sulfuric acid were placed in the anode section of the cell in which a lead anode is used. An electric current was passed through the cell at a current density of 0.2 ampere per square centimeter until the theoretical amount necessary for reduction has been used, that is, about four hours. The current density was then reduced to 0.13 ampere per square centimeter until the reduction was complete, about an hour additional. The temperature was maintained at about 65-71° C. throughout the run. The catholyte was then evaporated to dryness resulting in a residue of the sulfate of 2-amino-2-methyl-1,3-propanediol weighing 106 grams, corresponding to a 93% yield. The purity, as determined by nitrogen content, was 99%. The current yield was 77.2%.

EXAMPLE VII

Reduction of 2-nitro-2-methyl-1-propanol

The cell described above was used and into the cathode compartment thereof were introduced 289 g. of water, 32 g. of 100% sulfuric acid, and 35 g. of 2-nitro-2-methyl-1-propanol, resulting in a catholyte solution containing 9% by weight of sulfuric acid and 9.8% of 2-nitro-2-methyl-1-propanol. The catholyte mixture was in contact with a polished lead cathode. Twelve hundred milliliters of a 10% solution of sulfuric acid was used as anolyte and placed in the anode section of the cell, in which a carbon anode was used. An electric current was passed through the cell at a current density of 0.13 ampere per square centimeter throughout the duration of the run which lasted for four hours and fifteen minutes when the end point was determined by the Fehling's solution test. The temperature throughout the run was maintained between about 75-79° C. The catholyte solution was evaporated to dryness and the residual product, which was the sulfate of 2-amino-2-methyl-1-propanol, was recovered in an amount equal to 36.6 g., corresponding to a yield of 90.3%. The purity of the amino compound by nitrogen determination was 98.5% and by sulfate determination was 99.9%. The current yield was 50%.

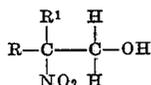
While the above describes the preferred embodiments of my invention, it is understood that departures may be made therefrom within the scope of the specification and claims.

What is claimed is:

1. In the electrolytic reduction of aliphatic nitroalcohols to the corresponding amino compounds, using an electrolytic cell containing an inert anode and an inert cathode suspended, respectively, in anode and cathode compartments separated by a semipermeable diaphragm, the process which comprises preparing an aqueous acid solution selected from a class consisting of

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hydrochloric acid solutions having a concentration ranging from about 1.4 to 10 per cent by weight and sulfuric acid solutions having a concentration ranging from about 9 to 24.4 per cent by weight, dissolving in said acid solution from about 9.2 to 24.4 per cent of a nitroalcohol having the structural formula:



wherein R represents a substituent selected from a class consisting of hydroxymethyl, methyl and ethyl groups and R¹ represents a substituent selected from a class consisting of hydrogen, methyl, ethyl and hydroxymethyl groups, placing the resulting solution in the cathode compartment of said electrolytic cell, placing in the anode compartment of said cell an aqueous acid solution containing from about 10 to 20 per cent by weight of a strong inorganic acid, maintaining the temperature of the cell within the temperature range of from about 50° to 100° C., passing an electric current through said cell from said anode to said cathode having a current density of about 0.2 ampere per square centimeter at least at the start of the process, continuing passing current through the solution until the nitroalcohol is reduced and recovering the resulting aminoalcohol thereby produced.

2. The process of claim 1 wherein the current density at the start of the process is maintained at a value of about 0.2 ampere per square centimeter until about 6 faradays have been passed through the cell and is then reduced within the

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range of about 0.1 to 0.13 ampere per square centimeter until complete reduction of the nitroalcohol has taken place.

GRAHAM W. McMILLAN.

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