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THE DEFINITION OF THE PROCESS OF ELECTROCHEMICAL IMPREGNATION OF NICKEL ELECTRODES

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16. Abstract Electrochemical impregnation was studied during a series of experiments designed to define the optimal conditions for the fabrication of dimensionally stable cell anodes of Ni-H2 and Ni-Cd systems. The influence of various parameters, such as current and duration of electrolysis, temperature and acidity of the chemical bath, the concentrations of Ni and Co as well as the use of ethanol was determined. Results show that the electrochemical impregnation process as defined is industrially feasible and it is suggested that Ni-H2 and Ni-Cd type electrodes be produced in sufficient quantity to further evaluate their performance characteristics.

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ABSTRACT

A parametric study of the process of electrochemical impregnation performed on positive Ni-H_2 electrodes 0.93 mm thick showed that electrodes can be fabricated which are dimensionally stable during the cycling process and which exhibit energy capacities similar to 100% chemically impregnated electrodes.

Comparable results are obtained with Ni-Cd positive electrodes 0.79 mm thick. The electrodes can be fabricated in well defined conditions and in a period of 2 to 3 hours at a temperature close to the boiling point of liquid.

The present results show that the presence of alcohol is necessary for stabilizing the electrodes dimensionally.

A complementary study was performed to define the critical impregnation factors (corrosion of the sintered support, role of the interelectrode distance, bath wear, impregnation bath additive).

A surface-active agent was used as an alcohol substitue and the first tests with this agent were performed at 80°C to fabricate dimensionally stable electrodes exhibiting an energy capacity of about 90% of that of standard chemically impregnated electrodes.

The overall results obtained in the laboratory, within the framework of the contract, show the benefit of the electrochemical impregnation process.

It is now desirable to fabricate an experimental impregnation unit to test the impregnated electrodes in accumulators according to the new process.

DEFINITION OF THE PROCESS OF ELECTROCHEMICAL IMPREGNATION OF NICKEL ELECTRODES

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Table 4.3	50%	"	"	80°C -	"	"	"	"
Table 4.4	50%	"	"	50°C -	"		"	"
Table 4.5	70%	"	"	80°C -	"	"	"	"
Table 4.6	50%	"	"	80°C -	"	"	"	=3M/1
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.Main parameters:

Table 5.1	0%	Alcohol -	Temperature	98°C -	Ph	1.5-2 -	Ni+Co	=2M/1
Table 5.2	20%	Alcohol -	"	84°C -	"	"	Ni+Co	=2M/1
Table 5.3	50%	Alcohol -	"	80°C -	"	"	"	"
Table 5.4	50%	"	"	50°C -	"	"	"	"
Table 5.5	70%	"	"	80°C -	"	"	"	"
Table 5.6	50%	"	"	80°C -	"	"	"	3M/1
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.Main Parameters:

Figure 5.1:	0%	Alcohol	- Temperature	98° C	- pH	1.5-2	- Ni+Co	= 2 M/l
Figure 5.2:	20%	Alcohol	- Temperature	84° C	- pH	"	"	"
Figure 5.3:	50%	"	"	80° C	- pH	"	"	"
Figure 5.4:	50%	"	"	50° C	- pH	"	"	"
Figure 5.5:	70%	"	"	80° C	- pH	"	"	"
Figure 5.6:	50%	"	"	80° C	- pH	"	"	= 3 M/l
Figure 5.7:	50%	"	"	80° C	- pH	3-3.5	"	= 2 M/l

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.Main Parameters:

Figure 6.1:	0%	Alcohol	- Temperature	98° C	- pH	1.5-2	- Ni+Co	= 2 M/l
Figure 6.2:	20%	"	"	84° C	- pH	"	"	"
Figure 6.3	50%	"	"	80° C	- pH	"	"	"
Figure 6.4	50%	"	"	50° C	- pH	"	"	"
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.Main Parameters:

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.Main Parameters:

FIGURE 21.1: 50% Alcohol - Temperature 80° C - pH 1.5-2 Ni+Co = 2M/l

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THE DEFINITION OF THE PROCESS OF ELECTROCHEMICAL IMPREGNATION OF THE NICKEL ELECTRODES

P. Antoine

1. PURPOSE OF THE STUDY /1*

Analyses of the failure of Ni-Cd and Ni-H2 elements brought to light a swelling of the positive electrodes which caused the interelectrode distance to decrease and the separator to dry, thereby resulting in a high internal resistance.

Such results were observed on Ni-Cd VO 10 S accumulators (1) for which the thickness of the positive electrodes increased by about 15% following 600 cycles at 40° C (charged for 7 hours at C/5, discharged for 1 hour at C/3) and 25% following 3,000 cycles at 20°C and 60% DOD.

Analyses of the Ni-H2 elements within the framework of the 75/CNES**/520 contract showed that the swelling of positive electrodes impregnated by 100% may reach 95% of the initial thickness after 3,300 cycles of low orbit type cycles.

A study conducted under CNES contract (2) showed that the electrochemical impregnation process corrected the swelling of the positive electrode and therefore the progression of its porous volume during cycling.

The purpose of the present study is to verify this property of electrochemically impregnated electrodes and to determine the electrochemical impregnation conditions whereby electrodes can be made at the same energy levels as the electrodes impregnated by chemical process.

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*Numbers in the margin indicate pagination in the original text.
**National Space Studies Center.

2. REFERENCE DOCUMENTS /2

-ESTEC Contract No. 3000/76/NL/HP (Sc)
-ECD technical specifications 2/Spec 31
-DT/SAS Report-616/77-PA/JM
-DT/SAS Report - 45/78 PA/JM

3. BIBLIOGRAPHIC RESEARCH

3.1. Facts Observed In the Ni-Cd and Ni-H2 Accumulators

Analyses performed of failures of Ni-Cd elements established a swelling of the positive electrodes causing compression of the separator and its progressive dewatering (drainage).

The excess quantity of electrolyte is then absorbed by the negative electrode as shown in figure 1.

In the case of the Ni-H2 elements, the phenomenon is similar. However, accounting for the hydrophobic nature of the negative electrode, part of the electrolyte initially contained in the separator is absorbed by the positive electrode (figure 2).

Corrections of the separator's (2) drainage was studied under CNES Contract 75/0590 and from this means were selected to limit or suppress the swelling of the positive nickel electrodes during cycling.

The results of this study established the following points:

-The positive nickel electrodes impregnated by chemical means swell progressively during cycling (figure 3).

Swelling may be limited by improving the initial strength of the sintered support (using a sintering of small porosity) or by protecting the support using an oxide layer obtained by passivation treatment.

Under-impregnation limits the swelling phenomenon, but does not suppress it.

Conversely, with the electrochemical impregnation method, electrodes exhibiting a high dimensional stability can be made (appendix 1).

Electrochemical Impregnation Tests

The first electrochemical impregnation tests were executed within the framework of the study on the draining of the separator.

The method consists of forming nickel hydroxide by electrolysis of a nickel nitrate solution in acidic medium, using ethanol as a solvent.

The results obtained by SAFT are:

- -Dimensional stability of electrodes during cycling (figure 3 - appendix 1).
- -Capacities close to standard electrodes impregnated chemically.
- -Little corrosion of sintered support during electrochemical impregnation.

3.3 Theoretical Aspect /4

-Advantage of the Method

Nickel hydroxide is the active substance of the positive electrodes used in the alkaline accumulators (nickel-cadmium, nickel-hydrogen, nickel-zinc).

The operating reaction commonly adopted is:

$$Ni\ (OH)_2 + OH^- \xrightleftharpoons[red]{ox} Ni\ OOH + H_2O + e^-$$

GLEMSER and EINERHAND (4), FEITCKNETCHT (5), TUOMI (6) and BODE (7) established two distinct phases of hydroxides with a valence of (II), i.e.:

α 3 $Ni(OH)_2$, 2 H_2O and β $Ni(OH)_2$

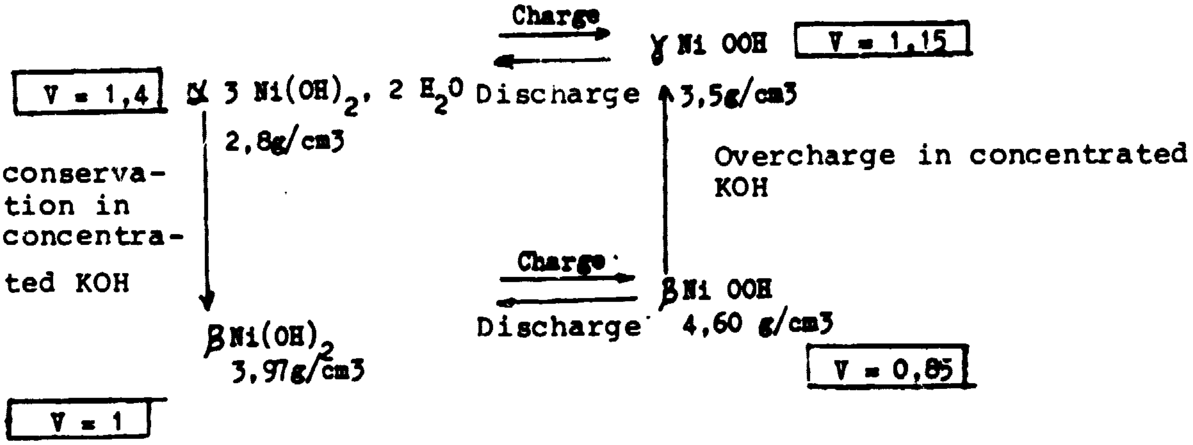
The densities measured from the RX diagrams are 2.82 g/cm3 and 3.97 g/cm3 respectively.

Hydroxide β 3 $Ni(OH)_2$, $2H_2$) is obtained by adding water to the system.

The oxidation of hydroxides with a valence of (II) causes the formation of higher hydroxides exhibiting different distinct phases established by BODE in 1979 (7):

-Phase βNi OOH whose axis C is between 4.6 and 4.8 A or a multiple.
-Phase αNi OOH possessing a C axis dilated by adding sodium or potassium.

The hydroxide transformation diagram proposed by BODE (7), then NESS (8) is the following: /5



Analyses performed by X-ray diffraction showed that the hydroxide $\beta Ni(OH)_2$ is obtained by chemical impregnation, and that a small quantity $\alpha 3\ Ni\ (OH)_2\ 2H_2O$ may come from the corrosion of the sintered support.

The hydroxide $\alpha 3\ Ni(OH)_2$, $2H_2O$ is obtained by electrochemical impregnation according to PICKETT's and PUGLISI's method (2).

It therefore seems that the swelling is due to the structural hydroxide transformations. In the case of chemical impregnation, the initially impregnated hydroxide $\beta Ni\ (OH)_2$ (relative volume 1) is converted into a thermodynamically stable hydroxide *Ni OOH during the overcharge (relative volume 1.15). This results in an increase in the volume occupied by the active substance causing internal stresses which are the cause of the swelling of the electrodes.

In the case of electrochemical impregnation, the hydroxide $\alpha 3\ Ni(OH)_2$, $2H_2O$ formed during the impregnation exhibits the smallest density.

The structural transformation can therefore not be the cause of the swelling during a cycle. This results in a high dimensional stability of the electrochemically impregnated electrodes.

-Principle of the Method /6

Nickel hydroxide is formed by the electrolysis of a nickel nitrate solution in acidic medium.

The sintered nickel plate constitutes the cell cathode.

The OH^- ions formed during nitrate ion reduction precipitate the Ni^{++} ions of the solution in the form of a nickel hydroxide.

The anode is made up of nickel or inert plates or of

---

*Illegible.

soluble nickel plates.

The advantage of the soluble nickel electrodes is to stabilize the concentrations of the Ni^{++} bath by oxidation of the electrode.

According to BEAUCHAMP (9) PICKETT and PUGLISI (10), the reactions to the electrodes are the following:

.<u>At the cathode</u>: $2 H_2O + 2 e \rightarrow H_2\uparrow + 20 H^-$

<u>Possible Reactions (Nitrate Reduction)</u>:

$NO_3- H_2O + e \rightarrow NO_2 + 2 OH^-$

$NO_3- + H_2O + 2e \rightarrow NO_2- + 2 OH^-$

$NO_3- + 2 H_2O + 3e \rightarrow NO + 4 OH^-$

$NO_3- + \frac{5}{2} H_2O + 4e \rightarrow 1/2 N_2O + 5 OH^-$

$NO_3- + 3 H_2O + 5e \rightarrow 1/2 N_2 + 6 OH^-$

$NO_3- + 5 H_2O = 6e \rightarrow N H_2OH + 7 OH^-$

$NO_3- + 5 H_2O + 7e \rightarrow 1/2 N_2H_4 + 8 OH^-$

$NO_3- + 6 H_2O + 8e \rightarrow HN_3 + OH^-$

.<u>At the Anode</u>: $2 H_2O \rightarrow 4 H^+ + O_2 + 4 e$ (inert anode) /7

$Ni \rightarrow Ni^{++} + 2e$ (soluble anode)

In the case of electrochemical impregnations using alcohol as a solvent, the alcohol is oxidized into an acetate: other Kolbe type secondary reactions may occur:

$H_3C - CH_2OH \quad H_3C - CHO + 2H^+ + 2e$
(acetaldehyde)

$H_3C - CH_2OH + H_2O$ * $H_3C - OOOH + 4H^+ + 4e$
(acetic acid)

---

*Illegible.

$$2H_3C - CH_2OH + \rightarrow H_3C - OOO - CH_2 - CH_3 + 4h^+ + 4e$$

$$H_3C - CH_2OH + H_3C - cooH \rightleftarrows H_3 - coo - C_2H_5 + H_2O$$

If we consider these equations, we see that for the passage of an electron, the quantity of NO_3 - consumed is always less than 1. Conversely, 1 to 2 OH^- may be formed.

For the same quantity of current, 0.1 to 1 gas molecule may be formed in the cavities of the sintered support. At 80°C (impregnation temperature), the volume occupied by one millimole of gas reaches 27 cm3.

H. SEIGER and C.J. PUFLISI (11) showed the important role which the gas formation in pores of the sintering could play.

The closed gas is most probably ammonia. KANDLER (12) and HAUSLER (13) confirmed this hypothesis.

In this case, H. SEIGER and V.J. PUGLISI (11) propose the following process.

The nitrate reduction occurs according to the equation:

$$NO_3- + (6 - x)H_2O + x\ H^+ + 8e \rightarrow NH_3 + (9 - x)\ OH-$$

When pH - 6.4, the nickel hydroxide $Ni(OH)_2$ precipitates. When pH - 8.4, the reaction is considered quantitative to pH = 13.5, the partial pressure of NH_3 reaches 1 atm.

The gas expels the liquid from the cavity. Then, when it is again in contact with the acid solution, dissolution occurs: $NH_3 + H^+ \rightarrow NH_4+$.

The vacuum thus created in the cavity causes a new solution to enter.

If an impregnation solution containing 3 M/l $Ni(NO_3)_2$ is used, the process indicated above will occur 7 times for it to be filled with the required amount of active substance. NH_3 is formed when the used solution evacuates from the pores and when hydroxide is not being produced.

3.4. Bibliographic Results /5

Numerous patents (15) to (35) describing the preparation of nickel hydroxide in the various parametric conditions according to the principle ofelectrolysis of a nickel nitrate solution.

The Most elaborate works known today are those of BEAUCHAMP (9) and D.F. PICKETT H.N. SEIGER and C.J. PUGLISI (3), (10), (11), (14).

However, BEAUCHAMP's works essentially account for the negative electrochemical impregnation ($Cd(OH)_2$ formation).

Two methods are proposed:

a. Method Using an Aqueous Solution:

The process is recommended by BEAUCHAMP (9).

The impregnation takes place using a nickel nitrate solution wihtout adding alcohol to the impregnation bath.

The impregnation conditions described by D.F. PICKETT (10) are:

nickel nitrate $Ni(NO_3)_2$ = 1.8 M/l
cobalt nitrate $Co(N0_3)_2$ = 0.2 M/l
sodium nitrate N_aNO_2 = 0.5 M/l
initial pH = 4

The current densities applied are on the order of 40 to 80 mA/cm2 per electrode.

b. Method Using an Alcoholic Solution (Denatured Alcohol)

The tests were carried out by D.F. PICKETT (3), (10), (11), (14). The impregnation conditions described were:

nickel nitrate = $Ni(NO_3)_2$ = 1.8 M/l
cobalt nitrate = $Co(NO_3)_2$ = 0.2 M/l
denatured ethanol concentration = 50%
pH = 1.5 - 2
Bath temperature: ≈ 80°C.

The current densities applied were on the order of 40 to /9
80 mA/cm2 per electrode.

The results obtained (3), (10) establish the following points:

-The electrochemical impregnation with or without alcohol makes it possible to prepare electrodes with an equivalent and comparable filling concentration to that of a standard chemical impregnation (hydrate weight - 1.9 to 2.2 g/cm3 vacuum).

-The dimensional stability of the 2 types of electrodes (11) seems to be appreciably identical in the following test conditions:

1st cycle: Charge 15 hours at C/10
Discharge at 1 A/dm2 for 2 hours.

6 cycles: Charge 2 hours at 1 A/dm2
Discharge 2 hours at 1 A/dm2

8 cycles (in cell): Charge 15 hours at 2 A/dm2
Discharge 15 hours at 2A/dm2

On the other hand, the dimensional cycling stability of electrodes impregnated without alcohol does not seem to be clearly demonstrated (10).

-The electrodes impregnated in an alcoholic solution present a thickness increase during the impregnation at the end of the filling (3). It would actually seem that a maximum filling concentration exists above which the swelling probability increases.

-An increase in the initial porosity of the sintered support improves the quantity of the impregnated hydroxide and the energy capacity of the electrodes.

3.5. Guidelines For the Experimental Study

The purpose of the present study is to optimize the method of electrochemical impregnation and to establish the definition of a pilot electrochemical impregnation unit.

We will therefore define the conditions for making Ni-Cd and Ni-H2 type Nickel electrodes with dimensional stability during cycling and whose energy characteristics are close to the standard chemically impregnated electrodes.

The parameters defining the fabrication of such electrodes /10 are of 2 types:

a. Nature of the Sintering:

-thickness
-porosity
-preliminary treatment.

b. Impregnation Conditions:

- concentration of the impregnation bath,
- temperature of the impregnation bath,
- bath acidity,
- presence of additives in the bath (alcohol, cobalt),
- current density,
- electrolysis time.

c. Nature of the Sintering:

-Thickness:

An increase in the initial thickness of the sintering can make it more difficult to obtain the correct active substance filling due to the obturation of the pores and precipitation of the hydroxide on the surface, particularly when the electrolysis occurs with a high current density.

The parametric study of the impregnation is conducted on thick Ni-H2 type electrodes (0.92 mm thick).

-Porosity:

A high porosity of the sintered support is equivalent to a larger vacuum volume, i.e. to a larger volume available for the hydroxide precipitation.

The electrode filling concentration should therefore be improved. However, the firmness of a support with high porosity is mediocre and the destruction of the support under the effect of internal stresses is that much easier. The dimensional stability of such a support during the electrical tests is guaranteed by the process.

The impregnation tests are executed with sintered supports of high porosity.

d. Impregnation Conditions - Concentration of the Impregnation Bath /11

A modification of the nickel concentration in the bath may have an influence on the crystallite formation and kinetics ics and may therefore change the rate of diffusion of nickel and of the concentration gradient in the sintering.

2 concentration levels were tested during the study:

-Temperature of the Impregnation Bath:

The bath temperature is a factor whose influence must be similar to that of the bath concentration.

In order to improve ion diffusion, the tests are carried out at a temperature similar to boiling point of the bath.

An impregnation test is carried out at a lower temperature.

-Bath Acidity:

The H^+ ions duffuse more rapdily than the Ni^{++} ions. For high acid concentrations in the bath, the H^+ ions can decrease the efficiency of the treatment in terms of the equation for nickel hydroxide formation.

$$NO_3^- + (6 - x)\ H_2O + x\ H^+ + 8e \rightarrow NH_3 + (9 - x)\ OH^-$$

For the passage of an electron, we have:

$$\frac{9 - x}{16}\ Ni^{++} + \frac{(9-x)}{8}\ OH^- \rightarrow \frac{9-x}{16}\ Ni(OH)_2$$

An increase in the H^+ quantity in the bath should therefore decelerate the rate of hydroxide precipitation and possibly modify morphology of the deposit.

The tests are performed for two acid concentrations in the bath.

-Presence of Additives In the Bath: /12

.Cobalt:

Cobalt improves the chargeability of the positive nickel electrodes by increasing the overvoltage with oxygen. It is usually present in 5% concentrations in chemically impregnated electrodes.

During the impregnation tests, the amount of cobalt syncrystallizing with nickel in the electrode will be determined as a function ofthe initial cobalt concentration in the bat and the bath acidity.

We will aslo try to make electordes whose cobalt concentrations with respect to the amount of active substance will be about 5%.

.Surface-Active

Additives having the property of decreasing the surface tension of the bath may have an effect on the ion diffusion rate and may thus increase the amount of hydroxide impregnated by a more uniform filling of the electrode.

Ethanol may have a similar effect. Impregnation tests will be performed with different alcohol concentrations.

Other surface-active agents will be tested.

-Current Density

Current density is the factor which has a direct influence on the OH^- ion concentration at the level of the pores of the sintering and influences the type of seeds formed, i.e. the type of deposition of the nickel hydroxide.

Moreover, a too high current density may favor a surface deposit and the obstruction of the pores limiting the filling concentration.

3 current densities, compatible with the manufacturing procedure and selected according to the bibliographic results, were tested.

-Electrolysis Time

The electrolysis time conditions the filling concentration of nickel hydroxide and therefore the electrode capacity.

At the beginning of impregnation, the amount of impregnated hydroxide for a given current density and for given impregnation conditions is proportional to the impregnation time, it then stabilizes and no longer changes when there is a surface deposition.

For each current density, three electrolysis phases will be /13 used. These phases will make it possible to determine the maximum amount of hydroxide formed in the defined impregnation conditions.

4. EXPERIMENTAL STUDY

4.1. Experimental Conditions

The experimental study was performed in accordance with the manufacturing and control diagram shown in appendix 3.

4.1.1. Preparation of the Sintered Support

The sintered support is fabricated by coating a nickel powder paste suspended in carboxymethylcellulose gel over a perforated ribbon.

After the binding agent decomposes at high temperatures in the presence of air, the sintering occurs at about 1,000°C under reducing atmosphere.

.Controls:

-Real Porosoity:

The porosity of the plates used for electrochemical impregnation is about 85%.

This percentage expresses the ratio of the vacuum volume to the apparent volume of the sintered nickel:

E = Thickness in 10^{-2}mm of sintered strip
e = Thickness in 10^{-2}mm of perforated nickel-coated ribbon
M = Mass in grams of 10 cm2 sintered strip sample
m = Mass in grams of the perforated nickel-coated ribbon sample (10 cm2).

11.2 = 100/real density of nickel

$$\% \text{ Porosity} = 100 \left(1 - 11.2 \frac{M - m}{E - e}\right)$$

-Accessible Porosity:

It expresses the accessible vacuum volume per unit of sintered mass.

It is determined by the water mass absorbable by the sintering.

-Firmness of the Sintering:

This measurement consists of using a penetrometer to determine the force required to perforate the sintered nickel with a determined needle.

-Thickness: /14

The thicknesses are measured on 4 points using a contact surface palmer equal to 0.5 cm2.

-Weight:

The sintered strips are weight to the nearest 1 mg.

4.1.2. Treatment of the Sintered Surface

Treatment of the sintering prior to impregnation is achieved with sinterings which have undergone a preliminary surface treatment.

4.1.3 Impregnation

The equipment consists of an electrolysis circuit described by appendix 4.

The photographies of appendices 5 and 6 represent the impregnation installation and the electrode support used.

4.1.3.1. Cell Composition

55 liter tank.

-Cathode: sintered plates (S_u = 0.58 dm2) situated half-way between the 2 counter-electrodes.

-2 soluble, pure nickel counter-electrodes
-Electrode/counter-electrode distance = 19 mm
-Teflon electrode support.

Agitation: thermomix pump flow rate: 13 l/mn.

4.1.3.2. Impregnation Conditions

The impregnation conditions are defined by the test grid of appendix 7.

-Nature of the sintered support

Sintered type Ni-H2 of high porosity.
-Thickness: 0.91 ± 0.03 mm
-Porosity: 86%
-Surface treatment: reduction.

-Bath concentration /15

2 concentrations are tested:
2 moles/l $Ni(NO_3)_2$ + $Co(NO_3)_2$ including 0.1 mole/l $Co(NO_3)_2$
3 moles/l $Ni(N)_3)_2$ + $Co(NO_3)_2$ including 0.1 mole/l $Co(N)_3)_2$

Bath temperature: 98°C - 84°C - 80°C - 50°C.

-Alcohol Concentration: 0 - 20 - 50 - 70%

The alcohol concentration indicates the percentage of alcohol with respect to the volume of nickel nitrate solvent.

-pH (indication of the calomel electrode)
2 pH zones:

pH 1.5 to 2
pH 3 to 3.5

Current density: 50 - 75 - 95 mA/cm2
Amount of current: 540 - 630 - 720 c/cm2

.Controls during impregnation:

-Ni and Co concentration:

The concentration is dosed by atomic absorption spectrophotometry.

-Alcohol Content:

Two measurements are used to control the alcohol concentration in the bath:

Density:

The calibration curve of the bath's density as a function of the alcohol concentration is given in appendix 10 for an $Ni(NO_3)_2+Co(NO_3)_2$ = 2 M/l concentration.

Surface Tension

Ethanol reduces the surface tension of the bath (appendix 9).

The surface tension is quickly measured using a DUCLAUX dropper or stalagmometer.

The measurement consists of determining the number of drops produced at 20°C to permit the flow of a given volume of solution.

The Surface Tension of the Liquid: $T = To\ D. \frac{N}{D}$ /16

T = Surface tension of liquid at 20°C (in dynes/cm)
To = Surface tension of water at 20°C = 72.7 dyn/cm
D = Density of analyzed solution
N = number of water droplets produced at 20°C
n = number of solution drops produced and analyzed at 20°C.

The calibration curve of thenumber of drops produced using the DUCLAUX dropper for a solution containing various alcohol concentrations is given in appendix 10.

-Bath Temperature:

The required temperature is regulated to the nearest ±0.5°C.

-Bath Acidity

The bath's acidity is controlled continuously to a pH meter using a calomel electrode.

The quantity of acid to be poured into the initial nitrate solution used for impregnation in order to obtain the same "pH" indication is appreciably identical in an aqueous and alcohol solution, i.e.:

approximately 5.10^{-3} H^+ to reach pH 3-3.5
approximately 0.5 H^+ to reach pH 1.5-2

The "pH" is controlled to the nearest ±0.3 unit.

-Current Density:

It is established with respect to the apparent sintering surface to be impregnated.

The current intensity is read to the nearest 1%.

-Electrolysis Time:

The time is controlled using a chronometer. The error range for the impregnation time is ±3 mn.

-Potentials:

The potential of the sintered support is recorded with respect to a platinum electrode during the impregnation.

-Bath Volume

A steady control makes it possible to maintain the bath volume constant to the nearest 1%.

4.1.4. Electrochemical Cleaning /17

The electrochemically impregnated plates are cleaned in the following manner:

-Electrode: impregnated plate
-Counter-electrode: negative cadmium
-Separator: miovyl
-Electrolyte: KOH 30%
Charge: 1 h at 6 A/dm2
Discharge at 1.5 A/dm2 up to OV/Hg-HgO

After cleaning, the plates are washed and rinsed in permuted water, then dried under nitrogen and preserved in an air-free environment.

-Controls Performed After Electrochemical Cleaning

-Weighings
-Thicknesses.

On a few specimens:
-Quantity of impregnated hydroxide
-Quantity of corroded nickel
-Cobalt concentration.

These analyses are performed by dissolutio n of the hydroxide in an acetic buffer, then dosage of Ni and Co in solution.

The weight of the residual support makes it possible to determine the corrosion of the sintering during impregnation.

4.1.5. Cycle Testing

After electrochemical cleaning, the plates undergo 4 growth (formation) cycle in the following manner:

-electrode: impregnated plate (0.58 dm2)
-separator: myovil
-electrolyte: KOH (d = 1.306).

Formation:

1st cycle: Charge: 15 hours at C/5 (C = 4.5 Ah)
Discharge at C/2 - Suspension at 0 V/Hg-HgO

2nd Cycle: Charge 6 hours at C/2
Discharge at C/2 - Suspension at 0 V/Hg-HgO

3rd cycle: Charge 4 hours at C/2
Discharge at C/2 - Suspension at 0 V/Hg-HgO

Controls Performed During Growth

The capacity restored by each electrode is determined for each growth cycle.

After the 4th cycle, the following controls are performed:

-Appearance
-Thickness
-Capacities.

Cycling:

For certain electrodes, elements of approximately 3 Ah are formed with noncompacted plate clusters.

Cycling Characteristics:

-Excess negative capacities,
-Separator: miovyl
-KOH electrolyte (d - 1.306).

Duration: 20 cyclces minimum
Charge: 6 hours at C/5
Discharge: 2 hours at C/2 or suspension at 1 V/element.

Control: - thicknesses after cycling
- capacities
- appearance.

4.2. Experimental Results

The test series was performed with Ni-H2 type electrodes (thickness = 0.91 ± 0.03 mm).

In order to make an accurate comparison with chemical impregnation, Ni-H2 type 100% chemically impregnated electrodes (Hydrate

= 14.5 ± 1.5 g/dm2) and current standard underimpregnated electrodes (Hydrate = 10.9 ± 1.5 g/dm2) were subjected to electrical tests under ths same conditions.

The electrochemical impregnation conditions required for reaching the defined objective being defined, a batch of 100 Ni-H2 electrodes was prepared to determine the discrepancy of the method. These electrodes were formed according to specification 340 115. Their energy characteristics were compared with the results of earlier fabrications of positive electrodes for Ni-H2 space elements.

Characteristics of the Sinterings Used /19

4 types of sintering were fabricated (Tables 1 and 10).

2 standard sinterings for the chemical impregnation of the electrodes used in the Ni-H2 and Ni-Cd elements.

2 high porosity sinterings for the electrochemical impregnation of electrodes for Ni-H2 and Ni-Cd elements.

These established characteristics characterize the vacuum volume which is accessible and capable of being filled with an active substance.

Appendix 12 shows the structure of the Ni-H_2 type high porosity type sintering observed in a scanning microscope.

Characteristics of the Impregnated Electrodes

The characteristics of the chemically impregnated electrodes used as reference are shown in table 2.

The characteristics of the electrodes electrochemically impregnated in compliance with the test grid of appendix 7 are shown in tables 3 to 6.

Appendix 13 shows the structure of the Ni-H_2 type sintering after electrochemical impregnation as observed in a scanning microscope.

The electrochemical impregnation method is defined as a function of 4 essential parameters:

-Filling
-Capacities (formation, cycling)
-Hydrate yield
-Electrode swelling.

4.2.1. Filling of the Electrodes

The filling curves, expressing the weight variation during impregnation, are transcribed on figures 5.

At the beginning of impregnation, the filling is achieved proportionally to the electrolysis time (figure 5.6.), then stabilizes progressively to reach a maximum value when the quantity of current is approximately 600 C/cm2 per impregnated plate.

This value varies with the impregnation conditions (figures 5) shown on the next page.

-Influence of the Alcohol Concentration

Tests were conducted at temperatrues close the the boiling point for a water-alcohol mixture (appendix 8).

In an alcoholic solution, one sees (figures 11 and 12) a decrease in the filling capabilities when the alcohol concentration increases between 50 and 70%. This phenomenon is probably due to

Maximum Filling Obtained (ΔP in g)

Current Density (mA/cm2) Impregnation Conditions			
Alcohol Conc. 0% - 98°C - Ni+Co=2M/l pH-1.5-2	14	12	14
" " 20% - "84°C - " = " "	11.5	11	10
" " 50% - "80°C - " " "	13	12	11
" " 50% - "50°C - " " "	> 9	10	10
" " 70% - "80°C - " " "	11	11	11
" " 50% - "80°C - " 3M/l "	11.5	11.5	11.5
" " 50% - 80°C - " 2M/l 3-3.5	11.5	8.5	8.5

the large increase in the bath viscosity thus limiting diffusion and promoting hydroxide deposition on the surface.

For concentrations varying from 20 to 50% alcohol, the quantities of impregnated hydrate are appreciably identical to the various current densities at impregnation temperatures close to the boiling point (figures 52 and 53).

In an aqueous solution, the given hydroxide quantities are greater than the quantities obtained in an alcoholic solution, and this is probably partially due to the large corrosion on the sintered support (table 3).

-"pH" Influence

The tests performed in an alcoholic solution (50% ethanol) for 2 "pH" zones; "pH" 1.5-2 and "pH" 3-3.5 showing a decrease in the filling concentration on the order of 10 to 15% when the "pH" is within the zone 3-3.5 (figures 53 and 57).

This phenomenon is probably due to a decrease in the corrosion on the sintered support when the acidity of the solution is lower (table 3) and to a more rapid surface deposition causing an obturation of the pores of the sintering.

-Temperature Influence /21

When the bath temperature is low with respect to the boiling point, there is a decrease in the filling and this is probably due to the fact that the diffusion phenomena are limited.

The filling at low current density (50 mA/cm2) therefore seems extremely slow (figures 53 and 54).

-Influence of the Ni-Co Concentration in the Bath

The tests were performed for 2 different concentrations Ni+Co = 2 Moles/l and 3 Moles/l.

It seems that the maximal filling rate is not affected by a variation in the salt concentration of the bath (figures 53 and 56).

However, the filling seems to be slower for low current densities (50 mA/cm2 when the Ni + Co concentration in the bath is low.

This observation seems to correlate well with the diffusion limitation phenomenon.

-Influence of the Current Density

The trend is a relatively large improvement in the filling rate when the current density decreases. This improvement is about 10% when the current density decreases from 95 to 75 mA/cm2 (figure 11).

In all cases, the maximum filling is obtained when the quantity of current reaches about 600 c/cm2 per plate.

The end of filling is indicated by an abrupt variation in the potential of the plate measured with respect to an electrode Pt (figures 51 to 54). We may therefore consider the absence of Ni^{2+} ions in the support.

A surface deposit then occurs and dissolves again on contact with the acid solution each time it forms.

The residual surface deposit, visible at the end of impregnation detaches during the electrochemical cleaning.

The end of filling signal is visible for high current densities and is virtually nonexistent when the current density is low, i.e. 50 mA/cm2 (figures 51 to 54).

4.2.2. Electrode Capacities (Formation - Cycling) /22

The Ni-H2 type electrode capacities are compared to the standard chemically underimpregnated and chemically 100% impregnated electrodes (Table 2).

The capacities of electrochemically impregnated electrodes in various impregnation conditions are presented in tables 4.

The variation of the capacities during formation is transcribed in figures 7.

In the case of chemically impregnated electrodes, the capacity increase is progressive: about 19% after the 4 formation cycles.

In the electrochemically impregnated electrodes, the capacity increase is relatively large between the 1st and the 2nd cycle. It may reach 10%, then stabilize during the next cycles (figure 16).

A capacity loss can sometimes be noted between the first cycle and the 2nd cycle. This fact might be due to a loss of the active surface substance. This observation is made on several impregnated electrodes in a bath of high alcohol concentration, i.e. 70% (figure 7.5) and in a bath with a high Hi + Co concentration (figure 7.6).

The influence of the impregnation parameters on the capaciites seems to be very similar to the influence on the filling of the electrodes (figures 6):

Maximum Capacities Obtained (Ah/dm2)

Impregnation Conditions	Current Density (mA/cm2) 50	75	95
Alcohol content 0% - 98°C - Ni+Co=2M/l - pH 1.5-2	4.3	4.0	4.4
" " 20% - 84°C - " " " "	4.2	4.1	3.5
" " 50% - 80°C - " " " "	4.2	3.8	3.7
" " 50% - 50°C - " " " "	2.3	3.5	3.2
" " 70% - 80°C - " " " "	3.5	3.3	3.3
" " 50% - 80°C - " 3M/l " "	4.1	3.6	3.4
" " 50% - 80°C - " 2M/l " 3.3-5	4.1	3.5	2.5

-Influence of the Alcohol Content /23

.In an alcoholic solution, we observe a definite decrease in the capacities of the impregnated plates in a highly alcoholic medium (70% ethanol - figure 12). This observation is especially true when the electrodes were impregnated with the lowest current densities.

For lower alcohol contents (20% to 50%), the specific capacities are appreciably similar and appreciably reach the capacities of 100% chemically impregnated plates (figures 6.2- 6.3.-6.7.).

.In an aqueous solution, the capacities obtained may be 5 to 10% greater than the capacities of the 100% chemically impegnated electrodes (figure 6.1.). This property is due to the corrosion of the sintered nickel support which is the cause of the formation of the supplementary active substance.

-Influence of the "pH"

The decrease in the filling defined in section 4.2.1. is shown by a decrease in the capacity which increases with the current density during electrolysis (figure 6.7.). This fact confirms that a too high "pH" is not favorable for the electrode energy capacities due to the surface deposit of the hydrate.

-Temperature Influence

The filling decrease observed during an electrolysis at low temperatures results in low electrode capacities (figure 6.4).

-Influence of the Ni + Co Concentration in the Bath

It seems that the filling is comparable when impregnation is performed at 2 Moles/l or at 3 Moles/l Ni+Co, it also seems that the electrode capacities achieved are noticeably identical (figures 6.3 and 6.6).

-Influence of the Current Density

Compared to the observation made of the filling rate, it seems that the maximal electrode capacities achieved in the various impregnation conditions descrease wehn the electrolysis current density is too high (i.e. 95 mA/cm2) (figure 12). However, it seems that the electrode capacities achieved with current densities on theorder of 50 mA/cm2 to 75 mA/cm2 are appreciably identical in alcoholic baths containing 20 and 50% ethanol.

4.2.3. Electrode Yield

The electrode yield expresses the capacity obtained during the 4th formation cycle per gram of impregnated substance (variation of the electrode weight before and after impregnation), and this does not take into account the corrosion on the sintered support.

Figures 8 show the variation of the hydrate yield as a function of the impregnationtime and of the current density applied during electrolysis. It seems (figure 13) that the yield obtained during the 4th formation cycle depends on the quantity of the impregnated hydrate and reaches its corresponding maximum of 11 to 12 g/dm2 of the electrode weight variation during impregnation.

This maximum may be explained as follows: when the support is underimpregnated, there is no electrode swelling, and therefore, the capacity obtained corresponds to the oxidation of the impregnated active substance.

When the impregnation is complete, there is probably an electrode swelling during the electrochemical impregnation (see 4.2.4.) at the beginning of an excess capacity and an increase in the "apparent" yield.

Conversely, when the filling reaches its optimal value, i.e about 12 g/cm2, it is probable that the supplementary hydrate is deposited on the surface and becomes detached from the support during the formation cycle. Therefore, compared to the initial hydrate weight determined, the electrode yield decreases (figure 13).

This observation demonstrates that the weight variation during impregnation should be on the order of 12 g/dm2 in order to make Positive Ni-H2 type electrodes with a high enough energy and without forming a surface deposit.

4.2.4. Variation of the Electrode Thicknesses

The electrode thicknesses prior to impregnation, before and after formation are shown in tables 5.

.Swelling Prior to Formation:

The thicknesses of chemically impregnated electrodes measured before impregnation and after electrochemical cleaning are appreciably identical. This is also true for the electrodes electrochemically impregnated in an aqueous solution.

Conversely, the electrodes electrochemically impregnated in an alcoholic solution exhibit a thickness increase which may reach 10 to 15% of the initial thickness, according to the amount of impregnated hydrate.

The "apparent" absence of modifications in the thickness of electrodes chemically or electrochemically impregnated in an aqueous solution may be due to a thickness decrease caused by corrosion of the sintering during impregnation and compensated by a swelling similar to that observed with electrodes electrochemically impregnated in an alcoholic solution after electrochemical cleaning.

.Swelling During Formation and Cycling:

Chemically impregnated electrodes swell rapidly during the first formation cycles, then continue to swell progressively during cycling.

The amount of swelling of 100% chemically impregnated electrodes reaches 60% after 200 cycles for Ni-H2 type electrodes (figure 17).

Figure 18 shows that the overall electrode swelling after formation depends on the amount of hydroxide impregnated. It is inexistent up to a minimum value of the impregnated hydroxide quantity (figure 18), i.e. capacity (figure 19), then increases exponentially for any impregnation condition (chemical or electrochemical) and for any corrosion on the sintered support.

However, the following fundamental difference exists: impregnation in an alcoholic medium stops the swelling process (figure 17) which is not the case of impregnations in an aqueous medium at "pH" 1.5-2.

Alcohol may be the cause of a special crystalline form of the hydroxide, the latter being able to temporarily contain a certain number of alcohol molecules in its system.

The support strength or the crystalline nature of the hydroxide may be an important factor in stopping the swelling process of electrodes impregnated in an aqueous solution.

Thus, an impregnation in an aqueous solution at a higher pH (pH = 3-3.5) might improve the strength of the impregnated support and thus stop the swelling process observed during the cycling.

Based on present knowledge, it seems desirable to use alcoholic baths to assure the dimensional stability of electrodes.

4.2.5. Secondary Parameters

-Hydration of the Hydroxide

A factor which may have an influence on the yield of the active substance is the hydration rate of the hydroxide $Ni(OH)_2$, x H_2O.

If we call the stoechiometric coefficient

$$S = \frac{M\ Ni(OH)2,\ xH20}{actif(Ni+Co)}$$

$$S = \frac{\Delta P+Ni\ corroded}{actif(Ni+Co)}$$

The hydroxide yield should increase with the value of S due to a better hydration.

This property seems to be confirmed by the curve in figure 14 giving the variations of the hydroxide yield.

.Cobalt Concentration in the Electrodes

The cobalt concentration present in the chemically impregnated electrodes is on the order of 5%.

The tests performed with $Co(NO_3)_2$ + $Ni(NO_3)_2$ solutions containing 2M/1 and 5% cobalt and various alcohol concentrations (pH = 1.5-2) causing a concentration of 6 to 8% cobalt with respect to the weight of the impregnated active metals in the electrode (figure 15A).

When the cobalt concentration in the solutionis half, i.e. 2.5%, it is also half in the impregnated electrode at pH - 1.5-2, i.e. 3 to 4% (figure 15B).

However, it seems that a decrease in the acidity of the impregnation bath favors a decrease in the impregnated cobalt concentration.

In conclusion, the impregnated cobalt concentration seems to be:

-Proportional cc the cobalt concentration in the impregnation bath at a given pH.
-Dependent upon the bath acidity.
-Not very dependent upon the presence of alcohol in the bath.

-Corrosion of the Support /27

The presence of alcohol makes it possible to decrease the corrosion of the sintered support by more than 50% (Table 3).

The corrosionof the support seems to be smaller when the bath acidity decreases.

These properties may explain the origin of the alcohol influence discussed in section 4.2.4.

4.2.6. Fabrication of Positive Ni-H_2 Type Electrodes

Based on the results obtained, one hundred electrodes were prepared without accounting for cost requirements in order to verify the dispersion of the method and to control the characteristics obtained compared to chemically impregnated electrodes.

The impregnation conditions were the following:

-alcoholic solution containing 50% ethanol
-"pH" acidity 1.5-2
-Ni + Co concentration = 2 M/l
-Co concentration = 2.5%
-impregnation temperature = 80 ± 0.5°C
-current density: 50 mA/cm2
-impregnation time: 4 hours.

After the electrochemical cleaning defined in section 4.1.4., the electrodes were formed in clusters of 10 in accordance with specification no. 340 115 (formation in a free electrolyte of positive clusters of Hr 23S type accumulators):

1st cycle: Charge 24 hours at C/10
Rest 1 hour
Discharge up to 0.8 V at C/2
2nd cycle: Charge 5 hours at C/4
Rest 1 hour
Discharge at C/2 up to 0.8 V
3rd cycle: Charge 5 hours at C/4
Rest 1 hour
Discharge up to 0.8V at C/2

The thickness and capacity results of these electrodes are shown in table 7.

Compared to the previous fabrication tests of 100% chemically impregnated Ni-H2 electrodes, the energies restored are similar:

	Formation - Capacities Ah/dm2			Ah/g plate
	1st cycle	2nd cycle	3rd cycle	3rd cycle
Electrochemically impregnated electrodes	4.0 ± 0.1	3.5 ± 0.1	3.6 ± 0.1	≃ 0.29
100% chemically impregnated electrodes	3.8 ± 0.1	3.6 ± 0.1	3.7 ± 0.2	≃ 0.27
Standard underimpregnated electrodes	2.9 ± 0.1	2.8 ± 0.1	2.75 ± 0.1	≃ 0.23

These results show that the specific capacities of the electrochemically impregnated electrodes are appreciably identical to the 100% chemically impregnated electrodes and about 30 to 35% greater tahn the standard underimpregnated electrodes.

From the standpoint of density, these electrodes have greater energy than the 100% chemically impregnated electrodes due to the greater yield obtained with the electrochemically impregnated hydroxide.

Finally, the discrepancy in the results obtained with the 100 electrodes is small, since it is on the order of 2 to 3%.

The electrochemical impregnation process therefore seems to be reliable, since dimensionally stable electrodes can be prepared with a much energy as the 100% chemically impregnated electrodes and with little fabrication discrepancy.

4.3 SUMMARY /29

The study of the definition of the electrochemical impregnation parameters demonstrated the feasibility of making thick Ni-H2 type nickel el-ctrodes exhibiting an energy capacity of about 95 to 100% of chemically impregnated electrodes and a high dimensional stability during cycling.

The main parameters defined during this study are:

.Role of Alcohol:

The impregnations performed in the baths, containing 20 to 50% alcohol assure the fabrication of dimensionally stable electrodes and with an energy capacity of about 95 to 100% of the chemically impregnated electrodes.

The presence of alcohol in the impregnation bath decreases the filling rate, but assures dimesnional stability during the cycling of electrochemically impregnated electrodes. It actually seems that the electrochemically impregnated electrodes in an aqueous medium swell during cycling. Thus, such electrodes exhibiting a specific capacity identical to the standard Ni-H2 electrodes swelled about 35% after 200 cycles.

Observation of the electrode thickness variation curves shows that the swelling of the impregnated electrodes (chemically or electrochemically) in an aqueous medium is progressive and continuous. Conversely, the electrodes impregnated in an hydro-alcoholic medium are dimensionally stable during cycling. Thus, the electrodes impregnated chemically in the presence of alcohol do not exhibit thickness increases during cycling after undergoing the formation cycles.

Alcohol probably has two types of roles:

-Decrease in the corrosion of the sintering and thus assuring a greater firmness of the impregnated support.

-Modification of the structure of the impregnated hydroxide (crystal size and shape).

This role is probably the cause of the suspendion of the swelling process observed with the electrodes impregnated in an aqueous medium.

The study of the critical impregnation parameters (section 6.4) tries to determine the role of alcohol.

.Electrolysis Current: /30

A decrease in thefilling is observed when the electrolysis current density is too high, i.e. 95 mA/cm2.

The best fillings are obtained for low current densities, i.e. 50 to 75 mA/cm2 and they assure the fabrication of electrodes with capacities comparable to chemically impregnated electrodes.

The impregnation time must be such that the amount of current used for electrolysis be greater than 600 C.cm2. A surface deposit then forms which is eliminated during the electrochemical cleaning. An observable potential jump corresponds to the formation of this deposit, particularly for high current densities (75 and 95 mA.cm2).

.Temperature of the Impregnation Bath

The tests were carried out at a temperature close to the mixture's boiling point.

It seems that a temperature decrease of the impregnation bath causes a decrease in the filling rate in the electrodes. This phenomenon is probably due to the decrease in the diffusion of Ni^{++} ions in the sintering.

.Bath Acidity:

The tests were carried out in two acidity conditions: "pH" 1.5-2 and "pH 3-3.5. This pH corresponds to the indication for calomel electrodes.

A decrease in the bath acidity causes a decrease in the filling and therefore in the capacities restored, particularly when the impregnation occurs under high current densities.

This phenomenon is due to a large rise in the pH on the plate surface, causing the surface hydroxide to precipitate and an obstruction of the sintering pores.

The optimal "pH" zone in an hydro-alcoholic medium therefore seems to be "pH" 1.5-2.

.Ni + Co Concentration in the Bath:

The tests were performed for concentrations of Ni + Co concentrations fo 2 M/l and 3 M/l.

The filling results are greater for an Ni + Co concentration in the bath equal to 3 M/l. However, the impregnated electrode capacities are similar in the 2 cases with a slight superiority of about 5 % for the impregnated electrodes in the bath containing 2 M/l Ni + Co.

.Cobalt Concentration in the Bath:

The impregnated cobalt concentration in the plate is a function of the cobalt concentration in the bath and of the bath acidity.

In an hydro-alcoholic medium at "pH" 1.5-2, the cobalt concentration in the bath must be on the order of 3% to fabricate electrodes containing about 5% cobalt which are comparable to the chemically impregnated electrodes.

The characteristics defined above made it possible to fabricate one hundred Ni-H2 type nickel electrodes with specific free electrolyte capacities identical to the 100% chemically impregnated electrodes.

The impregnation conditions are the following:

.Impregnation Bath: 50% ethylic alcohol
Ni + Co = 2 M/l
Co concentration: 2.5%
"pH": 1.5-2
Temperature: 80°C

.Electrolysis: Current density: 50 mA.cm2
Time: 4 hours

There is little discrepancy in the results as it is less than 3%.

The filling rate is about 15% slower than that of the chemically impregnated electrodes. However, the hydrate yield is about 10 to 15% greater than that of the hydrate obtained by chemical impregnation.

Consequently, the electrodes have: /32

-A specific energy per unit of mass identical to the 100% chemically impregnated electrodes.

-A higher energy per unit of volume due to the absence of swelling during cycling.

These results show that the impregnation of Ni-H2 type electrodes, whose specific energy is about 95 to 97% of chemically impregnated electrodes, may be performed in the following conditions:

.<u>Impregnation Bath</u>: 20% ehtylic alcohol
Ni + Co: 2 M/l
Co concentration: 2.5%
"pH": 1.5-2
Temperature: 84° C

.<u>Electrolysis</u>: Current density: 75 mA/cm2
Time: 2 hours 30 minutes

These conditions seem optimal for satisfying the objective established and for limiting the cost of impregnating the positive electrodes used for the Ni-H2 accumulators.

5. APPLICATION OF THE STUDY TO THE ELECTROCHEMICAL IMPREGNATION OF Ni-Cd TYPE POSITIVE ELECTRODES /33

5.1 REVIEW OF PREVIOUS RESULTS:

During the study on corrections to the dewatering of the separator within the framework of CNES Contract 75/0590, several electrochemical impregnation tests were made on Ni-Cd type nickel electrodes.

The set of results obtained presented in tables 8 and 9 show:

-the dimensional stability of the electrodes during cycling.

-an improvement in the filling and in the specific capacities obtained by increasing the initial porosity of the sintering.

-the capability of reaching capacities comparable to those of 100% chemically impregnated electrodes during the formation in a free electrolyte in the following conditions:

1st cycle:	Charge 10 hours at C/5
	Discharge C/2 - Stop at 0.5 V.
2nd cycle:	Charge 6 hours at C/3
	Discharge C/2 - Stop at 0.5 V.
3rd cycle:	Charge 4 hours at C/2
	Discharge C/2 - Stop at 0.5 V.

The results actually show that the electrode capacities could be improved by increasing the quantity of the electrolysis current (table 9).

Consequently, the impregnation tests performed within the framework of this study involve electrolysis current quantities greater than 450 C/cm2.

5.2 EXPERIMENTAL STUDY /34

The set of results obtained are summarized in appendix 11.

5.2.1 Experimental Conditions

The electrode fabrication and test conditions are identical to the conditions described in paragraph 4.1.

-Preparation of the Sintered Support:

The characteristics of the sinterings used are presented in table 10 (sintering number 4).

The porosity of the sintered support is high, i.e. 85%.

The accessible vacuum volume is 5.2 cm3/dm2, i.e. 6% greater than the vacuum volume of a standard type support.

-Impregnation:

The impregnation conditions are the same as those defined in paragraph 4.1.3:

55 liter Tank

-Cathode: sintered plate at mid-distance between the 2 counter-electrodes.
-2 counter-electrodes in pure soluble nickel.
-Electrode/counter-electrode distance = 19 mm
-Teflon electrode support
-Agitation - Thermomix pump flow rate 13 liters/minute.

The experimental conditions are summarized in appendix 11: /35

-Nature of the sintered support:

Ni-Cd type high porosity sintering
Thickness: 0.79 ± 0.02 mm
Surface treatment: reduction under H2.

-Bath concentration: 2 concentrations are tested

2 moles/l $Ni(NO_3)_2$ + Co $(NO_3)_2$ including 0.1 mole/l Co $(NO_3)_2$
3 moles/l $Ni(NO_3)_2$ + Co $(NO_3)_2$ including 0.1 mole/l $(NO_3)_2$

-Bath temperature: 80°C
-Alcohol content: 50%
-"pH": 1.5-2

-Electrochemical Cleaning

Identical conditions to those defined in paragraph 4.1.5:

Formation in free electrolyte (KOH d = 1.306) with negative cadmium electrodes with overcapacity.

1st cycle: Charge: 15 hours at C/5
2nd cycle: Discharge: at C/2 - Stop at 0 V/Hg-HgO
3rd & 4th cycle: Charge 4 hours at C/2
Discharge at C/2 - Stop at OV/Hg-HgO

C value = 4 Ah/dm2

5.2.2. Experimental Results /36

The tests were performed with Ni-Cd type sintered supports (thickness 0.79 ± 0.02 mm) with high porosity.

Standard Ni-Cd type positive electrodes were 100% chemically impregnated (hydrate = 12 g/dm2) to establish a comparison with the chemical impregnation of the electrode characteristics during electrical testing.

Characteristics of the Sinterings Used

2 types of sintering were made:

.1 standard sintering for the chemical impregnation of the positive electrodes used in the Ni-Cd elements.

.1 high porosity sintering (85) for the electrochemical impregntion of positive electrodes for Ni-Cd elements.

Their characteristics are presented in table 10.

The accessible vacuum volume of the "high porosity" sintered support capable of being filled with active substance exceeds the vaccum volume of the standar support by 6%, i.e 5.2 cm3 vacuum/dm2.

Characteristics of the Impregnated Electrodes

The characteristics of 100% chemically impregnated electrodes are presented in table 11.

The characteristics of electrochemically impregnated electrodes in the various conditions, in compliance with the test grid shown in appendix 11 are shown in tables 12 and 13 and in figures 20, 21 and 22.

3 essential parameters are examined to define the impregnation method:

-Filling
-Capacities (formation - cycling)
-Electrode swelling.

5.2.2.1 Electrode Filling (tables 12.1 and 12.2) /37

The tests were performed for 2 different Ni $(NO_3)_2$ + Co $(NO_3)_2$ concentrations of the impregnation bath:

-In Ni $(NO_3)_2$ + Co $(NO_3)_2$ = 2 M/l (table 12.1) the deposition of nickel hydroxide is achieved proportionally to the impregnation time for any electrolysis current density applied (figure 20.1).

However, it seems that the apparent thickness of the plate at the end of impregnation (Table 13.1) may increase by as much as 30% of the intitial thickness of the sintering.

During the formation, a weight loss of the impregnated plates is observed (table 12.1 - figure 20.1). This weight loss is associated with a thickness decrease (Table 13.1) and a capacity loss during the formation (figure 22.1).

At the beginning of impregnation, deposition occurs in the pores of the sintering, then on the surface at the end of impregnation (figure 20.1). The hydroxide quantities desposited are:

Current Densities Applied (mA/cm2)	50		75		95	
Impregnation time (minutes)	210	240	140	180	110	125
Specimen	3312	3313	3322	3323	3332	3333
Total hydroxide quantity deposited (g/dm2)	10.6	11.9	10.6	12.1	9.8	10.1
Hydroxide quantity deposited in the pores of the sintering (g/dm2)	9.3	9.2	8/9	8.4	7.4	8.1

The hydroxide quantities formed in the pores decrease as the electrolysis current density increases. This observation is correlated with the fact taht at high current densities, the pH gradient on the edge of the plate is greater and therefore the precipitation of hydroxide on the surface is facilitated.

-In Ni (NO_3) = 3 M/l solution, the diffusion of nickel nitrate improves and there is no increase in surface deposit at the end of impregnation (Table 12.2) unless there is a deposition with high current density: 95 mA.cm2.

For lower current densities (50 mA/cm2 and 75 mA.cm2) we observe the beginning of a filling stabilization (figure 20.2).

Electrodes thus impregnated do not exhibit capacity losses during formation (figure 22.2) nor large thickness decreases during formation (Table 13.2).

We observe an improvement in the filling when the current density increases:

Current Densities (mA/cm2)	50	75	95
Maximal filling obtained (*P in g/dm2) within the pores	10	10.5	11

In conclusion: The tests performed show a filling improvement within the pores of the sintering by using highly concentrated nickel nitrate solutions so as to avoid a large deposition on the plate surface.

The maximum filling rate is appreciably identical for the 3 current densities 50.75 and 95 mA.cm2.

5.2.2.2 Electrode Capacities (tables 12.1 and 12.2)

-In $Ni(NO_3)_2 + Co(NO_3)_2 = 3$ M/1 solution, the capacities obtained at the 4th formation cycle are low for the plates impregnated at high electrolysis current densities (figure 21.1).

The electrode capacities obtained by electrolysis at 50 mA/cm2 reach about 3.3 Ah.dm2, ie.e 90-95% of the capacity of 100% chemically impregnated electrodes at the 4th formation cycle. However,

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*Illegible.

after the loss of the surface deposit during the first formation cycle, the capacities obtained are stable (figure 22.1). This is not the case of the chemically impregnated reference specimen for which the capacity increase caused by the swelling in the free electrolyte is on the order of 4% for each cycle.

It is actually probable that the chemically impregnated electrodes compacted in a limited electrolyte undergoing the same type of formation would not show such a large capacity increase.

-In $Ni(NO_3)_2 + Co(NO_3)_2 = 3$ M/l solution, the capacities obtained during the 4th formation cycle are greater than in the Ni-Co - 2 M/l solution:

Current Density (mA/cm2)	Ni+Co (M/l) Bath Concentration 2	3
50	3.3	(2.9)
75	2.85	(3.1)
95	2.8	3.8

Electrodes electrochemically impregnated at 50 and 75 mA/cm2 have low formation capacities, whereas for an appreciably identical filling, the electrodes impregnated at 95 mA/cm2 show a high capacity. This property is due to an inadequate yield of electrodes impregnated at low current densities.

-<u>In Conclusion</u>:

Ni-Cd type electrodes impregnated electrochemically at high current densities (95 mA/cm2) in a highly concentrated solution (Ni+Co = 3M/l) are identical and are often greater than the capacities of 100% chemcially impregnated electrodes.

This characteristic is due to the proper filling within the pores of the sintering as a result of the increase in the diffusion of Ni^{++} ions.

5.2.2.3 Electrode Thickness Variation /40

Electrode thicknesses before and after formation are shown in tables 13.

-In Ni+Co = 2M/1 solution, one observes a large swelling duirng impregnation, particularly for electrodes impregnated at high current densities. This swelling may reach 30% when the quantity of the electrolysis current is on the order of 720 C/cm2. This swelling is due to the hydroxide deposit on the surface and a similar recuction in the electrode thickness is observed during its formation.

The overall swelling of formed electrodes is therefore less than 5%.

-In Ni+C0 = 3 M/1 solution, a swelling on the order of 5 to 15% is observed prior to electrode formation.

For electrodes impregnated at high current densities, this thickness increase is due to the deposit of hydroxides on the surface. This deposit detaches from the electrode during the formation and thus promotes a reduction in the electrode thickness (Table 13.2).

The overall thickness increase is less than 15%. It depends on the amount of hydroxide impregnated (figure 23) as we have already observed for Ni-H2 type electrodes.

5.3 SUMMARY

The results on the study of the electrochemical impregnation of positive nickel-hydrogen type electrodes (thickness 0.93 mm) applied to the study of the impregnation of Ni-Cd type electrodes (thickness 0.79 mm) shows that it is possible to make Ni-Cd type electrodes which are dimensionally stable during cycling and are just as energetic as the 100% chemically impregnated electrodes, by means of electrochemical impregnation in an hydro-alcoholic solution.

The electrochemical impregnation conditions tested demonstrate following points:

The impregnation must occur in a highly concentrated $Ni(NO_3)_2$ + $Co(NO_3)_2$ solution to improve the filling in the pores of the sintering and to avoid surface deposition.

In these conditions, the impregnated hydroxide quantities are satisfactory, i.e. about 2.1 g/cm3 empty, theoretically corresponding to filling the sintering porosity by about 75%.

The application of high electrolysis current densities does not influence the filling and makes it possible to fabricate high energy electrodes for an impregnation time of about 2 hours.

The thickness increase of the sintered support prior to the formation is on the order of 5 to 15%. /41

During the formation, the electrodes are dimensionally stable.

The optimal impregnation conditions tested are the following:

Temperature: 80°C
Ni+Co concentration: 3 M/l
Co Concentration: 0.1 M/l
Alcohol Content: 50%
"pH": 1.5-2

Electrolysis Conditions:

Current density: 95 mA/cm2
Impregnation time: 2 H 05

As shown in the study of the impregnation of Ni-H_2 type positive electrodes (Paragraph 4.2), it is probable that a decrease in the alcohol content in the batch does not influence the energy and stability characteristics of impregnated electrodes.

6. EXAMINATION OF THE CRITICAL FACTORS FOR THE DEFINITION OF AN ELECTROCHEMICAL IMPREGNATION UNIT

6.1 Corrosion of the Sintered Support Prior to Impregnation

The test consists of controlling the corrosion of the support immersed in an hydro-alcoholic solution before the beginning of electrolysis.

The tests were performed at 80°C (temperature of the impregnation bath) for 3 alcohol contents (20-30 and 50%) and for two nickel nitrate concentrations at "pH" 1.5-2.

The Ni-H2 type sintered supports of high porosity (Sintering no. 2 - Table 10) are immersed in the solution at 80°C according to 4 different immersion times: 2, 5, 10 and 20 minutes.

After the immersion, the samples are washed in distilled water, then are dried for 12 hours under nitrogen.

Control:

-The samples are weighed to the nearest ±1 mg.

-Corrosion of the sintering is controlled by dissolution of the nickel hydroxide capable of forming in an acetic buffer, then determining the corrosion by weighing. 3 tests were performed on the sinterings intact to correct the analysis error.

Results /42

The results obtained are shown in table 15 and in figure 24.

For any nickel nitrate concentration and nickel content, the corrosion seems to be appreciably nil during the first 15 minutes of immersion in the solution at pH 1.5-2.

After 15 minutes of immersion, the beginning of an observable corrosion seems to form in figure 24.

6.2 Wear of the Impregnation Bath

During the electrochemical impregnation test on the positive electrodes of Ni-H2 elements, we found a decrease in the filling and in the energy characteristics of the electrochemically impregnated electrodes (figure 25). After using the bath for 5 impregnations, the bath suddenly changed.

In this case, the impregnation conditions were as follows:

$$F = \frac{\text{Bath volume (dm3)}}{\text{Sintering surface (dm2)}} = \frac{55}{7.9} = 8$$

Temperature: 80°C
Alcohol content: 50%
Current density: 50 mA/cm2, i.e. I = 34.5 A
Impregnation time: 4 hours.

Another test for determining bath wear was performed in the following conditions:

$$R = \frac{\text{Bath volume (dm3)}}{\text{Sintering surface}}\ \text{(dm2)} = \frac{4.2}{0.58} = 7.2$$

Temperature: 80°C
Alcohol content: 50%
Current density: 50 mA.cm, i.e. I - 2.9 A
Impregnation time: 4 hours.

After impregnation, the filling rate is determined by weighing and by determining the formation capacities.

It seeems that after 21 impregnations in the same bath, /43 on the whole, we do not see any decrease in the filling characteristics and in the capacity of the impregnated plates (figure 25).

If we compare the characteristics of the 2 tests, we do not find an apparent correlation with the impregnation time and the amount of current passing through the bath.

Furthermore, the decrease in the performance of Ni-H2 type impregnated electrodes during the first test is possibly only temporary and would require a more thorough statistical determination on an experimental unit.

Note, however, that the oxidation of alcohol causes an increase in the acetate and in the acetic acid in the bath and this can have a buffer effect.

A possible regeneration might then be considered by distillation or by precipitation of the nickel hydroxide and decantation.

6.3 Characterization Tests of the Role of Alcohol

The results obtained in the first part of the electrochemical impregnation study (paragraph 4.2) show that the electrodes impregnated in an aqueous medium at pH 1.5-2 progressively increase in thickness during cycling.

Conversely, electrodes impregnated in an hydro-alcoholic medium are dimensionally stable.

Two hypotheses were therefore formulated to explain the role of alcohol:

-Alcohol decreases the corrosion on the sintering and therefore enables the sintering to preserve its initial firmness and thus prevents the corrosion process.

-Alcohol acts uon the structure of the hydroxide made and thereby assures homegeneity of the filling.

In order to confirm either of these hypotheses, the impregnation tests in an aqueous medium were carried out with sinterings of thickness 0.85 mm (porosity 85%) at pH 3-3.5 in the following conditions:

-Tmeperature = 98°C
-Alcohol content: 0%
-Ni + Co = 2 M/l
-Co content: 5%.

The low acidity of the bath should decrease the corrosion of the sintering during impregnation. If the brittleness of the support is an important cause of electrode swelling during cycling, an improvement in the dimensional stability of the electrodes should be verified.

The filling and capacity results obtained are shown in table 16. /44

The results of thickness variation are shown in table 17.

-Remarks:

One notes that all electrodes electrochemically impregnated in an aqueous solution exhibit a swelling during the relatively large cycling according to the filling rate and capacities restored (figure 26).

Note that the hydroxide yields obtained are small and that the electrodes showing a capacity similar to that of 100% chemically impregnated electrodes (sample type D) swell about 75% after 70 cycles.

Only specimen F has little swelling (about 10% after 70 cycles) for a capacity similar to that of a 100% chemically impregnated sample.

For the same capacity, the swelling rate of 100% chemically impregnated electrodes is greater than 40%.

It therefore seems that a decrease in the corrosion on the support electrochemically impregnated in an aqueous medium decreases the swelling of electrodes during cycling.

However, the tests performed did not make it possible to obtain a dimensional stability of electrodes impregnated in an aqueous medium. It is therefore probable that the role of alcohol is not limited to decreasing the corrosion of the sintered support.

Modification of the "bath acidity" parameter does not seem to be sufficient for obtaining dimensionally stable electrodes in an aqueous medium and a decrease in the impregnation temperature could be required at the cost of the cost of the impregnated hydroxide quantity.

Furthermore, the dimensional stability of electrodes impregnated in an aqueous medium at 98°C probably will not be obtained with high porosity sinterings for which the initial strength is low.

We therefore thought it would be a good idea to search for a product capable of having properties similar to those of alcohol, but making it possible to fabricate high energy and dimensionally stable electrodes in safe conditions and at the lower cost.

6.4 Alcohol Substitutes /45

Alcohol substitutes added to the impregnation bath are normally used in galvanic treatments to modify the surface tension of the liquid and its wetting power.

-Impregnation in the Presence of Surface-Active Agents

Several impregnations of high porosity (85%) sintered supports of type Ni-H2 (0.93 mm thickness) were performed in various baths containing surface active agents in the following conditions:

.Bath Temperature: 80°C
Ni + Co concentration: 2 M/l
Co concentration: 5%
Additive: surface active agent

.Electrolysis: Current density: 50 mA.cm2
Time: 4 hours.

The electrodes thus impregnated, then electrochemically cleaned were subjected to the 4 formation cycles defined in section 5.2.1.

The results obtained are shown in figure 27.

-Remarks:

Figure 27 shows that the maximal filling is obtained for an optimal surface active concentration.

The variation of the plate weight during impregnation is about 11.5 g/dm2, i.e. appreciably identical to that of an electrochemcially impregnated electrode in an hydro-alcoholic medium.

The capacities obtained during formation reach 3.8 Ah/dm2, i.e. about 90% of the capacity of the 100% chemically impregnated electrodes.

When the surface active quantity increases, the bath viscosity becomes too high and the hydroxide forms a deposit on the surface. The plate capacities thus achieved are therefore low.

The electrodes impregnated in the presence of surface active agents do not swell during formation and during cycling (50 cycles completed).

It is probable that the electrodes electrochemically impregnated in the presence of surface active agents at higher temperatures would make it possible to fabricate dimensionally stable electrodes showing the same energy levels as chemically impregnated electrodes.

6.5 SUMMARY /46

The study of the critical factros of impregnation established the following points:

-The corrosion on the sintered support in the impregnation bath seems to be appreciably nil for an immersion time lasting less than 15 minutes.

-A test for determining the possible number of impregnations with the same bath without observing a decrease in the filling characteristics and the capacity of the impregnated electrodes was performed for two different "Bath Volume/Sintering Surface" ratios.

This test did not make it possible to check whether the drop in the electrode characteristics observed after using the same bath several times was temporary or whether the bath was unusable.

This determination may be accomplished statistically on an experimental impregnation unit permitting a large number of electrodes to be fabricated.

-In order to improve our knowledge about the role of alcohol, an impregnation test in an aqueous medium was performed at pH = 3-3.5 to diminish the corrosion of the nickel sintering support. The results obtained show that the dimensional stablity of electrodes electrochemically impregnated at 98°C in an aqueous medium cannot be obtained. The role of alcohol therefore does not seem to be limited to a decrease in the corrosion on the sintered support.

A modification of the parametric factors "temperature" and "sintering porosity" might possibly make it possible to fabrication dimensionally stable electrodes at the cost of the amount of hydroxide impregnated.

Consequently, we thought it would be desirable to look for alcohol substitutes.

Among these products capable of lowering the surface tension of the bath, a surface active agent was tested at 80°C for various concentrations.

It seems that the filling capabilities of the impregnated electrodes reach a maximum level for an optimal surface active concentration.

The electrodes thus fabricated are dimenaionally stable during cycling and their energy capacities reach 90% of the 100% chemically impregnated electrodes.

It is probable that for higher impregnation temperatures, utilization of a surface active agent makes it possible to obtain results comparable to those of alcohol.

In conclusion, the overall results obtained both for the electrochemical impregnation of positive Ni-H2 type electrodes and for Ni-Cd type electrodes, show that the electrochemical impregnation makes it possible:

-To fabricate electrodes which are dimensionally stable during cycling.

-To obtain an energy capacity per unit of mass which is equivalent to 100% chemically impregnated electrodes. /47

The advantage of the process thus being demonstrated for laboratory conditions, it seems necessary to fabricate an experimental impregnation unit capable of impregnating electrodes in large enough quantities to be able to evaluate the performances of new electrodes in accumulators.

7. CONCLUSION

The entire study of electrochemical impregnation demonstrates the feasibility for making dimensionally stable Ni-H2 and Ni-Cd type psotive electrodes showing an energy capacity of about 95 to 100% of that of 100% chemcially impregnated electrodes.

The summary of the study presented in paragraphs 4.3 and 5.3 specifies the influence of the different parameters: current and electrolysis time, temperature and acidity of the impregnation bath, nickel and cobalt concentration, use of alcohol.

The impregnation conditions seeming to be optimal for obtaining electrodes showing the characteristics specified above, are the following:

Ni-H_2 type positive electrodes: (thickness 0.93 mm)

Impregnation bath:
-Temprature 84°C ± 1
-Alcohol content 20%
-Ni + Co = 2 M/l concentration
-Co = 0.05 M/l concentration
-"pH": 1.5-2.

Electrolyte conditions:
-Current density: 75 mA/cm2.
-Impregnation time: 2 hours 30 min.

Ni-Cd type positive electrodes: (thickness 0.79 mm)

Impregnation bath:
-Temperature 80°C ±1
-Alcohol content: 50%
-Ni + Co concentration = 3 M/l
-Co concentration: 0.1 M/l
"pH": 1.5 - 2

Electrolyte conditions:
Current density 95 mA/cm2
Impregnation time: 2 hours 05 min.

It seems that a decrease in the alcohol content by 20% should not modify the characteristics of impregnated electrodes and facilitate production in terms of fabrication safety. /48

All critical factors of electrochemical impregnation was discussed in paragraph 6.6.

What should be essentially noted are the tests performed to replace alcohol by an surface active agent.

These tests show that at 80°C, it is possible to obtain dimensionally stable electrodes by showing a capacity of about 90% of the chemically impregnated electrodes.

Electrodes which are dimensionally stable during cycling could not be fabricated in an aqueous medium by operating at 98°C for 2 bath acidity ranges.

A decrease in the impregnation temperature and of the initial porosity of the support might possibly have made it possible to fabricate stable electrodes, at the cost of their capacity, however.

Now that we have demonstrated the advantage of the electrochemical Impregnation process, it is important to fabricate electrodes in large enough numbers to be able to evaluate the performances obtained with the new impregnation process.

It therefore seems necessary to fabricate an experimental installation for making electrodes in the specified conditions. We may consider broadening the impregnation conditions to imporve the manufacturing cost and safety of these positive electrodes.

REFERENCES

1. Study of the polypropylene separators.

   Electrical and cycling characteristics of Vo 10S accumulators.
   SAFT Report DT/SAS-493/75-JG/JM.

2. Preliminary study of methods for correcting the dewatering of separators of Ni-H2 accumulators.
   F. PUTOIS - J. GOUALARD - P. ANTOINE
   DT/SAS-333/76 (Contract 75/CNES/0590).

3. V.I PUGLISI - H.N. SEIGER - D.F. PICKETT - AFAPL - TR.74.56.

   High energy density sintered plate type nickel-cadmium battery cells. Part II: Electrochemical Impregnation methods to produce Nickel-Oxide Electrodes.

4. O. GLEMSER - J. EINERHAND

   .Z Electrochem. 1950, 54, 302.
   .Z Anorg. Chem. 1950, 261, 28 and 43.

5. W. FREITKNECHT, H.R. CRISTEN, H. STUDER

   .Z Anorg. All. Chem. 1956, 283, 88

6. D. TUOMI - J. Electrochem. Soc 1965, 112.1
   J. Electrochem. Soc 1965, 115,450

7. H. BODE

   .Angew Chem. 1961, 16, 553
   .Electrochemica Acta 1966, 11, 1079
   .Electrochemica Acta 1972, 17, 609.
   .Z. Anorg Allg. Chem. 1969, 366, 1

8. P. NEES - SIE Conference, KELKHEIM, 1973.

9. R.L. BEAUCHAMP - Transcip of proceedings, NASA.GFSC Battery Workshop 1970.

10. D.F. PICKETT AFAPL-TR 75-34. Fabrication and investigation of nickel-alkaline cells. Part 1. Fabrication of Nickel-Hydroxide Electrodes using electrochemical impregnation technique.

    Part II. Analysis of ethanolic Metal nitrate solutions used in fabrication of nickel hydroxide electrodes.

11. Power Sources 1976.
H.N. SEIGER and V.J. PUGLISI - NIckel Oxide electrode development.

12. L. KANDLER, U.S. Patent 3.214,355 - 1965.

13. E. HAUSLER, Power Sources. D.H. COLLINS, Ed. Pergamon Press. N.Y. 1966.

14. V.J. PUGLISI - D.F. PICKETT. Development of nickel alkaline batteries for aerospace lightweight secondary Power AFAPL-TR-75-64.

15. Invention patent: M.J. BACSA 0 S. MILLTER No. 669.139.

16. Invention patent: J.G. FARBENINDUSTRIE AKTIENGESELLSCHAFT No. 828,247.

17. Invention patent: P. JACQUIER - No. L,017,110.

18. Invention patent: VARTA - No. 1,342,836.

19. Invention patent: Accumulatoren Fabrik Aktiengesellschaft No. 1,259,322.

20. Invention patent: GOULD NATIONAL BATTERIES No. 1,271,804.

21. Invention patent: L. KANLDER No. 1,234,074.

22. Invention patent: Western electric company No. 1,587,246.

23. Invention patent: Yardney International corporation No. 2,062,770.

24. Invention patent: Westinghouse Electric Corporation No. 2,062,770.

25. Invention patent: Western Electric Company Incorporated No. 2,075,968.

26. Invention patent: Matsushita Eelectric Industrial No. 2,053,167.

27. Invention patent: L. BEAUCHAMP No. 2,272,496.

28. U.S. Patent - T.A. Edison No. 1,402,751.

29. U.S. Patent - A.K. Smith No. 1,442,238.

30. U.S. Patent - R.S. Coolidge No. 2,737,541.

31. U.S. Patent - F.P. Kober No. 2,737,541.

32. U.S. Patent - G.A. Mueller No. 3,203,879.

33. U.S. Patent - D.F. Pickett No. 3,827,911.

34. Offenlegungschrift - VARTA AG No. 1671,993.

35. U.S. Patent - Rampel No. 3,248,266.

TABLE 1

Characteristics of the Ni-H2 Type Sinterings Used

Sintering Type	Standard Ni-H2 sintering no. 2	High porosity Ni-H2 sintering no. 2
Thickness (mm/100)	81 ± 3	92 ± 3
Sheet weight (g/dm2)	5,2	5,3
Nickel Coating Weight (g/dm2)	14,4 ± 0,4	9,85 ± 0,4
Theoretical porosity cm3/g sintered pl.	0,347	0,489
Open porosity cm3/g sintered pl.	0,296	0,405
Sintering strength	970	410
Positive sintering capacity (mAh/dm2)	13,8	13,4
Theoretical porosity (%)	80	86
Accessible vacuum volume (cm3/dm2)	5,8	6,1

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*Translator's Note: numbers at bottom center of page are foreign text page numbers.

TABLE 2

CHARACTERISTICS OF POSITIVE CHEMICALLY IMPREGNATED Ni-H2 PLATES

	Plate Type		Ni-H2 (100% impregn.)	Standard Ni-H2 (under-impregn.)
	Reference Sample		24	25
SINTERING	Refer. Sintering (no.)		1	1
	Thickness (mm/100)		91 ± 3	91 ± 3
	Porosity (%)		80	80
IMPREG-NATION	Hydrate Wt. (g/dm2)		14,2 Standard =14,5 ± 1,5)	11,[illegible] Standard =10,[illegible] ± 1,5)
	Corroded Nickel (%)		13	11
FORMATION	Capacities (Ah/dm2) Formation	1st cycle	3,72	3,[illegible]
		2nd cycle	3,96	3,2[illegible]
		3rd cycle	4,10	3,4[illegible]
		4th cycle	4,21	3,7[illegible]
	Yield (An/g hydrate)		0,296	0,3[illegible]9
	Thickness (mm/100)	Before formation		
		a.	86	84,[illegible]
		b.	87	85
		c.	88	85,[illegible]
		After formation		
		a.	109	91
		b.	110,5	93
		c.	111,5	92
		Swelling (%)	29	9

TABLE 3

CHEMICAL ANALYSIS OF ELECTROCHEMICALLY IMPREGNATED ELECTRODES

Reference Sample	Characteristic Parameters of the Impregnation Conditions			ΔP (g/dm2)	Corrod. Ni (g/dm2)	Activ. (Ni+Co) (g/dm2)	S=ΔP+N atta./ Ni+Co	Theoret. Yield (4th form. cycle Ah/g O/
1111 a	0 % Alcohol	50 mA/cm2	2 h 30	12,7	1,48	8,2	1,73	0,33
1122 a	0 % Alcohol	75 mA/cm2	2 h 20	11,6	1,08	7,2	1,77	0,34
1131 a	0 % Alcohol	95 mA/cm2	1 h 35	9,1	1,28	5,0	-	0,34
1211 c	20 % Alcohol	50 mA/cm2	3 h	10,4	0,59	6,1	1,80	0,36
1223 c	20 % Alcohol	75 mA/cm2	2 h 40	9,4	0,79	6,1	1,68	0,36
1232 c	20 % Alcohol	95 mA/cm2	1 h 50	9,6	0,79	5,7	1,81	0,34
1313 c	50 % Alcohol	50 mA/cm2	4 h	14,1	0,49	8,9	1,63	0,34
1322 c	50 % Alcohol	75 mA/cm2	2 h 20	11,7	0,49	7,35	1,66	0,33
1331 c	50 % Alcohol	95 mA/cm2	1 h 35	8,1	0,69	5,0	1,76	0,34
1612	70 % Alcohol	50 mA/cm2	3 h 30	10,9	0,55	7,4	1,55	0,32
1622	70 % Alcohol	75 mA/cm2	2 h 20	10,6	0,65	7,5	1,45	0,31
1632	70 % Alcohol	95 mA/cm2	1 h 50	10,7	0,15	7,0	1,55	0,32
1511	Ni + Co = 3M/l	50 mA/cm2	3 h	11,6	0,25	7,8	1,52	0,34
1522	Ni + Co = 3M/l	75 mA/cm2	2 h 20	11,8	0,15	7,7	1,55	0,31
1532	Ni + Co = 3M/l	95 mA/cm2	1 h 50	11,7	0,15	7,9	1,50	0,31
2113	Nonpass. Sintering	50 mA/cm2	4 h	11,0	0,75	8,0	1,47	0,32
2123	Nonpass. Sintering	75 mA/cm2	2 h 40	11,2	0,65	7,8	1,52	0,32
2313	Passiv. Sintering	50 mA/cm2	4 h	11,2	0,75	7,9	1,51	0,33
2323	Passiv. Sintering	75 mA/cm2	2 h 40	9,0	0,65	6,7	1,44	0,33
2413	Passiv. Sintering	50 mA/cm2	4 h	12,1	0,65	7,7	1,65	0,30
2423	Passivated Sintering + Reduced	75 mA/cm2	2 h 40	10,9	0,55	7,6	1,51	0,32
2222	pH 3-3.5	75 mA/cm2	2 h 20	10,6	0,15	8,0	1,34	0,33
2213	pH 3-3,5	50 mA/cm2	4 h	11,7	0,25	7,5	1,59	/
20	Electrode Ni-H2	50 mA/cm2		13,5	0,25	8,2	1,68	/
36	Electrode Ni-H2	50 mA/cm2		13,0	0,05	7,9	1,67	/
1	Chemical impregnation - Standard Labo.			11,6	1,60	8,2	1,60	0,24
2	Chemical impregnation - Alcohol			11,4	2,1	8,7	1,55	0,33

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TABLE 4.1

CHARACTERISTICS OF IMPREGNATED PLATES

Impregnation Bath pH: 1.5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content: 0%
Temperature: 98°C

Reference Sample		Impregnation Conditions		Characteristics of impregnated plates										
							Capacities in Ah (S = 0,58 dm2)							
		Current Density (mA/cm2)	Impregnation time (mn)	El. wt. before impregnat.	El. wt. after impregnat.	ΔP (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capac. 4th cyc Ah/dm2	Yield 4th cyc Q/ΔP (Ah/g)	Mean Yield Ah/g
1111	a	50	150	10,376	17,736	12,6)							
	b			10,384	17,488	12,2(12,3	2,14	2,32	2,29	2,30	4,00	0,324	0,328
	c			10,420	17,334	11,9)	2,08	2,32	2,23	2,30		0,333	
1112	a	50	210	10,390	18,490	14,0)							
	b			10,483	18,550	13,9(14,0	2,34	2,38	2,43	2,50	4,40	0,310	0,314
	c			10,430	18,600	14,0)	2,43	2,50	2,52	2,60		0,318	
1113	a	50	240	10,494	18,850	14,4)							
	b			10,500	18,850	14,4(14,3	2,34	2,43	2,47	[illegible]	4,33	0,300	0,304
	c			10,370	18,547	14,1)	2,25	2,34	2,38	[illegible]		0,308	
1121	a	75	120	10,400	17,312	11,9)	2,34	2,34	2,38	[illegible]	4,00	0,347	0,351
	b			10,300	16,500	10,7(11,0	2,10	2,32	2,14	2,20		0,355	
	c			10,405	16,500	10,5)							
1122	a	75	140	10,474	17,206	11,6)							
	b			10,577	17,550	12,0(11,9	2,20	2,32	2,29	2,40	4,05	0,344	0,336
	c			10,480	17,480	12,0)	2,20	2,30	2,29	2,30		0,328	
1123	a	75	160	10,317	17,100	11,7)							
	b			10,400	17,520	12,3(12,1	2,08	2,30	2,27	2,20	4,03	0,308	(0,327)
	c			10,360	17,526	12,3)	2,34	2,45	2,40	2,48		0,346	
1131	a	95	95	10,482	15,777	(9,13)							
	b			10,472	17,770	12,6(12,9	2,36	2,43	2,50	2,58	4,45	0,353	0,344
	c			10,562	18,245	13,2)	2,40	2,43	2,52	2,58		0,336	
1132	a	95	110	10,642	19,215	14,8)							
	b			10,493	18,050	13,0(13,6	2,45	2,52	2,54	2,58	4,45	0,341	0,340
	c			10,310	17,894	13,1)	2,32	2,45	2,50	2,58		0,340	
1133	a	95	125	10,314	18,784	14,6)							
	b			10,380	18,125	13,3(13,7	2,30	2,52	2,54	2,58	4,46	0,333	0,334
	c			10,305	18,050	13,3)	2,36	2,45	2,50	2,60		0,335	

TABLE 4.2
CHARACTERISTICS OF IMPREGNATED PLATES

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Impregnation Bath : Concentration Ni+Co = 2M/1
pH: 1.5-2 Concentration Co = 5 %

Alcohol Content: 20%
Temperature: 84°C

Reference Sample		Impregnation Conditions			Characteristics of impregnated plates									
									Capacities in Ah (S = 0,58 dm2)					
		Current density (mA/cm2)	Impregnation time (mn)	Current Quantity (C/cm2)	El wt. prior to impreg. (in g)	El wt. after impreg. (in g)	ΔP (g/dm2)	Mean ΔP (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity (4th cyc.) (Ah/dm2)	Yield 4th cycl Q/ΔP (Ah/g)
1211	a	50	180	540	10,247	16,710	11,1		2,01	2,14	2,27	2,38	4,14	0,368
	b				10,380	17,244	11,8	11,1	2,20	2,36	2,38	2,42		0,352
	c				10,160	16,170	10,4							
1212	a	50	210	630	10,300	17,488	12,4		2,12	2,36	2,38	2,45	4,17	0,341
	b				10,190	17,244	12,2	12,0	2,03	2,36	2,36	2,38		0,337
	c				10,424	17,077	11,5							
1213	a	50	240	720	10,220	13,280	(5,3)							
	b				10,275	16,980	11,6	(11,6)	2,03	2,17	2,27	2,25	(3,55)	0,335
	c				10,230	15,694	(9,4)		1,78	1,80	1,80	1,86		0,340
1221	a	75	120	540	10,422	16,762	10,9		1,88	2,12	2,10	2,25	3,93	0,355
	b				10,272	16,702	11,1	11,0	2,03	2,24	2,26	2,32		0,361
	c				10,432	16,843	11,0							
1222	a	75	140	630	10,280	16,880	11,4		2,20	2,24	2,34	2,38	4,00	0,361
	b				10,210	16,710	11,2	11,0	2,02	2,08	2,17	2,25		0,346
	c				10,220	16,260	10,4							
1223	a	75	160	720	10,293	16,780	11,2		2,04	2,14	2,25	2,36	4,12	0,364
	b				10,396	17,000	11,4	11,3	2,12	2,25	2,34	2,42		0,366
	c				10,260	15,690	(9,4)							
1231	a	95	95	540	10,010	15,452	9,4		1,73	1,80	1,86	1,88	3,22	0,345
	b				10,162	15,852	9,8	9,9	1,82	1,88	1,92	1,86		0,327
	c				10,056	16,150	10,5							
1232	a	95	110	630	10,013	15,783	9,9		1,82	1,97	1,95	2,04	3,48	0,353
	b				10,018	16,048	10,4	10,0	1,82	1,90	1,95	2,00		0,332
	c				9,975	15,540	9,6							
1233	a	95	125	720	10,030	15,610	9,6		1,67	1,80	1,86	1,97	3,46	0,353
	b				10,095	15,975	10,1	9,8	1,80	1,90	1,95	2,04		0,347
	c				10,000	14,665	(8,1)							

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TABLE 4.3
CHARACTERISTICS OF IMPREGNATED PLATES

Impregnation Bath : Concentration Ni+Co = 2M/1
Concentration Co = 5 %
pH : 1,5-2

Alcohol Content : 50
Temperature : 80°

Reference Sample		Impregnation Conditions			Impregnated plate characteristics				Capacities in Ah (S = 0,58 dm2)					
		Current Density (mA/cm2)	Impregnation time (mn)	Current Quantity (C/cm2)	El wt. before impreg. (in g)	El. wt. after impreg.	ΔP (g/dm2)	ΔP (mean) (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capaci. (4th cyc. Ah/dm2)	Yield 4th cycle $C/\Delta P$ (Ah/g)
1311	a	50	180	540	10,300	16,770	11,1	(9,6)	2,17	2,12	2,12	2,22	3,83	0,343
	b				10,142	12,000	-		-	1,62	0,80	-		-
	c				10,067	14,700	8,0							
1312	a	50	210	630	10,150	16,875	11,6	11,9	2,17	2,20	2,18	2,27	3,90	0,337
	b				10,156	16,834	11,5		2,23	2,20	2,18	2,27		0,339
	c				10,217	17,484	12,5							
1313	a	50	240	720	10,595	18,022	12,8	13,0	2,34	2,43	2,34	2,43	4,25	0,327
	b				10,360	17,590	12,5		-	2,43	2,54	2,50		0,345
	c				.0,327	18,547	14,1							
1321	a	75	120	540	10,320	17,104	11,7	11,8	2,12	2,12	2,08	2,12	3,81	0,312
	b				10,300	17,180	11,9		2,14	2,27	2,25	2,30		0,334
	c				10,430	15,317	(8,4)							
1322	a	75	140	630	10,194	16,932	11,6	11,6	2,23	2,27	2,27	2,12	3,85	0,315
	b				10,100	16,710	11,4		1,97	2,12	1,97	2,34		0,354
	c				10,313	17,090	11,7							
1323	a	75	160	720	10,225	17,332	12,2	12,5	1,97	2,06	2,08	2,12	3,65	0,298
	b				10,290	17,274	12,0		1,97	1,95	2,01	2,12		0,303
	c				10,226	18,046	13,4							
1331	a	95	95	540	10,250	16,300	10,4	10,7	2,12	2,12	2,06	2,12	3,64	0,350
	b				10,317	16,707	11,0		2,0	2,01	2,01	2,10		0,329
	c				10,335	15,033	(8,1)							
1332	a	95	110	630	10,285	16,802	11,2	11,2	1,71	1,95	2,06	1,77	3,45	0,271
	b				10,052	16,416	10,9		1,90	2,10	2,06	2,20		0,346
	c				10,340	16,940	11,4							
1333	a	95	125	720	10,137	16,430	10,9	11,3	2,06	2,10	2,15	2,06	3,75	0,327
	b				10,216	16,690	11,2		-	2,23	2,25	2,30		0,355
	c				10,274	17,790	11,9							

TABLE 4.4

CHARACTERISTICS OF IMPREGNATED PLATES

Impregnation Bath : Concentration Ni+Co = 2M/1
Concentration Co = 5 %
pH : 1,5-2

Alcohol content : 50 %
Temperature : 50°

Reference sample		Impregnation Conditions							Impregnated plates characteristics					
									Capacities in Ah (S = 0,58 dm2)					
		Current density (mA/cm2)	Impregnation time (mn) (mn)	Current Quantity (C/cm2)	El wt. before impreg. (in g.)	El wt. after impreg. (in g.)	ΔP (g/dm2)	mean ΔP (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity (4th cyc Ah/dm2)	Yield 4th cycle Q/ΔP (Ah/g)
1411	a	50	180	540	10,050	13,155	5,3		-	0,97	0,80	-		-
	b				10,032	13,860	6,6	6,0	-	1,08	0,98	1,00	1,72	0,261
	c				9,980	11,590	-							
1412	a	50	210	630	10,000	14,250	7,3		-	1,08	0,97	1,00	1,64	0,235
	b				10,283	13,490	5,5	6,2	-	0,97	1,04	0,90		0,281
	c				10,278	13,630	5,8							
1413	a	50	240	720	10,160	14,778	8,0		1,30	1,30	1,36	1,30	2,27	0,281
	b				10,060	14,642	7,9	8,4	1,30	1,21	1,21	1,34		0,292
	c				10,060	15,500	9,4							
1421	a	75	120	540	10,023	14,330	7,4		1,30	1,32	1,30	1,30	2,52	0,302
	b				10,100	15,280	8,9	8,2	1,56	1,62	1,62	1,62		0,313
	c				10,166	13,428	(5,6)							
1422	a	75	140	630	10,265	16,267	10,3		1,90	1,95	1,90	2,00	3,34	0,333
	b				9,990	15,890	10,2	10,5	1,90	1,92	1,93	1,92		0,325
	c				10,160	16,590	11,1							
1423	a	75	160	720	10,050	16,044	10,3		1,73	2,01	2,00	2,06	3,55	0,344
	b				10,150	15,867	9,9	10,3	1,95	2,01	2,00	2,06		0,360
	c				9,880	16,130	10,8							
1431	a	95	95	540	10,100	15,493	9,3		1,39	1,54	1,58	1,70	3,04	0,315
	b				10,100	15,640	9,5	9,4	1,43	1,62	1,73	1,82		0,328
	c				10,140	12,890	(4,7)							
1432	a	95	110	630	10,045	15,810	9,9		1,47	1,71	1,84	1,90	3,22	0,329
	b				10,240	15,882	9,7	9,7	1,54	1,75	1,75	1,84		0,326
	c				10,190	15,713	9,5							
1433	a	95	125	720	10,005	15,958	10,2		1,43	1,65	1,73	1,86	3,20	0,312
	b				10,130	15,916	10,0	10,1	1,50	1,73	1,82	1,86		0,321
	c				9,865	15,778	10,2							

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TABLE 4.5
CHARACTERISTICS OF IMPREGNATED PLATES

Impregnation Bath
pH: 1.5-2

Concentration NI+Co = 2M/1
Concentration Co = 5 %

Alcohol content: 70 %
Température : 80°C

Reference Sample		Impregnation conditions			Impregnated plate characteristics									
								Capacities in Ah (S = 0,50 dm2)						
		Current Density (mA/cm2)	Impreg. Time (mn)	Current Quantity (C/cm2)	El. wt. before impregn. (in g.)	El. wt. after impreg. (in g.)	ΔP (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity (4th cyc. Ah/dm2)	Yield 4th cycle Γ/ΔP (Ah/g)	Av. yiel Ah/g
1611	a	50	180	540	42,850	66,550	10,3	1,63	1,48	1,50	1,50	3,00	0,291	0,2
	b							1,63	1,34	1,39	1,50		0,291	
1612	a	50	210	630	41,600	66,640	10,9	1,70	1,74	1,74	1,71	3,50	0,314	0,3
	b							1,70	1,66	1,74	1,80		0,331	
1613	a	50	240	720	41,940	66,760	10,8	1,63	1,62	1,58	1,57	3,16	0,291	0,2
	b							1,63	1,62	1,67	1,59		0,295	
1621	a	75	120	540	42,770	67,200	10,6	1,60	1,60	1,67	1,69	3,40	0,318	0,3
	b							1,60	1,60	1,60	1,69		0,318	
1622	a	75	140	630	42,880	67,330	10,6	1,59	1,60	1,60	1,67	3,34	0,314	0,3
	b							1,71	1,69	1,60	1,67		0,314	
1623	a	75	160	720	43,050	68,050	10,9	1,56	1,60	1,50	1,67	3,24	0,307	0,3
	b							1,71	1,46	1,62	1,57		0,307	
1631	a	95	95	540	42,420	66,570	10,5	1,44	1,50	1,56	1,57	3,26	0,299	0,3
	b							1,74	1,70	1,62	1,69		0,321	
1632	a	95	110	630	44,420	69,140	10,7	1,87	1,74	1,80	1,80	3,40	0,335	0,3
	b							1,74	1,70	1,62	1,60		0,298	
1633	a	95	125	720	43,450	66,900	10,2	1,74	1,56	1,67	1,74	3,34	0,341	0,3
	b							1,74	1,56	1,62	1,60		0,314	

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TABLE 4.6

CHARACTERISTICS OF IMPREGNATED PLATES

Impregnation Bath
pH: 1.5-2

Concentration Ni+Co = 3 M/l
Concentration Co = 3,3 %

Alcohol content 50 %
Temperature : 80°C

Reference Sample		Impregnation conditions						Impregnated plate characteristics						
		Current density (mA/cm2)	impregnation time (mn)	Current quantity (C/cm2)	El. wt. before impregn. (in g.) S=2,3dm2	El wt. after impreg. (in g.) S=2,3dm2	ΔP (g/dm2)	Capacities in Ah (S = 0,50 dm2) 1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity Ah/dm2 4th cycle	Yield 4th cycle Q/ΔP Ah/g	Av. yield Ah/g
1511	a	50	180	540	43,700	70,480	11,6	-	1,99	1,98	2,00	4,00	0,343	0,34
	b							-	2,06	2,06	2,00		0,343	
512	a	50	210	630	42,230	67,840	11,1	-	1,87	1,80	1,84	3,74	0,330	0,[illegible]
	b							-	1,89	1,89	1,91		0,343	
513	a	50	240	720	43,950	70,220	11,4	-	1,97	1,97	2,10	4,14	0,368	0,[illegible]
	b							-	1,93	2,00	2,04		0,357	
1521	a	75	120	540	43,605	68,40	10,8	1,61	1,69	1,62	1,57	3,22	0,291	
	b							1,61	1,52	1,58	1,65		0,306	0,[illegible]
1522	a	75	140	630	43,560	70,810	11,8	1,74	1,69	1,67	1,86	3,72	0,314	0,31
	b							1,87	1,86	1,86	1,86		0,314	
1523	a	75	160	720	43,790	71,010	11,8	1,74	1,69	1,64	1,69	3,50	0,285	0,29
	b							1,82	1,74	1,78	1,80		0,304	
1531	a	95	95	540	42,390	67,210	10,8	1,74	1,69	1,65	1,61	3,26	0,298	0,30
	b							1,67	1,46	1,43	1,65		0,306	
1532	a	95	110	630	43,505	70,550	11,7	1,87	1,86	1,86	1,90	3,70	0,323	0,31
	b							1,74	1,69	1,82	1,80		0,306	
1533	a	95	125	720	43,900	71,260	11,9	1,91	1,70	1,76	1,74	3,46	0,292	0,2
	b							1,81	1,70	1,65	1,72		0,289	

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TABLE 4.7

IMPREGNATED PLATE CHARACTERISTICS

Impregnation bath

pH = 3-3,5

Concentration NI+Co = 2M/1
Concentration Co = 5 %

Alcohol content : 50 %
Temperature : 80° C

		Impregnation conditions						Impregnated plate characteristics						
								Capacities in Ah (S = 0,50 dm2)						
Reference sample		Current density (mA.cm2)	Impreg. time (mn)	Current Quantity (C/cm2)	El. wt. before impregn.	El. wt. after impreg. in g.	ΔP (g/dm2)	1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity 4th cycle (Ah/dm2)	Yield 4th cycle Q/ΔP (Ah/g)	Av. yield (Ah/
211	a	50	180	540	41,730	67,590	11,2	1,74	1,78	1,76	1,76	3,44	0,314	0,3
	b							1,72	1,78	1,76	1,68		0,300	
212	a	50	210	630	41,540	67,580	11,3	2,06	2,14	2,21	2,04	4,16	0,361	0,36
	b							1,94	2,02	2,08	2,12		0,375	
213	a	50	240	720	41,730	68,550	11,7	1,94	2,00	2,04	2,16	3,92	0,369	-
	b							1,72	1,85	1,82	1,76		0,309	
221	a	75	120	540	42,350	65,200	9,5	1,48	1,44	1,54	1,52	2,96	0,320	0,
	b							1,18	1,44	1,57	1,44		0,303	
222	a	75	140	630	41,590	65,880	10,6	1,74	1,76	1,76	1,76	3,54	0,332	0,3
	b							1,74	1,76	1,78	1,78		0,336	
223	a	75	160	720	42,180	65,580	10,2	1,50	1,57	1,76	1,74	3,46	0,341	0,3
	b							1,50	1,57	1,67	1,72		0,337	
2231	a	95	95	540	41,530	61,560	8,7	1,31	1,27	1,25	1,26		0,290	0,3
	b							1,31	1,33	1,52	1,46	2,72	0,335	
2232	a	95	110	630	41,570	60,570	8,3	1,00	1,16	1,25	1,24		0,299	0,3
	b							1,00	1,16	1,25	1,26	2,50	0,304	
2233	a	95	125	720	42,940	62,670	8,6	1,12	1,16	1,25	1,26	2,50	0,293	0,
	b							1,12	1,16	1,25	1,24		0,288	

MEASUREMENTS OF ELECTRODE THICKNESSES - TABLE 5.1

Impregnation Bath

pH : 1,5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content : 0 %
Temperature : 98° C

Reference Sample		Impregnation Conditions		Thickness before impregnation 4pts (mm/100)	Av. Thic mm/100	Nonformed impregnated thicknesses (mm/100)	Av. thic mm/100	Impregnated thicknesses formed (mm/100)	Av. thic mm/100	Thickness variation before formation (%)		Thickness variation during formation (%)		Thickness variation of formed electrodes (%)	
1111	a	50mA/cm2	180 mn	88 90 93 91	90,5	88 88 94 89	90			0					
	b			87 90 94 93	90	87 87 95 90	90	104 103 115 113	109	0	-1	+24	+23	+24	+21
	c			90 90 91 93	91	90 89 90 87	89	108 106 105 105	106	-2,5		+21,5		+19	
1112	a	"	210 mn	89 90 93 90	90,5	85 87 92 89	88			-3					
	b			89 90 90 91	90	90 90 87 92	90	119 122 119 122	120,5	0	-1	+39	+38	+39	+38
	c			90 90 94 90	91	91 91 91 91	91	119 118 119 118	120,5	0		+37		+37	
1113	a	"	240 mn	89 89 95 91	91	89 90 95 90	91			0					
	b			90 90 95 33	91,5	92 94 96 91	93	120 127 131 124	125,5	+2	0	+41	+42	+43	+43
	c			93 93 93 89	92	92 90 92 91	91	124 123 127 126	125	-1		+43		+42	
1121	a	75mA/cm2	120 mn	88 93 96 91	92	91 92 100 92	94	104 103 104 100	103	+2,5		+11	+9	+14	+11
	b			89 89 95 90	91	92 87 95 93	92	99 95 100 98	98	+1	0	+7,5		+9	
	c			91 96 96 95	94,5	93 93 92 94	93			-2					
1122	a	"	140 mn	94 94 90 90	92	94 96 93 92	94			+2,5					
	b			98 90 95 88	90	92 94 95 93	93,5	102 102 104 99	102	+4,5	+3	+11	+12	+15	+15
	c			86 92 94 93	91	91 93 95 94	93	106 108 99 99	103	+2,5		+13		+15	
1123	a	"	160 mn	88 96 96 90	92,5	91 99 103 92	96			+4					
	b			90 94 91 90	91	95 95 93 93	94	100 102 106 100	102	+4	+4	+10	+17	+14	+21
	c			89 92 93 92	91,5	93 95 97 94	95	113 117 120 111	115	+4		+25		+29	
1131	a	95mA/cm2	95 mn	90 91 92 91	91	92 90 87 92	90			-1					
	b			88 90 95 93	91,5	95 91 100 96	95,5	119 118 103 118	116	+5	+5	+26	+21	+31	+26
	c			89 89 92 88	89,5	95 93 96 93	94	106 103 108 109	106,5	+6		+16		+22	
1132	a	"	110 mn	93 97 96 98	56	99 105 102 98	101			+6					
	b			95 91 90 86	90,5	102 97 100 94	98	122 118 119 116	119	+9,5	+7,5	+26	+30	+36	+36
	c			91 93 92 86	90,5	97 97 97 93	96	124 133 120 116	123	+7		+34		+41	
1133	a		125 mn	88 89 91 90	89,5	95 97 97 95	96			+8					
	b			90 93 94 95	93	94 99 100 101	99	122 131 128 122	126	+7	+8	+33	+32	+40	+40
	c			88 91 92 90	90	94 97 100 96	97	120 120 122 122	121	+9		+31		+40	

MEASUREMENTS OF ELECTRODE THICKNESSES - Impregnation Bath

TABLE 5.2

pH : 1,5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content : 0 %
Temperature : 98° C

Reference Sample		Impregnation conditions		Thickness before impr 4pts (mm/100)	Nonformed impregnated thicknesses (mm/100)		Av. Thic mm/100	Impregnated thicknesses formed (mm/100)	Av. thi. mm/100	Thickness variation before formation (%)		Thickness variation during formation (%)		Thickness variation of formed electrodes (%)	
1111	a	50mA/cm2	180 mn	88 90 93 91	90,5	88 88 94 89	90			0					
	b			87 90 94 93	90	87 87 95 90	90	104 103 115 113	109	0	-1	+24	+23	+24	+21
	c			90 90 91 93	91	90 89 90 87	89	108 106 105 105	106	-2,5		+21,5		+19	
1112	a	"	210 mn	89 90 93 90	90,5	85 67 92 89	88			-3					
	b			89 90 90 91	90	90 90 87 92	90	119 22 119 122	120,5	0	-1	+39	+38	+39	+38
	c			90 90 94 90	91	91 91 91 91	91	119 [illegible] 119 118	120,5	0		+37		+37	
1113	a	"	240 mn	89 89 95 91	91	89 90 95 90	91			0					
	b			90 90 95 93	91,5	92 94 96 91	93	120 127 131 124	125,5	+2	0	+41	+42	+43	+43
	c			93 93 93 89	92	92 90 92 91	91	124 123 127 126	125	-1		+43		+42	
1121	a	75mA/cm2	120 mn	88 93 [illegible] 91	92	91 92 100 92	94	104 103 104 100	103	+2,5		+11	+9	+14	+11
	b			89 [illegible] [illegible] 90	91	92 87 95 93	92	99 95 100 98	98	+1	0	+7,5		+9	
	c			91 96 96 95	94,5	93 93 92 94	93			-2					
1122	a	"	140 mn	94 94 90 90	92	94 96 93 92	94			+2,5					
	b			88 90 95 88	90	92 94 95 93	93,5	102 102 104 99	102	+4,5	+3	+11	+12	+15	+15
	c			86 92 94 93	91	91 93 95 94	93	106 108 99 99	103	+2,5		+13		+15	
1123	a	"	160 mn	88 96 96 90	92,5	91 99 103 92	96			+4					
	b			90 94 91 90	91	95 95 93 93	94	100 102 105 100	102	+4	+4	+10	+17	+14	+21
	c			89 92 93 92	91,5	93 95 97 94	95	113 117 120 111	115	+4		+25		+29	
1131	a	95mA/cm2	95 mn	90 91 92 91	91	92 90 87 92	90			-1					
	b			88 90 95 93	91,5	95 91 100 96	95,5	119 118 109 118	116	+5	+5	+26	+21	+31	+26
	c			89 89 92 88	89,5	95 93 96 93	94	106 103 108 109	106,5	+6		+16		+22	
1132	a		110 mn	93 97 96 98	96	99 105 102 98	101			+6					
	b			95 91 90 86	90,5	102 97 100 94	98	122 118 119 116	119	+9,5	+7,5	+26	+30	+36	+38
	c			91 93 92 86	90,5	97 97 97 93	96	124 133 120 116	123	+7		+34		+41	
1133	a		125 mn	88 89 91 90	89,5	95 97 97 95	96			+8					
	b			90 93 94 95	93	94 99 100 101	99	122 131 128 122	126	+7	+8	+33	+32	+40	+40
	c			88 91 92 90	90	94 97 100 96	97	120 120 122 122	121	+9		+31		+40	

ELECTRODE THICKNESS MEASUREMENTS - TABLE 5.2.

Impregnation bath
pH : 1,5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content: 20 %
Température : 84° C

Reference Sample		Impregration conditions		Thickness before impregnation 4 pts (mm/100)				Av. thic, mm/100	Nonformed impregnated thicknesses (mm/100)				Av. thick mm/100	Formed impregnated thick. formées (mm/100)				Av. thick mm/100	Thickness variation before formation (%)	Thickness variation during formation (%)	Thickness variation of formed electrodes (%)	
1211	a	50mA/cm2	180mn	88	94	91	90	91	102	110	105	103	105	97	99	96	95	97	+18	-10	+8	+8
	b			92	95	98	87	93	100	101	105	97	101	96	99	105	94	98,5	+10	-3	+7	
	c			92	97	92	88	92	96	95	94	94	95						+4			
1212	a	"	210mn	92	90	97	90	92	98	97	103	97	99	96	96	106	100	99,5	+9	0	+9	+9
	b			86	90	96	88	90	95	98	105	97	99	93	99	103	97	98	+11,5	-1	+10	
	c			90	93	91	88	90,5	98	101	99	93	98						+9,5			
1213	a	"	240mn	92	92	94	93	93	87	86	94	86	88						-6			
	b			92	98	93	87	93	97	104	99	94	98,5	95	104	95	92	96,5	+7	-2,5	+4,5	+6
	c			89	90	93	88	90	91	91	97	92	93	94	98	100	92	96	+4	+4	+8	
1221	a	75mA/cm2	120mn	91	94	90	91	91,5	103	103	97	100	101	93	98	97	94	95,5	+12	-7	+5	+5
	b			95	90	90	90	91	101	94	95	93	96	97	94	96	92	95	+6	-1	+5	
	c			90	96	92	90	92	96	100	91	97	96						+5			
1222	a	"	140mn	87	98	88	92	91	96	103	92	99	97,5	91	101	94	98	96	+8	-2	+6	+5
	b			90	92	93	88	91	93	97	95	90	94	91	98	96	91	94,5	+4	0	+4	
	c			88	91	93	89	90	91	95	93	90	92						+2,5			
1223	a	"	160mn	84	91	91	93	90	95	101	98	103	99	90	95	95	98	94,5	+11,5	-6	+6	+5
	b			88	92	90	88	89,5	97	102	100	98	99	90	95	95	94	93,5	+12	-7	+5	
	c			92	89	93	92	91,5	95	97	100	94	96,5						+6			
1731	a	95mA/cm2	95mn	86	90	89	92	89	100	100	100	100	100	88	91	91	93	91	+14	-12	+2	+2
	b			88	94	95	90	92	97	100	99	97	98	91	94	98	91	93,5	+8	-6	+2	
	c			94	90	88	90	90,5	102	98	95	99	98,5						+10			
1232	a		110mn	91	90	90	91	90,5	100	100	94	98	98	96	92	94	95	94	+10	-5	+5	+5
	b			94	90	92	91	92	97	98	97	97	97	98	96	96	96	96,5	+6	0	+6	
	c			91	91	91	89	90,5	96	94	95	94	95						+6			
1233	a		125mn	93	93	90	87	91	106	103	106	99	103,5	95	92	92	90	92	+16	-14	+2	+2
	b			89	93	90	93	91	99	101	100	100	100	90	95	92	94	93	+11	-9	+2	
	c			93	90	90	87	90	93	92	92	91	92						+2,5			

ELECTRODE THICKNESSES MEASURED

TABLE 5.3

Impregnation Bath
pH : 1,5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content: 50 %
Température : 10° C

Reference Sample		Impregnation Conditions		Thickness before impr 4pts (mm/100)	Av. thick mm/100	Nonformed impregnated thicknesses (mm/100)	Av. thick mm/100	Formed impregnated thicknesses (mm/100)	Av. thick mm/100	Thickness variation before formation (%)	Thickness variation during formation (%)	Thickness variation of formed electrode	
1311	a	50mA/cm2	180mn	86 94 96 92	92	97 104 103 100	101	95 102 104 100	100	+11	-1	+10	
	b			88 91 91 90	90	88 87 90 89	88,5	84 88 87 86	86	-2	-3	-5	
	c			87 91 89 86	88	90 90 93 88	90			+3			
1312	a	"	210mn	90 89 91 92	90,5	102 101 104 103	102,5	99 98 101 101	100	+15	-3	+12)	+12
	b			89 90 91 88	89,5	98 100 105 99	[illegible]	98 100 98 100	100	+14	-1	+13 (	
	c			89 90 92 90	90	100 104 101 101	[illegible]			+14			
1313	a	"	240mn	96 94 94 92	94	108 104 108 106	107	107 103 108 107	106	+16	-1	+15)	+14
	b			92 95 95 93	94	106 111 109 [illegible]	108	103 109 106 104	105,5	+17	-3	+14 (	
	c			93 90 97 90	92,5	105 106 110 106	107			+18			
1321	a	75mA/cm2	120mn	90 89 95 92	91,5	99 101 102 101	101	95 100 102 98	99	+12	-2	+10)	+10
	b			88 90 92 87	89	96 97 101 98	98	95 95 98 98	96,5	+12	-2	+10 (	
	c			90 94 93 91	92	93 96 94 91	93,5			+2			
1322	a	"	140mn	93 90 95 88	91,5	101 100 107 100	102	96 94 102 97	97	+13	-6	+7)	+8
	b			91 88 90 90	90	100 98 98 96	98	100 95 100 97	98	+10	0	+10 (	
	c			86 88 89 90	88	92 96 96 93	94			+8			
1323	a	"	160mn	92 95 92 91	92,5	105 108 107 101	105	97 101 101 97	99	+15	-7	+8)	+9
	b			85 88 89 90	88	99 102 100 101	100,5	95 94 99 97	96	+16	-6	+10 (	
	c			90 89 92 88	90	105 102 104 100	103			+16			
1331	a	95mA/cm2	95mn	90 93 91 91	91	99 97 94 97	97	96 93 95 94	94,5	+7	-3	+4)	+5
	b			89 90 93 90	90,5	96 97 98 100	98	93 95 100 94	95,5	+9	-3	+6 (	
	c			89 89 94 90	89,5	91 90 93 90	91			+2			
1332	a		110mn	88 89 90 90	89	102 100 102 100	101	97 90 94 94	94	+15	-9	+6)	+8
	b			90 90 90 90	90	102 102 100 100	101	97 98 98 94	97	+14	-5	+9 (	
	c			90 90 90 90	90	100 97 100 95	98			+10			
1333	a		125mn	93 88 94 88	91	110 100 109 105	106	96 92 96 93	94	+19	-15	+4)	+6
	b			92 94 93 90	92	106 110 103 101	105	98 100 99 94	98	+16	-9	+7 (	
	c			90 91 92 92	91	103 106 109 105	106			+19			

ELECTRODE THICKNESS MEASUREMENTS

TABLE 5.4

Impregnation Bath
pH : 1,5 ~

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content 50 %
Température : 50° C

Reference	Sample	Impregnation Conditions		Thickness before impregnation 4 pts (mm/100)	Av. thick mm/100	Impregnated thicknesses formed (mm/100)	Av. thick mm/100	Impregnated thicknesses formed (mm/100)	Av. thick mm/100	Thickness variation before formation	Thickness Variation during formation (%)	Thickness variation of formed electrodes (%)	
1411	a	50mA/cm2	180mn	88 90 89 88	89	86 88 88 91	88	89 89 90 88	89	-1	+1	0)	+1
	b			88 88 86 87	87	90 88 89 87	88,5	91 89 89 86	89	+2	0	+2 (	
	c			88 89 90 88	89	85 87 91 88	88			+1			
1412	a	"	210mn	88 89 89 90	89	86 89 89 91	89	92 92 91 89	91	0	+2	+2)	+1
	b			88 90 91 85	88,5	90 90 91 87	89,5	88 90 89 86	88	+1	-1	0 (	
	c			90 89 90 88	89	90 91 92 87	90			+1			
1413	a	"	240mn	88 89 89 91	89	90 92 91 92	91	94 90 90 95	91	+2	0	+2)	+2
	b			87 88 89 91	89	90 90 91 94	91	91 92 91 95	92	+2	+1	+3 (	
	c			90 89 92 86	89	92 92 95 90	92			+4			
1421	a	75mA/cm2	120mn	90 90 91 93	91	89 89 91 92	90	90 92 93 92	92	-1	+2,5	+1,5)	+3
	b			87 90 88 87	88	89 89 89 91	89,5	91 91 91 91	91	+2	+2	+4 (	
	c			89 90 90 89	89,5	86 87 88 86	87			-3			
1422	a	"	140mn	90 90 90 88	89,5	95 99 104 98	99	93 94 96 93	94	+12	-6	+6)	+6
	b			88 88 90 88	88,5	96 93 92 96	94	93 94 92 91	93	+7	-1	+6 (	
	c			89 88 91 89	89	93 96 96 96	95			+8			
1423	a	"	160mn	88 88 91 87	88,5	100 96 100 96	98	92 93 92 93	92,5	+12	-7	+5)	+5
	b			90 90 88 86	88,5	95 97 97 96	96	96 94 93 90	92,5	+10	-4,5	+5,5 (	
	c			88 88 89 89	88,5	99 98 94 99	97,5			+12			
1431	a	95mA/cm2	95mn	93 90 93 90	91,5	98 92 92 93	94	96 94 97 92	95	+3	+1	+4)	+4
	b			87 91 92 88	89,5	90 96 93 93	93	91 96 95 92	93,5	+4	0	+4 (	
	c			87 91 91 89	89,5	87 92 93 89	91			+2			
1432	a	"	110mn	89 90 91 88	89,5	91 92 93 95	92	92 96 94 92	93,5	+3	+2	+5)	+5
	b			90 88 91 87	89	92 95 96 92	94	96 93 95 90	93,5	+6,5	-1	+5,5 (	
	c			89 89 91 93	90,5	90 90 94 94	92			+2			
1433	a	"	125mn	90 89 91 90	90	94 97 95 96	95,5	92 90 96 96	93,5	+7	-2,5	+4,5)	+4
	b			88 89 89 92	89,5	90 92 91 94	92	91 92 92 96	93	+3	+1	+4 (	
	c			88 90 89 92	90	90 92 91 93	91,5			+2			

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ELECTRODE THICKNESS MEASUREMENTS

TABLE 5.5

Impregnation Bath
pH : 1,5-2

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content 70
Temperature : 80° C

Reference Sample		Impregnation Conditions		Electrode thicknesses prior to formation (mm/100)	Mean Thickness (mm/100)	Electrode thicknesses after formation (mm/100)	Mean Thickness (mm/100)	Thickness variation before formation (%)	Thickness variation during formation (%)	Thickness variation of formed electrode (%)	
1611	a	50 mA/cm2	180 mn	99 99 89 95	95,5	98 100 91 94	95,5	+5	0	+5)	+3
	b			96 90 91 93	92,5	94 89 90 91	91	+1	-1	0 (	
1612	a	"	210 mn	94 93 97 92	94	94 91 97 91	93	+3	-1	+2)	+2
	b			92 98 93 96	95	92 96 92 92	93	+4	-2	+2 (	
1613	a	"	240 mn	95 94 94 95	94,5	93 90 90 93	91,5	+4	-4	0)	+1
	b			97 99 99 98	98	96 94 91 93	93,5	+8	-6	+2 (	
1621	a	75 mA/cm2	120 mn	96 93 92 95	94	94 92 92 96	93,5	+3	-1	+2)	[illegible]
	b			96 94 93 94	94	94 92 92 95	93	+3	-1	+2 (	
1622	a	"	140 mn	94 91 93 93	93	94 92 91 93	92	+2	-1	+1)	0
	b			93 90 92 94	92	93 88 89 93	91	+1	-1	0 (	
1623	a	"	160 mn	105 99 103 102	102	92 91 97 94	93,5	+13	-11	+2)	+1
	b			94 95 92 98	95	92 90 88 93	91	+4	-4	0 (	
1631	a	95 mA/cm2	95 mn	98 96 97 92	96	93 90 92 97	93	+6	-4	+2)	+3
	b			93 98 99 103	98	97 94 95 93	95	+8	-4	+4 (	
1632	a	"	110 mn	92 96 96 91	94	92 97 97 95	95	+3	+1	+4)	+4
	b			94 96 94 94	94,5	96 96 96 91	95	+4	0	+4 (	
1633	a	"	125 mn	96 91 93 89	92	92 92 93 90	92	+1	0	+1)	[illegible]
	b			93 90 91 91	91	92 91 89 91	91	0	0	0 (	

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ELECTRODE THICKNESS MEASUREMENTS

TABLE 5.6

Impregnation Bath
pH : 1,5-2

Concentration Ni+Co = 3 M/1
Concentration Co = 3,3 %

Alcohol content 50 %
Température : 30° C

Reference Sample		Impregnation Conditions		Impregnated electrode thicknesses before formation (mm/100)	Mean Thicknesses (mm/100)	Impregnated Electrode Thicknesses After Impregnation mm/100	Mean Thicknesses (mm/100)	Thickness variation before formation (%)	Thickness variation during formation (%)	Thickn. Varia. of formed Electr.
1511	a	50 mA/cm2	180 mn	100 102 102 99	101	98 98 101 99	99	+12	-2	+10)
	b			94 95 97 94	95	97 95 98 95	96	+4	+1	+5 (
1512	a	"	210 mn	98 91 92 98	95	98 91 93 93	94	+4	-1	+3)
	b			95 93 90 91	92	95 90 90 90	91	+1	-1	0 (
1513	a	"	240 mn	97 98 97 94	96,5	94 98 98 97	97	+6	0	+6)
	b			94 94 96 96	95	95 95 96 95	95	+4	0	+4 (
1521	a	75 mA/cm2	120 mn	105 102 99 99	101	105 100 98 100	101	+12	0	+12)
	b			104 100 103 100	102	99 98 100 98	98,5	+13	-4	+9 (
1522	a	"	140 mn	107 103 105 98	103	106 106 104 102	104,5	+14	+2	+16)
	b			106 104 107 107	106	105 106 104 108	106	+18	0	+18(
1523	a	"	160 mn	101 102 96 100	100	103 102 95 100	100	+11	0	+11)
	b			105 105 109 104	106	105 103 107 101	104	+18	-2	+16(
1531	a	95 mA/cm2	95 mn	100 96 94 96	96,5	95 96 100 93	96	+6	0	+6)
	b			100 99 101 95	99	95 97 92 95	95	+9	-5	+4 (
1532	a	"	110 mn	93 100 98 95	96,5	94 98 98 94	96	+6	0	+6
	b			93 103 103 92	98	94 103 103 98	99,5	+8	+2	+10
1533	a	"	125 mn	97 104 108 98	102	97 103 103 97	100	+13	-2	+11
	b			103 102 103 95	101	102 103 103 96	101	+12	0	+12

ELECTRODE THICKNESS MEASUREMENTS

TABLE 5.7

Impregnation Bath

pH : 3-3,5

Concentration Ni+Co = 2 M/1
Concentration Co = 5 %

Alcohol content 50 %
Temperature : 80°C

Reference Sample		Impregnation Conditions		Impregnated Electrode Thicknesses Before Formation (mm/100)	Mean Thicknesses (mm/100)	Impregnated Electrode Thicknesses after formation (mm/100)	Mean thicknesses (mm/100)	Thickness variation before formation (%)	Thickness variation during formation (%)	Thickn varia. of formed el. (%)
2211	a	50mA/cm2	180 mn	102 99 105 106	103	101 100 104 106	103	+14	0	+14
	b			100 100 96 98	98,5	101 101 97 98	99	+9	0	+9
2212	a	"	210 mn	102 96 102 94	98,5	97 95 95 97	96	+9	-3	+6
	b			96 92 94 96	94,5	98 95 96 95	96	+4	+2	+6
2213	a	"	240 mn	89 90 93 94	91,5	95 96 95 100	96,5	0	+6	+6
	b			90 92 91 91	91	94 95 94 93	94	0	+3	+3
2221	a	75 mA/cm2	120 mn	99 95 93 97	96	96 95 93 96	95	+6	-1	+5
	b			102 92 94 98	96,5	101 94 95 95	96	+6	0	+6
2222	a	"	140 mn	102 95 95 92	96	103 97 100 95	99	+6	+3	+9
	b			99 97 96 97	97	102 99 96 99	99	+7	+2	+9
2223	a	"	160 mn	100 101 101 98	100	100 101 99 96	99	+11	-1	+10
	b			97 98 97 93	96	99 97 95 90	95	+6	-1	+5
2231	a	95 mA/cm2	95 mn	94 95 91 96	94	94 90 91 95	92,5	+3	-2	+1
	b			89 86 89 88	88	91 88 88 90	89	-3	0	-3
2232	a		110 mn	95 92 95 94	94	98 93 96 92	95	+3	+2	+5
	b			93 94 89 95	93	93 91 91 92	92	+2	-1	+1
2233	a		125 mn	95 90 93 95	93	94 95 91 93	93	+2	0	[illegible]
	b			98 94 95 95	93,5	101 93 95 92	95	+5	0	[illegible]

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ELECTRODE CHARACTERISTICS DURING CYCLING

TABLE 6

Reference Sample	Thicknesses (mm/100)				Capacities (in Ah/dm2)				
	Prior to Formation	After Formation	After 21 cycles	After 180 cycles	4th cycle Formation	21st cycle (cyclage)	44th cycle	64th cycle	80th cycle
24*	87	110	120	131	4,21	4,08			
25*	85	92	98	106,5	3,70	3,50			
44*	76	92	103	109	3,62	3,45			
1122	93	102	112	121,5	4,05	3,87			
1211	103	98	99	110	4,14	3,63			
1223	99	94	95	102	4,12	3,50			
1313	108	106	104	111	4,25	4,07			
1511	105	105	104,5		4,06	4,09	4,27	4,36	
1522	105	105	106,5		3,72	3,58	3,73	3,90	
1532	97	98	96		3,70	3,70	3,85	3,95	
1612	94	93	93		3,50	3,55	3,67	3,72	
1622	92,5	91,5	92		3,34	3,44	3,52	3,57	
1632	94	95	93,5		3,40	3,53	3,62	3,64	
2113	96,5	95	94		3,50	3,37	3,53	3,55	
2313	99,5	99	100		3,68	3,85	4,12	4,12	
2413	99,5	99	101,5		3,60	4,03	3,91	3,87	
2123	99,5	97	96		3,58	3,70	3,80	3,85	
2323	95	94	92		3,00	3,08	3,16	3,21	
2423	96,5	94,5	95		3,48	3,44	3,50	3,64	
1**	90	98,5	107		4,00	4,07	4,22	4,30	
2***	90	101,5	101	102 (cycle 44)	3,35	3,65	3,92	3,79	

* Chemically impregnated electrodes (see table 2)
** Chemically impregnated electrodes without alcohol
*** Chemical impregnation in Ni(NO3)2 solution containing 50% alcohol.

TABLE 7 BY ELECTROCHEMICAL IMPREGNATION - Mean sintering wt. before imprg: 7.4 g/electrod

No. of Electrodes	Wt. (%)	Filling Δp (g/dm2)	Cluster No.	Formation Capacities (Ah/dm2)				Thicknesses		Swelling (%)
				1st cycle	2nd cycle	3rd cycle	Resid.	Before Formation	After Formation	
								mm/100		
1	12,3)	11,7	111 A					97		
7	12,0(	11,0		3,98	3,52	3,55	0,18	98	94	0
10	12,1)	11,2						101	99	0
13	13,1(12,38	13,8						102		
16	12,7)	12,8						102		
19	12,5(	12,1						99		
22	12,1)	11,2						98,5	98	0
26	12,4(	11,8						101		
29	12,2)	11,4						98,5	98	0
32	12,4)	11,8	111 B					99,5		
35	12,2(	11,4		3,88	3,52	3,55	0,14	98		
41	12,1)	11,2						98	98,5	0
44	12,2(12,40	11,4						100		
65	12,8)	12,4						97		
68	11,9(	11,7						95,5		
71	12,6)	12,4						101	102	1
74	12,5(	12,1						90	91	1
80	13,1)	13,6						102	103	1
83	13,0)	13,8	112 A					93	94	1
87	12,3(	11,7		4,07	3,58	3,64	0,14	100		
90	12,7)	12,6						103		
2	12,9(12,82	13,1						102	104	2
5	12,3)	11,7						95		
8	12,2(	11,4						94		
11	12,8)	12,8						105	106	0
14	12,9(	13,1						102	102	0
17	12,5)	12,1						101		
23	12,5)	12,1	112 B					102	102	0
27	12,7(	12,6		3,98	3,55	3,55	0,18	101		
30	12,5)	12,1						101	100	0
33	12,4(12,37	11,9						100		
42	12,7)	12,6						100		
45	11,7(	10,3						92		
64	12,3)	11,7						95,5	93	0
67	12,0(	11,0						97		
70	12,5)	12,1						100,5	100	0
73	12,6)	12,6	113 A					92	93	1
76	12,3(	11,7		4,07	3,58	3,58	0,20	100,5	100	0
82	12,8)	12,8						99		
86	12,6(12,57	12,4						101		
89	12,8)	12,8						102	102	0
3	12,7(	12,6						98		
6	12,4)	11,9						92		
9	12,4(	11,9						95	97	2
12	12,6)	12,4						102,5		
15	12,7)	12,6	113 B					100		
18	12,3(	11,7		3,98	3,52	3,55	0,14	97,5	99	2
21	13,0)	13,3						102	101,5	0
25	12,0(12,33	11,0						98		
28	12,1)	11,2						97		
31	11,8(	10,5						98	95	-1
40	12,1)	11,2						99		
43	12,6(	12,4						99		
66	12,4)	11,9						94	94	0
69	11,3)	9,3	114 A					94		
72	12,9(	13,1		3,89	3,52	3,52	0,18	101	101	0
75	12,7)	12,6						93		
78	12,6(12,42	12,4						101,5	101	
81	12,9)	13,1						98	[illegible]	1
84	13,1	13,6						94		
88	11,3.	11,7						95	96	1
4	12,0(	11,0						93		
77	12,4)	11,9						100		
46	13,1)	13,6	114 B					100		
52	12,6(	12,4		4,04	3,55	3,58	0,18	92,5	93	0
55	12,7)	12,8						91		
58	12,7(12,40	12,6						91,5	92	0
61	12,8)	12,8						98		
91	12,7(	12,7						101,5	102,5	1
94	11,6)	10,0						91,5		
97	11,4(	9,5						94,5		
100	12,0)	11,0						96	96	0
38	12,6)	12,4	115 A	4,12	3,46	3,52	0,18	98		
47	13,0(	13,3						98,5	101	+5
53	12,3)	11,7						91		
56	12,9(12,41	13,1						95	95,5	0
59	12,3)	11,7						91,5	92	0
62	12,0(	12,4						98		
92	12,8)	12,8						94,5	96	+2
95	11,5(	9,8						95		
101	11,7)	10,2						93,5		
39	12,2)	11,4	115 B	4,12	3,46	3,52	0,15			
48	12,9(	13,1						98	98	0
54	12,3)	11,7						97,5		
57	12,6(12,26	12,4						93		
63	12,8)	12,8						92	97	0
93	12,9(	13,1						101	100	0
[illegible]	10,2)	[illegible]						96		

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TABLE 8

CHARACTERISTICS OF Ni-Cd Type ELECTROCHEMICALLY IMPREGNATED PLATES*

	Type of impregnation		Electrochemical Impregnation						Chemic. impreg. refer.
	Thickness (mm/100)		75	75			76		75
	Porosity (%)		78	82			85		82
	Surface treatment		non-passiv.	passivated	non-passiv.	Reduced	Nonpassivated	passiv.	non-passiv.
	Reference Sample		111	242	211	231	311	321	221
IMPREGNATION	Filling : ΔP (g/dm2)		8,26	9,82	8,79	9,53	10,39	10,77	/
	Corroded Nickel (%)		0	0	1,5	2,5	3	3	22
FORMATION	Capacities: (Ah/dm2)	1st cycle	2,44	2,86	2,22	2,95	2,54	3,20	3,36
		2nd cycle	2,48	2,83	2,27	2,95	2,64	3,23	3,57
		3rd cycle	2,48	2,93	2,34	3,22	2,67	3,41	3,64
	Yield (Ah/g hydrate) (3rd cycle 0/ΔP)		0,300	0,298	0,266	0,338	0,257	0,317	/
	Thickness (mm/100)	Before formation	75	79.5	80,5	80	81	81	76,5
		After formation	75,5	80,5	80	80	84,5	82,5	96,5
		After cycling (50 cycles)	75,5	77	80	80	84	82,5	105
	Swelling after cycling (%)		0	0	0	0	4	2	44

Impregnation conditions: bath : Ni + Co = 2 M/l
Co = 5 %
pH = 1,5 - 2
Alcohol = 50 %
Temperature = 80° C

Electrolysis: current density = 50 mA/cm2
time = 2h 30

TABLE 9

CHARACTERISTICS OF Ni-Cd TYPE ELECTROCHEMICALLY IMPREGNATED PLATES* - INFLUENCE OF THE ELECTROLYSIS CONDITIONS

Reference Sample	Porosity (%)	Electrolysis			Capacities (Ah/dm2)
		Current density (mA/cm2)	Impregnation time (mn)	Current quantity (c/cm2)	
111	78	50	150	450	2,5
211	82	50	150	450	2,3
311	85	50	150	450	2,7
114	78	50	240	720	3,3
214	82	50	240	720	3,5
314	85	50	240	720	3,8
117	78	75	100	450	3,8
217	82	75	100	450	3,2
317	85	75	100	450	3,4

Impregnation Conditions : Bath :

: Ni + Co = 2 M/1
Co = 5 %
pH = 1,5 - 2
Alcohol = 50 %
Temperature = 80° C

*Contract 75/CNES/0590 - Report DT/SAS-333/76

** Reference - Chemical impregnation : 3,6 Ah/dm2
3rd formation cycle : charge 4 h à C/2
discharge C/2 (stop at 0,5 V).

TABLE 13

CHARACTERISTICS OF Ni-Cd TYPE SINTERINGS USED

TYPE OF SINTERING	Sintering No. 3 Ni-Cd (VO S)	Sintering No. 4 Ni-Cd High Porosity
Thickness (mm/100)	79 ± 2	79 ± 2
Sheet weight (g/dm2)	5,1	5,1
Nickel coating wt. (g/dm2)	10,8 ± 0,2	8,8 ± 0,2
Sintering strength (gf)	600	320
Positive capacity-sintering (mAh/dm2)	2,5	4,8
Theoretical porosity (cm3/g sintered pl	0,377	0,447
Open porosity (cm3/g sintered plate)	0,310	0,377
Theoretical poros. (%)	82	85,5
Accessible vacuum vol. (cm3/dm2)	4,9	5,2

TABLE 11

CHARACTERISTICS OF POSITIVE Ni-Cd CHEMICALLY IMPREGNATED PLATES

	Reference Sample		44
SINTERING	Sintering Reference (No.)		3
	Thickness (mm/100)		79 ± 2
	Porosity (%)		82
IMPREG-NATION	Hydrate weight (g.dm2)		12
	Corroded nickel (%)		14
FORMATION	Capacities (Ah/dm2) Formation	1st cycle	3,17
		2nd cycle	3,30
		3rd cycle	3,44
		4th cycle	3,62
	Yield (Ah/g hydrate)		0,302
	Thickness (mm/100)	Before formation a	76,5
		b	76
		c	76
		After formation: a	92
		b	92
		c	92
		Swelling (%)	24

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CHARACTERISTICS OF ELECTROCHEMICALLY IMPREGNATED Ni-Cd TYPE POSITIVE PLATES

TABLE 12.1

Impregnation Bath : / concentration Ni+Co=2M/1 /

concentration Co = 5 %

"pH" 1,5-2 Alcohol cont.: 50 %
Temperature : 80° C

Reference Sample	Impregnation Condition			Characteristics of impregnated electrodes								
	Current density	Impregnation time (mn)	Current quantity (c/cm2)	Electrode wt. before impregnation (g) S=2.3dm2	El.wt. after impregn. (g) S=2.3 dm2	ΔP (g.dm2)	Wt. loss during formation (g/dm2)	Capacities in Ah (S = 0,50 dm2)				Mean capac. 4th cycle Ah/dm2
								1st cycle	2nd cycle	3rd cycle	4th cycle	
3312 a	50	210	630	40,320	64,670	10,6	-1,6	1,76	1,69	1,70	1,70	
b							-1	1,76	1,69	1,72	1,70	
3313 a	50	240	720	40,190	67,550	11,9	-2,8	1,72	1,63	1,65	1,62	3,[illegible]
b							-2,6	1,72	1,63	1,65	1,62	
3322 a	75	140	630	40,010	64,490	10,6	-0,6	1,55	1,50	1,56	1,52	3,[illegible]
b							-2,8	1,70	1,57	1,51	1,50	
3323 a	75	160	720	40,450	68,400	12,1	-3,8	1,48	1,33	1,31	1,30	2,[illegible]
b							-3,6	1,48	1,41	1,44	1,41	
3332 a	95	110	630	40,260	62,840	9,8	-2,0	1,44	1,35	1,37	1,37	
b							-2,8	1,35	1,35	1,30	1,25	
3333 a	95	125	720	40,000	63,240	10,1	-2,4	1,48	1,39	1,39	1,37	
							-1,6	1,52	1,42	1,46	1,43	

C-2

CHARACTERISTICS OF ELECTROCHEMICALLY IMPREGNATED Ni-Cd TYPE POSITIVE PLATES

TABLE 12.2

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Impregnation Bath : / Concentration Ni+Co=3M/1 /

concentration Co = 3,3 %

"pH" 1,5-2 Alcohol content 50 %

Temperature : 80° C

Reference Sample		Impregnation condition			Characteristics of impregnated electrodes								
		Current density	Impreg-nation time (mn)	Current quantity (c/cm2)	Electrode weight before impregn. (g) S = 2,3 dm2)	Electrode weight after impregn. (g) S= 2.3 dm2)	ΔP (g/dm2)	Capacities in Ah (S = 0.50 dm2) 1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacities (4th cyc (Ah/dm2)	Yield 4th cycle Q/ΔP (Ah/g)
3211	a	50	180	540	40,040	63,000	10,0	/	1,37	1,37	1,35	2,8	0,28
	b							/	1,48	1,48	1,48		
3212	a	50	210	630	40,290	64,270	10,4	/	1,48	1,48	1,48	3,0	0,28
	b							/	1,52	1,52	1,54		
3213	a	50	240	720	39,910	62,000	9,6	/	1,43	1,43	1,43	2,8	0,291
	b							/	1,41	1,43	1,41		
3221	a	75	120	540	39,990	60,660	9,0	/	1,28	1,28	1,26	2,5	0,277
	b							/	1,28	1,20	1,22		
3222	a	75	140	630	40,570	63,160	9,8	/	1,30	1,30	1,30	2,7	0,27
	b							/	1,40	1,41	1,41		
3223	a	75	160	720	39,990	63,430	10,2	/	1,54	1,54	1,54	3,1	0,30
	b							/	1,52	1,54	1,57		
3231	a	95	95	540	38,580	60,480	9,5	/	1,41	1,41	1,57	2,9	0,
	b							/	1,52	1,54	1,35		
3232	a	95	110	630	38,490	63,880	11,0	1,79	1,79	1,68	1,83	3,5	0,
	b							1,68	1,68	1,81	1,68		
3233	a	95	125	720	39,010	68,430	12,8	1,81	1,83	1,88	1,90	3,8	0,
	b							1,77	1,81	1,81	1,85		

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ELECTRODE THICKNESS MEASUREMENTS

TABLE 13.1

Impregnation Bath : / concentration Ni + Co = 2 M/1 /
concentration Co = 5 %

"pH" = 1,5 - 2
Alcohol content: 50 %
Temperature : 80° C

Ref. Sampl.	Impregnation Conditions			Nonformed impregnated thickness (mm/100)				Mean thickness (mm/100)	Formed impregnated thickness (mm/100)				Mean thickness mm/100	Thickness variation before formation (%)	Thickness variation$_{a}$ during formation	Thick. var. of formed electr (%)
	Current Density	Impregnation time mn	Current quantity c/cm2													
3312 a	50	210	630	85	79	86	84	83	84	74	82	82	80,5	+6	-4	+[illegible]
b				88	83	85	95	88	83	81	83	85	83	+13	-7	+[illegible]
3313 a	50	240	720	105	88	88	107	97	80	80	80	80	80	+26	-25	+1
b				112	82	86	108	97	79	79	83	79	80	+26	-25	+1
3322 a	75	140	630	82	79	83	80	81	83	78	84	82	82	+3	+1	+[illegible]
b				85	94	84	80	86	78	78	80	80	79	+10	-10	[illegible]
3323 a	75	160	720	97	99	99	102	99	78	78	78	82	79	+30	-30	0
b				96	106	101	109	103	85	81	80	81	82	+35	-31	+[illegible]
3332 a	95	110	630	90	97	93	92	93	79	80	82	79	80	+20	-19	+1
b				90	97	95	106	97	78	82	81	80	80	+26	-25	+1
3333 a	95	125	720	100	96	91	110	99	79	79	80	81	80	+29	-28	[illegible]
b				104	92	86	106	97	79	78	80	81	79,5	+27	-26	[illegible]

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ELECTRODE THICKNESS MEASUREMENTS

TABLE 13.2.:

IMPREGNATION BATH : / concentration Ni + Co = 3M/1 /
concentration Co = 3,3 %

"pH" = 1,5 - 2
Alcohol content 50 %
Temperature : 80° C

Ref Samp	Impregnation Conditions			Nonformed impregnated thickness (mm/100)				Mean thickness (mm/100)	Formed impregnated thickness (mm/100)				Mean thickness mm/100	Thickness variation before formation (%)	Thickness variation during formation (%)	Thick. varia. of formed el.% (%)
	Current density mA/cm2	Impreg. Time mn	Current Quantity c/cm2													
211 a	50	180	540	86	85	86	82	85	84	82	87	86	85	+9	0	[illegible]
b				85	86	87	84	85,5	84	87	89	84	86	+10	0	[illegible]
212 a	50	210	630	85	84	90	81	85	84	90	90	85	87	+9	+3	[illegible]
b				83	87	87	86	86	88	89	90	85	88	+10	+3	[illegible]
213 a	50	240	720	81	86	86	82	84	82	86	86	83	84	+7	0	+7
b				81	88	87	81	84	86	89	88	83	86	+7	+3	[illegible]
221 a	75	120	540	86	81	81	86	83,5	86	85	89	82	85,5	+7	+3	[illegible]
b				87	83	81	83	83,5	84	84	84	86	84,5	+7	+1	[illegible]
222 a	75	140	630	86	86	85	84	85	88	84	81	85	84,5	+9	-1	+8
b				86	82	84	81	83	88	91	87	86	88	+6	+7	+1
223 a	75	160	720	82	84	82	82	82,5	82	85	82	81	82,5	+5	0	[illegible]
b				84	80	82	84	82,5	85	85	86	83	84,5	+5	+3	[illegible]
231 a	95	95	540	82	81	83	80	81,5	83	77	77	83	80	+3	-2	[illegible]
b				82	78	85	82	82	80	78	80	79	79	+4	-4	
232 a	95	110	630	84	83	86	86	85,5	80	80	82	80	80,5	+9	-7	[illegible]
b				87	83	86	86	85,5	82	82	85	80	82	+9	-5	[illegible]
233 a	95	125	720	88	86	88	86	87	80	83	82	80	81	+12	-9	[illegible]
b				87	87	87	87	87	78	77	82	76	78	+13	-1[illegible]	[illegible]

INFLUENCE OF THE INTERELECTRODE DISTANCE

- Ni-Cd type electrode
- Impregnation conditions :Alcohol content: 50 %

  Ni + Co = 2 M/l
  Co = 5 %
  pH = 1,5 - 2
  Temperature : 80° C

Electrode/counter-electrode distance (mm)		20 (standard)	10
Hydrate weight retained in plates (after formation) (in g/dm2)		9,2	11,3
Capacities (Ah/dm2) Formation	1st cycle	3,4	3,6
	2nd cycle	3,3	3,55
	3rd cycle	3,3	3,65
	4th cycle	3,25	3,6
Thickness :	Before impregnation (mm/100)	79	79
	After impregnation (mm/100)	97	86
	After formation (mm/100)	80	87,5
	Swelling before formation (%)	+26	+10
	Swelling during formation la forma- (%)	-25	+2
	Overall swelling	+1	+12

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TABLE 15

CONTROL OF CORROSION OF SINTERED SUPPORT IN THE IMPREGNATION BATH PRIOR TO ELECTROLYSIS

Conditions : pH : 1,5 - 2
Temperature : 80° C

Impregn. Baths		Impregnation Time (mn)	Reference Sample	Weight Prior to Immersion (g)	Weight after Immersion (g)	Weight variation during immersion (mg)	Wt. corroded nickel (g/dm2)	Nickel corrosion (%)
Alcohol (5) %)	Concentration Ni + Co (M/1)							
20	2	2	6a	2,6967	2,6796	17	0,06	< 1
		5	6b	2,6546	2,6440	11	0,04	< 1
		10	7a	2,7426	2,7050	38	0,31	3
		20	7b	2,6530	2,6010	52	0,42	4
30	2	2	4a	2,6030	2,5888	14	0,11	1
		5	4b	2,7115	2,6976	14	0,12	1
		10	5a	2,6378	2,6037	34	0,30	3
		20	5b	2,6994	2,6483	51	0,42	4
50	2	2	8a	2,7580	2,7417	16	0,19	2
		5	8b	2,7080	2,6924	16	0,20	2
		10	9a	2,7155	2,6605	35	0,37	4
		20	9b	2,6815	2,6444	37	0,25	3
50	3	2	10a	2,7085	2,6869	22	0,18	2
		5	10b	2,6143	2,5982	16	0,09	1
		10	11a	2,7650	2,7350	30	0,24	2,5
		20	11b	2,7460	2,6645	81	0,64	6

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TABLE 16

ELECTROCHEMICAL IMPREGNATION IN AN AQUEOUS MEDIUM

CHARACTERISTICS OF IMREGNATED PLATES

e = 0,84 mm

Impregnation Bath : concentration Ni + Co = 2 M/1
concentration Co = 5 %

pH : 3-3,5

Alcohol content: 0 %
Temperature : 98° C

Refer. Sample	Impregnation Conditions		Characteristics of Impregnated Plates								
	Current Density mA/cm2	Impregna-tion time mn	Electrode wt. before impregna-tion in g	Electrode weight after im-pregnation in g	ΔP (g/dm2)	Capacities in Ah (S = 0.45 dm2) 1st cycle	2nd cycle	3rd cycle	4th cycle	Mean capacity 4th cycle Ah/dm2	Yield 4th cycle Q/ΔP Ah/g)
B	50	2h 30	10,770	17,800	12,12	1,60	1,60	1,60	1,61	3,40	0,295
E	50	2h 30	10,800	17,320	11,24	1,52	1,43	1,41	1,43		0,28
D	50	3h 30	10,880	18,520	13,17	1,71	1,69	1,67	1,67	3,70	0,28
F	50	3h 30	10,970	18,220	12,50	1,67	1,67	1,66	1,65		0,2
A	50	4h	10,780	18,830	13,88	1,84	1,84	1,85	1,89	4,10	[illegible]
C	50	4h	11,150	19,240	13,95	1,82	1,82	1,81	1,81		0,

ELECTROCHEMICAL IMPREGNATION IN AN AQUEOUS

MEDIUM - THICKNESS MEASUREMENT OF ELECTRODES

TABLE 17

Impregnation Bath : concentration Ni + Co = 2 M/l
concentration Co = 5 %

pH = 3 - 3,5

Alcohol content: 0 %
Température : 98° C

Ref Sampl	Impregnation Condition	Thickness Prior to formation (mm/100)	Mean Thickness (mm/100)	Thickness after format. (mm/100)	Mean thickness (mm/100)	Thickness after cycling (70 cycles) (mm/100)	Mean thickness (mm/100)	Swelling during formation	Swelling after cycling %
	50 mA/cm2 150 mn	86 82 82 87	84	93 90 91 92	91,5	97 98 98 99	98	10	19
	50 mA/cm2 150 mn	85 82 84 86	84	89 83 86 87	86	94 86 91 91	90,5	3	9
	50 mA/cm2 210 mn	85 85 86 88	86	92 93 96 96	94	101 103 108 104	104	11	25
	50 mA/cm2 210 mn	88 80 84 86	84,5	89 89 88 91	89	94 96 96 96	95,5	6	9
	50 mA/cm2 240 mn	89 83 85 88	86	108 103 103 104	104,5	124 123 124 123	123,5	25	52
	50 mA/cm2 240 mn	85 84 84 87	85	100 103 102 104	102	114 119 115 116	116	24	43

FIGURES

FIGURE 1

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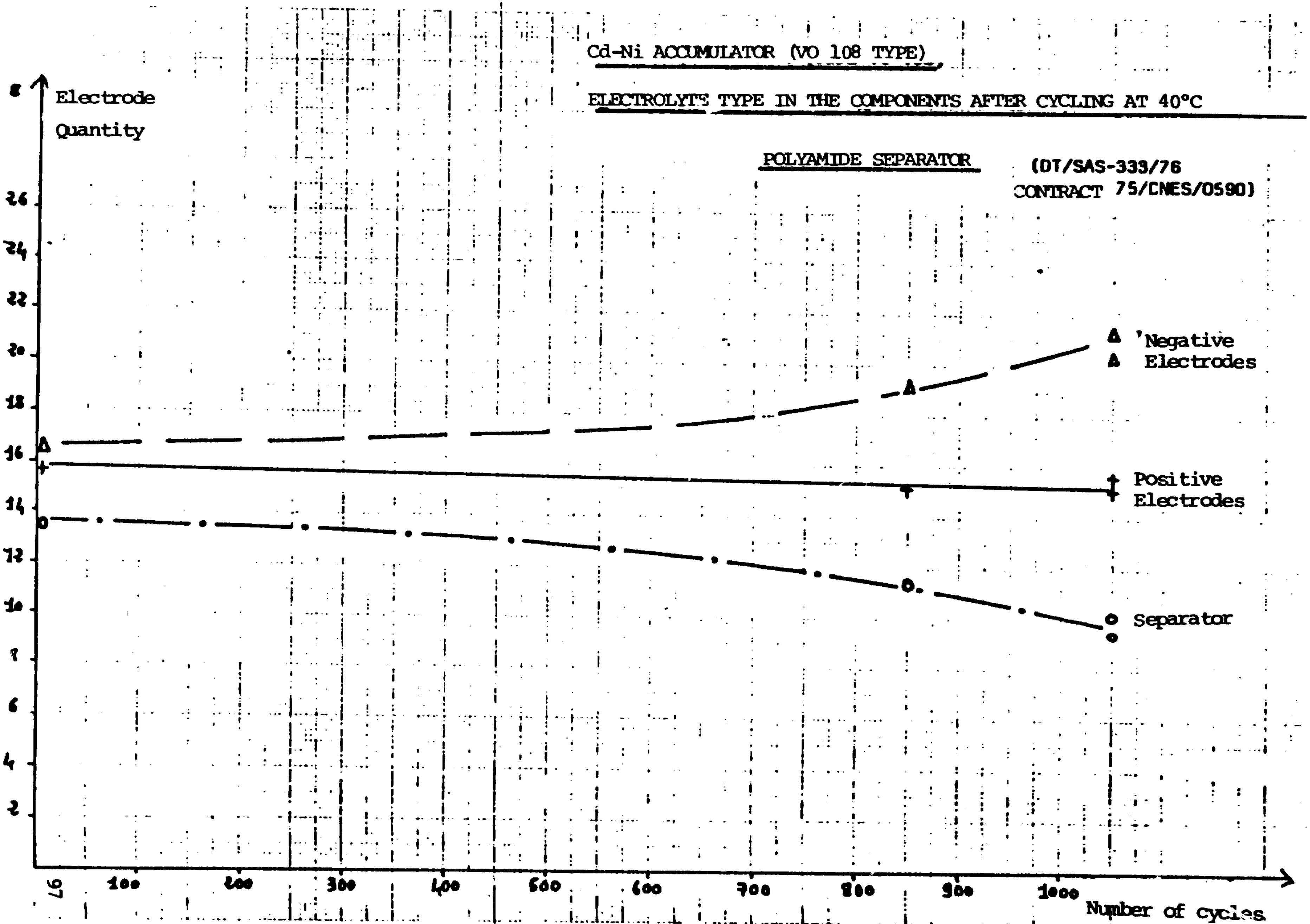


Cd-Ni ACCUMULATOR (VO 108 TYPE)

ELECTROLYTE TYPE IN THE COMPONENTS AFTER CYCLING AT 40°C

POLYAMIDE SEPARATOR

(DT/SAS-333/76
CONTRACT 75/CNES/0590)

FIGURE 2

ELECTROLYTE DISTRIBUTION IN THE COMPONENTS OF
Ni-H2 BATTERIES AS A FUNCTION OF THEIR
LOCATION IN THE CLUSTER

DT/SAS-333/78
Contract 75/CNES/0590)

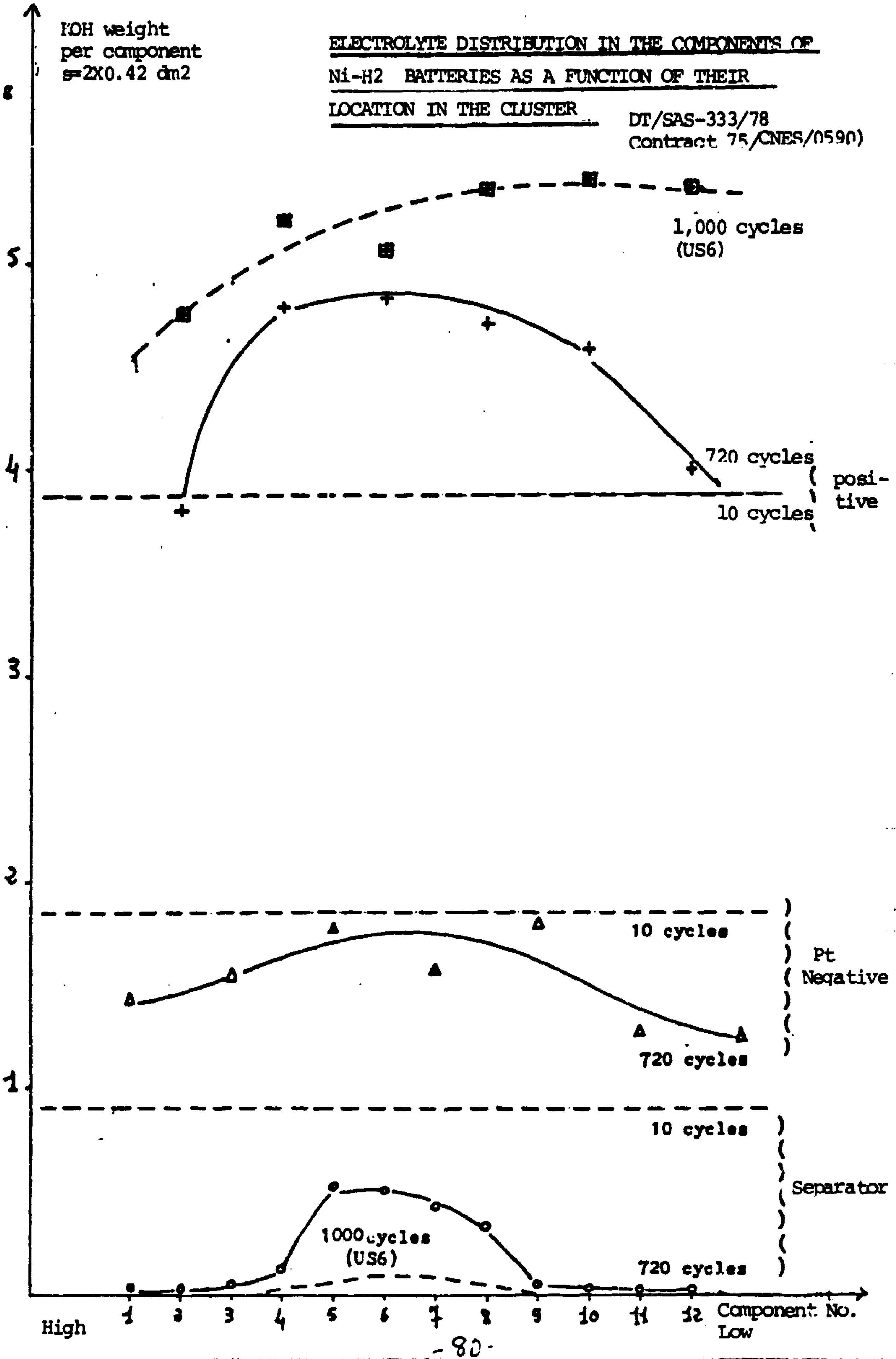
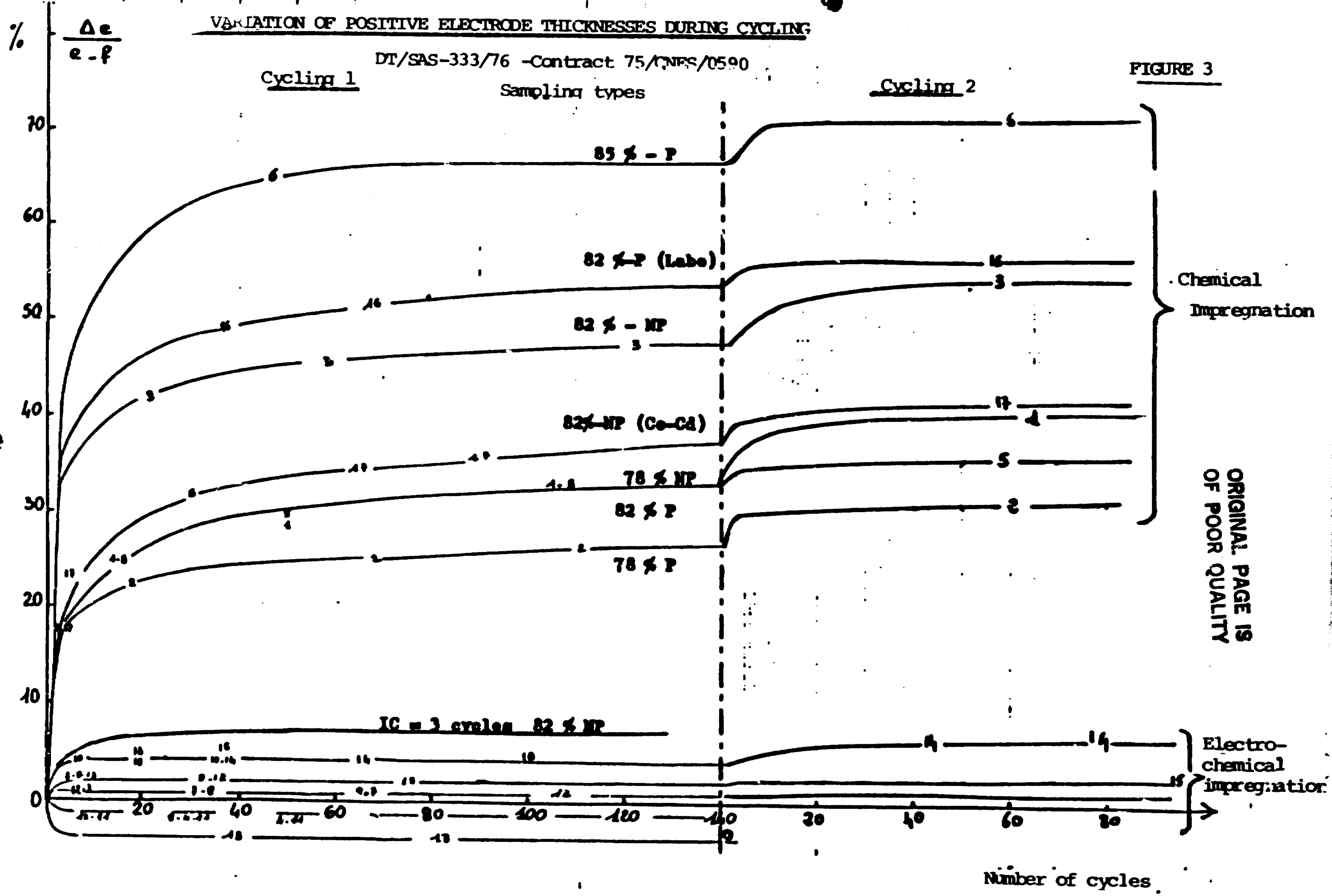


FIGURE 3



VARIATION OF POSITIVE ELECTRODE THICKNESSES DURING CYCLING

DT/SAS-333/76 -Contract 75/CNES/0590

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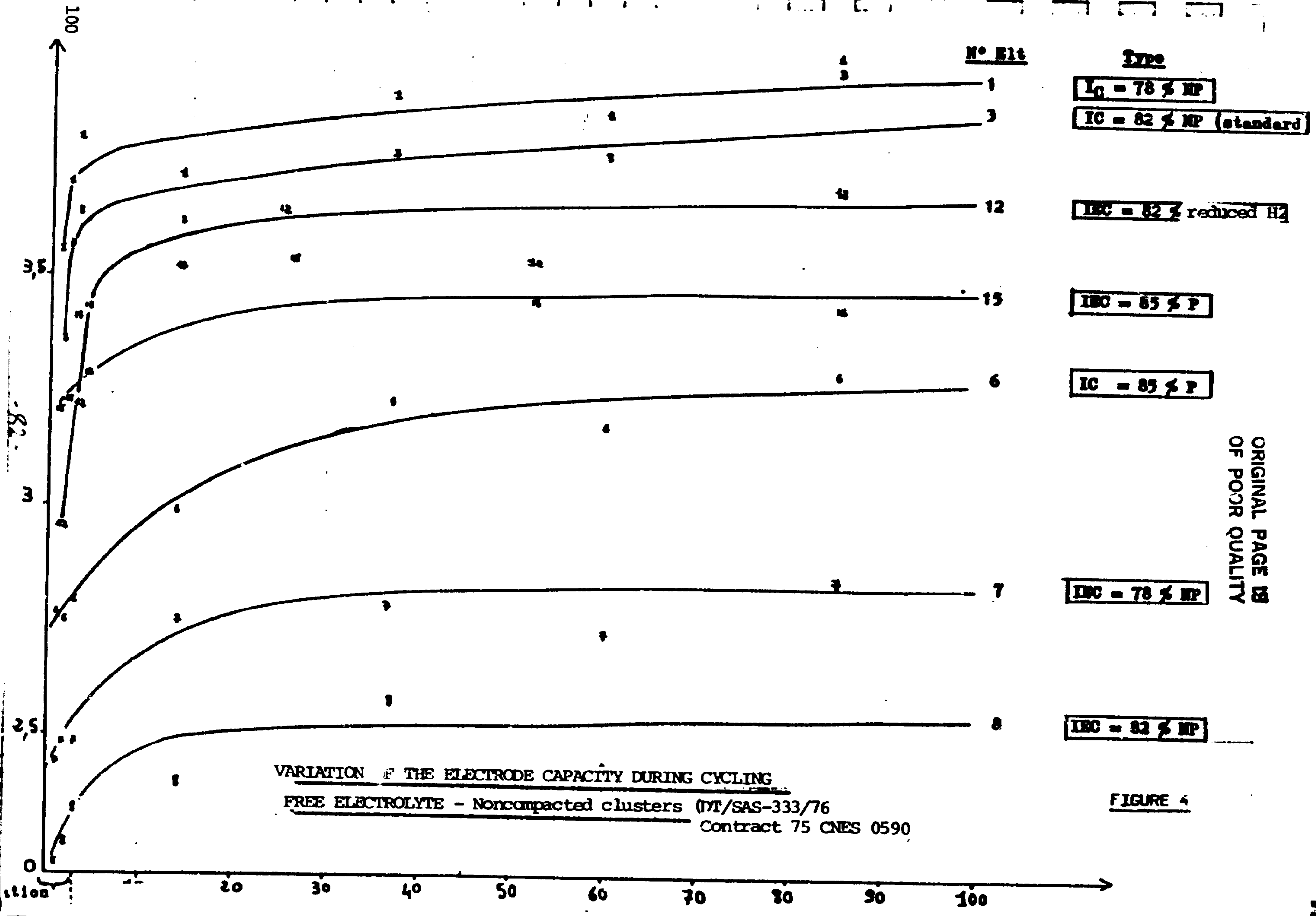


VARIATION OF THE ELECTRODE CAPACITY DURING CYCLING
FREE ELECTROLYTE - Noncompacted clusters (DT/SAS-333/76
Contract 75 CNES 0590

FIGURE 4

FIGURES 5

ELECTRODE FILLING AS A FUNCTION OF THE IMPREGNATION TIME AND OF THE CURRENT DENSITIES

Figure 5.1

IMPREGNATION
Conditions

ALCOHOL 0 %
temperature 98°C
pH 1.5 . 2
[Ni + Co] 2 M/l

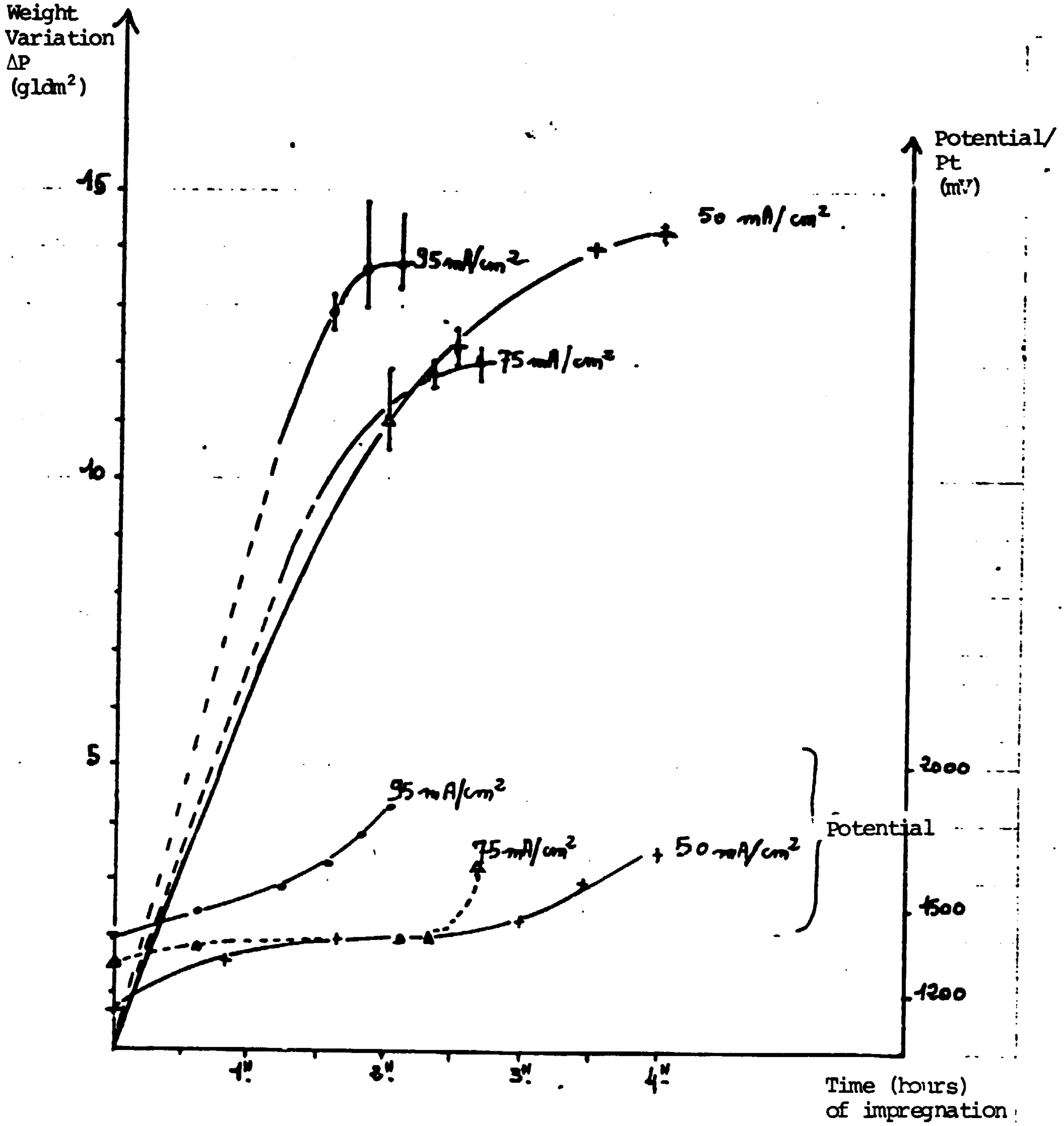


Figure 5.2

ELECTRODE FILLING PROCESS

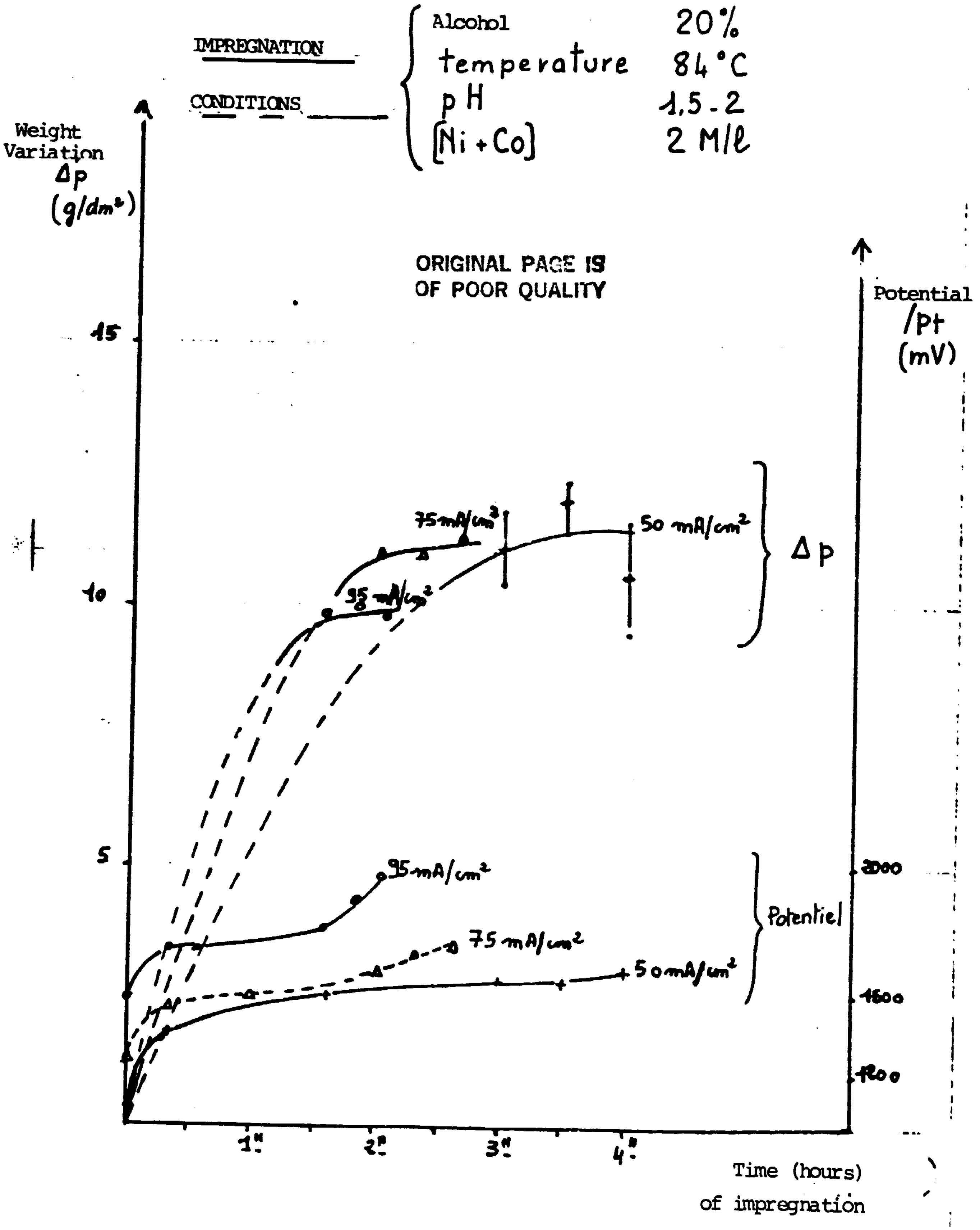


Figure 5.3

ELECTRODE FILLING PROCESS

IMPREGNATION
Conditions

Alcohol	50%
temperature	80°C
pH	1.5-2
[Ni + Co]	2 M/l

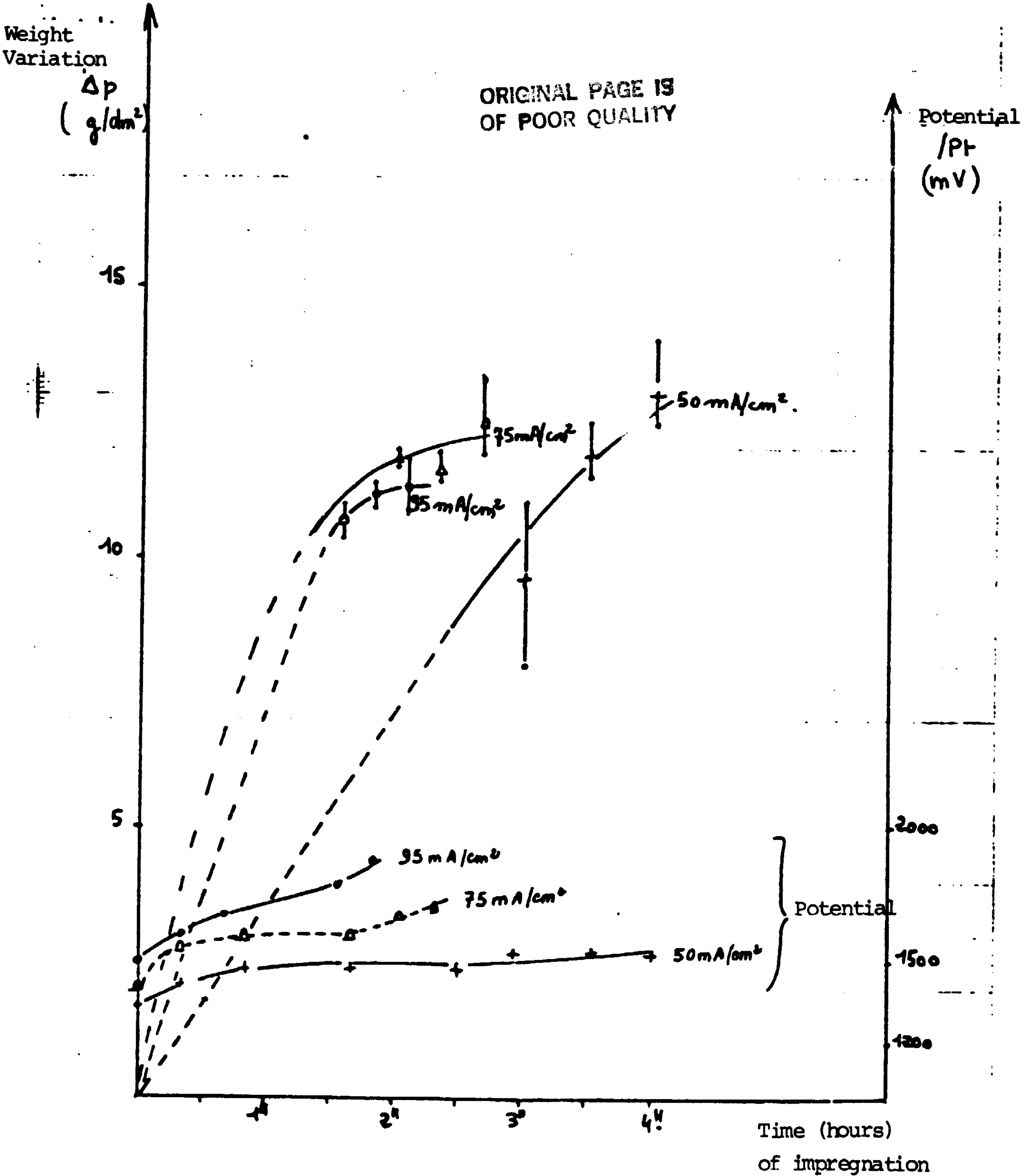


FIGURE 5.4 ELECTRODE FILLING PROCESS

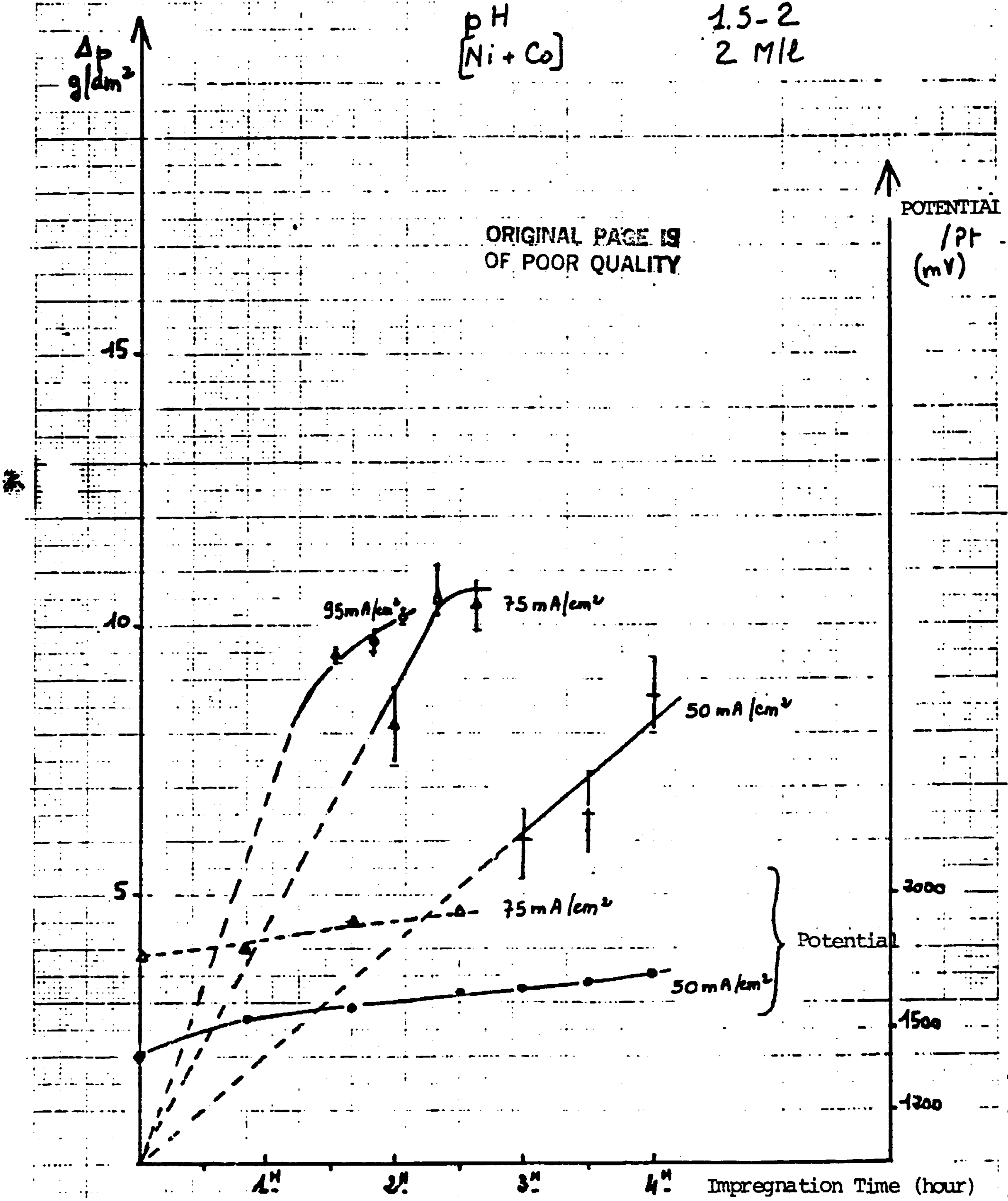
IMPREGNATION CONDITIONS:

ALCOHOL: 50%

temperature 50°C

pH 1.5-2

[Ni + Co] 2 M/l



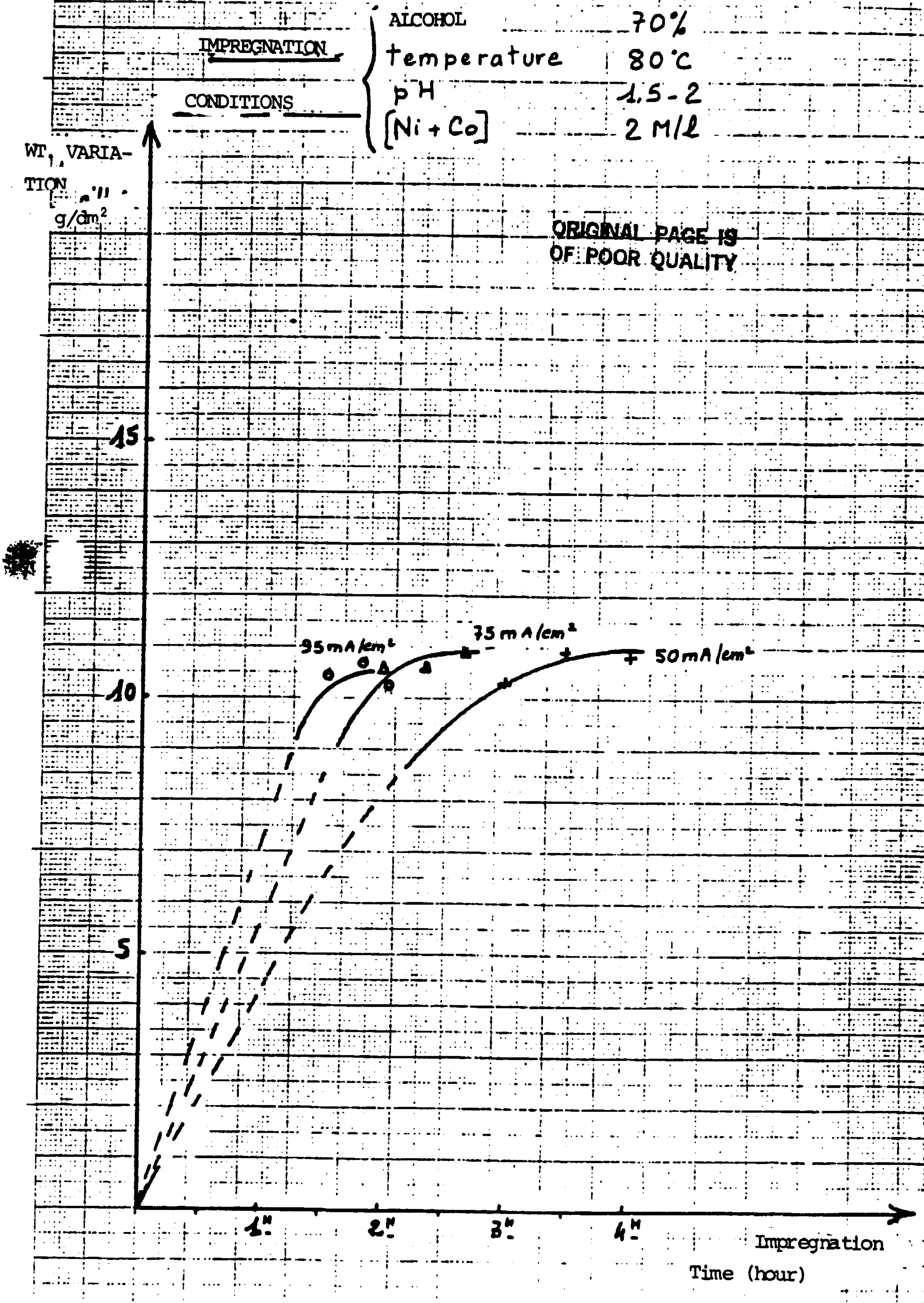

IMPREGNATION
CONDITIONS
ALCOHOL 70%
temperature 80°C
pH 1,5-2
[Ni + Co] 2 M/l
WT. VARIATION g/dm²
15
10
5
95 mA/cm²
75 mA/cm²
50 mA/cm²
1"
2"
3"
4"
Impregnation Time (hour)

FIGURE 5-6

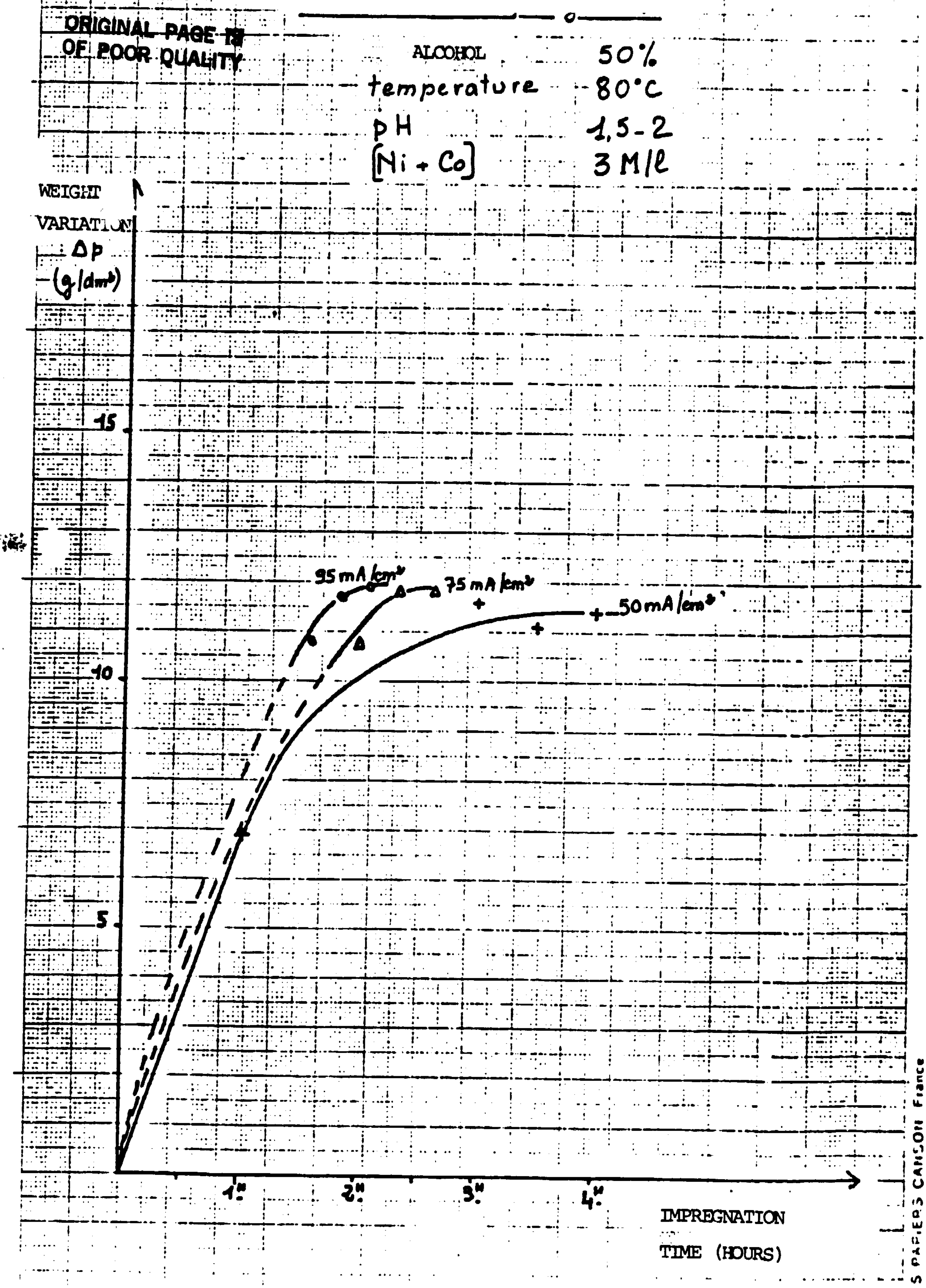
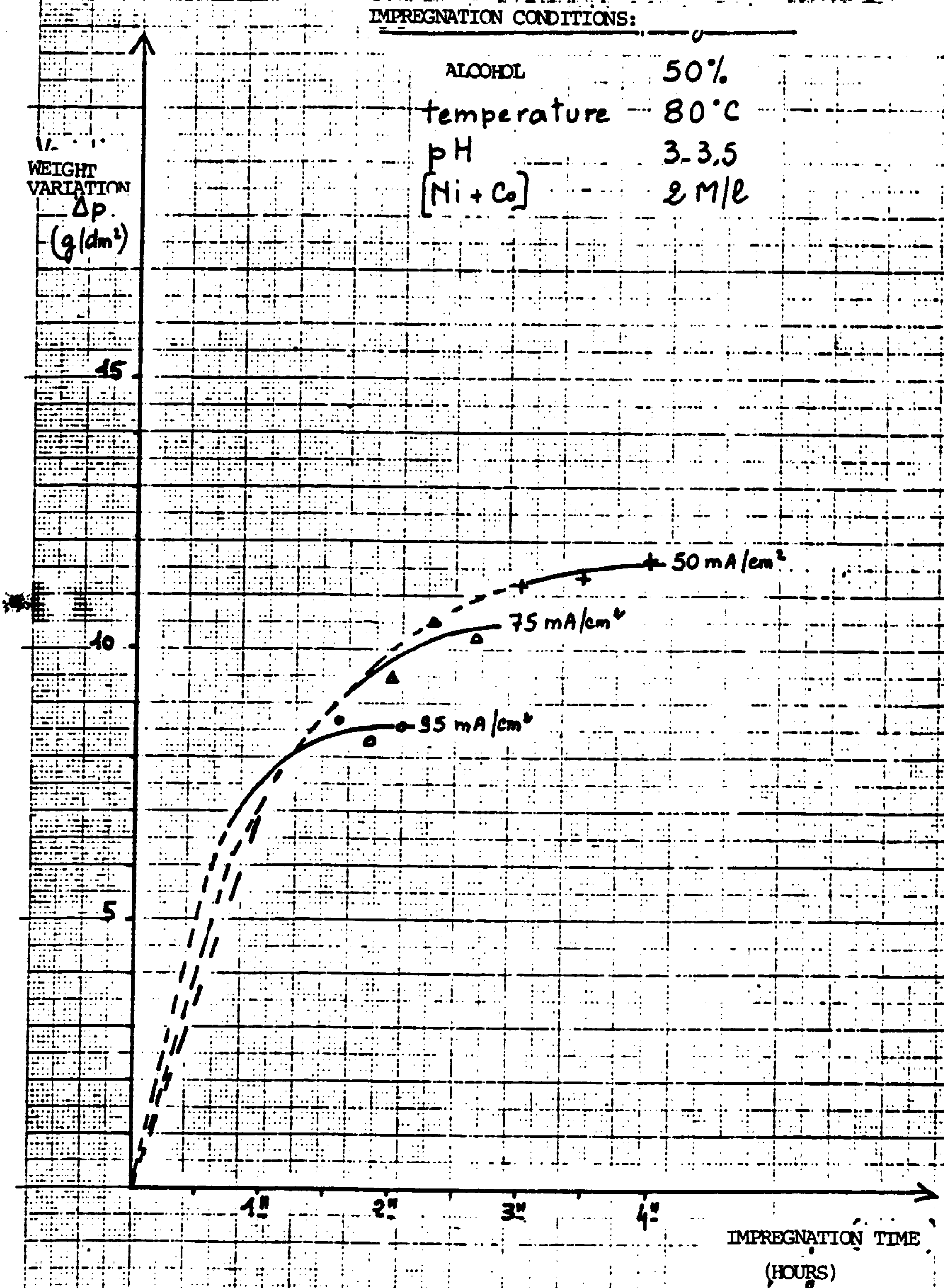

ALCOHOL 50%
temperature 80°C
pH 1,5-2
[Ni + Co] 3 M/l
WEIGHT VARIATION
Δp
(g/dm²)
15
10
5
95 mA/cm²
75 mA/cm²
50 mA/cm²
1h
2h
3h
4h
IMPREGNATION
TIME (HOURS)

Figure 5-7
FIGURE 5-7

ELECTRODE FILLING PROCESS



IMPREGNATION CONDITIONS:

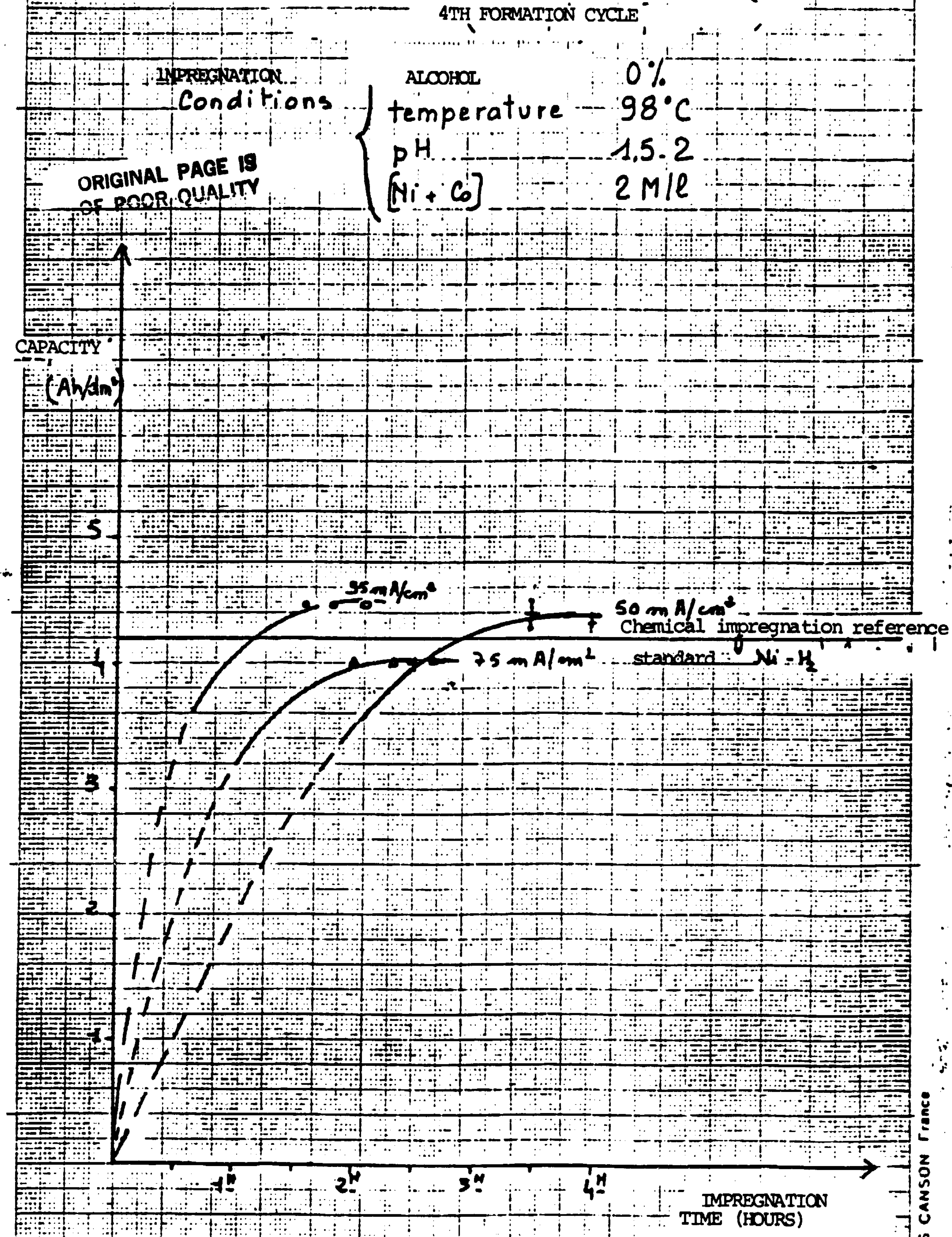
ALCOHOL 50%
temperature 80°C
pH 3-3,5
[Ni + Co] 2 M/l

FIGURES 6

INFLUENCE OF THE IMPREGNATION TIME AND OF THE CURRENT DENSITIES ON THE ELECTRODE CAPACITIES DURING THE 4TH FORMATION CYCLE

Figure 6-1

ELECTRODE CAPACITY



ELECTRODE CAPACITY

FIGURE 6.2

(1, 4th formation cycle

IMPREGNATION CONDITIONS

ALCOHOL	20%
temperature	84 °C
pH	1,5-2
[Ni + Co]	2 M/l

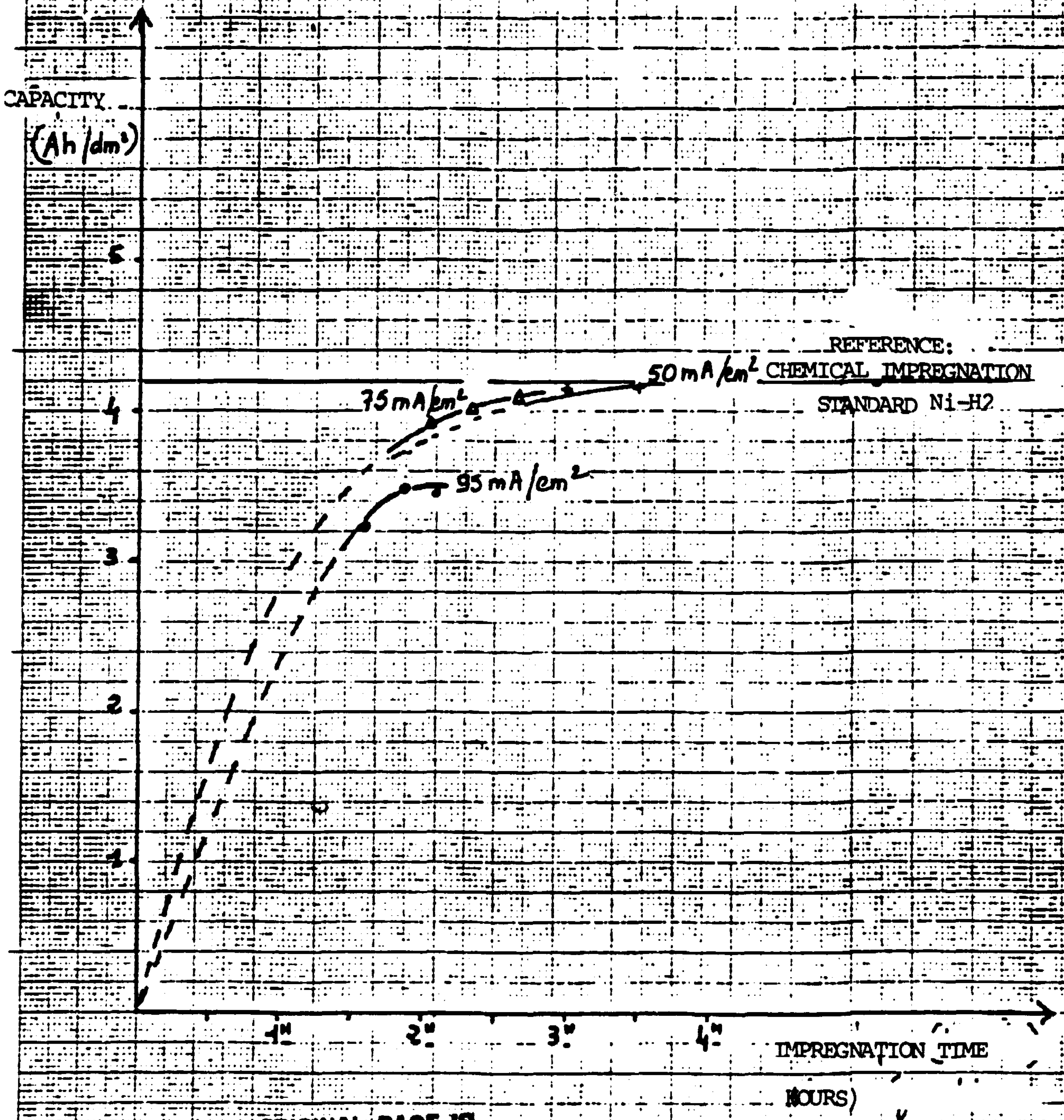


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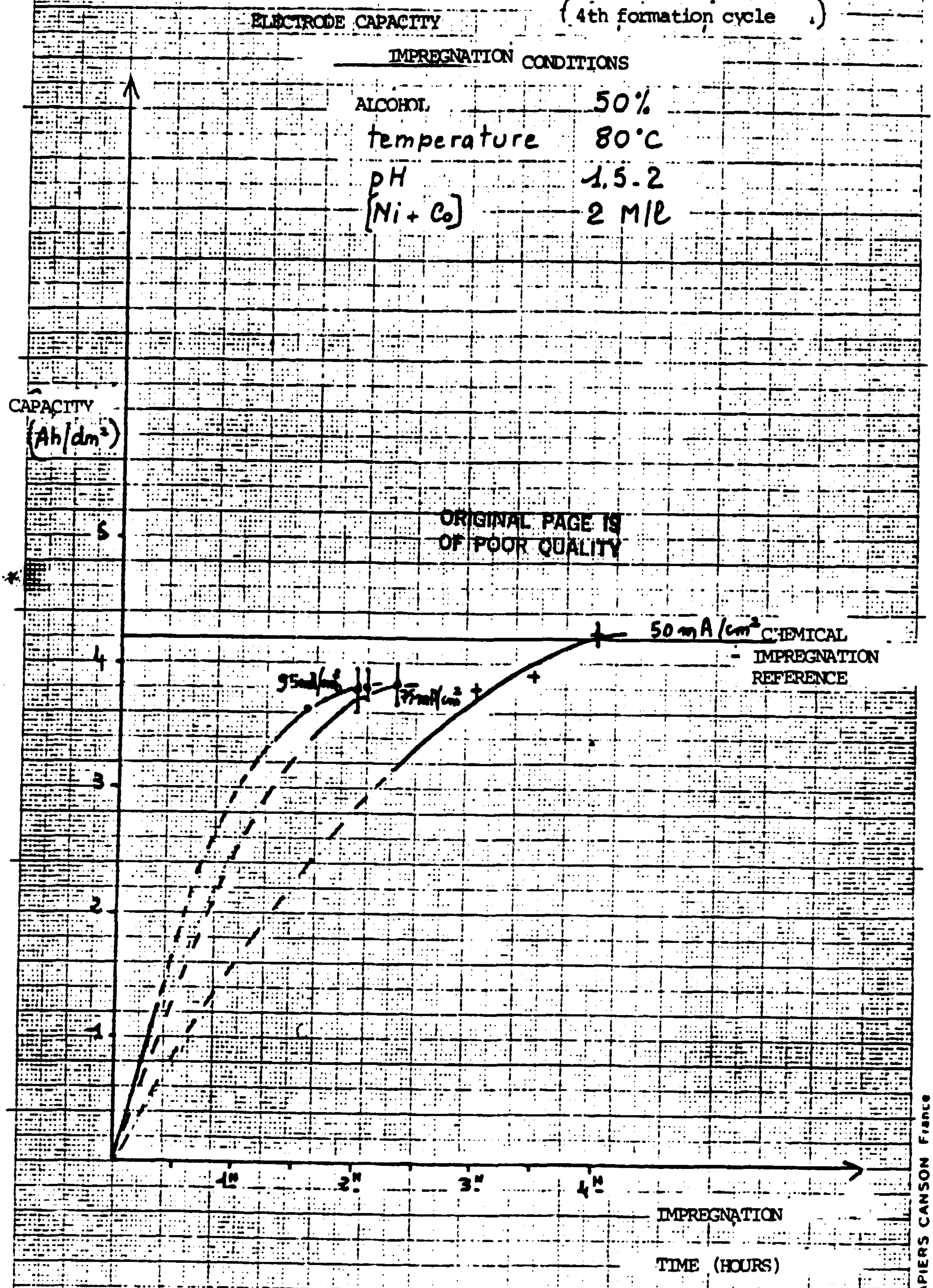
Figure 6.3


ELECTRODE CAPACITY
(4th formation cycle .)
IMPREGNATION CONDITIONS
ALCOHOL 50%
temperature 80°C
pH 1.5-2
[Ni + Co] 2 M/l
CAPACITY (Ah/dm²)
5
4
3
2
1
95 mA/cm²
75 mA/cm²
50 mA/cm²
CHEMICAL IMPREGNATION REFERENCE
1H
2H
3H
4H
IMPREGNATION TIME (HOURS)

ELECTRODE CAPACITY 4TH FORMATION CYCLE FIGURE 6-4

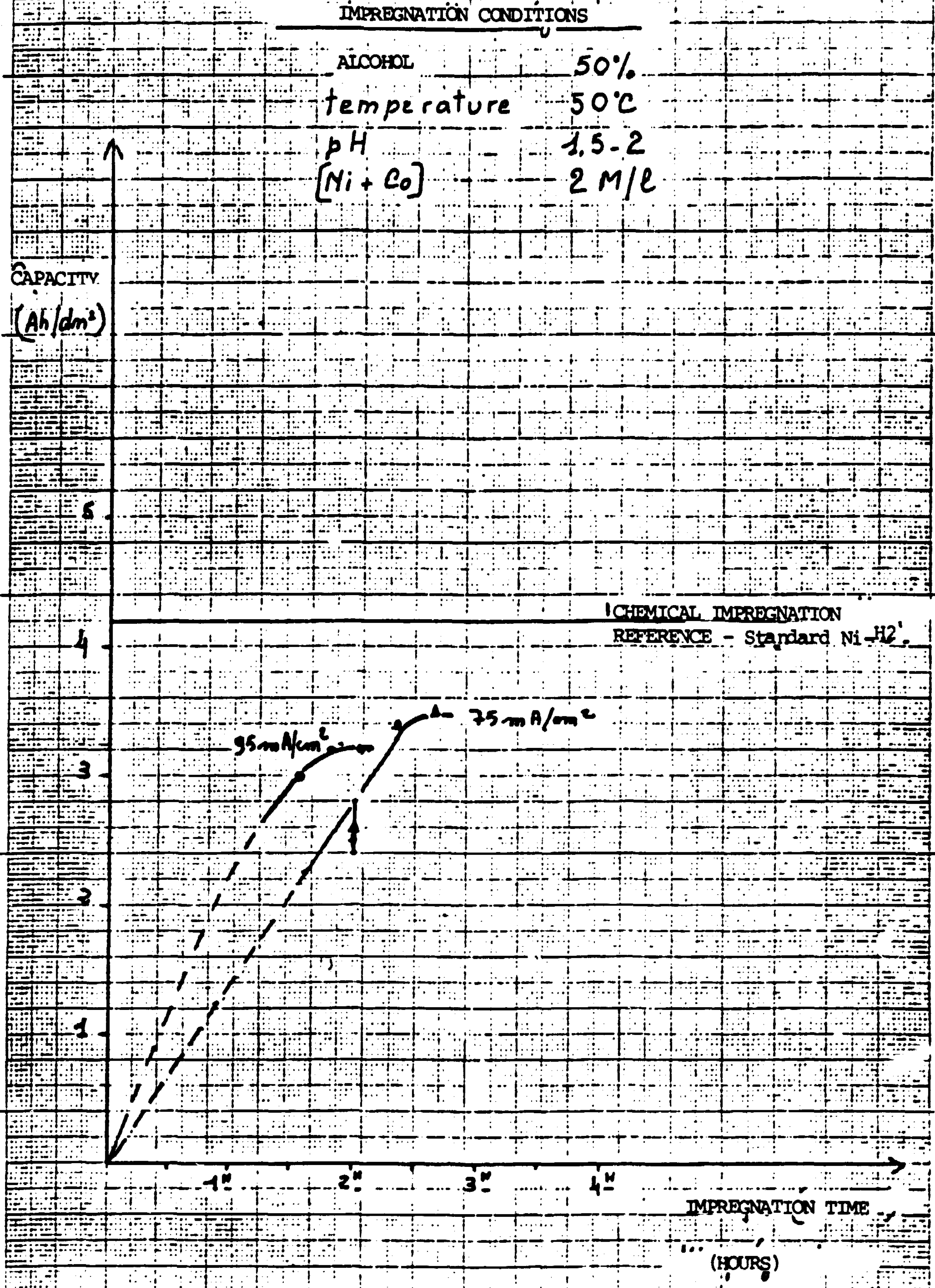


FIGURE 6-5

ELECTRODE CAPACITY
(4th FORMATION CYCLE)

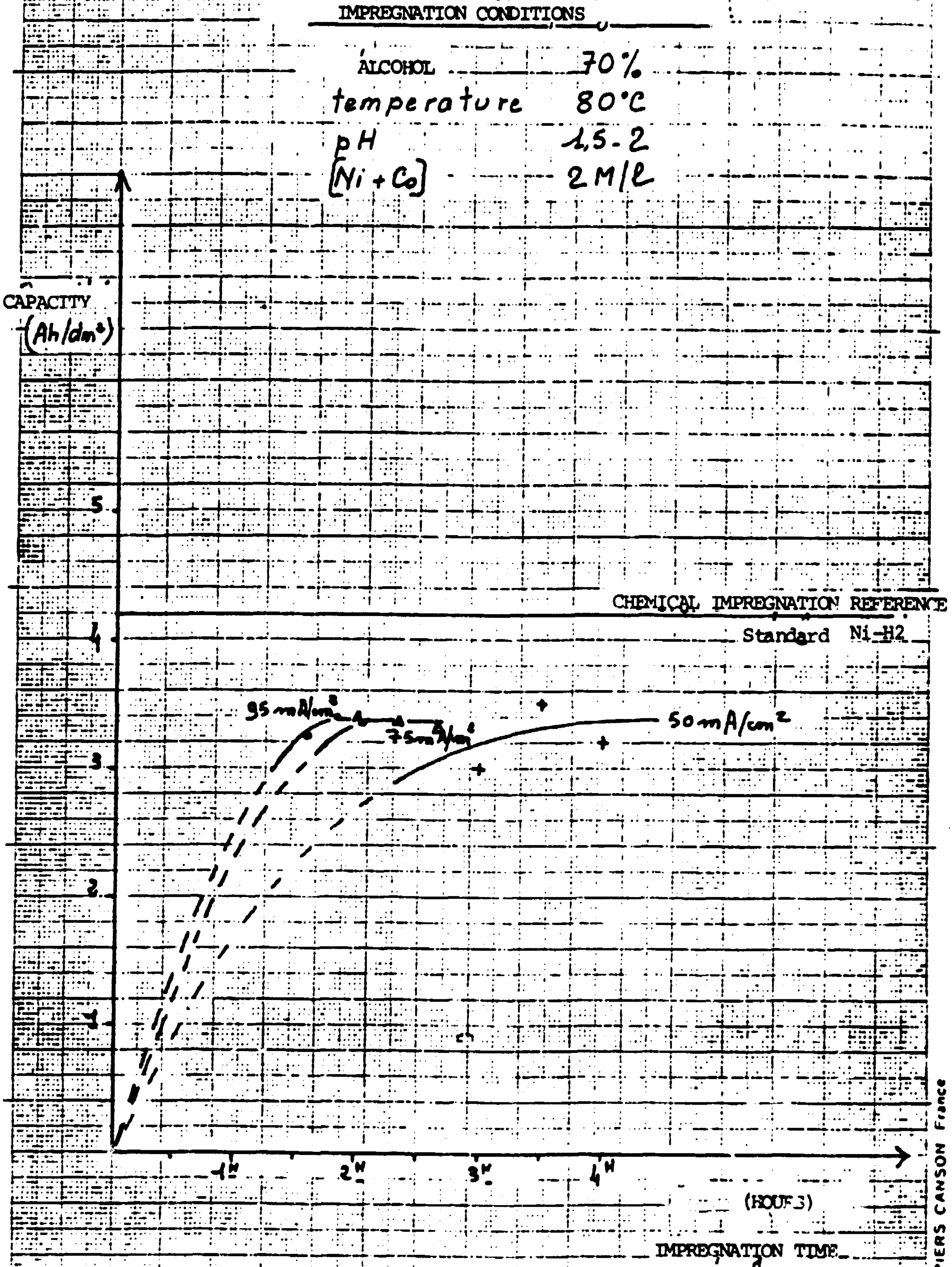


Figure 6.6

ELECTRODE CAPACITY (4th formation cycle) FIGURE 6-6

IMPREGNATION CONDITIONS

ALCOHOL	50%
temperature	80°C
pH	1,5-2
[Ni + Co]	3 M/l

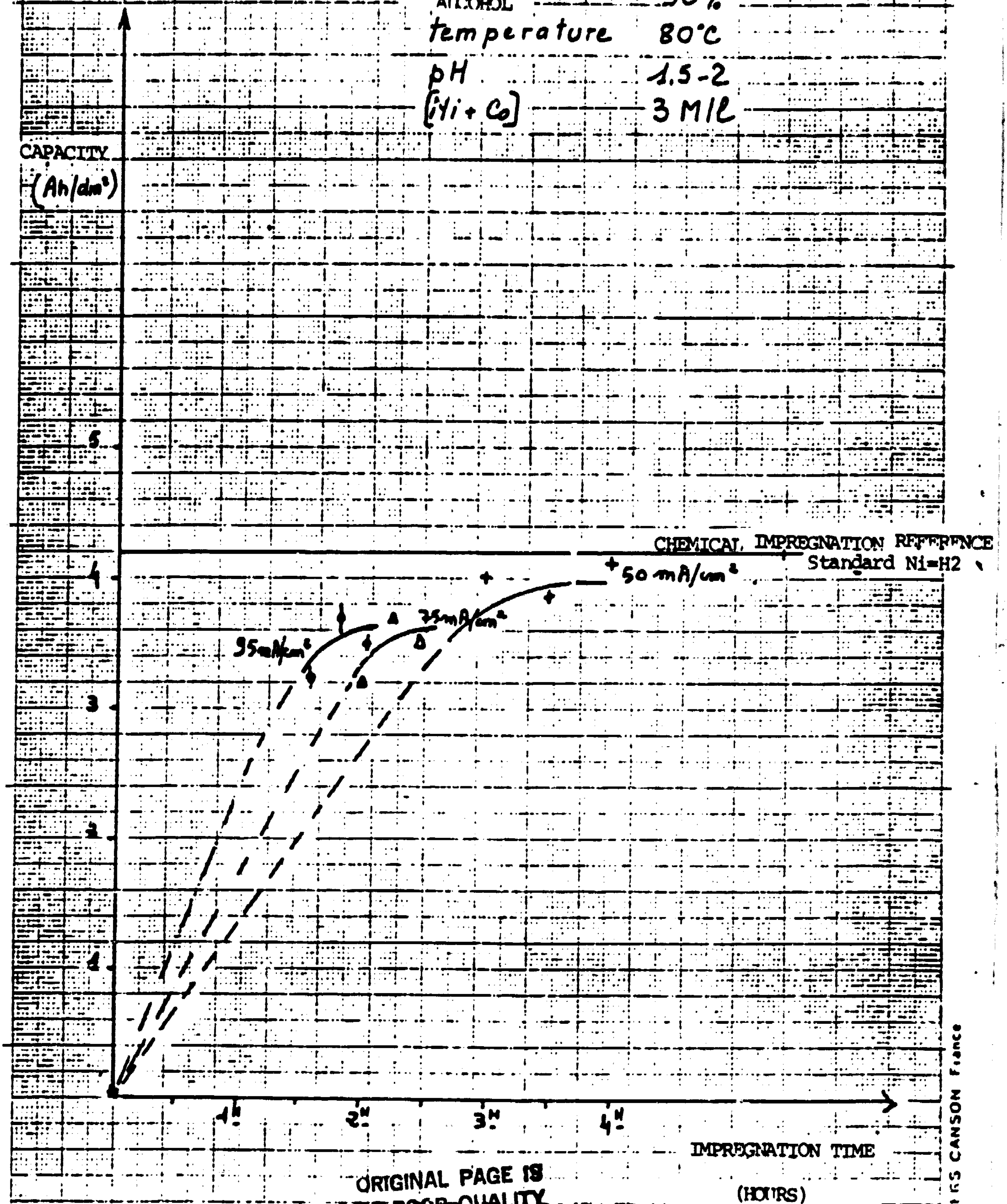


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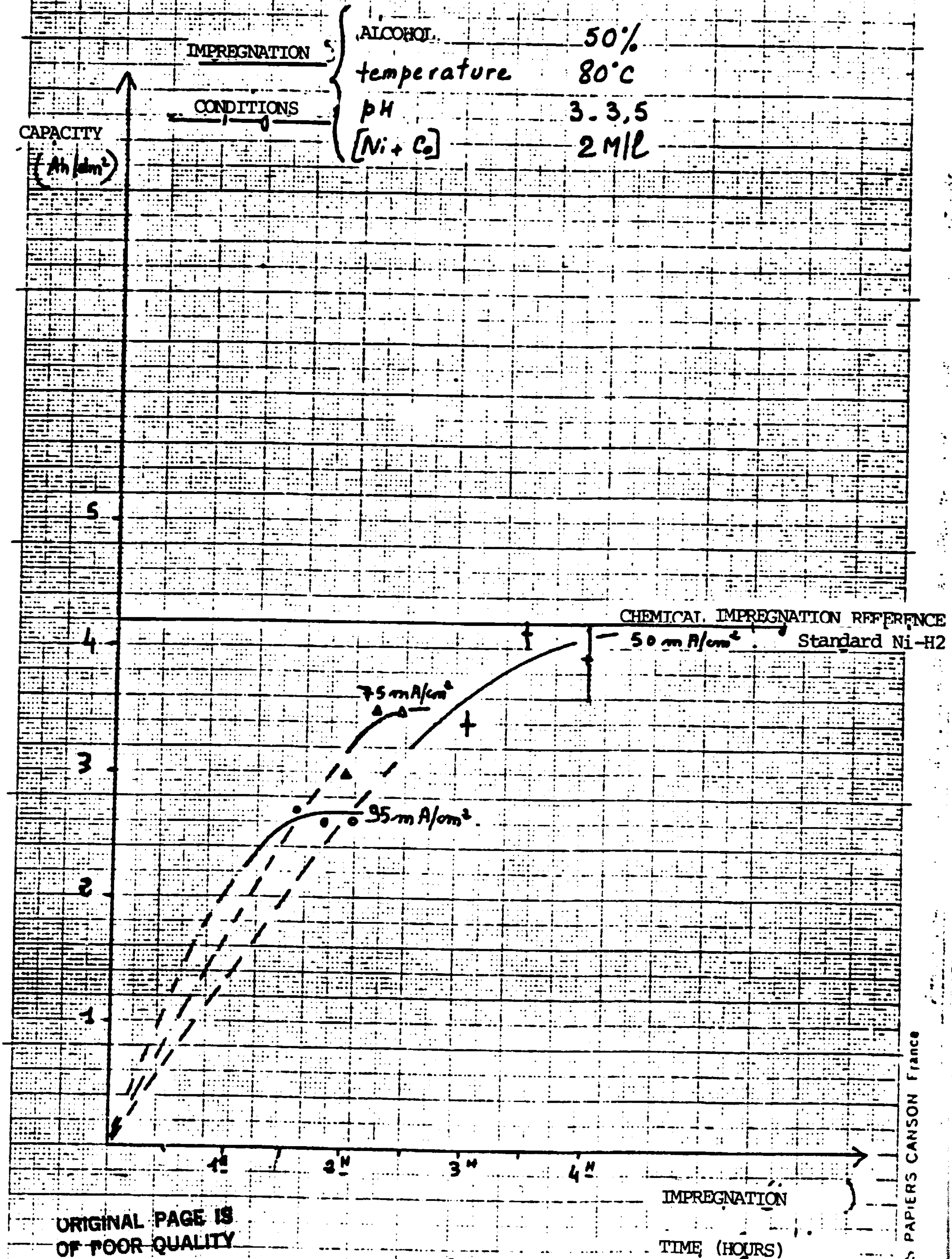
Figure 6-

FIGURE 6-7

ELECTRODE CAPACITY (4th FORMATION CYCLE)



IMPREGNATION CONDITIONS
ALCOHOL 50%
temperature 80°C
pH 3-3,5
[Ni + Co] 2 M/l

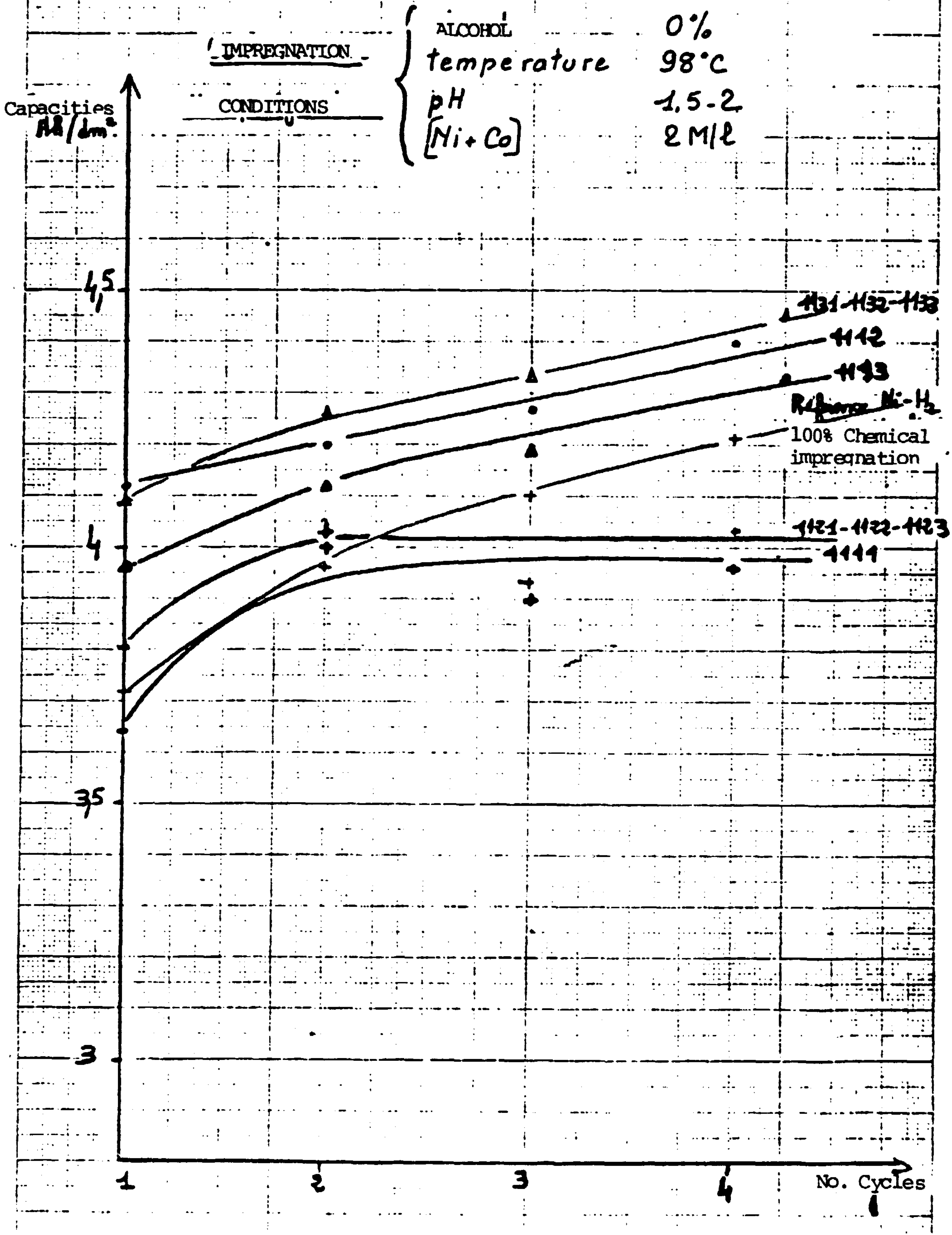
FIGURES 7

CAPACITY VARIATION DURING ELECTRODE FORMATION

CAPACITY VARIATION DURING

ELECTRODE FORMATION

Figure 7.1



· ICC

Figure 7-2

CAPACITY VARIATION DURING FORMATION

IMPREGNATION CONDITIONS

Alcohol	20%
temperature	84°C
pH	1,5-2
[Ni + Co]	2 M/l

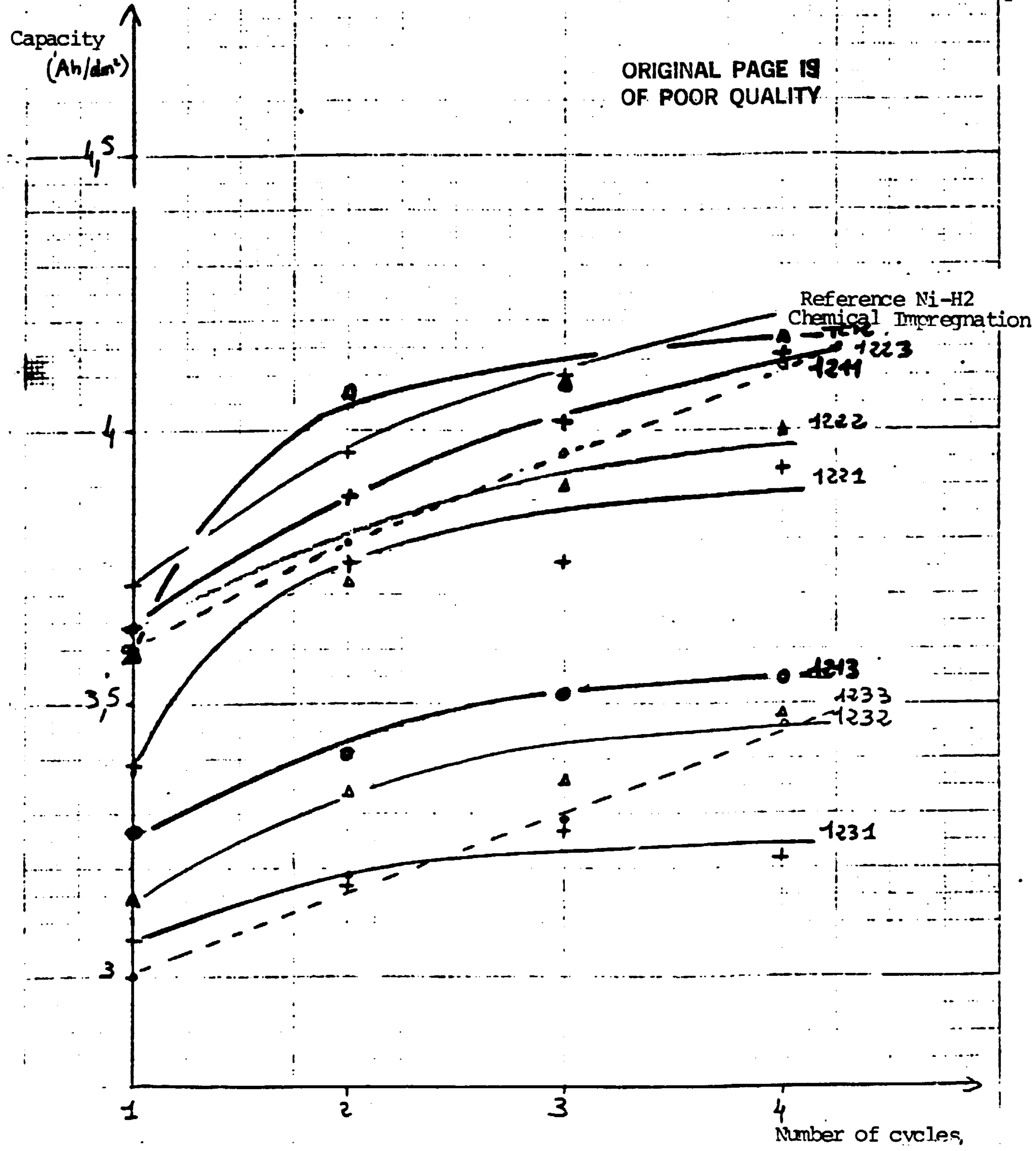


Figure 7.3
FIGURE 7-3

CAPACITY VARIATION DURING FORMATION

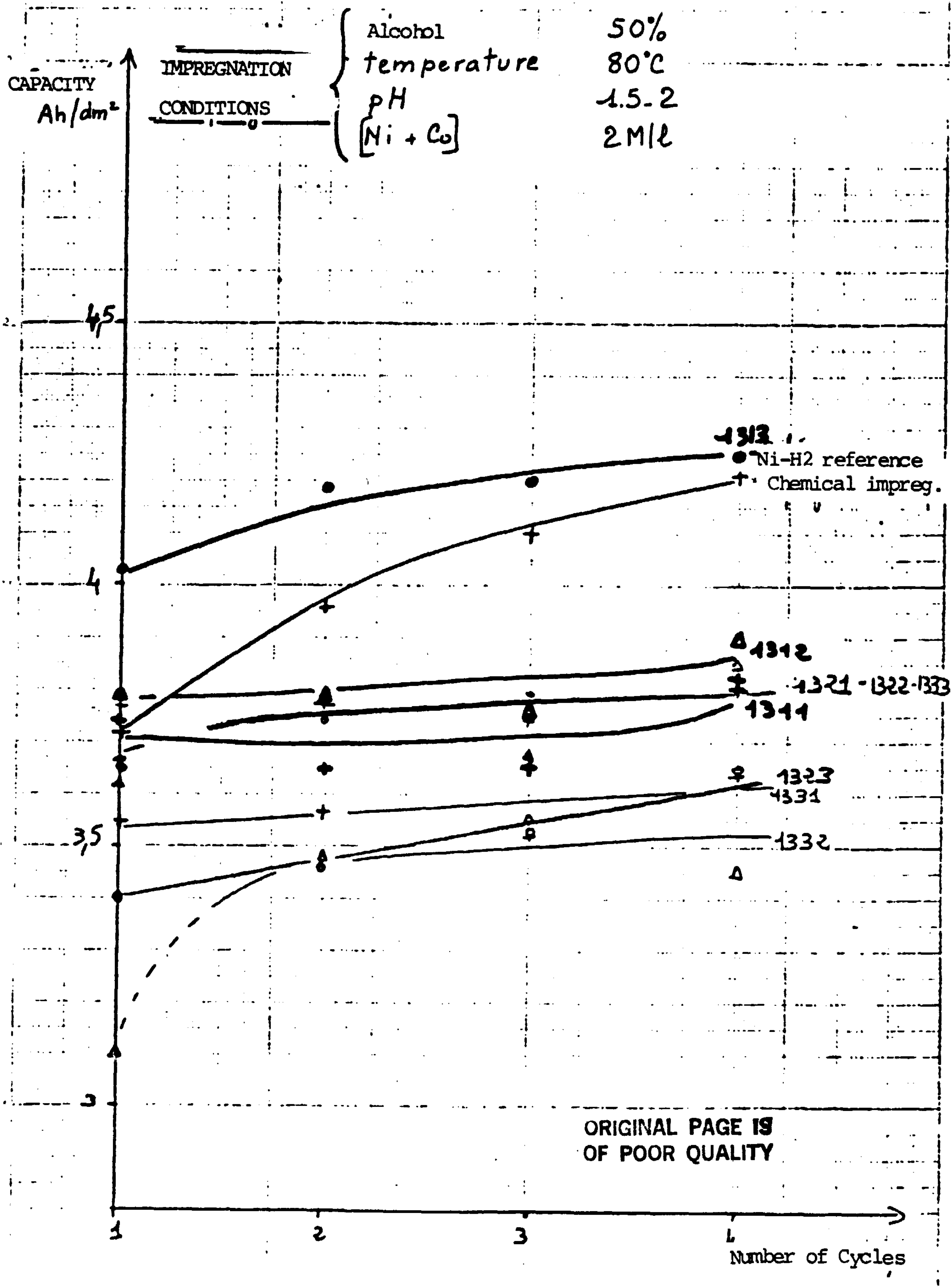


figure 7-4

CAPACITY VARIATION DURING FORMATION

FIGURE 7-4

IMPREGNATION CONDITIONS
- ALCOHOL 50%
- temperature 50°C
- pH 1,5-2
- [Ni + Co] 2M/l

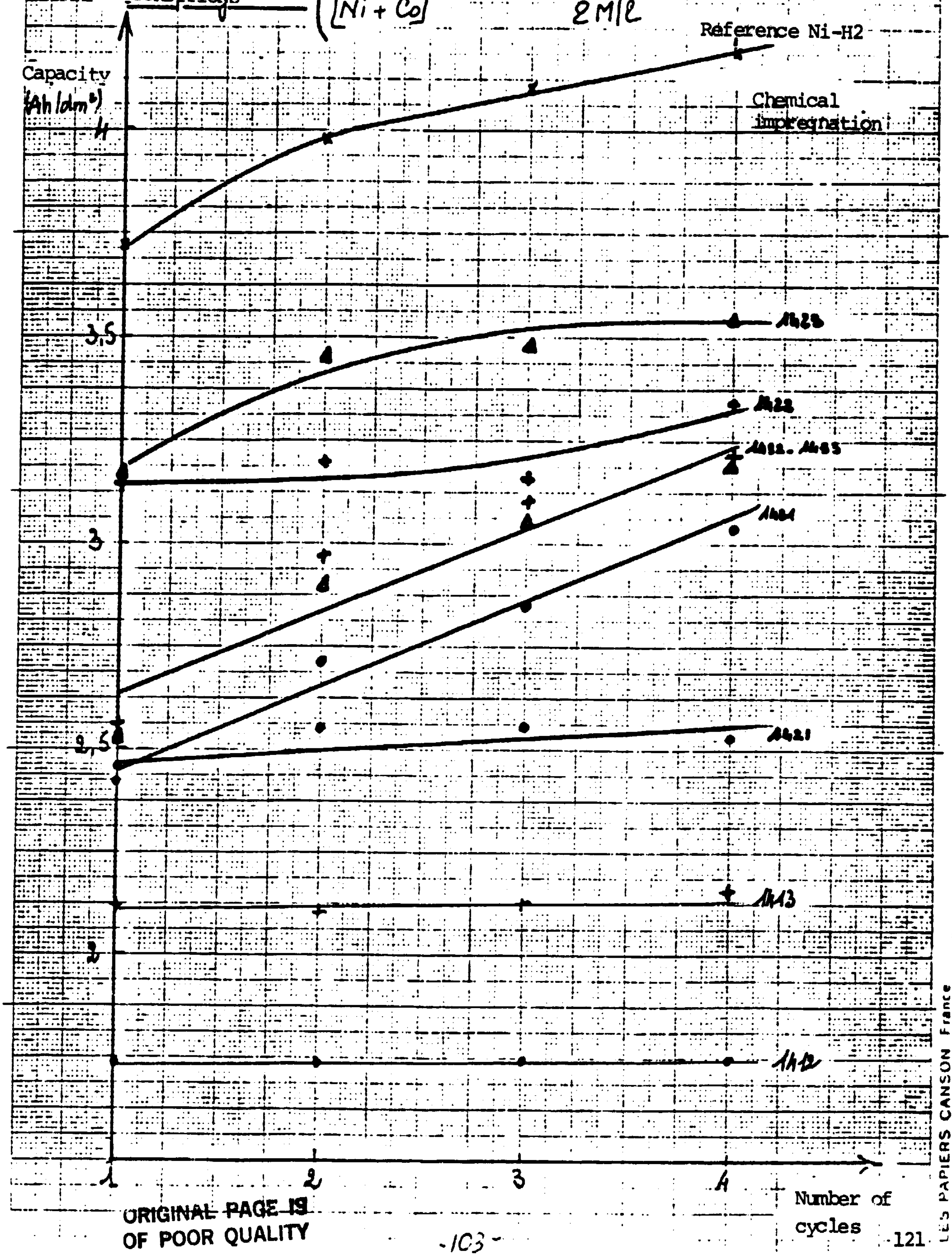


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FIGURE 7-5

CAPACITY VARIATION DURING FORMATION

Impregnation Conditions
- Alcohol 70%
- température 80°C
- pH 1.5 - 2
- [Ni + Co] 2 M/l

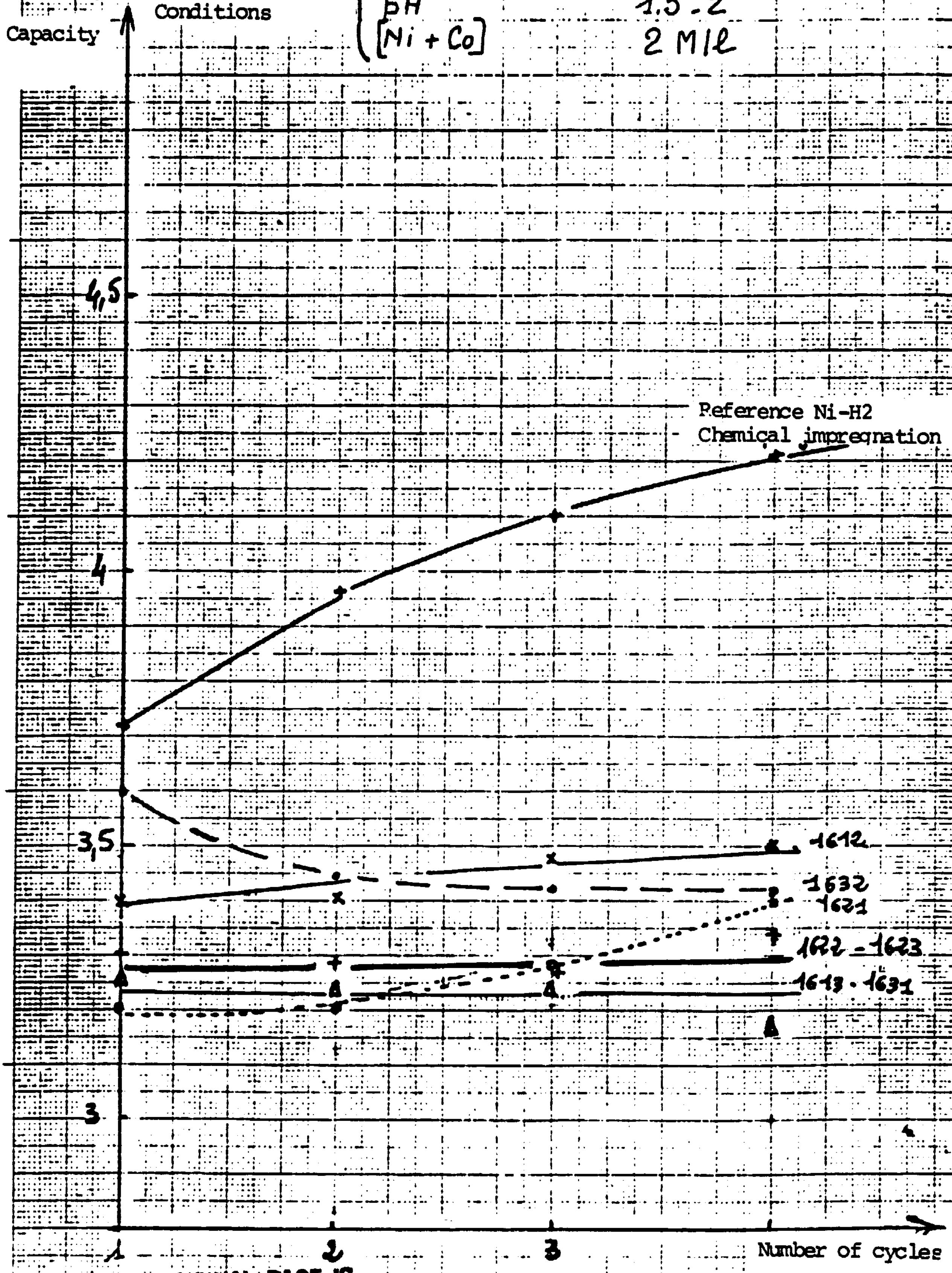

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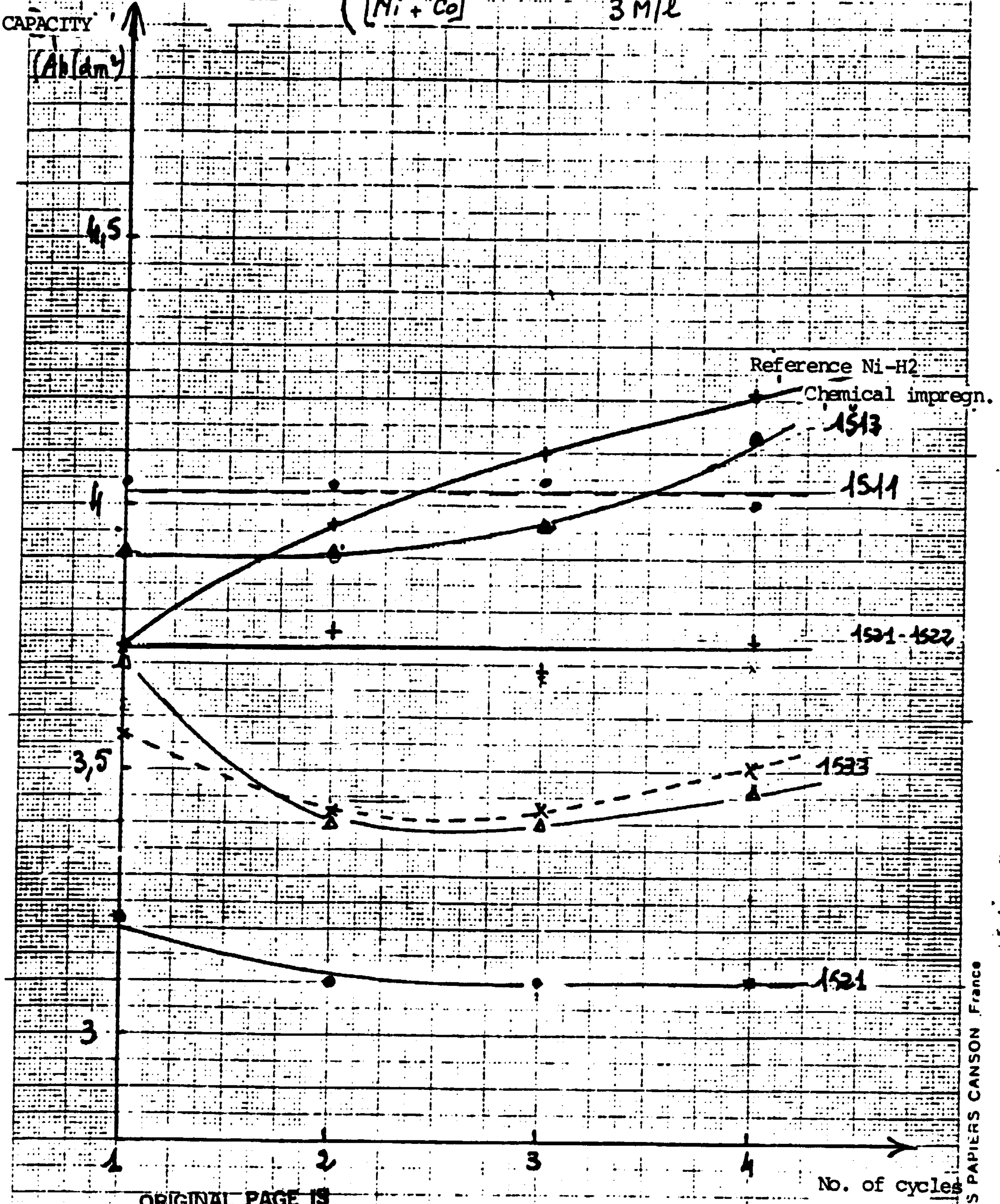
FIGURE 7-6

CAPACITY VARIATION DURING FORMATION

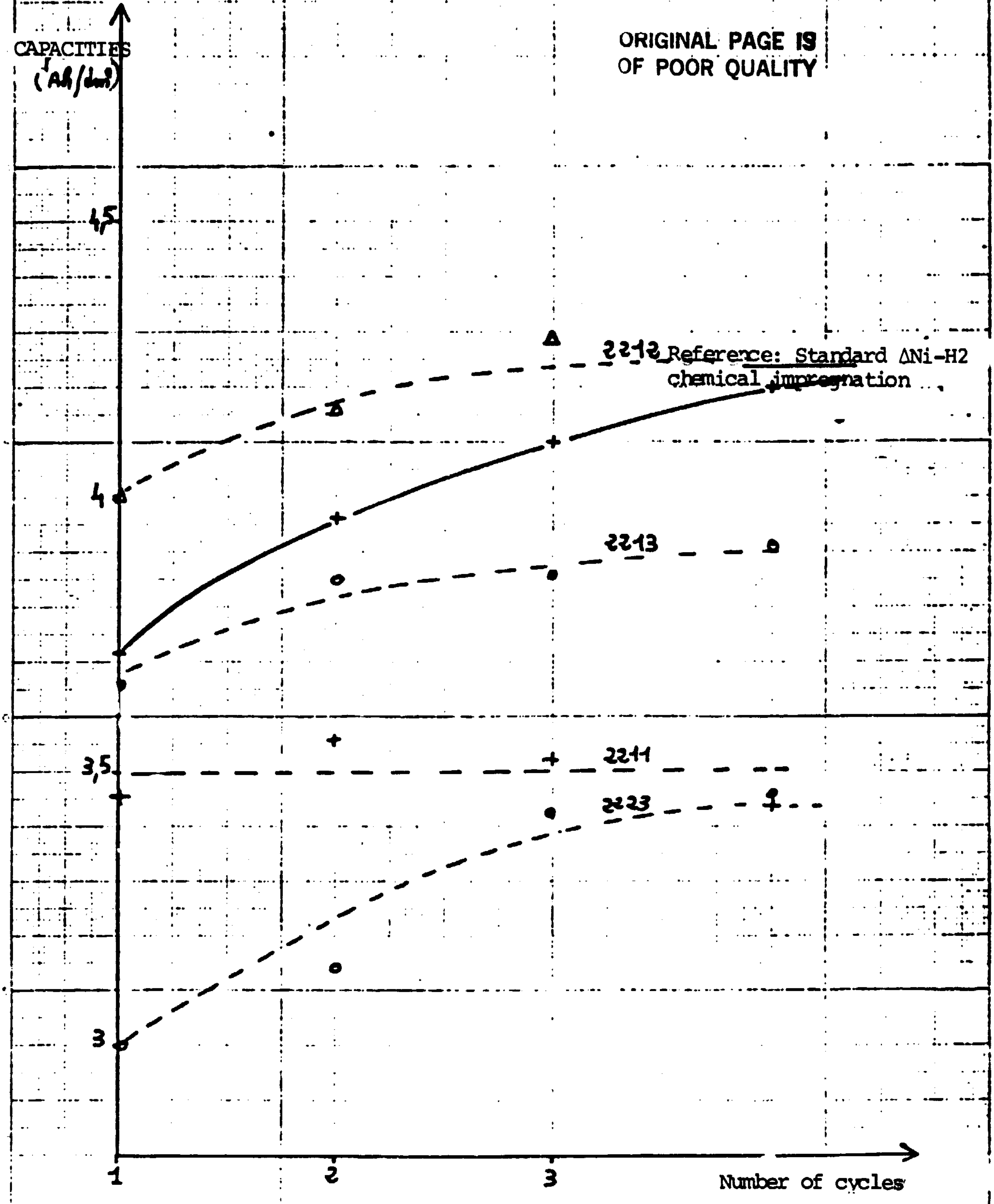
IMPREGNATION CONDITIONS		
	ALCOHOL	50%
	température	80°C
	pH	1,5-2
	[Ni + Co]	3 M/l



CAPACITY VARIATION
DURING FORMATION

FIGURE 7-7

IMPREGNATION CONDITIONS		
	ALCOHOL	50 %
	temperature	80 °C
	pH	3 - 3,5
	[Ni + Co]	2 M/l



FIGURES 8

AL ELECTRODE YIELD DUIRNG 4TH FORMATION CYCLE

ELECTRODE YIELD (4th Formation Cycle)

IMPREGNATION CONDITIONS		
	ALCOHOL	0%
	température	94°C
	pH	1,5-2
	[Ni+Co]	2M/l

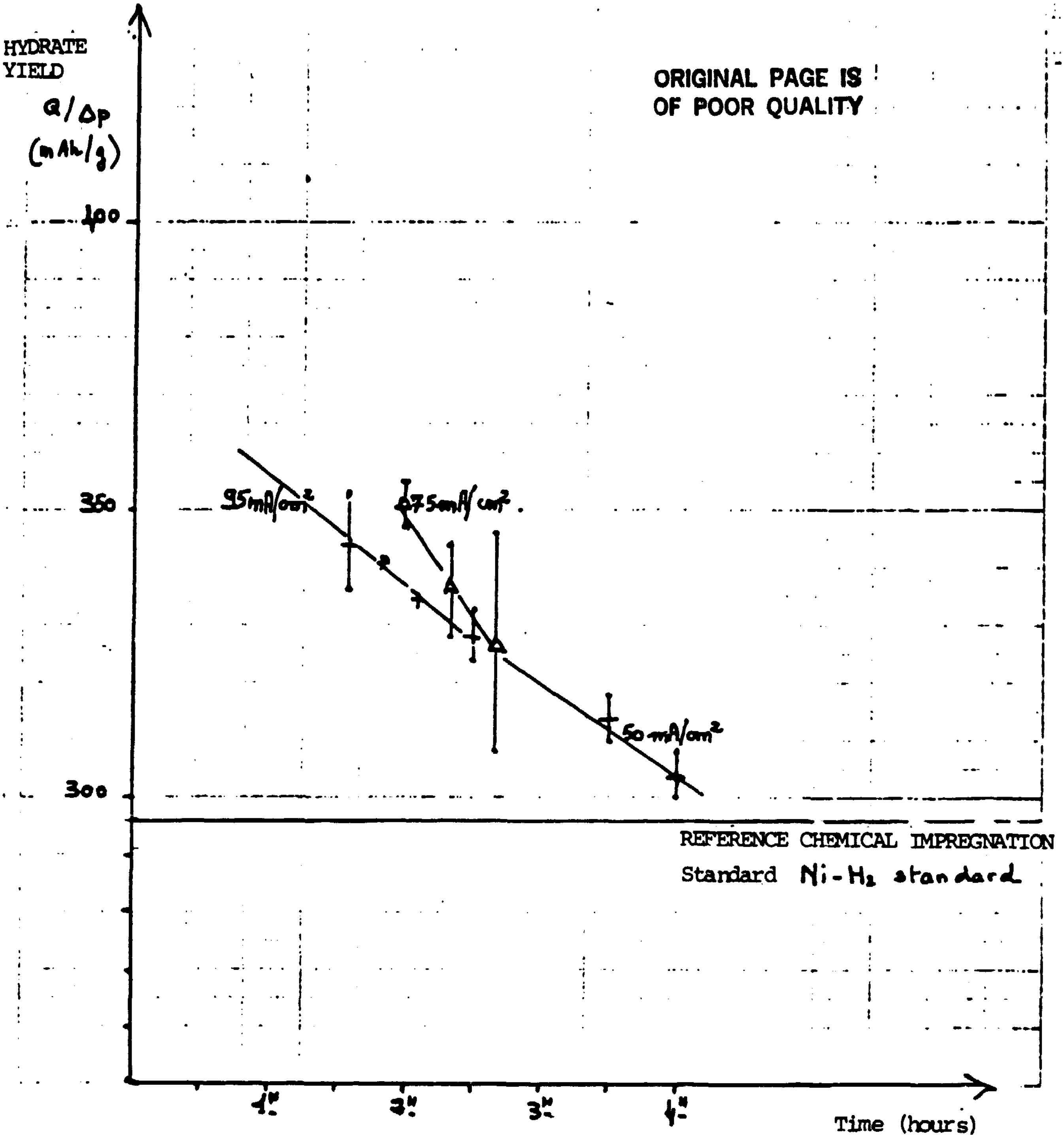


Figure 8.2
FIGURE 8-2

ELECTRODE YIELD (4th FORMATION CYCLE)

Impregnation Conditions

Alcohol 20%
temperature 84°C
pH 1,5-2
[Ni + Co] 2 M/l

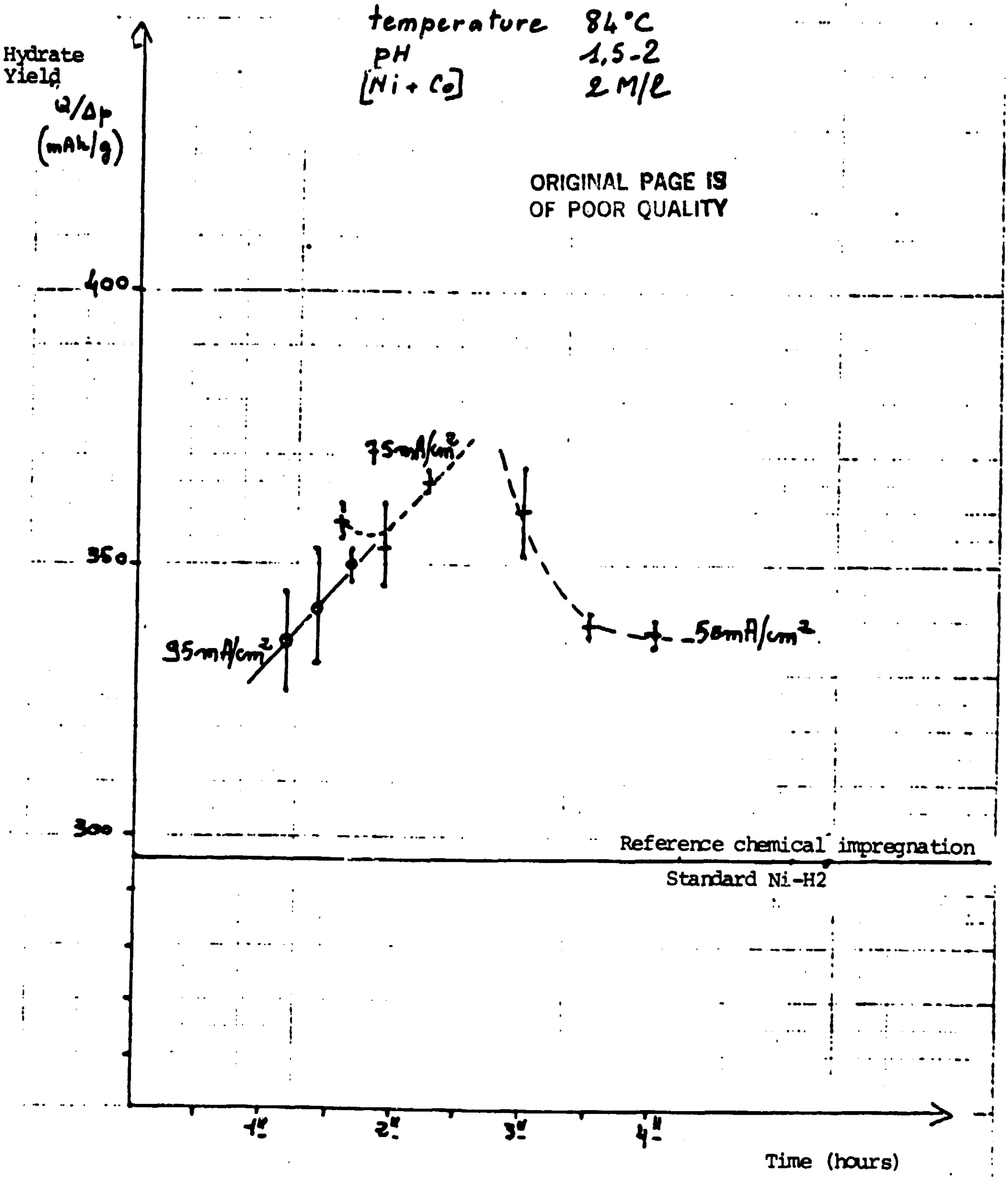


FIGURE 8-3

ELECTRODE YIELD - 4TH FORMATION CYCLE

IMPREGNATION CONDITIONS

ALCOHOL	50%
temperature	80°C
pH	1,5-2
[Ni + Co]	2 M/l

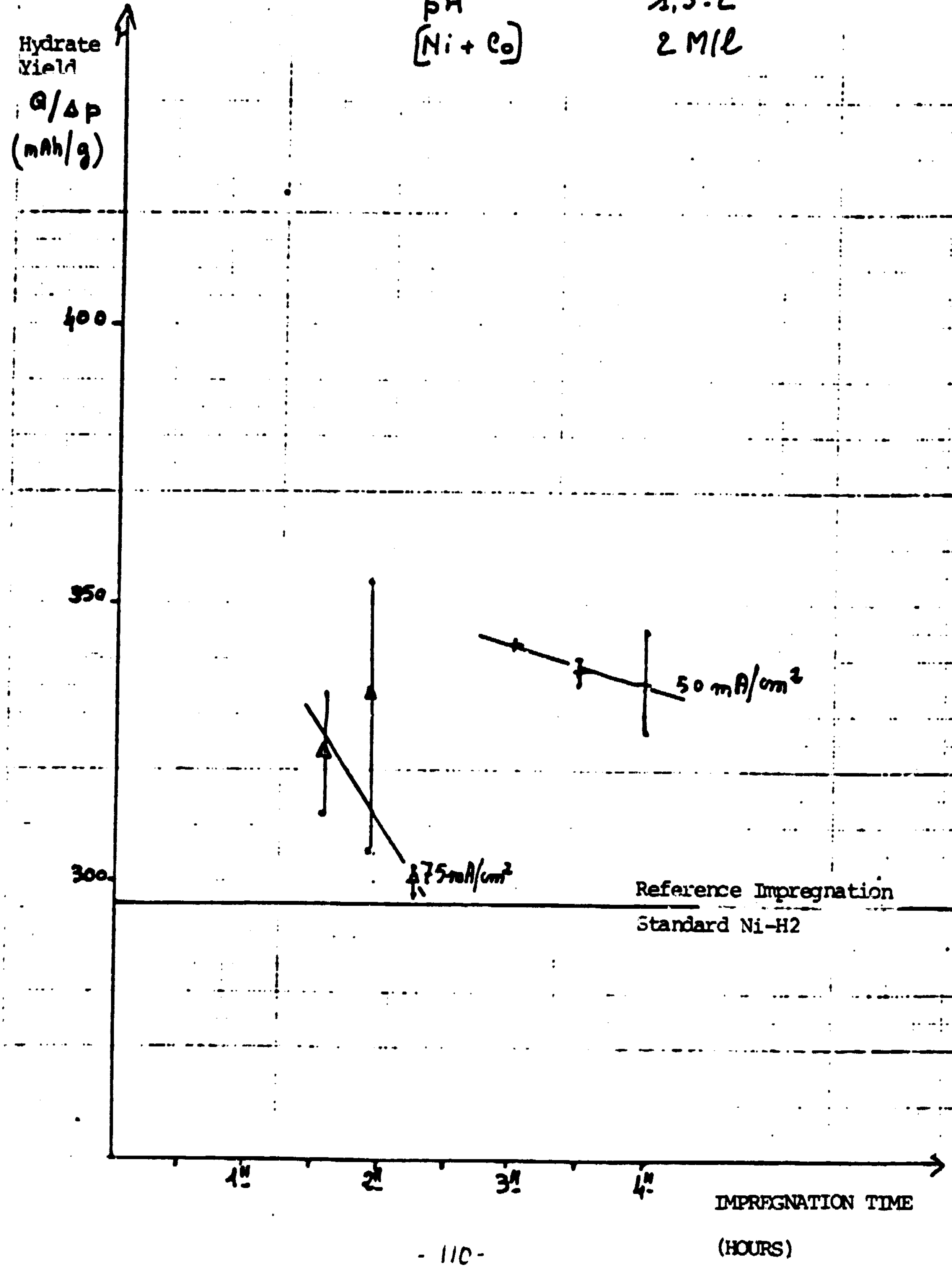
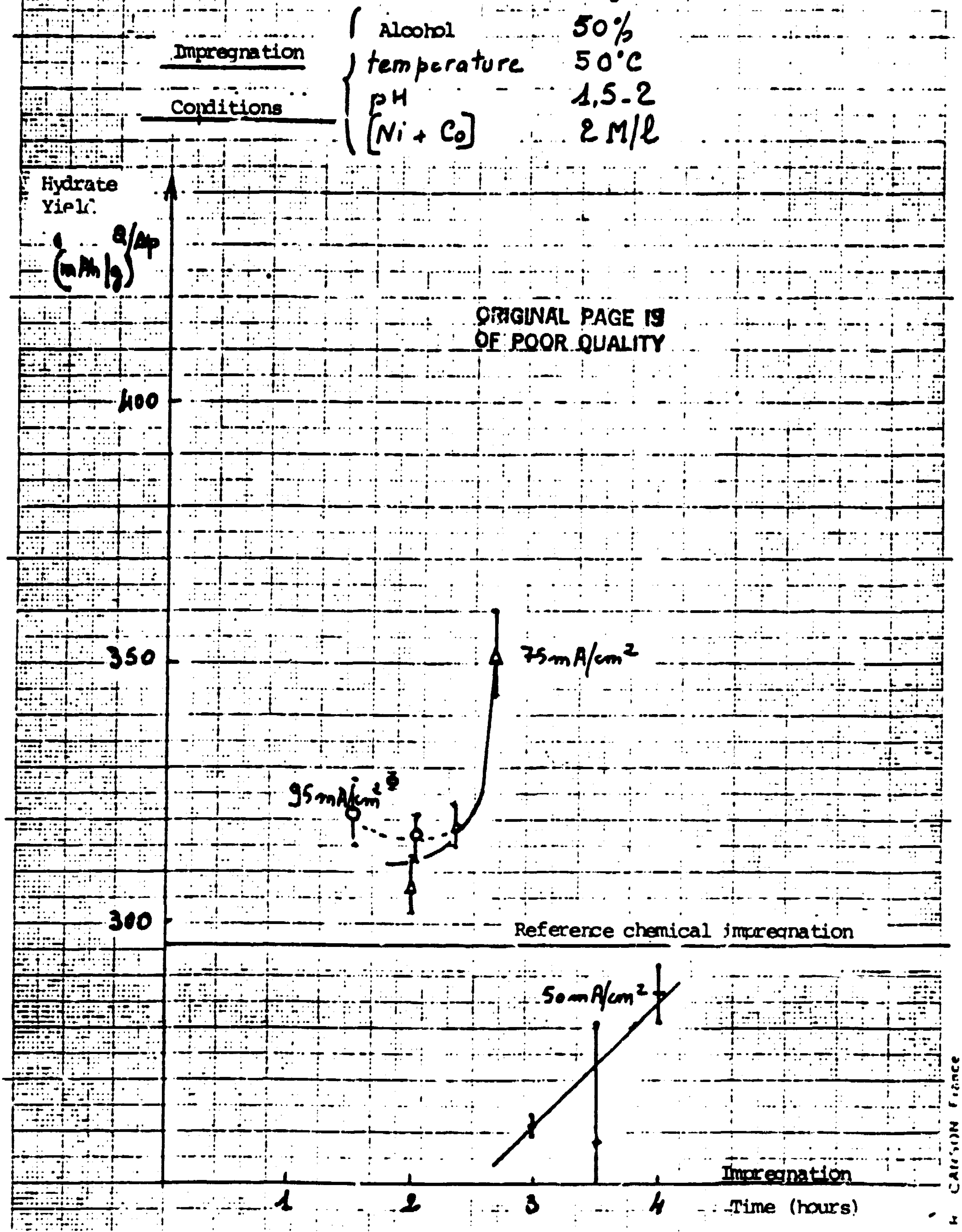


FIGURE 8-4

ELECTRODE YIELD (4TH FORMATION CYCLE)



Electrode Yield (4th formation cycle)

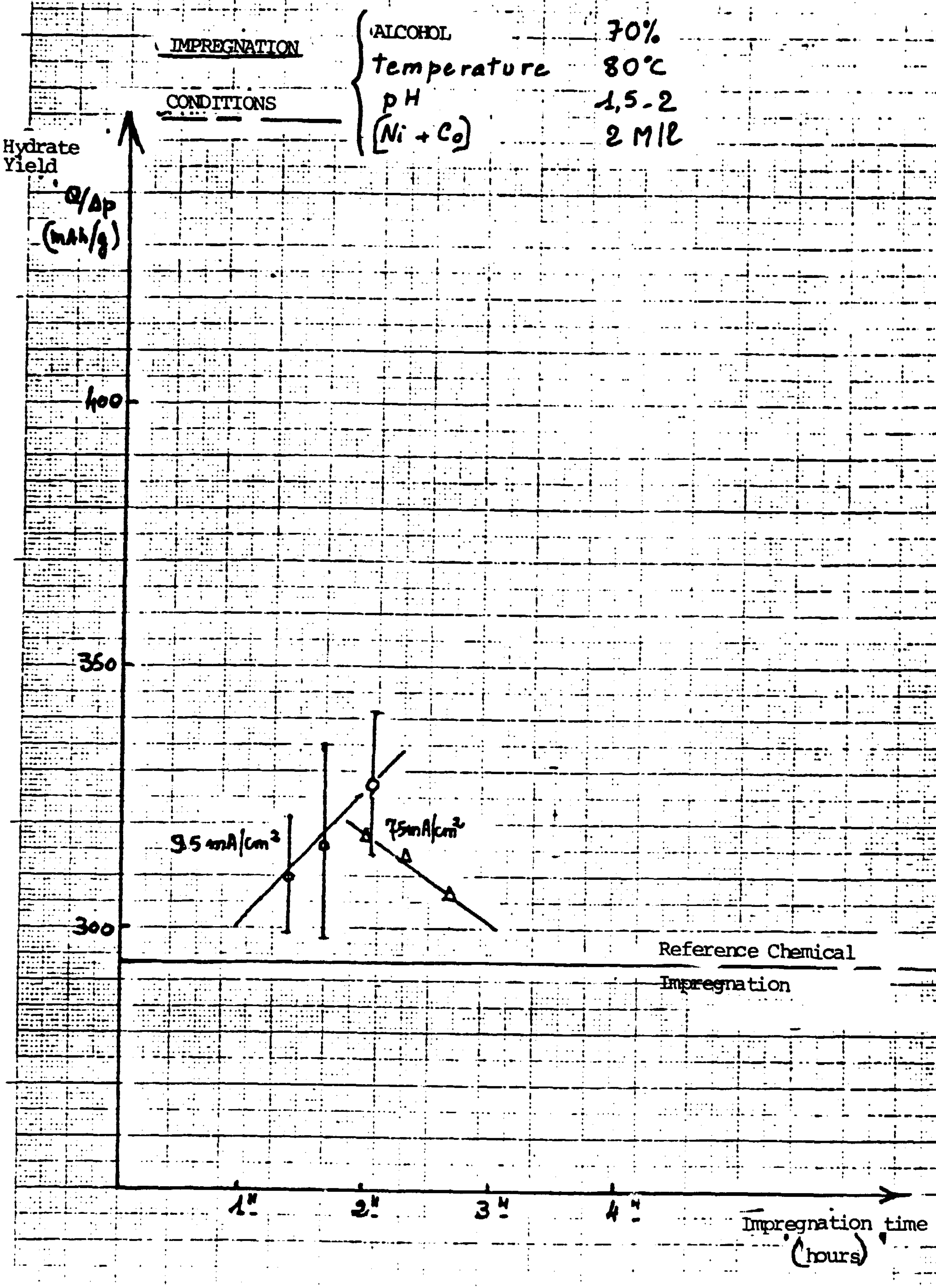


FIGURE 8-6

ELECTRODE YIELD
4TH FORMATION CYCLE

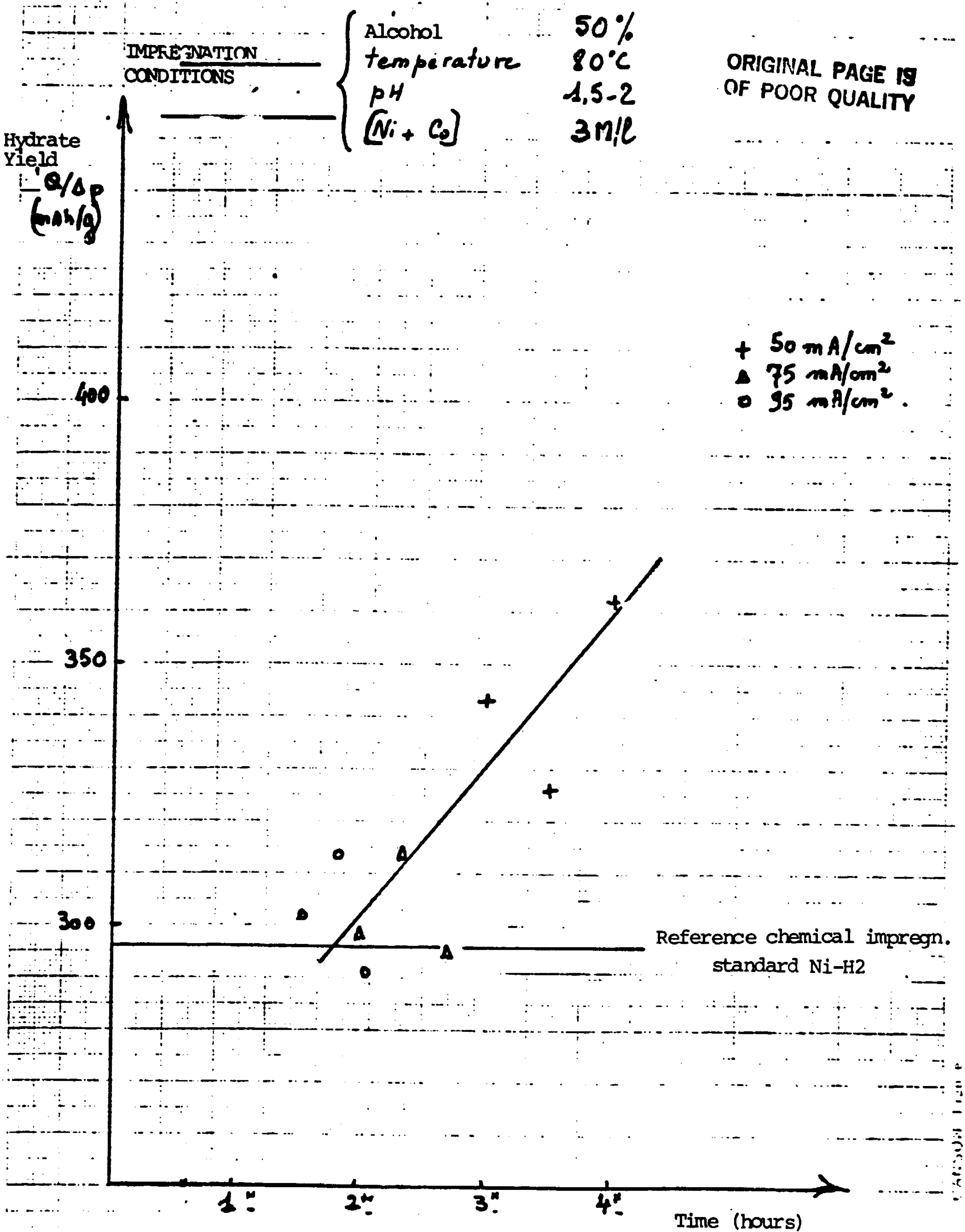
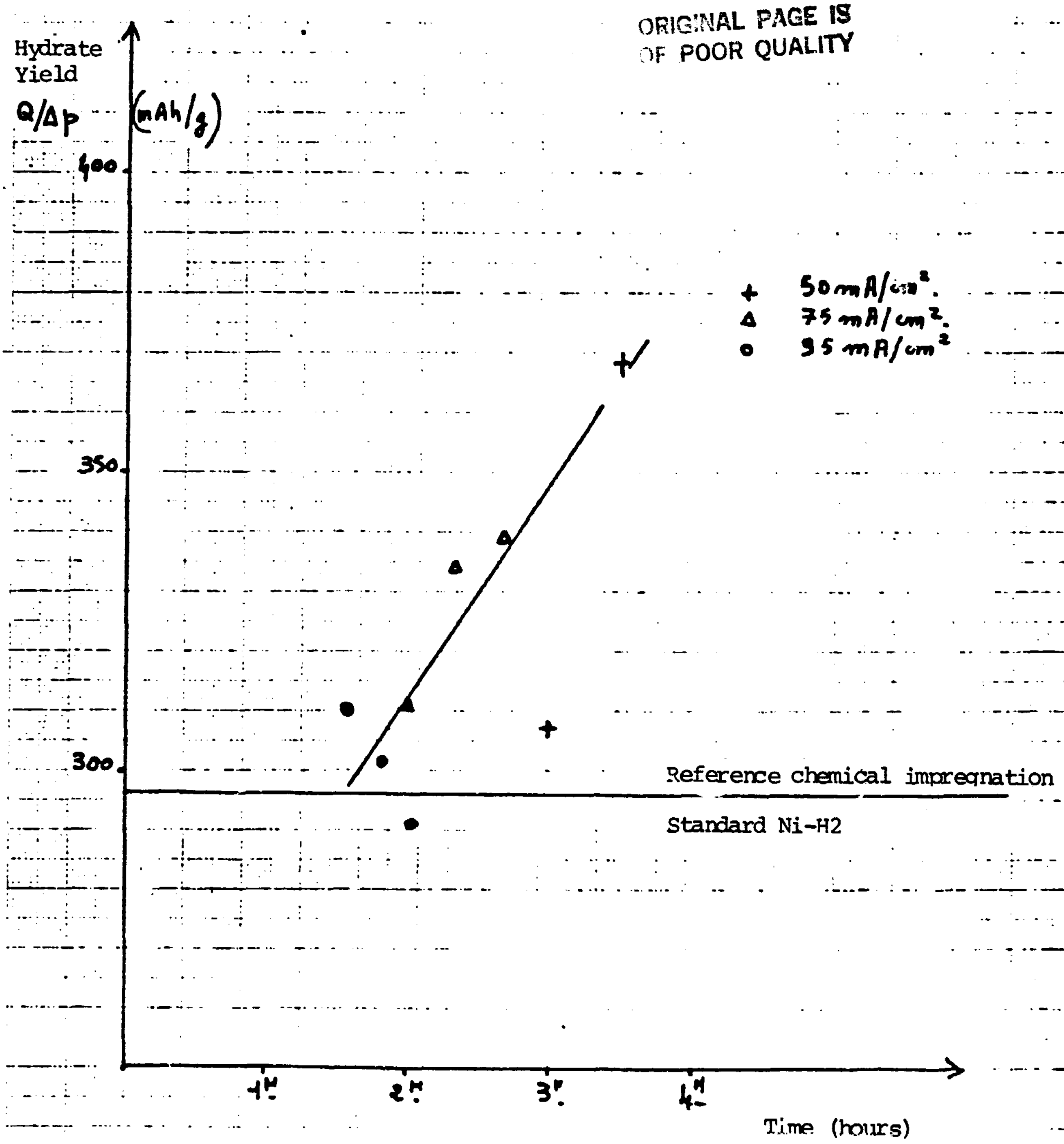


FIGURE 8-7

ELECTRODE YIELD (4TH FORMATION CYCLE)

IMPREGNATION CONDITIONS

Alcohol	50%
température	80°C
pH	3-3,5
[Ni + Co]	2 M/l



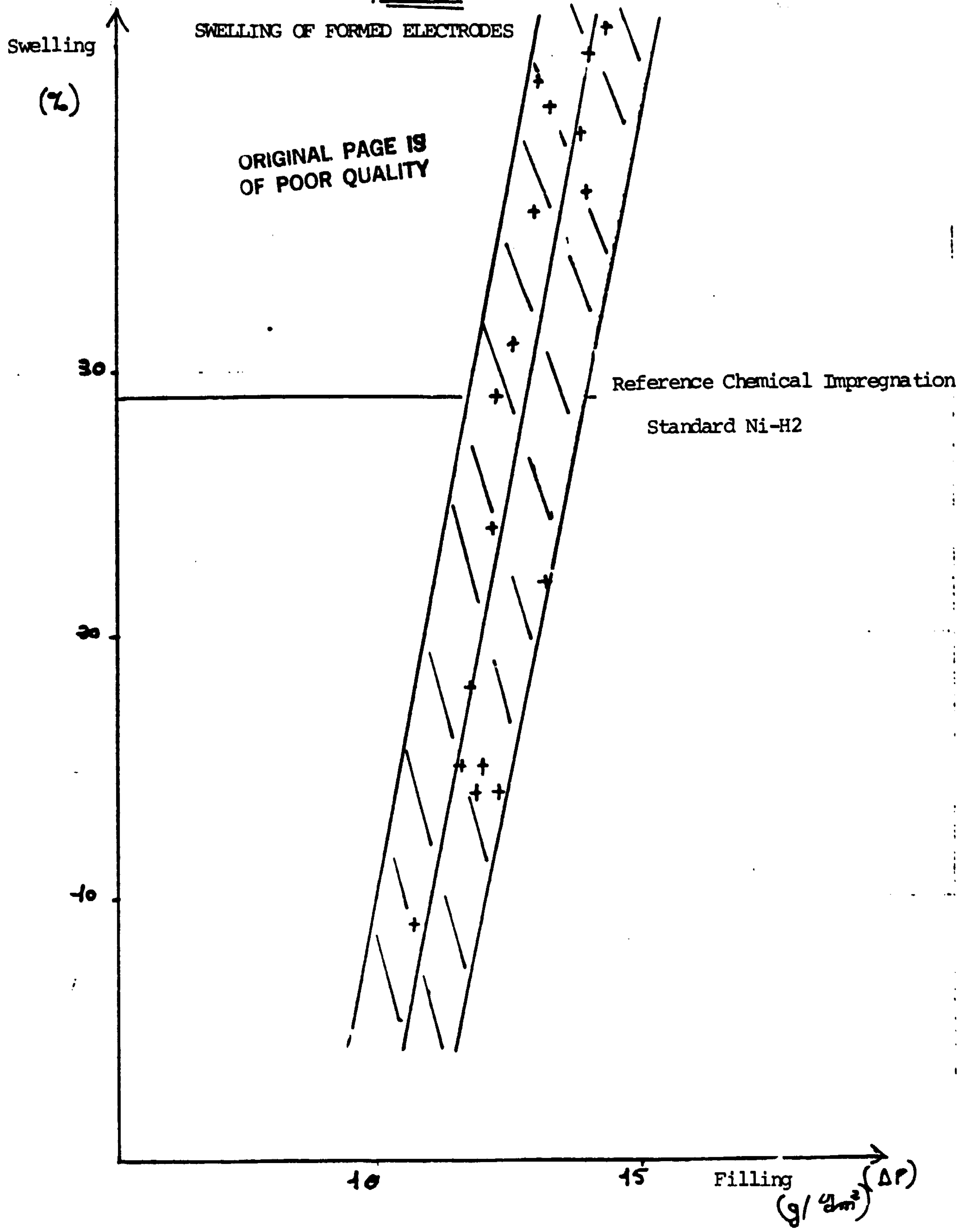
FIGURES 9

ELECTRODE THICKNESS INCREASE AFTER FORMATION

(% Initial Thickness of Sintering)

FIGURE 9.1

SWELLING OF FORMED ELECTRODES



Impregnation Conditions :

Alcohol	0 %	pH	1,5 - 2
Temperature	98°C	[Ni + Co]	2 M/l

Figure

FIGURE 9.2

SWELLING OF FORMED ELECTRODES

IMPREGNATION CONDITIONS:

ALCOHOL	20%
temperature	84°C
pH	1,5-2
[Ni + Co]	2 M/l

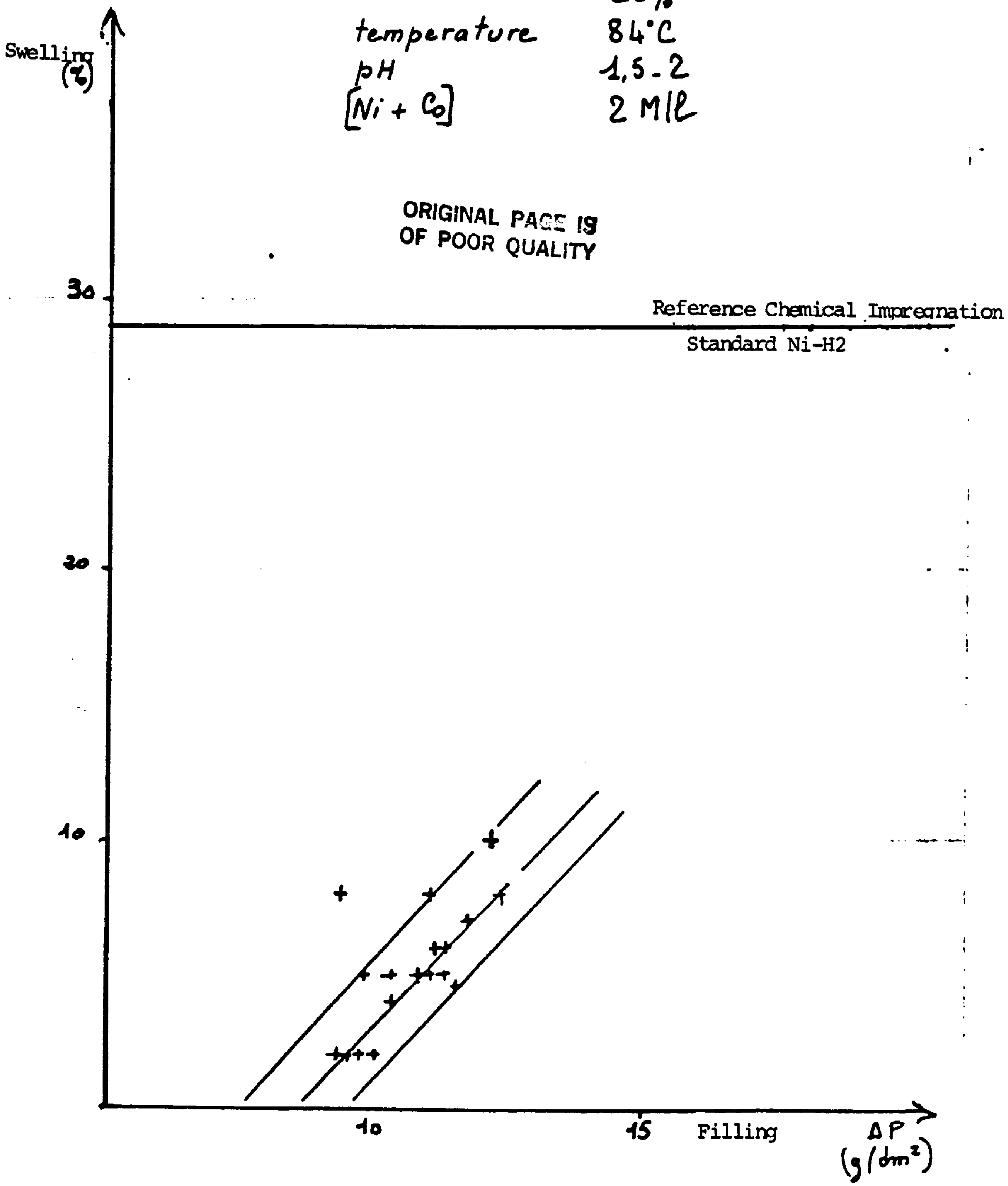


Figure 2-3

FIGURE 9.3

SWELLING
OF
Formed
Electrodes

IMPREGNATION CONDITIONS:

ALCOHOL: 50%
temperature 80°C
pH 1,5-2
[Ni + Co] 2 M/l

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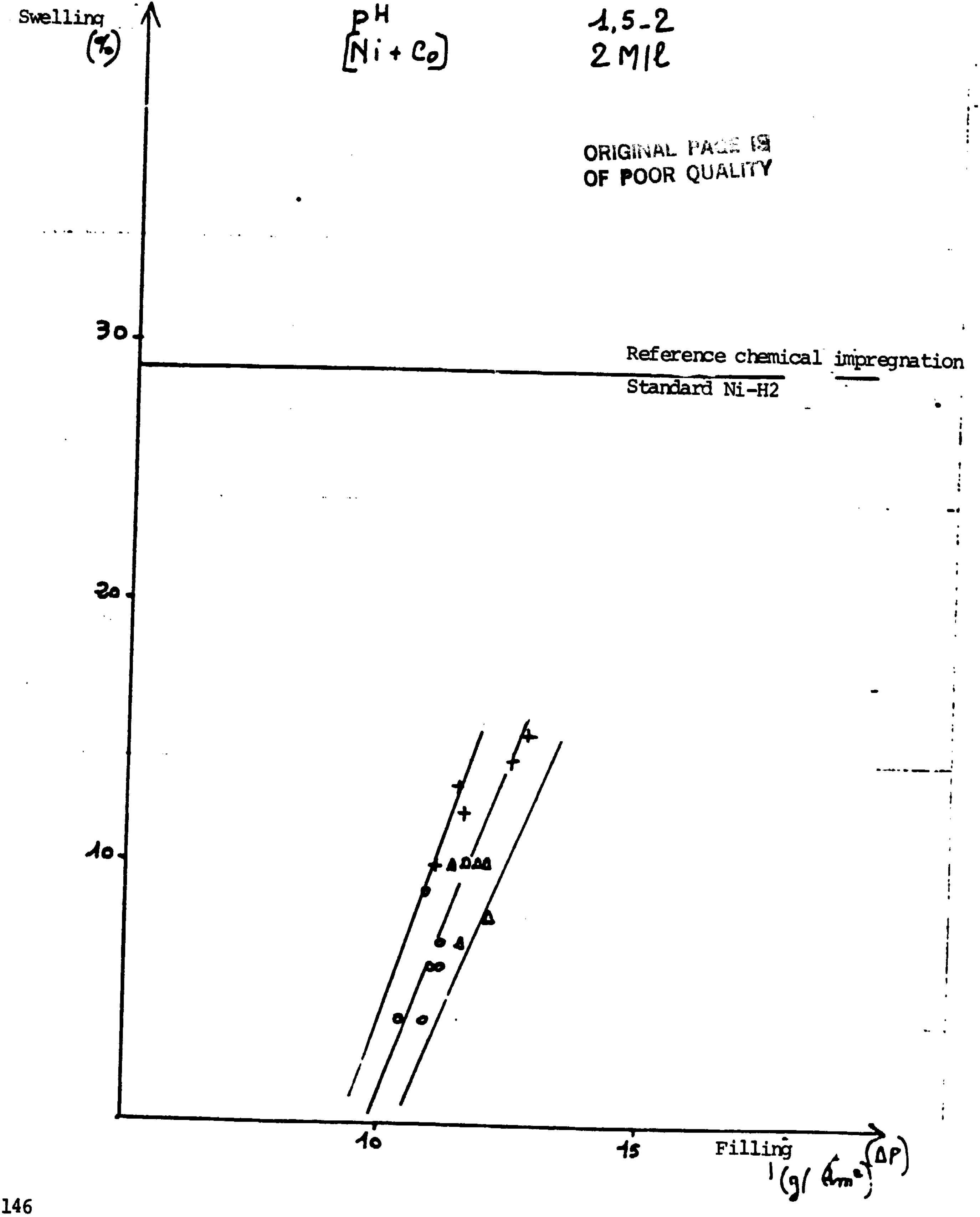

Figure 9-4
FIGURE 9-4

SWELLING
OF
FORMED
ELECTRODES

IMPREGNATION CONDITIONS:

Alcohol: 50%
temperature 50°C
pH 1,5 - 2
[Ni + Co] 2 M/l

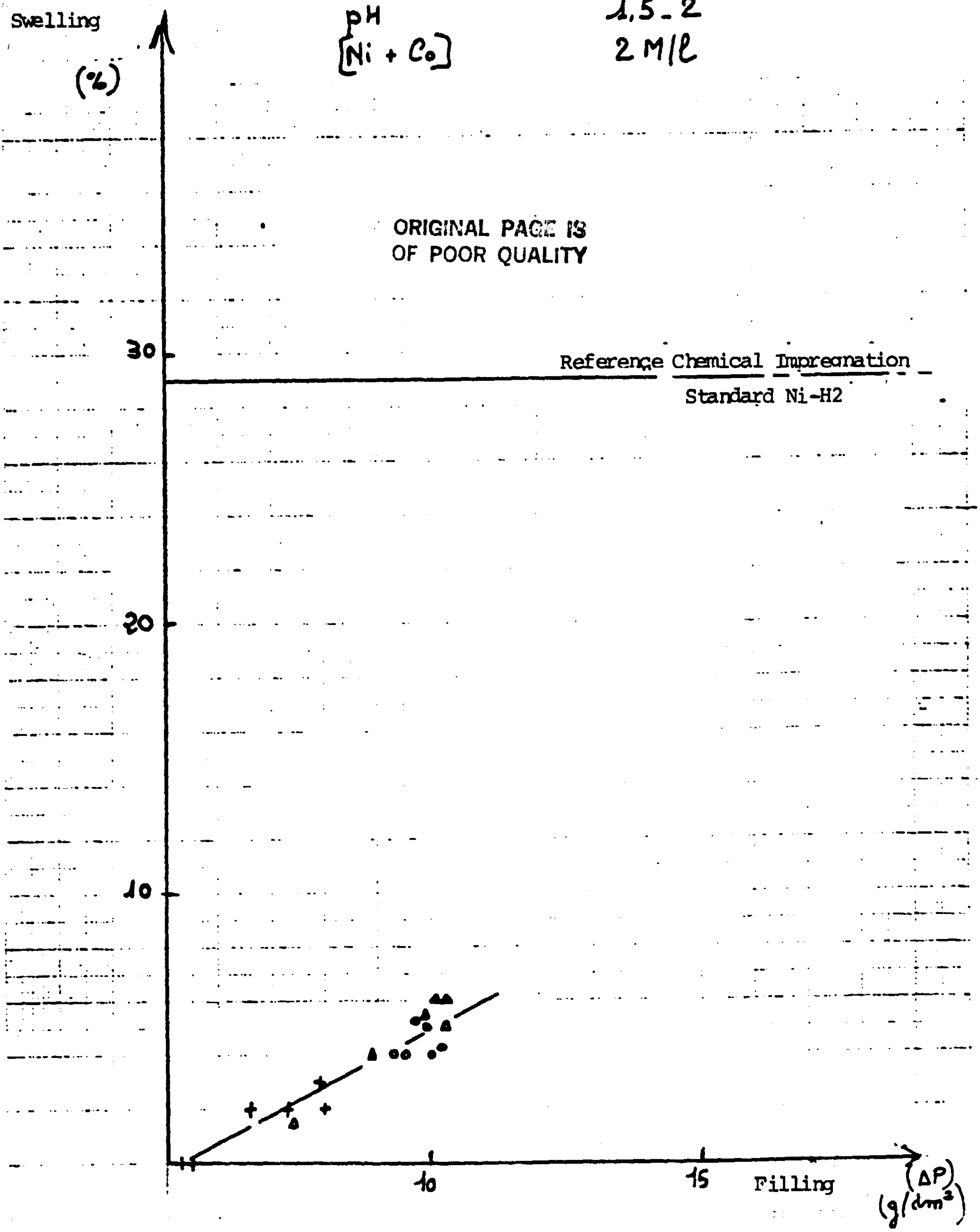

Figure 9.5

FIGURE 9.5

SWELLING OF FORMED ELECTRODES

IMPREGNATION CONDITIONS

Alcohol:	70%
temperature	80°C
pH	1.5-2
[Ni + Co]	2 M/l

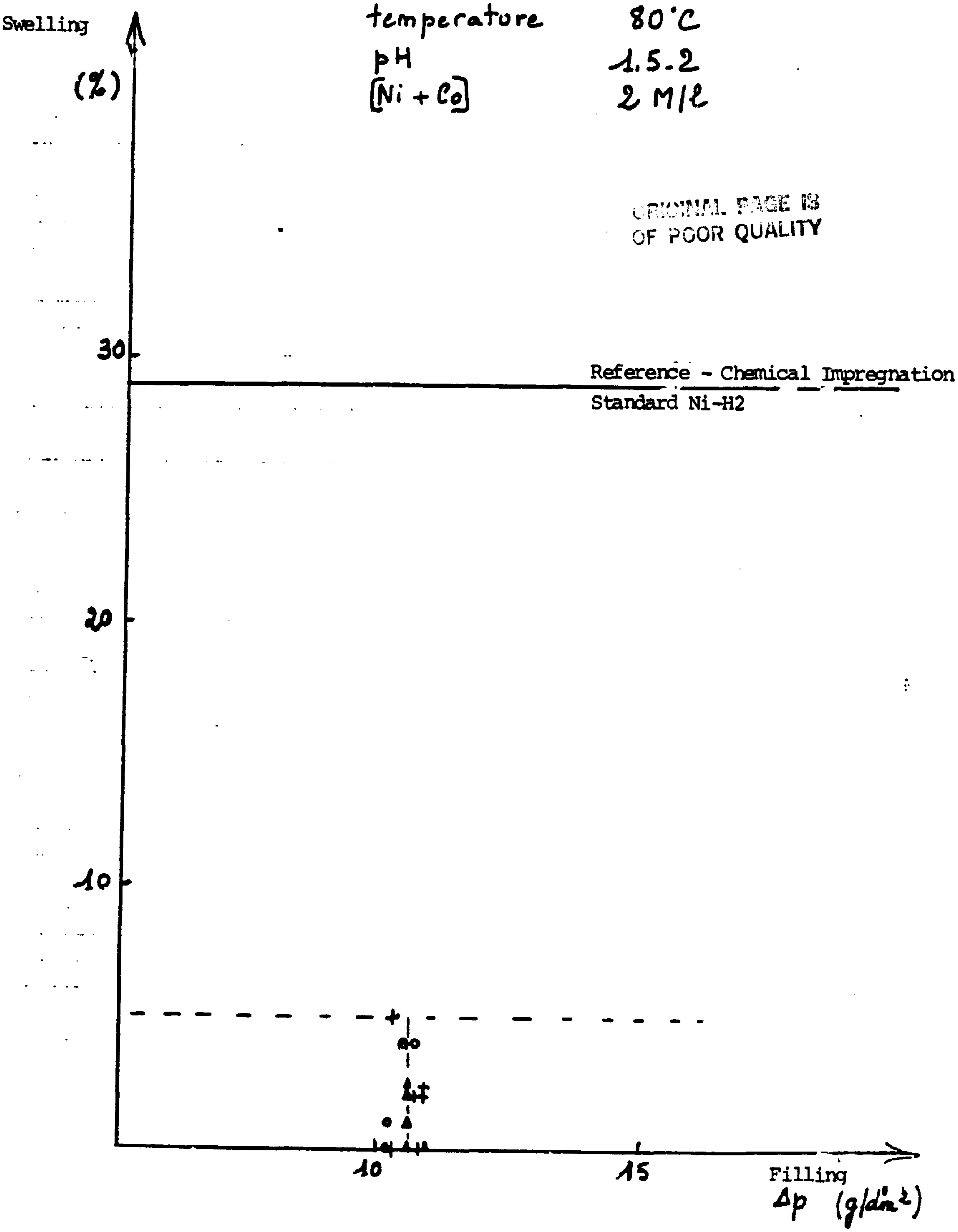

Figure 9.6

FIGURE 9.6

SWELLING
OF
FORMED
ELECTRODES

IMPREGNATION CONDITIONS :

ALCOHOL	50%
temperature	80°C
pH	1,5-2
[Ni + Co]	3 M/l

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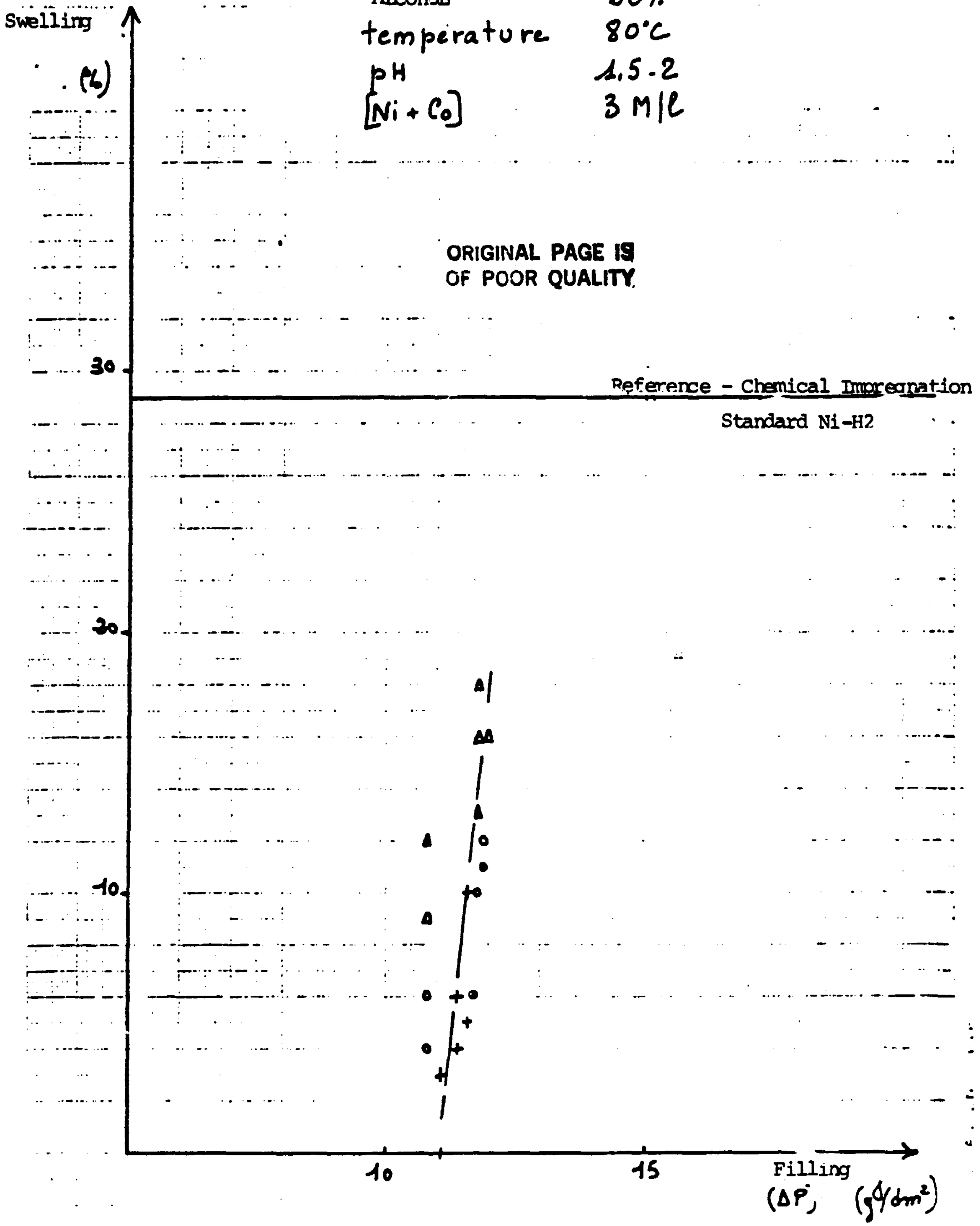


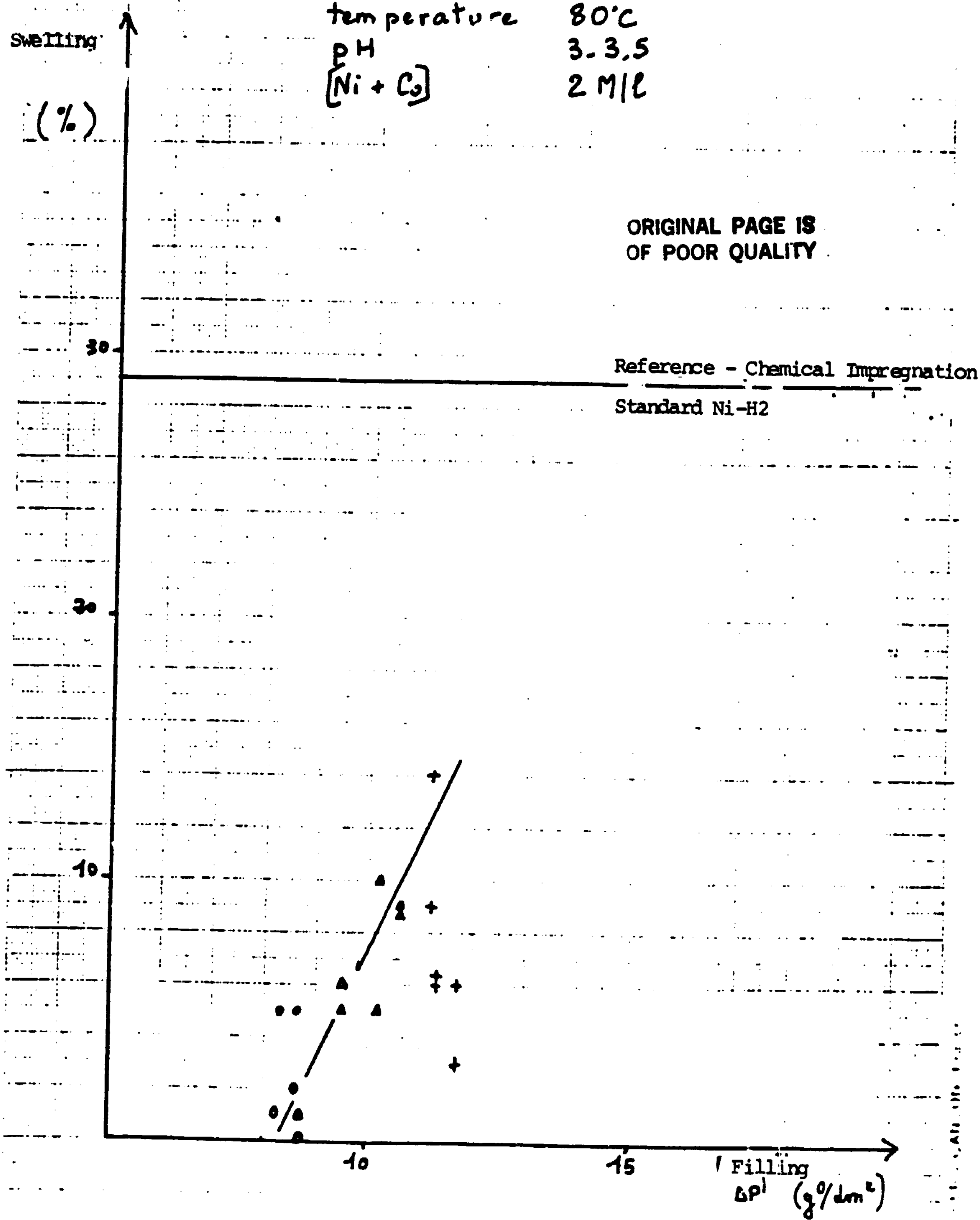
Figure [illegible]

FIGURE 9.7

SWELLING OF FORMED ELECTRODES

IMPREGNATION CONDITIONS:

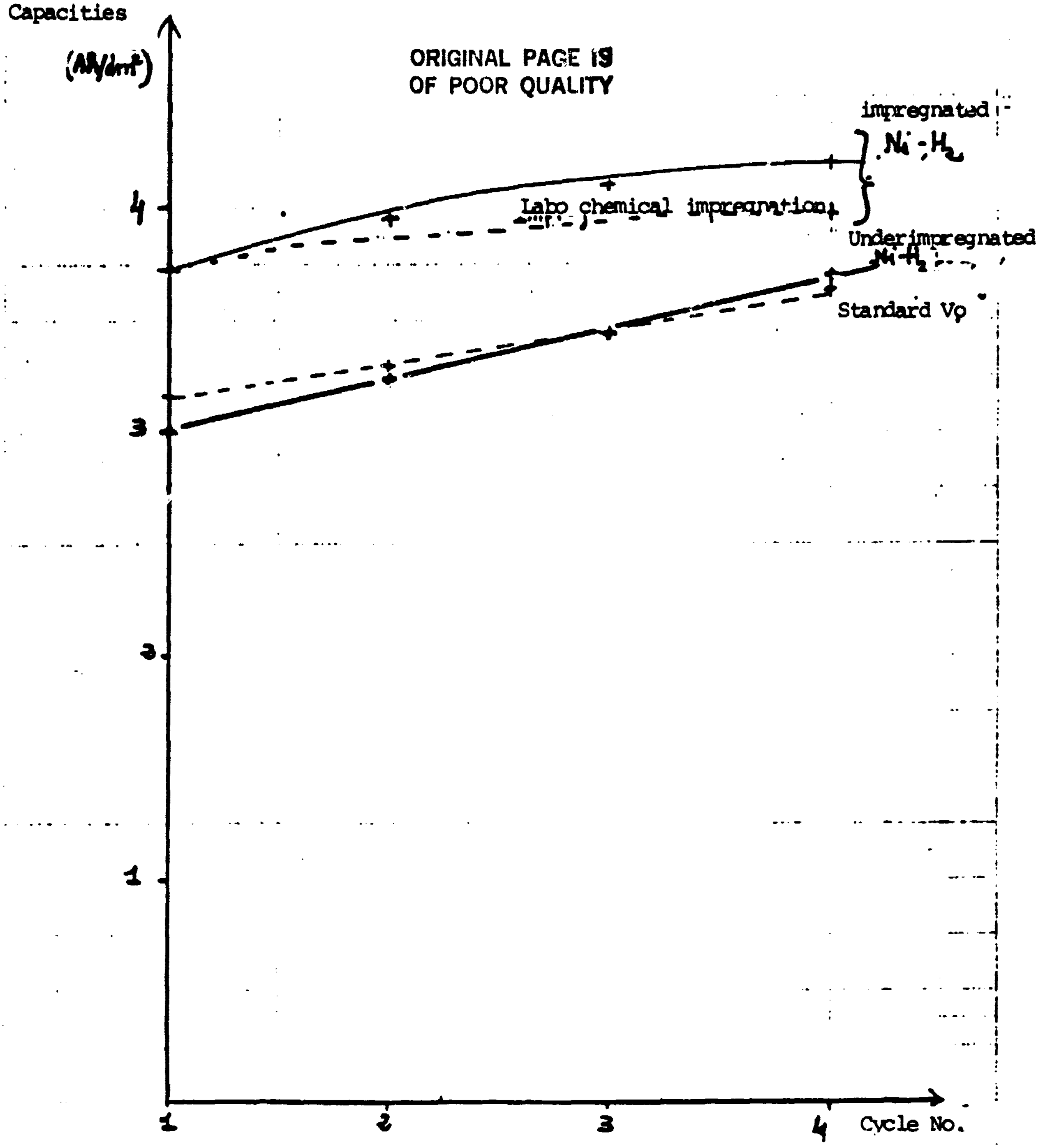
Alcohol	50%
temperature	80°C
pH	3-3.5
[Ni + Co]	2 M/l



SUMMARY CURVES

Figure 10

CHEMICAL IMPREGNATION - FORMATION OF Ni-H2 and VO TYPE PLATES - CAPACITY VARIATION DURING THE FORMATION CYCLES



IMPACT OF THE ALCOHOL CONTENT ON THE FILLING OF THE ELECTRODES

FIGURE 11

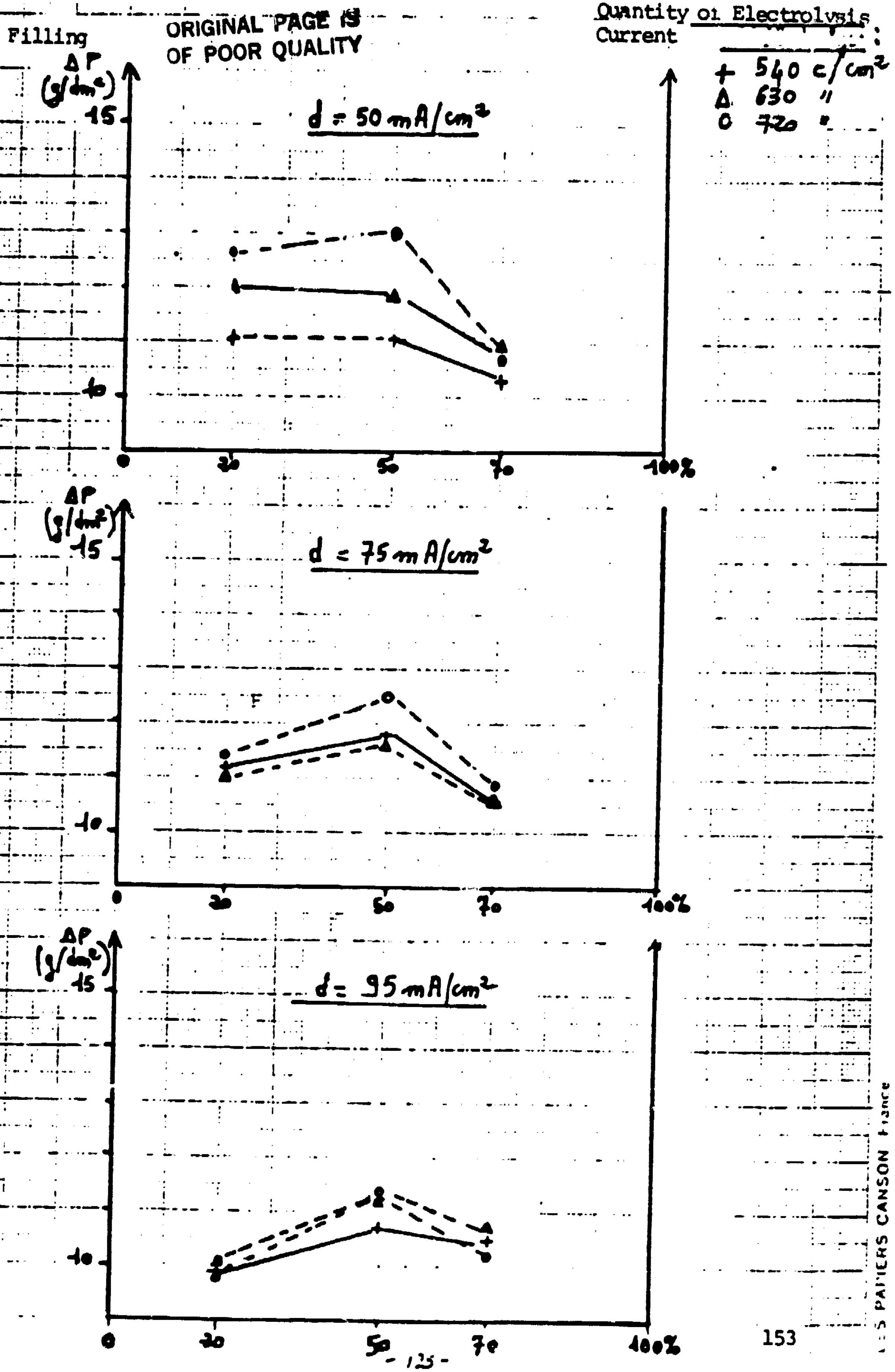
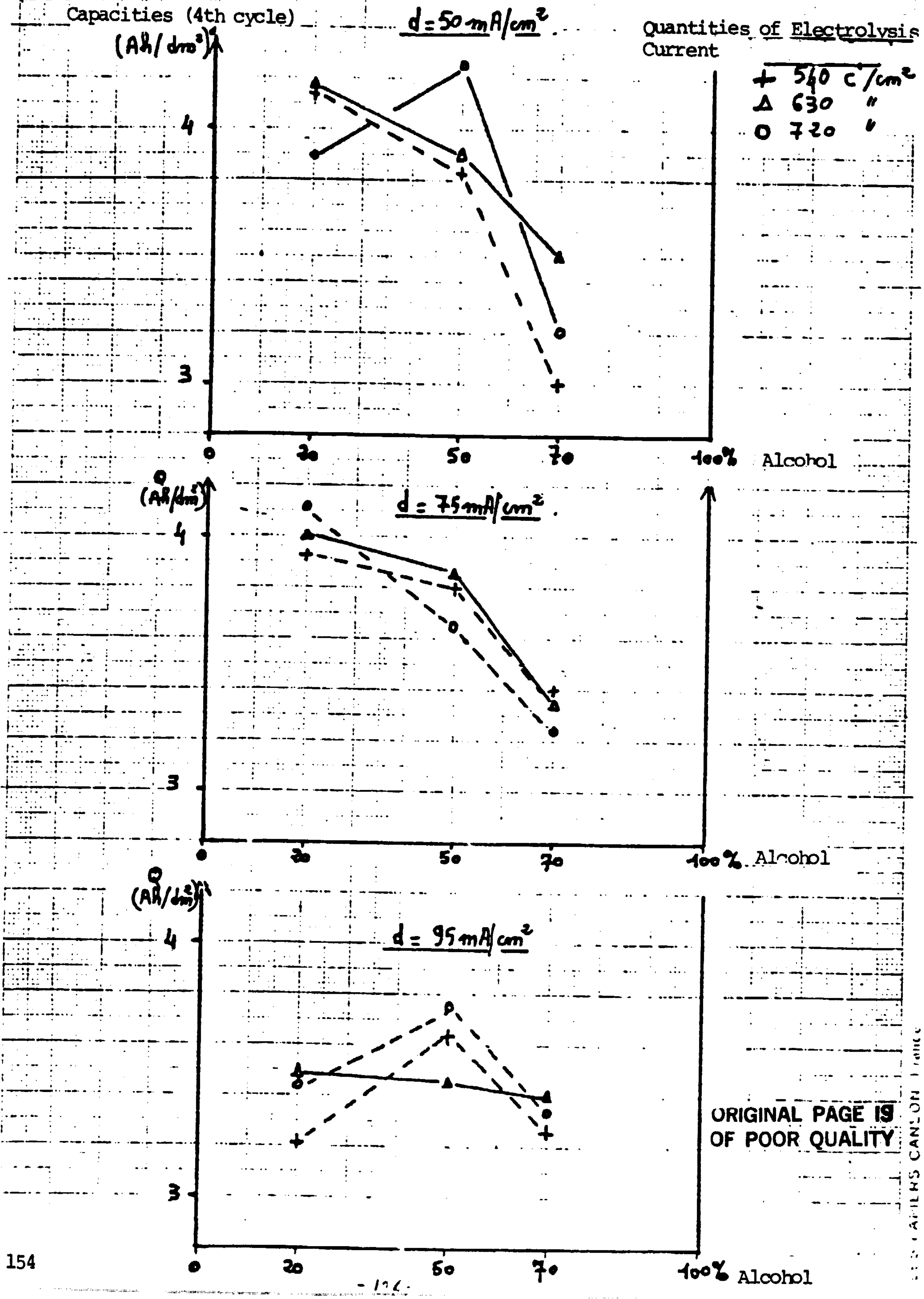


Figure 12

IMPACT OF THE ALCOHOL CONTENT ON THE CAPACITIES (4th FORMATION CYCLE)



IMPACT OF THE FILLING RATE ON THE HYDRATE YIELD FIGURE 13

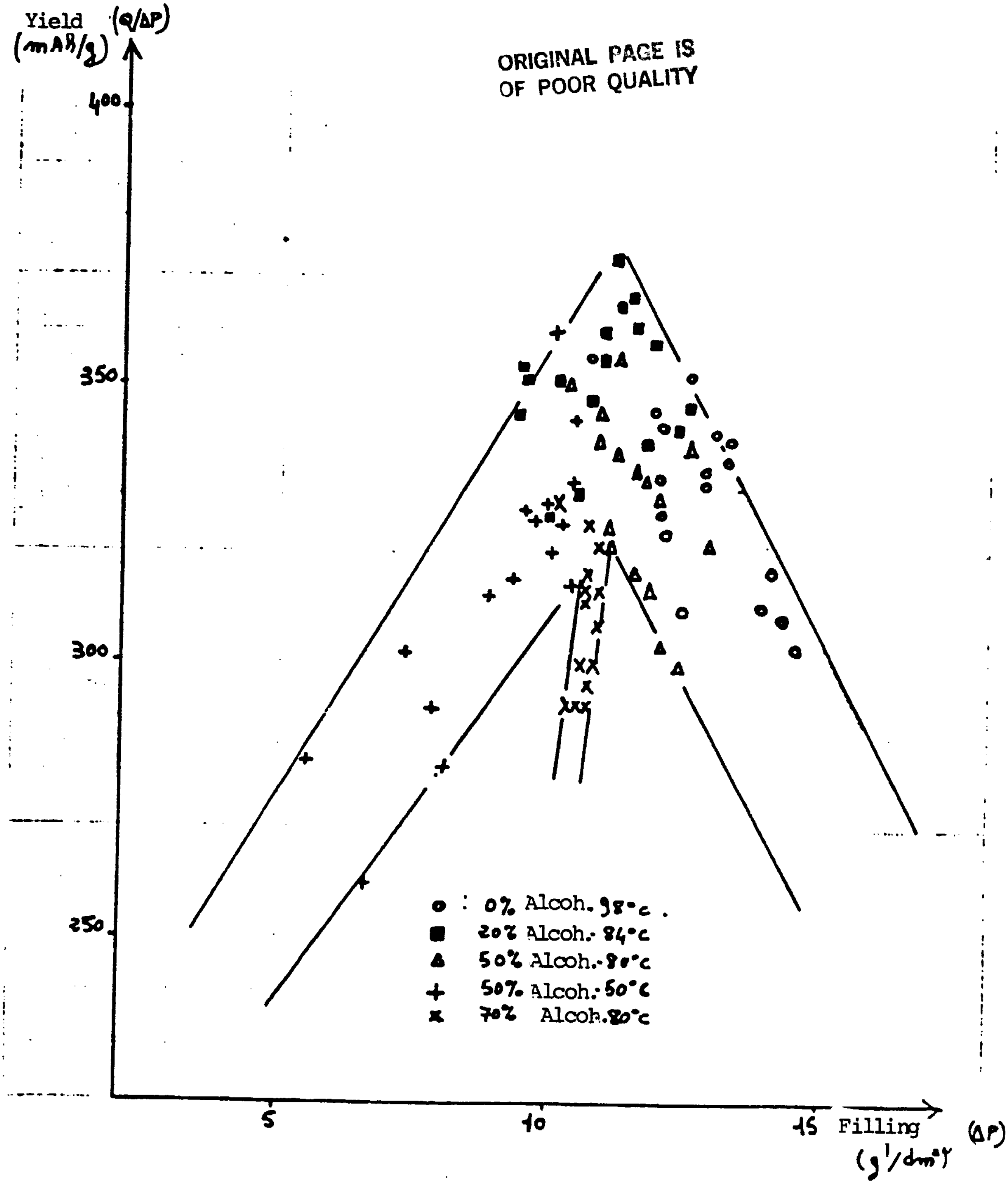


Figure 14

VARIATION OF THE HYDRATE HIELD AS A FUNCTION OF THE STOECHIMETRIC COEFFICIENT S.

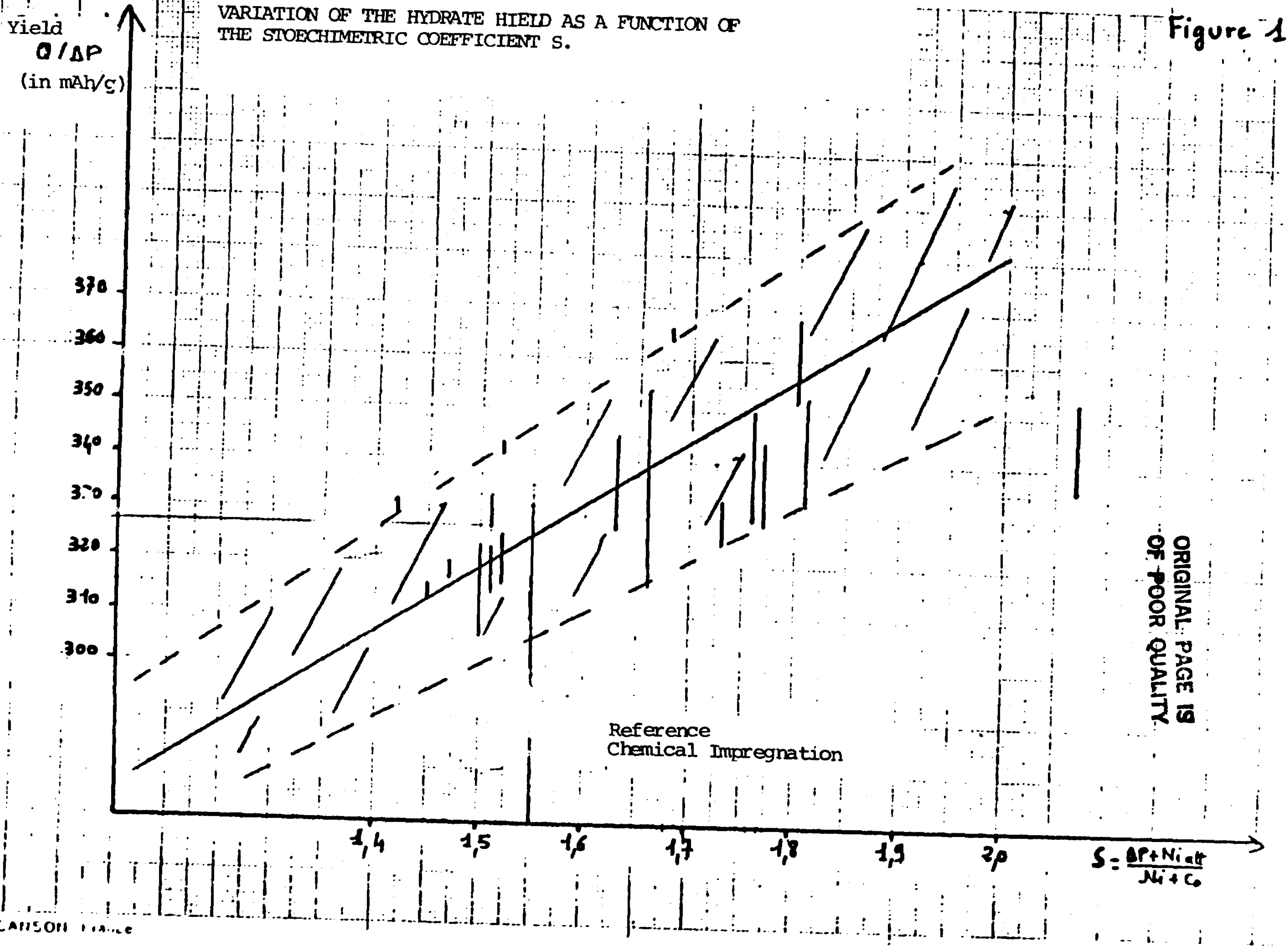


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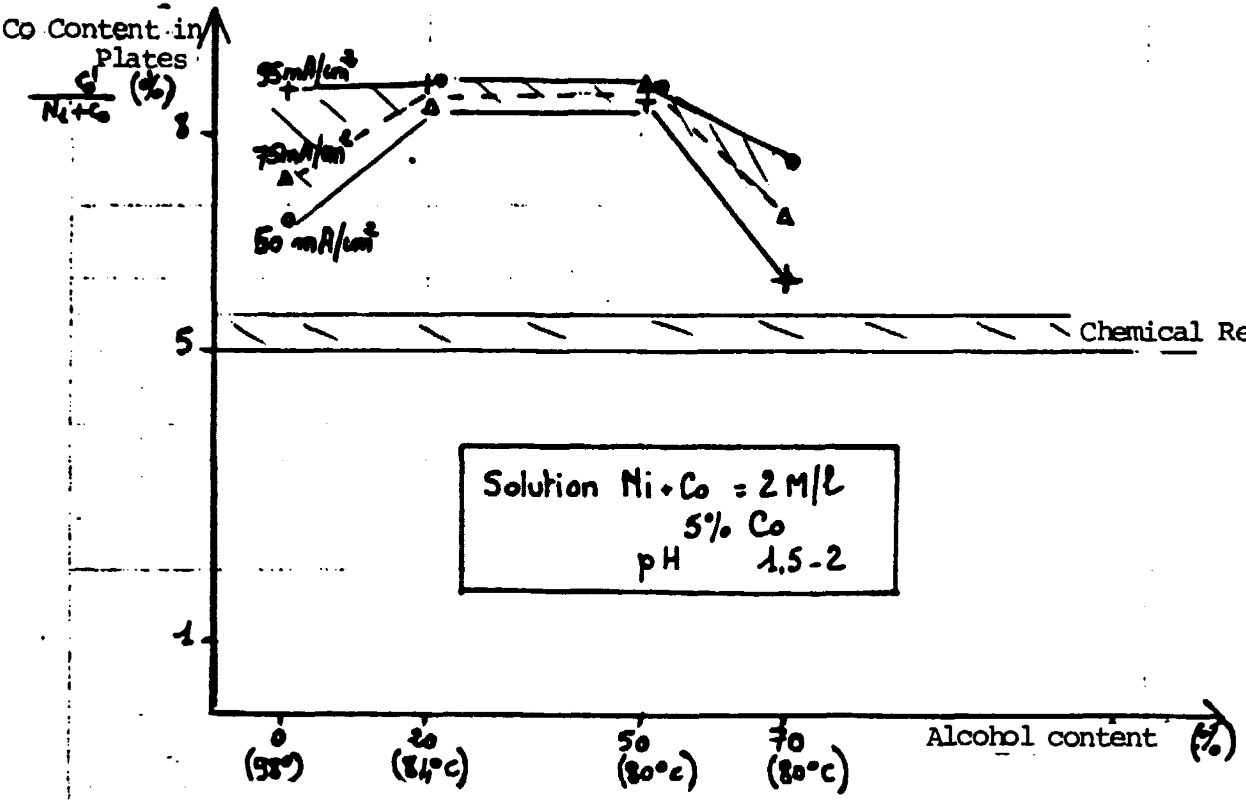
Figure 15

IMPACT OF THE IMPREGNATION CONDITIONS ON THE COBALT CONTENT IN THE PLATES.

FIGURE 15

- A - Impact of Alcohol and of the Electrolysis Current Densities



- B - Impact of Co content in the impregnation bath.

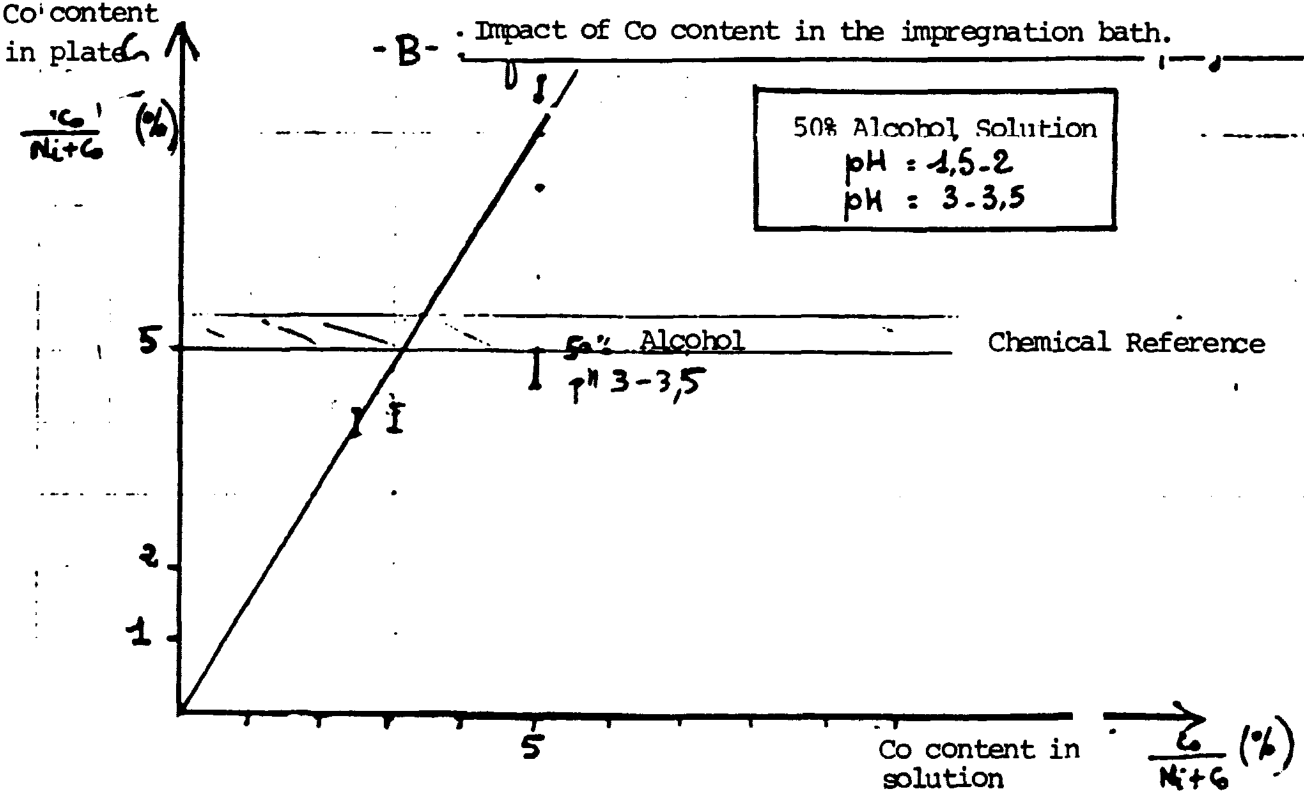


Figure 16

CAPACITY VARIATIONS OF ______ ELECTRODES DURING CYCLING

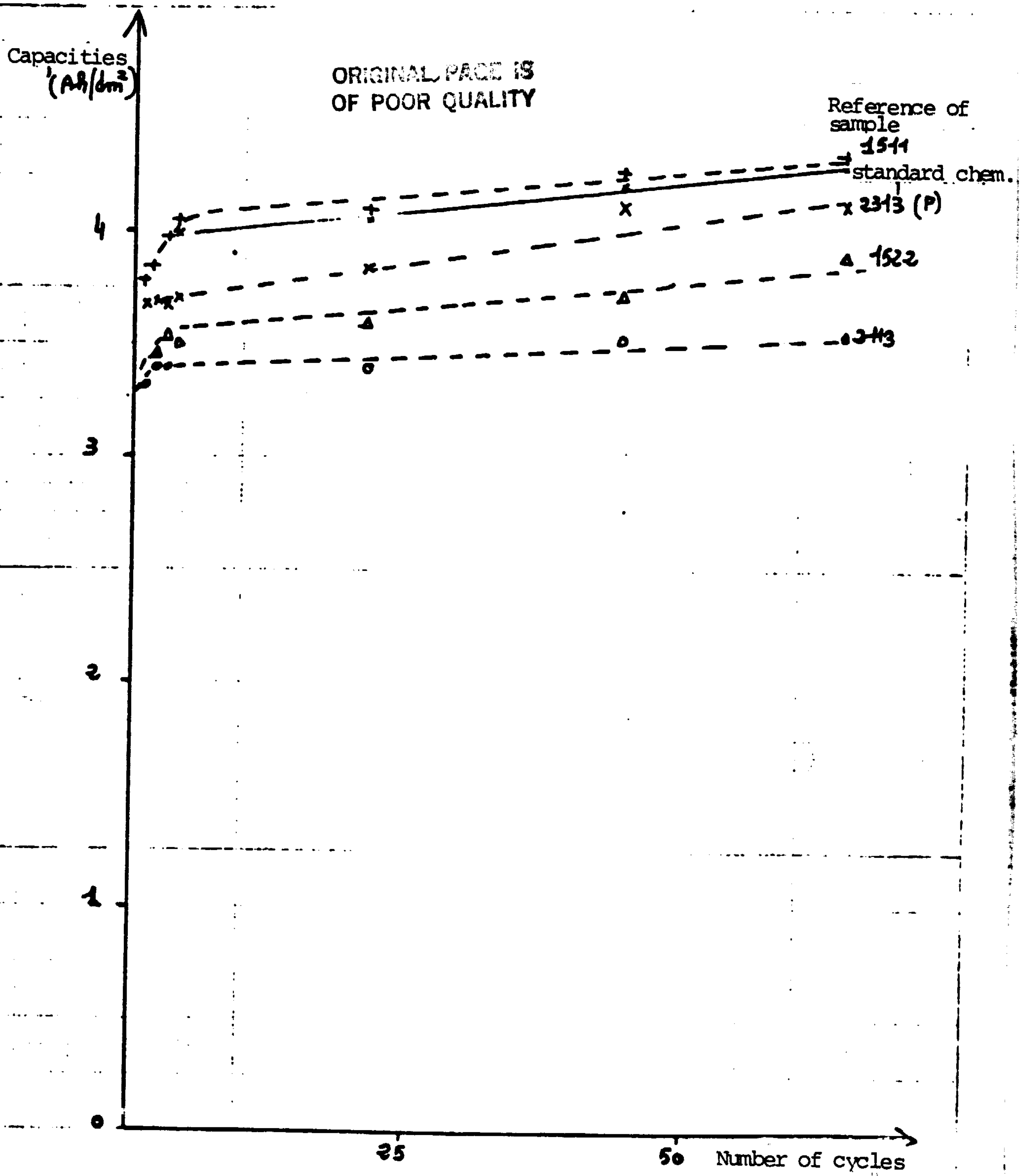
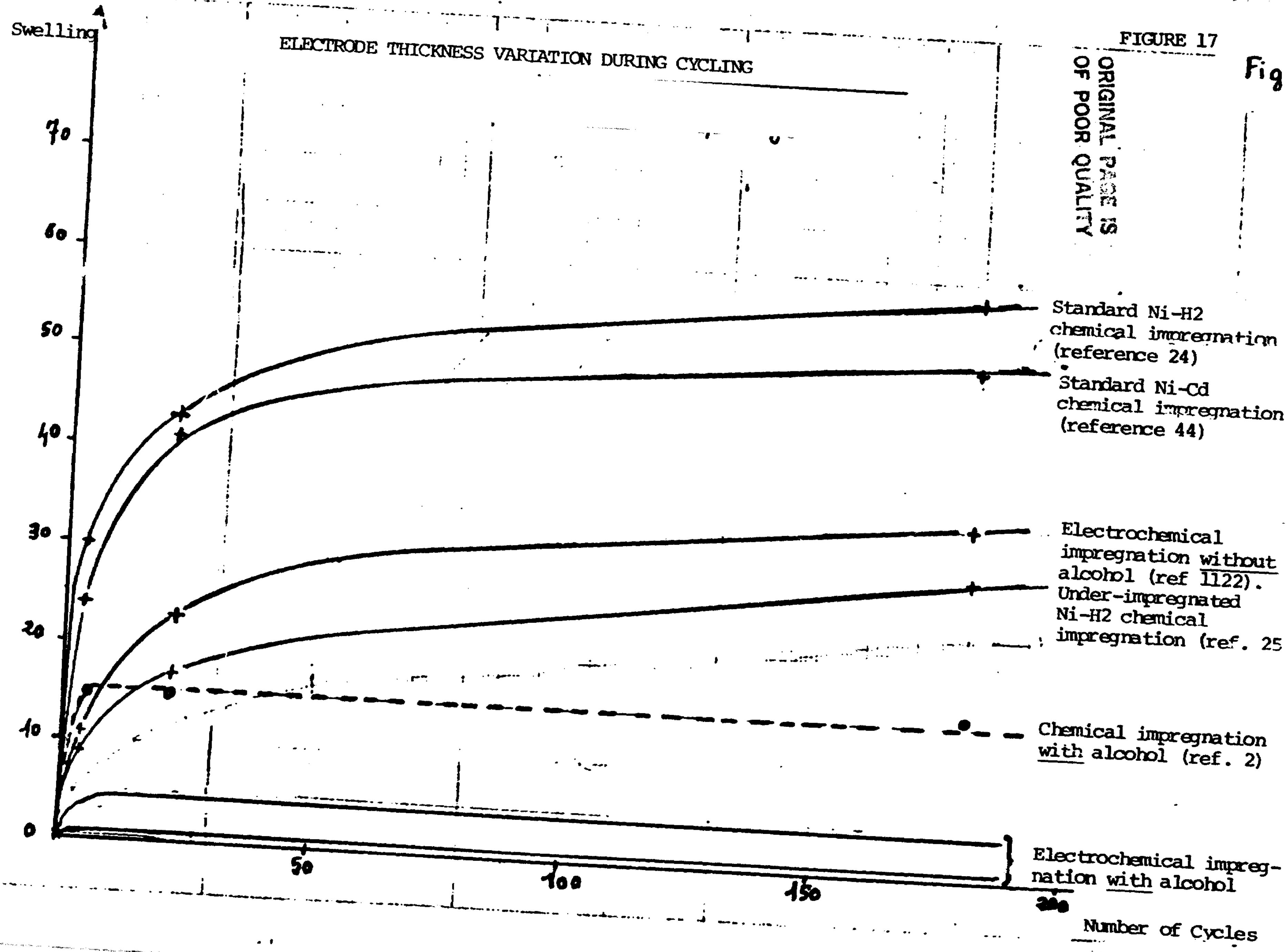


FIGURE 17

Figure 1



ELECTRODE THICKNESS VARIATION DURING CYCLING

ELECTRODE THICKNESS VARIATION
AS A FUNCTION OF THE FILLING PROCESS

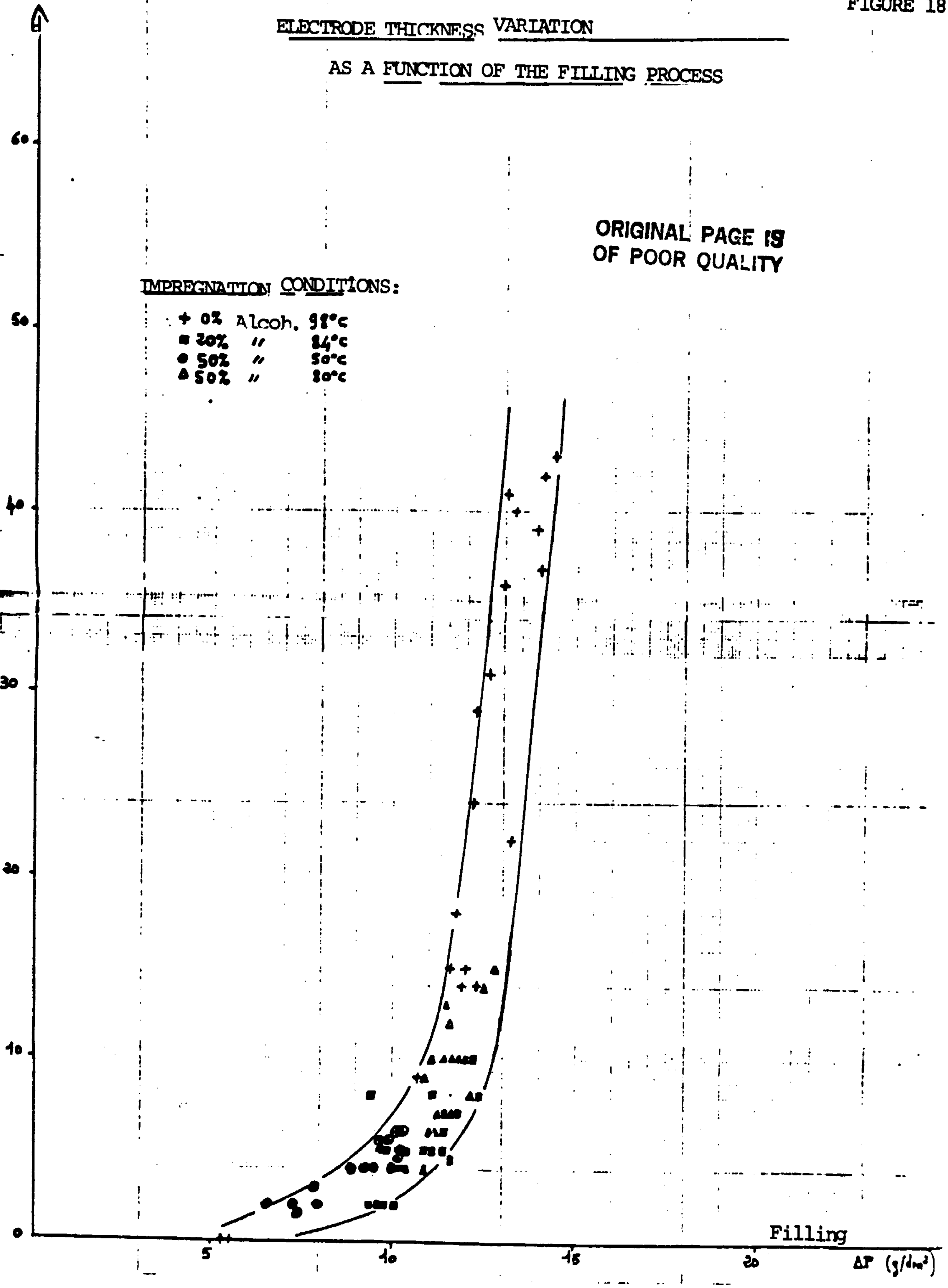


Figure 19

FIGURE 19

THICKNESS VARIATION OF ELECTRODES AS A FUNCTION OF THE ENERGY LEVEL

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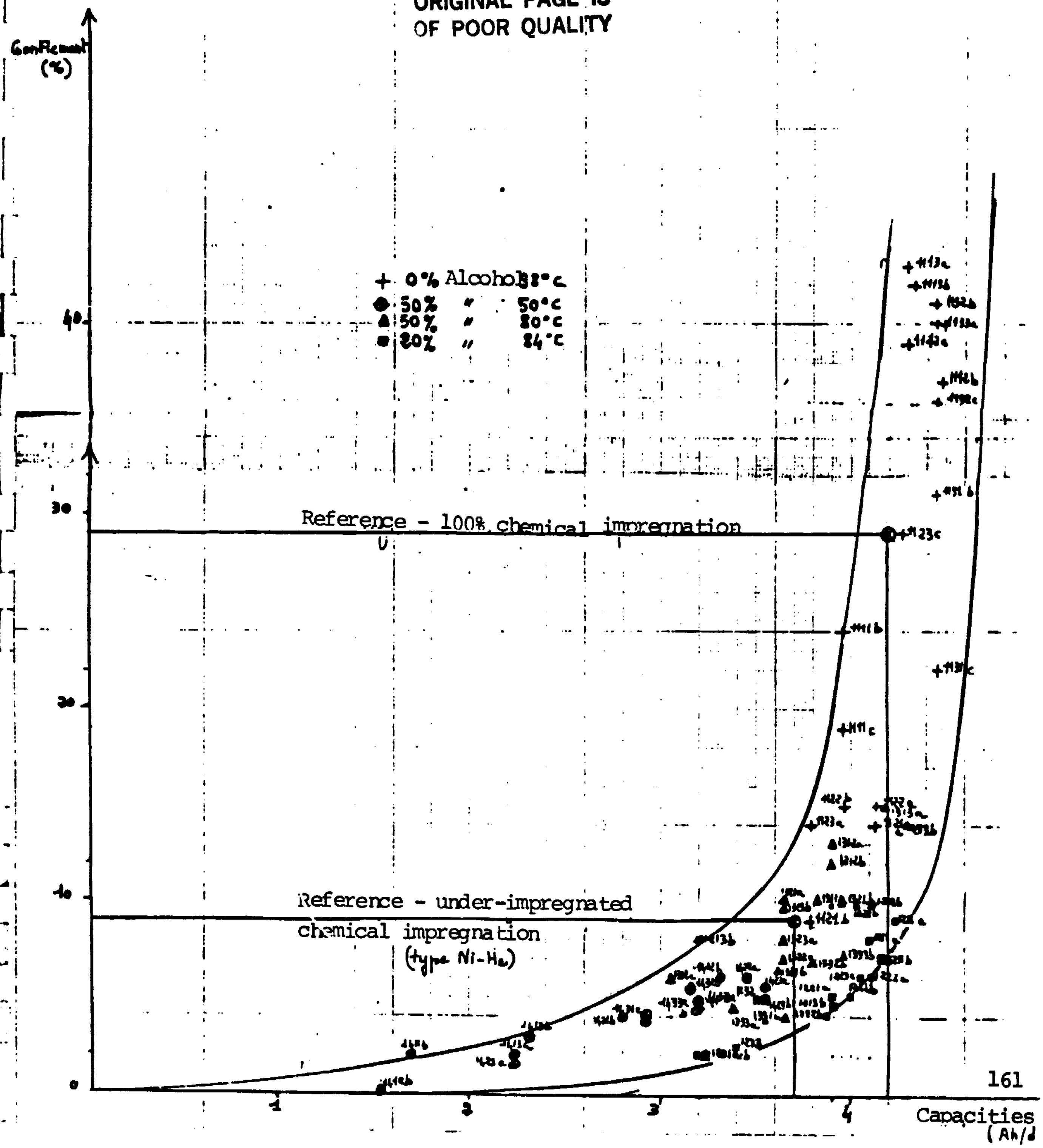
OF POOR QUALITY



ELECTRODE FILLING PROCESS (TYPE Ni-Cd)

Fig. 20.1.

FIG. 20.1

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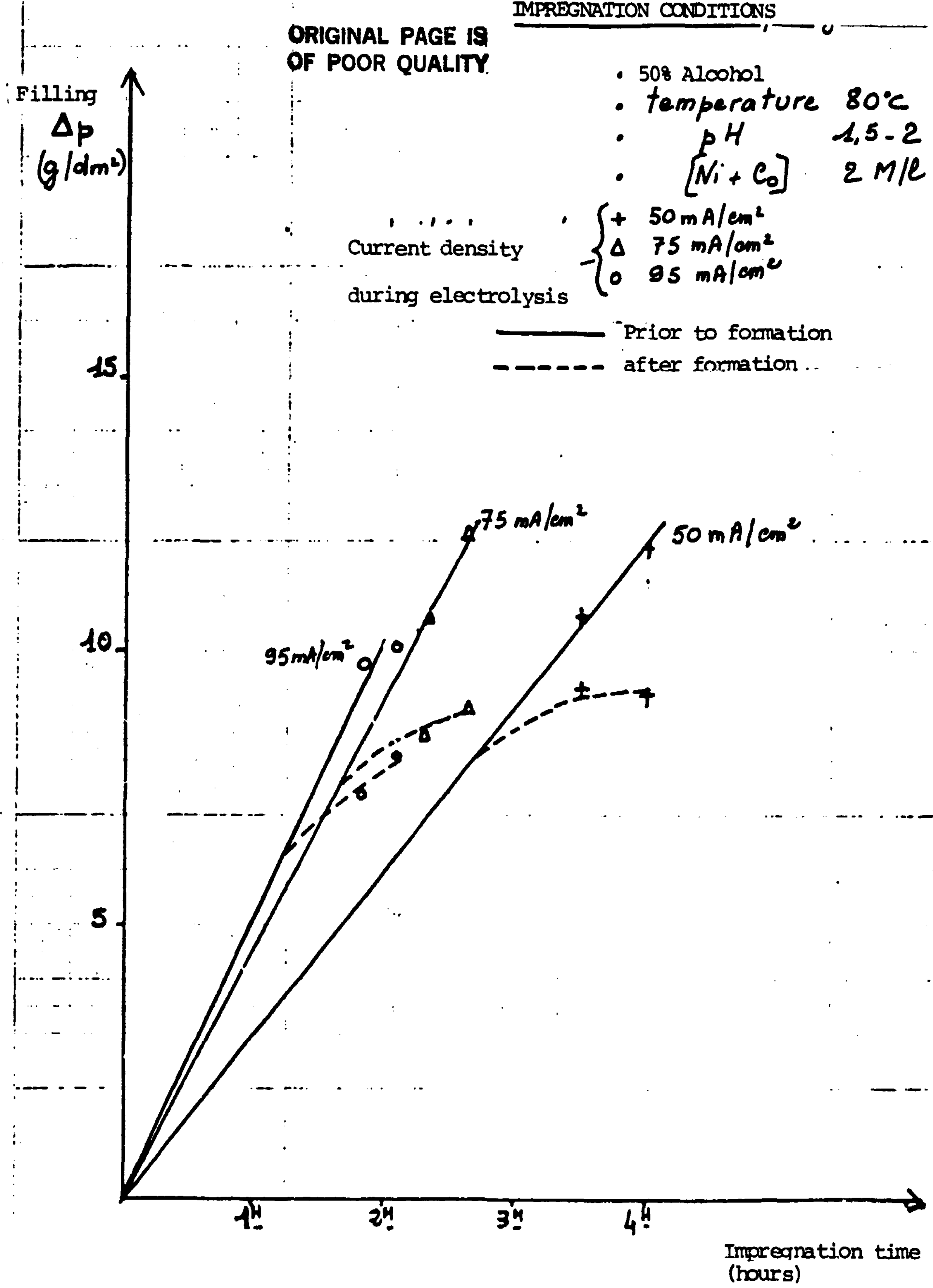


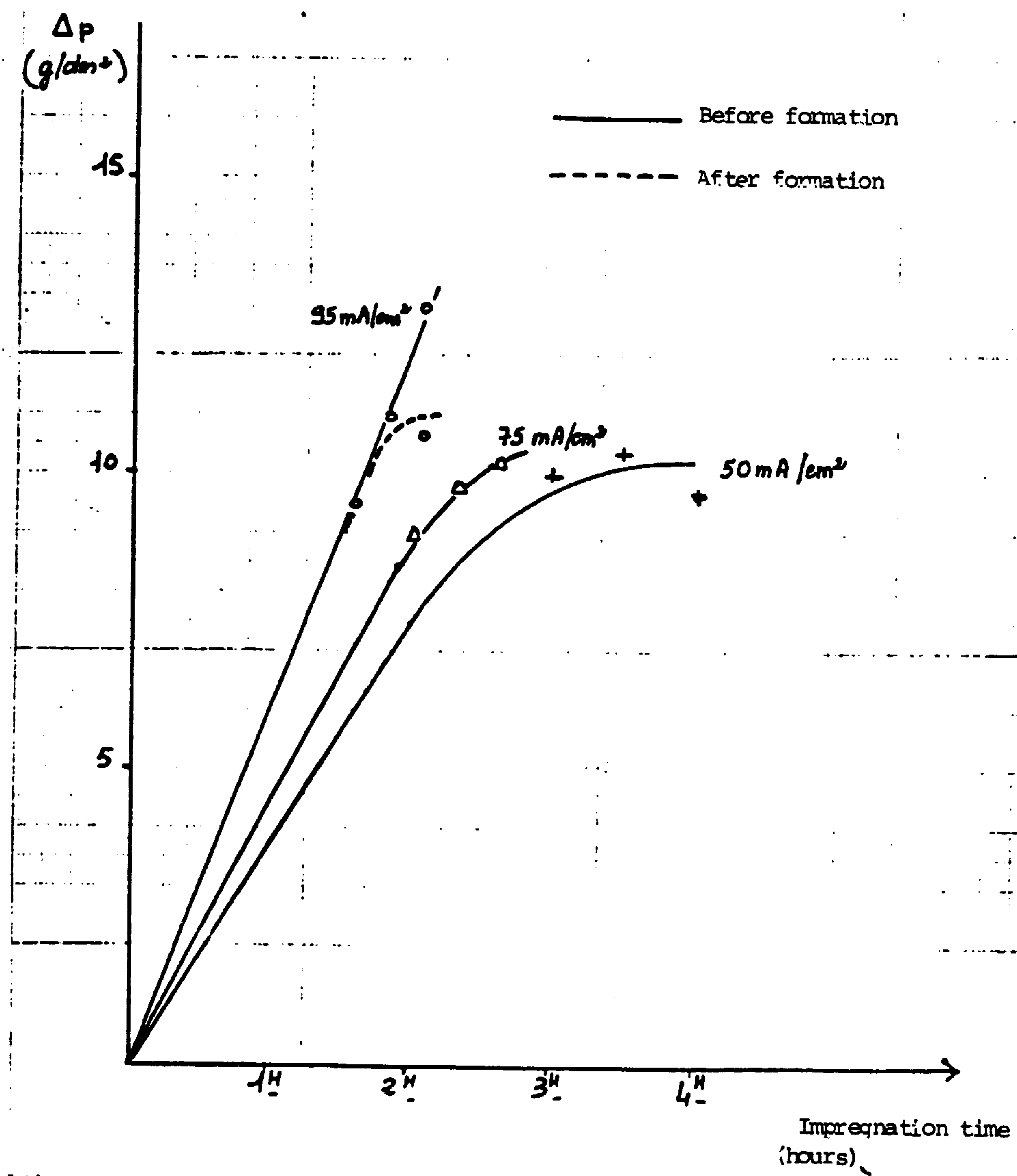
FILLING OF Ni-Cd TYPE ELECTRODES

Fig. 20.2.

FIG. 20.2

IMPREGNATION CONDITIONS

• Alcohol	50%
• temperature	80°C
• pH	1,5-2
• [Ni + Co]	3 M/l



CAPACITY OF Ni-Cd Type ELECTRODES (4th formation cycle)

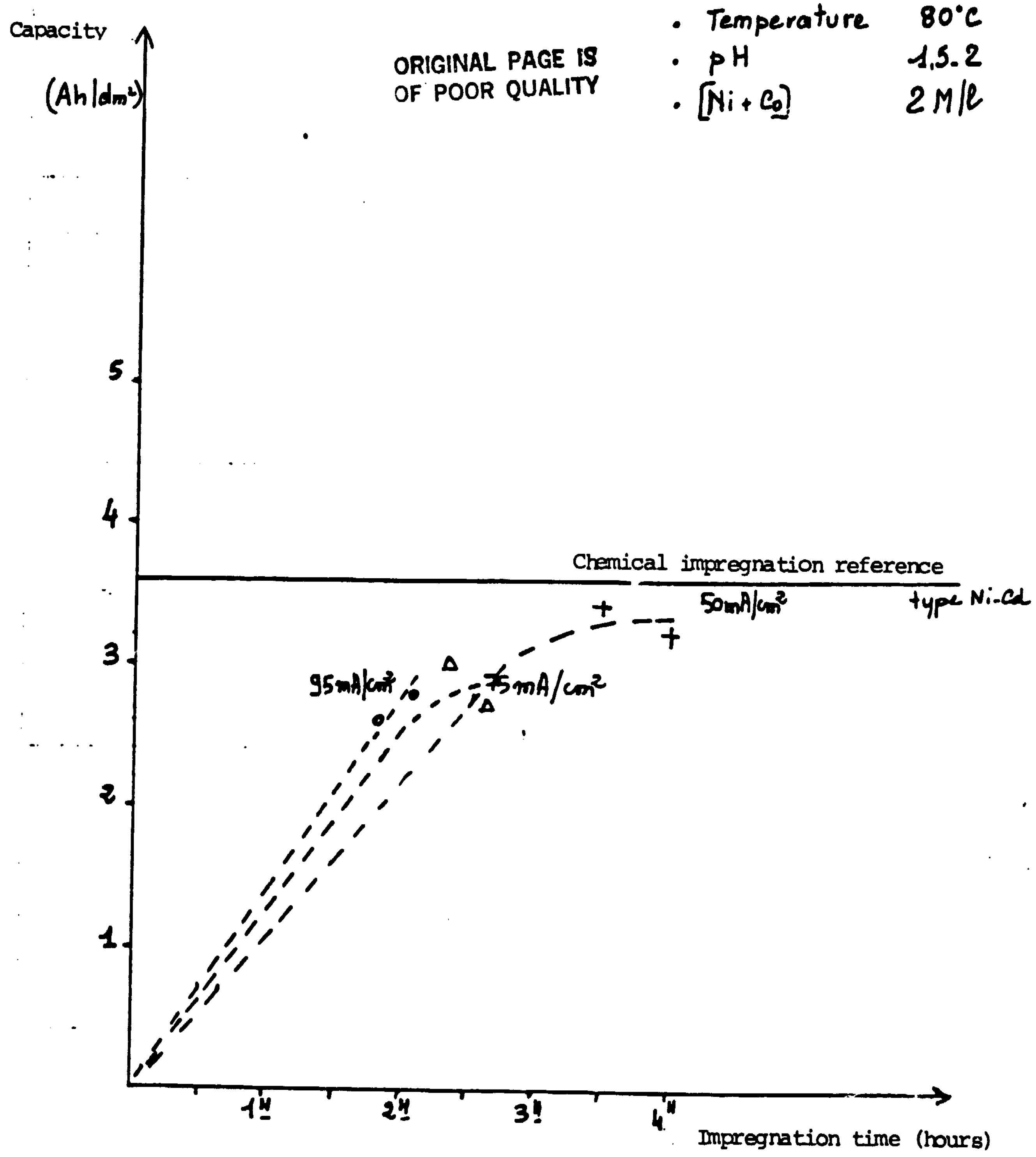
Fig 21.1.

Fig 21.1

(4th formation cycle)

IMPREGNATION CONDITIONS

Alcohol	50%
Temperature	80°C
pH	1,5.2
[Ni + Co]	2 M/l



CAPACITY OF Ni-Cd TYPE ELECTRODES

Fig. 21.2.

FIG 21.2

(4th formation cycle)

IMPREGNATION CONDITIONS

- 50%
- Temperature 80°C
- pH 1,5-2
- [Ni + Co] 3 M/l

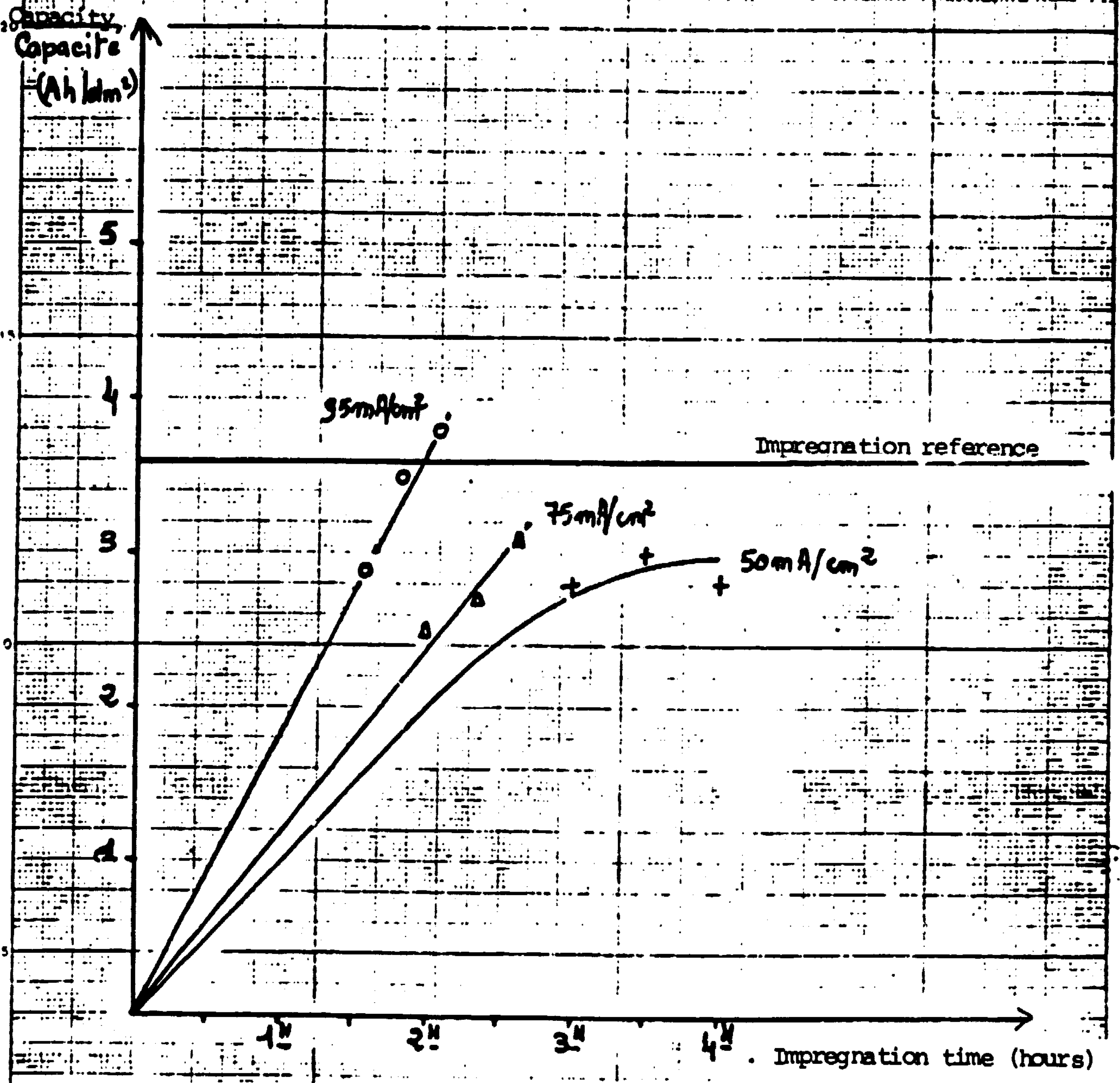


Fig 22.1.

FIG 22.1

CAPACITY VARIATION DURING FORMATION OF Ni-CD type ELECTRODES

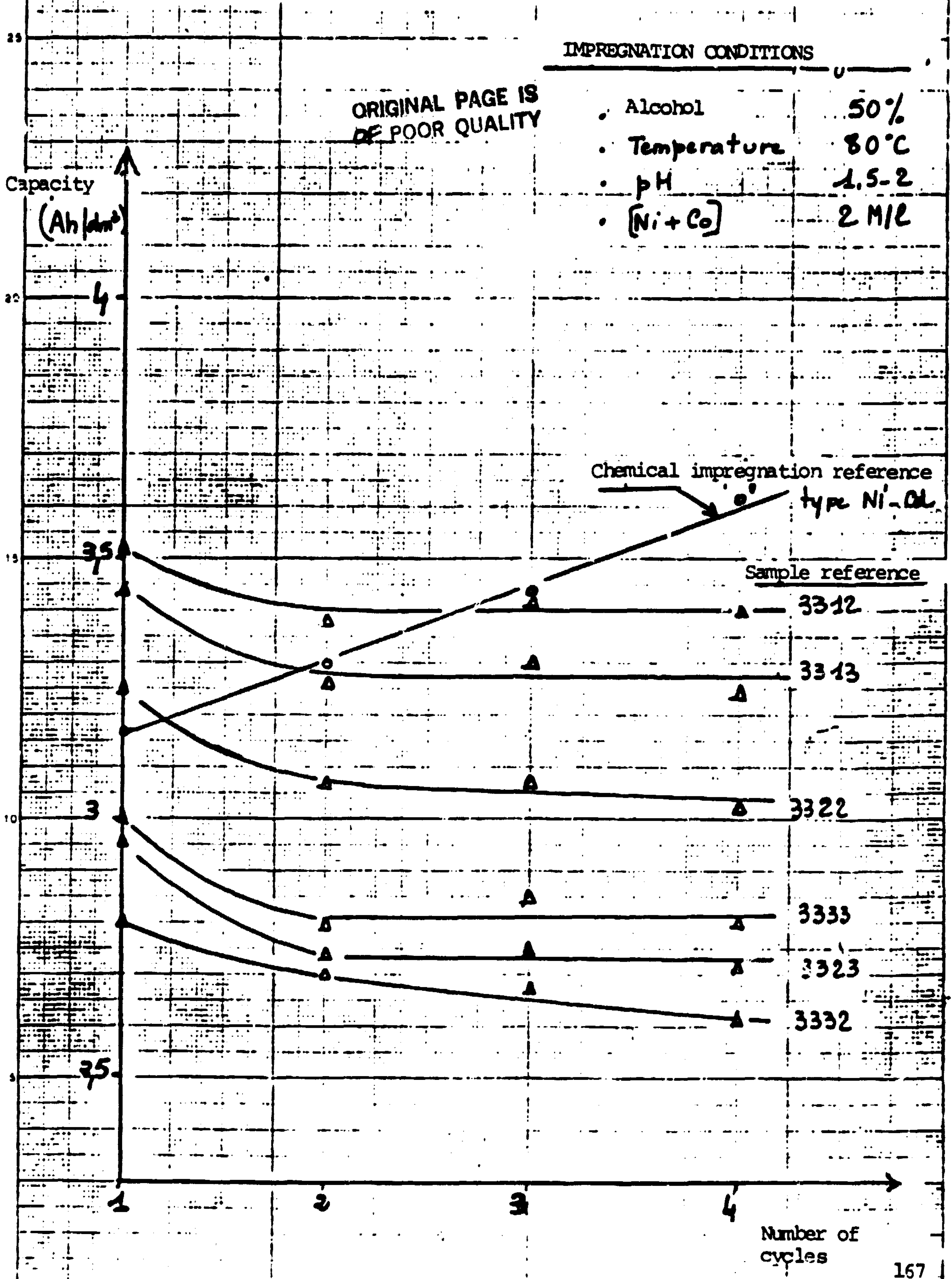


Fig 22.2.

Fig 22.2

CAPACITY VARIATION DURING FORMATION OF Ni-Cd TYPE ELECTRODES

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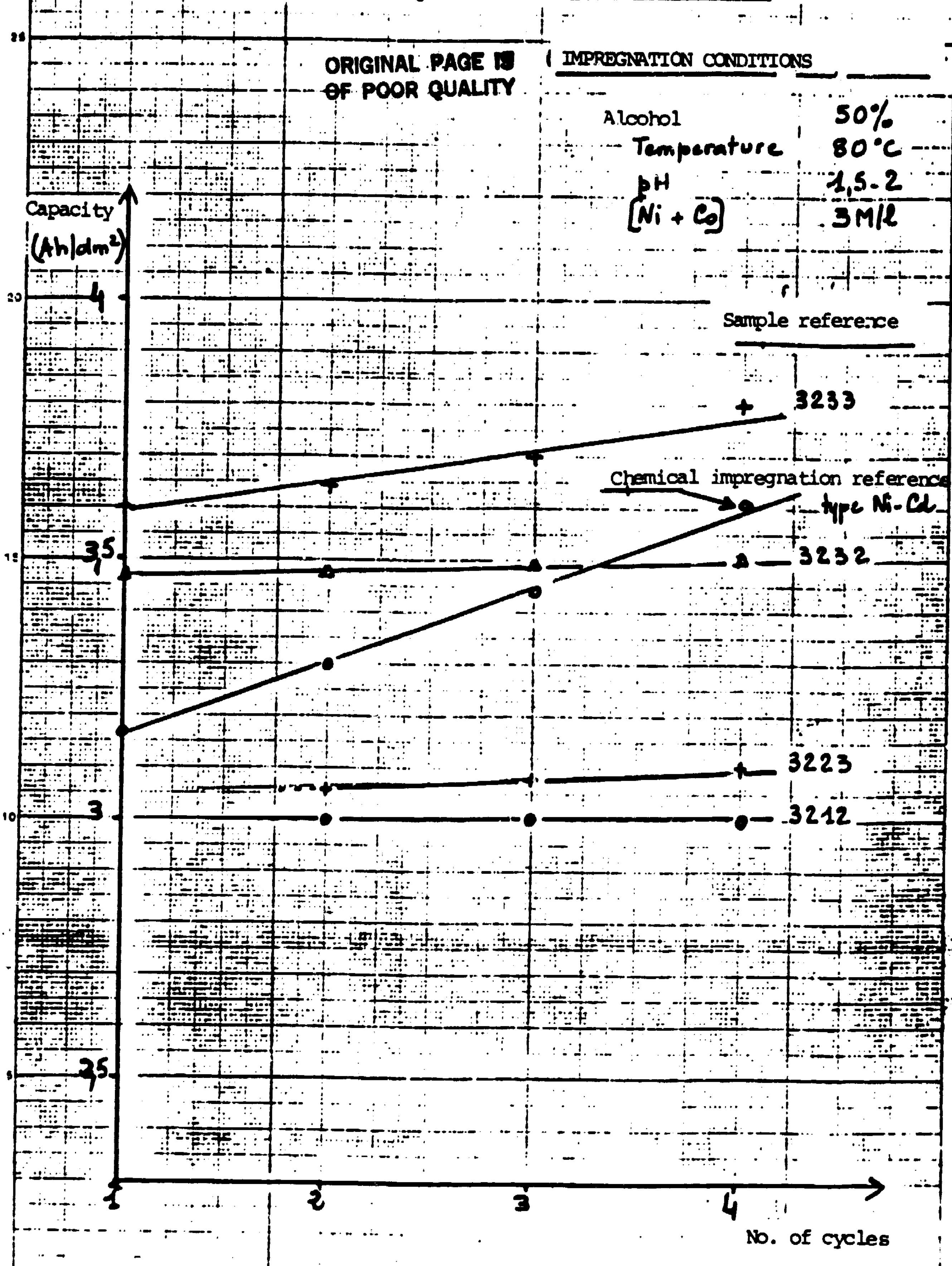


Fig 23

THICKNESS VARIATION OF Ni-Cd TYPE ELECTRODES AS A FUNCTION OF THE FILLING RATE

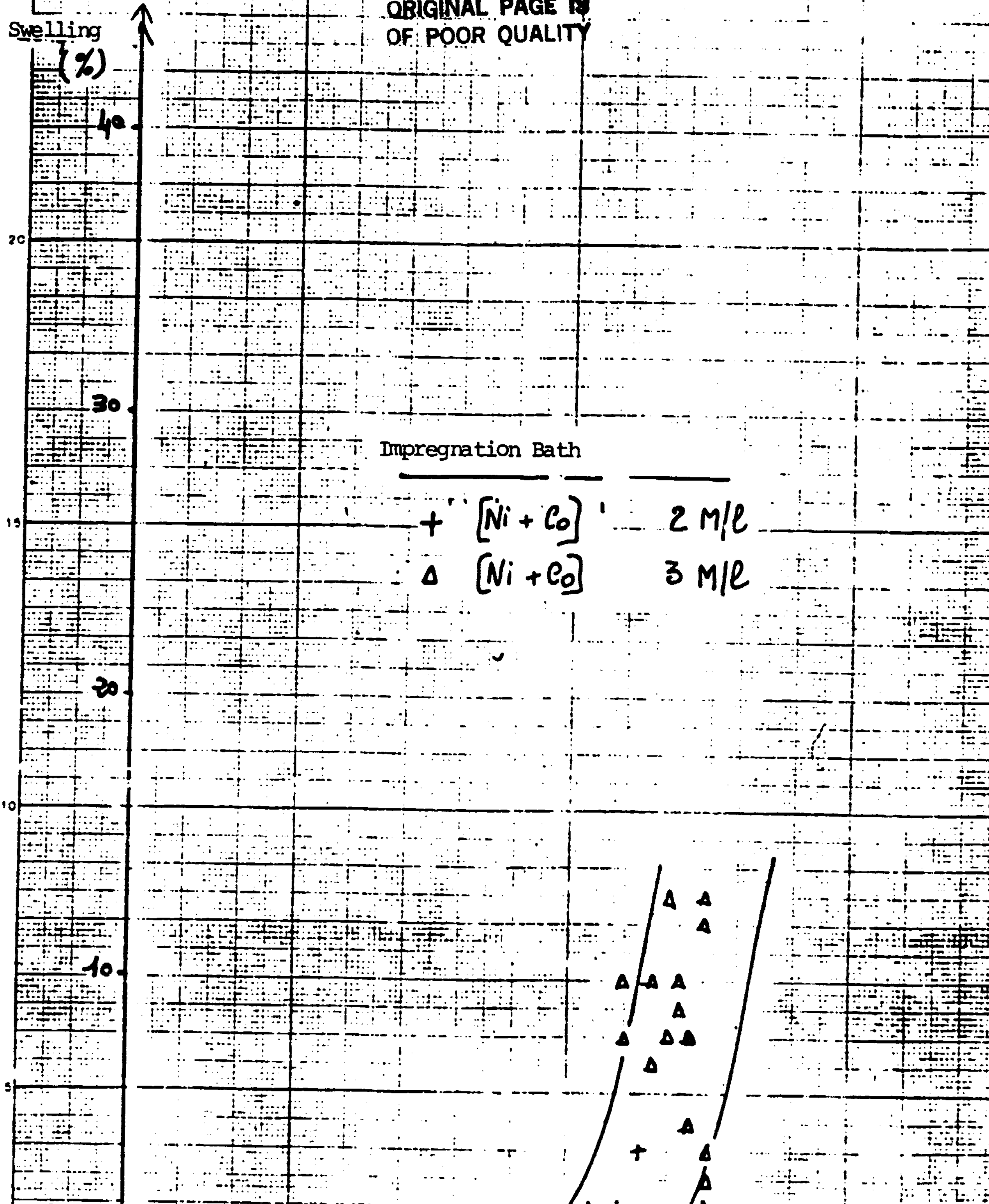


Fig. 24

CORROSION CONTROL OF THE SINTERED SUPPORT AS A FUNCTION OF THE IMMERSION TIME IN THE IMPREGNATION BATH PRIOR TO ELECTROLYSIS

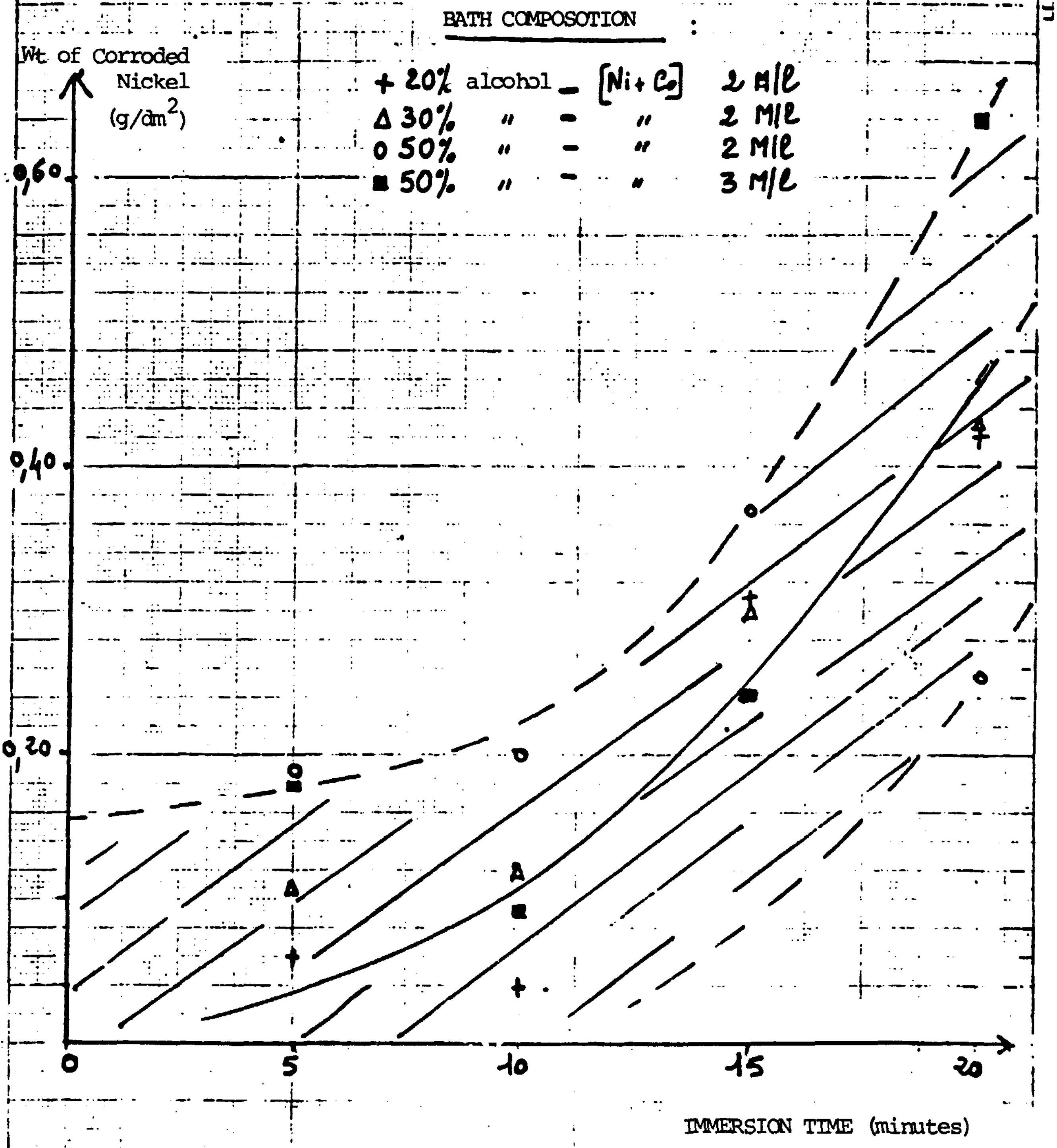


Fig. 25

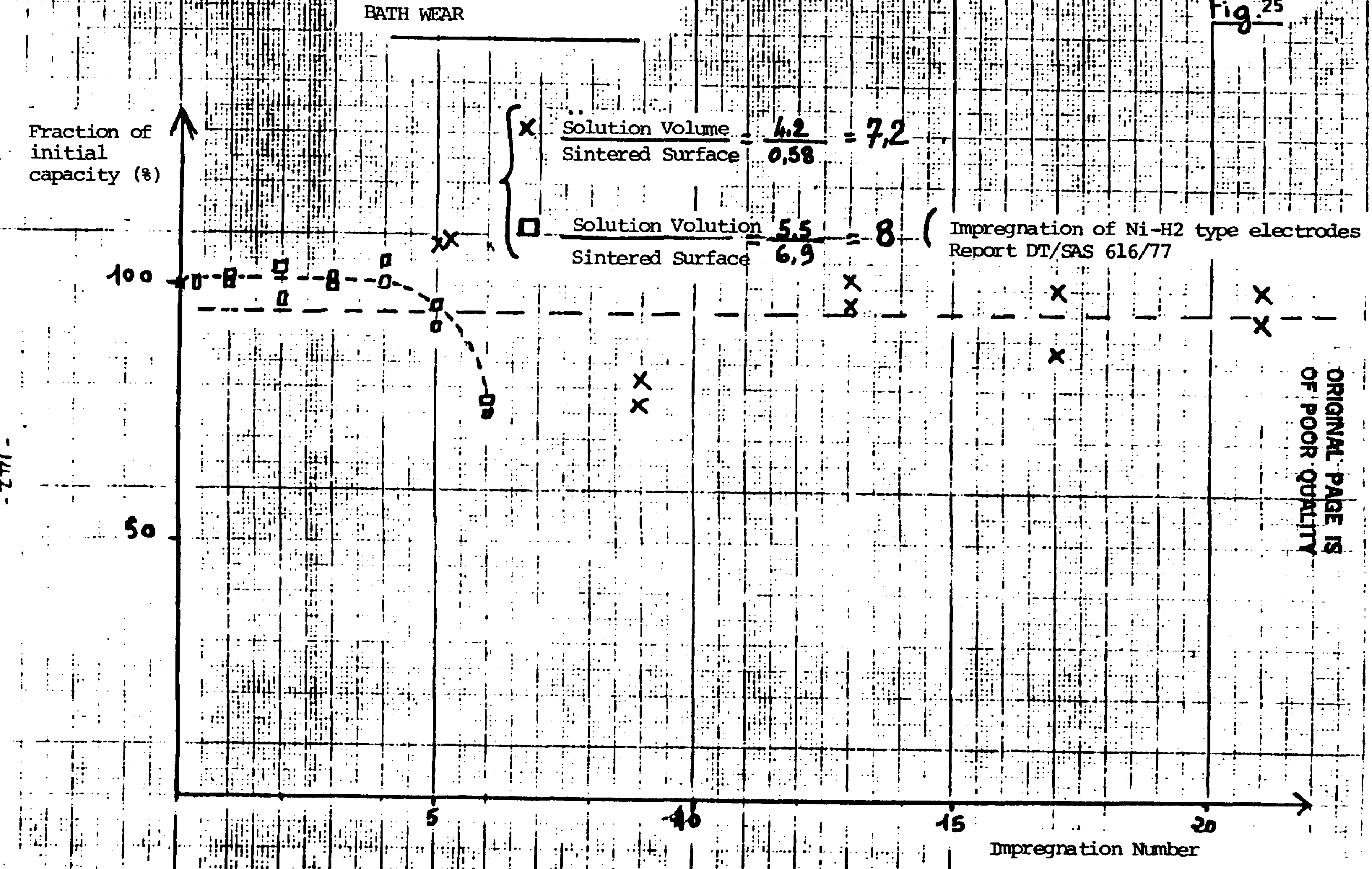

BATH WEAR
Fraction of initial capacity (%)
100
50
5
10
15
20
Impregnation Number
X Solution Volume / Sintered Surface = 4,2 / 0,58 = 7,2
□ Solution Volution / Sintered Surface = 5,5 / 6,9 = 8
Impregnation of Ni-H2 type electrodes
Report DT/SAS 616/77

Fig 28

VARIATION OF ELECTRODE THICKNESSES DURING CYCLING

IMPREGNATION : 0% ALCOHOL - 98°C - pH = 3-3,5

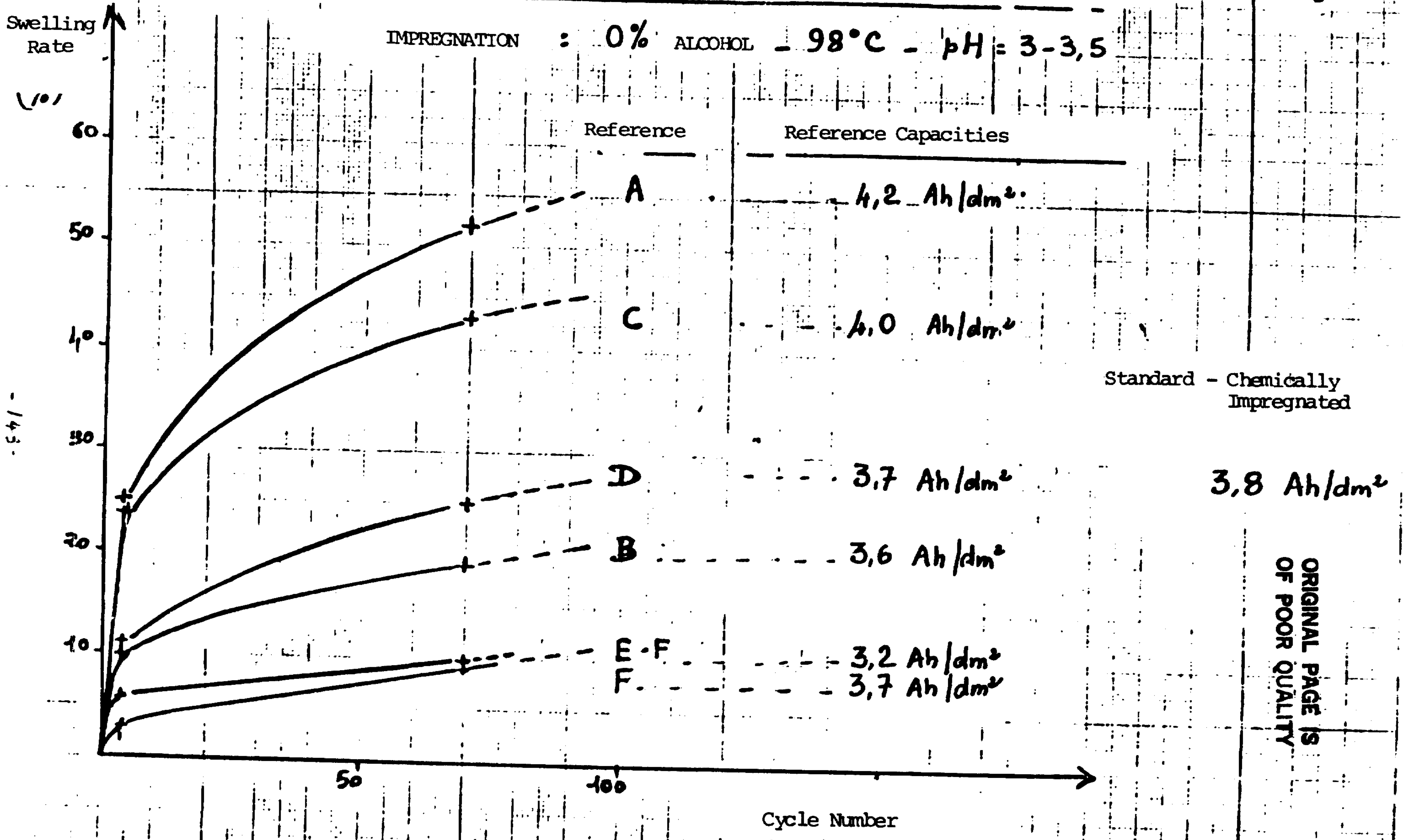
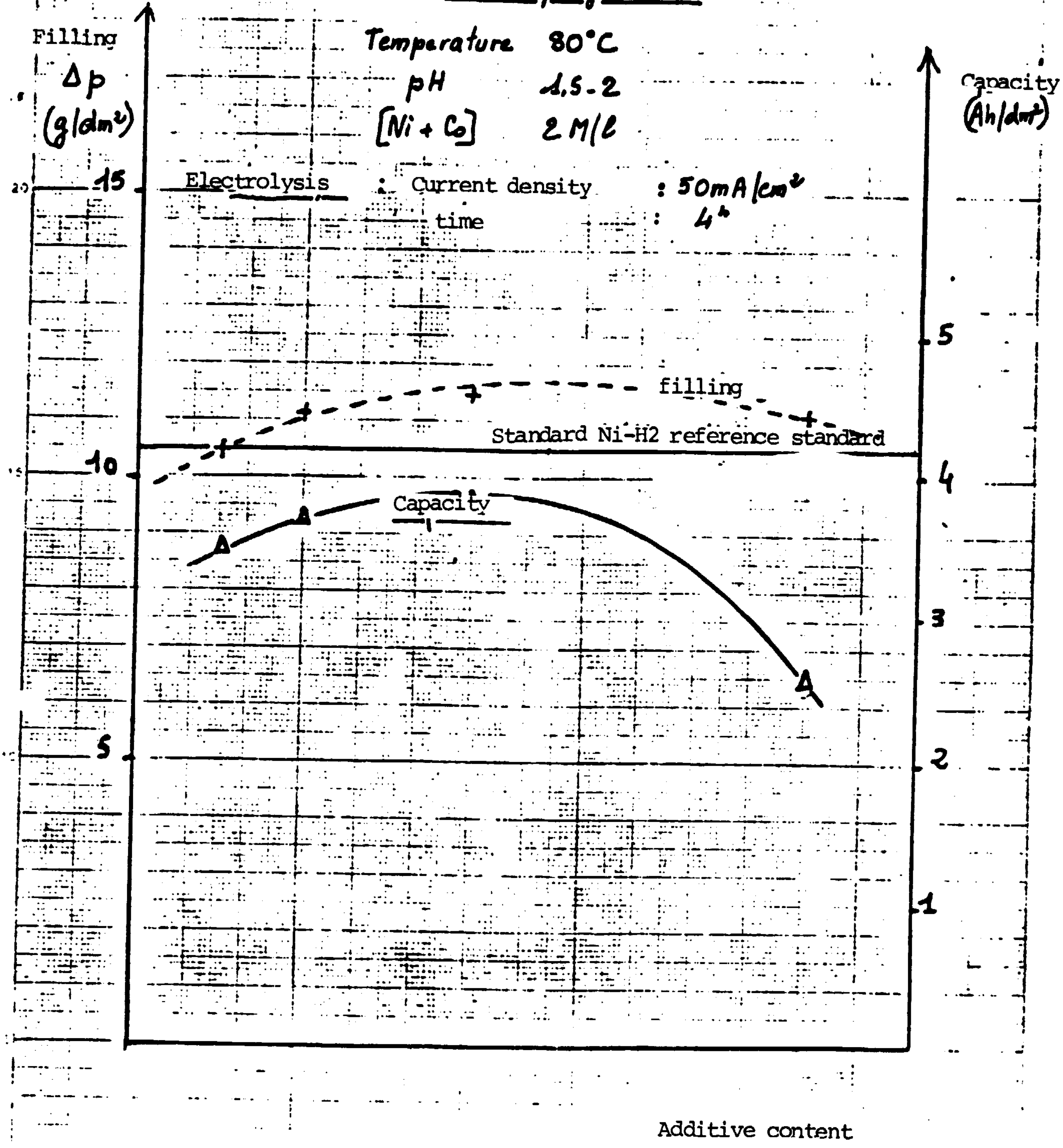


Fig. 27

FILLING AND CAPACITY (4th formation cycle)

CHARACTERISTICS OF ELECTRODES IMPREGNATED IN THE PRESENCE OF AN SURFACE-ACTIVE AGENT.

IMPREGNATION CONDITIONS



Temperature 80°C
pH 1.5-2
[Ni + Co] 2 M/l

Electrolysis : Current density : 50mA/cm²
time : 4h

APPENDICES

POSITIVE Ni-Cd ELECTRODES

(Type VR 6 FS)

Comparison of chemical and electrochemcial impregnation

Before cycling

After cycling

Impregnation by
chemical process
(6 cycles)

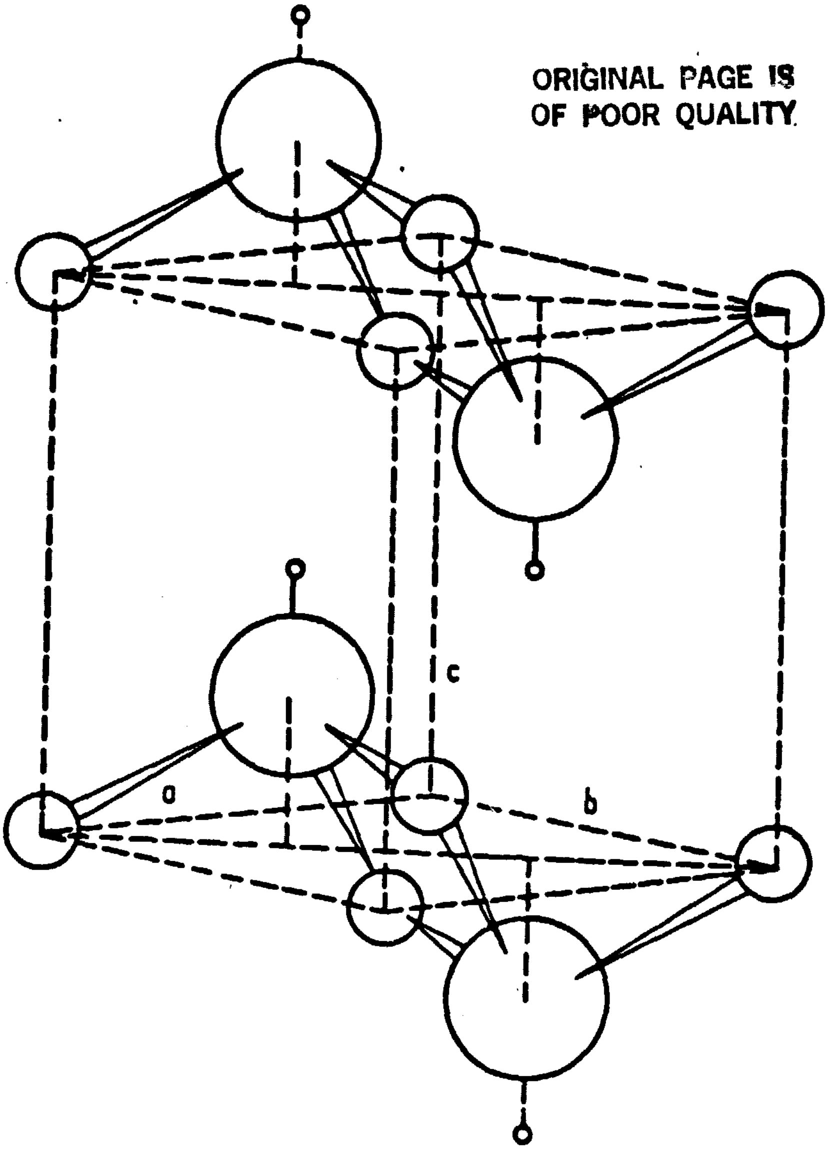
VR 6 F

Impregnation by
electrochemical
process

6 : 25

Before cycling

after cycling



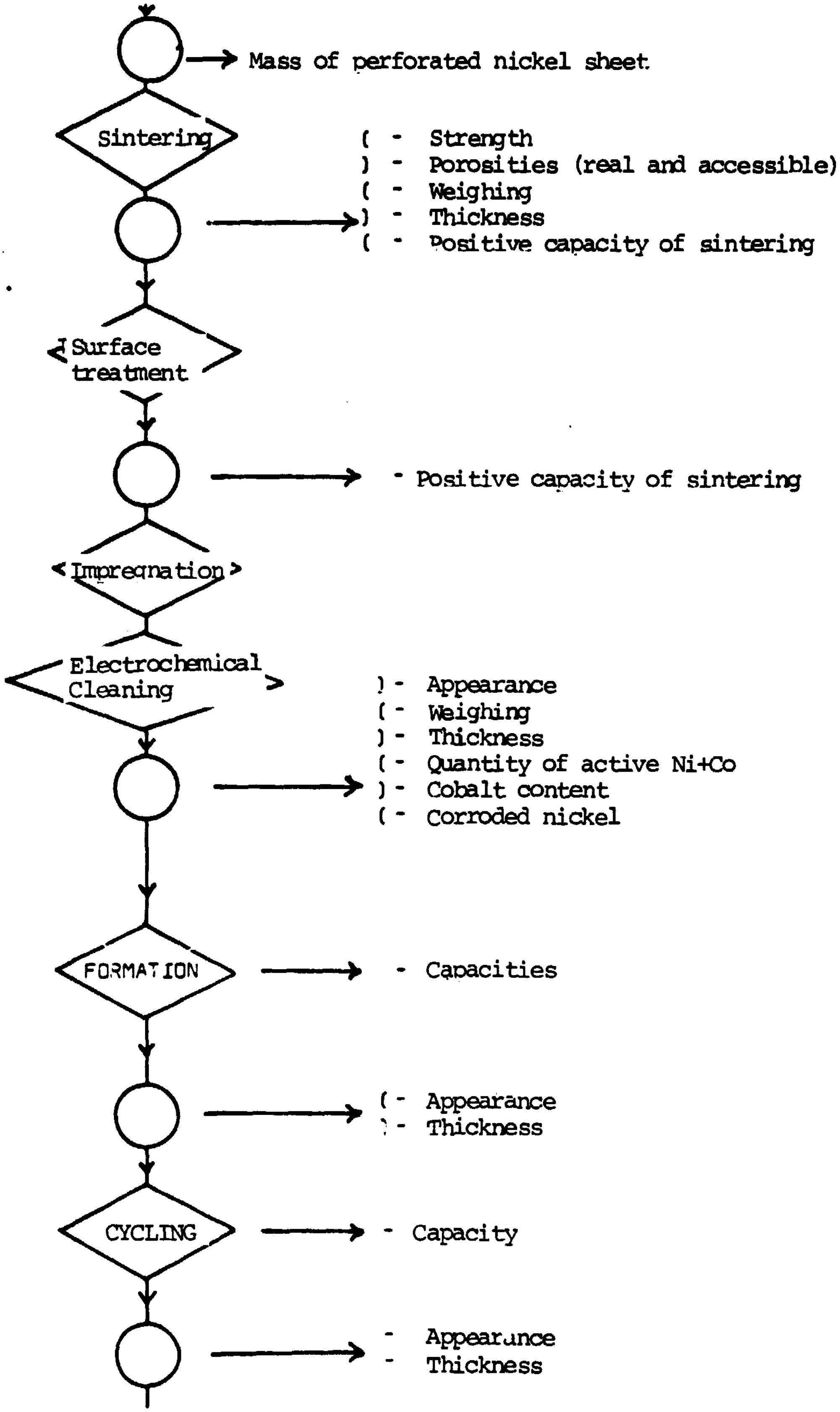
UNITARY PATTERN OF Ni$(OH)_2$ SHOWING THE HEXAGONAL SYMMETRY P$\bar{3}$m1.

◯ OXYGEN

○ NICKEL

∘ HYDROGEN

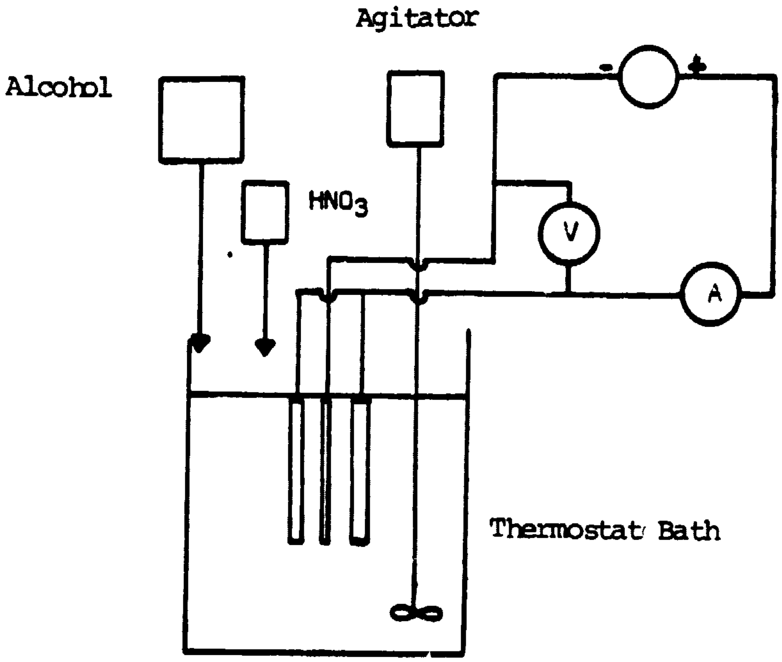
FABRICATION AND CONTROL DIAGRAM



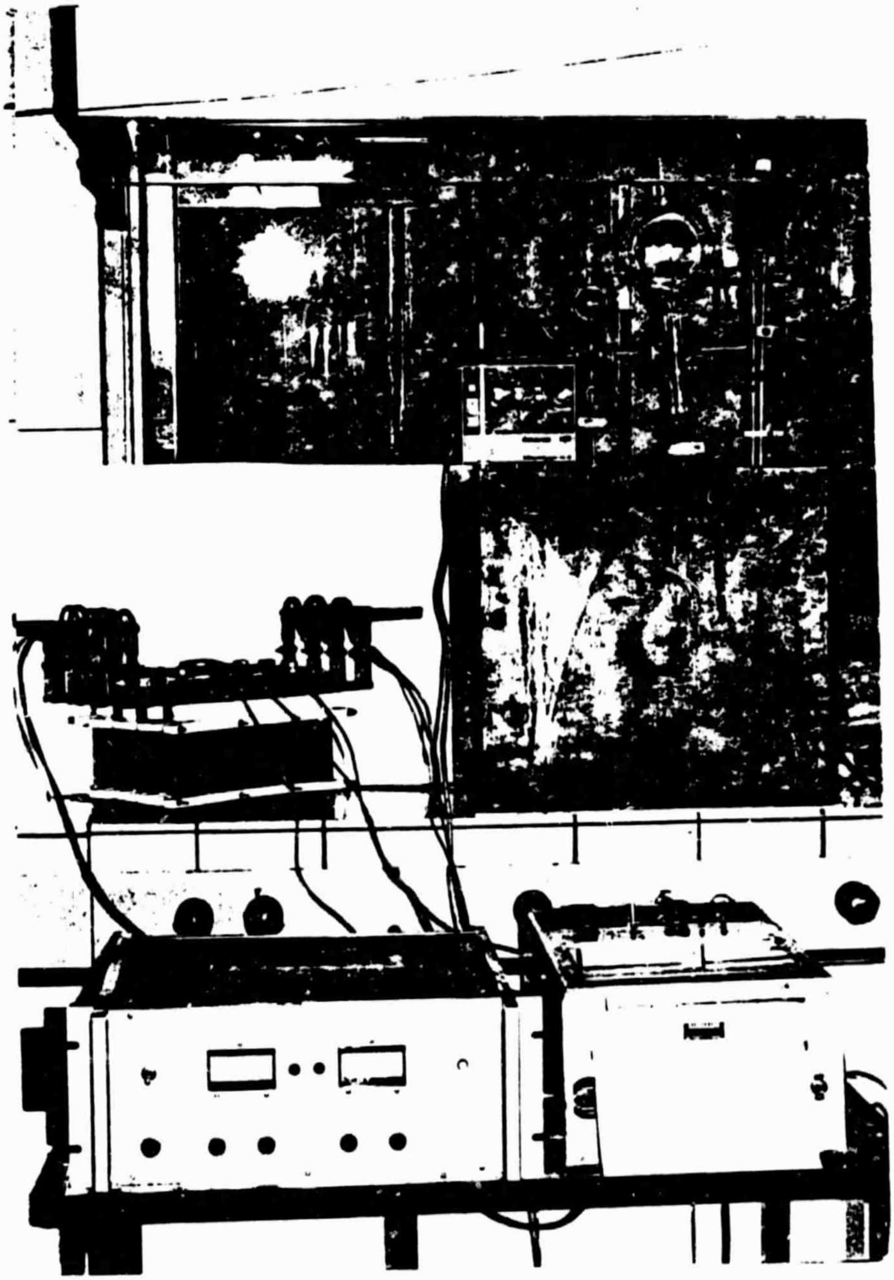
◇ OPERATION

○ CONTROLES

DIAGRAM OF THE ELECTROLYSIS CIRCUIT

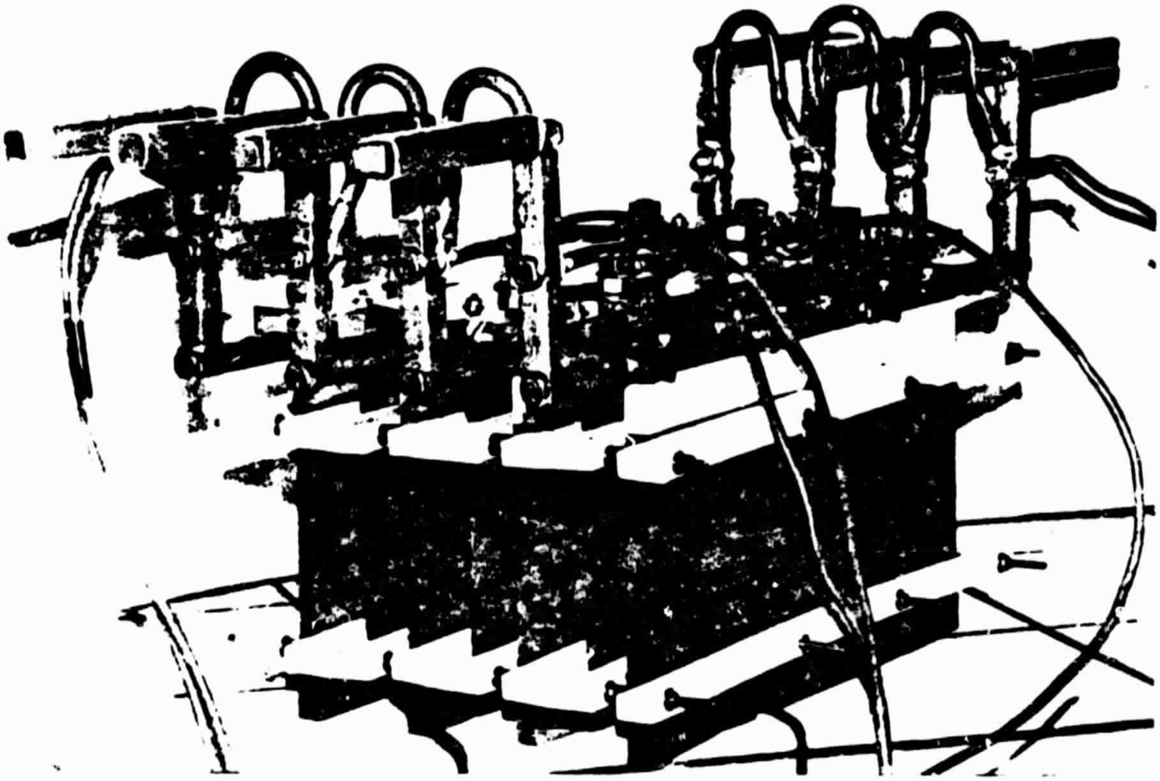


PHOTOGRAPHY OF THE STUDY AND ELECTROCHEMICAL IMPREGNATION INSTALLATION



Appendix C

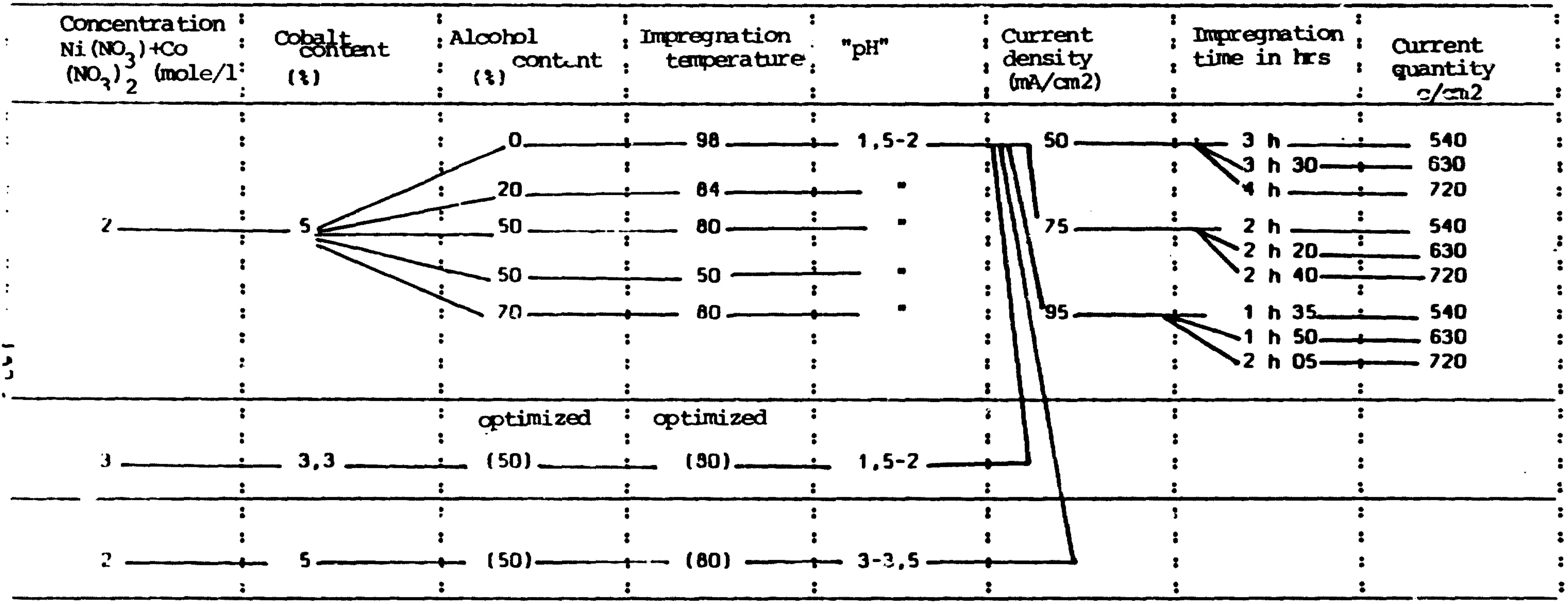
PHOTOGRAPHY OF THE ELECTRODE SUPPORT



ELECTROCHEMICAL IMPREGNATION

INFLUENCE OF THE IMPREGNATION CONDITIONS

Ni-H2 type sintering - Reference no. 21
after reduction treatment



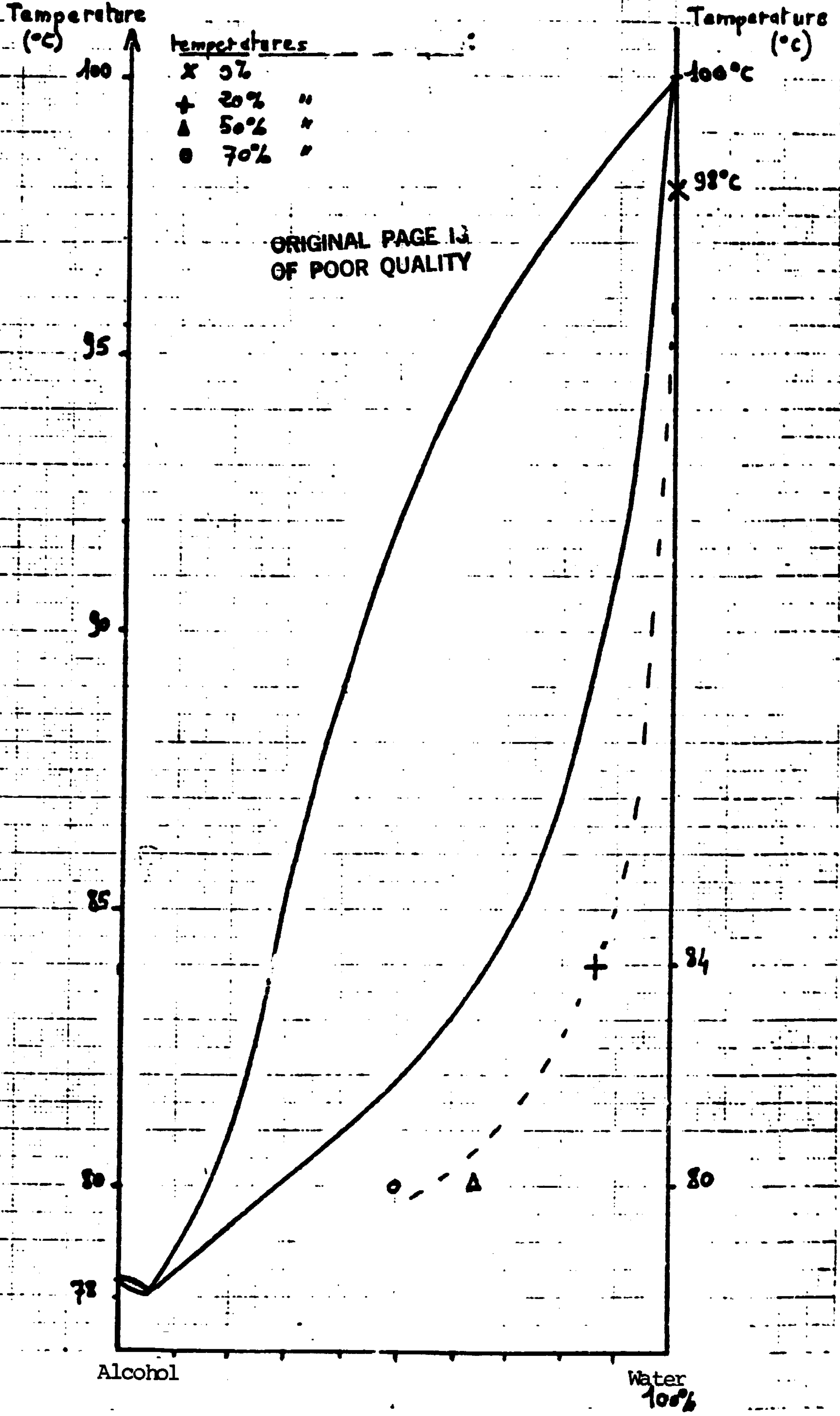
Concentration $Ni(NO_3)+Co(NO_3)_2$ (mole/l	Cobalt content (%)	Alcohol content (%)	Impregnation temperature	"pH"	Current density (mA/cm2)	Impregnation time in hrs	Current quantity c/cm2
		0	98	1,5-2	50	3 h	540
						3 h 30	630
		20	84	"		4 h	720
2	5	50	80	"	75	2 h	540
						2 h 20	630
		50	50	"		2 h 40	720
		70	80	"	95	1 h 35	540
						1 h 50	630
						2 h 05	720
		optimized	optimized				
3	3,3	(50)	(80)	1,5-2			
2	5	(50)	(80)	3-3,5			

ELECTROCHEMICAL IMPREGNATION

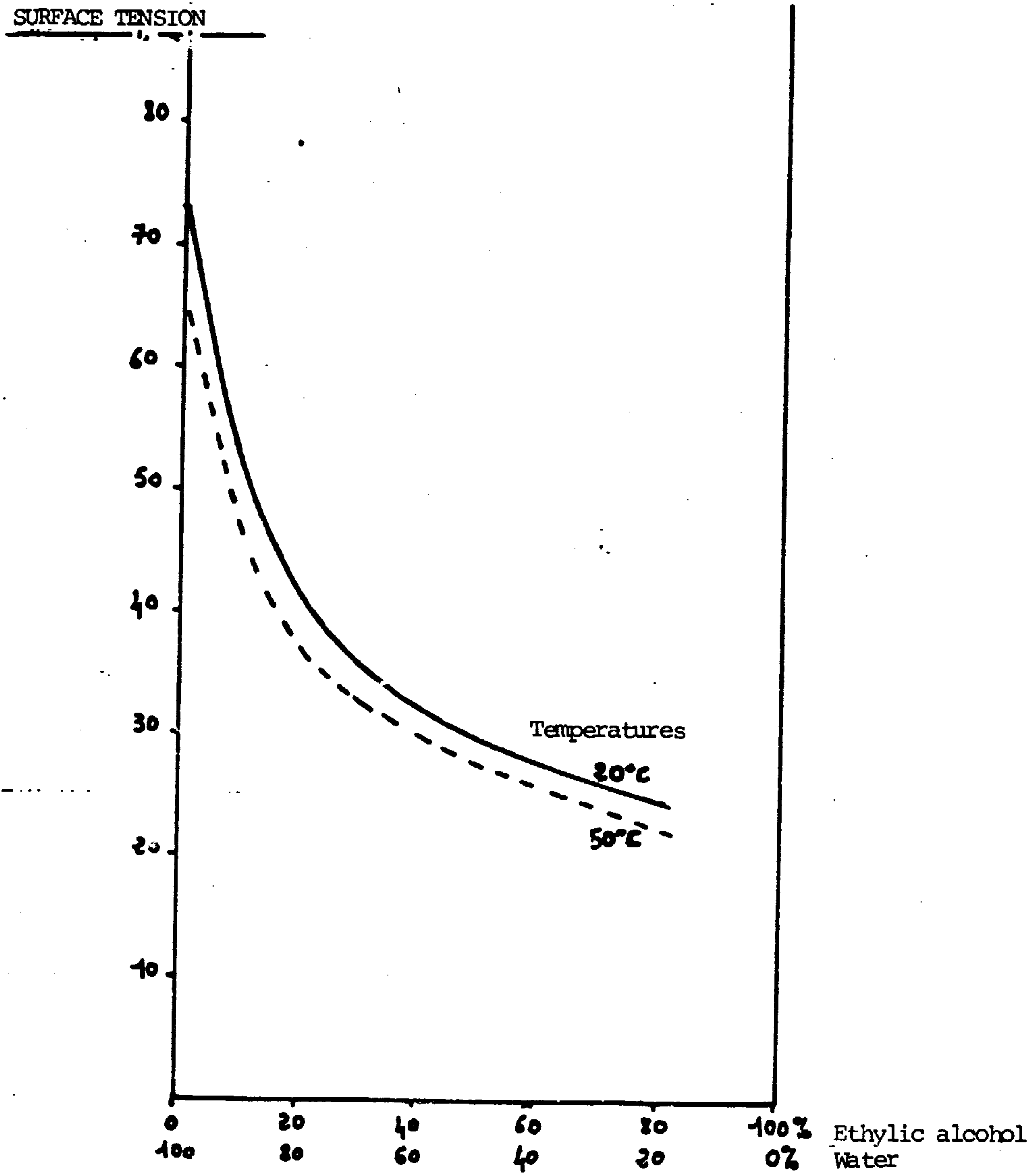
Type Ni-Cd - Sintering thickness: 0,78 mm ± 0,02
- Porosity : 85 ± 1 %

Impregnation Bath							
pH	Alcohol content	temperature	Ni+Co concentration (Mole/1)	Co content (%)	Current density (mA/cm2)	Impregnation time (hours)	Current quanity (c/cm2)
,5 - 2	50 %	80° C	2	5	50	3h 30'	630
						4h	720
	50 %	80° C	2	5	75	2h 20'	630
						2h 40'	720
	50 %	80° C	2	5	95	1h 50'	630
						2h 05'	720
,5 - 2	50 %	80° C	3	3,3	50	3h	540
						3h 30'	630
						4h	720
	50 %	80° C	3	3,3	75	2h	540
						2h 20'	630
						2h 40'	720
	50 %	80° C	3	3,3	95	1h 35'	540
						1h 50'	630
						2h 05'	720

DISTILLATION CURVE OF A WATER-ALCOHOL MIXTURE



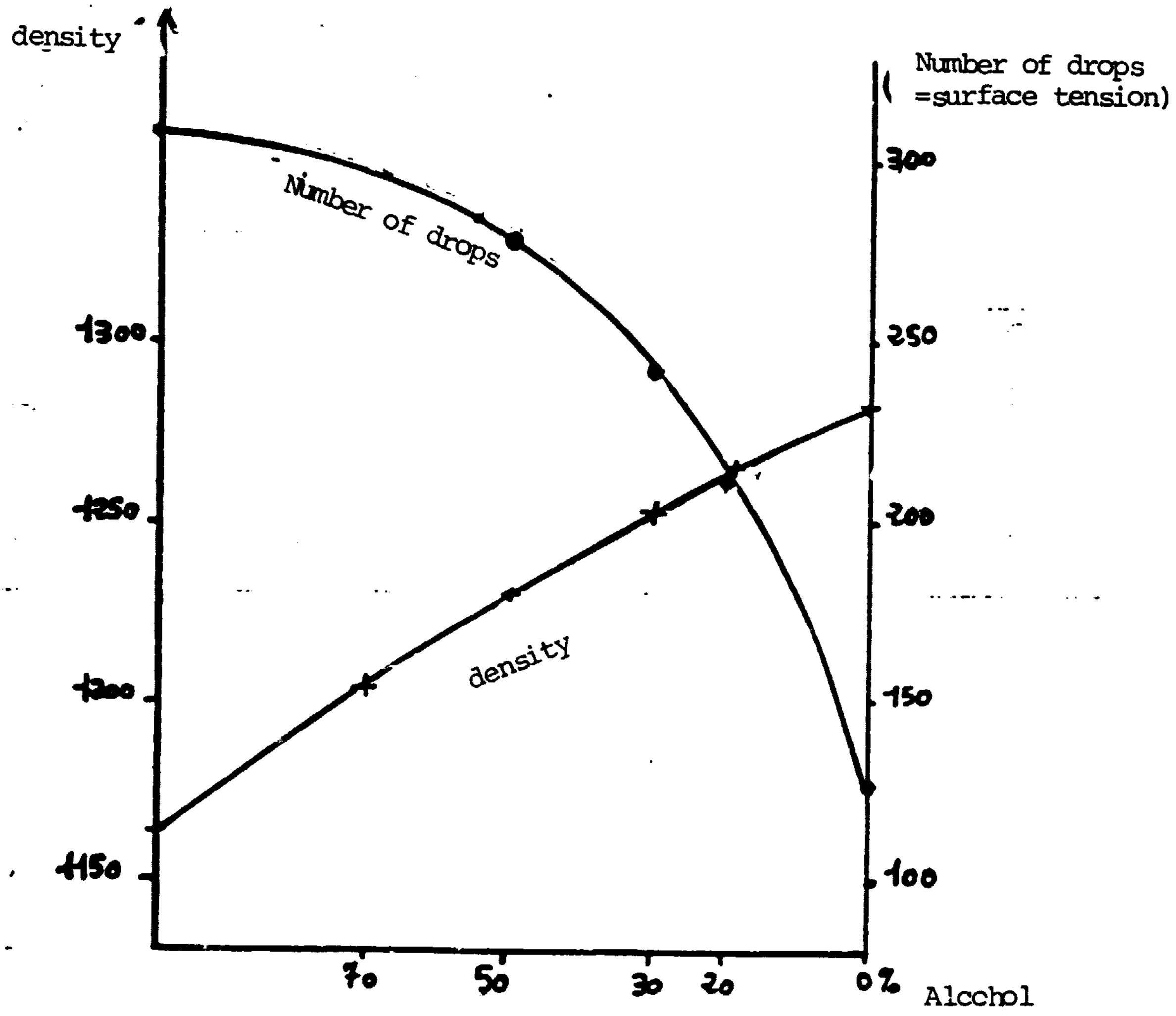
SURFACE TENSION OF A WATER-ALCOHOL MIXTURE



Control of the Alcohol Content in the Impregnation Bath

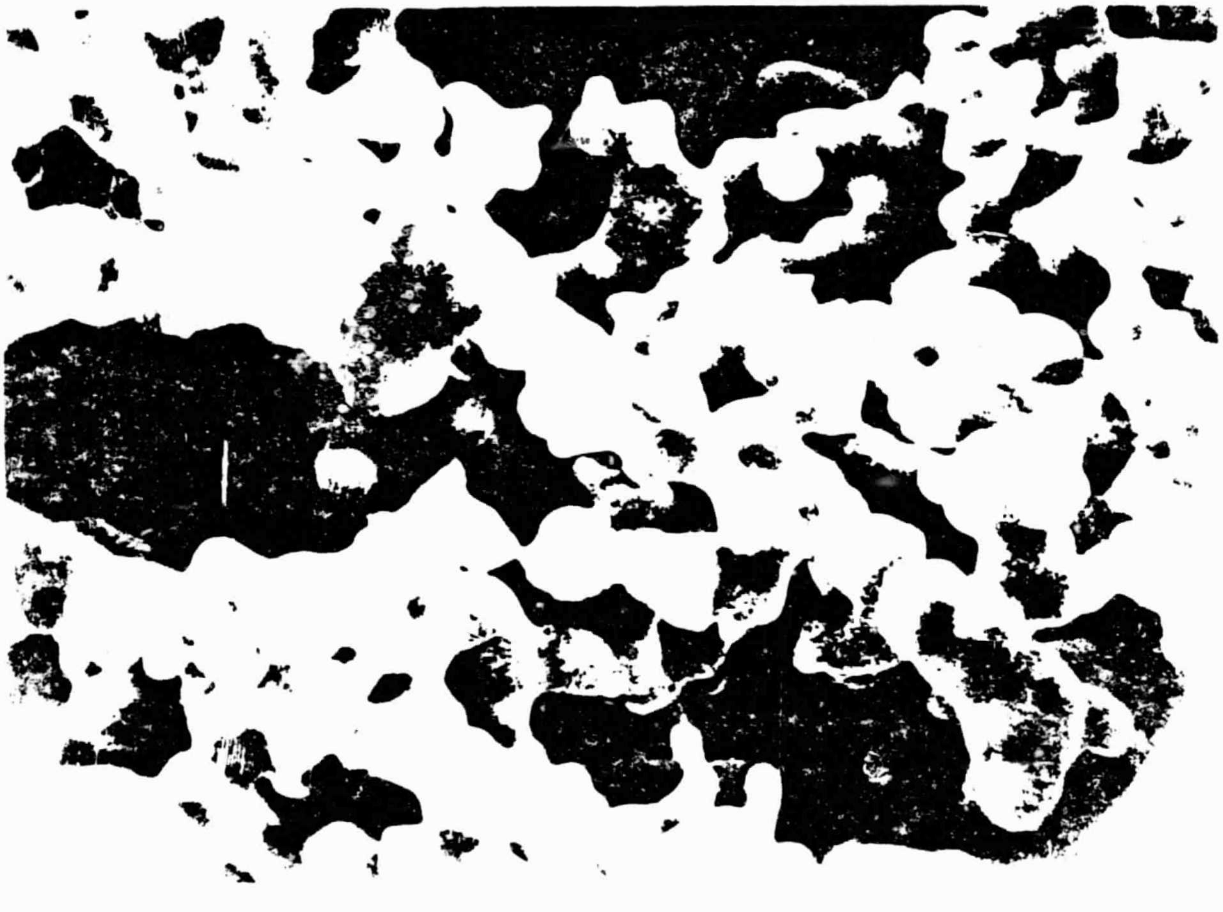
IMPREGNATION BATH :

$Ni + Co = 2M/l$.
$Ni(NO_3)_2 = 553 g/l$
$Co(NO_3)_2 = 29,1 g/l$



APPENDIX 12

STRUCTURE OF THE NICKEL SINTERING PRRIOR TO IMPREGNATION



STRUCTURE OF THE ELECTROCHEMICALLY IMPREGNATED SINTERING
(Ni-H2 type positive Electrode)