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# Weak Ammonia Liquor Stripping Facility
# Operating Manual
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INTERLAKE, INC.
WEAK AMMONIA LIQUOR STRIPPING FACILITY
CHICAGO COKE PLANT

OPERATING MANUAL

Dravo
Still

DRAVO/STILL

INTERLAKE, INC.

WEAK AMMONIA LIQUOR STRIPPING FACILITY
CHICAGO COKE PLANT

# OPERATING MANUAL

DRAVO/STILL 8040

Dravo Still

DRAVO/STILL

# Operating Manual
# for the
# Weak Ammonia Liquor Stripping Facility
# at
# Chicago, Illinois
# for
# Interlake, Inc.

Dravo Still

# TABLE OF CONTENTS

## 1.0 Process Description

Refer to Drawings C-6651, C-6652, C-6653, C-6654 and C-6655.

## 1.1 Overview

The Dravo/Still weak ammonia liquor stripping system consists of a steam stripping column (ammonia still) for both the free and fixed ammonia, a caustic soda storage tank, caustic unloading facilities, pumps, weak ammonia liquor storage tank and instrumentation. In this plant, the ammonia, both free and fixed, is steam stripped from the weak ammonia liquor. Caustic is added to the column to liberate the fixed ammonia. Future provisions have been made to add a second ammonia still as back-up to provide a 100% spare should the need develop.

## 1.3 Ammonia Stills

The ammonia still design used in this plant is unique in order to achieve a low effluent NH3 concentration. The still is one continuous, valve-trayed column for steam stripping weak ammonia liquor feed. The still contains twenty-two valve trays and one blank tray to facilitate the total vapor bypass. Caustic is fed to the column on Trays #7 and #13 to liberate "fixed" ammonia by adjusting the pH of the weak ammonia liquor. Also, a total vapor bypass from Tray #13 to Tray #7 of the column is provided to incorporate a two-stage stripping action for the fixed ammonia.

The still uses steam fed at two locations to strip the ammonia from the weak ammonia liquor. One steam feed is at the base of the still, and the other feed is above the blank tray (Tray #12).

Weak ammonia liquor is fed to the top of the column, and 50% caustic soda is added at Trays #7 and #13 (counting from the top) to liberate the "fixed" ammonia. The overhead vapor leaving the top of the still contains the stripped ammonia and flows to the existing 36" COG upstream of the existing ammonia absorber. In the ammonia absorber, some excess water from the vapor will condense, and the ammonia will be absorbed in sulfuric acid to produce ammonium sulfate solution which is sold. Liquor feed rate to the column from the new ammonia liquor pumps, one operating - one spare, is controlled via a panel-mounted controller. Total steam flow to the still is controlled via a panel-mounted controller to hold the column overhead temperature and ensure the proper ammonia removal. The individual flow rates on the split steam injections to the still are manually adjusted. Caustic addition to the column is on ratio control, based against the weak liquor feed rate to Tray #7. The still effluent pH is used to determine the caustic flow to Tray #13.

Steam usage for the still is reduced by use of a (heat pump) steam ejector to recover heat from the still bottoms. The ejector will lower the effluent liquor temperature from approximately 220 Degrees F to 190 Degrees F. The steam ejector recovers energy from the still effluent as steam and returns it to the ammona still and, thereby, reduces the steam required for the system.

## 1.3 Caustic Soda Storage and Feed

Caustic soda (50 wt % solution) for the ammonia stills is provided from an existing 15,440 gallon storage tank which has been relocated. The tank is equipped with electric panel heaters which maintain the caustic at temperature.

Facilities are provided for unloading caustic tank cars to the storage tank. Two new pumps, one operating and one spare, provide caustic soda to the ammonia still.

## 1.4 Weak Ammonia Liquor Storage

The weak ammonia liquor feed to the still system is by way of liquor storage tanks. In these tanks, the liquor is permitted to stand in order to settle the tar. The weak ammonia liquor feed to the still must be free of tar, to the greatest extent possible, in order to avoid fouling of the valve trays. The potential for the future installation of additional liquor storage, and thereby increased liquor settling to remove tar, has also been provided.

1.0 Process Description - Contd.

## 1.5 Still Cleaning Requirements

Use of caustic soda reduces the frequency for periodic cleaning of the stills due to solids accumulation. However, over time, the tar contained in the feed liquor will polymerize and accumulate in the the column. When this occurs, the accumulated tar will need to be removed by manually cleaning the column and trays.

## 1.6 Design Basis

| | | | |
|---|---|---|---|
| 1. | Ovens pushed per day | 140 | |
| 2. | Tons of coal per oven | 16.9 | |
| 3. | Coal moisture % | 8.5 | |
| 4. | Concentrations in W.A.L. to stills | | |
| | Free ammonia | 0.5 | g/l |
| | Fixed ammonia | 3.0 | g/l |
| | Total cyanide | 200 | mg/l |
| 5. | W.A.L. generated from all sources exclusive of direct steam to the NH3 stills | 120 | GPM |

### Effluent Quality

| | | | |
|---|---|---|---|
| 1. | Total ammonia | 50 | mg/l |

# 2.0 Operational Tests and Commissioning

## 2.1 Preparing the Plant for Commissioning

### 2.1.1 Checking the Plant

A detailed and careful check of the plant shall be made before commissioning is undertaken. This check is made by the responsible project engineer and operating foreman during the final stages of erection and before the plant is reported to be "mechanically complete" and ready for initial operation.

In this way, defects can be eliminated simultaneously to completing the remaining erection work. Any supplements or modifications, if desired, can be carried out without delaying the commissioning. When checking the plant, the following items are to be observed:

- Inspection of tanks before closing the manhole covers with regard to cleanliness, completeness and correct installation of internals.

- Examining all pipelines for proper connection, correct routing and good operation of valves.

- Checking the measuring and control devices for their proper position, measuring range, description, correct connection and operational direction (safety position, spring opens or closes) as well as agreement between valve position and valve position indication.

These inspections are made in addition to the pre-operational tests carried out during erection by the contractor.

Engineering and operation shall be responsible for checking the plant and shall be convinced that all equipment is in the proper condition and installed in a suitable way before accepting the plant from the constructor.

2.0 Operational Tests and Commissioning - Contd.

2.1.2 Flushing the Plant

The plant shall be cleaned of coarse contaminants by the constructor personnel while completing the erection work.

This includes:

- Removal of erection equipment, tools, etc. on site as well as on platforms. The site should be in such condition that it is possible to inspect all locations safely.

- Cleaning the equipment, vessels and pipelines of erection materials, miscellaneous contaminants (e.g. sand), temporary supports and auxiliary structures for erection, transportation, etc.

It is extremely beneficial if the careful flushing of the plant during commissioning is performed by the future operators. In this way, the operators become more familiar with the individual plant units, and by working with the equipment, they may realize and correct future problems.

Flushing shall be completed in stages according to a flushing program developed by the responsible engineer. The flushing routes are selected in such a way that media flows through the pipelines from top to bottom in order to dispose of contaminants in the flushing media by gravity flow. Any connecting pipes to machines are to be blanked off carefully. Tanks or equipment which are not affected by the discharged contaminants may be used as low points for the connected pipeline discharge. The flushing media can then be discharged from the bottom drains of these tanks. Subsequently, the manholes of these tanks should be reopened in order to thoroughly clean them.

For the duration of pipeline flushing, the orifices and flowmeters should be removed in order to avoid reduced velocity of flow as well as deposits of contaminants upstream of this equipment and to protect these instruments from damage.

2.0 Operational Tests and Commissioning - Contd.

Instead of the orifices, removable discs of the full pipeline cross-section should be installed.

Control valves may be removed and replaced by spool sections or blanked and by-passed. Instrumentation measuring lines are closed or dismantled and safety valves blanked off.

Water is to be used as the purging medium.

Surface lines and plant drainage systems should be completed at this stage and able to discharge the flushing water obtained without flooding. Some lines, e.g. instrumentation air lines, are carefully purged clean with dry air rather than by water.

Here, too, it is not enough to make a brief blow-through but to continue purging the gaseous or vaporous flushing media through the piping until the media leaving the piping is clean.

Water purgings may be partially superceded or even supplemented with steam purging. The overhead vapor piping from the still is a good example. As a result of the thermal expansions that occur with this type of purge, any rolled skin or welding cinder spall off would be removed from the system with the purge rather than with the hot product.

2.1.3 Operational Tests and Test Runs

Operational tests and test runs of equipment are initially carried out as dry runs, if possible. If flow of a medium is required, a harmless one is selected and passed through the flushing systems. For example:

- Trial runs of pumps with water

- Trial runs of compressors with air

- Running-in of control systems for combustible gases with air

Defects encountered during this operation will be harmless and not lead to an accident. Access to the equipment for repair or replacement of failures can be obtained relatively quickly and without difficulties.

The first phase of testing the equipment with non-dangerous flushing or process media is followed by the second phase, circulating these media. Temporary strainers are installed upstream of sensitive equipment and other locations. Typically, these locations include pumps, tank discharges, still inlets, etc.

The temporary strainers must be cleaned often to ensure free flow.

It is still possible to eliminate any weak points during the first extended process circulating period.

Orifices and flowmeters are removed from the pipeline, and the control valves are to be by-passed. Initially, the instrumentation connections are closed except for those which are not jeopardized by the circulating media. Examples of this are level indicators which supply valuable information regarding the fluid level. With the cleanliness of the plant systems improving, operational tests based on approximate process conditions are carried out for all measuring, control, alarm and safety devices. If possible, alarm and switch points are set and verified at this stage.

The data during this circulation period is to be carefully recorded!

The flushing media are discharged as soon as there is trouble-free operation of the equipment and when the temporary strainers remain clean.

2.1.3.1 Operational Tests and Test Runs with Flushing Media

The essential procedures of this step of commissioning are summarized in the following chart:

2.0 Operational Tests and Commissioning - Contd.

1. Check the equipment and auxiliary systems for cleanliness and remove any contamination.

2. Install temporary strainers (small mesh size) and use them for trapping the dirt.

3. Check auxiliary systems for leakages and fill the required equipment with lubricants.

4. Activate the power supply for the respective equipment and check rotation.

5. Put the auxiliary systems of equipment into operation and observe as well as check them for trouble-free functioning.

6. Check the free running of shafts, as well as the bearing and coupling clearances. Ensure that pipe is connected free of stress.

7. Activate the equipment and complete a test run for a maximum of one hour. Monitor the equipment for extra-ordinary temperature increases of bearings or shaft gaskets, running smoothly, pressure drop at the temporary strainer.

8. Remove and clean the temporary strainers.

9. Carry out longer test runs in the same fashion, e.g. for compressors increase the running time step-wise to 8 hours and then 24 hours.

10. Check the shut-off devices for proper functioning to detect any blockages or clogging due to foreign bodies or deposits. Remove and clean the valves if required.

11. Circulate harmless media.

12. Run operational tests with all measuring, regulating, control and safety devices.

2.0 Operational Tests and Commissioning - Contd.

13. Adjust the alarm and switch functions to the specified values.

14. Heat-up the media in the circulations and allow them to cool in order to break up the welding cinders, rust formations, rolled skins, etc. Adjust other measureing, regulating, control and alarm functions at higher temperatures.

15. Discharge the flushing media from the plant. The work should be perfomred by the operating personnel and the plant maintenance personnel who will maintain the equipment in the future.

2.1.4 Total Pressure Test

After draining the media, all opened drains and vents are closed, and by using air, the entire plant is adjusted to a pressure which is suitable for all the equipment.

A precondition of this test is that all parts have undergone the required water pressure test at an earlier stage of erection.

Flanged connections at all apparatus and piping are to be tightened and checked for leakage by brushing or spraying suitable leak-detection solutions. This is generally done at the completion of erection and prior to commissioning.

2.1.5 Purging the Plant

In plant units where little or no oxygen is allowed, and after the total pressure test is complete, purging the atmosphere inside the equipment is performed. An inert gas is injected at locations which have been predetermined at the design stage, or at locations which are determined during "plant checking." A clear flow of gas through partial systems is achieved by opening the valves at other adequate locations with the air being evacuated in this way. Care is to be taken to avoid excessive flow velocities and a mixture of atmosphere and inert gas "pushed forward." Thus, the air is evacuated.

This procedure is continued until a decrease of oxygen content (or of another important process value) below the allowable limit is analyzed at all venting points through the system. When the purging is completed, the plant unit is kept at a slight, positive pressure in all sections to prevent re-penetration of air. A leakage check is to be made again if there is evidence of a pressure drop.

## 2.2 Start-Up of the Plant

### 2.2.1 General

Some essential and general characteristics apply with respect to schedule, organization and plant preparation for commissioning.

A premature filling of the product, i.e. before the planned preparatory work has been satisfactorily completed, should not be permitted.

Each person involved in the start-up is to receive a copy of the written start-up description which must include a clear-cut distribution of functions and responsibilities.

A training course for operators and their superiors should be held prior to commissioning. Special attention should be paid to the unique features of the new operating unit.

The "hot" phase of start-up, with the product charged, is planned in single steps.

### 2.2.2 Commissioning of Ammonia Stripping

It is assumed that the ammonia still has been operated during operational tests and that the bolts have been re-tightened.

## 2.0 Operational Tests and Commissioning - Contd.

The most important prerequisite to commissioning steam-operated distillation columns ammonia still is slow heating-up without experiencing water and/or steam shocks. These shocks are caused by steam condensation and can involve so great a pressure wave, one cannot dismiss the possibility of material destruction.

A small amount of liquor (approx. 15 gpm) is fed to the ammonia still and 10 psig steam by way of PCV-2 is added manually through the 6" valve, 6" VGL041, upstream of the steam distribution at the stripper.

As the still temperature increases, more liquor and steam are added in step fashion corresponding to the temperature increase by observing the vessel temperature and sump temperature, TR-1, TR-2, and TI-101.

The steam ejector, including controller, FV-1, is not to be utilized at this time.

This increase in steam and liquor feed is to be done slowly to avoid the steam shocks mentioned earlier.

Steam is charged to the lower section of the ammonia still through the direct steam inlet line 12" S10-8-13 of the still. The quantity is adjusted by the 12" hand valve, VBY003.

The water in the still warms up in accordance with the steam charge. Thereby, the still is heated smoothly.

The vessel is heated until a temperature of approx. 98 Degrees C (209 Degrees F) at the still top (TR-1) is achieved.

### 2.2.3 Detailed Start-Up of Ammonia Still

This section presents a general guide for initial start-up but local preference or specific conditions may require revisions.

2.0 Operational Tests and Commissioning - Contd.

Before start-up, all lines and vessels will have been pressure tested, and all instruments will have been calibrated. All valves within battery limits shall be closed. Piping will have been checked for location and continuity.

Prior to start-up, check 1) instrument air to all components and check for proper control valve action, 2) open all valves to pressure indicators, pressure transmitters, upstream and downstream block valves for control valves (by-pass valves to be closed), low and high-pressure valves to flow transmitter devices, 3) check vendor information for proper operation of pressure reducing and flow control station.

<u>Sequence to Put Ammonia Still in Operation</u>

A. Set ammonia liquor flow

1. Open weak ammonia liquor tank valving from draw-off nozzles on storage tanks to the ammonia still feed pumps (41002).

2. Select one ammonia still feed pump for operation and open valve on suction line to pump.

3. Open valving on discharge of ammonia still pumps to the ammonia still. Set flow controller FIC-3 to 15 gpm with by-pass around the controller closed. Set ammonia liquor flow to top tray of ammonia still. Liquor flow to Tray #2 should be closed.

4. Open discharge flow from ammonia still to lime settling basin. pH probe and filter system are to be valved closed.

5. Start ammonia still feed pumps.

Note: The by-pass line for the weak ammonia liquor around the ammonia still will be utilized to isolate the ammonia still as required by operations. From this time on, the existing weak liquor pumps should not be utilized to pump liquor to the lime settling basin after this system is commissioned.

6. After establishing liquor flow to the lime settling basin through the ammonia still for a minimum of 1 hour, proceed to establish the steam flow on the still.

B. Ammonia Still Steam Flow

1. Open vapor valve 14" VGA 625 (at coke oven gas tie-in) in line 20"-14" AV-63-21.

2. Set steam flow to ammonia still through PCV-2. PCV-2 is to be set at 10 psig by-passing the steam ejector. Slowly open steam flow to the ammonia still below Tray #23 using 12" VBY 003.

3. As the temperature of the vapor leaving the ammonia still will begin to rise, the systems heat up. When the top of the ammonia still is approximately 98 Degrees C, open steam flow through FV-1 and steam ejector 42001. With still vapor line to ejector closed using valve 8" VGA603 in line 8" AL-12-15 slowly set TIC 3 at 98 Degrees C.

4. Close off steam flow to ammonia still through PCV-2 using valve 6" VGL-041 in line 10" S10-8-12. FIC-1 should begin to control steam flow to ammonia still based on top temperature (98 Degrees C) of ammonia still.

5. Slowly open vapor line (8" AL-12-15) from ammonia still to steam ejector. OPENED IN #3

6. Check all instrumentation on ammonia still.

2.0 Operational Tests and Commissioning - Contd.

7. Slowly increase the weak ammonia liquor flow to the ammonia still by FIC-3 to 100 - 120 gpm.

8. Slowly open steam flow to ammonia still below Tray #11, adjust flow to 5,000 lb/hr using 8" VBY 003 and reading FE-2. This flow will be adjusted during commissioning.

   The weak ammonia liquor temperature at the bottom of the still should be approximately 105 - 110 Degrees C as measured by TR-2.

Note: The ammonia still is now operating as a free still only. The caustic additions must now be started to adjust the pH of the weak ammonia liquor in order to remove fixed ammonia. The still should not be operated as only a free still for extended periods of time in order to avoid corrosion in the bottom of the still.

C. Caustic System

1. Open caustic storage tank valving from draw-off nozzle to caustic pumps 41001.

2. Select one caustic pump for operation and open valving on suction side of pump.

3. Open valving on discharge of caustic pumps to the ammonia still through FE-4. Set flow controller (FFIC-4) at 2 gpm and be sure by-pass valves are closed. Caustic flow is introduced to the ammonia still below Tray #6. Open caustic recycle line 1 1/2 CA-130-27.

4. Start caustic pumps.

5. Open valve for slip stream of discharge ammonia still flow to pH probe cooler and filters after introducing cooling water to cooler. Set pH controller at 10 to 11 pH. This will be adjusted during start-up.

6. Open caustic flow through FE-5 to ammonia still on Tray #13.

D. Shutdown

To take the ammonia still off line, the flow of weak ammonia liquor is diverted directly to the lime settling basin. Stop the flow of stripping steam to the still. Stop the flow of caustic to the still. Block off the appropriate valves, steam, vapor, caustic, liquor, etc. Maintain circulation of the caustic storage system. In freezing weather, drain or bleed freeze points in the piping and check the heat-tracing system.

## 3.0 Analytical Tests

The plant must be prepared to perform analysis of the weak ammonia liquor (inlet and outlet to the ammonia still) in order to make adjustments to the still operation to achieve the desired performance. Attached are two procedures which we have determined through experience to be appropriate for coke plant liquors. The first procedure (see 3.2) is applicable for the inlet weak ammonia liquor with higher ammonia concentration. The second procedure (see 3.3) is applicable for ammonia nitrogen concentration above 5 mg/l and is to be used for stripped weak ammonia liquors.

3.1 The plant must be able to check the pH of the stripped ammonia liquor independently to verify the in-line pH probe and to maintain calibration.

## 3.2 Analysis Procedure - Weak Ammonia Liquors

NH3 Determination in Water Solution

Required Equipment and Chemicals

Test bottles which can be closed
Pipettes 1, 2, 5, 10, 20, 25, 50 ml
Wide-neck Erlenmeyer flasks 300 ml, 500 ml
1 complete distillation equipment
Burette 1/10 ml graduations
Spray bottles
Caustic soda solution 20%
Sulfuric acid 1 N, 1/10N

Determination Method:

Total Ammonia — Procedure No. 1

A certain amount of test solution is boiled with 50 ml of 20% caustic soda solution in the distillation equipment. The freed ammonia is collected in 1 N H2SO4 and back-titrated with 1 N caustic soda solution while using a pH indicator.

Calculation

$$\frac{\text{ml H2SO4 consumed} \times 0.017 \times 1000}{\text{ml of sample}} = \text{g NH3/1 of sample}$$

3.0 Analytical Procedures - Contd.

Volatile Ammonia Procedure No. 2
(weakly bound or free ammonia)

The distillation and titration is done as described above, however, without adding the caustic soda solution. When making tests during operation, it is possible to have direct titration without first distilling.

Fixed Ammonia Procedure No. 3

Total ammonia-volatile ammonia = fixed ammonia

Empirical value

| | |
|---|---|
| Deacifier Bottom | $NH_3$ use 5 ml direct without distillation/ 7.05 ml 1 N $H_2SO_4$ 24 g/l |
| H2S Scrubber Bottoms | $NH_3$ use 2 ml direct without distillation/ 6.95 ml 1 N $H_2SO_4$ 23.7 g/l |
| Ammonia Scrubber Bottoms | $NH_3$ use 5 ml direct without distillation/ 5.60 ml 1 N $H_2SO_4$ 9.5 g/l |
| Mix Cooler | $NH_3$ use 2 ml direct without distillation/ 11.40 ml 1 N $H_2SO_4$ 37.7 g/l |
| Strong Liquor | $NH_3$ use 1 ml direct without distillation/ 12.40 ml 1 N $H_2SO_4$ 211.0 g/l |

Example: $\frac{7.05 \times 0.017 \times 1000}{5} = 24.0$ g/l

3.3 Analysis Procedures - Stripped Weak Ammonia Liquor

AMMONIA NITROGEN

Analysis Procedure
(Ammonium Nitrogen Specification)

3.0 Analytical Procedures - Contd.

A. General

Ammonium nitrogen can be found in many surfaces, some ground water, as well as in homes and often in industrial and commercial waste waters. The form in which ammonium nitrogen occurs in water, whether as NH4 ion or NH4OH, i.e. NH3, depends on its pH value as can be seen from the diagram at the end of this exhibit (Figure 1).

B. Analysis Following Prior Distillation

1. Theory

The ammonium nitrogen contained in the sample is distilled as ammonia, collected in a boric acid condensate tank, and analyzed. The ammonia can also be collected in sulfuric acid with the excess being titrated back.

2. Application

The method is applicable to measure ammonium nitrogen in concentrations above 5 mg/l.

3. Interruptions

Urea and other acid amides interrupt the measurement due to the separation of ammonia during the distillation with heavy alkaline solution. To reduce this error, the distillation takes place with a pH value of 7.5.

4. Instruments and Chemicals

Distillation instruments with condensate tank, content of distillation flask approximately 1 liter.

Phosphate buffer solution: 14.3 g potassium dihydrogen phosphate, KH2PO4, p.a., and 68.8 g dipotassium hydrogen phosphate, K2HPO4, water free, pure, are dissolved in distilled water; the solution is filled up to 1 liter with distilled water.

3.0 Analytical Procedures - Contd.

Boric acid solution: 40 g boric acid, H3BO3, p.a., and 1 liter ammonium carbon dioxide free distilled water.

Mix indicator solution: Solution a) 30 mg methyl red and 100 ml ethanol; Solution b) 100 mg methyl blue and 100 ml distilled water; 100 ml of solution a) are mixed with 15 ml of solution b).

Sulfuric acid 0.05 N

Manufacture of ammonium free water:

1. 1 liter of distilled water is distilled by addition of 3 ml of an alkaline potassium permanganate solution, which was made by dissolving 10 g potassium permanganate and 50 g sodium hydroxide. The first 200 ml of the distillate are discarded.

2. The distilled water is allowed to have a strong, acid cation exchange in the H form.

5. <u>Experimental</u>

500 ml or less of the filtered water sample is filled into a distillation flask and neutralized, if necessary. If a lower volume is used, it is replenished with ammonia free distrilled water to 500 ml. Following addition of 25 ml phosphate buffer solution, approximately 200 ml are distilled over in a condensate tank filled with 50 ml boric acid solution, where the cooler end is submerged into the condensate tank. Three drops of mix indicator solution are added to the distillate. The ammonium ions are titrated for a color comparison with 0.05 N sulfuric acid versus a blank sample of 50 ml of the boric acid solution filled to the same volume with ammonium free, carbonic acid free distilled water. The indicator color changes from purple to green.

3.0 Analytical Procedures - Contd.

6. Evaluation

1 ml 0.05 N sulfuric acid correspondends to 0.70 mg ammonium nitrogen. Calculation:

$$\frac{a \times 0.70 \times 1000}{b} = G$$

Expressions used in the formula:
a - consumption of 0.05 N sulfuric acid in ml
b - volume of the used water in ml
G - ammonium nitrogen (N) content in mg/l of the sample

7. Description of Results

The amounts are rounded off to 0.1 mg/l
Example:
ammonium nitrogen (N) = 9.3 mg/l

Conversion Factors

| | mg NH4 | mg NH3 | mg N | umol N |
|---|---|---|---|---|
| 1 mg NH4 corresponds to | 1 | 0.94 | 0.78 | 55.44 |
| 1 mg NH3 corresponds to | 1.06 | 1 | 0.82 | 58.72 |
| 1 mg N corresponds to | 1.29 | 1.22 | 1 | 71.39 |
| 1 umol N corresponds to | 0.018 | 0.017 | 0.014 | 1 |

3.0 Analytical Procedures - Contd.

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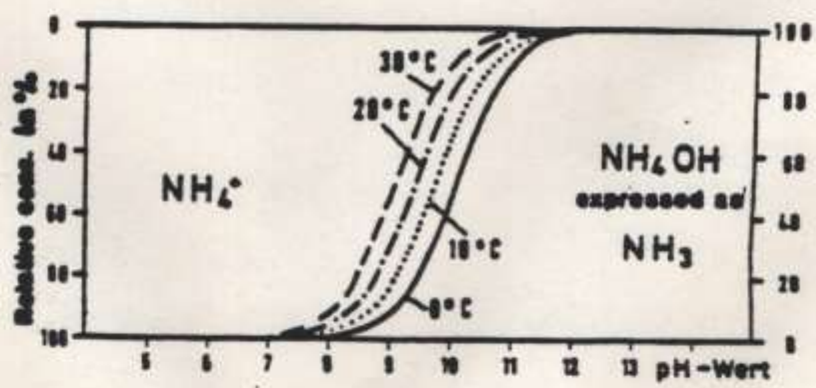



Figure 1

## 4.0 Normal Operation of the Plant

### 4.1 Ammonia Still Plant

The head temperature of the ammonia still is the single most important control value.

As determined by the water analyses, the head temperature of the deacifier must be increased or decreased. Thereby, the concentration of free ammonia in the deacified water is decreased or increased. The head temperature may only be changed in small steps of up to 0.5 Degrees C.

Another prerequisite to steady head temperature is a stable supply of steam. Therefore, the operations of the flow controller, FIC-1, must always be monitored. The ammonia still head temperature is automatically trimmed by TIC-3.

The feed temperature of the weak ammonia liquor charged to the ammonia still can also effect ammonia concentration in the effluent liquor. Changes of flow result in altered temperatures and thus greatly influence the head temperature.

The facility is equipped with a number of metering devices. During normal operation, they should indicate nearly constant values depending on the load. Data recording indicate any disturbance immediately.

### 4.2 Caustic System

50% caustic will be received by tank truck. Detailed instructions on the safe tne proper procedure for handling and unloading caustic are available from the supplier and must be followed in detail. Safety showers and eyewash stations are available in the sulfate building for immediate flushing of any caustic that accidentally contacts the body of any person in the area.

The caustic soda is unloaded from tank truck to the caustic storage tank C-120 with a capacity of 15,440 gallons. The storage tank liquid level is indicated locally and must be observed by the operator for stock purposes and to prevent overfilling.

4.0 Normal Operation of the Plant - Contd.

The temperature of the caustic solution, in the storage tank and in circulation, is maintained automatically by electrical heaters. (CAUTION: The freeze point of 50% caustic is 51 Degrees F.) Tank trucks are equipped with steam coils, and the caustic will have to be heated to approximately 80 Degrees F in cold weather before unloading. The stress corrosive cracking of carbon steel with caustic becomes critical above 140 Degrees F. The temperature in the tank is to be kept below this set point.

The caustic feed pumps (41001-1, 2, one operating, one spare) pump caustic from the storage tank, through a recirculating loop which returns to the tank. Caustic added to the ammonia still is bled off this recirculating loop. A flow-restricing orifice is installed to insure adquate back pressure in the recirculation loop to feed caustic to the ammonia stills.

All the caustic lines are heated to prevent freezing and help maintain the process temperature. The tracing, along with the storage tank heater, will prevent the caustic from falling below 80 Degrees F during operation. If for any reason circulation flow cannot be maintained or heating ability is lost, caustic lines should be blown clear with compressed air and to drain low points. Compressed air should be used to avoid temperatures over 140 Degrees F or exothermic reaction with water or steam.

4.3 Operating Parameters

NOTE: The following figures are from design calculations and may change when the units are put in operation and fine tuned.

Ammonia Still

| | |
|---|---|
| Feed - | 100 GPM |
| Steam - | 9,000 LB/HR |
| Top Temperature - | 208 Degrees F (98 Degrees C) |
| Bottoms Temperature - | 220 Degrees F (105 Degrees C) |
| Caustic Addition - | 1.5 GPM |
| pH Still Effluent - | 10.5 pH |

4.0 Normal Operation of the Plant - Contd.

<u>Caustic System</u>

Circulation Temperature 80 Degrees F

## 5.0 Troubleshooting

The operating data gathered during the initial operating period is to be retained. This information will serve as a benchmark upon which to gauge future operating performance and to indicate when some element of the still system is not operating properly.

The following are examples of possible difficulties which might be experienced during the operation of the ammonia still and a likely corrective action.

1. High-pressure drop across the still due to fouling of the valve trays. This condition will most likely result from an accummulation of tar. The temperature of the still can be raised by removing the liquor feed while maintaining steam flow. This step is intended to heat the tar, lower its viscosity, permitting it to flow and free up the valve trays. This action will also encourage accelerated polimerization of the tar. If this step does not lower the pressure drop across the column, the still and trays should be manually cleaned.

2. Increased ammonia concentration in the still effluent. Check still top temperature, steam flow and still effluent pH. These values can be raised in an effort to lower ammonia in still effluent. If difficulties continue, it is possible that chaneling in the still is occurring due to tar build-up. Heat column as in No. 1. Ultimate solution would be to clean the still.

3. The entrained steam picked up by the steam ejector from the still bottoms will ultimately drop off as the ejector nozzle wears. This can also be observed by an increase in the temperature of the still bottoms and an increase in the absolute pressure in the vacuum chamber. The steam ejector nozzles should be replaced. It would not be unusual to replace the ejector nozzle annually. Note: Replace the ejector nozzles only - not the entire unit.

5.0 Troubleshooting - Contd.

4. pH measurement of the still effluent by the in-line pH probe will require frequent checking and recalibration. It may also be necessary to clean or replace the probe periodically. Also check or change the filters in front of the pH probe frequently.

## 6.0 Operating Data Recording

The collection of operating data is essential for a successful start-up and the ability to monitor and review conditions that occur during the operation of the ammonia still. Basically, the data is comprised of temperatures, pressures, flow rates and chemical analysis of the various streams in the total process.

The accompanying operating data sheet lists the suggested information necessary to evaluate the operation of the unit. The flow, pressure and temperature recorders and indicators are listed by number. These numbers correspond to the instrument identification on the piping and instrumentation drawings.

Readings for the data sheet will be taken every two hours.

Immediate operating conditions are observed at the recorders on the control panels. Deviation from standard operating conditions will require adjustment and/or correction by the operators.

During the start-up and fine-tuning of the plant, definite operating conditions will be established. In the future, Interlake may wish to design their own operating data sheets and eliminate some data.

In addition to the specific instrument readings listed on the data sheet, the following additional tests should be made on a weekly (or more often as determined) basis during the commissioning period. Beyond start-up, some of these tests may not be necessary. However, all are significant in evaluating plant performance, status and operation. The Interlake operators and labs should be prepared to run the following analyses:

1. Ammonia (free and fixed) in the weak ammonia liquor

2. Ammonia (total) in the stripped ammonia liquor

3. pH of the stripped ammonia liquor

**DRAVO/STILL**

INTERLAKE, INC.
OPERATING DATA SHEET
AMMONIA STILL

Date:


| No. | DESCRIPTION | Dim. | Tag No. | 7 am | 9 am | 11 am | 1 pm | 3 pm | 5 pm | 7 pm | 9 pm | 11 pm | 1 am | 3 am | 5 am | ø |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Liquor Storage Tank 34001 - level | | LI 1 | | | | | | | | | | | | | |
| 2 | Liquor Storage Tank 34001 - temperature | | TI 100 | | | | | | | | | | | | | |
| 3 | Weak Liquor Storage Tank 37647-A - level | | LI | | | | | | | | | | | | | |
| 4 | Weak Liquor Storage Tank 37647-B - level | | LI | | | | | | | | | | | | | |
| 5 | Ammonia Still Feed Pump - 41002 | | PI 100 | | | | | | | | | | | | | |
| 6 | Ammonia Still Feed Flow | GPM | FR 3 | | | | | | | | | | | | | |
| 7 | Steam Flow to Still 380 psig | lb/hr | FIC 1 | | | | | | | | | | | | | |
| 8 | Low Pressure Steam Flow to Still | lb/hr | FRQ 8 | | | | | | | | | | | | | |
| 9 | Low Pressure Steam Flow - Tray #12 | lb/hr | FI 2 | | | | | | | | | | | | | |
| 10 | Steam Flow to Ammonia Still (Total) | lb/hr | FQI 7 | | | | | | | | | | | | | |
| 11 | Stripped Liquor out of Still - Temperat. | | TI 101 | | | | | | | | | | | | | |
| 12 | Ammonia Still Vacuum Pressure | | PI 104 | | | | | | | | | | | | | |
| 13 | Ammonia Still Bottom Steam Inlet - Press. | | PI 103 | | | | | | | | | | | | | |
| 14 | Ammonia Still Middle Steam Inlet - Press. | | PI 102 | | | | | | | | | | | | | |
| 15 | Ammonia Still Top Pressure | | PR 5 | | | | | | | | | | | | | |



Dravo Still

**DRAVO/STILL**

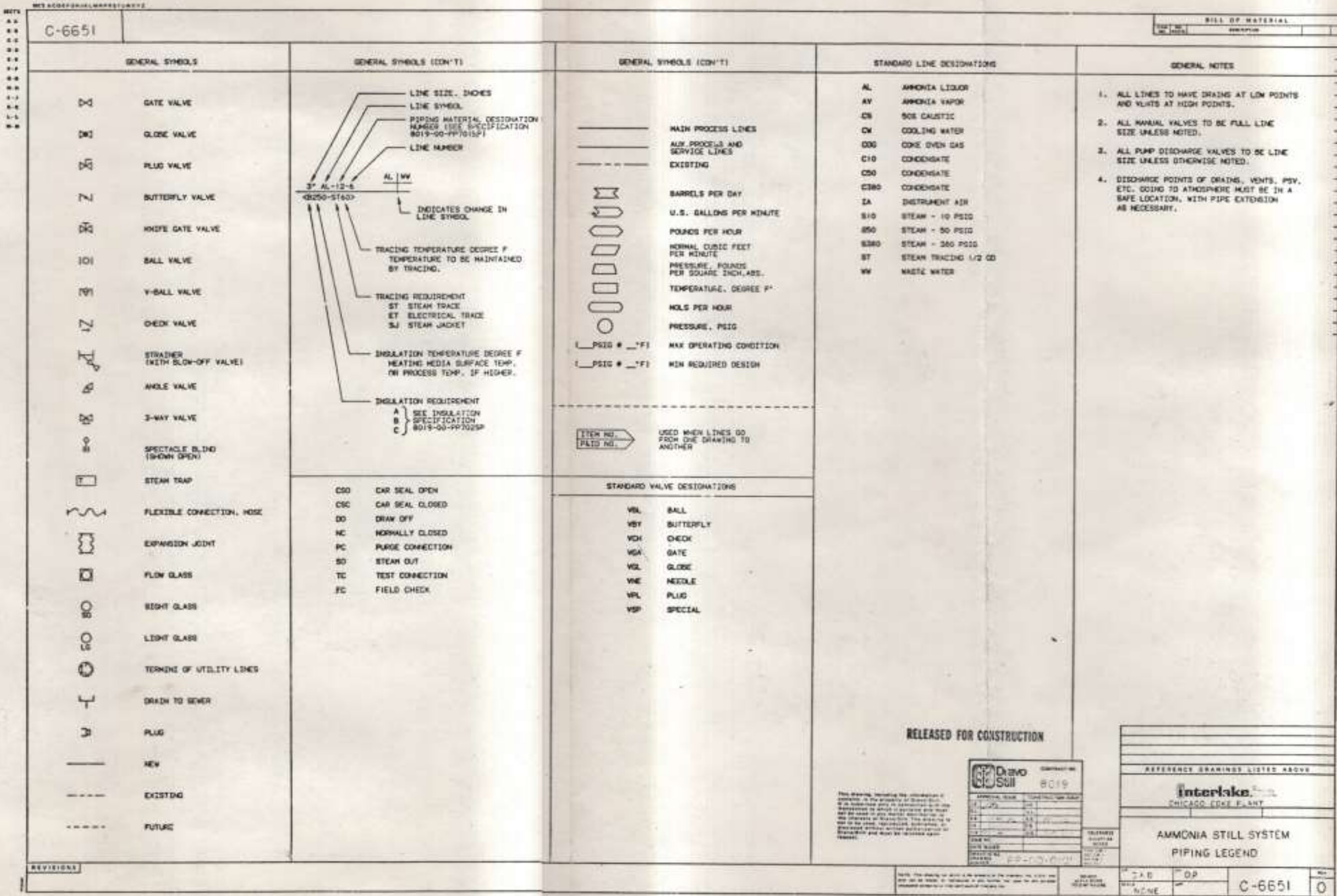
INTERLAKE, INC.
OPERATING DATA SHEET
AMMONIA STILL

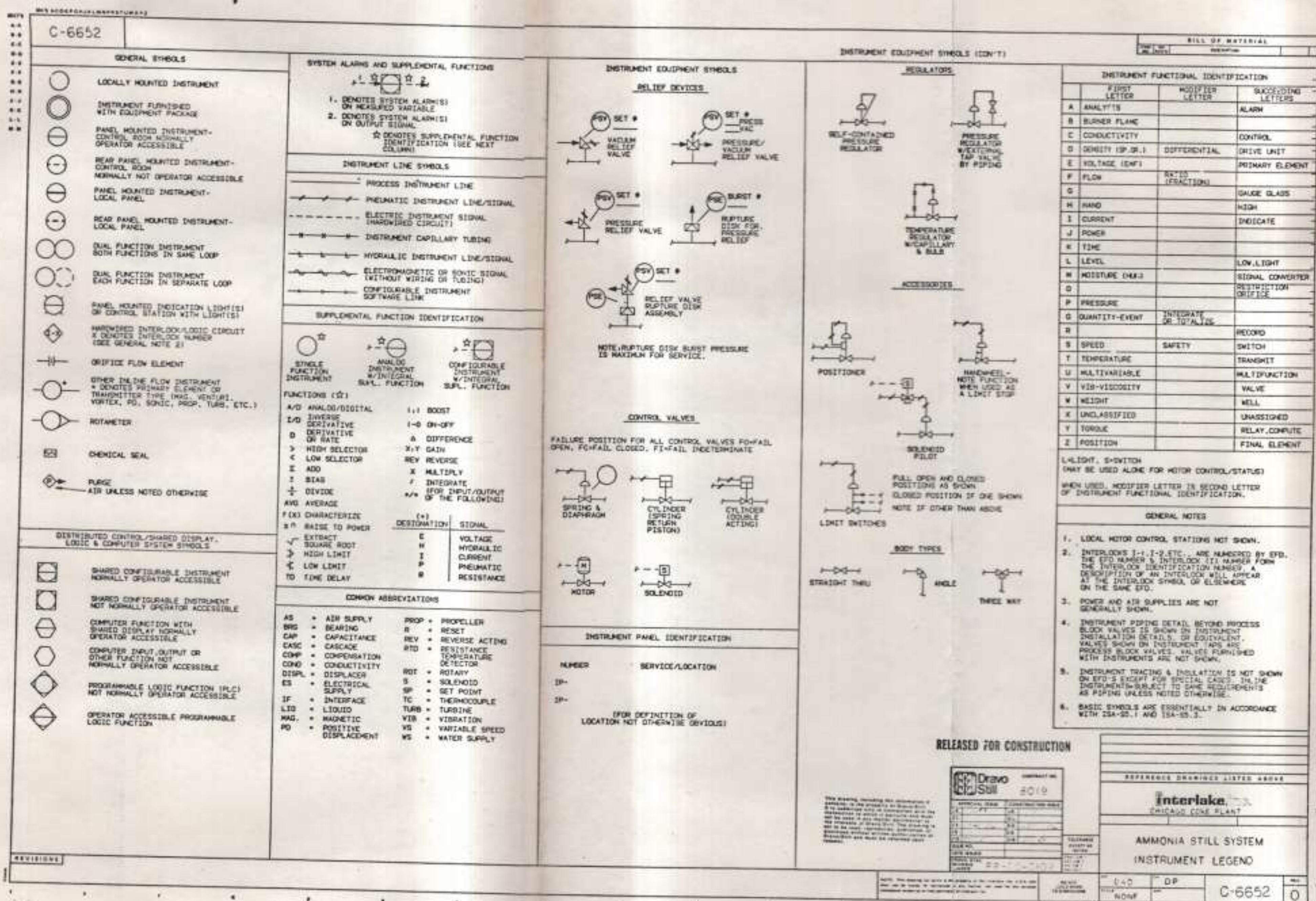
Date:


| No. | DESCRIPTION | Dim. | Tag No. | 7 am | 9 am | 11 am | 1 pm | 3 pm | 5 pm | 7 pm | 9 pm | 11 pm | 1 am | 3 am | 5 am | ø |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 16 | Ammonia Still Bottom Temperature | | TR 2 | | | | | | | | | | | | | |
| 17 | Ammonia Still Top Temperature | | TR 1 | | | | | | | | | | | | | |
| 18 | Ammonia Still Top Temperature | | TIC 3 | | | | | | | | | | | | | |
| 19 | Stripped Liquor pH | | AR 1 | | | | | | | | | | | | | |
| 20 | Stripped Liquor pH Temperature | | TI 103 | | | | | | | | | | | | | |
| 21 | Caustic Flow Ratio | | FFIC 4 | | | | | | | | | | | | | |
| 22 | Caustic Flow pH | | FIC 5 | | | | | | | | | | | | | |
| 23 | Caustic Pumps Discharge Pressure | | PI 106 | | | | | | | | | | | | | |
| 24 | Caustic Storage Tank - Level | | LG | | | | | | | | | | | | | |
| 25 | Low Pressure Steam Flow Pressure | | TI 102 | | | | | | | | | | | | | |
| 26 | Low Pressure Steam Flow Temperature | | PI 105 | | | | | | | | | | | | | |

Rev. 0
11/85

Dravo Still


C-6651
GENERAL SYMBOLS
GATE VALVE
GLOBE VALVE
PLUG VALVE
BUTTERFLY VALVE
KNIFE GATE VALVE
BALL VALVE
V-BALL VALVE
CHECK VALVE
STRAINER (WITH BLOW-OFF VALVE)
ANGLE VALVE
3-WAY VALVE
SPECTACLE BLIND (SHOWN OPEN)
STEAM TRAP
FLEXIBLE CONNECTION, HOSE
EXPANSION JOINT
FLOW GLASS
SIGHT GLASS
LIGHT GLASS
TERMINI OF UTILITY LINES
DRAIN TO SEWER
PLUG
NEW
EXISTING
FUTURE
GENERAL SYMBOLS (CON'T)
3" AL-12-6
<B250-ST60>
AL | WW
LINE SIZE, INCHES
LINE SYMBOL
PIPING MATERIAL DESIGNATION NUMBER (SEE SPECIFICATION 8019-00-PP7015P)
LINE NUMBER
INDICATES CHANGE IN LINE SYMBOL
TRACING TEMPERATURE DEGREE F TEMPERATURE TO BE MAINTAINED BY TRACING.
TRACING REQUIREMENT
ST STEAM TRACE
ET ELECTRICAL TRACE
SJ STEAM JACKET
INSULATION TEMPERATURE DEGREE F HEATING MEDIA SURFACE TEMP. OR PROCESS TEMP. IF HIGHER.
INSULATION REQUIREMENT
A B C SEE INSULATION SPECIFICATION 8019-00-PP7025P
CSO CAR SEAL OPEN
CSC CAR SEAL CLOSED
DO DRAW OFF
NC NORMALLY CLOSED
PC PURGE CONNECTION
SO STEAM OUT
TC TEST CONNECTION
FC FIELD CHECK
GENERAL SYMBOLS (CON'T)
MAIN PROCESS LINES
AUX PROCESS AND SERVICE LINES
EXISTING
BARRELS PER DAY
U.S. GALLONS PER MINUTE
POUNDS PER HOUR
NORMAL CUBIC FEET PER MINUTE
PRESSURE, POUNDS PER SQUARE INCH, ABS.
TEMPERATURE, DEGREE F°
MOLS PER HOUR
PRESSURE, PSIG
(__PSIG @ __°F) MAX OPERATING CONDITION
(__PSIG @ __°F) MIN REQUIRED DESIGN
ITEM NO. P&ID NO.
USED WHEN LINES GO FROM ONE DRAWING TO ANOTHER
STANDARD VALVE DESIGNATIONS
VBL BALL
VBY BUTTERFLY
VCH CHECK
VGA GATE
VGL GLOBE
VNE NEEDLE
VPL PLUG
VSP SPECIAL
STANDARD LINE DESIGNATIONS
AL AMMONIA LIQUOR
AV AMMONIA VAPOR
CS 50% CAUSTIC
CW COOLING WATER
COG COKE OVEN GAS
C10 CONDENSATE
C50 CONDENSATE
C380 CONDENSATE
IA INSTRUMENT AIR
S10 STEAM - 10 PSIG
S50 STEAM - 50 PSIG
S380 STEAM - 380 PSIG
ST STEAM TRACING 1/2 OD
WW WASTE WATER
GENERAL NOTES
1. ALL LINES TO HAVE DRAINS AT LOW POINTS AND VENTS AT HIGH POINTS.
2. ALL MANUAL VALVES TO BE FULL LINE SIZE UNLESS NOTED.
3. ALL PUMP DISCHARGE VALVES TO BE LINE SIZE UNLESS OTHERWISE NOTED.
4. DISCHARGE POINTS OF DRAINS, VENTS, PSV, ETC. GOING TO ATMOSPHERE MUST BE IN A SAFE LOCATION, WITH PIPE EXTENSION AS NECESSARY.
BILL OF MATERIAL
RELEASED FOR CONSTRUCTION
Dravo Still
8019
REFERENCE DRAWINGS LISTED ABOVE
Interlake, Inc.
CHICAGO COKE PLANT
AMMONIA STILL SYSTEM
PIPING LEGEND
NONE
C-6651
0
REVISIONS




INSTRUMENT FUNCTIONAL IDENTIFICATION

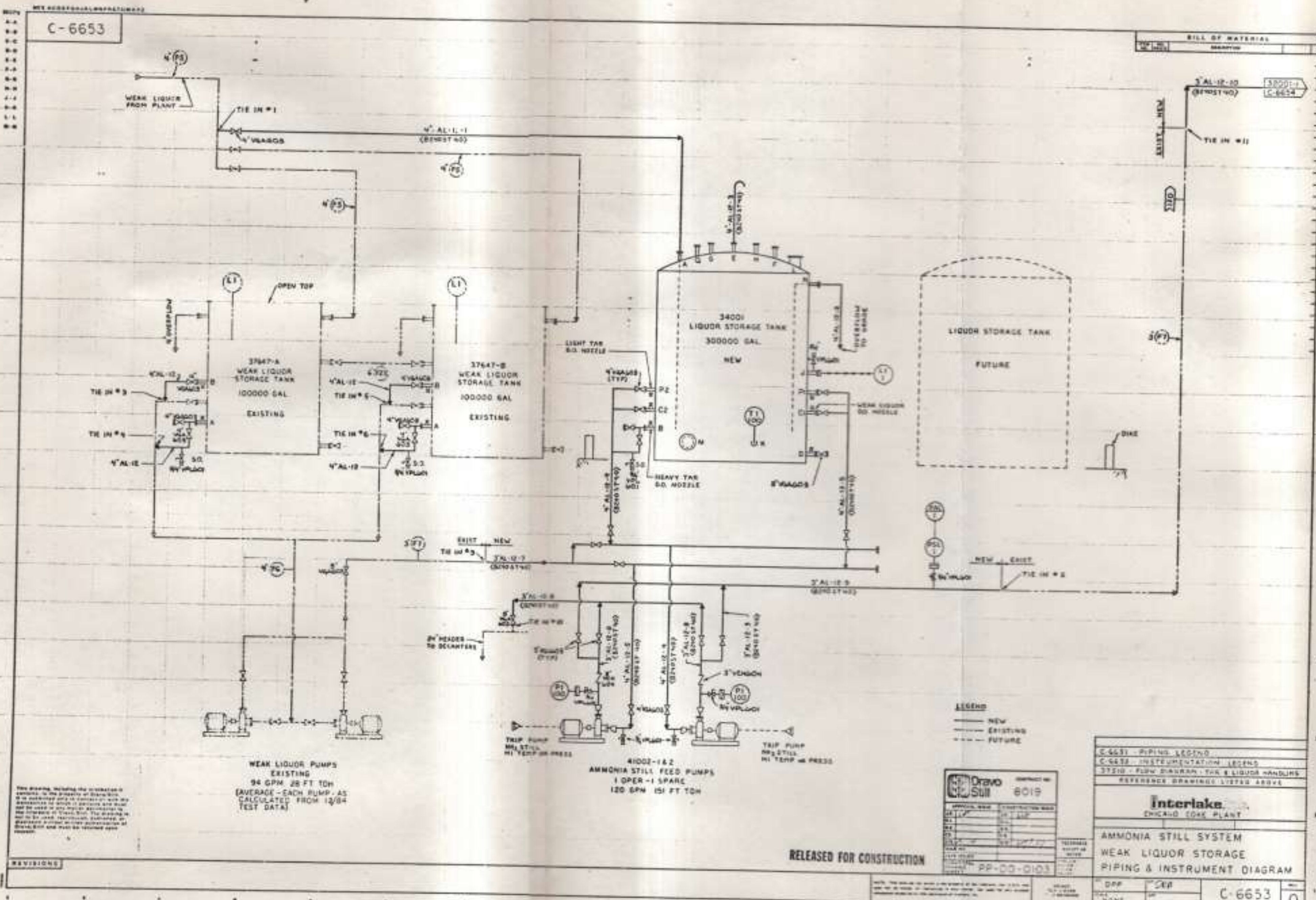
| | FIRST LETTER | MODIFIER LETTER | SUCCEEDING LETTERS |
|---|---|---|---|
| A | ANALYSIS | | ALARM |
| B | BURNER FLAME | | |
| C | CONDUCTIVITY | | CONTROL |
| D | DENSITY (SP.GR.) | DIFFERENTIAL | DRIVE UNIT |
| E | VOLTAGE (EMF) | | PRIMARY ELEMENT |
| F | FLOW | RATIO (FRACTION) | |
| G | | | GAUGE GLASS |
| H | HAND | | HIGH |
| I | CURRENT | | INDICATE |
| J | POWER | | |
| K | TIME | | |
| L | LEVEL | | LOW,LIGHT |
| M | MOISTURE (HUM.) | | SIGNAL CONVERTER |
| O | | | RESTRICTION ORIFICE |
| P | PRESSURE | | |
| Q | QUANTITY-EVENT | INTEGRATE OR TOTALIZE | |
| R | | | RECORD |
| S | SPEED | SAFETY | SWITCH |
| T | TEMPERATURE | | TRANSMIT |
| U | MULTIVARIABLE | | MULTIFUNCTION |
| V | VIB-VISCOSITY | | VALVE |
| W | WEIGHT | | WELL |
| X | UNCLASSIFIED | | UNASSIGNED |
| Y | TORQUE | | RELAY,COMPUTE |
| Z | POSITION | | FINAL ELEMENT |

L=LIGHT, S=SWITCH (MAY BE USED ALONE FOR MOTOR CONTROL/STATUS)

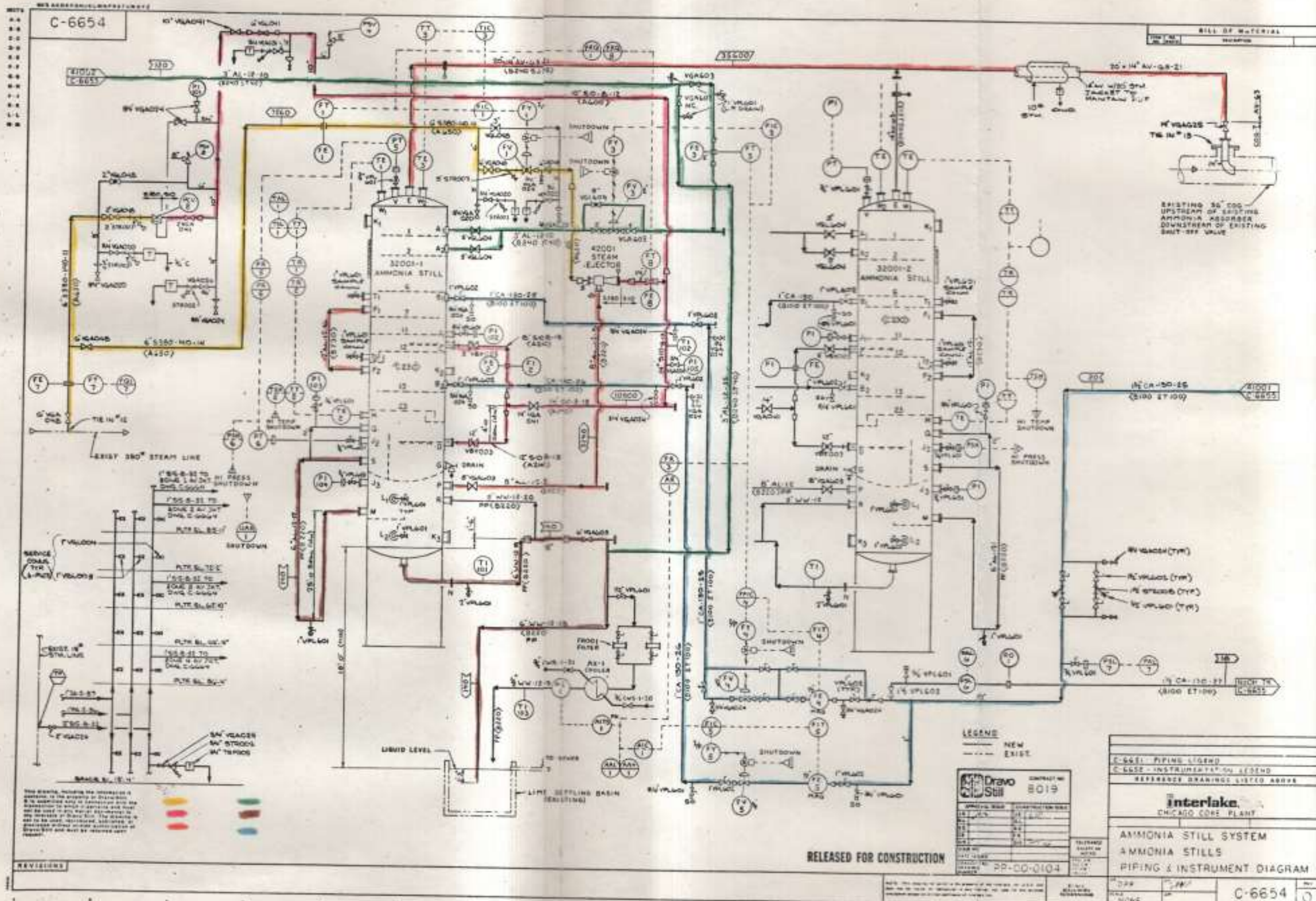
WHEN USED, MODIFIER LETTER IS SECOND LETTER OF INSTRUMENT FUNCTIONAL IDENTIFICATION.

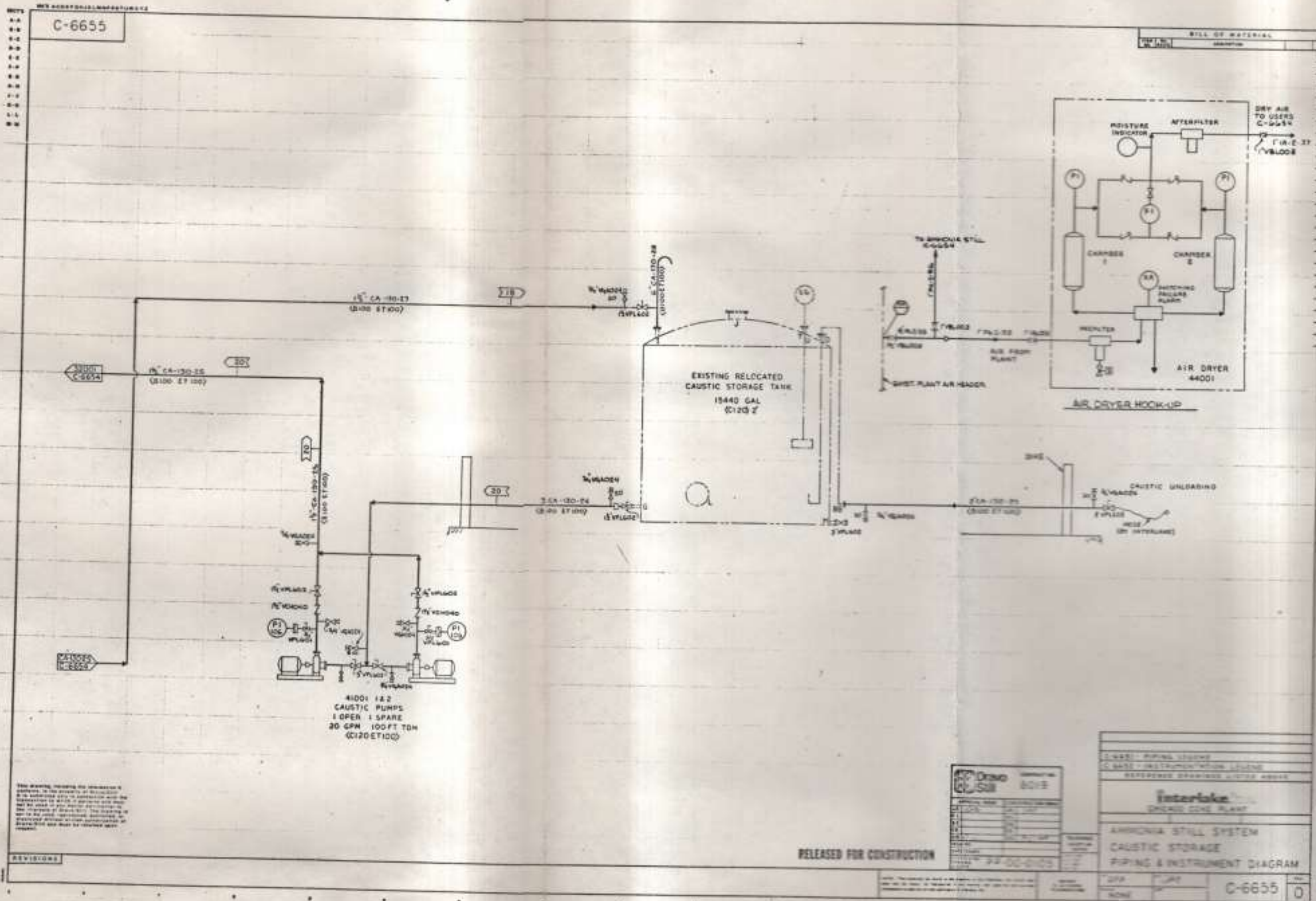
GENERAL NOTES

1. LOCAL MOTOR CONTROL STATIONS NOT SHOWN.
2. INTERLOCKS I-1, I-2, ETC., ARE NUMBERED BY EFD. THE EFD NUMBER & INTERLOCK (I) NUMBER FORM THE INTERLOCK IDENTIFICATION NUMBER. A DESCRIPTION OF AN INTERLOCK WILL APPEAR AT THE INTERLOCK SYMBOL OR ELSEWHERE ON THE SAME EFD.
3. POWER AND AIR SUPPLIES ARE NOT GENERALLY SHOWN.
4. INSTRUMENT PIPING DETAIL BEYOND PROCESS BLOCK VALVES IS SHOWN ON INSTRUMENT INSTALLATION DETAILS, OR EQUIVALENT. VALVES SHOWN ON INSTRUMENT TAPS ARE PROCESS BLOCK VALVES. VALVES FURNISHED WITH INSTRUMENTS ARE NOT SHOWN.
5. INSTRUMENT TRACING & INSULATION IS NOT SHOWN ON EFD'S EXCEPT FOR SPECIAL CASES. INLINE INSTRUMENTS SUBJECT TO SAME REQUIREMENTS AS PIPING UNLESS NOTED OTHERWISE.
6. BASIC SYMBOLS ARE ESSENTIALLY IN ACCORDANCE WITH ISA-S5.1 AND ISA-S5.3.



C-6653
WEAK LIQUOR FROM PLANT
TIE IN #1
4" AL-L-1 (B2405T40)
OPEN TOP
37647-A
WEAK LIQUOR STORAGE TANK
100000 GAL
EXISTING
37647-B
WEAK LIQUOR STORAGE TANK
100000 GAL
EXISTING
4" OVERFLOW
TIE IN #3
TIE IN #4
TIE IN #5
TIE IN #6
4" AL-12
LIGHT TAR D.D. NOZZLE
HEAVY TAR D.D. NOZZLE
34001
LIQUOR STORAGE TANK
300000 GAL
NEW
OVERFLOW TO GRADE
WEAK LIQUOR D.D. NOZZLE
LIQUOR STORAGE TANK
FUTURE
DIKE
EXIST NEW
TIE IN #9
TIE IN #2
TIE IN #11
3" AL-12-10
NEW EXIST
24" HEADER TO DECANTERS
WEAK LIQUOR PUMPS
EXISTING
94 GPM 28 FT TDH
[AVERAGE - EACH PUMP - AS CALCULATED FROM 12/84 TEST DATA]
TRIP PUMP
41002-1 & 2
AMMONIA STILL FEED PUMPS
1 OPER - 1 SPARE
120 GPM 151 FT TDH
LEGEND
NEW
EXISTING
FUTURE
BILL OF MATERIAL
C-6651 - PIPING LEGEND
C-6652 - INSTRUMENTATION LEGEND
REFERENCE DRAWINGS LISTED ABOVE
Dravo Still
6019
Interlake
CHICAGO COKE PLANT
RELEASED FOR CONSTRUCTION
AMMONIA STILL SYSTEM
WEAK LIQUOR STORAGE
PIPING & INSTRUMENT DIAGRAM
PP-00-0103
NONE
C-6653
0
REVISIONS


C-6654
32001-1 AMMONIA STILL
32001-2 AMMONIA STILL
42001 STEAM EJECTOR
SHUTDOWN
DRAIN
LIQUID LEVEL
LIME SETTLING BASIN (EXISTING)
TO SEWER
FROO1 FILTER
AX-1 COOLER
EXIST 350# STEAM LINE
TIE IN #12
TIE IN #13
EXISTING 30" COG UPSTREAM OF EXISTING AMMONIA ABSORBER DOWNSTREAM OF EXISTING SHUT-OFF VALUE
BILL OF MATERIAL
REVISIONS
LEGEND
NEW
EXIST.
RELEASED FOR CONSTRUCTION
Dravo Still
8019
PP-00-0104
C-6651 - PIPING LEGEND
C-6652 - INSTRUMENTATION LEGEND
REFERENCE DRAWINGS LISTED ABOVE
Interlake.
CHICAGO COKE PLANT
AMMONIA STILL SYSTEM
AMMONIA STILLS
PIPING & INSTRUMENT DIAGRAM
NONE
C-6654
0


C-6655
EXISTING RELOCATED
CAUSTIC STORAGE TANK
15440 GAL
(C120) 2'
41001 1&2
CAUSTIC PUMPS
1 OPER 1 SPARE
20 GPM 100 FT TDH
(C120 ET100)
AIR DRYER
44001
AIR DRYER HOOK-UP
MOISTURE INDICATOR
AFTERFILTER
CHAMBER 1
CHAMBER 2
DRY AIR TO USERS C-6654
CAUSTIC UNLOADING
AIR FROM PLANT
1½" CA-130-27
1½" CA-130-25
1½" CA-130-26
3" CA-130-24
RELEASED FOR CONSTRUCTION
BILL OF MATERIAL
REVISIONS
Interlake
AMMONIA STILL SYSTEM
CAUSTIC STORAGE
PIPING & INSTRUMENT DIAGRAM
C-6655
0