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**SANITIZED VERSION OF ELECTROLESS NICKEL PLATING OPERATIONS AT THE
OAK RIDGE AND PADUCAH GASEOUS DIFFUSION PLANTS**

(Sanitized Version of CRD Document KZ-7439)

Compiled by
S. G. Thornton
Environmental Management Division
OAK RIDGE K-25 SITE
for the Health Studies Agreement

July 31, 1996

Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7314
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the U.S. DEPARTMENT OF ENERGY
under Contract DE-AC05-84OR21400

This document has been approved for release
to the public by: *David B. Hilliland*

for A.S. Quist
Technical Information Officer
Oak Ridge K-25 Site

8/6/96
Date

1701

KZ. 7439

UNION CARBIDE NUCLEAR COMPANY

DIVISION OF UNION CARBIDE CORPORATION



POST OFFICE BOX P
OAK RIDGE, TENNESSEE

September 12, 1957

~~RESTRICTED DATA~~

This document is being disseminated in accordance with the provisions of the Atomic Energy Act of 1954 and is intended for the use of authorized personnel only.

U. S. Atomic Energy Commission
P. O. Box 3
Oak Ridge, Tennessee

Attention: Mr. S. W. Scott, Chief
Oak Ridge Patent Group

Subject: Electroless Nickel Plating Operations
at the Oak Ridge and Paducah Gaseous
Diffusion Plants

Enclosure:

Your attention is directed to the fact that electroless nickel plating operations are being conducted on a relatively small scale at our Oak Ridge and Paducah diffusion plants. These operations are being called to your attention because of their similarity to certain patented electroless nickel plating processes which will be identified in a succeeding paragraph.

To date, approximately seventy four pounds of nickel metal have been dissolved in hydrochloric acid to make up the nickel chloride used in these plating operations.

The electroless nickel plating facility at the Paducah Plant was designed in accordance with recommendations made by Metals Processing Company, Inc., and is operated with stock solutions purchased from that company. The stock solutions were procured under purchase orders containing standard patent indemnity clauses of the type included in Terms and Conditions Issued on August 3, 1955, and April 20, 1956. Exhibit 2 includes a schematic diagram and a process description of the Paducah facility, together with a calculation of the parts plated therein through July, 1957.

~~CONFIDENTIAL~~

~~CONFIDENTIAL~~

U.S.A.S.C.

September 11, 1957

Your attention is directed to the following United States patents dealing with electroless nickel plating:

- Patent No. 2,658,809, Process of Nickel Plating (Nov. 10, 1953)
- Patent No. 2,717,215, "Chemical Nickel Plating Methods and Apparatus" (Sept. 3, 1955)
- Patent No. 2,658,841, Process of Chemical Nickel Plating and Bath Therefor, (Nov. 10, 1953)
- Patent No. 2,658,841, Process of Nickel Plating and Bath Therefor, (Nov. 10, 1953)

These patents are assigned to General American Transportation Corporation. It is probable that a search would disclose other closely related patents of which we are not now aware.

Electroless nickel plating operations are continuing at the Oak Ridge and Paducah plating facilities.

It will be appreciated if you will determine whether or not operation of our electroless nickel plating facilities infringes the above mentioned patents or others. If so, please advise whether Carbide should negotiate for a license with the holder of patent interests which we may be infringing.

Very truly yours,

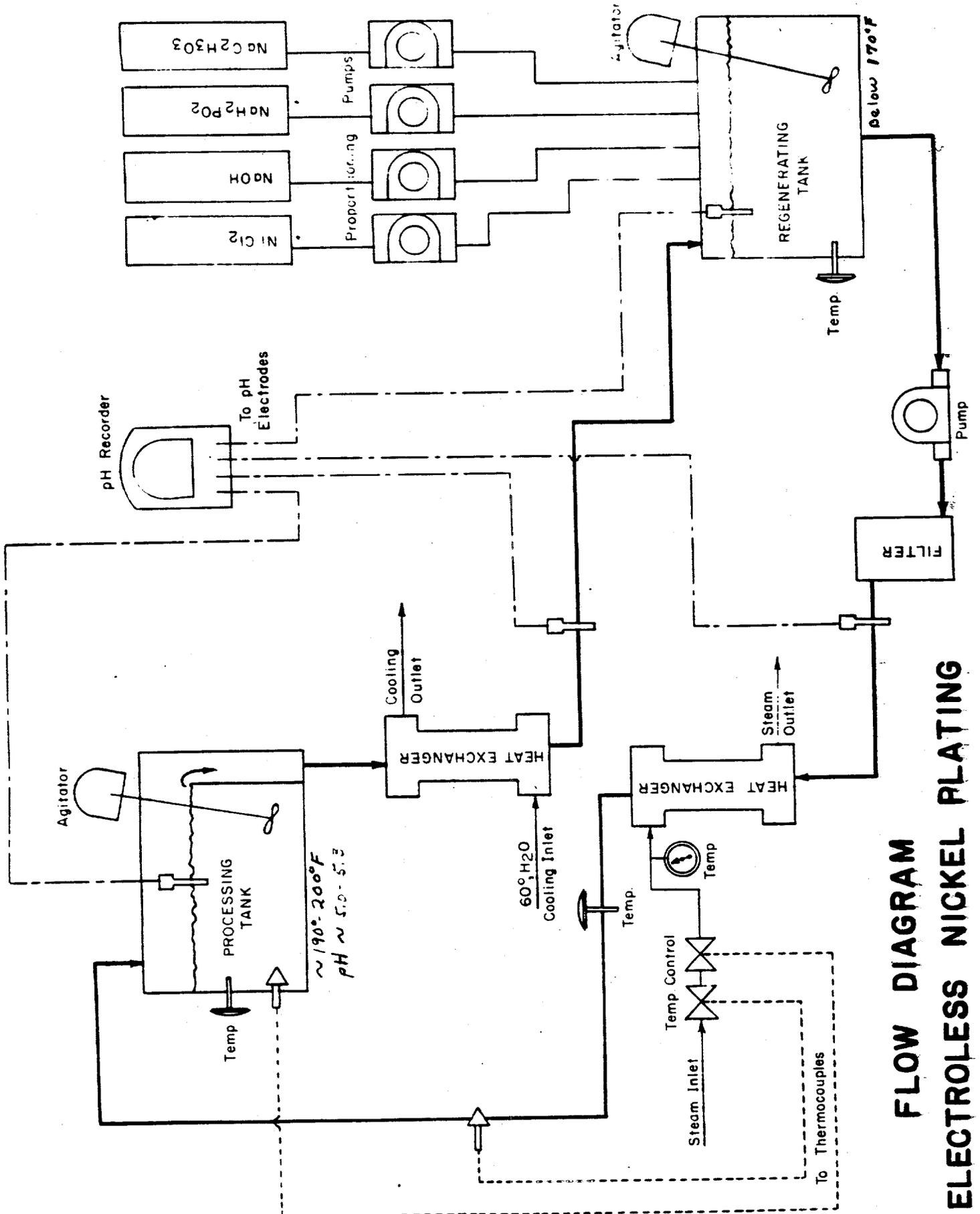
UNION CARBIDE NUCLEAR COMPANY


L.B. Emlet, Vice President

JEC:WLB:FCL:bdp (K-25 RC)
Enclosures (2)

- bc: L.B. Emlet, w/encls.
- A.P. Huber (3), w/encls.
- R.G. Jordan (3), w/encls.
- C.L. Gritzner, w/encls.
- M.A. Ladt, w/encls.
- J.A. Swartout, w/encls.
- J.P. Murray, w/encls.

~~CONFIDENTIAL~~



**FLOW DIAGRAM
ELECTROLESS NICKEL PLATING**

ATTACHMENT NO. 2

OPERATION SUMMARY SHEET - ELECTROLESS NICKEL-PLATING FACILITY

I. CHEMICAL MAKE-UP

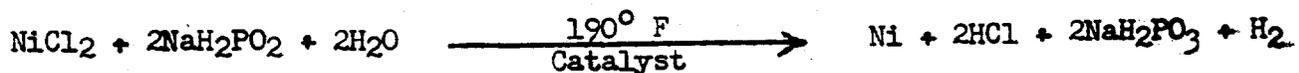
A. Plating Solution Feed Concentrations

1. Nickel Chloride (NiCl ₂ ·6H ₂ O)	-	300-600 gm/l
2. Sodium Hydroxacetate (NaC ₂ H ₃ O ₃)	-	200 gm/l
3. Sodium Hypophosphite (NaH ₂ PO ₂ ·H ₂ O)	-	120 gm/l
4. Sodium Hydroxide (NaOH)	-	3N

B. Bath Concentrations

1. Nickel Chloride	-	30 gm/l
2. Sodium Hydroxacetate	-	50 gm/l
3. Sodium Hypophosphite	-	9 gm/l
4. Sodium Hydroxide	-	Used to control pH

C. Chemical Reaction in Bath



D. Control

1. NaOH is added to hold the pH within the range of 5.0 to 5.3.
2. NiCl₂, NaH₂PO₂, and NaC₂H₃O₃ is added to raise the concentration of depleted solution to level shown in 'B' above.
3. NaC₂H₃O₃ is added as a buffer.
4. Temperature of solution in plating tank maintained between 190-200° F.
5. Temperature of solution in regenerating tank maintained below 170° F.

II. OPERATION

Solution is pumped from the regenerating tank through filters, through a heat exchanger, and into the constant level plating tank. Excess solution in plating tank flows through an overflow pipe, through a heat exchanger, and into the regenerating tank. Chemicals are added by proportioning pumps into the regenerating tank to restore the concentration of the solution and the cycle is repeated.

Control of the operation is maintained through the use of: (1) Temperature controllers, (2) Recording pH meter; and (3) Sampling and analysis for Nickel and Hypophosphite.

Parts being plated are immersed in the solution and suspended from a frame which is agitated with a vertical motion. The solutions in the plating and regenerating tanks are agitated with propeller-type agitators.

The plating rate varies between 0.4 and 0.6 mils/hr.

III. PREPARATION OF PARTS FOR PLATING

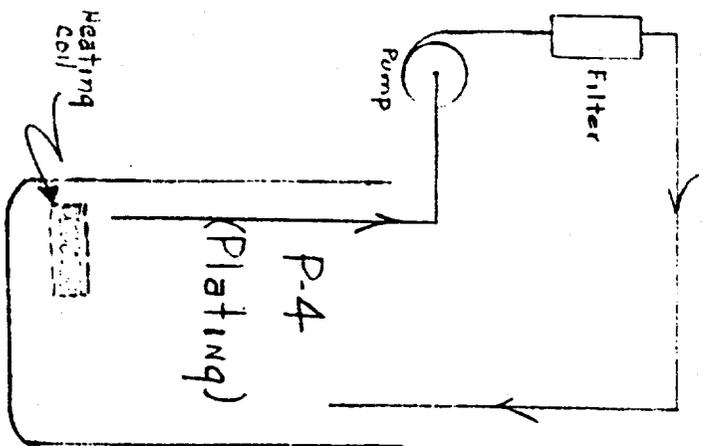
1. Normalize (steel only)
2. Demagnetize (steel only)
3. Degrease
4. Vapor blast
5. Rinse in cool water
6. Rinse in alcohol
7. Dry with air blast
8. Store in drying oven (to retard rusting)
9. Rinse in cool water (this step performed immediately before plating)

IV. EQUIPMENT

1. Plating tank - glass or steel w/glass lining, 3' x 3' x 6'
2. Regenerating tank - steel w/glass lining or stainless steel, 4' x 6' x 10'
3. Acid Storage Tank - stainless steel, 4' x 6' x 10'
4. Eight solution storage tanks - glass or steel w/glass lining, 3' x 3' x 5'
5. Pump - 25-gallons/minute, steel w/glass lining or stainless steel
6. Three proportioning pumps - stainless steel, 0-0.5 gallons/minute
7. Heat Exchanger, glass-lined, capacity = 25,000 BTU/hr.
8. Heat Exchanger, glass-lined, capacity = 40,000 BTU/hr.
9. Recorder, pH - L.N., Speedomax, Multi-Point
10. Electrodes, pH - 4 sets (two immersion, two flow)
11. Two filters - 40-gallons/minute, paper element
12. Three mixers - one-third hp, propeller type
13. Recorder, Temperature - L.N., Speedomax, Multi-Point
14. Miscellaneous equipment - valves, pipe, fittings, gages, motors, etc..

ELECTROLESS NICKEL-PLATING FACILITY-- ORGDP

Exhibit I



NaOH 20%	HCl 50%	H ₂ O
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LUSTRALLOY ELECTROLESS NICKEL PLATING APPARATUS

TABLE I

OPERATION OF THE ELECTROLESS NICKEL FACILITY

OPERATING CONDITIONS

Tank Capacity (liquid)	15 gallons
Operating Temperature	185-195°F
pH	5-5.5
Plating Solution	Concentrated stock solution purchased from Metal Processing Co., Inc. (P-4)
Plating Rate	0.0005 mil per hour
Filtration	Continuous
Regeneration of Solution	Continuous additions of reagents being depleted by plating action at rates prescribed in Tables III and IV.

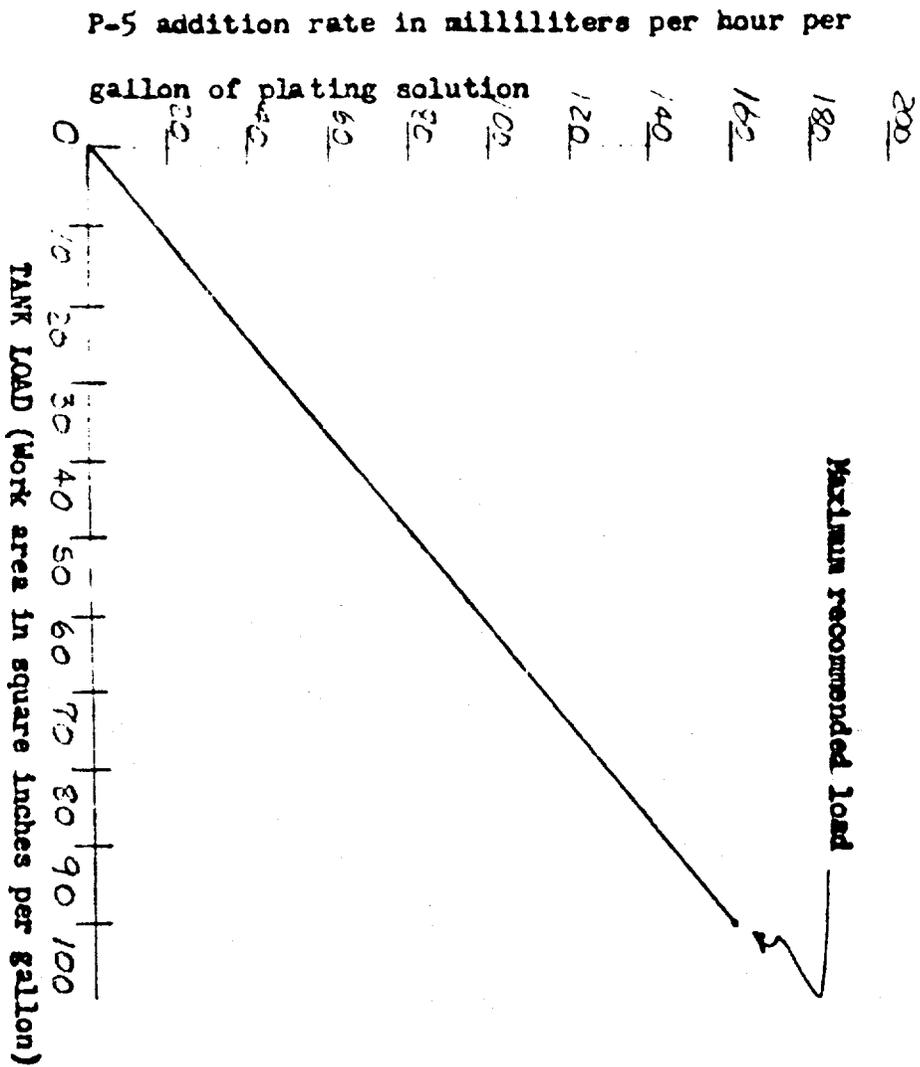
REAGENTS

P-4 Plating Solution	Nickel Chloride Sodium Hydroxyacetate Sodium Hypophosphite
P-5 (additive)	Nickel Chloride
P-6 (additive)	Sodium Hypophosphite
P-7 (additive-pH control)	Sodium Hydroxide

PROCEDURE

1. Vapor Degrease
2. Rinse in hot (165°F) HCl 50%
3. Rinse in cold H₂O
4. Rinse in 20% NaOH
5. Rinse in cold H₂O
6. Plate according to operating conditions.

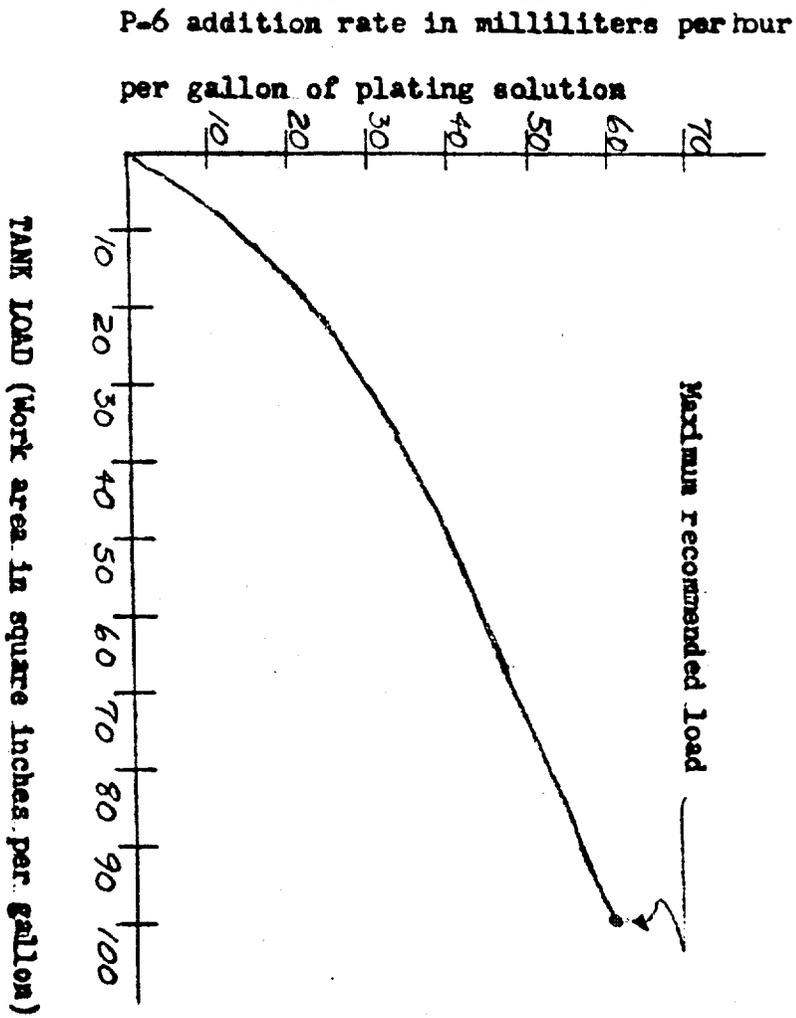
TABLE II



P-5

Notes: Continuous addition of P-5 is recommended but batch additions are permissible. Add $\frac{1}{4}$ of hourly requirement every 15 minutes.

TABLE III



Notes: The addition of P-6 should be made every 15 minutes at the rate of $\frac{1}{4}$ the hourly rate.

P-6

TABLE IV