I

E. I. DuPont de Nemours & Company

Jackson Laboratory

January 20, 1945

A-2557

JWD-45

Problem Report

Contract W-7412-eng-151

Plant for Packaging Fifteen Pounds C-216 Per Day By The Liquefaction Method

Work Done By:

J. F. Froning
M. K. Richards

Under Supervision of:

T. W. Stricklin, Jr.

Reported By:

J. F. Froning
M. K. Richards

338-1

Photostat Price $ 4.80
Microfilm Price $ 2.70

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

DISCLAIMER

II

TABLE OF CONTENTS

338-2

Plant for Packaging Fifteen Pounds C-216 Per Day By the Liquefaction Method

A-2557

Object:

To provide facilities for packaging approximately 15 pounds per day of specification grade C-216.

Period Covered by the Report:

July 1, 1943 to December 1, 1944.

Introduction:

The principle of this packaging operation is to condense C-216 at atmospheric pressure with liquid nitrogen and revaporize it in a system of such volume that a predetermined pressure (400 p.s.i.) obtains at normal air temperature. Approximately 98% of the volume of this system is provided by cylinders, which may be detached and shipped.

There are economic limitations to such a plant because of the high operating costs and the use of an expensive refrigerant, liquid nitrogen. However, the need for the plant was urgent, and at the inception of this problem, the chemical nature of the gas made the design of a mechanical compressor appear to be a long and difficult problem.

Discussion of experimental work leading to the design of this plant may be found in the following reports:

JWD-25 Reaction of HF with Sodium Fluoride - the Desorption of HF from Sodium Acid Fluoride.
JWD-34 Analysis of C-216.
JWD-43 Development of Cylinders for Shipment of Compressed C-216.
JWD-44 Development of Methods and Design of Equipment for Packaging C-216.

Summary:

The plant is an integral unit, including equipment for producing, purifying, bottling, and analyzing the gas as well as facilities for preparing cylinders for filling. The completed plant has demonstrated a capacity of 105 pounds a week (15 pounds a day), but a temporary lack of demand for the material has prevented continuous operation. It is estimated that with uninterrupted production experience, an 80% improvement in output will be accomplished. It is also estimated that if the gas production capacity were increased to meet the gas bottling capacity, a top production of 225 pounds a week would be possible.

338-3

The principle of operation is simple, but the physical and chemical properties of the gas demand extreme attention to details in construction, operation, and maintenance. Demonstration

of these details is made in this report. Relatively high operating costs and extremely high refrigeration costs make the plant uneconomical if a mechanical compressor can be developed.

Discussion:

I. General Plan for Building

The C-216 production and bottling facilities are housed in a wooden building 40' x 19' x 10'. Located outside are storage corrals for cylinders and liquid nitrogen containers, an emergency pressure relief tank, a vent line fume disposal unit, and a ventilating fan and most of the duct work. See Sketch I for a plan view of the building, and refer also to the cylinder processing flowsheet, Sketch III.

The electrolytic cells and any equipment processing compressed C-216 are operated from the outside of a barricade. Metal-covered walls and doors are provided around the cells to protect against gas or liquid spray.

The liquefaction unit and all C-216 pressure piping are barricaded with either one-quarter inch steel plate or two courses of brick, depending on the proximity of the equipment to the barricade. Double windows are installed, 6-8 inches apart; the glass is one-quarter inch "Tuf-Flex", a tempered, shock-resistant material. Protection is necessary against an impinging gas stream or a flame propelled by gas pressure; no explosion should occur.

The electrolytic cells and the liquefaction unit are located so that one operator can handle both. On the other side of the room are the cylinder processing facilities exclusive of actual filling.

The separate vent lines from the cells and from the pressure equipment lead to individual combustion chambers where the C-216 is diluted with air and burned with propane. The combustion products are washed with water.

II. Equipment

Refer to Sketches I and II, attached to this report.

Number	Description
1	Two 1500-ampere C-216 cells. For description refer to separate reports on C-216 manufacture.
2	Water circulating system, automatically controlled, used to maintain desired operating temperatures.
3	Two nickel condenser-receivers, volume 2.7 liters, designed for normal working pressure of 400 p.s.i. with 33% increase in wall thickness for corrosion

allowances. Two flanged, tongue-and-groove openings at the top (using annealed nickel gaskets). The thermowell is made of 1/8-inch nickel pipe. See Drawing C-155-1.

4. Nickel shipping cylinders, 50- and 125-pound water capacity, 3/4-inch opening, Pressed Steel Tank Co., ICC 3BN-400. See JLS-182 and JLS-314.

5. Expansion tank for safety disc vent, 18 inches in diameter by 12 feet high, working pressure 75 p.s.i. Provided with safety disc bursting at 150 p.s.i., pressure gage, 1-inch vent line (and valve) leading to fume disposal unit, two 2-inch inlet lines from bottling systems, bottom opening for cleaning.

6. Refrigerant bath, a standard American thermos bottle, No. 8623, 12-liter capacity, installed in a metal case.

7. Refrigerant bath cover; mounted on rubber shock absorbers over top of elevator lift for bath. Flat 16-gage steel disc, on which is cemented a 1-inch thick cork ring for contact against thermos lip. Provided with oversized holes for refrigerant and process gas lines so that they may be insulated from cover with rubber sleeves. Lagged on top with hair felt sealed from the air. See Drawing JLS-316.

8. Lifting device for liquid nitrogen bath, a simple elevator operated by a wheel on a horizontal shaft extending through the barricade. See Drawings L-181-1 and DWD-5607.

9. Heat exchanger for partial condensation of HF at -80°C. Copper coil draining into 2-liter copper receiver, both installed in large, lagged, closed dry ice chamber. Covered opening for 4-inch pieces of dry ice. Bottom outlet and valve from copper receiver for disposal of condensed HF to outside water ditch. See Drawing JLS-315.

10. Two sodium fluoride towers piped in parallel, made of 3-inch flanged steel pipe 5 feet long. Provided with internal and external thermocouples and an air jacket heated with electrical resistance heaters of 2500 watt capacity. One-inch pipe openings in top and bottom flanges for gas flow. A 12-mesh Monel screen mounted on tripod standing on bottom flange for support of 5/32-inch sodium fluoride pellets. See Sketch II and Drawing DWD-5686.

11. Dust filter, a 400-mesh Monel screen cylinder installed in steel cartridge. Ruggedly constructed to withstand hammering to loosen scale if formed on screen, with catch basin for solid deposits.

12. Ventilating fan, 9000 C.F.M. (total building volume 7600 cu.ft.). A 12-inch intake fed by individual ducts from enclosed operating compartments and an open hood over the cylinder washing jig. Intake air to compartments through individual roof openings. Fan discharges to a 30-inch stack 30 feet high. Ample damper facilities to control local degree of ventilation. See Drawing DW-5267.

13. Cylinder washing jig or rack, installed under open hood for removal of any solvent fumes. Drawings DWD-5914 and DWD-5915.

14. Venting and sampling booths, 3' by 3' x 7' of 1/4" steel plate. Permit sampling of filled cylinders for analysis, and venting and flushing of returned cylinders. Drawing DWD-5893.

15. Cylinder vise, vise for holding cylinders during valve removal and installation.

16. Fume disposal unit, consisting of burning chamber, stack, and water scrubber for combustion products.

17. Instrument board for:

Potentiometers
- Cells
- HF Scrubbers
- C-216 Condensers

Electrical meters and controls for cells
"Variac" temperature control for HF scrubbers

N.B. Miscellaneous drawings available:

(a) Architectural, arrangement, piping	L-171, L-179-1, L-178-1, DW-5213
(b) Original pilot plant assembly, including provisions for liquid piston compressor which was cancelled before completion.	L-158-1, DW-3162

III. Description of the Operation

A. Importance of Safety:

The hazards involved in compressing a gas as reactive as C-216 cannot be over-emphasized. Please refer to JWD-44 for a detailed discussion of safety practices supplementary to general precautions outlined in the description to follow.

338-6

B. Preparation of Cylinders for Filling:

1. Handling Returned Cylinders: Returned cylinders are treated as if full until vented of excess pressure. The equipment is operated through a barricade and includes a gage so that the operator may know when the cylinder pressure is zero and may dilute the residual gas twice with nitrogen at 50 p.s.i. to flush the cylinder. The valve may then be removed without discomfort to the operator.

2. Cleaning: The cylinder is washed with hot water and drained, using a jig for inverting the cylinder. If inspection shows it to be free of any extraneous matter, it is dried with steam; otherwise a solvent or more drastic cleaning solution must be employed to insure a perfectly clean vessel.

The valve, whether new or used, must be cleaned just before final assembly by a mechanic who should work at a dry, grease-free bench. This cleaning involves brushing the packing rings and individual valve parts with carbon tetrachloride to remove grease; it may also require removal of corrosion scale with hot water or dilute aqueous ammonia.

3. Inspection and Assembly of the Valve: A trained mechanic checks the individual valve parts for burrs or other mechanical imperfections. The seat and stem are faced off if necessary. The valve must work in perfect alignment, since the unusually hard packing does not permit self-alignment during seating action.

It should be emphasized that the packing rings are not re-used without inspection.

4. Installation of the Valve in the Cylinder: The gasketed joint is made up after taking two precautions: (1) Seeing that no recontamination occurs after cleaning of valve or cylinder, (2) checking all threads for burrs and gasket surfaces for imperfections. This must be a clean job.

5. Testing: A full 400 p.s.i. nitrogen test is applied to determine the tightness of the valve-to-cylinder joint, the valve packing and the valve seating action.

6. Evacuation and Torching: The cylinder is now evacuated to one millimeter of mercury pressure while keeping the cylinder walls hot (50-100°C.) with a gas flame. The operator retests the tightness of the valve by closing it and observing if the vacuum in the cylinder can be maintained. The cylinder is left evacuated and is now tagged for filling.

C. Production and Purification of the C-216 (Refer to Sketch II):

The C-216 is produced in an electrolytic cell with a capacity of 1-1/2 pounds per hour. The plant is provided with a stand-by cell and therefore production cannot be long delayed unless the source of the electric power breaks down.

The gas readily cools to air temperature in the pipe lines and the only processing required before it enters the liquid-nitrogen-cooled condenser-receiver is to remove any dust or mist to reduce the HF content to at least one per cent by volume.

Mist and dust are removed by a 400-mesh screen installed in such a way that the screen can be hand vibrated to dislodge any solid material which may plug the screen. A catch basin is provided for filtered solids.

Removal of most of the HF is required not only to meet specifications but to prevent solid HF (f.p. -93°C.) from freezing out on the walls of the condenser-receiver (temp. -196°C.) and building up an insulating layer that will first retard and eventually stop the condensation. First the C-216-HF mixture passes through a copper heat exchanger embedded in a dry ice bed. The HF dew point is reached at approximately -40°C., and when the gas has cooled to -78°C., the HF vapor pressure has been lowered to about 4.5 mm. of mercury. However, since HF is associated at this temperature, the actual volume per cent in terms of a nonassociated molecule is 2.1. Further reduction to less than 1% on this basis is accomplished by an absorption tower charged with sodium fluoride.

The NaF is packed in the form of 5/32" pellets in a 3-inch pipe tower to a depth of four feet; this volume corresponds roughly to 15 pounds. The tower is provided with an electrically heated air jacket which maintains the pellets at about 100°C. At this temperature absorption of HF is slow enough that it will take place roughly throughout the length of the tower instead of at one end where plugging would occur. When the value of "x" in $NaF \cdot (HF)_x$ reaches 1 at 100°C., the vapor pressure of the solid is such that the volume per cent of HF in the gas can be reduced to a minimum of 0.18%.

Operation of the HF removal apparatus may be summarized as follows:

(a) The process gas leaving the dust filter is allowed to pass through the dry ice cooled trap. Attention by the operator is necessary only to keep the dry ice chamber full, to occasionally drain off the liquid HF which is collecting, and to check performance by gas temperature and/or HF content.

(b) The noncondensibles leaving the cold trap are next passed over sodium fluoride.

The absorption tower is operated continuously until analysis of the outlet gas shows an undesirably high HF value (maximum allowed, one per cent). Then the gas flow is diverted to an alternate tower in parallel. The first is meanwhile heated gradually to 400°C. and swept with nitrogen to liberate

338-8

and remove the HF. Upon cooling to 100°C. the regenerated sodium fluoride is ready for reuse.

Performance data on these sodium fluoride scrubbers are meager; such data will be available when the plant goes into steady operation. It is known that the adsorption units installed for this operation have run successfully for 60 hours, purifying 1.5 pounds per hour of C-216. Five regenerating cycles have been performed without sufficient deterioration of the pellets to cause plugging of the tower. There is no reason to believe that a scrubber of this size could not run 500 hours without recharging if the precooler performed satisfactorily, but no efficiency data are yet available to prove or disprove this. Certainly it is expected that the pellets will be physically damaged by the volume changes due to alternate loss and gain of HF as well as by the tendency to ward uneven absorption of HF throughout the pellet. Please refer to a separate research report on a laboratory study of the combination of HF with NaF pellets (JWD-25).

In operating all HF-removal equipment it is <u>important</u> to keep them <u>dry</u> when shut down. Occasionally a dismantling and thorough cleaning is necessary to remove deposits of sodium acid fluoride both in the equipment and in the connecting lines.

The process gas leaves the sodium fluoride tower containing less than 1% HF, less than 1% O2, less than 3% N2. If the production rate is one pound per hour, gas enters the condenser-receiver at the rate of approximately 11 cu.ft. per hour, at air temperature and pressure.

There is a possibility that under unknown circumstances hydrogen is present in C-216 systems. No proof has been developed, but several minor fires and explosions occurring in C-216 use have led to suggestions that hydrogen may be a contributing factor.

D. Condensation:

The manner of condensing C-216 in the plant unit coincides exactly with that described in JWD-44 in connection with the pilot plant. The condenser-receiver is a cylindrical nickel vessel with hemispherical ends, 10-3/4" long, 5-1/4" in diameter, with a volume of 2700 cc. Immersed in a liquid nitrogen bath, it will condense at rates up to 1-1/2 pounds per hour until it is practically full of liquid C-216. This amounts to 6-3/4 pounds. The capacity to condense drops to zero within five or ten minutes, indicating that only a small area of nickel is required to maintain sufficient heat transfer, and this area is not covered with liquid until the receiver is nearly full. The end point is readily detected by an increasing supply line pressure as well as a lowering of the vacuum beyond the throttling valve.

During the condensation the throttling valve must be continuously adjusted to compensate for fluctuations in gas production rate and for the gradually decreasing vacuum in the condenser-receiver (due to increasing temperature, in turn resulting from loss of cooling capacity as the receiver fills). The use of a valve automatically adjusted by C-216 supply line pressure has been suggested but has not been given a trial. The maintenance of a line pressure of + 3 inches of water is a prerequisite of the electrolytic cell operation.

The liquid nitrogen make-up is added to the cooling bath in as few batches as possible to avoid the high refrigeration losses from the transfer lines between the carboy and the bath. Transfer of liquid nitrogen is accomplished by blowing it from the shipping container with nitrogen. A nitrogen gas bleed valve for this purpose is located near the C-216 throttling valve, and both are below the window through which the vacuum gage is visible. Liquid nitrogen requirements, as shipped in 25-liter containers, have been 4 liters per pound of C-216 packaged when in regular production.

The condenser is double-valved from any line leading to the air to reduce the possibility of introducing air into the charge. Otherwise the failure of a single valve would cause air to condense in the receiver as fast as it could be sucked in through the leak.

Once the liquefaction has begun, an operator may not enter the compartment enclosing the liquefier and pressure equipment until the system pressure has been reduced to atmospheric or lower, except for the C-216 in closed cylinders. Facilities are provided for performing all necessary manipulations from the outside of the barricade.

If for any reason a liquid charge is considered to be of low quality, it is vaporized slowly to the fume disposal unit.

Refer to Part G in this section for a discussion of maintenance requirements.

E. <u>Filling the Cylinders:</u>

1. <u>Operation.</u> At the end of a condensation, the pressure shut-off valve at the inlet to the liquefaction unit is closed. A short section on the side leading to the gas supply is vented to prevent damage to the electrolytic cell or vacuum gage should the pressure shut-off valve fail during the vaporization (pressure) cycle. Always working outside the barricade, by mechanical means, the operator lowers the liquid nitrogen bath away from the C-216 container and slips a cover over the bath. The pressure gage is carefully observed, and as vaporization begins in the closed system the pressure builds up to 20 p.s.i. in a few minutes. The feed valve leading to one of the two evacuated cylinders is cracked until the pressure in the system is equalized. The valve is then opened wide and the vaporization allowed to continue and the cylinders to fill. A vapor pressure-temperature curve for the above system has not been obtained.

338-10

If the only heat supplied to the nickel container is by natural air convection, it will require an hour to completely vaporize the 6-3/4 pounds of C-216. This process can be markedly hastened, if necessary, by blowing dry air on the sides of the vessel containing the liquid.

Assuming no deviation from the gas laws, a 125-pound water volume cylinder contains 5.4 pounds of C-216 at 400 p.s.i. and 86°F. Therefore, when the system pressure reaches 400 p.s.i., one cylinder valve must be shut and the other opened. Equilibrium will result at about 100 p.s.i. system pressure when the vaporization is completed to the second cylinder. The second cylinder valve is shut and the condenser-receiver is cooled again with liquid nitrogen until a vacuum of 18-19 inches (Hg) is obtained. The two cylinder feed valves are shut and the vent valve on the cylinder connector leading to the full cylinder is opened.

The operator dons protective clothing, removes the cylinder valve extension handle, breaks the spud connection, and rolls the full cylinder a few feet through the nearby door. The cylinder is checked for leaks, the valve outlet cap is put on with a copper gasket, the packing nut "snugged up" a bit, the cylindor protecting cap screwed on securely, and the cylinder is rolled to the covered corral.

Another evacuated cylinder replaces the one removed, and a new condensation can begin. The new vaporization is begun by first completing the filling of the cylinder already at 100 p.s.i. Eventually a cycle will end with two full cylinders and they will both be replaced.

2. <u>Time Cycles and Production.</u> Two identical packaging units have been installed. Since there is only one source of gas, with no excess capacity, the extra unit is used to carry on condensation during the vaporization cycle on the first unit, permitting full utilization of gas production capacity, or to serve as a stand-by when repairs are required on the one which has been operating.

Normally, two hours are required between condensations to accomplish cylinder filling and changing, switching of liquid nitrogen carboys, and other miscellaneous duties. <u>One</u> bottling unit (6-3/4 pounds capacity per cycle) therefore performs as follows:

Condition	Condensation Rate - Pounds Per Hour	Condensation Time - Hours	Per Cent Attainment, Operating	Daily Production Pounds
A	1.35	5	100	23.15
B	1.35	5	65	15.00
C	1.5	4.5	75	18.75

Due to a temporary lack of demand for the material, only one bottling unit of the plant has been operated at a time, and that has been only infrequently and for short periods. The condition reached in the above table has been "B". It is believed that condition "C" will be obtained once the experience of continuous production is gained. If both units are operated, condensation will be continuous, i.e., the two-hour lapse will be eliminated. Therefore, at 75% attainment and at a rate of 1.5 pounds per hour, daily production would be 24 x 1.5 x 0.75 = 27 pounds per day.

Observation of the refrigeration capacity exhibited by the C-216 condenser leads to an assumption that if increased gas production rate were available, the 27 pounds per day could be raised to 32. Only steady operational experience at any level will test such an assumption. The possibility exists, too, for an improvement in the 75% attainment figure.

3. Precautions. The following precautions are observed to preserve safety on the cylinder filling operation:

(a) The operator performs every step through a protecting wall except handling closed cylinders.

(b) The safety disc vent line discharges into an expansion tank rather than to air. After a rupture, the entire system is vented through a fume disposal unit. (See Sketch II).

(c) Safety discs are renewed at regular intervals because of rapid corrosion of the thin nickel, especially if the system is down for other repairs and is alternately exposed to both air and C-216. Actually there are two discs in series, with a gage between to indicate leakage through the first one if corroded.

(d) If a leak develops during the vaporization step, the only action is to close the cylinders and vent the system or complete the vaporization as soon as possible. Recondensation by raising the refrigerant bath is not recommended unless it is a small leak that can be quickly fixed by simply tightening up. The safe way to locate a leak is to first shut off both cylinders and then empty the system of C-216 until the condenser is at room temperature, and the gauge shows only 50-100 p.s.i. Then test with wet potassium iodide paper held at two-foot length (using a stick) by an operator wearing protective clothing.

(e) Never transport a full cylinder without having the valve-protecting cap on.

F. Analyzing, Storing and Shipping of Cylinders:

(Refer to Cylinder Processing Flowsheet)

Each cylinder must be analyzed for nitrogen, oxygen and HF to determine whether it meets the 3%, 1% and 1% limitations,

338-12

respectively. The same safety precautions are observed in sampling as in filling. The chemistry of the analytical method is described in a separate report. The time required per cylinder is about an hour. (See JWD-34).

Full cylinders are stored in a corral with a roof but no sides. This arrangement provides a rain shed to keep water from collecting under the valve-protecting cap but eliminates necessity for a fire-proof, ventilated building in case leaks develop. The area around the corral is fenced in.

Even though a cylinder has been checked immediately after filling, it should be re-checked for leaks just before shipping and periodically in storage. If found leaking, a cylinder should be plainly marked and set aside so that it will be handled with due care.

Due to the great reactivity of the gas, it is especially desirable to keep the cylinders clean and clearly identified with the 3-inch yellow stripe.

G. Operating Maintenance:

1. Cleaning. All new or repaired vessels, valves and pipe lines are separately and carefully cleaned before installation. To minimize the possibility of dirt or solvent being overlooked or trapped, numerous unions are provided to facilitate handling and draining. Valves are completely disassembled for this job; small parts and packing rings are kept in a closed, clean box. Equipment must be free of extraneous matter and dry when installed.

Used equipment containing fluorides may be cleaned by washing with copious quantities of hot water. It is usually dried with steam, followed by air. The fluorides collecting in small copper vessels have been most conveniently removed by rinsing with aqueous ammonia. Valves otherwise in good condition may be simply broken at the bonnet and the bonnet dried in air up to 100°C. Gages must be scrupulously cleaned, especially when new. The best procedure is to first evacuate the Bourdon tube and then allow it to fill with the cleaning fluid, then re-evacuate and dry.

Steel equipment, once washed and ready for installation, should be kept dry. If possible the surface should be treated with a rust inhibitor and then sealed from the air. Rusting is not dangerous but produces very undesirable scale and dust when it reacts with C-216. In fact, large steel vessels are best cleaned by sweeping slowly with C-216 until all rust is consumed; the fluoride solids then can be dumped out.

2. Preconditioning. It is recommended that any new or cleaned equipment be conditioned at atmospheric pressure by cautious C-216 sweeping for at least an hour before subjecting it to pressure. When the sweeping is first begun, careful

observation should be made to note any hot spots. Conditioning is not complete if the metal surfaces are above air temperature. If this preconditioning is not carried out, introduction of C-216 under pressure may cause a violent flame at the point of reaction with the contaminated metal. Improperly prepared steel valves will literally "go up in smoke".

3. Testing Pressure Equipment. To test a pressure system containing packed valves, first eliminate those leaks detectable with a working pressure of nitrogen (and soapy water). Whether or not the system is now tight to nitrogen, a 50 p.s.i. C-216 test is applied. If the system is small and is valved off from the pressure supply source, the operator may stand on the equipment side of the barricade provided he wears protective clothing. He then searches for the leak with a piece of wet potassium iodide paper placed on the end of a short stick. Any significant leak can thus be detected.

Often a vessel will leak to C-216 when tight to other gases. A large one, therefore, should first be tested at low C-216 pressure. In this case it is safe to stand near the tank if it has been previously conditioned properly and is not leaking so badly that the operator will be hit by a stream of gas. The difference between this case and that of a system containing valves is the presence of packing. A slowly leaking packing has been known to suddenly fire, with resulting ignition of the metal valve bonnet and a severe flame hurled by the force of escaping gas.

4. Vent System. It is imperative that the vent header be kept dry, especially if the pipe lines are of steel. It has been found impossible to do this if the vent outlet leads to the air or to a water-scrubber type of fume disposal unit. Aqueous acid collects in the lines due to the dehydrating action of HF. Careful sloping of the entire header and incorporation of traps and drains is only of partial help. Water vapor is drawn to all parts of the system, even if the vent stack leads only to the air.

Furthermore, even if the system is swept thoroughly with nitrogen before a shut-down, the fluoride corrosion scale in iron pipe will hydrate if air is admitted. Hydration causes swelling, and a line as big as one inch will eventually plug.

When shut down, blank all parts of the system not being repaired. An example of the importance of this is that when repairs have been frequent in operation of the present plant, even the nickel safety discs have corroded faster during shutdowns than during operation.

Safety discs are best replaced frequently (every two weeks) as a matter of routine.

After it was diagnosed that water introduced from the vent lines caused two dangerous metal-C-216 fires on the DuPont

338-14

pilot plant, a dry vent system was obtained by installing a gas burning chamber at the vent header outlet. Excess propane reacts with the C-216 vented off, converting it to less harmful products. The combustion gases are scrubbed with an obnoxious fume absorber with the result that a slight vacuum exists in the vent system.

5. Valves. Process valves in the high-pressure lines require careful maintenance in two respects: (1) condition of the packing gland, and (2) mechanical perfection of extension handles, i.e., with respect to alignment, tightness of any set screws, etc.

Packing nuts should be checked frequently to see that they have not become loose. As previously mentioned, a leaking gland can result in a fire. If the packing becomes so tight that the valve is difficult to turn-(a situation that can easily develop with K-416)--the packing should be replaced. This will prevent the undue wear on the seating surfaces which occurs when a hard-turning stem is forced shut by the operator.

The York ammonia valve, a cast steel needle type with a hardened steel stem, has proved very satisfactory for pressure line service. Five of these valves installed in one bottling unit are in good condition after several months of intermittent operation; they have been repacked twice as a matter of routine. Such a valve will soon fail unless the system is kept clean and dry, for the internal threads will corrode and jam. As for other types of valves that have been tried, smaller ones are not rugged enough, and nickel and nickel alloy types have either been susceptible to galling and seizing of the rubbing surfaces or require too much care in tightening the packing in comparison to the York valve. Refer to the discussion of valves in JWD-43.

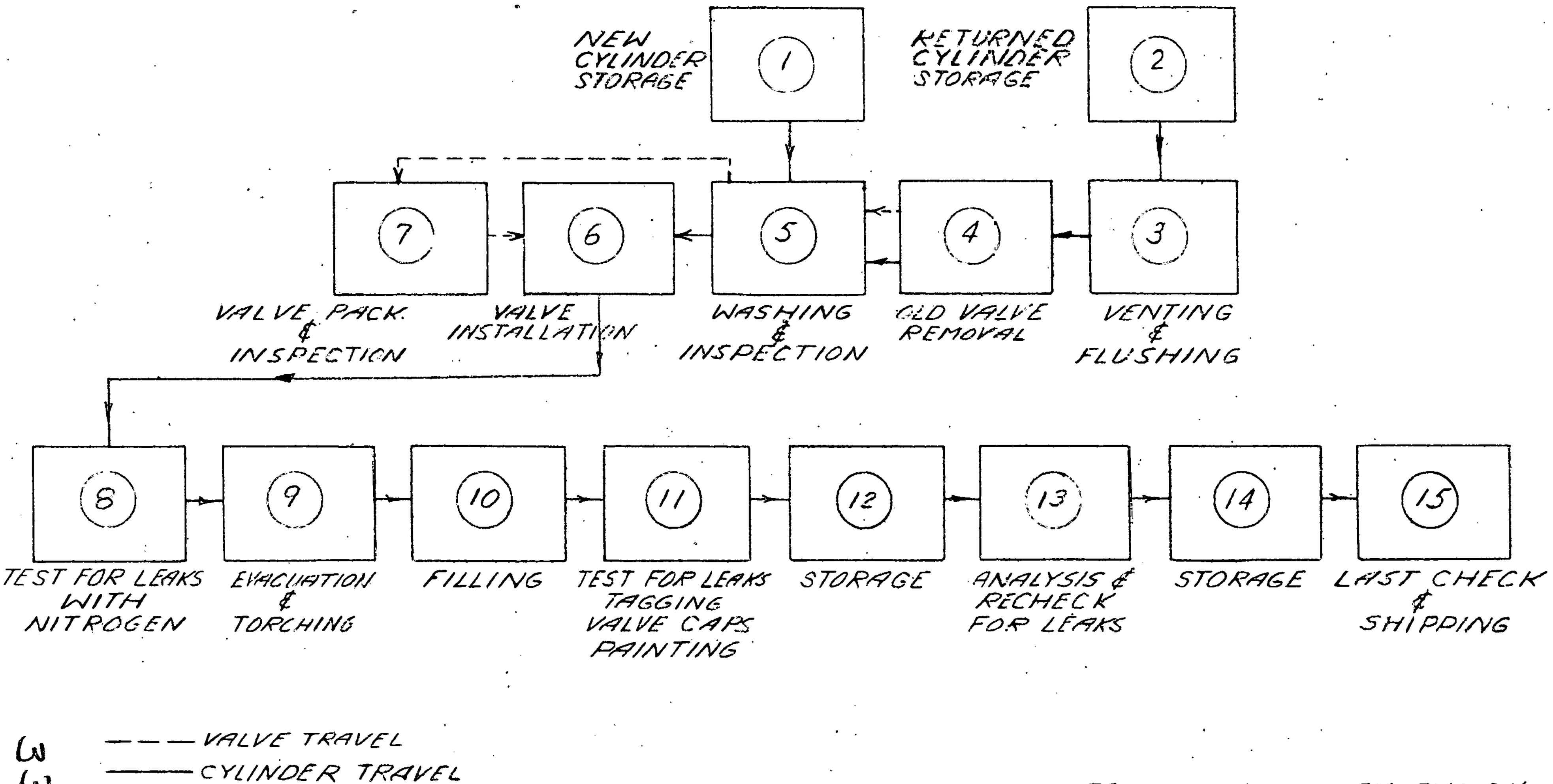
6. Refrigerant Bath Cover and Lifting Device. Two important considerations obtain: (1) liquid nitrogen consumption due to heat transfer into the cooling system from the surroundings represents the major refrigeration requirement under the best of conditions. It is of paramount importance, therefore, to keep the bath lid in proper condition. (2) The remotely controlled "elevator" moving the bath up and down must not fail mechanically during operation. If it could not be lowered, there would be a delay while the liquid nitrogen evaporated away to permit vaporization of the C-216. If it could not be raised, a charge might be lost in an emergency during the pressure step. Safety rules would prevent entrance into the operating compartment to move a defective lift by hand.

May 26, 1945
LED

338-15

CYLINDER PROCESSING FLOWSHEET

REPORT REFERENCE - JWD-45



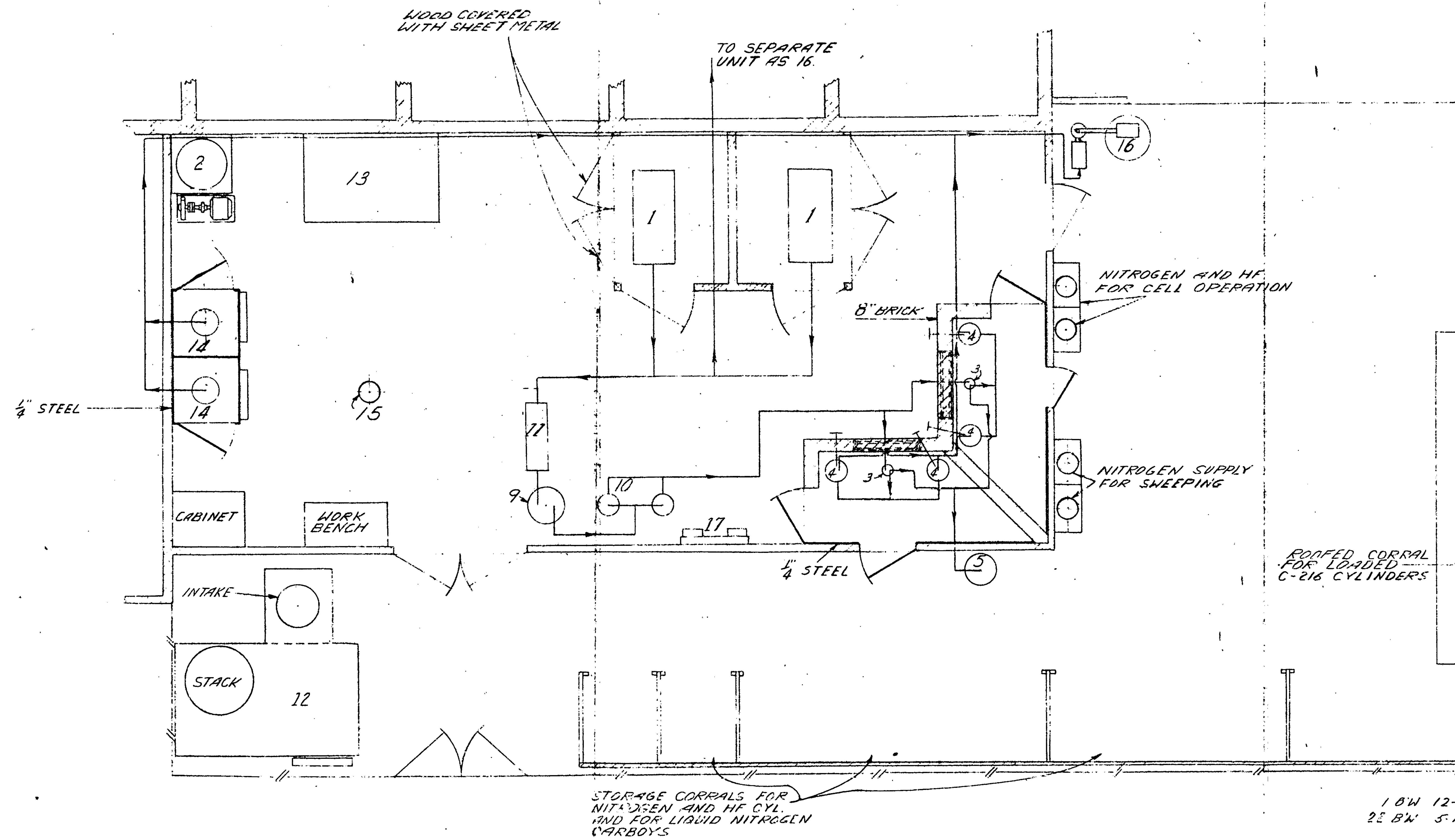
338-16

JACKSON LAB. CHEM. ENG. DIV.
DATE: OCT. 3, 1944
DRAWN: [illegible]
APP: [illegible]

JLS-318

SKETCH 1
SCHEMATIC PLAN VIEW
C-216 PACKAGING BLDG.

40' x 19' x 10' WOODEN EXCEPT WHERE NOTED.
REPORT REF. JWD-45



1. C-216 CELLS
2. HOT WATER SYSTEM FOR CELLS
3. LIQUIFIER
4. SHIPPING CYLINDERS
5. EXPANSION TANK FOR SAFETY DISC DISCHARGE
9. PARTIAL CONDENSER FOR HF
10. SODIUM FLUORIDE SCRUBBERS FOR HF
11. DUST FILTER
12. VENTILATING FAN
13. HOOD FOR CYLINDER CLEANING
14. CYL. SAMPLING & VENTING BOOTH
15. CYLINDER VISE
16. FUME DISPOSAL UNIT
17. INSTRUMENT BOARD

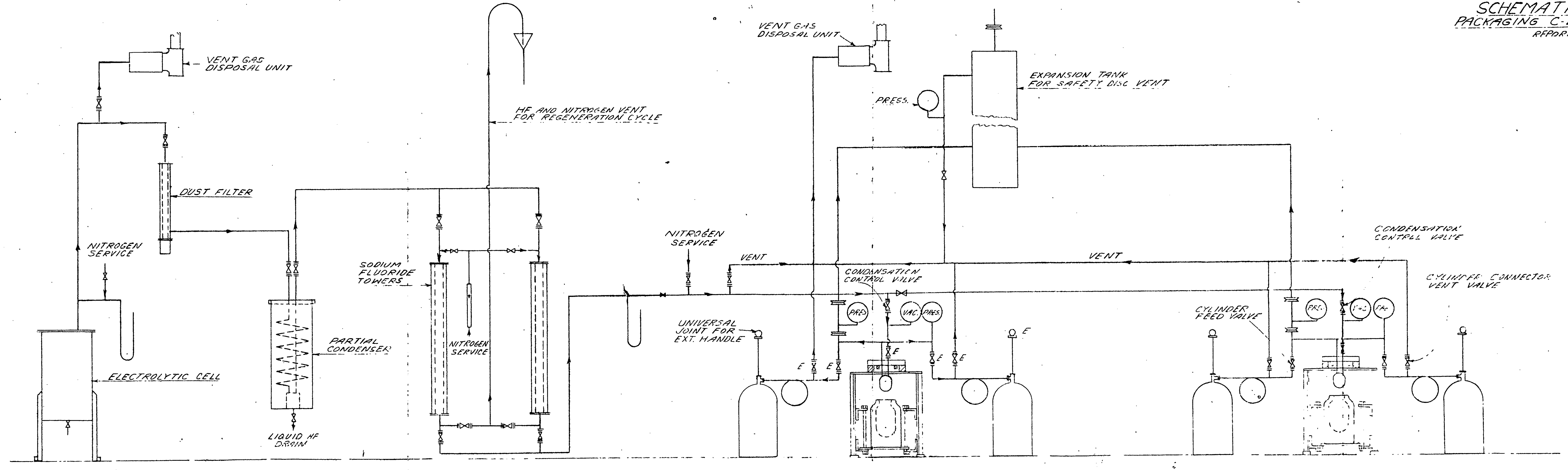
1 BW 12-6-44 COPY NO —
2E BW 5-19 45

JACKSON LAB. CHEM. ENG. DIV.
DATE: NOV. 1, 1944
DRAWN: EJ [illegible]
SCALE: NONE
APP. [illegible]

338-17

JLS-319

SKETCH II
SCHEMATIC PIPING DIAGRAM
PACKAGING C-216 - LIQUEFACTION METHOD
REPORT REF. JLD-44 & 45



JACKSON LAB. CHEM. ENG. DIV.
SCALE:- NONE
DATE:- DEC. 6, 1944
DRAWN: C. J. Clark
APP.

JLS-320

338-18

I-41 AND

1-21