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Title and author (if document is unnumbered)
*Please Note - Articles: "Methods of Determining Fluorides" from Journal of the American Water Works Assoc. Vol. 33, No. 11, Nov. 1941, pages 1965 thru 2020; and "Caustic Soda Safety Practices in Handling" from Chemical and Engineering News, July 25, 1944 were removed from this report. LMES can not release magazine article.

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Date submitted to ADC 10/17/95

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Date received from CICO 10/20/95

Date submitted to ChemRisk/Shonka and DOE 11/1/95

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Date document received

Signature

with removal of arsenic
"Methods of Softening Fluorides"
from Journal of the American Water Works Assn.
and "Caustic Soda..." News
from Chemical Eng

Carbide and Carbon Chemicals Corporation Operating Contractor for the U.S. Atomic Energy Commission.

CONTENTS

1. Introduction

C-216 DISPOSAL PLANT

INTRODUCTION

The material in this booklet has been compiled to aid the Disposal Plant operator by furnishing him with the basic theory underlying the plant operations, a description of the units of equipment, instruments and controls, operating and laboratory procedures. It outlines the data needed for successful plant control. A suggested report form has been included, also useful physical and chemical data and a chapter each on maintenance and safety practice.

The operator can of course add to it from time to time from his own experience.

W. C. Moore
Asst. Area Supv'r.

2. Theory of the Process

THEORY

The removal of C-216 & HF from the waste gases fed to the Disposal Plant is accomplished by neutralization with 10% Sodium Hydroxide in an absorption tower and precipitation as a calcium salt by treatment with lime slurry in an agitator and subsequently collecting the precipitate in a sedimentation tank.

The essential reactions involved are:

$$3(C\text{-}216)_2 + 6H_2O \longrightarrow 6H(C\text{-}216) + 3O_2$$

$$HF + NaOH \longrightarrow NaF + H_2O$$

$$Ca(OH)_2 + 2NaF \longrightarrow CaF_2 + 2NaOH$$

The absorption by NaOH proceeds best at temperatures above normal (150°F) and at high concentrations of NaOH. On the other hand sedimentation efficiencies are higher with low concentrations of NaOH (due to decreased viscosity) and less power is required to circulate the caustic solution. A satisfactory compromise is obtained with a 10% solution at 130°F. The governing temperature, however, should be the minimum required to reduce the C-216 concentration in the tower outlet gases to below say 10 p.p.m.

The plant should never be operated at caustic concentrations below 5%. This is necessary in order to prevent the formation of C-212, a substance even more toxic than C-216. It is produced in quantity by a brief contact with caustic of from 1/2 to 2% concentration.

$$4(C\text{-}216) + O_2 \longrightarrow 2(C\text{-}216)_2O$$

The solubility of Sodium Fluoride in water is 4 gms per 100 cc. at 15°C. But in 5% NaOH this is reduced to 1.3 gms per 100 cc.

From this fact it is apparent that lime should be fed continuously, whenever there is a possibility of obtaining a concentration of NaF above 1%.

A study of the solubility of $Ca(OH)_2$ in strong caustic will reveal that the amount of calcium in solution at any one time is very small (1-2 p.p.m.). Consequently an appreciable time for completion of the precipitation reaction is anticipated. Therefore a detention period of from 20 to 30 minutes in the agitator has been provided.

Preliminary tests in the laboratory indicate that best sedimentation will be obtained if an excess (12.5%) of $Ca(OH)_2$ over the amount theoretically required to precipitate all the S-215 is used.

Some fluoride will remain in solution due to the slight solubility of CaF_2 (about 1000 mg/l. with 10% caustic).

3. Description of Plant

DESCRIPTION OF PLANT

The plant consists essentially of an absorption tower, lime reaction tank, and sedimentation tank. Referring to the flow sheet enclosed, it can be seen that C-216 gas enters the base of the tower and flows upward against a descending stream of caustic. The absorbed gas is carried in the flow of caustic to the base of the tower and from thence is pumped into a reaction tank where the spent caustic is mixed with lime slurry. The mixture from the reaction tanks flows by gravity to the settling tank, where the calcium fluoride and excess lime settle out and the clear regenerated caustic solution flows over the effluent weir and is pumped again to the top of the tower.

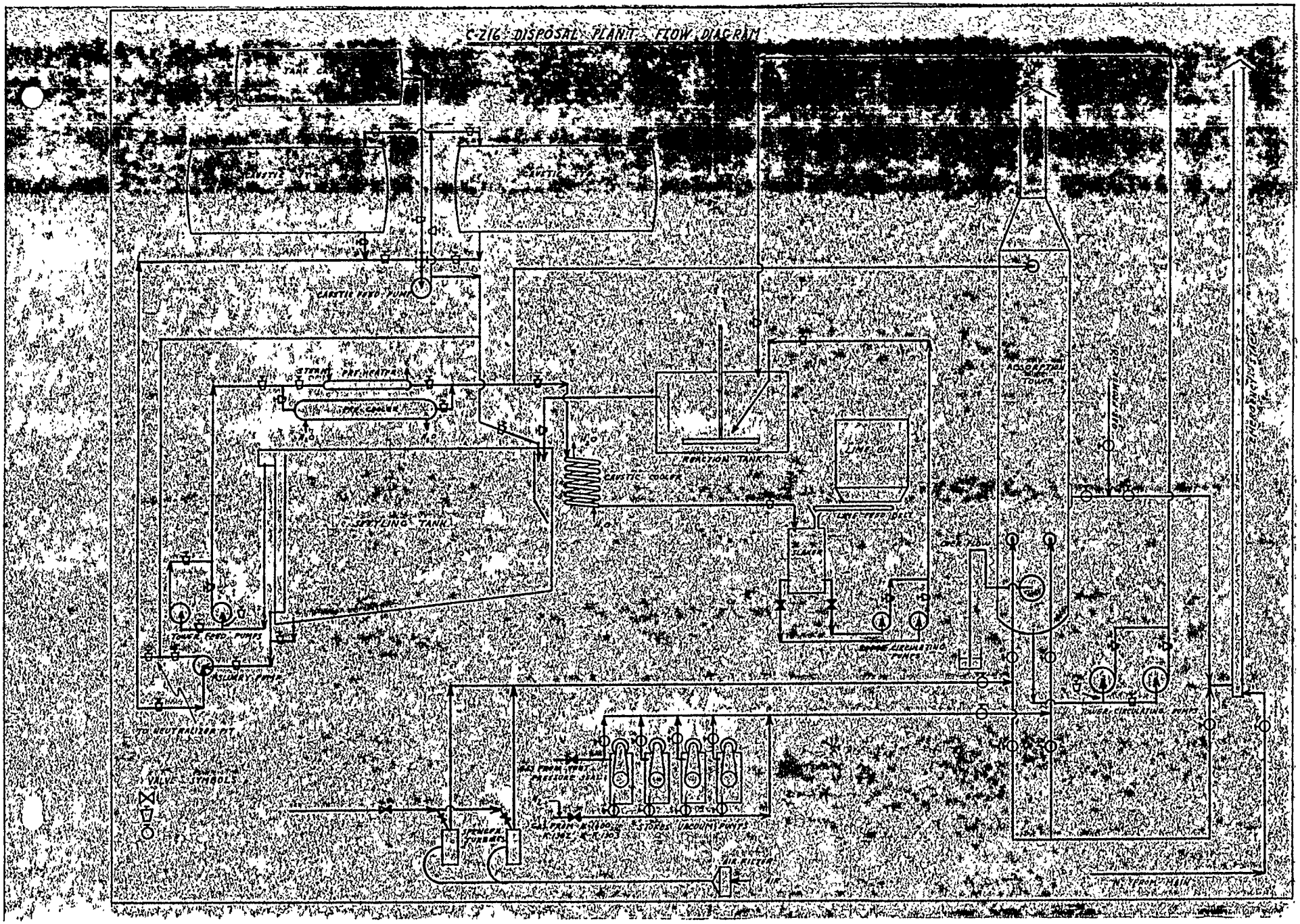
The tower which has an internal diameter of 4½ ft. is constructed of steel and lined with carbon brick. It is packed with 3" carbon Raschig rings set in place to a depth of 1 ft. and 2" rings dumped in at random to a depth of 12 ft., making a total packing depth of 13 ft.

Several layers of copper mesh are placed above the caustic manifold to function as a spray absorber. C-216 inlets are constructed of carbon pipe; all C-216 piping is constructed of monel. Caustic lines are made of ordinary black steel pipe.

An emergency monel exhaust stack has been provided for use when it is desirable to by-pass the tower.

In the event of failure of caustic flow, automatic valves will by-pass the gases containing C-216 to this emergency stack. This is to prevent the deposit of salt on the tower packing.

On the inlet side of the tower moisture traps are installed in each C-216 line. Following each moisture trap a large inverted "U" prevents possible backflow of caustic. In addition a caustic overflow has been


C-216 DISPOSAL PLANT FLOW DIAGRAM
CAUSTIC FEED PUMP
STEAM PRE-HEATER
REACTION TANK
CAUSTIC COOLER
SETTLING TANK
TOWER FEED PUMPS
SALINITY PIPE
TO NEUTRALISER PIT
VALVE SYMBOL
AIR FILTER
AIR

installed to prevent possible submergence of the gas inlets.

Lime is fed into the reaction tank in the form of a slurry with pre-cooled caustic which is prepared in a lime slaker, equipped with recirculating pumps. Agitation is obtained with either compressed air or an electric mixer and dry pebble quicklime is fed into the slaker at the desired rate through a Link Belt dry feed mechanism. The lime feeder has a capacity of 150 to 1500 lbs. per day and holds 6 tons of lime.

A 500 lb. electric hoist provides a convenient means of loading lime into the storage hopper.

The reaction tank is provided with a Turbo-Mixer rotary agitator which thoroughly mixes the lime slurry with the spent caustic.

At normal operating conditions the settling tank, which is constructed of steel and has a capacity of 22,500 gallons will have a detention period of 5 hrs. (at 75 gpm). Even distribution of the feed is obtained by the use of five inlets behind a baffle, and a low escape velocity is provided by the use of an effluent weir extending the full width of the tank.

Sampling outlets for determining the depth of accumulated sediment are provided on each side of the tank. The bottom has a slight slope toward the effluent end to facilitate cleaning and sludge removal.

Temperature control of the caustic is attained by the use of a steam jacketed parallel tube heat exchanger and a water jacketed cooler, connected in parallel. Caustic from the settling tank is pumped through this temperature regulator to the tower. A temperature controller and recorder operates a pair of three way automatic valves which route the liquid through either the heater or cooler as may be required.

Four 100 CFM Stokes vacuum pumps have been provided to evacuate C-216 lines between the plant and other buildings in case of emergency.

Two Spencer turbines have been provided to supply a constant flow of heated air to the tower. The purpose is to dilute C-216 from manifold vents in building K-1302 and cubicle vent from Bldg. 1303.

Caustic storage tanks are of all steel welded construction with a capacity of 25,000 gallons each. A caustic pump serves both tanks for either pumping from tank cars to storage or from storage to settling tank.

Note[1]: The original carbon-gas inlets plugged up with CaF and cracked from thermal shock when pure C-216 was fed into the tower during a test run. Therefore they have been replaced with monel inlets of modified design.

4. Instruments & Controls

INSTRUMENTS & CONTROLS

The caustic flow to the tower is metered thru an orifice. The flow rate can be read either from an indicator on the first floor of the plant or from a recording controller on the instrument panel on the second floor. A failure of caustic flow or reduction below any desired rate will actuate three pairs of motor operated valves on the gas inlets which divert gases from the tower to the emergency vent. An alarm is simultaneously sounded when this occurs.

Caustic flow and make-up water flow to the lime slaker are metered with recording instruments actuated by orifices.

Liquid level in the tower is governed by the simultaneous action of a reverse acting diaphragm valve on the tower feed line and a direct acting valve in the line from the tower to the reaction tank. Both valves are actuated by a level control at the tower. The flow rate can be controlled by manually throttling any valve in the caustic circulation system. Or if desired, a three way valve in the instrument air line at the rear of the instrument panel can be positioned to give level control from the diaphragm valve in the caustic line to the reaction tank only, and remote control of the flow rate from the instrument panel.

High or low levels in either the tower or reaction tank will actuate an alarm.

A recording instrument at the tower charts the fluctuations in liquid level in the tower.

Gas inlet pressures are recorded on a three point instrument actuated by transmitters on each inlet. When the instrument installation is completed an inlet pressure rise above any desired value will sound an alarm and divert gases from the tower to the emergendy vent.

Caustic temperature is controlled by a pair of three way valves which regulate the flow through the heater, cooler or by-pass to the rate required to maintain a desired temperature. These valves are actuated by a recording temperature controller on the instrument panel. Temperature indicators are installed on tower feed, recirculation, and calcium hydroxide pump discharge lines, also on the cooling water outlet from caustic coil.

Pressure indicators are installed on each pump discharge, also on both high and low pressure air, water, steam, and C-74 lines. Vacuum gauges measure the pressure in vacuum pump suction lines.

Flow of air from the Spencer turbines is metered through an orifice and the differential pressure transmitted to a pressure indicator.

The liquid level in caustic storage tanks is indicated by pressure gauges.

A temporary C-216 flow meter has been installed in the purge line from Bldg. 1302 for use in controlled test runs.

Liquid level in the lime slaker will be controlled with a float valve in the inlet line.

5. Operating Procedures

DUTIES AND RESPONSIBILITIES

The following duties and responsibilities pertaining to the operation of the C-216 Disposal Plant are herewith outlined for your information and guidance:

I. Each shift supervisor is responsible for operation, maintenance and analytical control on his shift in accordance with the following requirements:

A. The plant supervisor will be kept informed of all difficulties with equipment, personnel, or control by appropriate entries in the log book. Each shift supervisor will initial entries made during previous shifts to signify that he has read and understands their problems and activities.

B. Each supervisor will maintain a close liaison with the Hooker shift supervisor and will pay a visit to the Hooker Plant at the beginning of each shift for the purpose of familiarizing himself with Hooker operations and inform the Hooker supervisor of any changes at the Disposal Plant which might affect them. The time of such visit and the person contacted will be recorded in the log book.

C. He will keep himself informed of all personal problems affecting his men which might influence the quality of their work and report such information to the plant supervisor.

D. The Instrument Department will change all charts daily and check each instrument periodically. Failure of this check to be accomplished should be recorded.

E. All maintanence work will be performed under supervision of Mr. Long. Request for maintenance will be made in writing, outling in detail the work desired accompanied by sketches if needed. In case of an emergency, maintenance will be requested verbally and confirmed in writing as outlined above. A duplicate copy of all requests will be retained for the Disposal Plant file.

F. Each supervisor will be responsible for the safety of his personnel and no hazardous work of what-so-ever nature will be accomplished without his personal supervision. Operators will be required to wear rubber gloves and rubber goggles when operating caustic valves or pumps. When operating C-216 pumps and valves, they will be required to wear Bausch & Lomb goggles and asbestor gloves. On the first of each month and on the fifteenth of each month each supervisor will hold a gas mask drill and review thoroughly the proper methods of using masks and conditions under which they are unsafe.

G. Each supervisor will familiarize himself with the manufacturers' instructions pertaining to each item of equipment installed at the Disposal Plant, including instruments and electric controls, in order that he may have a thorough

understanding of the proper usage and care of such equipment.

H. Each supervisor will make a personal check of plant conditions at least once during each shift and will observe in particular the following points:

1. Are all pumps operating properly at normal discharge pressures without noise and vibration? Are the pump stuffing boxes receiving the right amount of lubrication?
2. Are motor and pump bearings operating at normal temperatures?
3. Are the exhaust fans in the vacuum pump room and over the caustic tank operating?
4. Are vacuum pumps ready for emergency operation with body and base heaters at a temperature of 140° F.?
5. Has the instrument department made its routine check as required?
6. Is the plant clean? In partic lar, it is undesirable to have bottles, loose bolts and nuts and other objects of any description placed on horizontal studding, in corners, on top of lockers, etc. Check to see that all equipment has been cleaned and polished, when required.
7. Are all gas lines properly valved into the tow r or emergency stack?
8. Is there sufficient liquid in the system?
9. Are caustic flow, lime-slurry flow, and lime feed rates, temperatures and pressures at the values instructed by the latest log book entry?
10. Do the analytical results indicate desirable operating conditions?

I. On the first day of each week arrangements will be made with the maintenance department to open all safety switches and motor starters and blow them free of dust with dry air. All motors will be cleaned likewise. For this operation the air pressure should not exceed 50 pounds.

J. A maintenance record system consisting of card index file with a separate card for each item of equipment will be kept up to date as maintenance performed on any item is accomplished.

K. Lubrication of all equipment shall be accomplished in accordance with the manufacturers' recommendations and except in case of those points of lubrication requiring daily attention a tag shall be attached to each item of equipment at the date of lubrication showing the type of lubricant used and the date lubrication is next required.

L. Each shift supervisor will personally perform such analytical work as required by the plant supervisor with the exception that routine analyses may be delegated to an operator at the discretion of the shift supervisor providing weekly checks on the operator's results are made.

M. No caustic will be discharged from the plant except through the neutralization pit and prior arrangement with 1407 Bldg. operator.

N. In the case of items of equipment installed in duplicate, one unit will be operated continuously for one week, then the alternate unit will be operated for a week and so on. All change overs will be made by the 12-8 shift on Mondays.

O. Normally idle equipment such as caustic unloading pump,

slurry pump, and vacuum pumps will be operated for a period of 30 minutes each week as a check on its condition. This will be accomplished by each shift on the following schedule:

12-8 shift Monday
8-4 shift Wednesday
4-12 shift Friday

P. Each shift supervisor will familiarize himself with the emergency alarm and telephone systems, and instruct his men in emergency procedures.

W. C. Moore
Asst. Area Supv'r.

DUTIES OF OPERATORS

For your information and guidance, the duties and responsibilities of Disposal Plant operators are listed below:

1. Each operator is responsible for the maintenance of the caustic flow, slurry flow, and lime feed rates, temperatures, pressures, and fluid rates prescribed by the shift supervisor, or the plant superintendent.
2. One operator will be designated by the shift supervisor to record hourly on the daily report form, all data called for.
3. The operator will assist in any laboratory procedures requested by the shift supervisor.
4. He will familiarize himself with the proper care and operation of each item of equipment and request the shift supervisor for instruction on any points about which he is in doubt.
5. He will leave the adjustment or repair of all instruments or controls to the Instrument Department. In case of emergency, manual control will be used.
6. Major repairs to equipment will be made by the Maintenance Department; minor adjustments by the operator.
7. He will familiarize himself with and observe the safety rules.
8. A good operator should take pride in a clean, efficiently operated plant, and strive at all times to keep it that way.
9. He will never leave the plant until his relief arrives, unless given permission by the shift supervisor.
10. He will protect the security of the war effort by refusing to discuss the plant in any way with any individual whomsoever except his fellow operators, the shift supervisors, and the plant superintendent. All requests for information from any other source should be politely but firmly referred to the shift supervisor.

W. C. Moose
Asst. Area Supv'r.

9-30-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

TOWER CIRCULATION

A. Start up procedure:

1. Check liquid level in settling tank weir trough. If low add water through reaction tank.
2. Open plug valve on suction side of tower feed pump.
3. Open vent valve on top of pump casing until a steady stream of caustic flows indicating pump is primed. Leave vent valve partially open.
4. Open plug valve on discharge side of tower feed pump.
5. Press starting button on pump control switch. If pump fails to start check safety switch to see if handle is in "on" position, press "reset" button in all the way on magnetic starter. If pump still fails to start call maintenance electrician.
6. After pump has run a few seconds, close vent valve on top of pump casing.
7. Start up recirculating pump exactly as outlined above for tower feed pump. This must be done without delay to prevent caustic from overflowing in tower.
8. If neither recirculating pump can be started shut down tower feed pump immediately.
9. After circulation has continued for a few minutes adjust liquid level in settling tank weir trough by adding water through reaction tank. If level is too high let it adjust itself through evaporation.

B. Operating procedure:

1. Check liquid level in settling tank weir trough each hour. If it is more than 6 inches below edge of weir add water through reaction tank until level is 3" below edge of weir.
2. Check liquid level in tower each hour. If out of range of float control request service from instrument department. Tower level can be controlled manually by by-passing control valve next to reaction tank and throttling plug valve on recirculating pump discharge.
3. Check caustic flow rate each hour. Adjust to desired value with flow control valve on instrument panel.

9-30-44

4. Read and record pressures and temperatures on tower feed pump and recirculating pump discharge lines once each hour.
5. Check pumps for excessive vibration or noise each hour. Add lubricant to stuffing box the first of each shift with two turns to the right on lubricator. Check pump and motor bearing temperatures. If excessively hot determine bearing temperature with thermometer. If motor bearing temperature is above 120°F. or pump bearing temperature is more than 30°F above caustic temperature change to alternate unit and request service from maintenance department.
6. To change to alternate unit start idle pump first, then shut down unit in operation.

C. Shut down procedure: (only after routing gases to the emergency vent.)

1. Press "off" button on motor switch to tower feed pump.
2. Close suction and discharge valves.
3. Shut down recirculating pump as above.

D. Precautions:

Never operate a centrifugal pump against a closed discharge valve for over 10 minutes. The heat produced by liquid friction will cause the wearing rings to expand and rub against each other, ruining the pump. The packing gland should be tight enough to stop caustic leakage but loose enough to permit free rotation of the shaft by hand. Smothering gland should not be used.

9-29-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

LIME SLAKER

A. Start up procedure

1. Close drain valve on slaker can (No. 11).
2. Open valve on caustic line (#6) to can and fill can with caustic.
3. Close drain valve on slurry pump suction (No. 10)
4. Open valve on pump suction line to can. (No. 9).
5. Open vent valve on top of pump casing (No. 14) until all air is vented then close vent valve.
6. Open valve on pump discharge (No. 8).
7. Be sure that valve in water connection to pump discharge line (No. 3) above can is closed and globe valve in pump discharge line to reaction tank is open. (No. 2)
8. Press pump starting button.
9. Open and close vent valve at top of pump casing to be sure all air is vented from pump. (No. 14)
9a. Throttle caustic flow with pump discharge valve (No. 8) until liquid level remains in constant position.
10. Open cooling water valve (not shown on sketch) on caustic cooling coil enough to cool caustic to desired temperature without waste of cooling water.
11. Open air valve 1/8 turn (No. 13) or enough to obtain good agitation.
12. Press starting button on lime feed machine.
13. Regulate lime feed to desired rate.

B. Operating procedure

1. Read and record caustic flow rate to can at hourly intervals.
2. Check slurry pumps for condition of packing gland and bearings each hour.
3. Check bearing temperatures in lime feed machine each hour, and if over 120°F change to alternate unit and request maintenance.
4. Check liquid level in can. It should be about 6" below overflow.
5. Be sure belt is traveling in center of rollers. If off center release tension on off side by turning adjusting screw.

9-29-44

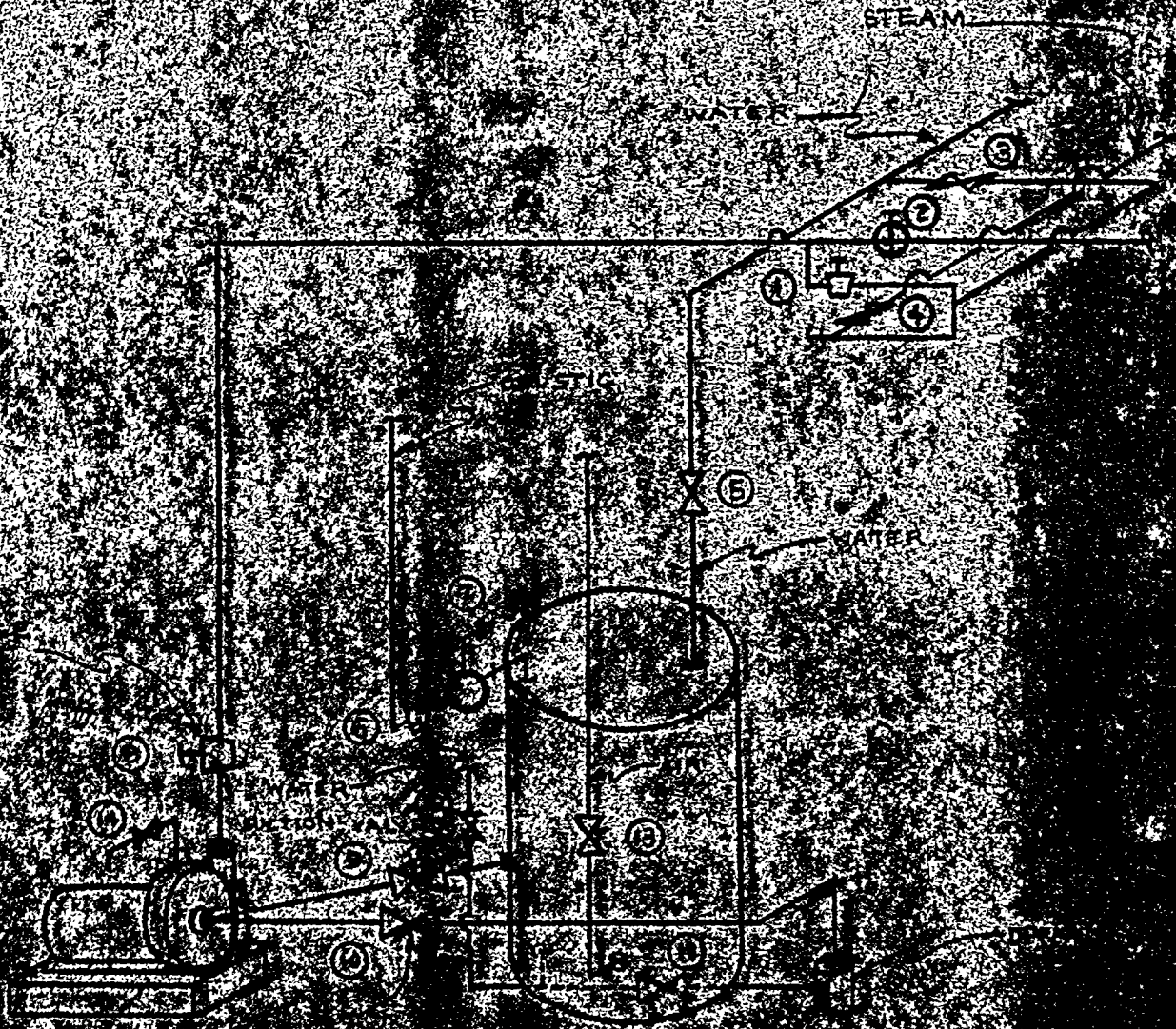
C. Shut down Procedure

1. Press "stop" button on lime feed motor control switch.
2. Close caustic valve in line to slaker can. (No. 6).
3. Press "stop" button on pump motor control switch.
4. Close pump discharge and suction valves. No. 8 and 9.
5. Close air valve. (No. 13).

D. Precautions

1. If system becomes clogged with lime shut down and backwash through pumps to drain. Flush discharge line to reaction tank with smallest possible quantity of water.
2. Never tighten one side of belt only; this will put a permanent set in belt.
3. If belt slips change over to alternate feeder and request maintenance.
4. If cooling coil is not operated hot water vapor from caustic will rise through lime feed huts and moisten lime prematurely. This may clog idle unit with plaster.
5. Unslaked lime will burn as badly as caustic so use rubber gloves.
6. Do not permit loss of caustic through overflow.
7. Never try to adjust varispeed drive unless machine is running.

LIME SLAKER & PIPING



GATE VALVE

GLOBE VALVE

FLOAT CONTROL VALVE

FORD BACON & DAVIS

216 DISPOSAL

DATE 3-28-44

9-29-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

VACUUM PUMPS & PIPING

A. Start up procedure

1. BASE & BODY HEATERS should be left on at all times. Caution--never start a pump unless both heaters have been on at least 8 hours.
2. Open discharge valve B.
3. Check to see that cooling water valves C and E are open.
4. Turn pump over once or twice by hand to make sure that piston turns freely.
5. Press pump starting button,
6. When motor starts solenoid valve D should open and body heater switch should close automatically. Otherwise pump should be stopped at once.
7. Open suction valve (A) gradually maintaining 27" vacuum.
8. Adjust flow of cooling water with angle valve E to 1 or 2 gal/min.

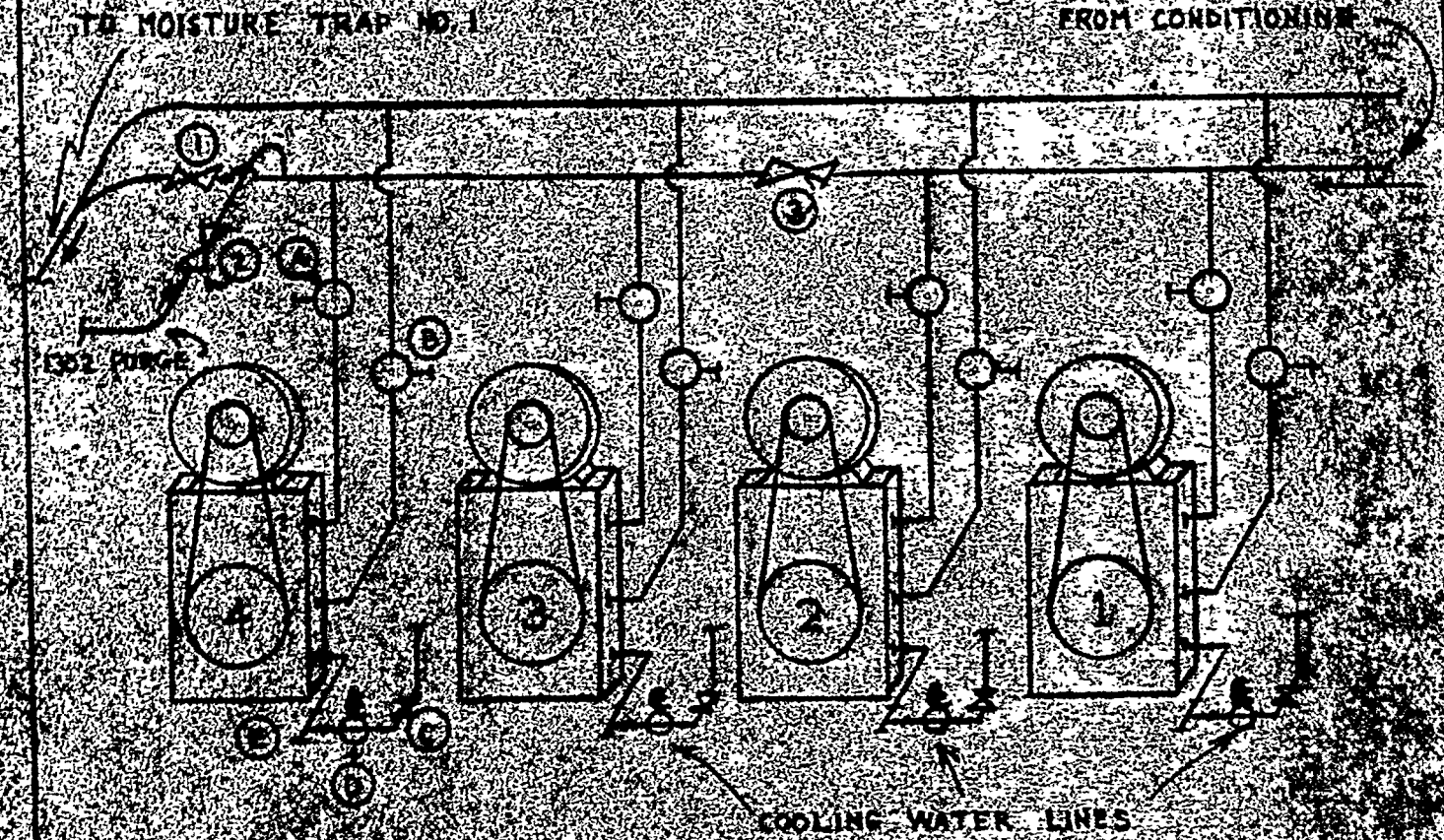
B. Shut down procedure

1. Before shutting down purge lines and pump with G-74 see procedure on piping from conditioning bldg.
2. Press pump "stop" button.
3. Break vacuum with G-74 by cracking G-74 valve open.
4. Close G-74 valve.
5. Close suction valve (A).
6. Close discharge valve (B).
7. Leave heaters on and cooling water valves open.

C. Precautions

1. Maintenance on vacuum pumps will be accomplished only under Mr. Barclay's supervision.
2. Never start a pump cold.
3. Never start a pump against a closed discharge valve.
4. Pumps will not operate against a back pressure greater than 812 mm (about 1 lb. gage).
5. Never open suction valve wide until vacuum of 27" is obtained. Operation at lower degree of vacuum will result in loss of C-2144 oil through vaporization.

VACUUM PUMPS & PIPING



SYMBOLS
- ANGLE VALVE
- GLOBE VALVE
- GATE VALVE
- SOLENOID VALVE

FORD, BACON & DAVIS INC.

G-216 DISPOSAL PLANT

DATE: 9-28-44 BY: J.H.S.

9-29-44

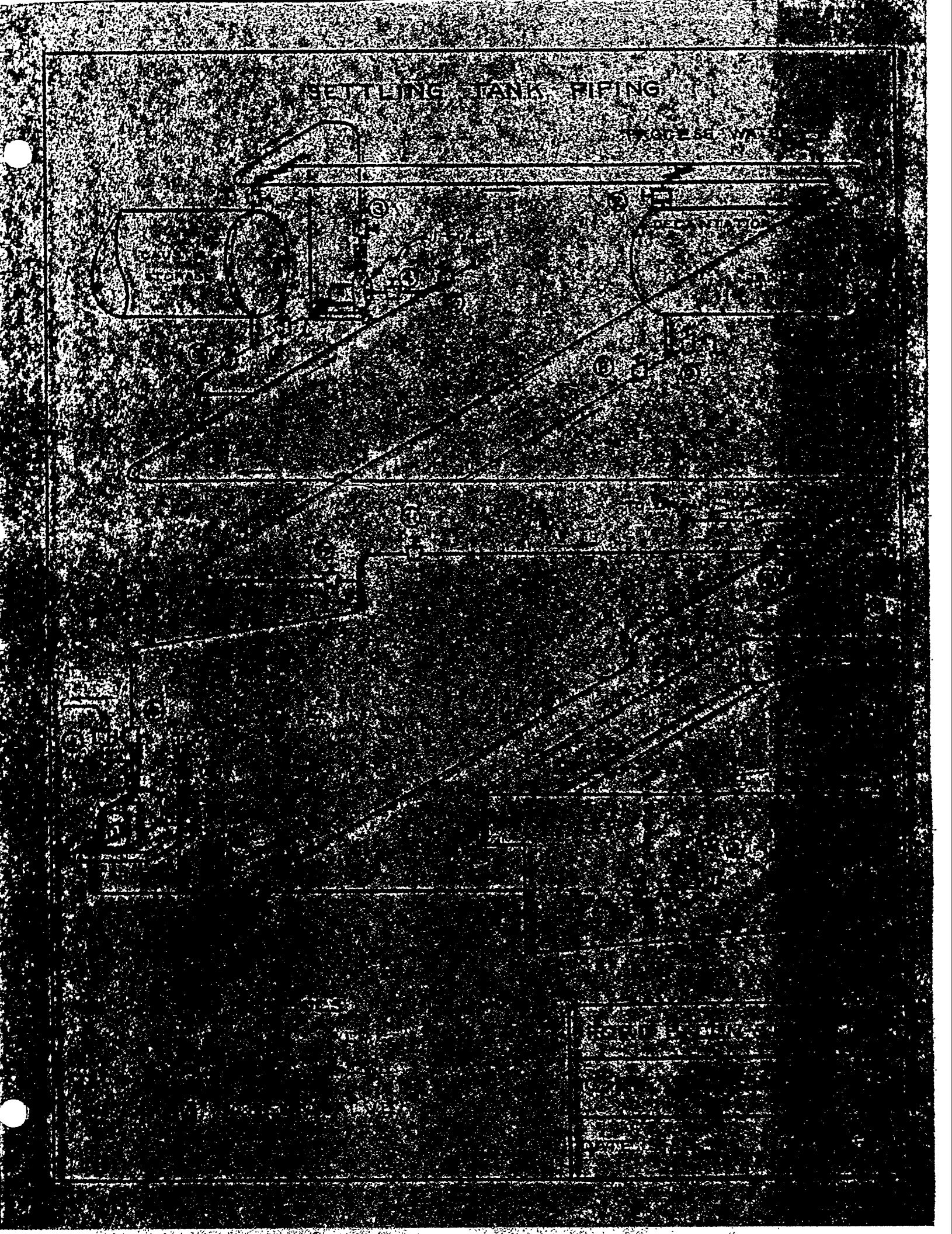
DISPOSAL PLANT OPERATING INSTRUCTIONS

SETTLING TANK

A. For removal of slurry with caustic unloading pump.

1. Valves 1, 5, 6, 7, 8, 9, 10, 11, 12, 13, 16, 20 should be closed.
2. Lower swing pipes into caustic until just above slurry depth.
3. Open valves 2, 3, 4, 14, 15.
4. Open vent valve on top of caustic unloading pump J-604 and when all air is removed from pump casing, close vent valve.
5. Press pump starting button.
6. Allow pump to run until swing pipes begin to draw in air.
7. Stop pump.
8. Connect hose to outlet at reaction tank and insert end of hose down reaction tank outlet.
9. Close valve 2.
10. Open valves 10, 12, 13, 16.
11. Start pump.
12. Open water valve at hose connections.
13. Allow pump to run until all slurry has been removed.
14. Close water valve at hose connection.
15. When all water has been removed from settling tank stop pump.
16. Close valves 12, 13, 16, 17, 19.
17. Open valve 8, 11, 18.
18. Start pump.
19. Allow pump to run until settling tank level is at edge of weir.
20. Open valve 17 and close valve 18.
21. When reaction tank starts to overflow stop pump.
22. Close valve 8.
23. Open valve 21 and flush system with water through pump into reaction tank.
24. Close valve 21.
25. Open valve 20 and drain piping.
26. Drain pump from plug in bottom of casing.
27. Start tower circulation.

SETTLING TANK PIPING



9-30-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

C-216 PIPING FROM CONDITIONING BLDG.

A. To By-Pass Vacuum Pumps
   1. Open valves 1, 2, 3.
   2. Close valves 4, 5.

B. To Evacuate Common Line
   1. Open valve 4, (1 and 2 should be open).
   2. Close valve 3, (5 should be closed).
   3. Follow vacuum pump procedure.

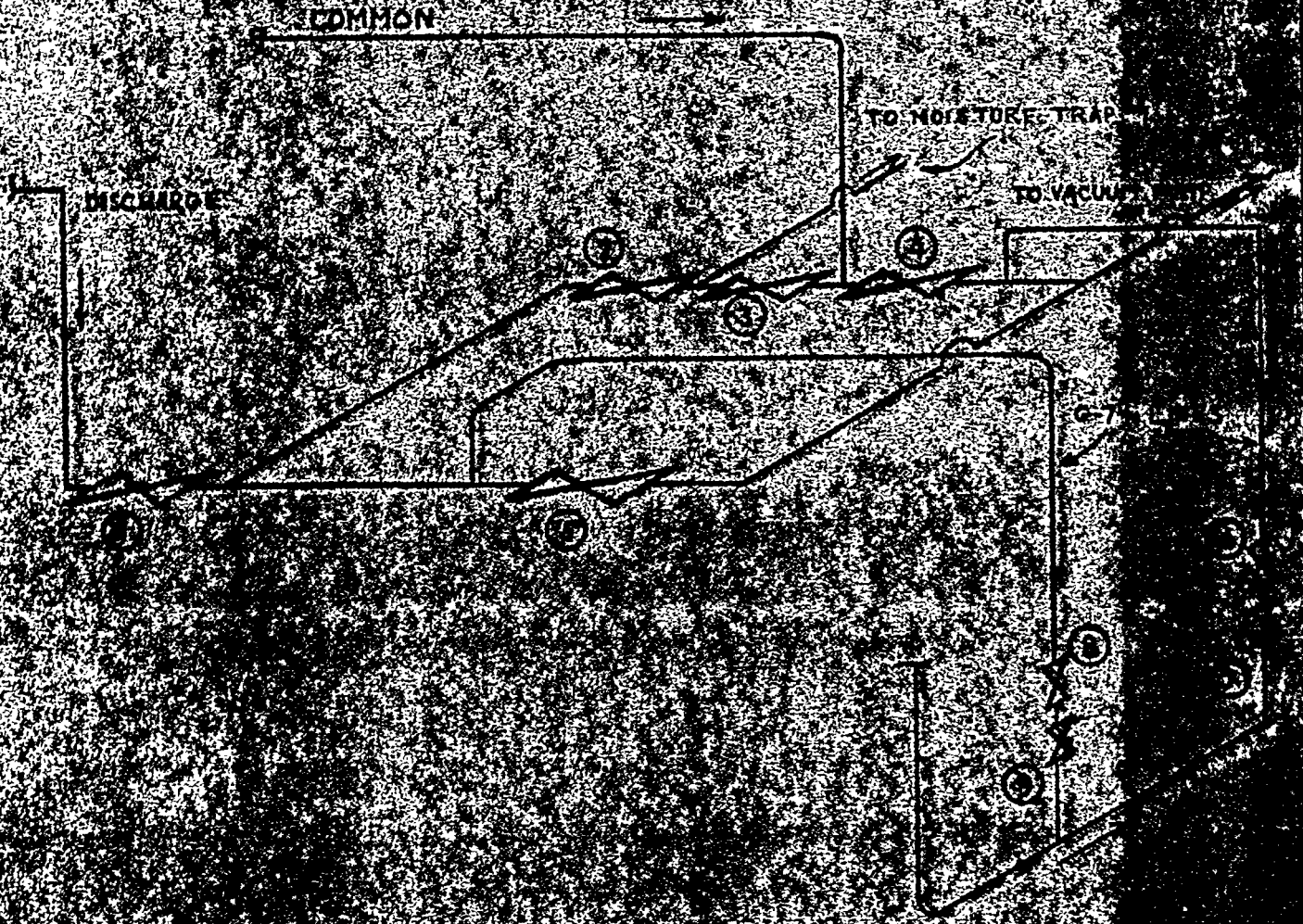
C. To Evacuate Discharge Line
   1. Open valve 5, (1 and 3 should be open).
   2. Close valve 2, (4 should be closed).
   3. Follow vacuum pump procedure.

D. To Purge Vacuum Pumps
   1. Close valves 4 and 5. (1, 2, and 3 should be open).
   2. Open G-74 valves 6 and 7 one fourth turn each.
   3. Operate vacuum pump for 30 minutes.
   4. After lines are purged close valves 6 and 7.

Precautions:

Never open G-74 valves under conditions that may build up back pressure as little as 1" of water in Hooker or Conditioning Bldg. lines without notifying Hooker and/or Conditioning.

G-216 PIPING FROM CONDITIONING BUILDING



FORD, BACON & DAVIS

G-216 DISPOSAL

DATE: 8-24-44

9-25-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

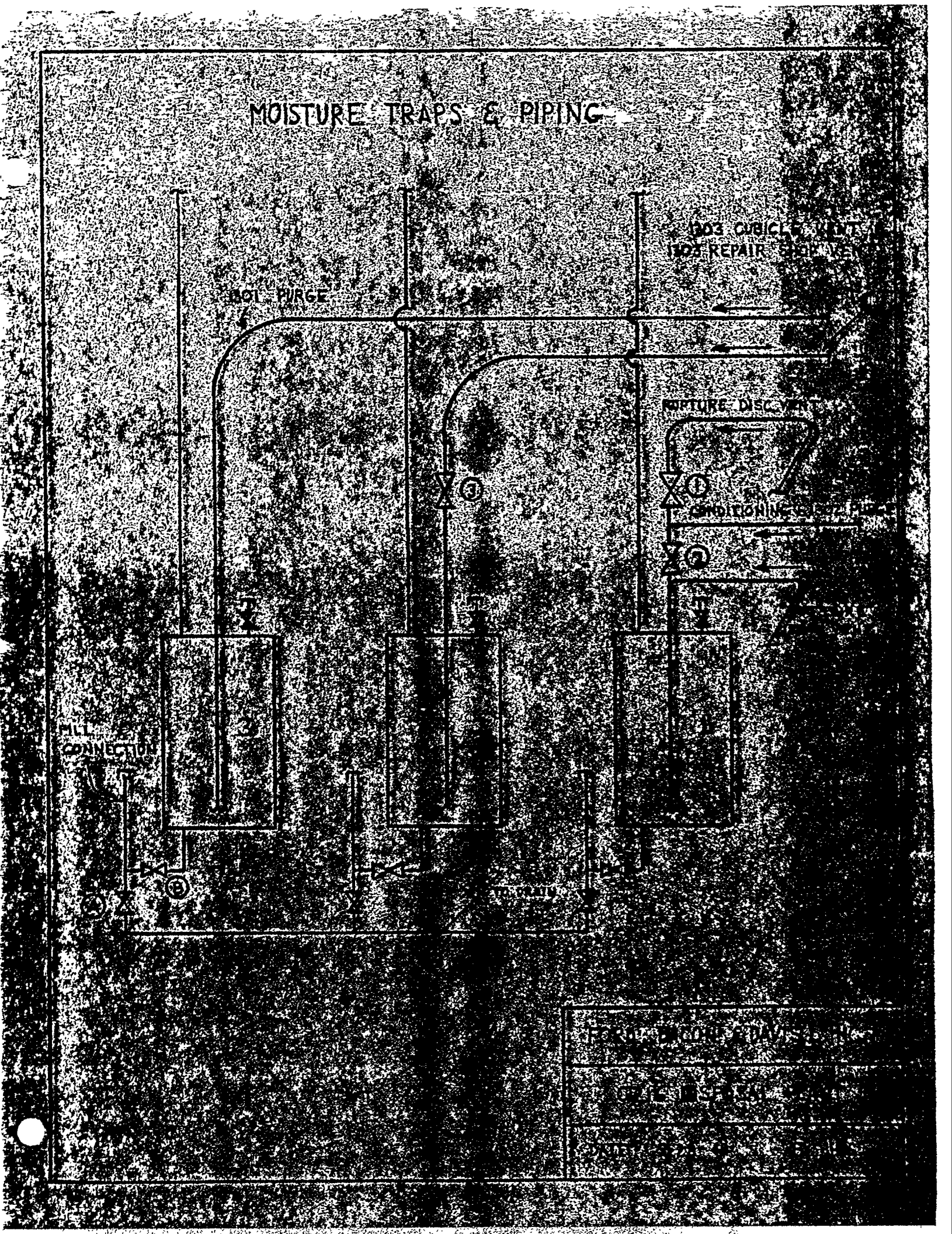
MOISTURE TRAPS & PIPING

1. Valves 1, 2, and 3 should be locked in open position at all times. Never close one of these valves without prior arrangement with Hooker and/or conditioning as required.

2. Fill traps with 85% Phosphoric Acid to depth of 1" as follows:
   a. Unscrew cap on fill connection after first checking to be sure valves A and B are closed.
   b. Place funnel in fill connection and pour in acid to top.
   c. Open valve B and allow acid to flow into moisture trap.
   d. Repeat until required depth is obtained as measured from top of fill connection. Allow $\frac{1}{8}$" for thickness of bottom plate. 1 1/3 gallons of acid will be required.
   e. Withdraw and analyze sample of acid weekly. If acid strength drops below 80% drain and replace.

Caution - do not remove caps to drain trap.

MOISTURE TRAPS & PIPING



9-25-44

DISPOSAL PLANT OPERATING OPERATIONS

MANUAL CONTROL OF HEATER

A. Start up procedure

1. Close valves 1, 2, 3, 4, 5, 6, 7, 8, 10, and 12 and both drain valves.
2. Open valves 9 and 11. This puts caustic flow thru heater.
3. Open steam valve ¼ turn.
4. Open globe valve on by-pass around steam trap.
5. Allow heater to warm up.
6. Open gate valves on each side of steam trap.
7. Close globe valve on trap by-pass.
8. Open steam valve wide.

B. Operating procedure

1. Read and record caustic temperature each hour if over 130°F., open valve 10. This by-passes half of flow. If temperature is below 125°F. close valve (10). This puts full flow through heater.

C. Shut down procedure

1. Close steam valve.
2. Open valve (10).
3. Close valves 9 and 11.
4. To drain heater open both drain valves.

D. Precautions:

1. Never close or throttle caustic valves to heater when steam is on.
2. Do not use heater and cooler at same time.

CAUSTIC TEMPERATURE CONTROLLER

9-25-44

DISPOSAL PLANT OPERATING INSTRUCTIONS

C-216 VALVE NEST AT TOWER

A. For Manual Control

1. To vent to emergency stack
   a. Open valve (2) then close valve (1)
   b. Close valves 3, 5, 6, 8.
2. To vent to tower
   a. Open valve (1) then close valve (2)
   b. Close valves 6, 8, 3, 5.

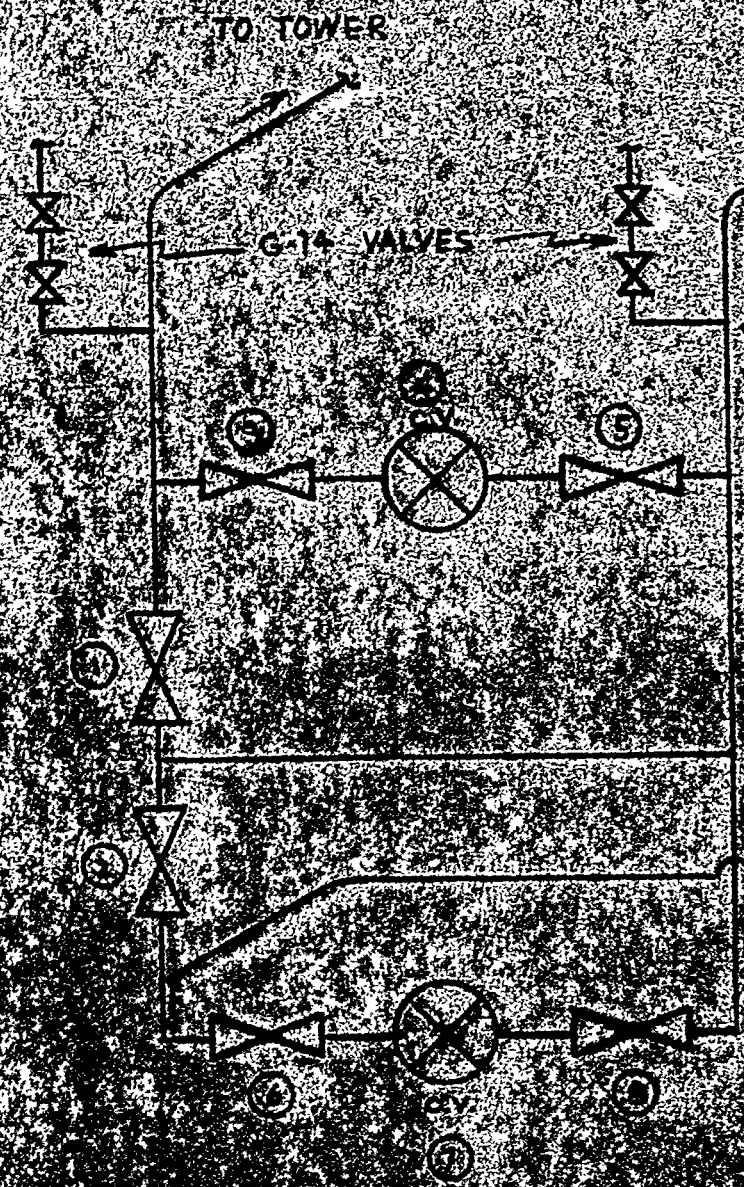
B. For Automatic Control

1. Open valves 3, 5, 6, 8.
2. Close valves (1), 2.

Precautions:

1. Never turn gas into tower if:
   a. Caustic flow is less than 50 G.P.M. or
   b. Caustic strength less than 5% NaOH or
   c. Liquid level is below center line of manhole.
2. Never open C-74 valves without notifying Hooker and Conditioning that back pressure may be built up in their lines.

C-216 VALVE NEST AT TOWER



FORD, BACON & DAVIS INC.

C-216 DISPOSAL PLANT

DATE 9-28-44

6. Laboratory Procedures

LABORATORY PROCEDURES

For routine analytical control methods emphasis should be placed upon simplicity and rapidity rather than extreme accuracy. This insures the most efficient use of time spent in the laboratory. The following methods were selected with this thought in mind:

A. Sampling-

A great deal of time can be wasted by careful analytical work spent on improperly collected samples. Therefore, the analyst should give some thought to the method of collecting his samples.

Caustic samples are most easily collected from the sampling outlets provided on pump discharge lines. The sampling valve should be opened and enough of the first portion of the caustic which flows wasted to insure thorough purging of the sampling line. At least 3 cm. of sample should be collected.

Samples taken from either storage tanks, tank car, or settling tank should be composited from individual portions collected from various depths.

Gas samples should be collected with extreme care. The temperature and pressure of the gas must be known as well as the volume. In this case also, enough of the first gas collected should be wasted to insure thorough purging of sampling lines.

B. Reagents-

1. Hydrochloric Acid

Data: Specific Gravity = 1.012 approx. percentage HCl = 3.58%

Procedure: Take 1/12 of the volume of normal acid required of pure laboratory hydrochloric acid. Add to it 11/12 volume of distilled water. This will give a solution of slightly higher concentration than normal. Standardize against 3 gms pure sodium carbonate, using 2 drops methyl orange as an indicator. Adjust concentration to between 1.01 N and 0.99 N.

Calculations:

$$\text{Normality} = \frac{\text{gms } Na_2CO_3}{\text{mls Acid} \times .05303}$$

2. N/10 $KMnO_4$

Weigh out approximately 3.2 gms $KMnO_4$ per liter of solution desired. Boil the solution 10-15 minutes and allow to stand overnight. Filter through asbestos.

Standardization:

Weigh on watch glass .3 gms. (accurately) Sodium Oxalate. Transfer to 600 ml beaker. Add 250 ml 2-1 Sulphuric Acid, and stir until all Oxalate has dissolved. Add 39-40 ml. $KMnO_4$ solution at fast rate. Heat solution to 55-60°C. and finish titration.

Data: 1 ml N/10 $KMnO_4$ = .00670 gms.

3. Ferric Chloride

Ferric Chloride solution is standardized according to the following procedure:

15 ml. of $FeCl_3$ solution is taken for a sample. 3 grams KI are added and the solution is allowed to stand for about 5 minutes. Dilute to 100 ml. and titrate with N/10 $Na_2S_2O_3$ to a pale yellow color. Add starch as indicator and finish

titration.

Molecular wt. $FeCl_3 - 6H_2O$ 270.31

Molecular Wt. KCNS 97.17

Grams Fe in 1 gm $FeCl_3 - 6H_2O$

4. Ammonium Oxalate

10% solution. No standardization necessary.

5. Barium Chloride

N solution. No standardization necessary.

Weigh out 208 gms $BaCl_2$. Dissolve in enough water to make 1 L of solution.

6. Sodium Fluoride

Normal solution. No standardization necessary.

Weigh out 42 gms NaF. Dissolve in enough water to make 1 L of solution.

7. Potassium Thiocyanate

Make accurately. No standardization necessary.

Weigh out 97.2 gms KCNS. Dissolve in enough water to make 1 L of solution.

8. Dilute acids and bases

By 2-1 Acid is meant quantity of water that is to be added to unit quantity of acid. In this case 2 volumes distilled water are to be added to one volume concentrated acid.

Acids or bases made according to this notation are never standardized.

C. Analytical Methods

I. Sodium Hydroxide

Dilute a 2.5 ml. sample to 150 ml. using distilled water. Add 3 drops of methyl orange indicator and titrate to a pink end point with standard 1 N hydrochloric acid. Calculate the normality of the caustic solution as follows:

$$\frac{\text{ml acid used}}{\text{ml sample}} \times \text{normality of acid used} = \text{normality of sample}$$

II. Turbidity

See instructions with turbidimeter.

III. Fluoride-Colorimetric Method

Reagents:
- Sodium Fluoride 1 gm F^-/l solu.
- N/10 $FeCl_3$
- N KCNS
- 10% Commercial Caustic
- N HCl
- N/10 NaOH
- N/100 HCl
- Phenolpthalien, Brown Cresol Green)- Indicator

General:

The pH of the test solution must be controlled very accurately. This is done in the following way. A few drops of phenolpthalein are added to the sample and it is titrated with N HCl to a colorless endpoint. The color is restored with N/10 NaOH. 6 drops Brown Cresol Green is added and the sample very carefully titrated with N/100 HCl to a yellow endpoint (nearly colorless). This gives a pH of 5.4 -3.8. With care the pH can be held to 5.4 - 4.9.

Samples:

In the range of 1000 to 1500 mgm/l, 5 ml samples are sufficient. The solution should be centrifuged for 1 minute at a high speed to eliminate suspended lime. The sample is taken after centrifuging.

Standards:

Standards for the range 1000 - 1500 should be made as follows:

To each of six numbered 150 c.c. erlenmeyers, add 5 ml 10% commercial caustic. To number 1 add 5 ml Sodium Fluoride solution. To No. 2 add 5 1/2 ml., to No. 3 - 6 ml., to No. 4 - 6 1/2 ml., to No. 5 - 7 ml., to No. 6 - 7 1/2 ml., The pH of each of these should be adjusted as in general paragraph. The solutions are then transferred to Nessler tubes and the tube diluted to the mark. To each of these tubes is added 1 ml N KCNS and 1 ml. N/10 FeCl3. Tubes are then shaken to uniform color and labeled.

Unknown:

The 5 ml. sample is adjusted to the right pH as shown in general paragraph. The solution transferred to a Nessler tube, diluted to the mark, 1 ml. N KCNS and 1 ml. N/10 FeCl3 added. Color of unknown and color of standard compared by looking both at the sides of the Nessler tubes and looking down through the Nessler. Interpolation can be made on the basis of color change.

IV. C-216 Analysis

Sampling lines from each of the three gas inlets and from the top of the tower have been installed in the laboratory as a convenience to the analyst, also air and C-74 lines and a small air ejector. The sampling of C-216 requires special precautions.

1. All lines and glass apparatus must be clean and perfectly dry.

2. Use only C-2144 oil for stopcock grease on glass sampling bulbs.

3. Do not use rubber connections but only standard tubing fittings, copper glass seals, and ground glass joints lubricated with C-2144 oil.

4. When analyzing gas of over 20% concentration it is advisable to collect the sample through a clean dry metal tube of ample dimensions packed with oven dried sodium chloride.

The C-216 is then converted to chlorine which may be more safely handled.

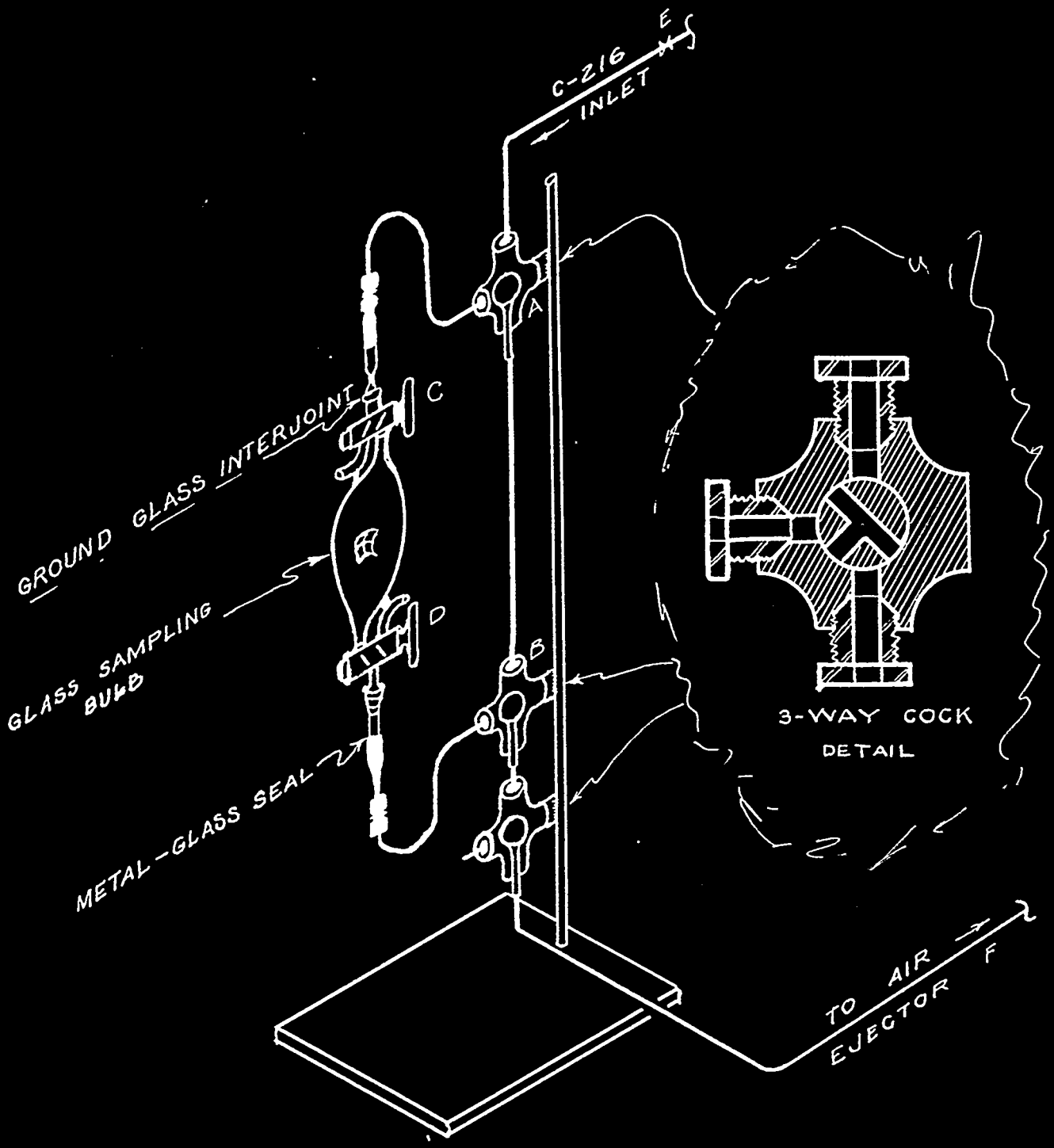
IVa. Inlet gases -

If the concentration of C-216 is known to be less than 20% it may be safely collected directly in a clean dry evacuated gas sampling bulb as follows: (Reference is made to the drawing showing the sampling device.)

1. Purge the sampling line by connecting the sampling device to the suction fitting on the air ejector. Turn the two way valves at A and B so as to by-pass the sampling bulb. Open the valve on the sampling line and then open the ejector valve. After the line has been thoroughly purged with the gas to be sampled, turn the three way valves A and B so as to force gas through the sampling bulb. The two way stop cocks C and D on the sampling bulb should previously be set in their proper positions. After the bulb has filled with the gas to be analyzed, close the valve on the sampling line and turn the three way valve so as to connect the tubing leading to the sampling bulb with the ejector suction. Then open the two way stop-cocks C and D to the atmosphere. After the lines have been purged with air shut off the ejector and remove the sampling bulb. Note the temperature. Connect one end of the sampling bulb to a burette containing neutral 10% KI and introduce as much of the solution as possible into the sampling bulb. Then close the stop-cock and shake the bulb vigorously to insure completion of the reaction. Empty the contents of the bulb into a clean Erlenmeyer flask, wash the bulb into the flask with distilled water and titrate the liberated iodine in an aliquot portion with standard N/10 sodium thiosulphate solution using starch as an indicator. Titrate another aliquot for HF formed with standard N/10 sodium hydroxide. Calculate

C-216 SAMPLING APPARATUS

DISPOSAL PLANT



NOTE: ALL TUBING $\frac{1}{8}$" COPPER

(78)

the percent C-216 from the sum of the two titrations, correcting the gas volume for deviations from standard conditions.

If the C-216 concentration is over 20% it is unsafe to make the determination directly. Instead the gas must first be passed through a clean dry salt tube before collection, to convert it to chlorine, otherwise the procedure is the same.

IVb. Outlet gases -

Connect the sampling line to the water ejector suction and purge for 5 minutes or until the collection of a representative sample is insured. The amount of C-216 can then be determined by aspirating directly from the sampling line into a Chrysler C-216 detector, using a one-hole rubber stopper to make the connection. See instructions with the detector for details.

NOTE:

The following material is included because a satisfactory short method of fluoride analysis suitable for use at the Disposal Plant has not yet been selected. The data presented may be useful in solving this problem.

7. Reports

Reports

A good set of records is of prime importance to the plant operator. The data collected is invaluable in determining better methods of operations, ways and means of reducing operating expenses, and settling controversies which may arise. For this reason suggested daily and monthly operating reports have been included.

In addition adequate maintenance records are desirable. A card index file with a separate card for each piece of equipment showing: date of installation, initial cost, dates and costs of repairs, and stock of repair parts needed will pay dividends.

A system of lubrication with adequate records and a log showing operating hours of each unit of equipment are also essential to good operation.

[redacted]

DAILY OPERATING REPORT
C-216 DISPOSAL PLANT

DATE:

Time	Caustic Flow G.P.M.	Heater		Temp Tower Eff. °F	Makeup Water G.P.M.	Caustic to Slaker				Lb/Hr	Steam		Air		C-74		Process Water		Liquid Levels			Ventilation	Turbine Dischg. Press	Line Vacuum (inches Hg.)	C-216 Inlets Max. Press. During Prev. Hr-In. Water			Remarks
		Inlet Temp °F	Outlet Temp °F			Flow G.P.M.	Inlet Temp °F	Outlet Temp °F	Temp Cooling Water Outlet °F	Lime Feed Rate	High Press PSIG	Low Press PSIG	High Press PSIG	Low Press PSIG	High Press PSIG	Low Press PSIG	Supply Press.	Booster Pump Dischg. Press.	Settling Tank	Tower	Lime Slaker				1	2	3	
1																												
2																												
3																												
4																												
5																												
6																												
7																												
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24																												

UNCLASSIFIED
Classification changed to: (level and category)
Gary W. Hall 10/19/95
ADC or ADD signature (first reviewer) Date
Sharon W. Kelly 10/19/95
ADD signature (final reviewer) Date

UNCLASSIFIED

Jas. W. Hill 10/19/55

W. Ailing 10/19/55

CONFIDENTIAL

C-216 DISPOSAL PLANT

DATE:

DAILY ANALYTICAL RECORD

BY:

LOCATION	ANALYSIS	SHIFT A								SHIFT B								SHIFT C							
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	
SETTLING TANK	% NaOH																								
	% F^-																								
	TURBIDITY P.P.M.																								
TOWER DISCHARGE	% F^-																								
	TURBIDITY P.P.M.																								
EXHAUST GAS	% C-216																								
INLET GAS	% C-216																								
REACTION TANK	% $Ca(OH)_2$ EXCESS																								

MONTHLY OPERATING REPORT

C-216 DISPOSAL PLANT

Pg. 1

Month:
Year:
By:

DATE	
MAX. C-216 LEAVING TOWER ppm.	
AVG. CAUSTIC FLOW G.P.M.	
C-216 USED BY CONDITIONING (lbs.)	
C-216 VENTED BY HOOKER (lbs.)	
TOTAL C-216 lbs./day	
TOWER BYPASS TOTAL HOURS	
LIME USED lbs./day	
AVG. SLURRY FLOW G.P.M.	
AVG. CAUSTIC TEMP. (TOWER FEED) °F.	
OUTSIDE TEMP. °F.	
VACUUM PUMPS HOURS OPERATED	
AVG. C-216 IN CAUSTIC LEAV. TOW. mg/l	
AVG. C-216 IN CAUSTIC ENT. TOW. mg/l	
EXCESS LIME %	
TURBIDITY OF REACTION MIXTURE	
TURBIDITY OF SETT. TANK EFF.	
SOLIDS IN SLUDGE %	
CAUSTIC IN SLUDGE %	
SLUDGE WASTED Gals.	
MAX. INLET PRESSURES Inches of Water — 1	
2	
3	

10/9/95

10/11/95

[redacted]

MONTHLY OPERATING REPORT

[illegible] PLANT

Month:
Year:
By:

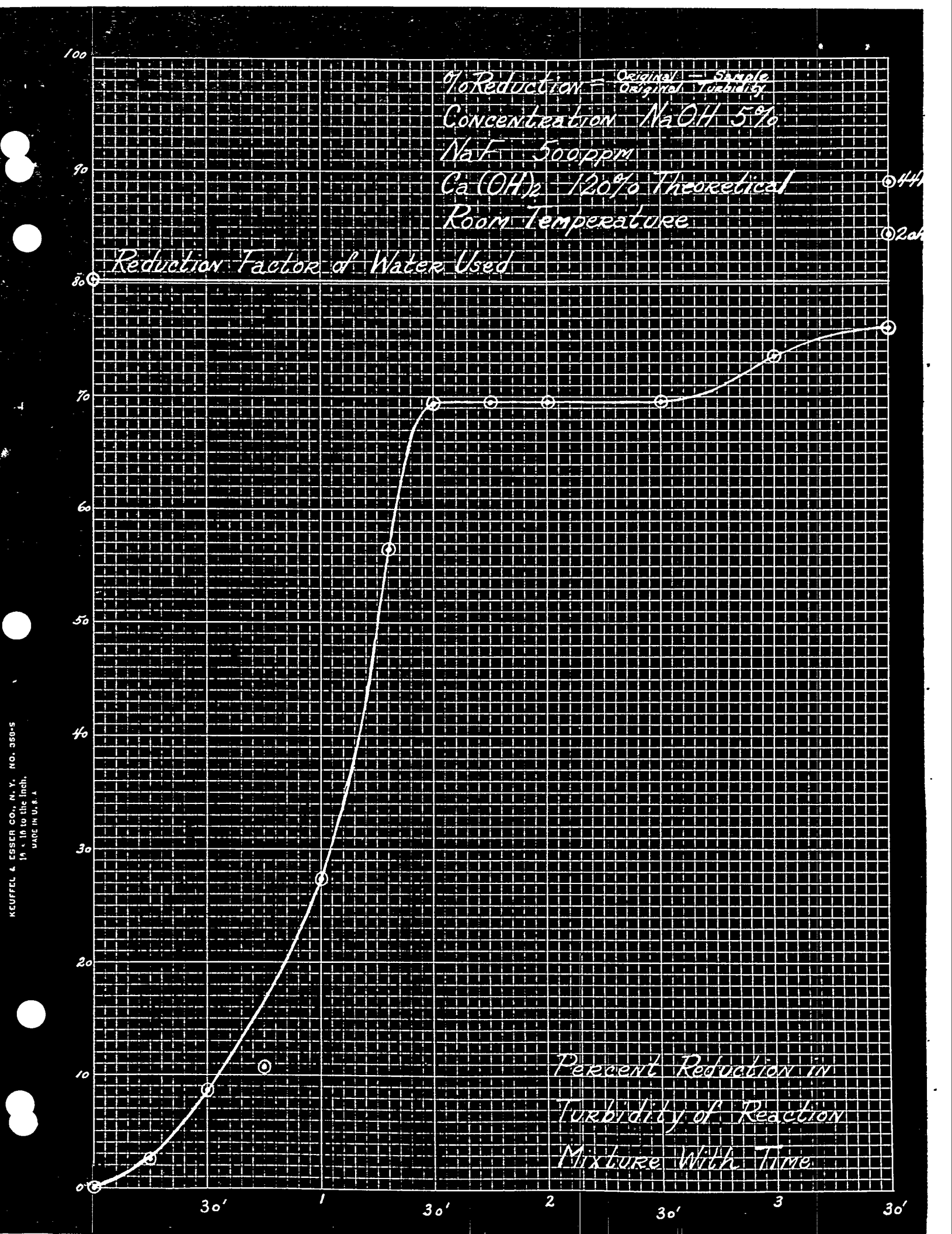
	Quantity	Unit	Unit Cost	Total
Estimated Power Consumption for Month		KW-H		
Estimated Steam Consumption for Month		Lbs.		
Total Lime Used		Lbs.		
Caustic Used		Lbs.		
Caustic on Hand at End of Month		Lbs.		
Salaries				
Maintenance and Repair				
New Construction				
G-311A Oil Used		Gals.		
Total Operating Costs				

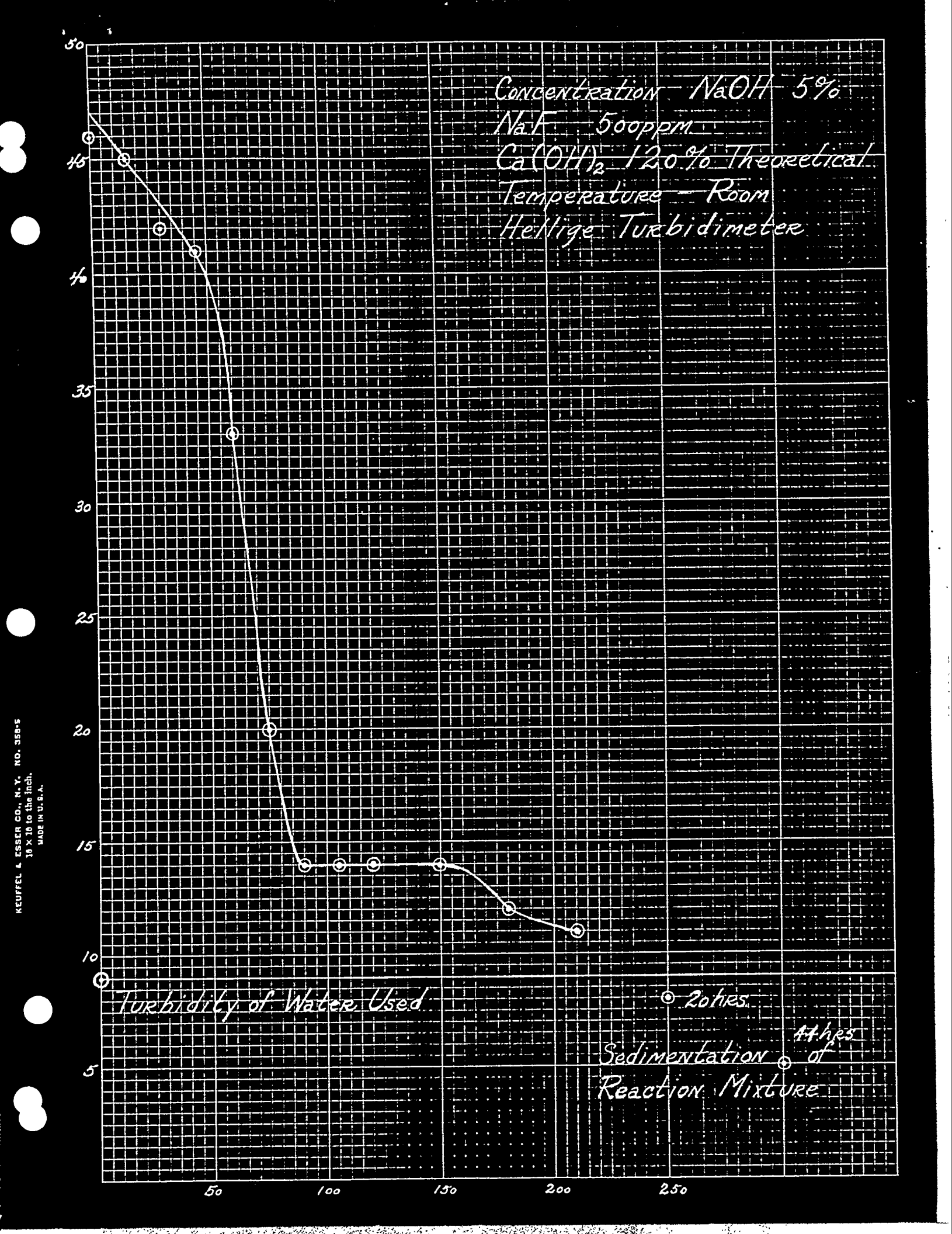
Classification changed to UNCLASSIFIED

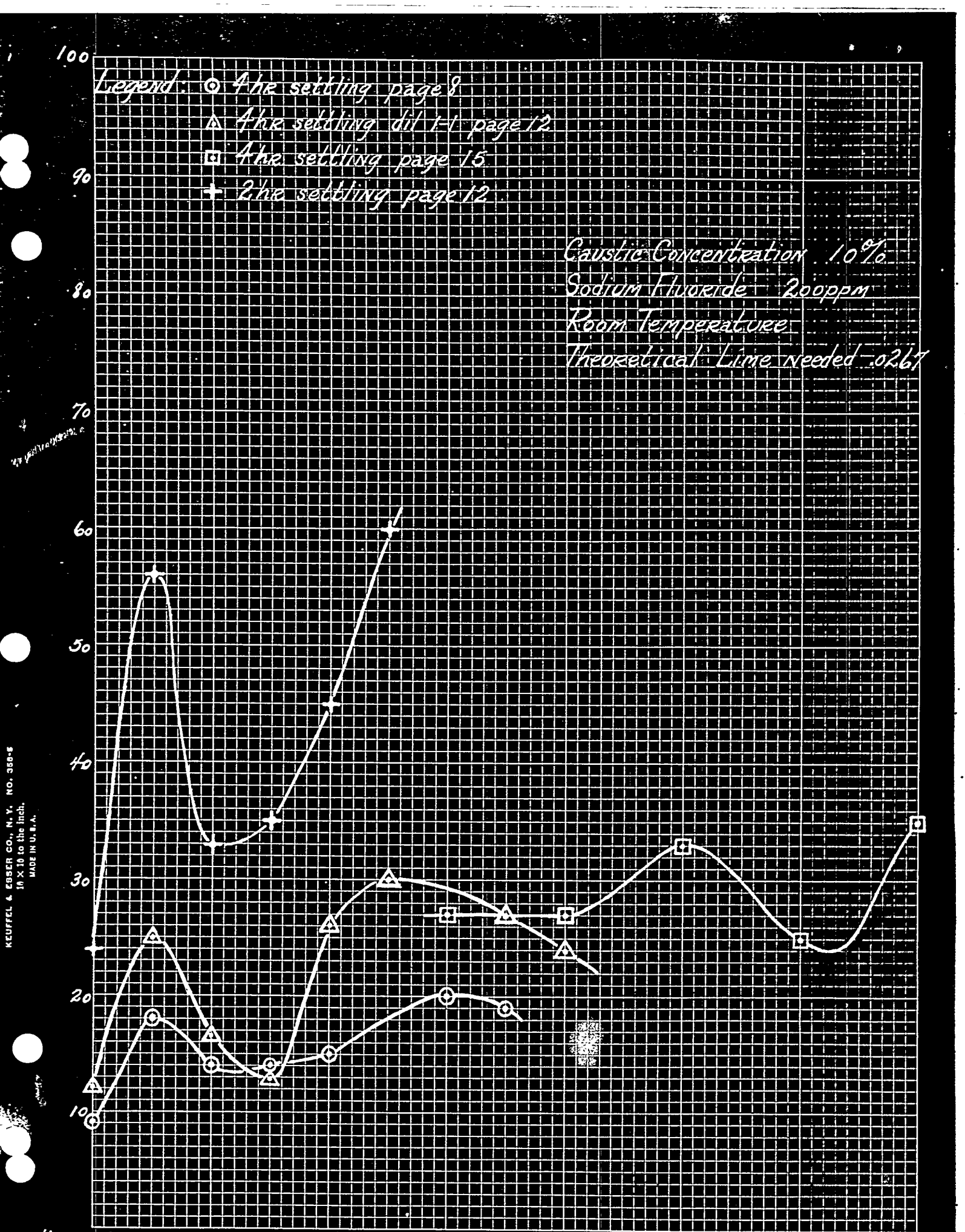
10/9/95

10/9/95

8. Useful Physical & Chemical Data


% Reduction = Original − Sample / Original Turbidity
Concentration NaOH 5%
NaF 500 ppm
Ca(OH)2 120% Theoretical
Room Temperature
Reduction Factor of Water Used
100
90
80
70
60
50
40
30
20
10
0
30'
1
30'
2
30'
3
30'
Percent Reduction in Turbidity of Reaction Mixture With Time


Concentration NaOH 5%
NaF 500ppm
Ca(OH)2 120% Theoretical
Temperature — Room
Hellige Turbidimeter
50
45
40
35
30
25
20
15
10
5
50
100
150
200
250
Turbidity of Water Used
20hrs.
44hrs
Sedimentation of
Reaction Mixture
KEUFFEL & ESSER CO., N.Y. NO. 358-S
10 X 10 to the Inch.
MADE IN U.S.A.



KEUFFEL & ESSER CO., N. Y. NO. 358-S
1/8 X 10 to the inch.
MADE IN U.S.A.

CALIBRATION OF CAUSTIC STORAGE TANKS

Ht. of Liquid Ft.	In.	Volume Gallons
0	6	424
0	9	756
1	0	1154
1	3	1605
1	6	2086
1	9	2608
2	0	3174
2	3	3751
2	6	4399
2	9	5035
3	0	5697
3	3	6383
3	6	7088
3	9	7804
4	0	8528
4	3	9265
4	6	9989
4	9	10,757
5	0	11,517
5	3	12,287
5	6	13,068
5	9	13,849
6	0	14,619
6	3	15,379
6	6	16,147
6	9	16,871
7	0	17,608
7	3	18,332
7	6	19,048
7	9	19,748
8	0	20,439
8	3	21,101
8	6	21,737
8	9	22,385
9	0	22,968
9	3	23,528
9	6	24,050
9	9	24,531
10	0	24,982
10	3	25,380
10	6	25,712
10	9	25,930
11	0	26,176

9. Maintenance

MAINTENANCE

The efficient operation of any plant depends not only on such factors as process control, analytical data, and well trained operators but also on adequate and properly planned maintenance. Although it is anticipated that maintenance work at the Disposal Plant will be accomplished by a separate department, the immediate responsibility for maintenance should be retained by the plant supervisor. He should know what constitutes good maintenance and demand it.

Manufacturers' instructions pertaining to the various items of plant equipment have been obtained and are on file at the plant. These should be reviewed and supplemented as desired. Construction drawings are available in the Kellex manual, "Final Operating Instructions for North Area".

A few maintenance tips are given in the section devoted to operation in "Duties of Shift Supervisors".

Close attention should be given to the care of the centrifugal pumps. Packing glands should be tight enough to stop caustic leakage, but not too tight to permit free rotation of the pump shaft by hand. The packing selected should be recommended for caustic service and high speed - (3600 rpm) operation. The use of metallic packings is not recommended. It is true that repacking will be required less frequently with this type of packing, but scoring of shaft sleeves from excessive friction is less easily avoided. A good grade of braided asbestos, graphited and treated with a caustic resistant grease, or a plastic packing, similarly treated is recommended. Packing rings should be cut accurately to size and replaced one at a time with alternate rings staggered 90° or 180° with respect to the junction between the ends of the packing. Ends

should be cut square, not beveled. The lantern ring should be properly centered to match the grease opening from the lubricator. Lubricant should be applied until it is forced out between packing and shaft. Then a little extra lubricant should be forced out on each shift with two or more turns on the lubricator.

Electrical controls and motors should be freed from dust at periodic intervals depending on conditions, preferably once each week. The dust acts as an insulator and prevents proper cooling of the windings in motors and starter coils, thus shortening the life of the equipment. This can be most easily accomplished by blowing with dry air.

Lubrication of all bearings should be accomplished according to manufacturers' recommendations on a systematic schedule. Over-lubrication of anti-friction bearings should be avoided because it causes overheating. Lubricant should not be selected for use at higher temperatures than those actually encountered.

Leaks of any description should be stopped as soon as possible. Bellows type C-216 valves should be tested for leaks yearly. The seal connection can be used for this purpose.

In accition to the above the following texts are recommended:

"Lubrication" - Glower McGraw-Hill
"Pumps" - Krystal & Annett McGraw-Hill
"Wartime Care of Centrifugal Pumps" - Allis Chalmers Mfg. Co.
" Wartime Care of Electric Motors" - Allis Chalmers Mfg. Co.
"Catechism of Electrical Machinery" - Fairbanks Morse & Co.
"Piping Pointers" -Crane Co
"Lubrication of Anti-Friction Bearings" - Tide Water Oil Co.

The above mfgrs. publications may be obtained free of charge.

In conclusion, an important principle of good maintenance is to have a clean, neatly painted, and orderly plant. It will pay dividends.

10. Safety

SAFETY RULES

C-216 DISPOSAL PLANT

1. Remember that 90% of all accidents are avoidable.

2. Wear rubber gloves and goggles when handling caustic pumps and valves or unslaked lime.

3. Wear asbestos gloves and metal frame goggles when handling C-216 pumps.

4. Learn the proper use of gas masks and their limitations.

5. Let the electrician repair electrical equipment.

6. Report all accidents immediately to your supervisor.

7. For treatment of either C-216 or caustic burns wash the affected part of the body at once with large quantities of water, then go to the dispensary for check up or treatment.

8. Stay out from under the hoist regardless of whether or not it is in operation.

9. Avoid contact with uninsulated parts of steam lines and valves.

10. In case of C-216 leaks inside the bldg. leave the bldg. at once and arrange with Hooker or Conditioning to shut off the gas at its source immediately.