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……"DEHYDRATION OF REFRIGERATION SYSTEMS"……………………

was submitted by

……………………Sri P.D. Patel……………………

and accepted for the award of Degree of Master of Engineering in ……………………

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DEHYDRATION OF REFRIGERATION SYSTEMS

P. D. PATEL

M. E. THESIS

UNIVERSITY OF ROORKEE

1962

[illegible] OF REFRIGERATION [illegible]

A THESIS SUBMITTED IN
PARTIAL FULFILMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
MASTER OF ENGINEERING
IN APPLIED THERMODYNAMICS
(REFRIGERATION & AIR CONDITIONING)

BY
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19[illegible].

-: CERTIFICATE :-

Certified that the dissertation entitled "DEHYDRATION OF REFRIGERATION SYSTEMS" which is being submitted by Sri P.D. Patel, in partial fulfilment for the award of the Degree of Master of Engineering in APPLIED THERMODYNAMICS (REFRIGERATION AND AIR CONDITIONING) of the University of Roorkee is a record of student's own work carried by him under my supervision and guidance. The matter embodied in this dissertation has not been submitted for the award of any other Degree or Diploma.

This is further to certify that he has worked for a period of Six months from Dec. 1, 1961 to June 1, 1962 for preparing dissertation for Master of Engineering Degree at the University.

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ABSTRACT.

Success of the operation of a refrigeration plant or air-conditioning plant depends on its availability. This can be brought about by minimizing the moisture in the system. Whatever may be the initial cause leading to a failure, the final failure is always due to the corrosion of the material used in the refrigeration system construction. Moisture, over and above the final failure, causes operational difficulties such as mechanical freeze-ups at the low temperature points and binding of the moving parts such as compressors, valves, bearing surfaces due to the formation of sludges and/or copper deposition on these parts.

Fundamental aspects of moisture sources and effects are reviewed in this dissertation. Moisture, its control, its measurements and removal from a refrigerant, and refrigerating system are also reviewed.

There are disagreements between various authorities on certain points as safe limit of moisture in a refrigeration system, conditions inside the system when the system is operating, desiccants and their characteristics etc. However, effort has been made to present opinions of various persons and manufacturing companies which they have derived after extensive experimental work or after sufficient experience in the respective field. Most of the persons have worked with Freon-12, being a more commonly used refrigerant.

CHAPTER I.

INTRODUCTION

INTRODUCTION

1.1. General

Refrigerants used in the early days of mechanical refrigeration were mainly ammonia and carbon dioxide, which were not materially affected by the presence of moisture. Ammonia and water form ammonium hydroxide which does not freeze readily and is not corrosive to common metals other than copper and its alloys. Carbon dioxide and water are quite corrosive in air but do not cause trouble in a closed system.

When sulfur dioxide came into use it was necessary to exclude moisture from the refrigeration system, as they chemically react to form corrosive acid. Due to toxicity sulfur dioxide was mainly used in small units either factory assembled or field assembled with factory sealed elements and tubings. Reasonable care was sufficient to prevent moisture migration into the system during installation and operation.

The modern refrigeration system is designed to operate with a given refrigerant, the inclusion of any foreign material is harmful. Dirt, air, noncondensible gases, and water are commonly encountered and must be eliminated.

1.2. Dirt.

It can be avoided by care in original installation. The major pieces of equipment such as compressors, condensers, evaporators, receivers, water chillers etc. are kept clean during manufacture, are sealed at the factory and left closed as long as possible during installation. Other parts vulnerable to dirt are usually protected by internal strainers as a further precaution.

1.3. Air and non-condensible gases.

These are undesirable in the refrigeration system because of artificially high head pressure which they cause with accompanying water wastage and increased operating cost. Air accelerates the formation of certain harmful by products by oxidation with oil and refrigerant. They are removed by evacuation in factory and by proper care during installation their introduction into the system is avoided.

1.4. Moisture.

This leaves moisture as the main trouble maker especially for halogenated hydrocarbon refrigerants because of their low water solubility and low working temperatures of the modern systems. Moisture causes freeze-ups and corrosion in the system.

1.5. Anti-freeze agents.

When the engineers and servicemen were not aware of corrosion due to moisture, they use to introduce anti-freeze agent namely methanol into the system to prevent ice formation, knowing that it does not reduce amount of moisture in the system.

In case of hydrocarbon refrigerants methanol goes in solution with the moisture and form a non-freezing solution which may circulate into the system without causing any trouble. In case of chlorinated refrigerants it is more soluble in refrigerants than in moisture and do not serve the purpose.

Methanol itself reacts with the refrigerants and lubricating oil even in bone-dry-systems to form unstable compounds which are corrosive in character.

1.3. Moisture, the insidious enemy.

It was found at the conference on Corrosion held at the January 1950 A.S.R.E.(1a*) meeting that many troubles blamed on refrigerants and oils are really traceable to the presence of excessive moisture. Therefore, the complete removal of moisture entrapped in a refrigeration system was felt necessary, for the system to give continuous, repair-free service for an extended period. Although it may seem that it should be easy to construct a moisture free system, the actual realization of such a unit is very difficult, if not impossible.

The subjects of moisture in refrigerating system, namely, corrosion and methods of testing for moisture, moisture characteristics and its safe limit, dehydration methods

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* Numbers in the parenthesis refer to the references given at the end of the thesis.

etc. have created great interest among engineers, research workers, and manufacturers of equipment.

CHAPTER II.

MOISTURE, ITS SOURCES AND EFFECTS

MOISTURE, AIR [illegible] & [illegible]

2.1. Moisture, Air content.

The following are the sources of moisture migration into the refrigeration system :

a) Manufacturers try to remove any moisture when equipment is shipped, but where assembly line methods are used, each individual unit may not get all the attention it requires, this leaves system initially wetter than it should be.

b) Pressure Vessels are usually tested hydrostatically. Coils and another parts are tested for leaks by immersion in tanks of water and a few drops are apt to get in.

c) When assembling a system in the field, where parts are subject to variations in temperature, there may be appreciable amount of condensation, some of which will be inside the system.

d) The use of torches burning C_2H_2 gas may introduce moisture into the system as one of the products of combustion.

e) Soldering fluxes may have affinity for water.

f) After installation moisture may be introduced by air used in testing or by mistake the system if purged down to a slight vacuum instead of a positive pressure, air and moisture will enter when it is opened up.

g) Refrigerant and lubricating oil may contain water. Also pouring oil into the machine is a sure way of adding air and moisture as the oil stream traps air and carries it along.

h) All solid metallic or non-metallic and porous or fibrous surfaces either in a system or outside absorb moisture e.g. hermetic motor stator insulation which has high moisture absorption capacity.

It will be also obvious that the introduction of a stator with a cotton insulation introduces a potential source for the production of water and other undesirable by-products in the system should the stator decompose of any reason, such as an electric fault, high operating temperatures or presence of acidic substances.

i) Oxidation of certain hydrocarbons of oil to produce moisture.

j) When a system has been in operation for a considerable period of time, there may be number of troubles that develop due to wear and normal servicing, if great care is not taken, they make it possible for water to get into the system.

2.2. EFFECTS OF MOISTURE IN A REFRIGERATION SYSTEM

With the presence of moisture in a refrigeration system the frequent undesirable effects are :

2.2.1 Mechanical Freeze-ups

This term is applied to the freezing up of the moisture in refrigerant at a pressure reducing valve or capillary tube where the refrigerant pressure and consequently refrigerant temperature is reduced. They are more common in systems using dry type evaporators than in flooded type of systems, as in the dry type of evaporators the moisture which may freeze up into ice in evaporator is carried through the evaporator by the velocity of gases and it thereby returns to compressor and circulates through the system so that it is in a position to freeze up again as it returns through the expansion valve.

In flooded type systems the ice remain in header of the evaporator and may not return to compressor so as to recirculate through the pressure reducing valve.(2)

When a great percentage of freeze-up results in complete blockage of the expansion valve orifice, it is not uncommon to have a partial blockage which not only starves the system during running cycle but prevents valve from closing properly during off-cycle. Off-cycle feeding, as is well known, usually results in frostback and generally erratic operation.

On the low temperature jobs, those operating below -10°C, the excess moisture precipitates out as ice crystals.

On a single stage job the crystals might not cause trouble, as the precipitation might occur after passing the expansion valve. However, on multi-stage jobs it is customary to subcool the liquid before it enters the expansion valve, which may result in ice crystals in the liquid line and clogging the valves and strainers. Ice is difficult to remove because the system must be pumped down, and warming up of the parts allowed before opening up for cleaning. After this has been done, the moisture will have evaporated and disappeared in the system(9).

However, in low temperature jobs a plugged expansion device may result due to separation of wax from oil-refrigerant mixture. The presence of wax may be demonstrated by washing the expansion device with a small amount carbon-tetrachloride and evaporating the solution so obtained. White wax of extremely viscous oil-wax mixture proves that wax is the offending element although it does not rule out moisture.

3.2.2 CORROSION OF METALS TO FORM SLUDGE.

One major concern is the damage caused by the chemical reactions which occur when oil, water, and refrigerant placed in intimate contact in any refrigeration system. The water permit a hydrolysis in presence of refrigerant and oil to form acids contributing to the formation of sludges - metallic salts.

Approximately 90 percent of the sludges produced in refrigerating systems are due to moisture, the other associated with oil or minor causes. Corrosion in sulfur dioxide system proceeds within few days or even hours, provided enough water is present, where it is much slower for methyl chloride and freon-12. This is due to very rapid action of water on sulfur dioxide and rather slow action on other two refrigerants, to produce acids for corrosion.

The process of hydrolysis is greatly accelerated by high temperatures for short or long periods or by presence of certain metals, depending upon the refrigerant.

The effects of hydrolysis will show up in etched or pitted and leaking valves and seals and excessively worn parts. Acids also tend to corrode moving parts such as pistons, bearing surfaces, causing damage and binding at these points. In hermetically sealed units it may also breakdown motor insulation or cause other trouble. The sludge formation may plug screens and increase back pressures.

3.2.3 Copper Plating.

This is not a truly descriptive term but covers the precipitation of copper on various parts of the unit. It has been demonstrated fairly conclusively that the condition incidental to copper plating is dependent upon the presence of oil and moisture or some material other than refrigerant. The effect is similar to that of sludge formation except that copper seems to be precipitated on moving parts preferentially. If carried to extremes, this precipitation will cause compressors to stick.(23)

2.2.4 Oxide formation on copper tubing

Copper tubings after annealing are cooled and if they contain excessive moisture then even stored for short periods get badly corroded. At a maximum dew point of 30°F (270 ppm water/cu.ft inner space) rate of oxide formation is very low and tolerable.(6)

2.2.5 Flapper-valve failure.

Testing has shown that relatively large quantity of moisture in a refrigeration system using Freon-12 as refrigerant, results in accelerated flapper valve breakage. The same system overhauled completely and equipped with an efficient drier, operated indefinitely without flapper valve breakage. Incipient corrosion, probably of an intercrystalline nature, was advanced as the cause of fatigue failure of the flapper-valves.(7)

CHAPTER III.

MOISTURE, ITS CHARACTERISTICS, CONTROL AND LIFT

MOISTURE, ITS CHARACTERISTICS, CONTROL & LIMIT

3.1. Moisture Characteristics.

3.1.1 Operating System

Figures 1 to 4** present moisture characteristics for the hermetic units using Freon-12 and Freon-22 as refrigerant and of capacity 5 tons and 7.5(8) tons respectively. They represent moisture characteristics under nearly stable operating and room conditions. The general character of the curves is valid for other operating conditions and total moisture content.

Figure 1 shows that a transient condition exists following the start up of a refrigeration unit charged with Freon-12. An initial increase in the moisture content in the suction line as seen in the figure 1 may be explained by the change of the evaporator from initial room temperature down to evaporating temperature. If a film of lubricating oil is present at the surface, the solubility of moisture in this film is decreased by reduction in temperature. This should cause release of moisture, which may account for the initial high value. The moisture in the suction line then decreases rapidly to a stable level.

It is also observed that moisture in the condenser vapor drops immediately to low values. As known the short term-

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** Figures are given in appendix.

over time of the charge does not permit the moisture content to be different for vapor and liquid phases during operation. The condenser vapor is assuming the average moisture current content of the total charge. Mean time the motor temperature rise caused release of moisture from the insulation. Approximately one hour is requiredto reach stable conditions and also final motor temperature, with initial moisture content of this order, refrigerant - moisture equilibria are reached in less than two hours.

The moisture characteristics of refrigeration system charged with Freon-22 is considerably different from that charged with Freon-12 as shown by Figure 8. It indicates that a rapid drop in moisture content in the condenser vapor occurs in the first few minutes of operation. It then increases slowly to reach a stable level after approximately 3 hours of operation.

When a refrigeration system is shut down, the liquid refrigerant normally transfers into the crank case where it remains in solution with the lubricating oil. During the first few minutes of operation, the liquid boils of progressively cooling the remaining solution at the same time. It may be assumed that the vapor boiling off during the initial period is carrying a low moisture content leaving most of the moisture in the liquid portion and also in the oil. With the progressing time, this moisture is released and is responsible for the increase shown in Figure 8. This condition could not occur in a unit charged with Freon-12 because here the larger portion of moisture is contained in the vapor.

During nonoperating conditions the cellulose may be exposed to the moisture contained in the vapor phase of the refrigerant. During operation the entire refrigerant is carrying the average moisture content which means for the vapor phase a considerably higher relative moisture content. The unit operating at low winding temperatures justifies the opinion that the moisture has been absorbed by the cellulose. This condition is not probable on units charged with Freon-22 since, due to the moisture equilibria for this refrigerant, the windings are exposed to lower relative moisture content during operation than under non-operating conditions.

3.1.2. After Shut Down.

Refrigerant systems charged with Freon-12 and Freon-22 respectively show great differences in their moisture characteristics after shut down also. Figure 3 presents the characteristic of the system charged with Freon-12.

Immediately after shut-down, there is a steep rise to a very high moisture value which is maintained only momentarily. The moisture content then drops rapidly and after approximately 1½ hours reaches the level which existed prior to start up of the unit. This shows the tendency to reestablish immediately equilibrium between liquid and vapor phase in accordance with data of Figure 6. However, the total moisture content is first still higher than before start up. It takes approximately 1½ hours for reabsorbing the moisture which was released from the cellulose during the first hour of operation.

Figure 4 presents the moisture characteristics after shut down for the refrigeration unit charged with Freon-22. A rapid decrease of moisture content occurs during the first few minutes after shut down. This represents again the tendency to establish moisture equilibrium between the vapor and liquid immediately after circulation of refrigerant is stopped. Moisture migrates from the vapor into the liquid phase.

If the final equilibrium were established immediately, the moisture content should be equivalent to the values for non-operating conditions of Figure 2. However, great unbalance exist for a long time after shut down. Condenser becomes the hottest part in the system due to conducted and radiated heat from the motor (condenser water being shut off and evaporator fan still operating). Refrigerant is evaporated in the condenser and transferred into the evaporator where it condenses. From there it transfers through the suction line into the crankcase and gets into solution with the lubricating oil. During this process an excess amount of moisture is left in the vapor phase accounting for the high values on the curve. Diffusion from the vapour phase into the liquid phase is greatly slowed down due to an oil-rich layer on the liquid in the crank case which presents a barrier to flow of moisture. Finally moisture is released from the motor insulation to the vapor phase which then is lower in moisture content than during operating conditions. It is this reasoning which might explain the long time required for returning to equilibrium conditions within this unit after shut down. The very slow return to equilibrium conditions is characteristic for refrigeration systems charged with Freon-12.

Winding Temperature.

Figure 6 presents effect of motor winding temperature on the moisture content of system charged with Freon-12. The increase in moisture content with winding temperature for the unit as received from the assembly line is rather small.

The increase in moisture level with rising winding temperature is steeper for the higher total moisture contents. It is important to recognize this fact. A moisture determination might greatly mislead if the operating conditions of the unit are not well defined. If for instance, a moisture test is made in the liquid phase of Freon-12 at non-operating conditions, the test tolerance should be extremely low. A small test error results in a misjudgement of the moisture under operating conditions by a multiple of its value which is the larger the higher the winding temperature is.

3.1.4 Total moisture.

Figure 7 is a work chart which gives information on moisture distribution of any unit of one particular type from a single moisture test at specified conditions. In combination with a diagram like figure 6 sufficient data on moisture characteristics are available for further study of the effects of moisture in refrigeration systems.

2.1.3 Equilibria in Freon-12 - Water Systems.

When equilibrium between Freon-12 vapor and water vapor is established then the composition by weight of the gaseous mix-ture is a function of the densities of the components and if accurate data are available, the composition can be calculated without resort to the gas laws. It is only necessary to assume that the density will follow the simple additive relations based on Dalton's law.

If this assumption is valid, the following equation(D) will hold for composition of gaseous mixture.

$$\text{Percent water} = \frac{100\ W_{water}}{W_{Freon}} = \frac{100\ d_{water}}{d_{Freon}} \quad .... (1)$$

where W_{water} and W_{Freon} are weights of water and Freon respectively in the vapor phase.

d_{water} and d_{Freon} are densities of pure saturated water vapor and pure saturated Freon vapor respectively.

Percent water is the percent by weight of water in the vapor phase based on the anhydrous Freon. This value may also considered to be the percent of water in the total vapor mixture, since there would be a negligible addition by including the density of water vapor in the denominator.

Liquid composition data for saturated solutions are given by the solubility chart, Figure 8,(7) and may be applied over the same temperature range. These data combined with the calculated vapor composition data to derive the relationship between the moisture contents in the vapor and liquid phases of saturated solutions of water in Freon-12. This relationship is most simply expressed as a ratio of the concentrations of water in the two phases. Such ratios as well as data from which they can be derived are tabulated in Table 1.(8)

It is safe to assume that the ratios of moisture distribution shown in the last column of Table 1 will apply to less than saturated solutions of water in Freon, if it can be assumed that the Henry's Law is obeyed. Exact obedience to this law is to be had only when the dissolving gas (vapor) follows the gas law, and water vapor does not act as an ideal gas at temperatures under consideration. It is not safe to assume that solutions of Freon-12 vapor in water would confirm to Henry's law except very low Freon vapor pressures.

It is concluded that Henry's law does apply to solutions of water in liquid Freon-12 and that the ratio derived for saturated solutions may also be used for more dilute solutions. Thus for any concentration of water in Freon-12 it is possible to calculate the moisture distribution between liquid and vapor phases and from a knowledge of water concentration in one phase to predict accurately the concentration in the other phase at equilibrium.

3.2 MOISTURE CONTROL.

3.2.1. Hermetic Units.

In hermetic units amount of moisture can be controlled initially and in service life within a tolerable limit by the following ways :

1. Low Initial Moisture Content.

A compressor motor stator insulation should be such that its moisture holding capacity is low. Small size electric motors currently use Paper - Formvar - cotton insulation(20). This has disadvantages such as high initial moisture content, low thermal stability and evolution of water during operation.

A high initial moisture content compells the manufacturer to perform lengthy dehydration operations and low thermal stability limits dehydration and operational temperatures.

A Polyester - Acrylic insulation in place of former one is better because of its low moisture holding capacity, high thermal stability and low moisture evolution. This requires less dehydration time and can work safely at high temperatures.

By taking proper care during transportation and installation of a dehydrated compressor-motor unit initial moisture content can be reduced.

2. Service Life Moisture Content.

By properly selecting insulation material and controlling operation temperature within limit prescribed by manufacturers, water evolution and subsequently its bad effects can be eliminated.

3.2.3. Non-hermetic units.

In Non-hermetic units amount of moisture can be kept within tolerable limits by thoroughly dehydrating the equipment, with reasonable care during installation and during operation, to avoid moisture introduction into the system and finally by selecting proper lubricating oil.

3.3. Safe Moisture Limit.

The charge of a refrigeration system consists of variety of substances such as refrigerant, lubricating oil, and other impurities, one of which is moisture. The charge is exposed to and in contact with numerous different metals and perhaps their oxides and organic materials. Also great temperature difference exists at various points in the system. From all this and other reasons it is rather difficult and also not significant to give a single answer to safe moisture limit in the system(9).

With regard to mechanical freeze up safe limit can be determined by knowing temperature at the expansion device. The dew point of refrigerant should not exceed this temperature. However, this is not correct because there is no place in the machine where ~~is~~ there is a complete cross-section of travelling liquid, and water solubility in liquid is phase and vapor phase of refrigerant is not same.

Some limited information regarding the water content of machines that are operating satisfactorily is as follows :

For hermetic units; small and large commercial units using F-12 safe moisture limit is 25 to 30 PPM by weight and for domestic hermetic units upto 37 PPM by weight. These small commercial units using CH_3Cl operate satisfactorily at 50 to 60 PPM by weight(7).

The corrosion of metals occurs whenever the quantity of water exceeds certain values. Table 8 contains the results of tests conducted(9) on steel in glass tubes where the quantity of water could be closely defined.

Limits for copper, brass and aluminium are slightly higher in all three refrigerants. Aluminium in methyl chloride is eliminated from moisture corrosion consideration, since a direct action occurs resulting in the formation of spontaneously flammable products. Higher temperatures, encountered in the compressor and condenser, will lower these limits appreciably, so that corrosion occurs with less moisture in a machine at these points than with steel at room temperature.

METHODS OF MOISTURE DETERMINATION
IN REFRIGERANTS AND REFRIGERATION [illegible].

4.1 REFRIGERANT SAMPLING :

Refrigerant samples are drawn either in gaseous form or liquid form from a refrigerant cylinder or a refrigeration unit whose moisture content is to be determined or from a reference refrigerant cylinder used for comparison. A poor sample will not yield good result regardless of how good the moisture determination method may be.

4.1.1 SINGLE CYLINDER OR TANK :

(a) GAS SAMPLING :-

It is impossible to take a gas sample of a refrigerant from a single cylinder or tank containing liquid and have it representative of the whole unless all of the material is vaporized.(21) Daley and Flowers(D) have shown that the liquid analysis of Freon-12 can be deduced from the vapor analysis. Often this is quite difficult because of the small amount of moisture in the vapor phase. If some Freon-12 vapor is removed, the new vapor formed to take its place will be drier.

With rapid refluxing the total water can be obtained by taking no more than 10 per cent of the total refrigerant. If weight of the refrigerant is known then PPM water content can be calculated otherwise only total amount of water in the given refrigerant can be known. This method cannot be used for Freon-22 and methyl chloride or such refrigerants.

(b) LIQUID SAMPLING :-

Liquid sample can be far from representative for those refrigerants which are auto-drying(12), i.e., which dry themselves in the liquid phase on boiling e.g. Freon-11, Freon-12, Freon-113 Freon-114. For sampling a single tank or drum, the liquid is preferred and should be taken immediately after the whole is shaken thoroughly. However, the gaseous sample can be shipped without the hazard as in case of liquid sample.

4.1.3 REFRIGERATION UNIT :-

To sample a refrigerant from a refrigeration unit correctly, one must take certain precautions depending upon what the analysis is to be used for and what condition the unit is in. If it is desired to learn precisely how much water is in the refrigerant, when the refrigerant is at equilibrium in an operating unit, the system should be operated until steady state run conditions are established. Valves should be installed in the system, so that the liquid refrigerant can finally be trapped between them. The trapped refrigerant can be taken through the exit valve by either the liquid or gas sampling technique using the ice water around the receiving cylinder.

An alternative method would not make use of valves to trap the refrigerant sample. This is especially appropriate for commercial units having no valves in the circulating system.

Figure 9 illustrates how sample is taken. The refrigerant is obtained through an exit valve placed between the compressor and the condenser. With valve in this location, the liquid will be pushed immediately into the sampling cylinder. However, good samples can be obtained, if gas is taken from the condenser.

The sampling cylinder is dehydrated in an oven at 300° F, nitrogen being passed through the cylinder. While still hot it is removed and pumped to a pressure as low as 25 microns. Once the cylinder is connected in place to receive the sample, it is again evacuated to about 40 microns, primarily to ensure that there will be no leaks of a fairly large magnitude. Loss of small amount of the refrigerant will change the moisture content by a rather large amount.

Once the sampling is started, the exit valve from the unit is left open until all the liquid has left the evaporator. Immediately after the sample cylinder has been disconnected, tests should be made to find if the refrigerant is leaking out. Due to the high tendency toward fractionation in the water and the Freon-12 system, leakage cannot be tolerated.

The other advantage of operating unit till steady state is reached is that such precaution prevents the samples being contaminated with oil which would ruin analysis and wreck Phosphorous pentoxide absorbent.

If conditions of operating were such as to give wide variations in temperature then the units using the method of trapping do require temperature control during sampling. If trap method of sampling is used then desiccant temperature must be approximately constant.

A commercial unit containing drier unit (where desiccant holds more water than refrigerant) and an exit valve can be examined without its being necessary to capture the factory charge.

The line can be cut, after unit has operated for 3 hours, allowing the charge to escape. Valves now can be installed and the unit recharged with refrigerant and the moisture content of the unit do not change.

4.2 MOISTURE DETERMINATION METHODS :-

The main requirements for the method used for determining moisture in operating refrigeration system are :-

i) No changes should be made on standard refrigeration system.

ii) The charge of the system should not be affected by the moisture tests to such an extent that the operation or characteristics of the unit would be changed.

iii) The moisture determination should be reasonably accurate for the refrigerant even if contaminants like oil, and its additives were present in the form of a solution and / or mixture with the refrigerant.

It should be realized that for the very low moisture contents desired in refrigeration systems, the effect of impurities is of greatest importance. Each time lines are connected the danger of introducing moisture is leakage. Minute amounts can effect a proper measurement. Generally, a method should be preferred for repeated measurements at which the instrument for measuring the moisture is constantly connected to the refrigeration system. Furthermore, a single error in measurement will not be of detrimental effect in a method which determines an instantaneous moisture content since at least a few measurements are made. A measurement that is in doubt can in most instances be repeated while an integrating method does not provide this opportunity.

CHAPTER - IV.

METHODS OF MOISTURE DETERMINATION IN REFRIGERANTS AND REFRIGERATION SYSTEMS.

prepared by shaking in a stoppered bottle two parts by volume of phosphorus pentoxide with one part of Gooch asbestos fibers which has been dried at 105° C. The reagent is packed loosely in the jars on a ½ inch layer of glass wool and a layer of glass wool is also provided over the reagent. These jars should be kept in a constant temperature cabinet during the analysis steps and be removed only for weighing.

6.3.1 PRELIMINARY SAMPLING :-

Due to the well known effect of flash gas, the sample will have an interesting inaccuracy depending on the refrigerant used. Table 3(12) shows the amount of liquid taken from the drum in order to obtain 100 CC sample in test flask. However, the flash gas does not carry its equivalent amount of moisture hence the percentages obtained in this test will be slightly high or low depending upon the refrigerant.

Table - 4(13) Gives the results of analysis made on a warm, humid summer day. It shows effect of different sampling methods on moisture content of sulfur dioxide. The results given by the ordinary method were obtained(13) by drying the sampling flasks at 105° C and then without preliminary washing and purging of the flasks with sulfur dioxide, the samples were run directly from the cylinder into the flasks without the use of connecting nipples. The results given by standard method(13) were obtained by observing the precautions in sampling.

6.3.2. [illegible] :-

All the moisture is picked up in the first absorption jar. Any change in weight in the next two jars is averaged and used as correction factor for the gain of the jar number one. The change in weight of other two jars is due to changes in barometric pressure or small changes from the temperature at which the jars were first weighed. The differences encountered are significant but are small and may be either positive or negative. They serve as a practical method of arriving at the correction which should be made to the weight of the moisture collected in the first jar.

Then percent moisture by weight =

$$= \frac{\left[\Delta W1 - \left(\frac{\Delta W2 + \Delta W3}{2}\right)\right] \times 100}{VG} \qquad (8)$$

where, $\Delta W1$ = grams. Net gain in weight of jar No. 1.
$\Delta W2$ = grams. Net gain in weight of jar No. 2.
$\Delta W3$ = grams. Net gain in weight of jar No. 3.
V = CC. volume of liquid refrigerant sample.
G = specific gravity of liquid refrigerant at its boiling point.

If the absorption jars could be weighed filled with air, the preliminary procedure of gassing them with the refrigerant could be eliminated. However, long time required for in sweeping the refrigerant and if not swept properly then due to great difference in density of the refrigerant and air erratic results will be obtained. But the other materials in the vapor sample are also absorbed by the absorbent on its surface hence flushing of the jars with dry air before and after running the sample is sometimes adopted.

4.3.3 [illegible] :-

1) To get the accurate results i.e. to minimise error in

weighing more amount of sample has to be taken.

ii) It requires very long time to obtain measurement.

iii) It fails when oil or other impurities are present in the refrigerant sample, because the increase in weight does not represent the moisture content of the refrigerant only, but of the moisture plus oil or impurity.

However, silica gel (24) used as an absorbent and the refrigerant drier installed in the refrigeration system removes moisture from the oil by repeated recirculation and drying. The moisture thus absorbed is transferred to absorption jars and thus separated from the oil trapped in the drier.

iv) Elaborate temperature controls are necessary because of the necessity for controlling temperatures in various parts of the system.

From all these it is concluded that this method is not suitable for moisture determination of an operating refrigeration system. It is suitable for pure refrigerants only.

INFRA-RED SPECTROPHOTOMETRIC METHOD.

Gravimetric method has been accepted as a standard method for many years, but because of its lengthy and complicated procedure with good many precautions required for accurate results, it is not a widely used method. Also modern units requires very low moisture content and [illegible] 1 within this limit necessitates a rapid and accurate method for analysis. This requirement is fully met by this method, as it eliminates human error, as in case of gravimetric method and make it possible for the average analyst to obtain good result.

4.4.1 THEORY :

In the small portion of the spectrum represented by visible light we are familiar with changes in colour with wave-length. The violet light at the short end of the visible range has a wavelength of about 0.4 micron. (The micron is equal to 0.0001 cm) At the other end of the visible spectrum, dark red light has a wave-length of about 0.7 micron. Immediately beyond the red lies a much broader segment designated 'infrared' and extending to about 1000 micron.

The region of 2 to 25 micron is particularly useful as within these limits many chemical compounds show selective absorption, characteristic of their molecular structures.

The modern infrared spectrophotometer includes a source of radiation, a cell for confinement of the sample, a monochromator for dispersion of the transmitted light over the spectrum and isolation of any desired segment thereof, a detector which responds to infrared radiation falling upon it, an amplifier, and finally, an indicator. (See Fig. 11).

1. The light source is usually a substance heated electrically to a dull red heat. Most commercial instruments employ an incandescent rod of silicon carbide [illegible] (a globar) or of rare earth oxides (a Nernst Glower). Optical parts used to focus the beam before passage through the sample are either front surface mirrors or lenses made from a transparent salt crystal.
2. The sample is contained in a cell ~~fitted with~~ which is a metallic tube with windows which transmit infrared light of the desired wave length. In passing through the sample, the beam loses to various extents those wavelengths absorbed, and the characteristic absorption spectrum of the sample replaces the essentially continuous spectrum of the source. However, the change in the nature of the beam is not apparent at this point in the instrument.
3. In the monochromator, the beam is dispersed (or refracted to a degree varying with the wavelength) to permit the detector to measure the energy of transmitted radiation along the spectrum. Infrared radiation, like visible light, is dispersed by a prism. However, since ordinary glass is opaque to infrared light, prisms for this purpose are constructed of sodium chloride, fluorite, or other material transparent at the required wavelength. The beam from the cell enters the monochromator through a slit and passes through the prism. Many instruments obtain increased dispersion ('stretching' of the spectrum) by passing the beam through the prism twice. An exit slit allows only a

narrow, essentially a monochromatic section of the spectrum to be passed over the exit slit - for example, by rotation of a mirror in the optical path.

4. Infrared radiation may be detected and measured by either the electromotive force of a thermocouple or the change in conductivity of a bolometer.
5. The effect of the infrared radiation upon the detector is transmitted, as an electric signal, to an amplifier where it is built up to a level suitable for operation of the indicator.
6. A milliammeter is usually the most suitable indicator, being much more stable than a galvanometer . The deflection of the needle is thus a function of the radiant energy reaching the detector and in turn of the absorption in the sample cell.

The absorption of infrared light, in the absence of complicating factors, follows Beer's law which may be expressed as follows :-

$$- \log (I/I_0) = klc \qquad \text{———————} (3)$$

In this expression,

I_0 = energy of incident beam.

I = energy of emergent beam.

k = a constant for a given substance, usually called the extinction coefficient.

l = length of path through the sample.

c = concentration of sample.

The term $-\log (I/I_0)$ is called the optical density and is a measure of the opacity of the sample; (I/I_0) (100) may be termed the percent transmission.

In the experimental evaluation of infrared spectra, the quantities directly measured are I_0 (with empty cell) and I (wit sample cell filled). Their ratio, I/I_0, multiplied by 100 gives the percent transmission and most infrared spectra are plotted with percent transmission as the ordinate and wavelength as the abscissa. (See Fig. 12). However, when infrared absorption is used as a measure of concentration as in the subject method, the plot of percent transmission vs concentration is not linear.

A linear plot, more useful for interpolation and extrapolation, is obtained by plotting the optical density vs concentration in accord with Beer's Law.

As mentioned, the complete infrared absorption spectrum of a compound is a unique property resulting from its molecular structure. However, at one wavelength it is possible that two or more compounds may show appreciable absorption, and in such a case they would interfere with analysis by infrared absorption at that wavelength. The subject method is made possible by the fact that water strongly absorbs infrared light of 2.67 micron wavelength and liquid Freon-12 does not.

Referring to Fig. 12, the very strong band at 2.0 micron is due to absorption by liquid Freon - 12. The instrument should be set as closely as earlier calibration would permit to a wavelength of 2.67 micron and then adjusted very carefully to a point of maximum absorption. The optical density of the sample is determined at this point and converted to water concentration in ppm (parts per million) by reference to Fig. 10. This curve is based on the measurement of optical densities of a number of reference samples with water contents carefully determined by a standard phosphorus pentoxide method(25). A 4 inch cell was used in obtaining these data(15) and if a cell of different length must be used, the results should be corrected in accordance with Beer's Law.

4.4.2 DEMERITS :-

a) This method requires a relatively large sample of refrigerant to assure high accuracy of the measurement especially for low moisture contents, and also requires elaborate and expensive apparatus and highly specialised skill.

b) It is possible to use this method on a microscale for high moisture concentrations but the technical difficulties involved in a micro determination when very low moisture contents are present, make it impractical for refrigeration systems.

c) Oil like most hydrogen containing compounds shows absorption of infrared light at nearly the same wavelength (2.67 microns) as the hydroxyl band in water (26). If any oil is present in the sample, its broad absorption in this region greatly decreases the water sensitivity. Separation of small amounts of oil in solution with the refrigerants is very difficult. For these reasons, this method is not considered practical for testing operating refrigeration systems.

4.6 DEW POINT METHOD :-

Determination of water content of a sample of Freon - 12 has been a laborious and time consuming task which has called for the most painstaking work not only in analysing the sample but in preparing it for analysis and in guarding it against the entrance of additional moisture before and during the course of analysis.

Here presented a dew point method with its limitations. It has been used for moisture determination in air, refrigerant and many industrial gases. Here a simple apparatus is described to avoid the use of expensive equipment and skilled technicians.(17)

4.6.1 DESCRIPTION OF APPARATUS :-

In Fig. 14, A and B are two copper drums fitted with Hoke precision needle valves. The drums are attached through compression couplings to a copper tubing manifold which is joined through rubber tubing G to the dew point cell C, which consists of two mating vessels joined at the top by a glass ring seal. The inner vessel is closed at the bottom by a metal cup made by Kovar alloy.

After the Kovar cup has been sealed to the glass, it is highly polished and plated with bright nickel. The incipient 'Dulling' of this mirror like surface through the deposition of dew and frost is the end point to be sought. The lower half of the outer vessel can be removed by separating the ground glass joint. This gives access to the Kovar Cup so that it can be cleaned and repolished if it becomes dirty.

The glass side tube through which the Freon is admitted to the apparatus extends into the vessel and almost touches the Cover Cap. This construction tends to localise the area on which dew initially forms and thus increases the sensitivity of the apparatus. The Freon leaves through the side tube and flexible rubber sleeves G into the rotameter F. The rate of Freon flow should be kept constant.

A rapid flow of gas increases the amount of water vapor passing through the apparatus, and thus the amount of dew that may be formed, but it also makes it more difficult to keep the temperature of the mirror and of the coolant the same.

4.6.7 PROCEDURE FOR ANALYSIS :-

To prepare for an analysis, the assembled apparatus is flushed out with dry gas from drum D. The dry gas may most conveniently be Freon - 12 although it can any other gas which is non-reactive and non-condensible under the conditions of the test. Oxygen, Nitrogen or Hydrogen can be used to flush the cell and to stir the coolant.

After flushing is completed, the inner vessel is filled with the coolant. Freon - 113 which boils slightly above room temperature is most satisfactory liquid for this purpose. It is denser than dry ice which is used as the cooling agent and hence the dry ice floats on the surface of Freon - 113. The use of a less dense coolant in which the dry ice will sink, is thoroughly unsatisfactory as bumping and boiling, even to overflow, will occur. Freon-

-113 vapors are also nontoxic and the freezing point of this liquid is well below the range of any dew point found with Freon - 12, samples.

A well evacuated sample drum is attached to the gas phase of the liquid receiver of a unit and filled with Freon - 12 vapor. It is assumed that the removal of one or two ounces of gas from a large receiver will not materially disturb the equilibrium. Now the sample drum is attached to the apparatus, and dry ice in small quantities is added to the coolant which is stirred continuously by bubbling a nonreactive gas through it and as the temperature of the dew point cup slowly falls, a mirror like surface is observed for the appearance of the visible dew or frost. As soon as the dew appears the cooling of the cup is stopped and without disturbing the flow of the sample, dry gas from drum B is introduced into the cell along with gas under test. The introduction of this dry gas dilutes the water present in the sample so that the gas phase becomes unsaturated with respect to water and the dew evaporates, then the gas is turned off. If dew immediately reforms, it is obvious that the temperature of the cup has been lowered well below the dew point. Hence the supply of dry ice is cut off while stirring of the coolant is continued. This causes the Kovar Cup slowly to rise in temperature until the dew no longer condenses. Juggling of the temperature up and down by fractions of a degree will allow the maximum temperature at which dew can just be made to form to be estimated and this may be taken as the dew point.

The temperature read by thermometer D will not approach that of the metal surface on which dew collects unless the coolant is well stirred and is not changing in temperature rapidly and if too rapid a flow of gas through the cell does not warm the metal faster than it can transfer the heat to the coolant. A simple and accurate method to measure the dew point of Freon - 12 is that refrigerant from the sample drum is passed through a magnesium perchlorate absorption train and the water in the gas sample is determined. Immediately after this determination gas sample is passed through the dew point cell at the same rate as it had passed the absorption apparatus and then a moisture content is found and compared with that found by absorption train.

4.6.3 DEGREE OF ACCURACY :-

Fig. 15(17) is based on Stock's data for the dew point of air and on the Kinetic Chemicals thermodynamic tables for Freon - 12. This relation is true when the Freon - 12 is at 14.7 psia, for other pressures it can be corrected.

Practically, though not theoretically, the precision of the dew point method at least with the simple apparatus described here, falls rapidly as the dryness of the Freon increases. The Kovar cup has the large surface and dew will collect over all of the metal when its temperature is at or below the dew point of the gas flowing through the same cell, except on the spot which is directly opposite the jet of the incoming gas. This spot is warmed by the hot gas is never coated by dew unless the cup is far below the true dew point. The accuracy of measurement increases at any temperature as the time taken to build up visible film is increased. The more nearly the cup temperature approaches the true saturation temperature or dew point, the longer time will be required to build up a visible dew or frost film.

localizing the cooling of mirror by directing the cooling gas against its rear surface at the point of attachment of thermocouple, is desirable and is responsible for the quick response and the high sensitivity of the instrument even at low temperatures.

The dew point method has no value in estimating the wetness of supply Freon - 12. The temperature at which dew will separate from a gas sample drawn from receiver is determined only by the water content of the refrigerant gas and not at all by any other gaseous compounds which are with water vapor unless they are very soluble in water or chemically react with it.

After the dew point has been satisfactorily determined, dry ice is added to the coolant and the temperature of the dew point cup is lowered until Freon 12 itself condenses. The temperature at which Freon will just condense is estimated as accurately as possible and by the use of Table 8, and the measured barometric pressure at the time of the experiment, the partial pressure of the noncondensible gas and of the Freon can be estimated readily.

4.0 ELECTRIC CONDUCTOMETRIC METHOD :-

The determination of the water content of Freon-12 (dichlorodifluoromethane), or of any other refrigerant that is circulating in a refrigerating system is a problem that has not yet been solved adequately. Although several methods for determining the moisture content of refrigerants by direct sampling are available, none of these is readily adaptable to a circulating refrigerant. Two serious objections can be raised against them. First, the removal of any sizable sample of the refrigerant will upset the steady state condition of the cycle; and second, unless the sampling is carried out with extreme care, the portion obtained may not be a representative one.

4.0.1 GENERAL METHOD :-

An electrical method for measuring the water content of gases developed and used for many years by E.R.Weaver(10) has been successfully applied in measuring the water content of Freon - 12, circulating in a refrigerating system. The method is fast, simple, sensitive and does not require the removal of any sample for the determination. Furthermore, determinations may be made repeatedly without difficulty. Using this method, it is possible not only to determine the wetness of a refrigerant, but also to follow and determine continuously by its instantaneous water content before and after passage through a drier.

In this method a film of hygroscopic material (usually a mixture of sulfuric and phosphoric acids) is applied to a detector in a pressure-tight enclosure and exposed to the gas containing the water vapor that is to be determined. The film reaches equilibrium rapidly with the water vapor in the gas, forming a solution the electrical conductivity of which is a measure of the water content of the gas. To make the electrical measurement, a simple electronic circuit and a microammeter are used. A gas of known moisture content is used as a comparison gas to calibrate the film after each reading. This is done by adjusting the pressures of the two gases (or simply by adjusting the pressure of either one of the gases) until the same conductivity is obtained. The unknown water content can then be calculated from the two pressures and the known water content of the comparison gas.

As a first approximation the following formula may be used for computing the unknown water content :-

$$W_x = W_c \frac{P_c}{P_x} \qquad \text{———————}(6)$$

where, W_x = unknown water content at atmospheric pressure (14.7 psi) in ppm by weight, or any other convenient unit.

W_c = known water content of comparison gas at atmospheric pressure in the same unit as above,

P_x = absolute pressure at which a conductivity reading is obtained on the gas under test (absolute pressure equals gage pressure plus 14.7 psi)

P_0 = absolute pressure of the comparison gas at which the same conductivity reading as for the gas under test is obtained.

For more precise work, especially at higher pressures, the deviations from the ideal gas laws of all the gases involved including water vapor, have to be taken into account. At the low working pressures of a refrigerating unit, the difference between the deviations of the water vapor-Freon system and those of the water vapor air system from the ideal gas law is so small that it may be neglected without introducing serious error.

The comparison gas generally used is one that has been saturated at a high pressure (about 1000 psi) at room temperature in a specially designed saturator and permitted to expand to get a comparison reading. For very dry systems, a secondary comparison gas is used. The latter is nothing more than a cylinder of fairly dry air, the moisture content of which has been previously determined by comparison with the saturated air.

4.6.3 APPLICATION OF METHOD :-

Because of the pressure relationships involved and because a liquid would wash any part of the hygroscopic film, the electrical method can only be used to make moisture determinations in the gaseous state. There are two possible ways of making such determinations on a circulating refrigerant: First, directly on the refrigerant vapor in any space not completely filled with liquid (preferably the receiver); or second, on a completely vaporized portion of the liquid refrigerant. In the first method, vapor from the receiver is passed through a pressure-tight enclosure holding the detector, where the moisture determination is made, and is then returned to the system by way of the suction line. This method is simple and direct, but unless the water distribution ratio between the vapor and liquid phases in the receiver is known at all temperatures, and under all conditions of operation, it can lead to erroneous results. Elsey and Flowers(D) present water distribution data based on vapor density calculations but whether this information is applicable to the complex situation found in a circulating refrigerant must be determined by further studies before the reliability of this method is established.

The second method avoids such complications, but it necessitates the use of a somewhat more involved sampling system, which includes a heater for vaporizing the liquid Freon. To prevent fractionation which would take place when only a part of the liquid sample is vaporized, it is necessary to withdraw the sample through a capillary tube (or equivalent) so that the entire sample drawn is continuously vaporized. Once the sampling and vaporizing system is installed, moisture determinations can be made rapidly and continuously.

Provided the reliability of the first method is adequately established, either one or the other of the two methods can be used to make moisture determinations on a circulating refrigerant. For some applications the first method is suitable; for others the second method must be used.

6.6.2 APPARATUS :-

The dual sampling and vaporizing is shown diagrammatically in Fig. 16. The two refrigerant sampling points are located close to and on either side of the drier. A capillary tube dipping into the main stream of liquid Freon at the sampling point on the inlet to the drier carries a portion of the liquid Freon to an electrical resistance heater, the electrical input of which is adjusted to supply the required amount of heat to vaporize the Freon. To insure complete vaporization, the tubing containing the liquid Freon is wound loosely around the heater in a helix and is thermally insulated from the ambient air. The vaporized Freon is then led to the detector block - a specially built, pressure tight enclosure containing the detector. Details of the detector block are shown in Fig. 17.

By manipulating three control valves in the detector-block, either Freon vapor or the comparison gas can be caused to flow past the hygroscopic film on the detector. The detector-block is so constructed, however, that the flow of Freon vapor through it is never interrupted; it is merely cut off from the detector. At all times the Freon flows through a passage in the detector-block, then through the annular space around the stem of the middle control valve, and out through the other side of the detector block. Since the sensing surface of the detector is very close (within 1/8 in) to the seat of the middle valve, when this valve is opened the flowing Freon immediately comes in contact with the hygroscopic film. To prevent condensation of the Freon in the detector-block, the heater is placed near enough to keep the Freon heated to just above the saturation temperature, at the pressure of the liquid line. (For the usual pressure range encountered, a temperature of 100° F was sufficient to prevent condensation). After passing through the detector-block, the vapor proceeds to a valve block where a regulating valve limits the flow through the circuit so that very little refrigerant by-passes the evaporator, and from there it is returned to the suction line for recirculation. The sampling and vaporizing system for the outlet of the drier consists of an identical arrangement.

The modified mechanical connections are recommended for testing compressed gases to meet the requirements of the job at the hand. The gas connections are so arranged that:
(1) the same cylinder of compressed air supplies the 'dry' as well as the 'wet' comparison gas, and (2) either of the comparison gases can be directed to the two detector-blocks. Fig. 18 shows the arrangement diagrammatically. To prevent the incoming comparison gas from cooling the detector-block during the comparison reading, the tubing carrying the comparison gas to the detector-block is wound loosely around the same resistance heater used to vaporize the Freon. Thermocouple measurements have shown that(10) for any given series of readings the temperature of the detector-block and of the gases flowing through it remained constant with this arrangement.

The refrigerating unit used (shown in Fig. 10) is a ½ hp. model equipped with a belt driven, open type compressor, an air cooled condenser, a liquid receiver, and an oil separator. Refrigerating load is imposed by means of electrical resistance heaters applied to the evaporator coil. The load is controlled by using a Variac to adjust the amount of current through the heaters. It also shows point for water addition.

(19)

4.4.3 PROCEDURE AND EXPERIMENTAL RESULTS :-

Fig. 20 presents the type of drying curves plotted against time obtained by adding specified amounts of water to the refrigerating system equipped with a silica gel drier. The curve for instantaneous moisture content of the inlet side of the drier is shown by the solid line, that for the outlet side by the broken line. The irregularities in the descending slopes of the curves are due to chiefly to local moisture variations through out the circulating refrigerant. They become quite pronounced when a large amount of moisture is added. When a system is over loaded with water so that some free water remains undissolved either in the form of an emulsion or floating on the refrigerant in the liquid receiver or both, an irregular platform rather than a peak is obtained in addition to the marked irregularities.

4.4.4 EVALUATION OF METHOD :-

(20)

Fig. 21 presents the comparison of methods for determining the moisture in Freon - 12. The methods compared are electrical and gravimetric methods and Fig. 21 A is by first method in which sample Freon is obtained from the vapor phase of a cylinder of commercial Freon. The area under the curves gives the weight of water as determined electrically. The irregularities in this and in the subsequent curve are caused by the changing water content of the vapor as influenced by the changes in the temperature and in the rate of flow at the source. Any point along the electrically determined curve gives the instantaneous water content of the Freon vapor. The weight of the water obtained gravimetrically is indicated by the horizontal broken line. No point along this line has individual significance because the gravimetric method is incapable of detecting rapid changes and can only measure the total moisture of the selected time interval of the test.

In the second method, the detector block is placed directly in the line from the cylinder to the absorption train so that electrical determination could be made on the sample of Freon that flows through the absorption train. The type of curve is in figure 21 B.

4.6.6 CONCLUSIONS :-

This method provides a rapid and reliable means of measuring water content of Freon-12 circulating in a refrigerating system. Repeated moisture determinations of the instantaneous water content of the refrigerant can be made without difficulty and the behaviour of the system under various conditions of water content can be closely followed . Very small sample for determination purpose and to bleed the lines is required.

(D)

The effect of contaminants from a refrigeration system. Oil is objectionable when it deposits on the surface of the hygroscopic film on the detector but cleaning and recoating of the detector can be done in about one minute.

However, care is necessary to prevent hygroscopic materials from entering the indicator system because these may affect moisture determinations by absorbing or releasing moisture. This contamination could result in a change in the moisture content of the sample.

4.7 ELECTROLYTIC WATER ANALYZER METHOD :-

In the last few years the moisture content of 'Freon' products has steadily decreased. With this lower moisture level, in the 0-10 ppm by weight range, there have been analytical problems, to analyze for moisture at this level accurately, precisely and rapidly. The need was felt for equipment that would analyze for water continuously and automatically in the process stream. An instrument has been developed, (DP) an electrolytic analyzer, which is applicable to the moisture analysis of continuous as well as discrete samples.

PRINCIPLE :-

The principle of analysis is based on the electrolysis of water, which has been absorbed by a thin viscous film of phosphoric acid. The analyzer requires a gaseous sample; so liquid 'Freon' compounds are continuously vaporized. The vapor is passed through a sensitive element where the phosphoric acid film absorbs the moisture. The phosphoric acid film surrounds two platinum electrodes imbedded in the sensitive element. A dc voltage applied across the electrodes continuously electrolyzes the absorbed water in the film to hydrogen and oxygen. The dc current flow resulting from the electrolysis is directly related to the amount of water vapor entering the sensitive element according to Faraday's Law. At a sample flow rate of 100 cc per minute of vapor, this current is 13.2 μamp per ppm by volume of water. The current is directly proportional to water concentration, the vapor flow rate, and the density of the vapor at any given temperature.

Faraday's Law for gas samples containing water says that

2 x 96,500 or 193,000 coulombs are required to electrolyze 1 mole of water. By application of the gas laws, it is found that at a sample temperature of 25° C, an ambient pressure of 1 atmosphere and a vapor flow rate of 100 cc per minute, a gas containing water at a concentration of 1 ppm by volume will carry 4.09×10^{-9} moles of water through the sensitive element per minute. The size of the theoretical electrolytic current under these conditions is, therefore,

$(2)(96,500)(4.09 \times 10^{-9})(1/60)$ coulombs/sec/ppm by volume of water, or 13.2×10^{-6} amp per ppm by volume water.

So long as vaporization of the liquid 'Freon' compound is 100% complete, the concentration of water in the liquid and vaporized samples is the same. This volume percentage of water is converted to a weight percentage by dividing by the ratio of the molecular weight of the 'Freon' compound being analyzed to the molecular weight of water.

FLOW DIAGRAM :-

Fig. 23 is a schematic diagram of the analyzer and its sampling system. The liquid 'Freon' compound enters an adjustable vaporizer, a finned and elongated needle valve. The vapor sample enters the analyzer and passes first through the sensitive element to minimize response time. The remaining components control and allow flow measurement: a Moore controller, a [illegible] adjustable restrictor, and a flow meter. A bypass valve and flow meter are provided also to improve the response time. Some models with ac actuated power supplies are equipped with current actuated solenoid valves which automatically permit the sample to bypass the sensitive element when the water level exceeds a prescribed limit. This arrangement can be used to saturate a high-level alarm. Excess water in the sensitive element, above 2% by weight, results in saturation, the sensitive element has a finite capacity for electrolyzing water.

The electrical circuits, Fig. 23, are extremely simple, consisting essentially of a source of direct current and a micro-ammeter. Generally this current is supplied by a 45-v battery but some models use a 75 or 90-v dc power supply. A range selector switch with appropriate shunt resistors is provided to furnish full-scale ranges of 10,20,100,200 and 1000 ppm by volume of water.

CALIBRATION :-

There is one factor to be considered when a liquid "Freon" sample contains less than about 30 ppm by weight of water. The analyzer does not absorb 100% of the water in the vapor sample when a 20-in. length of the electrode assembly is used. The efficiency of operation for a given length of electrodes is a function of sample flow rate and the water level of the sample being analyzed.

The analyzer is calibrated empirically with one assumption, as the vapor flow rate approaches zero, the contact time of the mixture and phosphoric acid becomes large, and, therefore, the efficiency approaches 100%. This assumption is shown to be correct by the correlation of results obtained(29) with the electrolytic analyzer and the P_2O_5 and Karl Fischer methods. Data are obtained which relate the instrument indicated water and the vapor flow rate in the range from 15-100 cc per minute. Curves are plotted and extrapolated to zero flow rate. The point of intersection of the curve and water axis is the true moisture level of the sample. Once this point is established, the points corresponding to different efficiencies can be obtained. With a family of these curves in the moisture range of interest, a nomograph may be constructed if curves are drawn through the points of equal efficiency. The nomograph relates vapor flow rate, water indicated by the instrument, and efficiency. The actual water is the indicated water divided by the efficiency.

Fig. 24 is the nomograph for the determination of water in 'Freon-12' refrigerant, using a 22-in. length of electrodes. This nomograph applies to all instruments using the same length of electrodes. Similar nomographs must be prepared for each different 'Freon' compound to be analyzed.

ACTUAL WATER CONCENTRATION :-

The method of calculating the actual water concentration in parts per million by weight in a 'Freon' compound is found by using Equations No. 5 and 6. The water indicated by the instrument is:

$$W_1 = \frac{(S)(M)}{(R)(F)} \text{ ppm by wt } H_2O \quad \text{------------} (5)$$

where W_1 = the water indicated by the analyzer, ppm by wt.

S = scale the instrument is operating on (100, 200 etc ppm by volume water)

R = ratio of molecular weights of 'Freon' compound being analyzed and water.

M = microammeter reading, expressed as a percentage of full scale reading, 0-100%.

F = a dimensionless number equal to the vapor flow rate

F = a dimensionless number equal to the vapor flow rate of sample in cc per minute measured at atmospheric pressure.

From the nomograph for a particular 'Freon' compound, the efficiency of operation corresponding to a particular vapor flow rate and microammeter reading is found. Therefore, the actual water content of the sample is :

$$W_2 = W_1(100/E) \text{ ppm by wt. of water} \quad \text{———} (6)$$

where W_1 = the water indicated by the instrument, ppm by wt.

W_2 = the actual water content of the sample, ppm by wt.

E = the efficiency of operation expressed as a %age.

A second nomograph can be prepared from the available data to eliminate the need for all calculations. For a particular instrument (sample flow meter), and operating range (x10, x20 etc) this nomograph relates microammeter readings, flow meter readings, and the actual water in the sample.

OPERATING VARIABLES :-

Voltage :- Voltage has no effect on the analyser so long as it is sufficiently high and is above the thermodynamic decomposition potential of water, 2 v. Below this voltage, no water is electrolysed regardless of its concentration. Some units operate with 90-v dc power supplies. If the moisture levels of interest are above 1000 ppm by weight, lower voltage must be used to prevent overheating the sensitive element. Fluctuating voltages of say 5% or more should be avoided to prevent transient errors, if difficulty of this sort is expected, constant-voltage transformers may be used.

Temperature :- Sample temperature affects the gas density and therefore, the total mass of water entering the sensitive element. The specific resistance of the acid film is a function of sample temperature and the latter should therefore, be kept reasonably constant, within 10° F. The sensitive elements can operate at any temperature from 30-300° F. Changes in the ambient or sample temperature should be avoided. The resulting transient phenomena destroy the equilibrium conditions that exist between the moisture in the sample, the sample tubing, the valves, etc. Analyzers used indoors should be sheltered from drafts, those installed out of doors should be housed in insulated enclosures.

Pressure :- The magnitude of the sample vapor pressure, downstream from the vaporizer, should be low enough to prevent a liquid sample from entering the sensitive element. If a liquid sample enters the sensitive element, the tendency is to wash away the phosphoric acid, this has not been found a consideration for vapor samples. The temperature of the liquid sample and analyzer must be high enough to permit a 10 psig operating vapor pressure, the Moore controller does not function well below this pressure.

RESPONSE TIME - CONTINUOUS SAMPLE :-

The response time of the analyzer depends on whether the analyzer is used on a continuous or a discrete sample. For continuous sample in which equilibrium exists between the moisture in the sample and the tubing through which it is flowing, and in which sudden change upward occurs in the moisture level. The time for the analyzer to reach 63% of its new final reading is about 1 min. If the sudden change in the moisture level had been downward, the response time would be from 8-9 minutes.

In actual practice step changes of this sort are improbable. Consider what is involved: suppose a process is running smoothly and the product moisture level is 2 ppm by weight. Suppose a small condenser leak suddenly permits gross water to enter the system. The product moisture level does not jump to some high level due to the leak. The rise in the moisture level is very rapid, perhaps only seconds, but there is no discontinuity in the moisture level before and after the leak. A finite time is required for the moisture level to rise. This finite time is essentially the time required for the moisture to dissolve in the liquid 'Freon' compound and for the water to 'wet' the pipes, valves, etc., through which the process stream is flowing.

The time required for 'wetting' is a function of the process flow rate, the effective surface area of the process piping, and the temperature. Similarly the time required for 'drying' the process piping when the moisture level drops is a function of the same parameters as for 'wetting', and in this case may require hours. The same situation holds true for the electrolytic analyzer. So long as liquid sample hold up is negligible, and the sample flow rate, effective surface area of the sample tubing, and the sample temperature are comparable to the corresponding process parameters, it is felt that the electrolytic analyzer faithfully follows the moisture level of the process. The response time of the analyser in this case is essentially that of the process itself.

RESPONSE TIME - DECREASE CHANGE :-

In connecting the analyser to the cylinder the response time is merely the time required for equilibrium to be established between the moisture in the sample and in those components with which the sample comes in contact. Techniques have been developed (10) whereby the final answer, within 0.5-1.0 ppm by weight of water, is achieved inside of 20 minutes.

The reason for the longer response time when the moisture level decreases, as opposed to a rise, lies in the diffusional properties of water in the acid film. It takes longer to dry the acid film than it does to 'wet' it. Consequently, the analysers are maintained in a drier state than the material in the discrete samples to be analysed. This is accomplished by passing dry material through the analyser when it is not in use.

APPLICATION :-

The electrolytic analyser can be used to determine the water content of many gases or vapours. The major 'Freon' products and the minimum allowable sample temperatures that permit proper vaporization are as follows :-

Minimum Allowable Sample Temperature.

Refrigerant	Minimum temperature of sample for vaporization.
'Freon-11'	100° F
'Freon-12'	0 F
'Freon-22'	-20 F

Refrigerant.	Minimum Temp.of Samples for vaporization.
'Freon-113'	160 F
'Freon-114'	60 F

It will be noted that there is never apt to be any difficulty in analyzing 'Freon-12' and 'Freon-22' because of the low boiling points of these two materials under normal atmospheric conditions. For the others, precautions in varying degree must be taken regarding the sample and instrument temperature. If the sample temperature must be elevated to achieve vaporization, the instrument must be maintained at a higher temperature to prevent condensation in the sensitive element and other components.

ADVANTAGES :-

It is felt that the electrolytic analyser is precise and accurate for the moisture analysis of 'Freon-12' and that it has several other distinct advantages. In particular, it operates continuously and automatically on either a plant product stream or in the laboratory on discrete samples. It is relatively inexpensive; it is dependable and requires minimum maintenance and gives rapid results. It is believed that these remarks apply to the moisture analysis of other 'Freon' compounds as well.

There is no background of knowledge as yet with the electrolytic analyser for the moisture analysis of high-boiling 'Freon' compounds such as 'Freon-11' and Freon -113' compounds ; but it is known that with these compounds it is difficult to obtain precise analytical results using the P_2O_5 and Karl Fischer methods. The latter two methods for precise results require vapor samples.

in the two curves is due to the 'tramp' water that is present on various sample fittings. This 'tramp' water must be purged from the analyzer and fittings to obtain the correct answer in a minimum of time. A single discrete sample brought to the laboratory for analysis generally contains this 'tramp' water. In the moisture range of interest, below 20 ppm, by weight, the presence of 'tramp' water can contribute from 1-6 ppm by weight to the final answer when using discrete sampling methods and laboratory apparatus. In addition poor sampling technique can raise this figure even higher.

6.0 KARL FISCHER METHOD FOR REFRIGERANT-OIL MIXTURES [illegible]

Each unit does contain some water, but the total amount is unknown. To assist in determining the amount of moisture circulating in the refrigerant-oil solution, an analytical procedure has been developed for its measurement.(32)

The procedure described is an adaptation of the Karl Fischer method. It is a well established method which has been applied to a variety of problems requiring the analysis of materials for moisture. Recently it has been reported that the Karl Fischer method has been applied to the determination of water in refrigeration oils with excellent results.(33) The procedure described here for analyzing solutions of refrigerant-oil in Freon fluorinated hydrocarbon refrigerants is, in essence, a modification of this method.

Although this method does not have the accuracy of the P_2O_5 and infrared methods, particularly at moisture concentrations less than about 15 ppm, it does have a number of practical advantages, especially for the problem to which it has been applied. It is a reasonably straightforward procedure and does not require a highly trained technician. Although assembly of the apparatus requires some glass blowing, the component parts of the apparatus are readily available from commercial sources. The procedure is somewhat faster than the P_2O_5 method, particularly when two sets of equipment are used concurrently. A determination can be completed in about one hour but the analyst's attention is required for an only half this time. Other

In the P_2O_5 method the sample tends to condense preferentially in the first Nesbitt bulb. In the Karl Fischer method, the sample tends to remain in the titration flask which is apt to affect the conductivity of the solution therein.

LOW MOISTURE REFRIGERANTS :-

The use of the electrolytic analyzer has furnished an insight into the problem of analyzing refrigerants of very low moisture content. The single most important factor found that contributes to precise moisture analyses is the attainment of equilibrium between the moisture in the sample and the moisture in or on those materials with which the sample comes in contact. This says that a single laboratory analysis of a discrete sample will rarely give the correct answer; usually it will be high. The reason for this is the presence of excess water on cylinder valves, in sampling tubes, in intermediate sampling containers, and in the atmosphere.

The only way to avoid these errors is to have dynamic equilibrium, the moisture in a flowing sample and in those materials which contact the sample must be in equilibrium. This point is demonstrated in Fig. 25 which graphically shows what has been found when analyzing liquid phase a cylinder of "Freon-12" for moisture.(20) The top curve in Fig. 25 is a plot of the actual measured moisture vs time when the analyzer was connected and the cylinder valve was opened with no purging. The second curve was obtained by first bypassing the sample around the sensitive element for 10 minutes after opening the cylinder valve.

Both curves approach asymptotically the same final reading, but the lower one arrives there first. The reason for the difference attractive features of the method include the wide range of water concentration over which it is applicable and the complete range of refrigerant-oil solutions which can be analysed.

In making a quantitative determination for amounts of water less than, roughly, 50 ppm., the analyst must exercise extreme caution to prevent contamination by extraneous moisture. This is paticularly true to the procedure described here, not only with respect to the analysis itself but also with respect to the equipment and procedure used in obtaining the sample to be analysed.

A second warning, which is extremely important in analysing refrigeration systems for moisture, concerns the uniformity of the sample. The fluid at various points in a refrigeration unit will not be uniform in composition. Therefore, some correlation must be established between the results of an analysis of fluid withdrawn from some known point and the moisture in the entire system. On the other hand, if the procedure is to be used as a control method, where a knowledge of the absolute water content is not required, some level of water concentration may be set as standard and all analyses referred to this standard. The successful application of such a scheme requires that all samples be removed from the same location in the unit after it has been operated for a sufficient length of time to attain equilibrium.

4.2.1 [illegible] :-

A sample of the refrigerant-oil mixture to be analysed is condensed at dry ice-acetone temperature into a solution of chloroform, methyl alcohol, and Karl Fischer reagent. The resulting mixture is allowed to return to room temperature very slowly, during which time the bulk of the refrigerant is evaporated. After reaching room temperature, the solution is titrated immediately with Karl Fischer reagent and standard alcohol solution to a sharp end point using the 'dead-stop' end point technique. The water content is calculated from the weight of sample used, the volume of Karl Fischer reagent, the volume of alcohol, and the solution standardization factors.

4.3.2 APPARATUS :-

The apparatus found most satisfactory for this analysis is diagramed in Fig. 37. The titration is carried out in a 500-ml, 4-neck, round flask, which has been flattened on the bottom. Agitation is accomplished by use of the magnetic stirrer L.

Karl Fischer reagent is supplied from a stock solution to the 10-ml. microburet A which is connected to the flask by narrow bore glass tubing sealed into a standard taper glass fitting at the center neck. For a permanent piece of apparatus it is recommended that the glass tubing lines be continuous throughout. If this is not practical, glass tubing lines may be joined satisfactorily with Tygon tubing. As little of the tubing as possible should be exposed to the Karl Fischer reagent or alcohol. Standard methanol solution is similarly connected to the flask through the 50-ml., buret B. Both burets and stock solution bottles are protected by drying tubes packed with calcium chloride and indicating Drierite. No completely satisfactory lubricating grease for the burets and glass joints is known, but a silicone type is recommended as the best available.

The left-hand neck of the flask contains a standard taper glass fitting through which two platinum electrodes E are sealed at the side. Through the top of the same fitting is sealed a length of glass tubing which extends about halfway into the flask. A small female ball and socket joint is sealed to the top of this line. A male ball and socket joint is suitably connected to the sampling container by a short length of rubber tubing and this in turn is connected to the apparatus at C. The sampling container is a Pyrex glass pressure tube. When the sampling container is not connected to the apparatus, a male ball and socket joint plug is kept in place at C. The platinum electrodes are connected to the binding posts of the titrimeter box D. A diagram of the 'Dead-stop' titrimeter circuit appears in Fig. 26.

To the right-hand neck of the flask is connected the auxiliary equipment for maintaining a dry atmosphere in the flask and for removing the solution at the completion of the analysis. Two lines of glass tubing are sealed to the fitting on the right-hand neck. Line F extends through the top of the fitting down to half an inch from the bottom of the flask. The other end of the line F is connected through a stop-cock to a vacuum outlet with a suitable liquid trap located conveniently in the line. Line G is sealed to the side of the right-hand fitting and is connected through a stopcock to drying tower H packed with a mixture of asbestos and P_2O_5. This in turn is joined to a second drying tower J packed with anhydrous calcium chloride and indicating Drierite. The legs [illegible] [illegible] of the tee leaving tower J are connected to an air line and a mercury bubbler K. The center tube of the bubbler extends only 1 or 2 cm below the surface of the mercury dry air through line G and permitting it to escape at C. The stopcock in limb F is closed. Since moisture contamination by room air in the flask is a serious source of error, the following additional drying step is recommended. About 200 ml of the chloroform-methanol solvent used for the analysis should be introduced through the fourth neck of the flask. (It is advisable to keep a supply of this solvent in a separate stock bottle, suitably arranged to permit transfer with a minimum of exposure). A plug is placed in the opening of the sample line at C, and a gentle flow of air is passed through line G during addition of the solvent. To be certain of a good flow of air into the flask, it is well to pinch off the leg of the tee going to the bubbler K in line G whenever this fourth neck is open. After addition of the solution is complete, and agitation is started. The water in the chloroform-methanol solution is neutralized by addition of Karl Fischer reagent until a sharp end point is reached. The stirring motor is lowered and a bath of dry ice and acetone is placed around the flask. The motor is returned to position and, while being stirred, the solution is allowed to cool for 5 to 10 minutes. The cooling bath is then withdrawn and the flask and contents are permitted to warm to room temperature. Karl Fischer reagent is again added to a sharp end point. If more than 0.05 to 0.10 ml is required, the process should be repeated until only a titre of that amount is required. This procedure is very effective in extracting moisture from the flask.

At all times during these preliminary steps and during the course of the analysis, except when indicated, a constant positive pressure of dry air is maintained in the flask. This is done by keeping a flow of air through the drying towers and line G. The and the side arm dips below the surface of a small beaker of high boiling liquid such as Nujol heavy mineral oil. The fourth neck of the flask, not shown in the drawing, is stoppered with a standard taper glass fitting. This neck is used as a port for introducing the solvent into the flask.

<u>REAGENTS</u> :-

<u>Anhydrous Methanol Reagent</u> :- Analytical reagent grade absolute methyl alcohol was found to be satisfactory without further drying. Standardization of this reagent was accomplished by direct titration with the Karl Fischer reagent.

<u>Chloroform Methanol Solvent</u> :- This solution was prepared with U.S.P. grade chloroform and anhydrous methanol (see above) without further drying. Its composition is [illegible] percent of chloroform by volume.

<u>Karl Fischer Reagent</u> :- The preparation and standardization of this reagent are described adequately by Mitchell, J. and D.M. Smith; Aquametry, Interscience Publishers, Inc. New York, City, 1948. A reagent strength of about one-half that normally used is recommended - i.e., 1 to 2 mg of water equivalent to 1 ml of Karl Fischer reagent.

4.24 <u>PROCEDURE</u> :-

All components of the apparatus should be thoroughly dried by oven baking at 100 C before assembling. Prior to actual use or after the apparatus has been standing unused for any period of time, the air in [illegible] the system should be displaced with dry air. This can be accomplished by introducing a gentle stream of dry air through line G and permitting it to escape at C. The stopcock in limb F is closed. Since moisture contamination by room air in the flask is a serious source of error, the following additional drying step is recommended. About 200 ml of the chloroform-methanol solvent used for the analysis should be introduced through the fourth neck of the flask. (It is advisable to keep a supply of this solvent in a separate stock bottle, suitably arranged to permit transfer with a minimum of exposure). A plug is placed in the opening of the sample line at C, and a gentle flow of air is passed through line G during addition of the solvent. To be certain of a good flow of air into the flask, it is well to pinch off the leg of the tee going to the bubbler K in line G whenever this fourth neck is open. After addition of the solution is complete, and agitation is started. The water in the chloroform-methanol solution is neutralized by addition of Karl Fischer reagent until a sharp end point is reached. The stirring motor is lowered and a bath of dry ice and acetone is placed around the flask. The motor is returned to position and, while being stirred, the solution is allowed to cool for 5 to 10 minutes. The cooling bath is then withdrawn and the flask and contents are permitted to warm to room temperature. Karl Fischer reagent is again added to a sharp end point. If more than 0.05 to 0.10 ml is required, the process should be repeated until only a titre of that amount is required. This procedure is very effective in extracting moisture from the flask.

At all times during these preliminary steps and during the course of the analysis, except when indicated, a constant positive pressure of dry air is maintained in the flask. This is done by keeping a flow of air through the drying towers and line G. The mercury bubbler K serves the purpose of maintaining this positive pressure as well as indicating a positive flow of air. This precaution is necessary to prevent contamination of the system by moist air. As mentioned earlier, no grease is known which maintains leak-free joints for any extended period of time. If a positive pressure of dry air is maintained at all times, any small leaks can be ignored since there is no opportunity for moist air to enter the flask.

For the analysis itself, a sample of 50 to 60 gm of the refrigerant-oil solution is collected in a suitable small cylinder, taking every precaution possible to prevent contamination by moisture. The sample and container are carefully weighed, shaken vigorously, and then connected at C. The contents of the flask, remaining from either the preliminary drying or a previous analysis, are sucked out slowly through line F while a positive pressure of dry air is continuously maintained through line G.

Chloroform-methanol solution is introduced into the flask through the fourth neck and the stopper replaced immediately. The amount of solution required is about 100 ml, but this can be varied according to the quantity of oil in the sample. Sufficient solvent should be used to give a homogeneous solution at the end of the analysis. After the stirring is started, the Karl Fischer reagent is added carefully until an end point within one drop of reagent is reached. The permanent deflection of microammeter for two minutes is considered the end point. This will vary with each set up and the analyst should establish his own end point requirements. The level of Karl Fischer reagent is then read and a known excess of reagent added. Usually 2 ml is sufficient.

The stirring motor is lowered and a dry ice-acetone bath is placed around the flask. The motor is raised, stirring is started, and the flask is allowed to cool a few minutes. The valve on the sampling flask is cut off. This is done in such a way that the pressure in the flask is always kept on the positive side as indicated by no rise of mercury in the center tube of the bubbler. Once the source of air is shut off, the sample is condensed in the flask as rapidly as practical without loss of gas through line G. Again the mercury bubbler can be used as an indicator. After the desired amount of sample is in the flask, the cylinder valve is closed, the cooling bath is removed, and the solution in the flask is slowly brought to room temperature. As this occurs, the Freon will boil off. The rate of evolution of gas is controlled by raising or lowering the cold bath as required, and the rate of gas flow could be followed by immersing the side arm of the mercury bubbler in a small beaker of Nujol heavy mineral oil and observing the rate of bubbling. A rate of 2 to 4 bubbles a second is a good standard. Any sudden evolution of gas should be avoided. Generally the time required to bring the flask to room temperature is about half an hour. As soon as the evolution of gas has essentially stopped, the air is again passed through line G.

At this point there normally is an excess of Karl Fischer reagent and the microammeter shows a positive deflection. Sufficient alcohol from buret D is added to return the reading to zero. The volume used is recorded. Karl Fischer reagent is again added until the same one-drop end point is reached. The analysis now is complete, and the sampling cylinder is removed and reweighed. Before removing the sampling cylinder, it is advisable to check the external surface of the sample line and the ball and socket joints for condensed moisture. If any is present, it should be removed to prevent any droplets from entering the flask when the cylinder is removed.

The amount of moisture present is calculated as follows :-

$$\frac{\left[\text{ml KF reagent} - (\text{ml methanol} \times K)\right] \times S \times 10^3}{\text{Gm of sample}} = \text{ppm water} \qquad (7)$$

where, K = ml of Karl Fischer reagent equivalent to 1 ml of methanol.

S = mg of water equivalent to 1 ml of Karl Fischer reagent.

The time required to carry out a single analysis should average about one hour. This figure may vary somewhat according to the relative amount of fluorinated hydrocarbon refrigerant in the sample, which determines the time required for the evaporation step. Of the total time, however, only half is actual working time.

4.8.3 RESULTS :-

The analytical data obtained(21) on several solutions of Freon-12 and Freon - 22 with refrigeration oil in which the refrigeration oil was analysed by the Karl Fischer method and the refrigerants by P_2O_5 method and the combination of this results compared with Karl Fischer values of refrigerant-oil mixtures indicated the accuracy of the Karl Fischer Method.

6.0 THE CHEMICAL METHOD :-

It is known that the vapor pressure of water dissolved in refrigerants is dependent upon the relative degree of saturation of water in the refrigerant. This vapor pressure is approximately equal to the vapor pressure of water in air.

From equation (1) the water content of the vapor phase over a saturated solution of water in Freon - 12 can be computed. Also for less than saturated solutions Henry's law is followed which states that the partial vapor pressure of the solute (water) is proportional to the mole fraction of the solute (water).

Salts which form hydrates vary in the degree of hydration according to the partial pressure of the water in the air in contact with the hydrated salts. From above, it can be expected that hydrated salts will vary in the degree of hydration according to the partial pressure of water in refrigerant in contact with them. These hydrated salts that change in colour according to the degree of hydration are possible candidates for consideration as means for visually indicating the approximate amount of moisture in refrigerant upto the point of saturation i.e. upto the point where the free water is present.

A number of salts which change colour as the degree of hydration changes are(23) :-

Cobaltous bromide ($CoBr_2$)
Cobaltous chloride ($CoCl_2$)
Cobaltous sulphate ($CoSO_4$)
Cobaltous acetate $Co(C_2H_3O_2)_2$
Cobaltous nitrate $Co(NO_3)_2$
Copper (ic) chloride $CuCl_2$
Copper (ic) sulphate $CuSO_4$

4.3.6 SUMMARY :-

This method can be used for individual moisture determinations of refrigeration systems and also for comparative tests of various methods. Its use is satisfactory at higher moisture concentrations that is those above 15 ppm.

However, the required sample is too large for conducting repetitive tests. Recharging of the unit would be necessary which may not reconstruct the identical conditions within the unit. It is also time consuming and short time changes in a unit cannot be observed.

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It is known that the vapor pressure of water dissolved in refrigerants is dependent upon the relative degree of saturation of water in the refrigerant. This vapor pressure is approximately equal to the vapor pressure of water in air.

From equation (1) the water content of the vapor phase over a saturated solution of water in Freon - 12 can be computed. Also for less than saturated solutions Henry's Law is followed which states that the partial vapor pressure of the solute (water) is proportional to the mole fraction of the solute (water).

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A number of salts which change colour as the degree of hydration changes are(20) :-

Cobaltous bromide	($CoBr_2$)
Cobaltous chloride	($CoCl_2$)
Cobaltous sulphate	($CoSO_4$)
Cobaltous acetate	$Co(C_2H_3O_2)_2$
Cobaltous nitrate	$Co(NO_3)_2$
Copper (ic) chloride	$CuCl_2$
Copper (ic) sulphate	$CuSO_4$

Chromic Chloride $CrCl_3$

Nickel(ous)chloride $NiCl_2$

Nickel(ous) sulphate $NiSO_4$

Obviously, if any salt is to be suitable as an indicator of the relative amount of moisture in refrigerant, a definite change in colour must take place at the moisture concentration considered to be the maximum desirable. There is probably general agreement that the ideal would be for refrigerant to be absolutely anhydrous. Practically, however, some amount of moisture must be tolerated.

The maximum amount of water in refrigerant that can be tolerated without damage to a system is still a matter upon which there is disagreement. Manufacturers of refrigerants specify the maximum moisture content of their products to 10 ppm by weight.

It is felt that visual moisture indicator for Freon -12 should have a transition point in the neighbourhood of 15 ppm moisture content so as to have a safety factor in view of the present limited knowledge of the amount of water that can be safely tolerated.

Cobaltous salts are commonly used on paper to indicate humidity changes and cobaltous bromide(25) has the sharpest colour transition in refrigerant in the range of moisture concentration to be indicated. It goes through the following colour transitions :-

(8). $CoBr_2 + H_2O = CoBr_2 . H_2O$ (Blue)
(green)

(9). $CoBr_2 . H_2O$ (Blue) $+ H_2O = CoBr_2 . 2H_2O$ (purple blue)

(10). $CoBr_2 . 2H_2O + 4H_2O = CoBr_2 . 6H_2O$ (pink)

Cobaltous bromide in contact with cellulosic material apparently forms a complex in which $CoBr_2$ combines with one of the hydroxyl (-OH) groups of cellulose . Accordingly when cellulosic material is the carrier of the $CoBr_2$, a deep blue colour indicates the driest condition. The concentration of $CoBr_2$ on cellulosic material such as ordinary paper or cotton determines within certain limits the point of colour transition from blue to pink or vice versa.

PAPER -BASE INDICATOR :-

The presence of normal quantities of oil in the refrigerant has no harmful effects on the indicator. A paper-base mixture indicator reaches its minimum sensitivity in both Freon-22 and methyl chloride at high moisture concentrations even at low liquid line temperatures and hence a more sensitive indicator is necessary for these refrigerants.

A more sensitive indicator for Freon-22 is $CoBr_2$ deposited on an inert carrier such as glass fiber, asbestos or aluminium silicate fibre paper. The anhydrous $CoBr_2$ is green and the blue colour of the paper base indicator is due to the formation of a complex salt having one degree of hydration. It is obvious that the transition to the anhydrous state represents a condition of equilibrium with a system having a lower partial pressure of water i.e. a lower relative saturation of water in refrigerant.

When this indicator was used in an operating unit,(23) using Freon-22 it was found that the colour transition from green to pink or vice versa took place at moisture concentrations of 2.5 to 3.5 ppm at 80° F. At no time is a blue or purple blue colour found. This indicator showed a colour transition point at 24 ppm water for a system charged with Freon-22 and operating at liquid line temperature of 80 F.

INERT - BASE INDICATOR :-

When inert-base indicating element is green, the moisture content of the system is 43 ppm or below in Freon - 22 and 10 ppm or below in Freon - 12. If the colour of the element is pink, the moisture content of the system is 28 ppm or above in Freon-22 and 8 ppm or above in Freon-12 depending upon the refrigerant temperature.

LIMITATIONS :-

a) The moisture sensitive elements are not instantaneous in their indication of the relative saturation of a refrigeration system. The indicating elements are usually pink at the time of installation. If installed in an operating system containing Freon-12 having less moisture concentration than 10 ppm, the paper base indicator will turn light blue in 15 minutes and light green for Freon-22 in an hour if the operating system have moisture concentration less than 25 ppm.

b) All substances considered foreign to a refrigeration system will have some undesirable effects on the moisture indicator. Liquid anti-freezers are most subtle in their action since the presence of these substances in an operating system containing moisture will generally cause the indicator to signify that the system is dry. Liquid-anti-freezers serve only to increase the solubility of water in refrigerants which, for a given moisture content, reduces the relative saturation. Since the colour of the moisture indicator is dependent upon the relative saturation of water in refrigerant, any increase in solubility results in an indicated, but false low moisture content.

c) Free water in an operating system will leach out the $CoBr_2$. If the water is dispersed in the form of small droplets, the indicators show white or pink.

d) Leak detectors will discolour the indicating elements since most of them are highly coloured dyes.

e) The indicator should not be placed in a system after a motor burn out has occurred until the system has been thoroughly cleaned by the use of driers. The acid formed turned the paper-base indicator an irreversible bluish green. Sludges, too, discolour the elements and mask any colour changes which may occur.

f) Either excessive quantities of moisture or motor burn-outs results in the formation of $CuCl_2$ and iron salts, which collect and discolour the indicating elements.

g) Both ammonia and sulfur dioxide form salmon-coloured complexes with $CoBr_2$. The $CoBr_2$ - ammonia complex is very stable and appears irreversible but the sulfur dioxide complex is unstable and when the indicator is exposed to air the pink colour soon returns.

ADVANTAGES :-

1. A moisture indicator can be installed on the original equipment by the manufacturer, the contractor, or the serviceman,

2. Continuous visual indication is obtained as to whether a refrigerating system is operating within safe moisture limits without the necessity for sampling followed by laboratory determinations or the use of specialized apparatus.

CHAPTER - V.

MOISTURE DEHYDRATION METHODS.

MOISTURE DEHYDRATION METHODS :

Removing moisture from a new refrigeration system is a very important step. No reliable contractor or manufacturer fails to do it. Special facilities and good instrumentation are employed to dehydrate refrigerant systems and equipment thoroughly in the factory where the process can be carefully controlled by the manufacturer. These facilities are impractical for use on field installations, because they usually consist of ovens which heat refrigerant containing parts to a carefully controlled high temperature for a long period.

However, the field engineer must accomplish some low moisture level with portable equipment and in most cases without a means of determining how well he has accomplished the job. The accepted methods of dehydration are described here. The field engineer should thoroughly acquaint himself with each of these methods and be able to select the method most suitable for use on any particular occasion - it may be even advisable to use more than one method on some occasions.

5.1 EVACUATION METHOD :-

Referring to saturation curve for water in Fig. 28, it is found that the system can be dehydrated by using a vacuum pump capable of reducing the absolute pressure within the system to such a value that at the existing room temperature, any water would be boiled off and the water vapor would be removed along with air by the vacuum pump. E.g. pull a vacuum of 0.5" mercury absolute pressure in the system with an ambient temperature above 60° F. When desired pressure is reached all free moisture will have "boiled" into water vapor and the moisture will have been pumped out by the vacuum pump so system will be dry.(24) (Fig.29)

Only equipment needed for dehydrating is a good vacuum pump and a vacuum indicator. If no indicator then one can make easily on the job, from a large test tube, an accurate thermometer, some wicking, corks, copper tubing and paraffin (See Fig. 31). Pack the test tube in a container with insulation as shown. A window is cut in the insulation so that the thermometer can be read. Take care that indicator is perfectly air tight and handle it very gently. Use paraffin to seal around tubes so it is air tight.

5.1.1 NEW SYSTEM :-

To dehydrate a new system, connect a tee to the charging connection as shown in Fig. 30. Connect vacuum pump to one branch of tee with copper tubing. Attach a vacuum indicator to the other branch, with shut off valves in each line. Open valves in the system except compressor service valve, vacuum indicator valve, and valve to the high pressure gage.

Compressor is usually dehydrated at factory(25,26) and vacuum will injure high pressure gage if it is not a compound gage. Open the indicator valve only to take readings. If valve is left open, water in that test tube will boil off. Then trouble will be to replace the water often and pump will have more water vapor to handle. After opening valve allow 5 minutes for conditions to steady before taking the reading.

Start the vacuum pump. If two stage, operate pump parallel at first. As pressure in system is reduced, boiling poi of water within it lowers until finally all moisture is removed, When 55° F (0.43" abs.) is reached, change pump to series operation (if that type); pump until 35° F (0.2" abs.) shows on vacuum indicator. Then close pump valve and stop pump. Hold vacuum for about 5 hours, but check hourly by opening valve to vacuum indicator. If vacuum holds, disconnect pump, connect Freon cylinder and break vacuum with Freon. Thus has got a good moisture free start in life.

Keep system in this dehydrated condition by taking extreme care to prevent air from entering e.g. when opening a system for repairs, be sure to purge all open parts with Freon gas, to expel the air before closing the system. Keep on toes by repairing leaks promptly as soon as found. That is very important in systems operating below atmospheric pressure on the low side.

If water pressure is found higher than the normal refrigerant pressure, then blow off the contaminated freon charge, and repair leak and use a vacuum pump as in a new system. But this is expensive. Not only is refrigerant charge is lost, needing costly replacement in large systems, but labour also runs high.

Amount of vapor handled by vacuum pump is small, so cut system at all low points and drain water and oil from these sections. Then repair these cuts. It is also important to remove all possible oil from the system. That includes the compressor case, as boiling refrigerant out of oil with vacuum pump is a long process.

Less costly repair job, and almost as effective, (if properly carried out), can be done with a "system washer"(24).

6.1.2 DEFECTS :- (27)

a) In any refrigeration system, there are a number of crevices at piping joints, including evaporator and turns etc., which give up their included moisture most reluctantly. The rate of heat transfer from the ambient to the various parts of the system, is low. The rate of evaporating liquid water is a direct function of area of the exposed surface of the water which may be very small, thus explaining slow evaporation rate. Surface tension is also an important factor and the presence of oil tends to retard the evaporation of the water. Hence, successful dehydration can be accomplished by this method only if indefinitely long time is available to complete the process.

b) One complication arises in that, if vacuum is reduced below 4 m.m. mercury, the water will tend to freeze at the existing temperatures. This is of importance only in that it slows down the action from then on, as water will sublimate directly to vapor over a period of time as is commonly recognised from the experience of having clothes dried in freezing weather.

c) Accurate measurement of vacuum is difficult because of large volume of gas being handled, the pressure drop may be appreciable and vacuum in one part of the system may be much different from the vacuum in another. To check the possibility of pressure drop, it is recommended that a mercury manometer may be connected into the system at the point most remote from the pump so that its readings can be checked with that of a similar manometer located at the pump. It is a glass U-tube, closed on one end. All air has been removed from the close end. It does not begin to register until a vacuum of approximately 20" in. of mercury is obtained and then it reads absolute pressure in inches of mercury as the difference of level of two mercury columns. If the two columns were at the same level, a perfect vacuum would be indicated.

An indirect method of pressure measurement using a vacuum indicator consisting a thermometer has been suggested. Low pressures determined by this method will probably agree closely with those determined by direct measurement method, but former is felt to be preferable because it is direct rather than indirect and, also, because the temperature method involves introducing a certain amount of moisture into the system tending to prolong if not prevent dehydration.

6.2 HEAT AND VACUUM METHOD :-

As seen , evacuation method of dehydration requires too much time and still it may not dehydrate the system completely and hence it prohibits its use in a factory. Efforts have been made to overcome this by applying heat to the piping and other accessible parts of the system. This may help the same if the heat happens to fall directly on or near a puddle of water inside the system, but it is likely that vapor from such a pool of water may recondense into another cooler part of the system instead being withdrawn entirely. Hence heat is usually applied by placing the completely assembled refrigeration unit in a temperature control oven in which there is vacuum.(22,20)

As known water like refrigerant, has a definite pressure temperature relation or saturation curve. Application of vacuum lowers the boiling point. Thus by simultaneous application of heat and vacuum, the water in the refrigeration can be made to boil off quickly.

A simple method of heating the equipment on the job is by infra red lamps. They should be directed at every part of the equipment including the evaporator, with possible exception of expansion valve, tubing, and compressor, while a deep vacuum is drawn on the system. Some types of expansion valves and controls should be removed from the system during this operation. The heat and vacuum should be applied for a minimum of 24 hours. It is imperative that sufficient heat and vacuum be applied if satisfactory dehydration is to be accomplished. The following figures show the boiling points of water at different vacuums. 26 in. Hg.-125 F ; 28 in.Hg-101 F; 28.5 in. Hg-84 F; and 29 in.Hg-76 F; 29.5 in.Hg-54 F; and 29.72 in.Hg-35 F. Even with every part of the system at 125 F, a vacuum of 26 in., Hg is necessary to vaporize any water present. It is necessary, therefore, in view of these data, to be extremely efficient in the operation. If a converted refrigerant compressor is to be used as a vacuum pump, be sure it will pull 26 or 27 in. Hg vacuum and, to be safe, figure on raising the equipment temperature to more than 125 F, but not more than 150 F. A clip-on thermometer, located out of the direct infrared field, should give a satisfactory indication of the equipment temperature. Even this simple Heat and Vacuum method requires care if the dehydration is to be accomplished successfully.

6.2.1 DEMERITS :-

If heat is to be applied by putting the whole system into an oven then this method is impracticable in the field.

Even heating by infra red lamps, it is difficult and costly to carry them in the field. Therefore, this method is good for factory use.

6.3 HOT AIR METHOD :-

Fig. 38 illustrates this method of field dehydration(20). Air is drawn through the equipment to be dehydrated - in this case an evaporator - by means of a vacuum pump. Room air enters a heated tank through a large cartridge type absorption drier which removes most of the moisture from the air. The relatively dry air is then heated to a temperature not to exceed 200° F. and then passed through the evaporator, heating it, and causing the internal water to vaporise. The air and water vapor then form a mixture and are passed out of the system by a vacuum pump. This operation can also be accomplished by compressed air released through the cartridge drier, tank, and evaporator. However, in such a case, the compressed air should be precooled to room temperature to insure the absorption type drier being effective in initially drying the air. Since the absorption type drier picks up only water, it can be reactivated by placing it in a 275° F oven. It should be pointed out that reactivation of such a drier taken from a refrigerating system is seldom advisable since the oil film collected by the absorption agent will bake on during a heating process.

A lb. of dry air will pick up, or become saturated with, a much greater weight of water vapor at high temperature than at low temperature. Thus, to make this method of dehydration effective, the air must be heated to a temperature near 200° F.

The use of a thermostat on the heating tank can limit the maximum temperature. The inner surface of the tubes in the evaporator of Fig. 38 will eventually assume the air temperature, thus causing vaporization of any moisture present. However, remember that the air in this case is the heat carrier and will lose some, or all, of its heat to the water and metal during the early stages of the operation. A clip-on thermometer placed on the evaporator outlet tube will indicate when the temperature of the very last section is hot enough to have been effectively dehydrated. The use of a psychrometer (wet and dry bulb thermometers) at the evaporator outlet when using the pressure system gives an accurate indication of the dew point to which the evaporator has been dehydrated.

When the refrigeration equipment uses conventional external motor drive, drying of components such as vaporizers and condensers is done by passing hot air before assembling. This frequency shortens the drying time required for the final assembled unit. Since such units donot usually contain organic materials, temperatures higher than 275° F may be used.

In the hermetic units, using an internal motor drive, the presence of organic materials such as cotton windings, cellulose, slot insulation and nylon or Formex wire coatings limits the maximum temperature to about 275° F.

6.2.3 A WARNING :-

This method is not absolutely effective when the equipment has parallel paths, as in the case of a waffle type plate evaporator, due to bypassing of the hot air. Therefore, use this method only where suitable.

6.4 CO_2 AND ALCOHOL METHOD :-

This method of field dehydration is advisable where large quantities of water must be removed from permanently located equipment such as evaporators and refrigerant lines. A good example is a system filled with water due to a ruptured condenser tube. As much of the equipment as possible should be removed, cleaned, and dehydrated by heat and vacuum. The balance, or immovable part of the equipment can then be initially dried by the CO_2 and alcohol method.(20) This should be supplemented by one of the other methods after the system is reassembled. Fig. 24 illustrates the necessary equipment for utilizing this method of dehydration. A small service drum is equipped with a valve and funnel, a tee connection for attaching CO_2 drum, a pressure gage, and a bottom outlet. The procedure is simple. After connecting as shown in Fig. 24, charge the drum with approximately one quart of methyl alcohol, close valve C, open valve B on CO_2 drum, and blow the alcohol through the equipment as shown. Open valve B on CO_2 drum carefully and do not allow the drum pressure to exceed 200 psi. Atomized alcohol will form an explosive mixture, so use the vent tube as shown in Fig. 24. After sufficient alcohol has been blown through the equipment, continue blowing CO_2 to dry up the alcohol. Warning: Alcohol and some drying agents do not mix. Be sure no alcohol remains in the equipment.

As an extra precaution the CO_2 should be safe-dried. Commercial CO_2 may be contain a small amount of water which will gradually settle out during storage. If the CO_2 drum is left in an inverted position for three days, this water will settle out and can be eliminated by unscrewing the safety cap approximately ½ turn and then opening the shut off valve for a short time or until the water is blown out.

6.5 VACUUM DEHYDRATION METHOD :-

This method of field dehydration(3,7), while requiring precision equipment and a much longer drying time, is the most effective since it results in an almost bone dry system with positively no guessing. Fig. 33 illustrates the necessary equipment namely a precision vacuum pump and a moisture indicator, hooked up to the completely assembled refrigerating system. The moisture indicator is tied into the vacuum pump suction line which is connected to the compressor suction valve. This particular hook up is advisable because manipulation of the compressor discharge valve results in a complete "sweep" of the system.

The time needed for complete dehydration depends on the vacuum pump capacity and the amount of free water that must be removed and since the time will vary greatly in different systems, it is necessary to actually record the conditions existing within the system at frequent intervals. This is where the moisture indicator (Fig. 32) is of value. When it is connected in the vacuum pump suction lines as shown in Fig. 33, the test tube will carry the same vacuum as the rest of the system. Thus, the distilled water will have the same saturation temperature as any free water in the system and this temperature can be read on the thermometer within the test tube.

Fig. 35 shows a typical saturation curve to be expected during dehydration by this method. The indicated boiling point of approximately 53° F existing for several hours in a 70° F room shows that free water is present in the system and is being removed at a rate equivalent to (70-53) or 17 deg. F temperature difference. After the free water is removed, the pressure is further reduced and eventually a temperature of 35° F is shown by the moisture indicator. Considerable moisture is still present in the system at this time and it is doubtful that continued evacuation would removed any appreciable additional amount. However, if this remaining moisture is diluted with room air, the pump in re-evacuating the system to the same 35 deg. F saturated condition will remove the major part and end up with a moisture content of approximately 1 ppm.

The mechanics of this method of dehydration can best be explained as follows. The moisture condition present at the end of dehydration, during which any free water has been removed from the system, is as follows.

If we consider that all air has been removed and only water vapor is present at this time, we have established a condition where the greatest possible weight of water vapor is present. We can then establish by reference to the thermodynamic properties of steam the actual maximum weight of water vapor still present in the system. Since the moisture indicator shows a temperature of 35° F, reference to the steam tables shows the saturation pressure of the water vapor to be 0.2035 in. Hg absolute. At the same time the tables show that the specific volume of steam vapor at this low pressure is 2947 cu.ft. per lb. Now 7000 grains of water equal one pound of water. By dividing 7000 grains by 2947 cu.ft. we get 2.37

grains of water vapor present in each cu.ft. of the system. Remember that one grain of water has a diameter of approximately 3/16 in. Therefore, the amount of water present at the end of the initial dehydration is still excessive for satisfactory operation and, even though the vacuum pump continues to operate, it is working at such a low vacuum that its capacity is practically nil. However, if the 2.37 grains of water vapor still present are mixed with, let us say, 100 parts of dry air at a higher pressure, the pump in re-evacuating the system will remove a major portion of the 2.37 grains because of the dilution. It is impossible to sweep the system with dry air as it will be apparent that when the system is swept out with air, an additional quantity of water vapor is added to the 2.37 grains already present.

The procedure and explanation then is as follows: After the moisture indicator has reached 35 deg. F during the first evacuation, close the compressor discharge valve and break the discharge line connection with the vacuum pump still running. The moisture indicator temperature will rise rapidly and again approach the initial starting temperature, in this case 70 deg. F. Average air at 70 deg. F contains approximately 5.0 grains of water vapor per cu.ft. The addition of this water vapor to that left in the system after the initial dehydration brings the total amount now present to 5.0 plus 2.37 or 7.37 grains for each cu.ft. of the system.

The next procedure is to tighten the discharge line, open the discharge valve, and allow the vacuum pump to re-evacuate the system until the moisture indicator again reads 35 deg. F. At the end of the second evacuation the pressure has been reduced from

29.5 in. Hg absolute to 0.2035 in., this being a minimum compression ratio of 147 to 1. Since the 7.87 grains of water are uniformly mixed with the air present, it is drawn off uniformly and the moisture content is reduced in proportion to the compression ratio. Therefore, the weight of water vapor remaining equals 7.87 grains divided by 147 or 0.054 grain per cu.ft. of the system, this being equivalent to approximately 1 ppm.

Use of air for sweeping the system introduces considerable extra moisture into the system, but, even so, the the second evacuation reduces the system to an almost bone dry condition. Also the second evacuation requires less time. The use of Freon-12 as a sweep rather than air will be much more effective, but also rather costly since the Freon-12 would be lost during the second evacuation.

5.5.1 TEMPERATURE EFFECT ON DEHYDRATION RATE :-

In Fig. 36 the curve (30) shows that the time of dehydration of a 50 deg. F. system is reduced appreciably by warming it to 80 deg. F. Temperatures of 100 deg. F., and above did not decrease the dehydration time much more than that for 80°F. The dehydration time may be reduced ½ hour by leaving the wet bulb moisture indicator out of the circuit during the dehydration as shown by the lower curve.

5.6 CHEMICAL DEHYDRATION :-

As seen in the evacuation method that one may not be sure that the system is dehydrated. Therefore, use of a chemical dryer after the system is evacuated to an indicated pressure below 6 mm. mercury charged with refrigerant is recommended.

Fig. 37 is a cross section of a new type chemical dehydrator(37). This type is made in suitable sizes and has the advantage of being refillable in the field. The 25 ton will remove a pint of water. The dehydrators are installed in the liquid line for economy and are left in operation for a minimum of 48 hours. The capacity of a dehydrator is based upon the expected quantity of moisture, refrigerant pressure drop data, and amount of refrigerant charge. It is essential that a dehydrator of sufficient capacity be used and an over-sized one could be used except that all of the refrigerant would tend to collect in the dehydrator if too large. These dehydrators are charged with CaO and charcoal. CaO is an excellent drier and acid neutraliser, and a reasonable amount of charcoal not only removes alcohol, tars, and similar substances, but also serves as a spacer for CaO. Silica gel, Activated Alumina, and $CaSO_4$ will remove more moisture but will not removed acids to a satisfactory degree. The strainer which is used with the felt filter, prevents the chemicals of the drier from going to refrigeration system.

8.C.2 FOR LOW TEMPERATURE JOBS :-

The usual practice is to dry the refrigerant for the low temperature jobs i.e. operating below - 20 deg. F by means of dehydrator in the liquid line. Sometimes the refrigerant is charged into the system through a dehydrator. Low moisture content can be obtained in this manner, but the process is rather slow and large dehydrators are necessary.

Drierite has been recommended for low temperature jobs, perhaps, because of its chemical action, which does not depend on vapor pressure difference. Its action is very slow so that it may take some time to get the job operating smoothly.

A rather unique application of Dehydrators(9) has been used in some instances. One was a Cascade installation operating at -100° F., a valve in the low temperature liquid line was clogging with ice every few minutes. A Dehydrator was installed in this line as shown in Fig. 36, located in an insulated room at normal temperature. The shut off valves were left open so the liquid flow was divided, part through the dehydrator and part through the by-pass. The ice crystals being lighter than refrigerant floated and were carried into the dehydrator. Here they collected and eventually blocked the flow, but the refrigerant continued through the by-pass and kept the plant in operation. Meanwhile the dehydrator warmed up, melting the ice, and flow was then gradually resumed. The refrigerant with the water in high concentration flowed into the dehydrator, where the water was readily absorbed. This appears to represent very efficient use of the dehydrator, as it combines the advantage of low temperature and high moisture concentration.

Removing moisture from the system in which new refrigerants are used, has taxed the ingenuity of refrigerating engineers.

CHAPTER - VI.

DRIERS.

DRIERS.

A drier is a manufactured device for use in a refrigeration system whose primary purpose is to collect and hold at its location that water in the system which is in excess of the amount which can be tolerated free in the system. The moisture may cause corrosion due to acid formation and/or freeze up at low temperature points in the system. The drier is equipped with screens and filter pads to prevent solid desiccant from entering the refrigerating system. The drier unit, in addition to holding the desiccant functions as a filter and removes any solid found in the refrigerant reaching it.

It must be recognized that no desiccant can reduce the moisture content to zero even under ideal conditions, while under practical refrigeration conditions the efficiency of the desiccant is apt to be considered less than the ideal. However, drier dries down to a point that any remaining moisture is absolutely harmless.

The question of evaporator temperature vs dryness requirement is often asked. The solubility of water in refrigerant is fundamental in this regard. The very low solubility of water in commonly used refrigerants emphasises the absolute necessity of drying to the lowest possible level for best operation.e.g. An ordinary commercial unit using Freon - 12 will operate with 20 to approximately 40 ppm water. Considering solubility data for water in Freon-12, it is evident that these systems must operate in a super saturated condition or circulate ice or hydrate crystals without difficulty. Another possibility is that the water present freeze out at some point where it causes no serious difficulty. Freezing depends on the physical factors such as type of expansion valve, or restrictor used and the relative expansion permitted at each point. A long large diameter restrictor is less likely to freeze at a given temperature and water content than a smaller size restrictor or valve.

C.11 ANALYSIS :-

Driers have been installed in almost every conceivable position and in most cases, they have worked fairly well. It is recommended that the drier construction be analyzed for the use intended to determine the best position of the drier to give ultimate contact between the desiccant and the refrigerant. Where the desiccant in granule form is used attention should be given to positioning the drier so that the flow of refrigerant will not contribute to the attrition of the desiccant.

Some people want driers to remove water and acids, and similar non-polar substances and noncondensible gases, with equal efficiency. However, sole economical justifiable reason for adding drier is the removal of moisture from the refrigeration system to prevent freeze-ups. If damaging acid concentrations do develop when halogenated hydro-carbon refrigerant is used, then change the refrigerant.

C.1.2 MATERIAL :-

Drier shells are generally fabricated from either copper, copper alloys or steel. The form of these shells vary with the ingenuity of the design engineers. Shell constructions available are of the throw-away or non-serviceable type and the refillable or replaceable cartridge type. The features claimed for the various shell constructions must be evaluated by the application engineer.

To prevent tumbling and attrition of the granules when used in this form, the desiccant bed can be compacted by spring pressure. Driers are sometimes equipped with a disperser tube at the inlet connections to provide more even distribution of the refrigerant through out the desiccant bed.

G.1.3 BED GEOMETRY :-

Size and shape of the desiccant bed is referred to as bed geometry. The shape of the desiccant bed should be determined by economics of shell construction and pressure drop considerations. A small cross section deep bed removes water much faster during the first few passes of the refrigerant as compared to a large cross-sectional area shallow bed. In order to do a good drying job, the desiccant must come in intimate contact with the refrigerant and must be in contact with it for a reasonable length of time.

The period of time the refrigerant is in contact with the desiccant depends on the length of the path through the desiccant and the velocity of the refrigerant. Obviously bed geometry has no effect when equilibrium has been established between the moisture in the refrigerant and the moist in the desiccant. At equilibrium conditions the water is distributed through-out the bed in a uniform manner.

G.1.4 RATE OF REMOVAL :-

A very important factor regarding use of a drier is the question of rate of moisture removal and the time a drier unit must be left in the machine. A critical glance at the problem shows that the time is longer than is often assumed. To take a specific example, suppose that three drops of water present in a system containing three pounds of refrigerant is about 0.03% moisture and to dry the system to 0.01% moisture level. Now if 1/2 pound of refrigerant circulates/hour, then in the first hour not more than 1/6th of the moisture would be removed, even if the desiccant remaining all moisture entering it. Further more, since the returning dry refrigerant would dilute the wet refrigerant, the feed liquid would become progressively drier and less and less water could be removed by the desiccant. Hence, under even these ideal conditions, considerably more than six hours would be required. In addition moisture will tend to collect in the evaporator of a flooded system because even in relatively small concentrations it vaporizes much less readily, than the refrigerant.

Also in either a flooded or dry type system, it is highly probable that at least part of the water will be dissolved or entrained in the oil entering the compressor and will take a long time to be carried over into the refrigerant cycle where it can be removed.

Fig. 30 shows the rates of drying of Freon-12 using silica gel, sovabead and activated alumina(31). Drying rate is extremely rapid during the first hour, most of the water in the system being removed in this period. An initial advantage is noted for granular gel, this being due to its greater exterior surface area because of its smaller size, (14-20 mesh) which permits more efficient contact with the refrigerant. Activated alumina dried the refrigerant less rapidly and under the conditions of operation, it was not possible to produce the same extent of dryness with this desiccant. The maximum drying effect for the desiccant is apparent after ten hours residence time in the system. On the basis of these results, it is felt that the rate of drying does not play too important role in a refrigerating system in view of the short time involved for drying to a reasonably low moisture content for all desiccants tested. This indicates a little difference in rates of drying and the differences obtained are more probably reflection of the particle size of the desiccants than of any other property.

6.1.5 IDEAL DRIER :-

For example, from the manufacturer's point of view, an ideal drier will be filled with a desiccant which is poured into the shell as a liquid, after which it will solidify to a solid foam. This foam will be mechanically tough and strong and will be bonded tightly to the walls of the shell so that it will act as a filter for all solid dirt. The cells of foam will be small and uniform in size. The cell walls will be thin and freely permeable to all fluids. If the above requirements are met, the pressure drop through this ideal drier will be very low and it will purify the refrigerant as it passes through.

In this ideal drier, the desiccant in its foam form will not suffer thermal damage, unless temperatures are well above those used in furnace brazing. It will be readily reactivated by heat not over 300 deg. F. and at temperatures below 200 deg.F. it will quantitatively remove all substances which might endanger the useful life of and performance of the refrigerating unit.

C.1.6 SELECTION :-

The major problem confronting in the selection of driers is the determination of the proper size drier for a given application. Driers should be selected by comparison with drier capacities under specified conditions to known and estimated total water content in the entire refrigeration system. There are many variables involved and unless these factors are evaluated, the drier cannot be properly selected.

Selection of the size of a drier is actually a split application - on original and on field equipment. Manufacturers can properly apply a drier as they have a knowledge of their dehydration procedures and can estimate accurately residual moisture content of equipment, where as, the service man installs the drier as a precautionary measure or applies to correct system failures.

The manufacturer must put a "size" on a given drier unit. Unfortunately, the amount of water in a system cannot be correlated to the horse power or tonnage of a refrigeration system. Some years ago, a commercial standard was evolved stating a drier containing 12 cu.in. of desiccant should be rated as a 1 hp. unit. As hp was increased, the cubical content per hp. was proportionately reduced. However, 12 cu.in. of desiccant has no significance since the desiccants themselves vary as to density and water absorbing ability, hence drier capacity cannot be related to cubical content and horse power.

The data given in Fig. [illegible] can be used to size a drier employing the desiccants studied. For the purpose of illustration the problem is worked out using silica gel as an example.(38) The first step is to determine the total amount of water that the drier will be called upon to pick up. This is done in the following manner :-

1. 1½ lb. of Freon-12 at 20 ppm water content.

$$1.5 \times 453.4 \times 0.000020 \times \underline{1000} = 13.6 \text{ m.g.}$$

2. ½ lb. of oil at 20 ppm water content.

$$0.5 \times 453.4 \times 0.000020 \times 1000 = 4.5 \text{ m.g.}$$

3. Water left in the unit after factory drying = 100 mg.

Total :- 118.1 m.g.

∴ % water possible in Freon-12 is ;

$$\frac{0.1181 \times 100}{2 \times 453.4} = 0.013 \text{ or } 130 \text{ ppm.}$$

If 10 ppm water is the desired level on which to base calculations, then in the 2 lbs. of liquid there will remain,

$$2 \times 453.4 \times 0.000010 \times 1000 = 9.1 \text{ m.g. water.}$$

∴ 118.1 − 9.1 = 109 m.g. to remove by desiccation to realize 10 ppm level.

At a 10 ppm equilibrium level silica gel will hold 2.5 gms of water / 100 grams of desiccant at 77 deg. F. Calculations are based on 77 deg. F. because comparable data are available on all desiccants at this temperature. Actual operating conditions may attain 120 deg. F. depending upon the location of the drier. Therefore, 0.109 x 100/2.5 = 4.3 gms. of silica gel is required.

Generally 100% safety factor is applied to the actual calculated value of desiccant requirement and a package containing approximately 9 gms. would be recommended for this specific case. Where the size of drier is not important a 200 or even 300% safety factor may be recommended.

A similar calculation for sovabeads at the 10 ppm equilibrium level gives a requirement of :-

$\frac{0.109 \times 100}{1.3}$ = 8.4 gm required or 16.8 gms. with 100% safety factor.

Relative requirements of the several desiccants can be compared at 20 ppm equilibrium water content in Freon-12. At the 20 ppm water level, the several desiccants will hold the following weights of water / 100 gm of desiccants.

Silica gel = 5.0 gm.
Activated alumina = 3.8 gms.
Sova beads = 2.7 gms
Sporlan 'catch all' 1.00 gms.

Using these figures the minimum amount of each of the desiccants required to remove the 100 mg. of water to a 20 ppm level will be

Silica gel = 1.0 gm
Activitated alumina = 2.6 gms.
Sovabeads = 3.7 gm
Spolan 'catch all' = 10 gms.

It is of interest to know desiccant requirements to dry water saturated Freon -12 to a desired level. In Fig. 41

plotted fundamental desiccant requirement data for Freon only using 100 gm. of Freon-12 saturated at 100 deg.F. as a datum point. Freon-12 under these conditions contain 167 ppm water. The number of grams of desiccant required to dry the Freon-12 to the indicated percent of dryness is shown as the ordinate with the equilibrium water content of Freon-12 as abscissa. These are the same data as shown on the previous Fig. 40 but presented in a manner to emphasise the importance of thoroughly understanding the absolute level to which a given system is to be dried in order to size a drier adequately to suit the situation. Remembering that at relatively high equilibrium water concentrations in the Freon-12 these data indicate smaller differences between the several desiccants, but if dryness below approximately 50 ppm is desired the desiccant requirement is much more difficult to meet. These data are most extensive for the two siliceous desiccants namely silica gel and sovabeads. The similarity of these materials is clearly shown by the shape of the curves. At approximately 35 ppm level the relative weight ratio of desiccant required is 0.28 to 0.15 or 1.86. Comparing drierite with silica gel at 35 ppm level, the weight ratio is 1.89 to 0.15 or 12.6.

6.1.7 CLASSIFICATION OF DRIERS :-

Driers can be placed in three categories, the classification of which depends upon the location of the drier in the system(23).

a) High side liquid line drier.

b) Low side drier.

c) Suction line drier.

a) High side liquid line driers:- is usually a cartridge with suitable connections provided internally for retaining the desiccant and is located in the liquid line of the refrigeration system. It is placed after the liquid receiver and, by a series of valves, the refrigerant could be made to go through the desiccant or to by-pass it completely.

b) A low side drier is a shell with suitable connections and in which the desiccant is usually retained by a screen. It is normally located between the expansion device and the evaporator surface. Full refrigerant does not pass through the drier and moisture pick up is accomplished by transfer of the moisture to the desiccant, through the medium of the refrigerant. A variation of this type of drier is to place desiccant in a sack or some other suitable porous container or use the desiccant in a molded form and suspend it in the low side of the system. This type of installation is usually confined to domestic refrigerators and small fractional horse power appliances.

c) The suction line drier is similar in construction to the high side liquid line drier. In many instances identical driers are used with the exception of the size of the connection fitting. Where a drier is designed particularly for suction line applications, special consideration is given to pressure drop through the drier unit as suction line driers are normally installed so that full refrigerant passed through the desiccant.

6.1.3 MERITS & DEMERITS :-

The following is the list of merits and demerits of driers located as above. A drier's moisture absorbing capacity at a given equilibrium point dryness and temperature is a function of the quantity of a given desiccant contained within the drier shell. Therefore, drier cost is dependent upon the cost of the desiccant and the size of the shell to house it. Many of the demerits of any location can be offset by greater quantity of the desiccant.

HIGH SIDE DRIERS :-

MERITS :-

1. Moisture is removed from the refrigerant ahead of the expansion (metering) device which greatly reduces the possibility of freeze up.

2. Makes possible a time of contact between refrigerant and desiccant without concern as to excessive pressure drop since the velocity of the refrigerant in the liquid line is comparatively low.

3. This point of installation provides minimum pressure drop in the refrigerant line if the drier is properly selected with respect to capacity.

4. Little possibility of trapping oil in the drier.

5. Drier can be installed downstream of the heat exchanger or installed inside of the refrigerated space where an appreciable temperature difference between ambient exists, therefore, greater capacity at a given End Point Dryness can be obtained from adsorbent type desiccants since the desiccant is at a lower temperature.

6. If filtering pads or other filtering media are incorporated into the drier, the unit provides protection to the expansion device from dirt, solder particles, scale etc.

7. It is convenient to install a drier in this location.

2. In this location there is less possibility for moisture to enter the evaporator.

DEMERITS :-

1. Possible release of moisture from adsorbent type desiccants at high liquid line temperatures.

2. When the liquid feed is at the bottom of the drier, pulsations of refrigerant may lift and drop the desiccant in a loosely packed unit resulting in the formation of excessive dust which may cause plugging of the screens or filter pads.

3. Sulphur dioxide cannot be adequately dried in the liquid phase.

4. Greater capacity at a given end point dryness can be obtained from adsorbent type desiccants at lower refrigerant temperatures.

Note :- This is a good example of drier economics. The capacity loss at higher liquid line temperatures can be offset by using a greater quantity of desiccant whereas a more expensive shell must be considered for suction line installation to provide for low pressure drop through the drier. A 1/2 lb. pressure drop in the suction line is important as compared to a relatively unimportant 2 lb. drop in the liquid line.

LOW SIDE DRIERS :-

MERITS :- 1. Cold location of drier provides for greater capacity from adsorbent type desiccants.

2. In this location there is less possibility for moisture to enter evaporator.

3. Convenient and economical for manufacturer of some types

of original equipment to install the drier in this location.

4. Less possibility for release of moisture from adsorbent type desiccant during "off-cycle".

DISADVANTAGES :-

1. Full refrigerant flow does not pass through drier; moisture pick up accomplished by transfer from refrigerant to desiccant. Therefore, a greater time is required to obtain equilibrium between the moisture in the refrigerant and the desiccant.
2. Greater possibility of trapping oil in the drier.
3. Inconvenient to replace drier on original equipment in the field.
4. Not practical to install drier in this location in the field.
5. Does not stop moisture from freezing in the expansion device
6. Filtering of the refrigerant medium is not obtained since the full flow of refrigerant does not pass through the drier.

SUCTION LINE DRIERS :-

ADVANTAGES :-

1. Cold location of drier provides for greater capacity from adsorbent type desiccant.
2. Less possibility for release of moisture from adsorbent type desiccant during "off-cycle".
3. Sulphur dioxide can be adequately dried in the vapor side of the system.

DISADVANTAGES :-

1. Greater possibility for excessive pressure drop to develop

through the drier. Pressure drop is objectionable in suction line as it materially affects system capacity. A 1 lb., pressure drop in the suction line at minus 25 deg. F on Freon-12 represents a 6 to 8% loss of capacity. Since the consumer is paying for Btu per unit he is also being penalised for extra power consumption.

2. Greater possibility of trapping oil in the drier if drier is not properly designed.

3. Does not stop moisture from freezing in the expansion device.

4. Does not provide means of filtration ahead of the expansion device. Usually necessary to install a filter in addition to the drier.

5. Comparatively high refrigerant velocity provides for less intimate contact between refrigerant and desiccant which requires a greater period of time to reach equilibrium between the moisture in the refrigerant and the moisture in the desiccant.

6. Moisture can enter the evaporator.

6.2 DESICCANTS :-

A desiccant is a chemical compound capable of absorbing or reacting chemically with the moisture contained in the liquid or gaseous refrigerant-oil mixture. A brief discussion of the various desiccants which have been used in the refrigeration industry is given here.

Desiccants may be divided as follows :-

6.2.1 THE CHEMICAL ACTION TYPE :-

They remove moisture from the refrigerant by chemical action. When they react with water form either hydroxides or hydrates. They have certain disadvantages of undergoing chemical change often accom-

-panied by liberation of minute quantities of dust or sediment, which may affect their physical structure. Some of them, particularly, some of those forming hydrates, are deliquescent i.e. will take up moisture to a point where the entire mass will liquify, which will cause serious trouble. Also one or two of these materials are corrosive in the liquid phase. They may also change volume resulting in an increased resistance to flow. The desiccant can be reactivated.

a) $CaCl_2$:- $CaCl_2$ has been extensively used and is the fastest drying agent in common use. It is not capable of reducing moisture content to a low level. It may be used with all refrigerants. Excess moisture produces a solution of $CaCl_2$ which is highly corrosive. It will not remove acid and should not be left on a machine longer than a few days. It should be used with caution.

b) CaO :- It removes water and acid but slow in action. While uniting with water it forms $CaOH$ or slaked lime which is a fine powder, but not harmful except sometime plugging the screens. It can be used with all refrigerants except Sulfur dioxide.

c) Drierite :- It is prepared as a granular white solid. It dusts somewhat more than activated alumina but does not much cause trouble. It is also cast in sticks. It removes water by chemical action as well as physical action but other remarks are same as for activated alumina.

d) Acid in the system causes corrosion and hence the corrosion arrestors are used. They make use of feathered zinc or zinc mass. The acid in the system is neutralized by these arrestors.

6.2.2 THE PHYSICAL AGENT TYPE :-

These materials which take up moisture and other undesirable compounds principally through physical means and they can be reactivated.

a) Silica gel is a glass like substance (silicon dioxide) which is very good adsorber for moisture acids and also refrigerant oil. It does not dust . Activated alumina is a granular aluminum oxide which also removes moisture and acids by adsorption. It dusts slightly on handling but this is not a source of trouble.

Both of them had extensive and satisfactory use with sulfur dioxide, methylene chloride, methyl chloride, Freon-11, and Freon-12 and they are used with sulfur dioxide in vapor phase only, with other refrigerants in either suction or liquid lines usually latter. They usually become clogged with refrigerant oil which nullify their adsorbing qualities. They may be left on the machine indefinitely.

Silica gel has the following action. It becomes completely saturated with refrigerant and because it has preference for inorganic compounds, over organic compounds, the refrigerant and oil are replaced instantaneously by moisture or acids passed through the silica gel charged dehydrator. It has a capacity in the dehydration of refrigerants under normal conditions of operation from 10 to as high as 20 % of its weight in water or other undesirable compounds.

It has been proved experimentally that inspite of its composition same as sand, it is not abrasive at all.

Above are the most commonly used desiccants.

6.2.3 THEORIES OF ADSORPTION

Numerous theories of adsorption have been proposed but no theory is universally acceptable or applicable to all adsorption phenomena(29). Present day theories may be roughly divided into two groups. The first group is based on the general picture of the adsorbed layer as monomolecular at low relative pressures and becomes much multi-molecular as the relative pressure approaches unity.

The second group usually referred to as the 'capillary condensation theories', generally pictures the adsorbate as condensing to the liquid state in the pores of the adsorbent after the walls of the pores are covered with a monolayer. The capillary condensation view point is particularly favoured by some since it offers a conveniently visualizable picture of working model for understanding the adsorption of materials by silica gel.

6.3 DESICCANT CHARACTERISTICS :-

The desiccant must be able to remove water from the refrigeration system efficiently, be physically strong, nondeliquescent and chemically inert. With the increasing emphasis being placed on solid desiccants as dehydrating agents in refrigerating systems, evaluation of the properties of the various materials employed becomes increasingly important. The design of driers for refrigerating systems requires data on the characteristics of the desiccants without which drier design may not be proper.

6.3.1 ADSORPTION CHARACTERISTICS :-

Dusting has been one of the serious difficulties attendant to the use of desiccants in a refrigeration system since the abrasive desiccant dust has a detrimental effect on compressor parts and may contribute to the clogging of the system. For this reason it has been common to run attrition tests on desiccants considered for use in refrigeration systems. It seemed probable that the results of attrition would depend on the details of the test used. Since such tests can be designed, such that different forces can be applied to the particles under test. For this reason tests emphasising the action of different forces were applied. These tests were carried out by B.J. Stork(31) on siliceous desiccants only. Activated alumina was not included since the only material available was obviously much more friable than the others and the comparison with the siliceous desiccants would have had little meaning.

Four ounce bottles loosely filled with the desiccant tested from which 50 mesh material had been screened. In the first test desiccant filled bottles were subjected to a sudden and steady vertical jolting somewhat analogous to that obtained in a pressure system. The impact produced was achieved by a sudden drop of ½ in., 105 times per minute. A horizontal motion, 280 times per minute with a displacement of 2 in., was imparted to the desiccant filled bottles in the second test. The test were run for 24 hours and seven days periods, following which screening was carried out.

In the vertical motion tests, comminution is accomplished predominantly by compressive forces while grinding is very limited. Face to face grinding is the dominant force of attrition in the horizontal motion test and it is to be expected that these types of motion will create a larger amount of fines. The results of the horizontal motion attrition test on silica gel and sova bead indicate that the rate of producing fines (i.e. 50 mesh material) is not a linear function of time. Observations of the desiccants after conclusions of the attrition tests showed a dust film on the outer surface. It seems probable that this dust film protects the desiccant from further rapid attrition. For granular silica gel, one week's production of fines only doubled the first days' production. Sova bead fines increased four folds in the same period. This is understandable in the light of the greater weight of sova bead which can more easily penetrate the protective dust film.

The vertical motion test reported somewhat different picture and it is apparent that different forces are acting to produce comminution. The compressive forces emphasized in this test, have a more serious effect on silica gel than on sova bead. These results probably reflect the greater strength of particles formed by the bead technique.

Comparison of the results obtained with the two types of tests indicate that the no single test can be used to indicate the resistance of desiccants to all type of attrition. Further comparison with the results of P.L. Veltman and C.S. Ucr[illegible] these tests based on tumbling particle against particle in air or carbon tetrachloride liquid medium. 120 mesh material had been screened out. The results are shown graphically in Fig. 42. It should be emphasized that these tests are obtained using an accelerated test and that probably in actual operation far less attrition is realised with any of the materials, hence most weight should be given to that test which emphasises those forces which are probably causing attrition in particular application. In a refrigeration system it seems probable that the attrition is caused largely by compressive forces resulting from collision of the particles with one another or with the container walls, while the refrigerant acts as a lubricant and diminishes face to face attrition. This indicates that results of the vertical motion tests should deserve most weight. However, these data do indicate on a relative basis the respective resistance to attrition for the several desiccants. Increased resistance to attrition insures improved performance.

6.3.2 WATER HOLDING CAPACITY OR EQUILIBRIUM CAPACITY :-

Water removing ability is most important from the stand point of the equilibrium water capacity. High equilibrium capacity (useful concentration) is especially desired for long term operation. Certainly a refrigeration unit could be operated until a steady state occured. After this attainment a constant amount of moisture would be circulating around the system at all cross - sections in a unit time. There must then be established some equilibrium between the moisture in the desiccant and moisture in the refrigerant.

Some data have been published by Voltman and Waring(32), Stark(33) and Pennington(34) on moisture equilibria between Freon - 12 and the more commonly used commercial desiccants is given here.

A single drop of water weighs approximately 0.05 gms. and if it is present in 1 lb. of refrigerant amounts to approximately 110 ppm water concentration. It is rather hard to remove the equivalent of a drop of water when spread throughout a system containing organic materials and somewhat porous metal walls, which act as fairly good water retainers or desiccants in themselves.

Experimentally the evaluation of the utility of a desiccant is not easy. This is particularly true with respect to water removal ability and attrition characteristics. A new method for determination of the absolute capacity of a desiccant for removing water from refrigeration systems is given here.(32) It consists of presaturating the portions of the desiccant under examination to the known water content and then testing the drying ability employing water saturated refrigerant. By this technique the equilibrium water content of Freon-12 in contact with desiccants containing known amounts of water is determined. Equilibrium is established by repeatedly passing the Freon-12 over the desiccant using a weight ratio of approximately 125 parts of Freon-12 per part of desiccant. This simulates closely what is experienced in the field when the desiccant picks up water left in the system by the manufacturer. Water content is determined by vaporizing the Freon-12 under controlled conditions and collecting the water vapor with P_2O_5.

In Fig. 40 is shown a comparison of drierite, activated alumina, sova beads, Sporlan 'catch all' drier and silica gel on an absolute basis of equilibrium capacity versus the equilibrium water content in Freon - 12. These data were obtained under identical conditions(32). The Figure shows the relative ability of the several desiccants to remove water from a refrigera-

-tion system. At approximately 12 ppm of water content in the Freon-12, silica gel will hold three times as much water as the activated alumina employed. At the 36 ppm level silica gel will hold 8.5 units compared with 0.75 units for Drierite or a ratio of 11 : 1 on weight basis.

(31)

Results of experiments carried out by B.J.Stork with sova bead, silica gel (14-20 mesh refrigeration grade) and activated alumina (4-8 mesh) are given below :-

Efforts were made for obtaining information under conditions approximately as closely as possible those existing in service. The experimental work was performed on a carrier food freezer unit.

The curves shown in Fig. 43 indicate the equilibrium capacity of the desiccant on the high pressure high temperature side for water in Freon-12, when equilibrium exists. Each point on the curve gives the moisture content of Freon-12 in equilibrium with the desiccant of the corresponding moisture content under operating conditions used. If sova bead or silica gel of 10.5 per cent moisture content were contacted with Freon-12 at equilibrium, Freon-12 would assume a moisture content of 27 ppm.

The curves for granular silica gel and sova bead coincide. It is seen that even with 2 to 3 per cent water in either desiccant, relatively dry Freon may be obtained. Possibly then, desiccants may be of considerable help in factory drying of systems. Investigation was not done on the high pressure side at low temperatures as it is time consuming and costly.

Fig. 44 shows degree of dryness attainable with sova bead and silica gel tested on the low pressure side. Because of very violent motion imparted to the desiccant activated alumina was not used. Activated alumina except in molded form has low resistivity to attrition.

By assuming i) that the concentration of moisture for a given desiccant is directly proportional to the saturation value and ii) that the saturation capacity of a desiccant is reduced by the presence of the refrigerant and that the reduction is proportional to the pressure of the refrigerant. Pennington(34) derived a mathematical relation to determine the water in the desiccant by the refrigerant analysis, and also carried out work on refrigeration units to derive equilibrium relations and conclusions drawn are as follows :-

CONCLUSION :-

1. Silica gel and sova bead have been found to have equal capacity for moisture absorption in Freon-12.
2. These desiccants can best be dehydrated to absolute bone dryness by vacuum at an elevated temperature.
3. Freon-12 in equilibrium with bone dry adsorption type desiccant will contain 0.8 ppm of water.
4. The equilibrium relation for the low side may be expressed as a straight line upto about 50% relative humidity.
5. A straight line can also be used for the high side but should be restricted to moisture levels lower than 10 ppm.
6. The relations for all conditions can be represented by a family of parabolas.

7. These parabolas may be deduced from a single one determined experimentally.
8. A drier containing the desiccants tested when placed at the beginning of the evaporator is more than twice as effective as one in the suction line, Freon-12 being the refrigerant.
9. This same drier is about 6 times as effective as one on the high side if the drier temperature is as much as 130 degree F.

Methods described previously were tedious and time consuming . A method is presented by A.J.Gully, H. A. Tooke and Luis H.Bartlett(35) which yields reliable data on moisture equilibria between refrigerants and solid desiccants. The equipment is simple and easy to operate with minimum sources of variation. Data are presented which indicated that the equilibrium relations expressed in terms of desiccant moisture content vs relative water saturation of Freon-12 are virtually independent of temperature. The data verifies Pennington's(34) first assumption but are not in agreement with the assumption No.2. The data indicate that the temperature is the determining factor and that pressure has no significant effect.

6.3.4 FRICTION FACTORS OF DESICCANTS IN REFRIGERATION SYSTEMS:-

The disadvantage of using a drier, besides the initial cost, is the pressure drop resulting from fluid friction in the drier system. There is very little information available on the magnitude of such losses and on the laws [illegible] governing the behaviour of flow through driers . Hence investigation was made by Daniel. G. Schlarman( ) to study the flow of refrigerants to liquid driers and to make a theoretical and experimental analysis of the pressure drop encountered. The effects of the various variables involved were investigated so that data could be presented in a simplified form which can be used to predict the pressure drop across any drier under various operating conditions. The factors studied included the effects of void volume and mass flow on pressure drop, with the main objective being to establish a Reynolds number- friction factor relationship for the different materials. Tests were made on three widely used desiccants in the refrigeration industry namely activated alumina, silica gel and sova bead. Freon-12 was used as the refrigerant for all test work. Results of this investigation showed a good correlation between the Reynolds number and friction factors.

Now $$R_m = \frac{G\,D_p}{6\mu}\left(2/3\ \frac{e}{1-e}\right) \qquad \text{———}(22)$$

where,
R_m = modified Reynold's number.
G = mass flow per unit of area or lb/sec./s.ft.
D_p = mean particle diameter.
μ = absolute viscosity of the refrigerant lb/sec/s.ft.
e = fraction void volume
$$= \frac{\text{empty space in packed drier}}{\text{total volume of drier.}}$$

1. PRESSURE DROP AGAINST MASS FLOW :-

The curves for pressure drop against the mass flow found to be similar to those obtained for flow in pipes. Each curve consisted of two parts, one for laminar flow and other for turbulent flow. In every case the laminar flow portion had a slope of approximately one, indicating a linear relationship between flow and pressure losses. For the turbulent region the slope was approximately 1.8 for all curves again corresponding to a value for rough pipes.

The results indicate clearly that the resistance to flow is a function of the particle size. For each material the pressure loss for a given set of operating conditions increased as the particle size decreased. Indications are that the shape of the particles has an important influence on the pressure drop. The effect is probably due to expansions and contractions introduced & the resulting shape of flow channels. The more uniformly shaped particles appears to be by far the more desirous.

The data verified the fact that the void space has no direct relationship for pressure loss. The void volume remain almost constant with particle size whereas the pressure loss showed a marked increase with a decrease in mean particle size.

D. REYNOLDS NUMBER - FRICTION FACTOR RELATIONSHIP :-

The data for activated alumina plot as a single curve for both laminar and turbulent regions. The straight line relationship in the laminar range established the expression :

$$f = 10/R_e \quad \text{————————————} \quad (12)$$

It should be noted that this relationship is not obtained for non-circular cross sections.

Each particle size of silica gel has its own

Reynolds number - friction factor relationship. The granules of silica gel resembles small rectangular parallelepipeds, characterized by sharp edges and corners.

The three curves are seen to give a common straight line relationship for the laminar region. Again this line has a slope of -1 and can be expressed by ;

$$f = 170/R_e \quad \text{———————} \quad (13)$$

The relationship for sorbead indicate that, of the three materials tested, it offers least resistance to fluid flow. This should be expected since this material has the smoothest surface and is by far the most uniform shape, the latter probably being the more important. For the laminar region, the friction factor is ;

$$f = 70/R_e \quad \text{———————} \quad (14)$$

Fig. 45 shows these results in the form of a set of master curves.

The pressure drop due to refrigerant friction in drier is given by the following derived relationship ;

$$\Delta p = \frac{G^2}{2g\rho} \cdot f \cdot L/D_p \left\{ 3/2 \, \frac{1-e}{e} \right\} \text{————} (15)$$

Substituting this value of 'f' in equation (15) one can get pressure drop across the drier.

In Eq.(15) Δp = pressure drop across the drier. lb/s.ft.
L = length of the drier in ft.
ρ = Refrigerant density. lb/cu.ft.
g = gravity factor ft/sec^2

Since dehydrating by adsorption is a surface phenomenon, it is desirable to have as much refrigerant as possible come in contact with the desiccant. To insure good mixing, a moderate degree of turbulence is definitely required i.e. the Reynolds number should be well above the critical value. The upper critical values for the various commercial sizes are as follows :-

Activated alumina	- 70
Silica gel	- 22
Sova bead	- 60

The mass flow through any drier should be selected to give a Reynolds number in excess of the above values. Since the pressure drop increases almost as the square of the mass flow, some discretion must be exercised in determining just how high above the critical value the Reynolds number should be ? The designer of drier should take the operating conditions into consideration in order to prevent the pressure drop from becoming excessive.

6.3.5 OTHER SORPTION CHARACTERISTICS OF SILICA GEL :-

Little specific information exist as to the effect of repeated activation. Most investigators prefer to use the zero-sorption point (ZSP) as a standard of reference in reporting results. This point is defined as the one at which there is no further loss of weight up when the desiccant is activated under specific conditions.

Some investigators used the ignited weights of desiccant as a zero point claiming that the reproducibility of the Z-SP was not good enough to allow its weight as a reference point. This ignited weight can be obtained by heating the desiccant to a constant temperature of 800° C.

However, it is known that desiccants such as silica gel, activated alumina and drierite loose their desiccating properties under such treatment. In addition silica gel and activated alumina, when activated contain a certain amount of bound water without which they cannot function as desiccants. This bound water is lost during ignition at 800 deg. C. There is, therefore, valid objection to the use of ignited weights.

OBJECT OF RESEARCH :-

The object of this research (37) was in part to determine the water zero-sorption points of silica gel, activated alumina and drierite, and to consider reproducibility when the desiccants were activated at atmospheric pressure and under vacuum.

CONCLUSIONS :-

a) The Z-SP for silica gel is reproducible after repeated activation - water exposure cycle, either at atmospheric pressure or at vacuum.

b) The Z-SP for activated alumina is not reproducible after activation - water exposure cycles, either at atmospheric pressure or under vacuum. This indicates the formation of a new phase.

c) The Z-SP for drierite is less reproducible than that of a silica gel after repeated activation - water exposure cycles - either at atmospheric pressure or under vacuum.

d) There is no significant difference in reproducibility of the Z-SP for atmospheric activation as compared with vacuum activation except for activated alumina.

e) The Z-SP and the ignited weight point can be consistently reproduced.

6.4 MOLECULAR SIEVE - A REFRIGERANT DESICCANT :-

A new class of adsorbents, called Molecular sieves, has been developed recently(23). They are crystalline alumino - silicates. Outstanding characteristic is their ability, when heated, to give up their water of hydration with little or no change in crystal structure. The dehydrated crystals are thus interlaced with regularly spaced channels of uniform size and of molecular dimensions in which adsorption may occur. Molecules that are too large, to enter these channels cannot be adsorbed while smaller molecules of suitable size and shape may enter the inter-crystalline pore system and be adsorbed.

Due to the molecular size effect, both Freon - 12 and Freon - 22 and oil are excluded from molecular sieves ; thus the adsorbent volume is used only for water adsorption, and performance is not affected by the presence of oil.

Molecular sieves are selective adsorbents which have a high affinity for water. They have a high water capacity at temperatures as high as 140 deg. F. and at water concentrations as low as 10 ppm by weight. Therefore, a smaller size drier cartridge is required for drying refrigeration system to prevent freeze-ups and corrosion and permit longer utilization of the present equipment. A smaller cartridge can offer economic advantages as it occupies less space and requires less metal to manufacture. The high water capacity of molecular sieves permit use of this desiccant to dry unit after assembly which eliminates the costly and time consuming compressor drying operation. Because of its high water capacity at high temperatures the drier cartridge can be located close to the compressor without experiencing a significant loss in water capacity. Additionally, with water capacity being practically unaffected, by the presence of oil, a drier cartridge possibly could be placed in the compressor housing.

Tests(23) have shown that molecular sieve perform effectively in refrigeration systems. Under severe conditions of vibrations, some dusting of the molecular sieve particles occur, but by use of a proper filter this dust can be retained. Moreover, if molecular sieve dust should enter the refrigeration unit, no damage will be done to the system. No pressure is built up across the drier and the system operates efficiently.

CONCLUSIONS.

CONCLUSIONS:

In the earlier pages an attempt has been made to present the available information on moisture, its sources and effects, methods of moisture determination and removal from the refrigeration systems.

However, no general statement as regarding the applicability of the data presented can be made as no identical operating conditions are found on actual machines. Manufacturers of refrigeration equipment, contractors and field engineers have to be acquainted with moisture and its behaviour under operating and non-operating conditions. They have to set their own safe limit of moisture and then select methods for moisture determination and removal which is best suited to their equipment and existing conditions.

In India, refrigeration and air-conditioning industry has not developed but in future this subject will be one of those in which the manufacturers of the refrigeration equipment and its users are most interested.

-: O :-

APPENDIX.

TABLES AND FIGURES.

TABLE - 1.

Moisture Distribution between Gaseous & Liquid Phases of Saturated Solutions of Water in Freon-12.

Temperature. °F	Density of saturated Freon-12 vapor lb/cu.ft.	Density of saturated water vapor lb/cu.ft.	% water in vapor phase saturated with both water & Freon % by wt.	% water in liquid phase at saturation (solubility) % by wt.	Ratio of water contents, vapor to liquid.
-40	0.2557	0.764×10^{-5}	0.0029	0.00017	17.1
-20	0.4049	2.36 x 〃	0.0058	0.00038	15.3
0	0.6100	6.75 x 〃	0.0110	0.00083	13.3
20	0.9071	1.765×10^{-4}	0.0195	0.00163	11.9
30	1.065	2.772 x 〃	0.0260	0.00230	11.3
40	1.263	4.09 x 〃	0.0324	0.00320	10.1
50	1.445	5.86 x 〃	0.0406	0.0041	9.9
60	1.743	8.30 x 〃	0.0476	0.0058	8.2
70	2.028	1.15×10^{-3}	0.0567	0.0075	7.6
80	2.353	1.58 x 〃	0.0671	0.0098	6.9
90	2.721	2.14 x 〃	0.0785	0.0128	6.1
100	3.125	2.86 x 〃	0.0915	0.0164	5.6

TABLE - 11

Refrigerant	Water by weight, %	Results at room temperature.
Sulfur dioxide	0.03	Slight discoloration
	0.10	Slight scale
	0.16	Heavy scale
		Presence of air did not affect results.
Methyl chloride	0.02	Slight discoloration
	0.03	Marked discoloration Very slight scale.
	0.05	Moderate to heavy scale.
		Presence of air increased corrosion in all cases.
Freon - 12		Similar to methyl chloride.

TABLE - 3.

		Freon	CH_3Cl	SO_2
1.	Volume of liquid removed from cylinder at 70° F - CC	100	100	100
2.	Weight of liquid removed from cylinder at 70° F - grams	132.5	100	143
3.	Weight of "Flash" gas - gms.	32.5	20	20
4.	Weight of liquid sample remaining in flask - grams.	100	100	143
5.	Volume of liquid sample remaining in flask - CC	100	100	100
6.	Temperature of liquid sample remaining in flask - Deg. F.	-20	-22	-10
7.	Original sample remaining in flask - percent.	75	80	80
8.	Grams of water in 100 CC. sample which are equivalent to 0.0025% (25 ppm) water by weight. (This figure to show delicacy of test). - grams.	0.0033	0.0025	0.0037

TABLE - 4

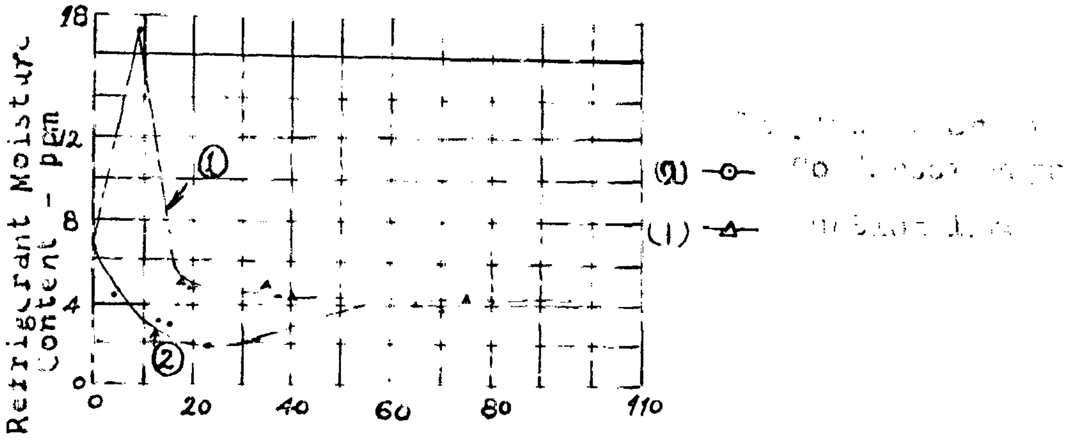
Effect of different sampling methods on moisture content of sulfur dioxide.

Cylinder No.	Moisture by ordinary method	Moisture by standard method.
403	0.0020 %	0.0019 %
405	0.0028 %	0.0031 %
301	0.0034 %	0.0014 %
304	0.0074 %	0.0024 %
303	0.0038 %	0.0019 %
307	0.0034 %	0.0024 %
574	0.0036 %	0.0032 %
107	0.0030 %	0.0025 %
Average	0.0034 %	Average 0.0029 %

TABLE - 5.

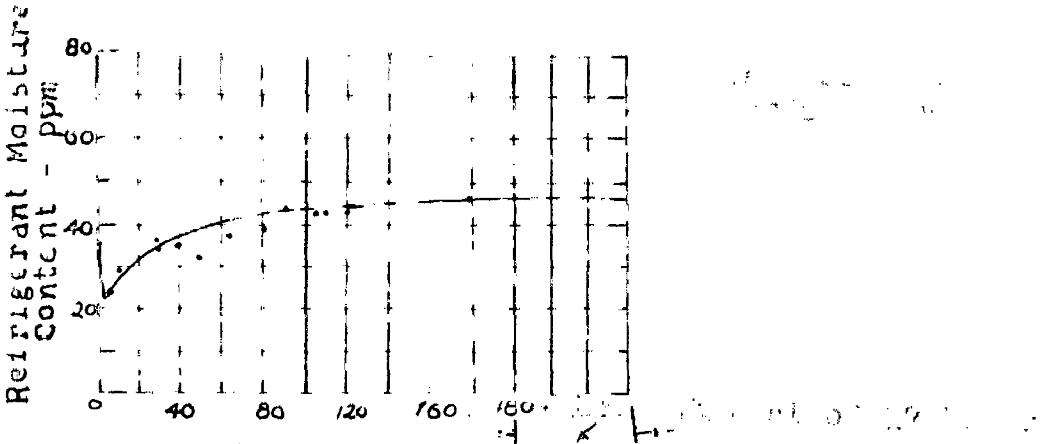
Vapor pressure and Density of Saturated Freon-12.

Temperature o F	Vapor pressure m.m.	Density lb/cu.ft.
-70	205	0.116
-65	239	0.134
-60	277	0.154
-55	320	0.175
-50	368	0.200
-45	422	0.226
-40	482	0.256
-35	548	0.288
-30	621	0.324
-25	702	0.364
-20	790	0.404


Refrigerant Moisture Content - ppm
18
12
8
4
0
0
20
40
60
80
110
①
②
(a) -o-
(1) -Δ-

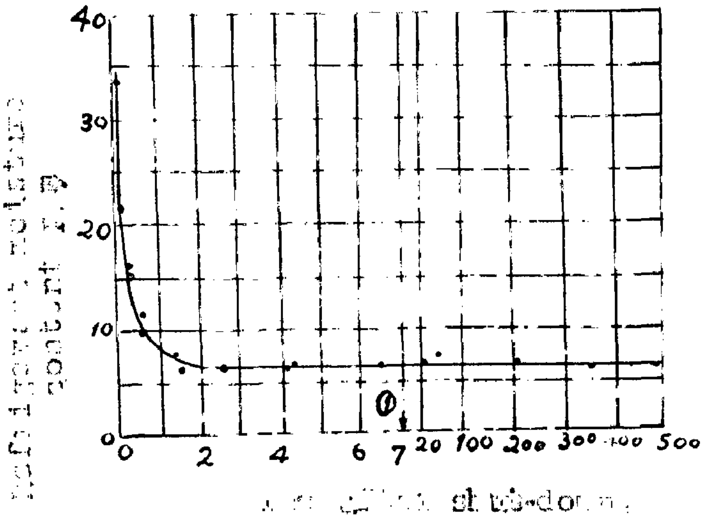
[illegible]

[illegible]


Refrigerant Moisture Content - ppm
80
60
40
20
0
40
80
120
160
180

[illegible]

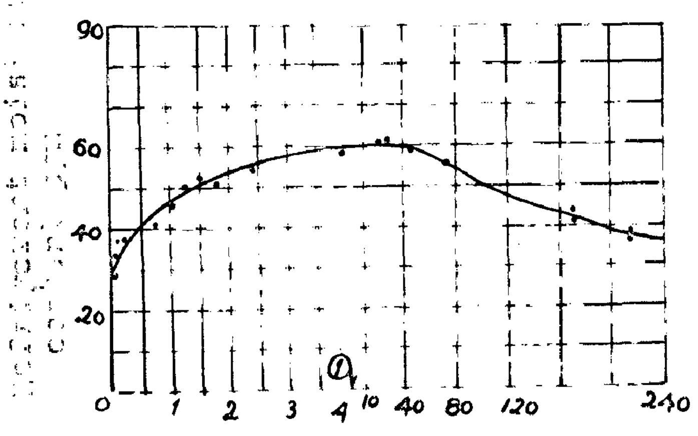
[illegible]



Samples taken fro[illegible]
[illegible] vapour

1 [illegible]

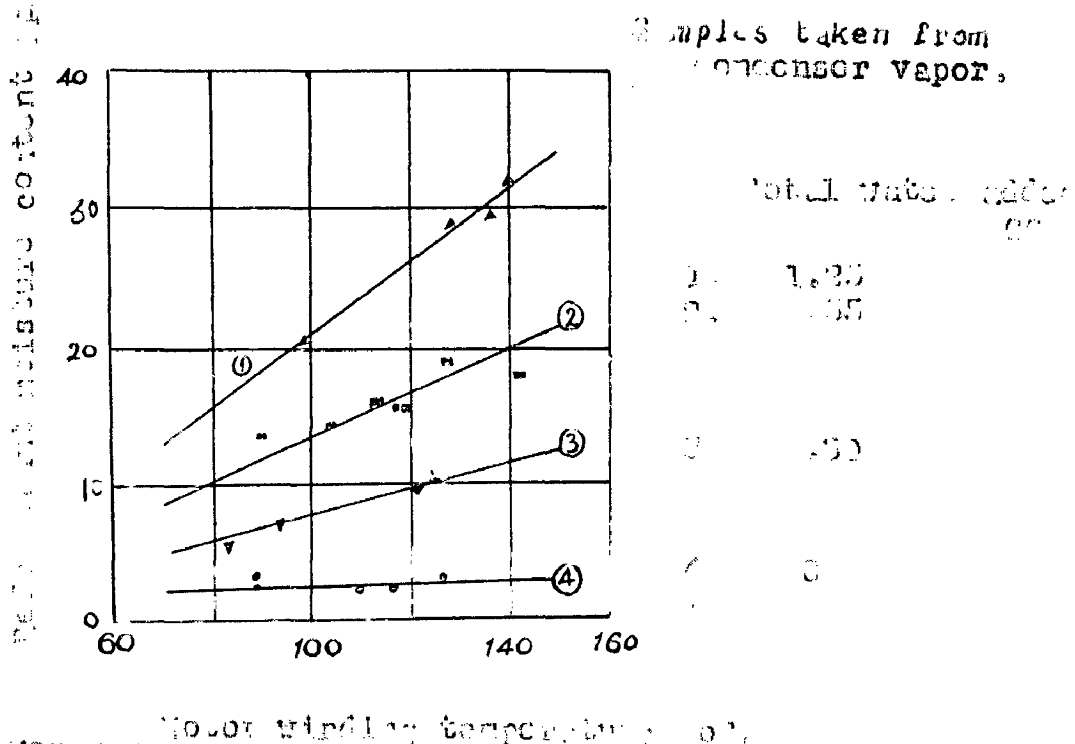
Fig. 8. [illegible] content as a function of [illegible] shut-down [illegible] refrigeration system [illegible]



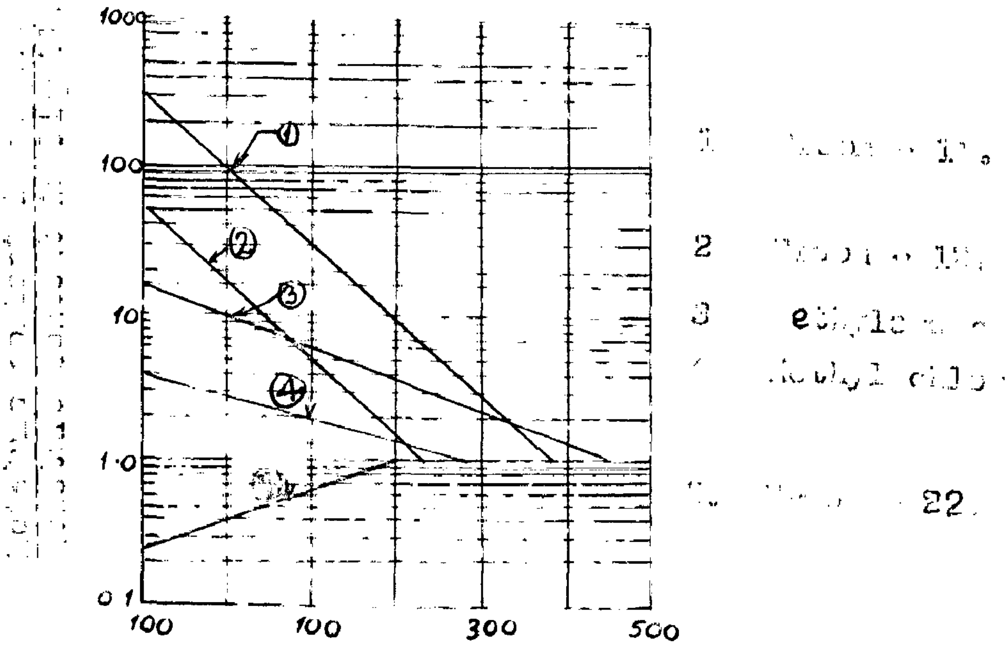
[illegible]

1 [illegible]

[illegible]



[illegible] at [illegible]
of motor winding [illegible] system
[illegible]



[illegible]

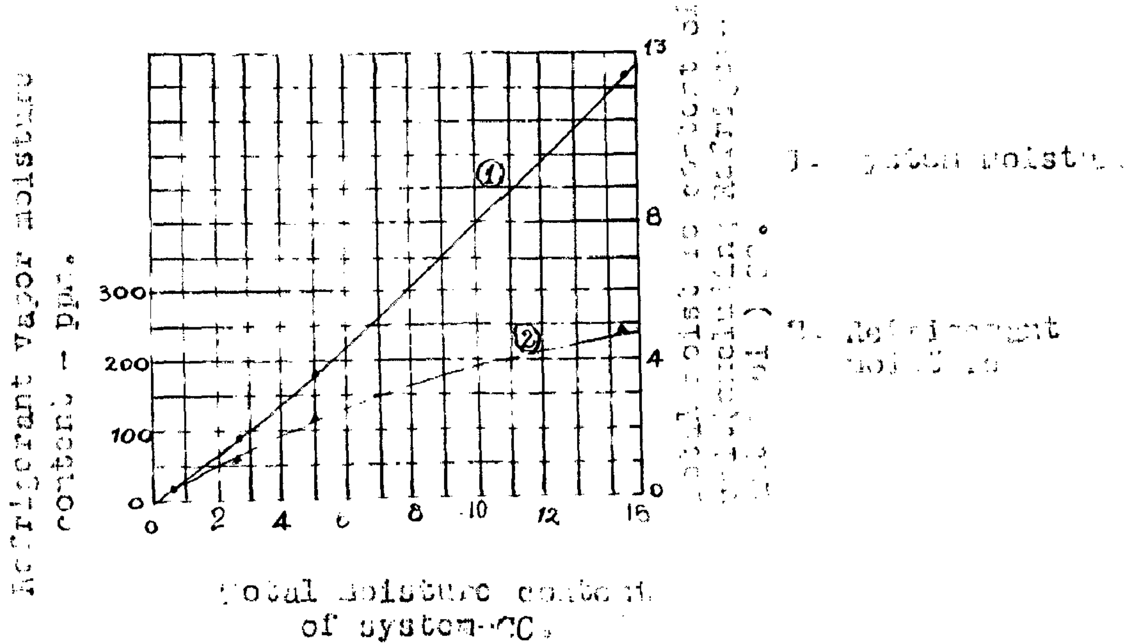


Fig. 7. WORK CHART for determining of [illegible] moisture in system (including and excluding [illegible]) from single moisture test at [illegible] refrigeration system charged with [illegible]

Ref. 3

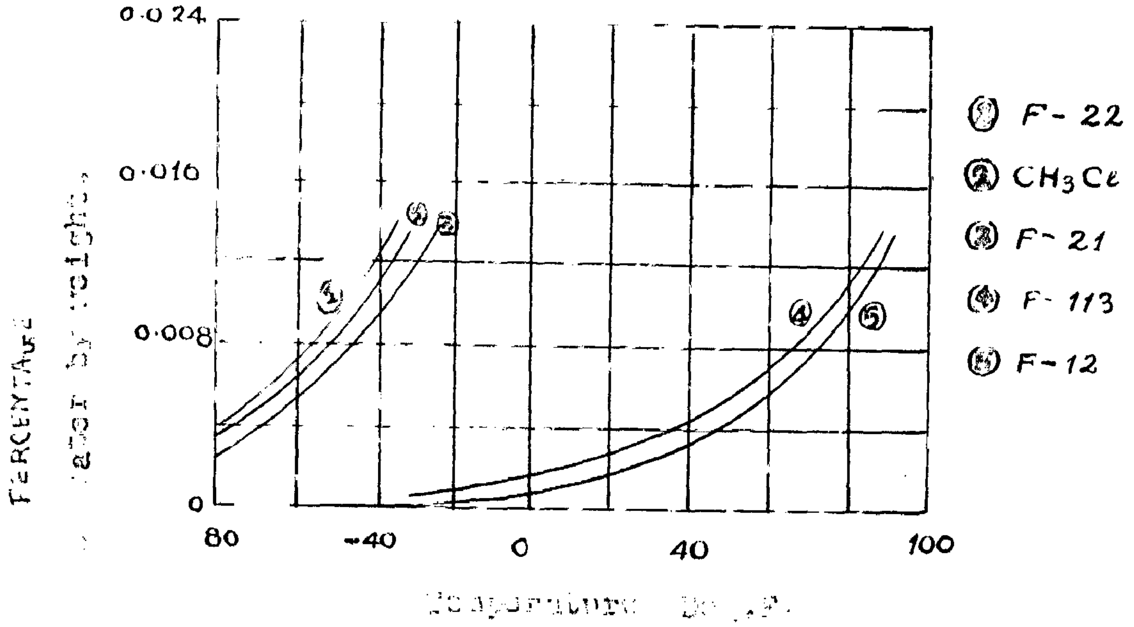
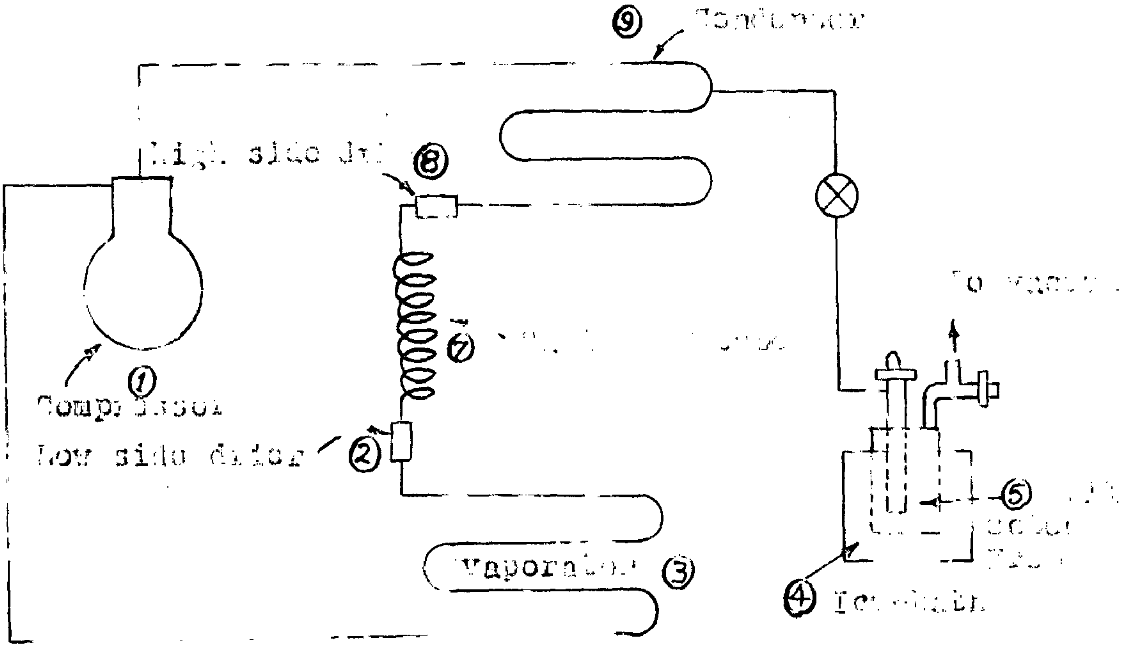


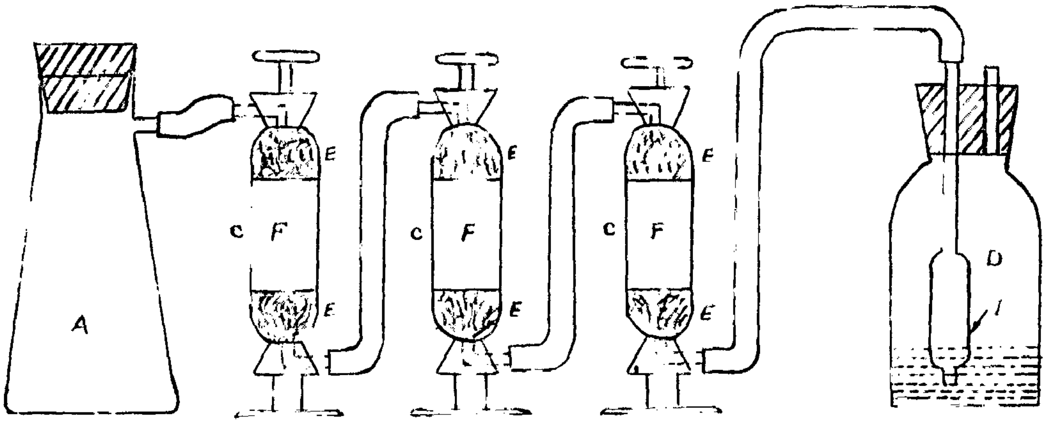
Fig. [illegible] Solubility of Water in Refrigerants.

Ref. 7



[illegible] Schematic diagram that shows sampling technique for refrigeration system.

Ref [illegible]



[illegible] for determination [illegible] in liquid [illegible]

A [illegible] flask
Absorption [illegible]
D [illegible] wash bottle.

E [illegible] glass wool
F [illegible]
F [illegible] solution
[illegible]

Ref [illegible]

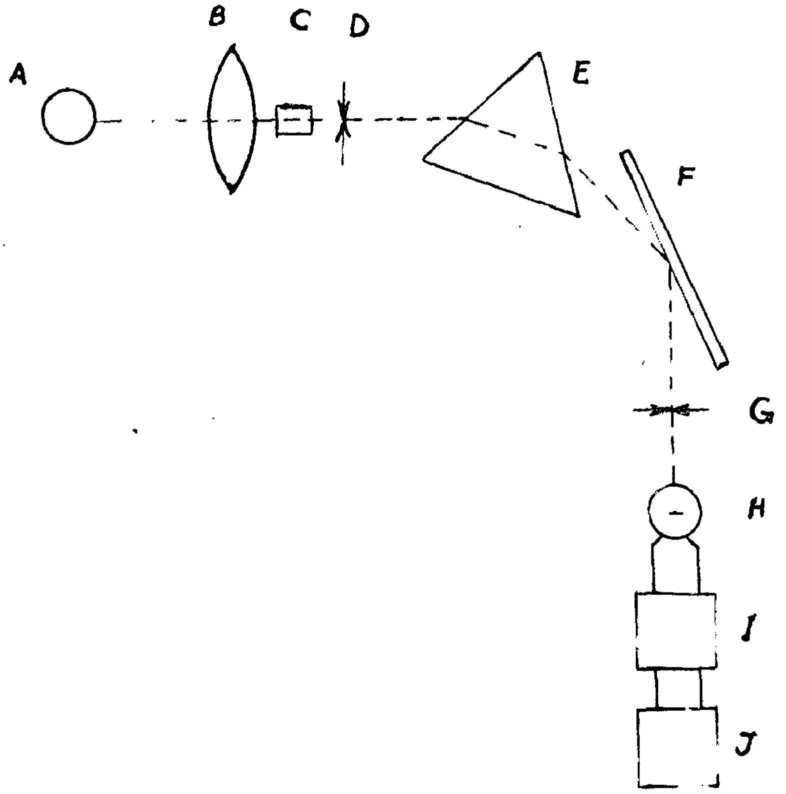


Fig. 11. Simplified schematic diagram of an infrared spectrophotometer.

A - Source

B - Focusing system

C - Sample cell

D - Entrance slit

E - Prism

F - Focusing mirror

G - Exit slit

H - Detector

I - Amplifier.

J - Indicator.

Ref. 15

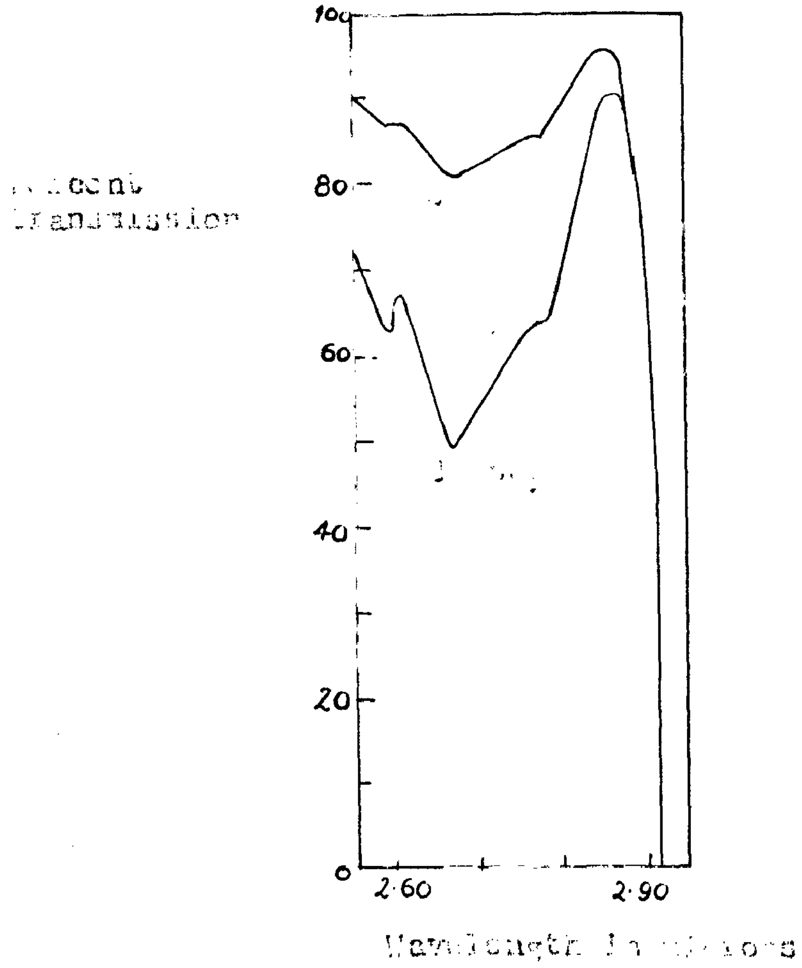


Fig. 12. A portion of the infrared absorption spectra of 3 and 20 ppm water in [illegible].

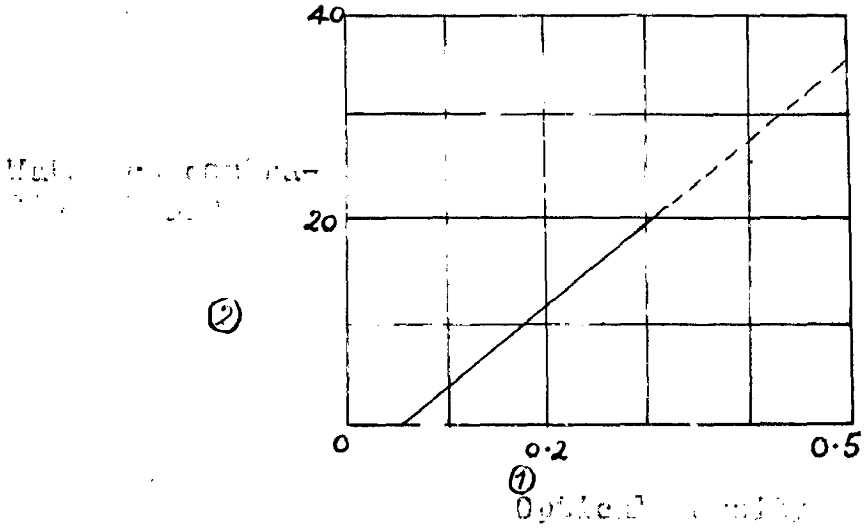


Fig. 13. Calibration chart showing relation between optical density and water content in ppm.

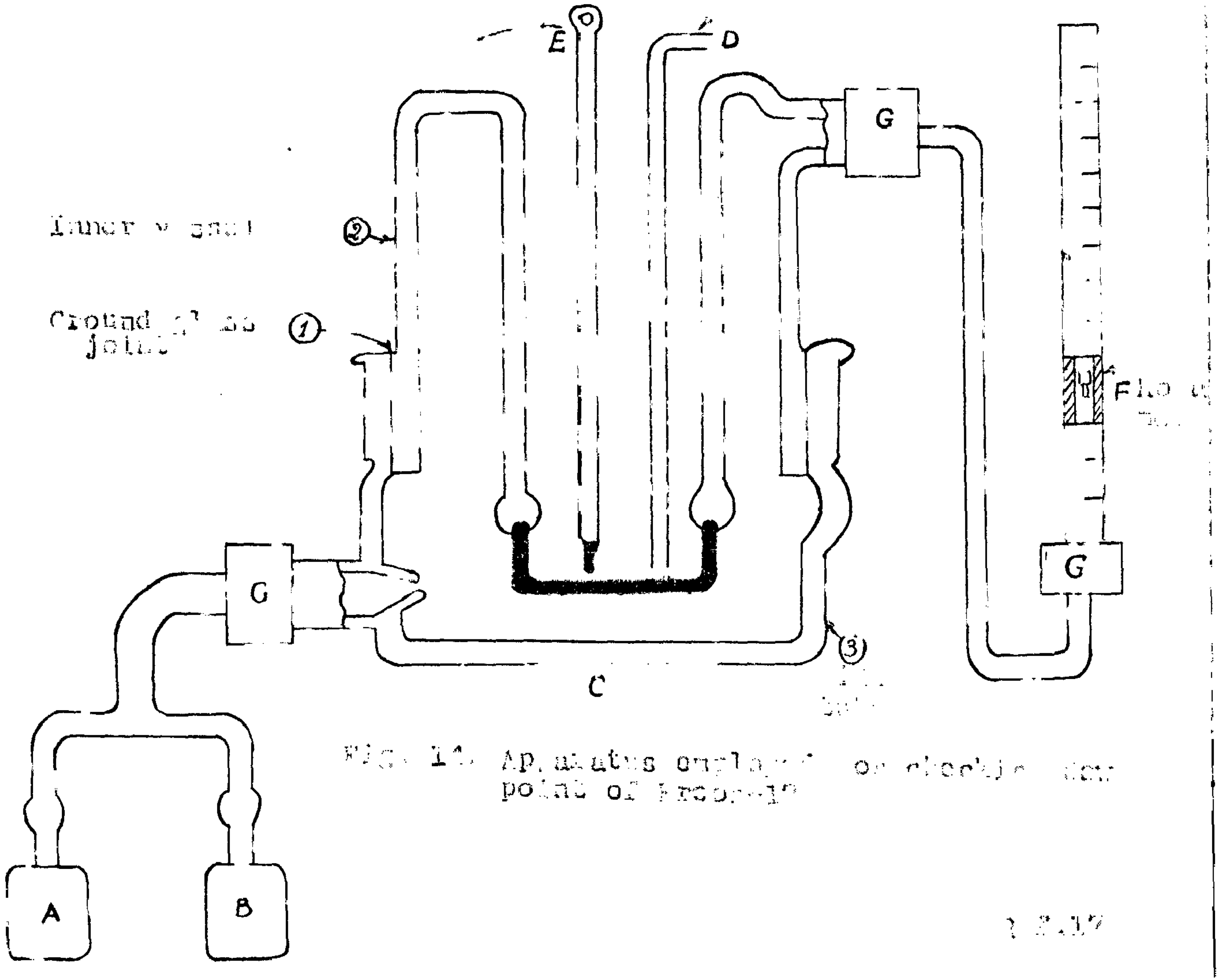
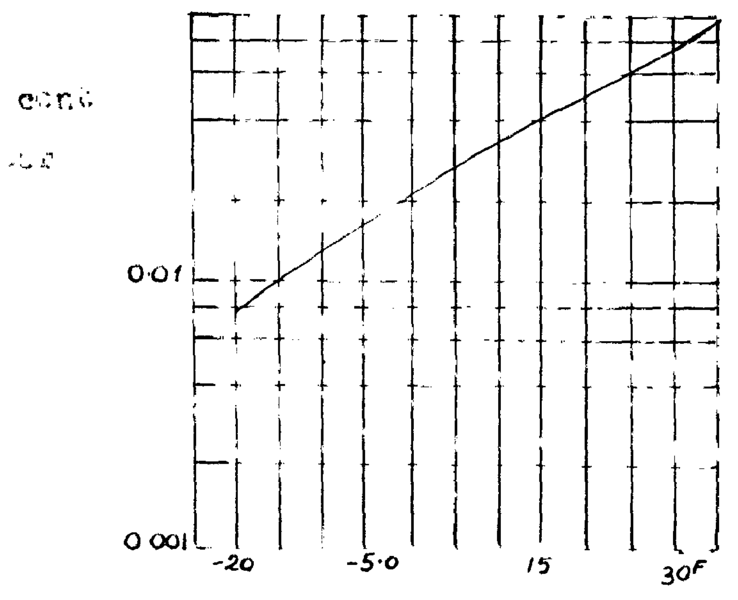
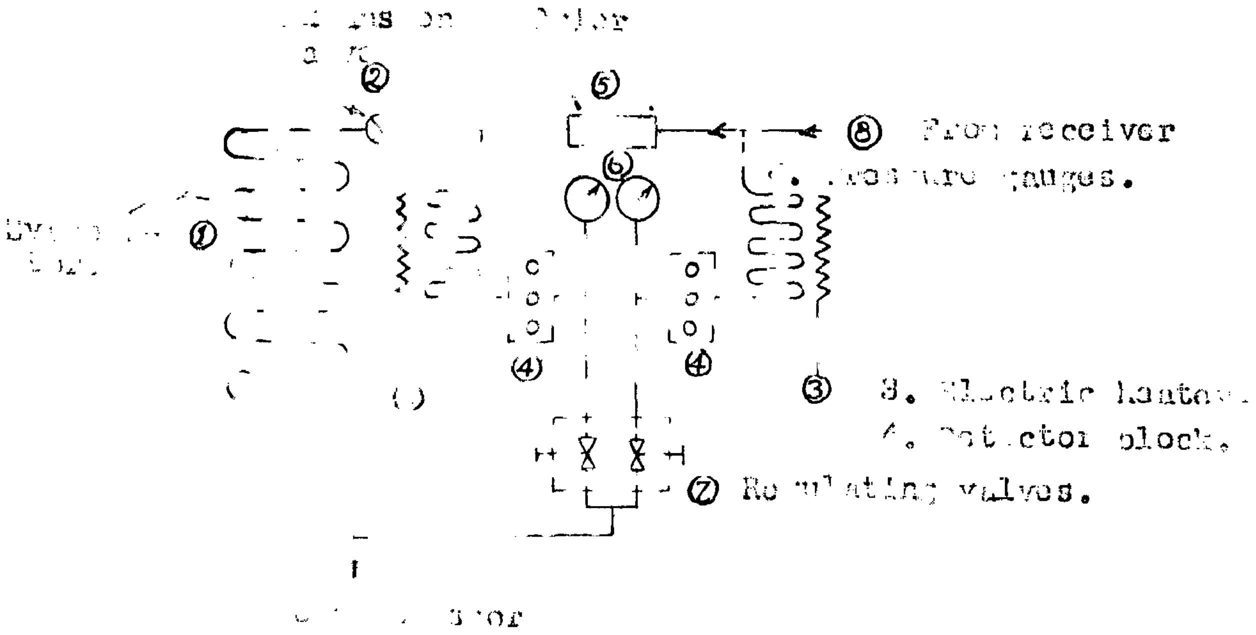


Fig. 14. Apparatus employed for checking dew point of propane

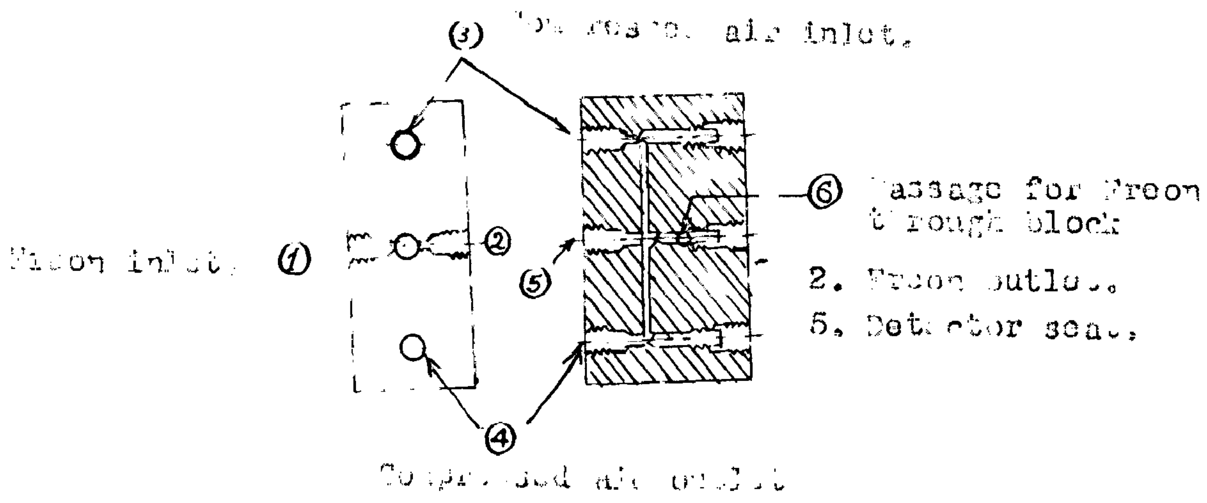


[illegible] relation between [illegible] content of [illegible]



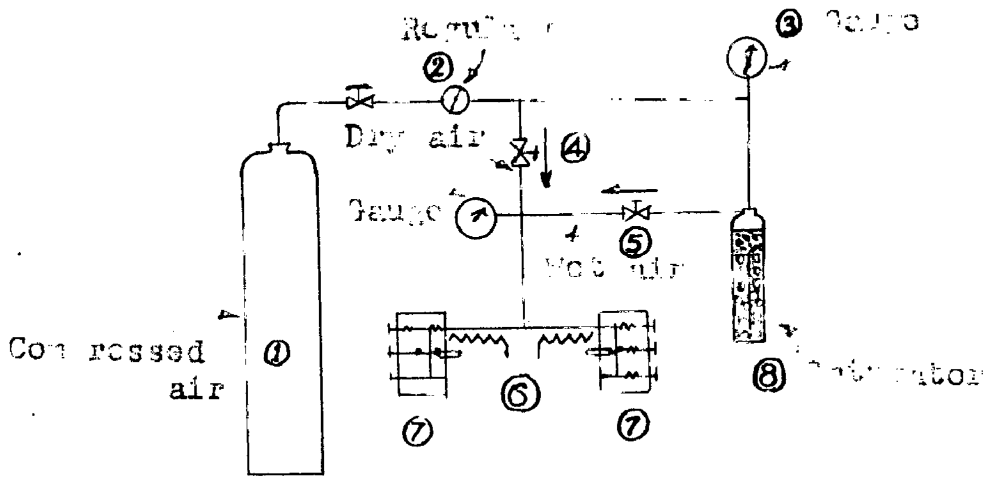
[illegible] diagram showing [illegible] the vaporizing syste[illegible] [illegible] [illegible] constant [illegible] Freon-12 circulation, in [illegible]

Ref. 19



[illegible] The Detector-Block.

Ref. 19



6. Electric heaters.
7. Detector block.

Fig. 18. Method of making gas connections.

Ref. 1[illegible]

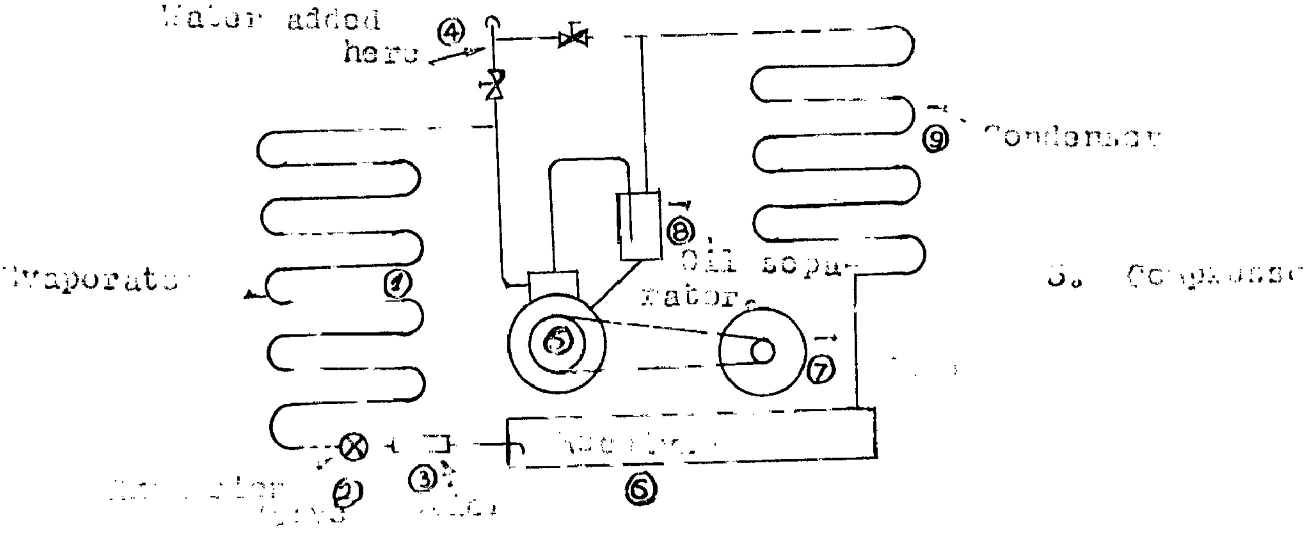


Fig. [illegible] Sketch of Refrigeration system showing [illegible] by-pass for introducing water.

[illegible]

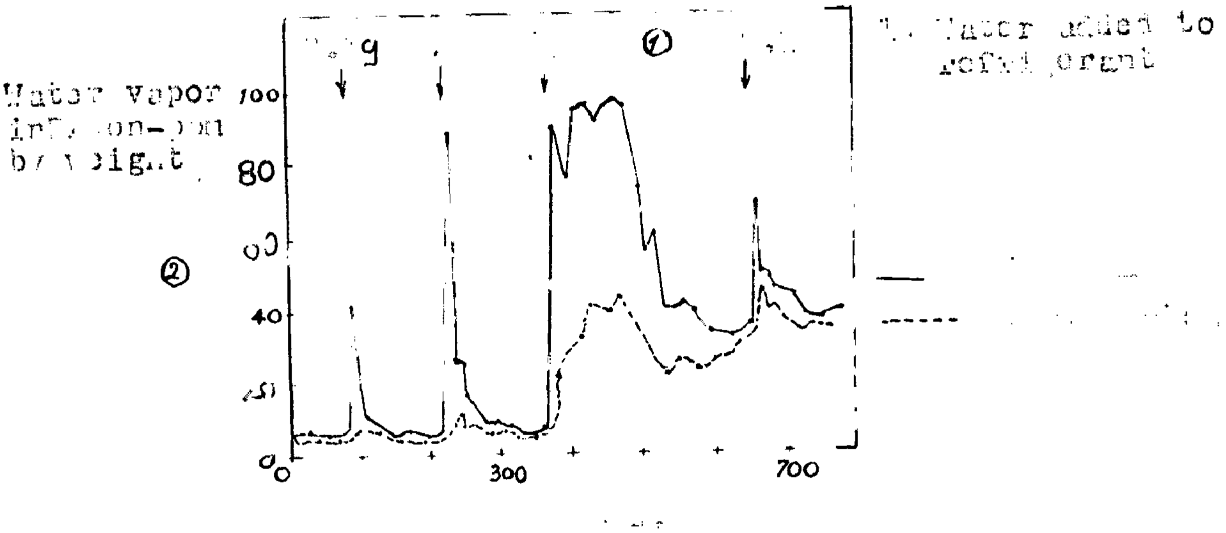


Fig. 20. Drying curves.

Ref. 19

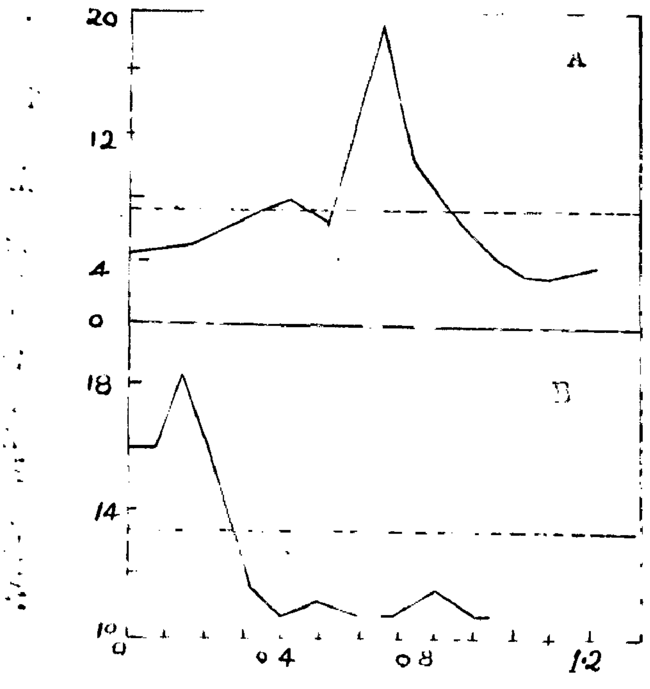


Fig. 21. [illegible] to determine [illegible]
[illegible]

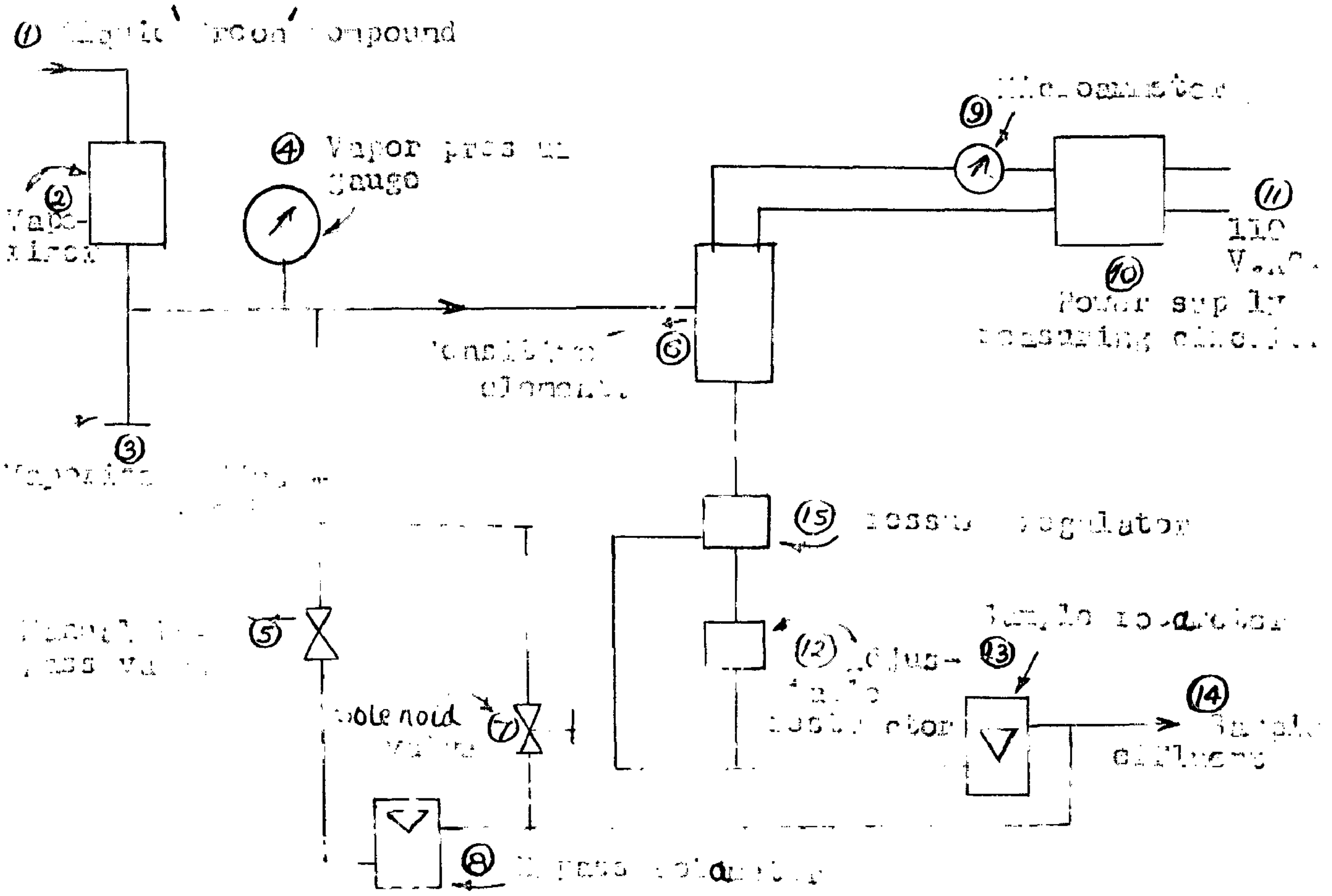
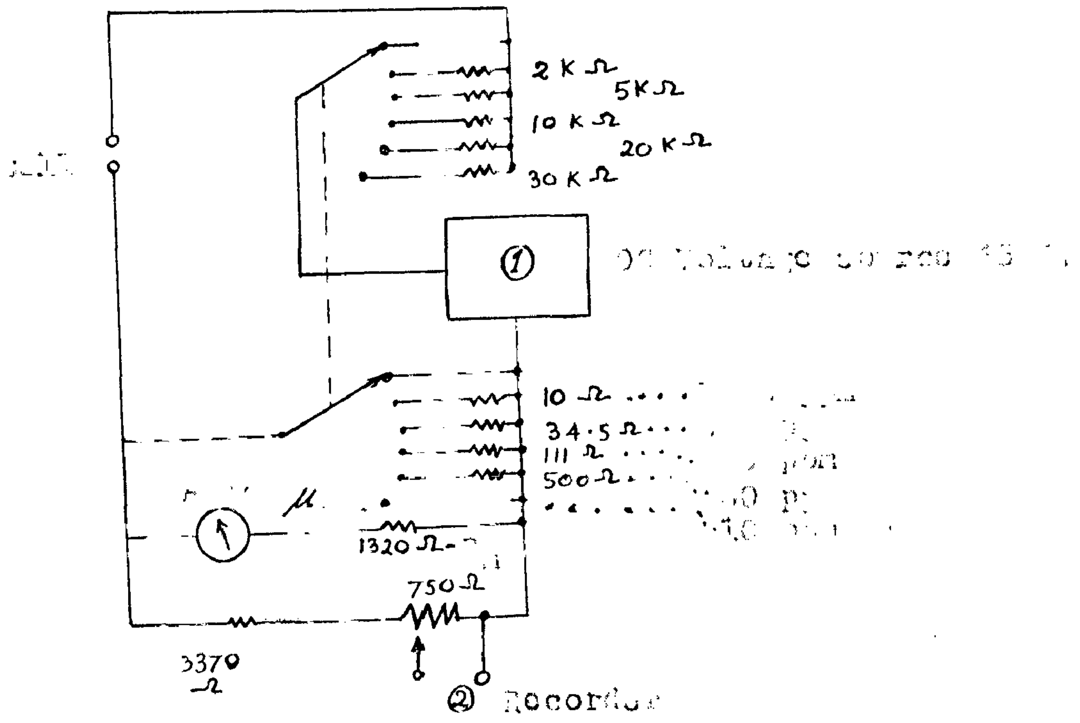


Fig.29. Schematic diagram of the analyzer and its sampling system

Ref. 30



[illegible] .23 Electric circuit of the [illegible]

Ref. 20

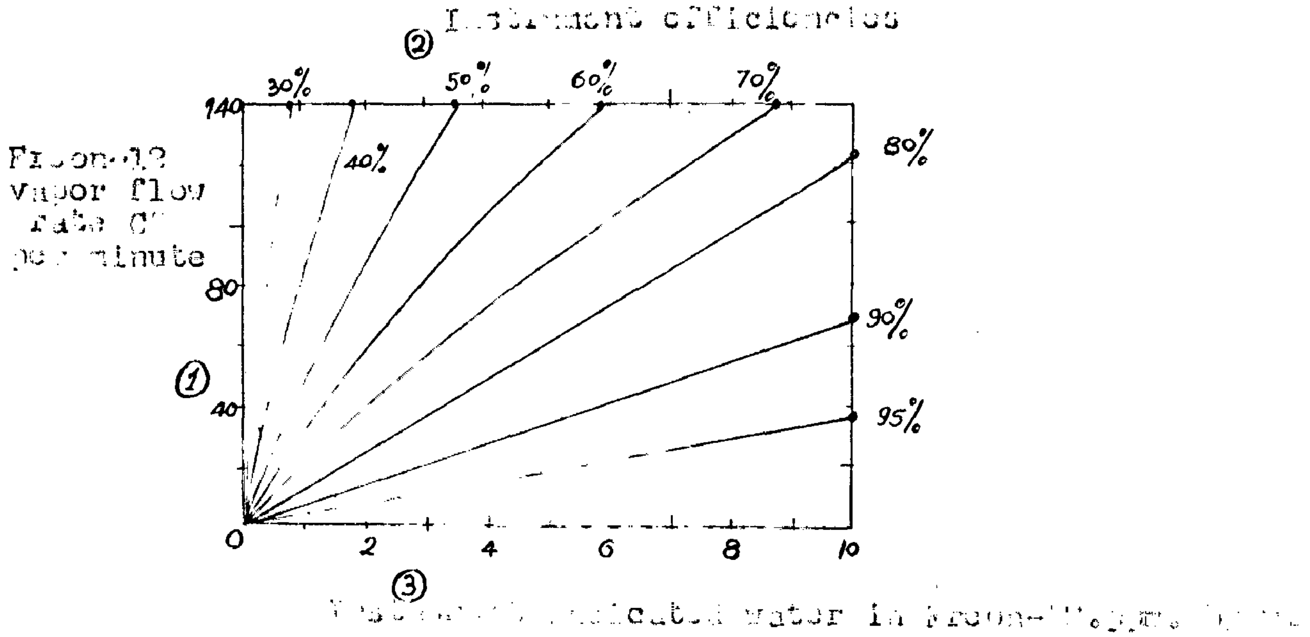


Fig. 24. General nomogram [illegible] analysis of water in Freon-12.

Ref. 20

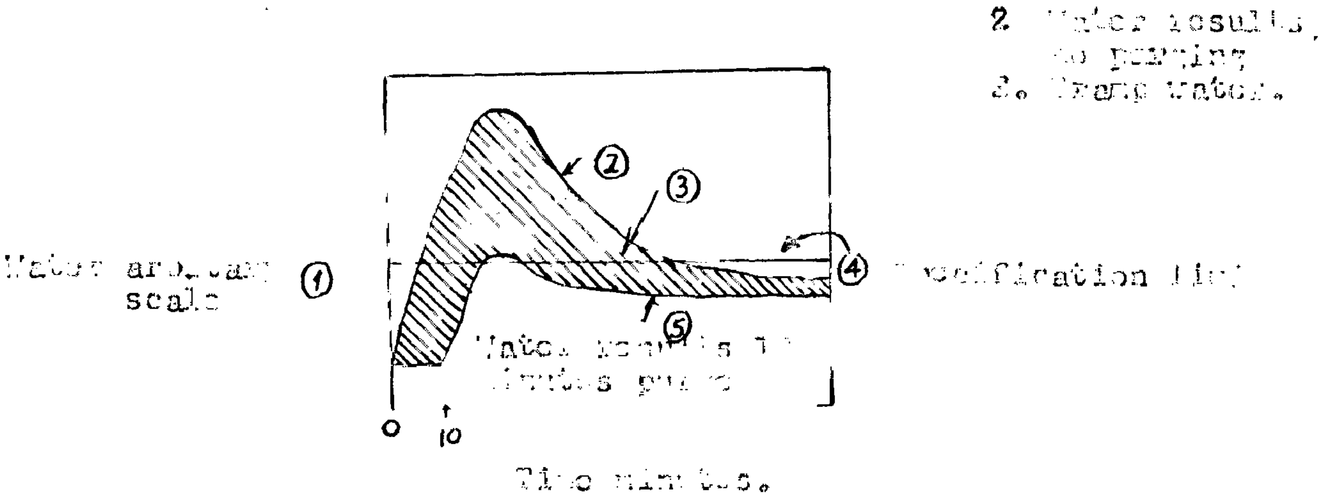
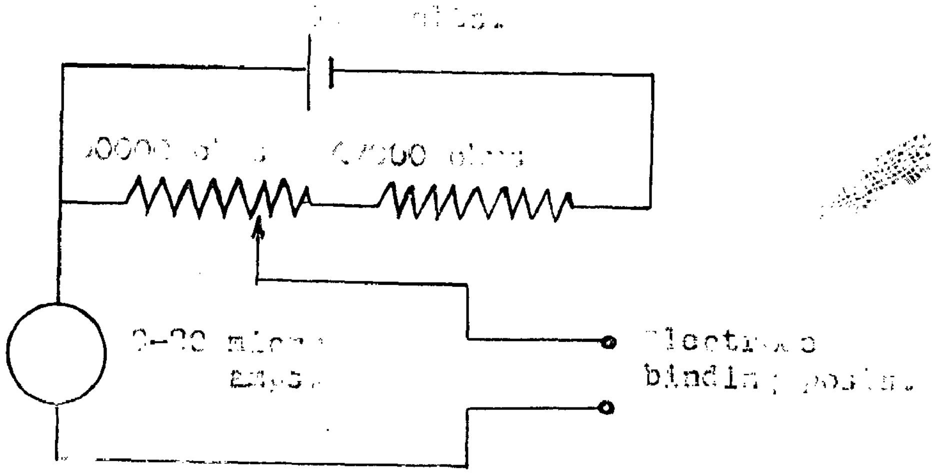


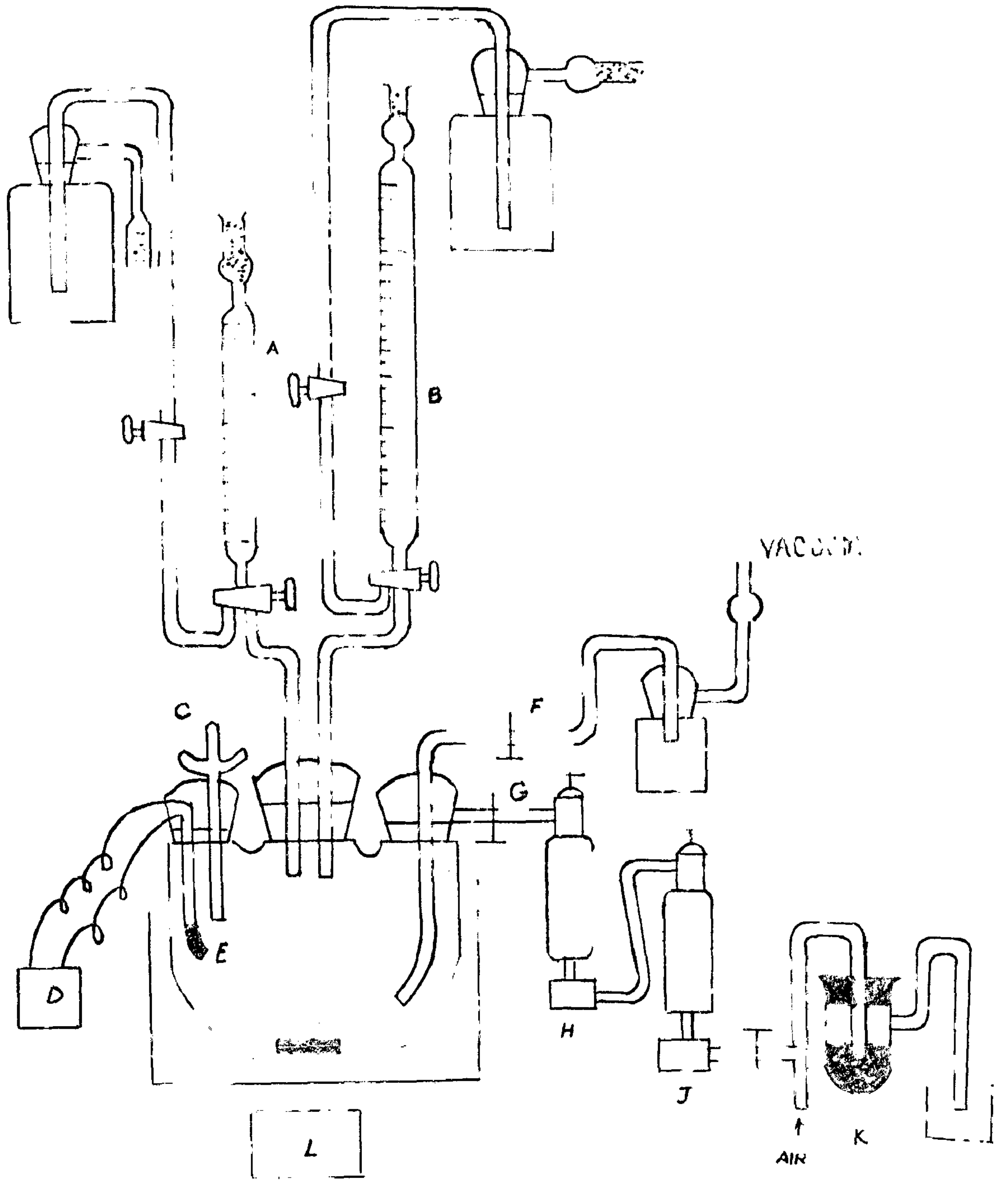
Fig.20. [illegible] of trench water - [illegible]

[illegible]

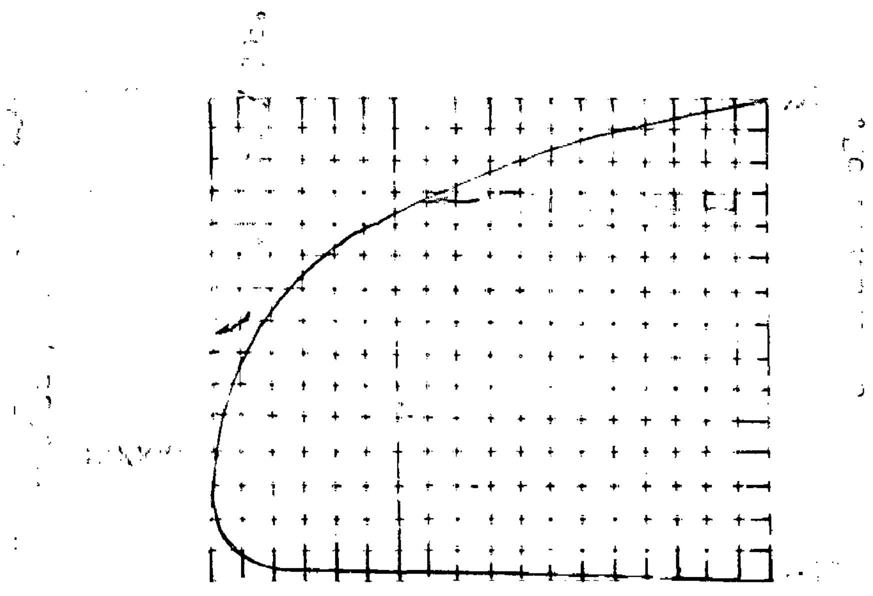


[illegible] [illegible] ical diagram [illegible]

[illegible]

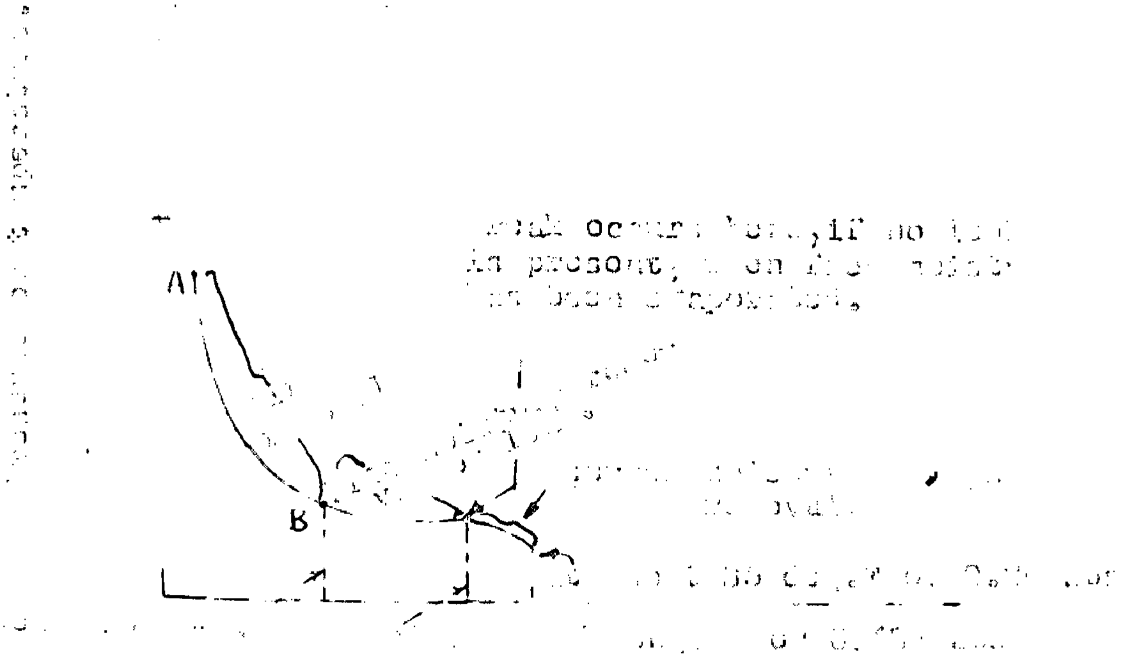

A
B
C
D
E
F
G
H
J
K
L
VACUUM
AIR

[illegible]



[illegible] of

Ref [illegible]



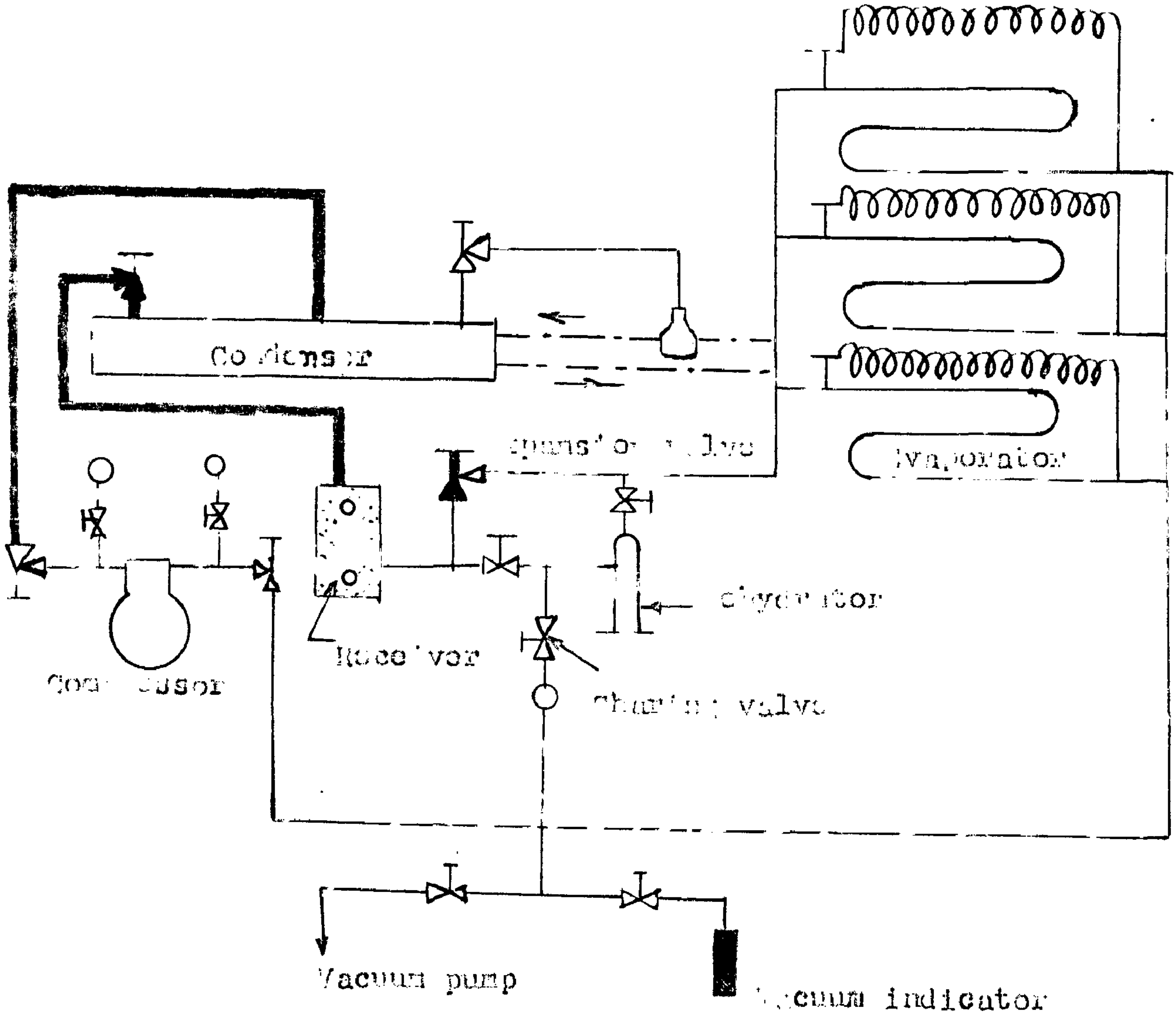
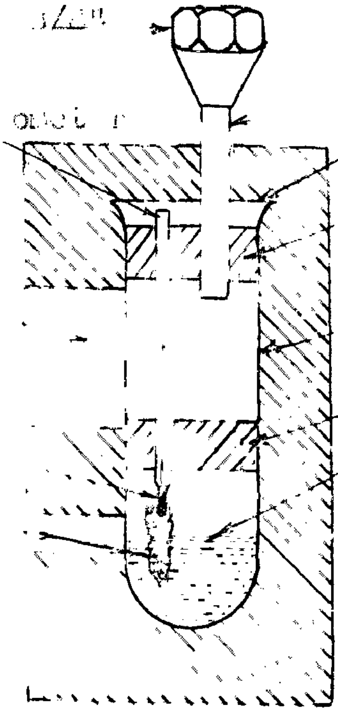
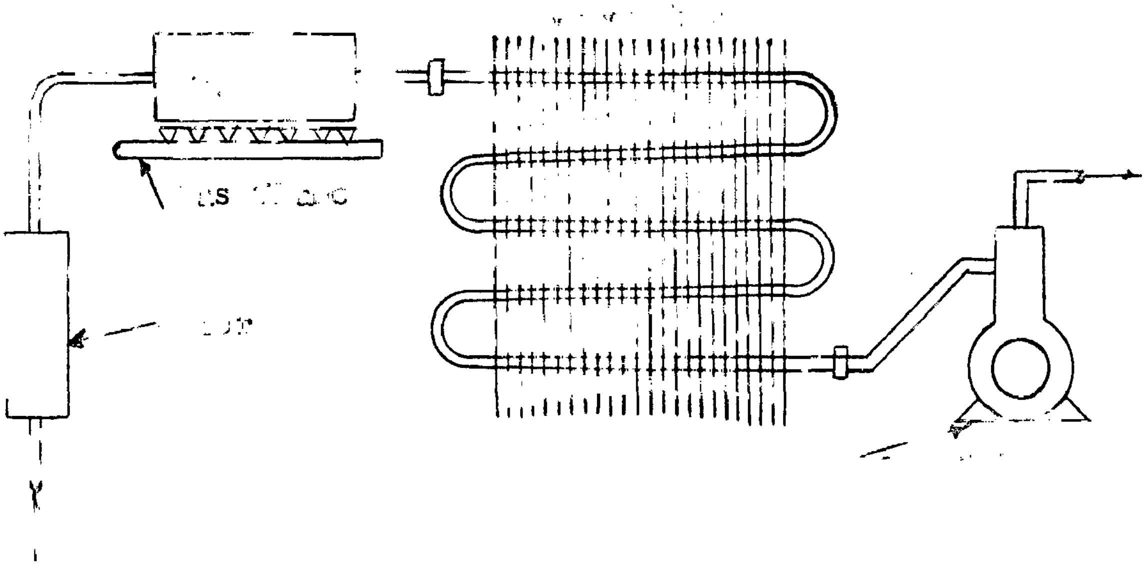


Fig.30. Vacuum pump and indicator are hooked into charging line. If system has been flooded from Condenser leak, one has to break low points in lines, drain out water.

Ref. 24


Wick

[illegible]



[illegible]

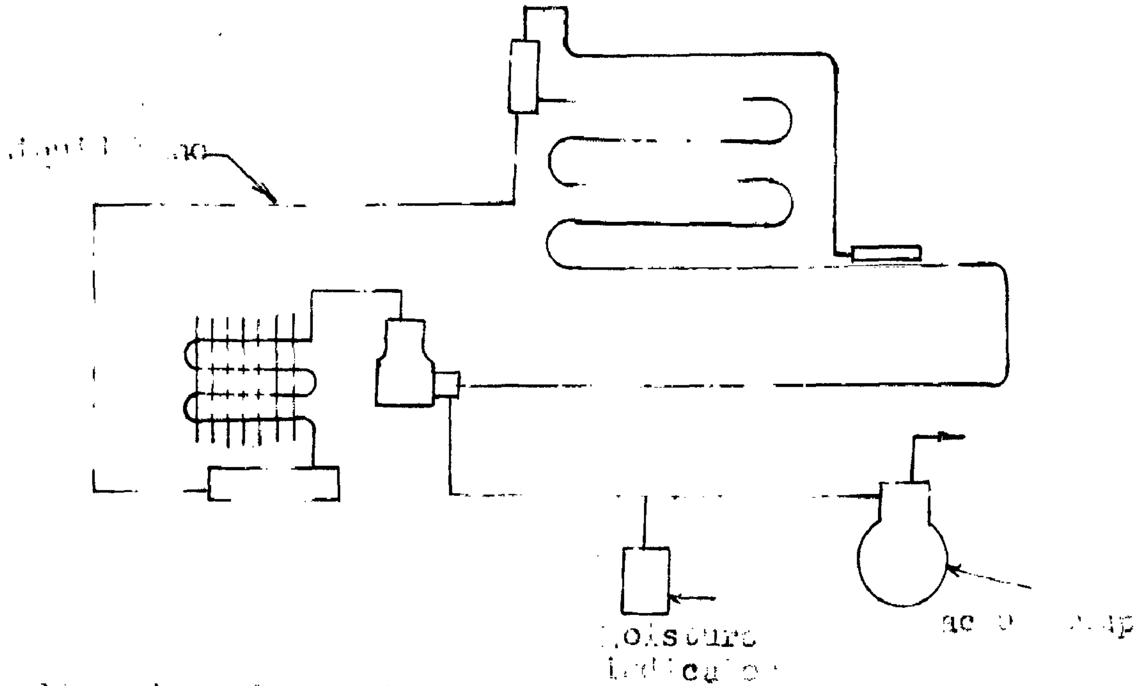


Fig. 33. Set up for employing the double evacuation method of moisture removal.

[illegible]

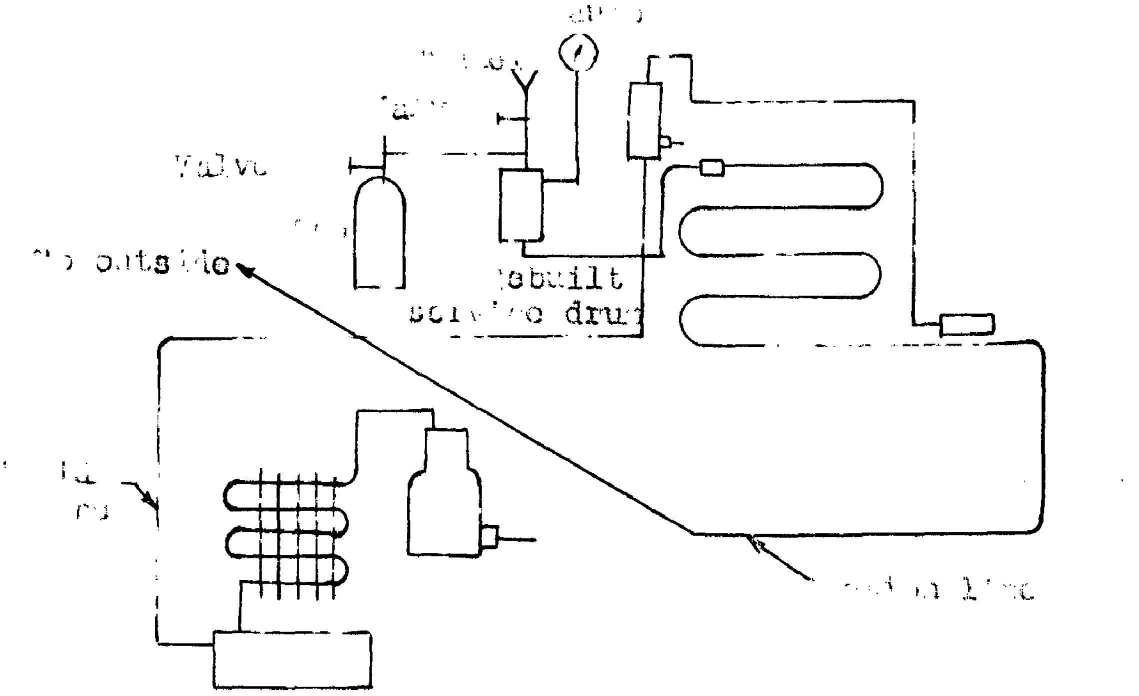
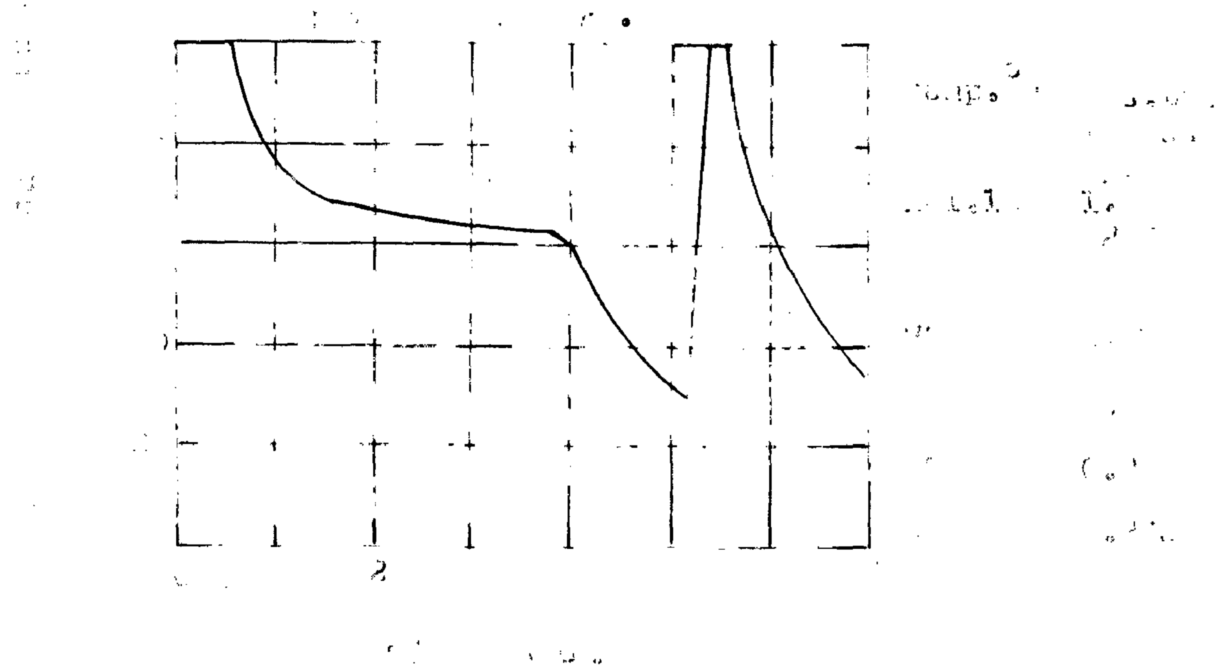


Fig. 34. Carbon dioxide and alcohol method for removing moisture from refrigeration system.



[illegible]

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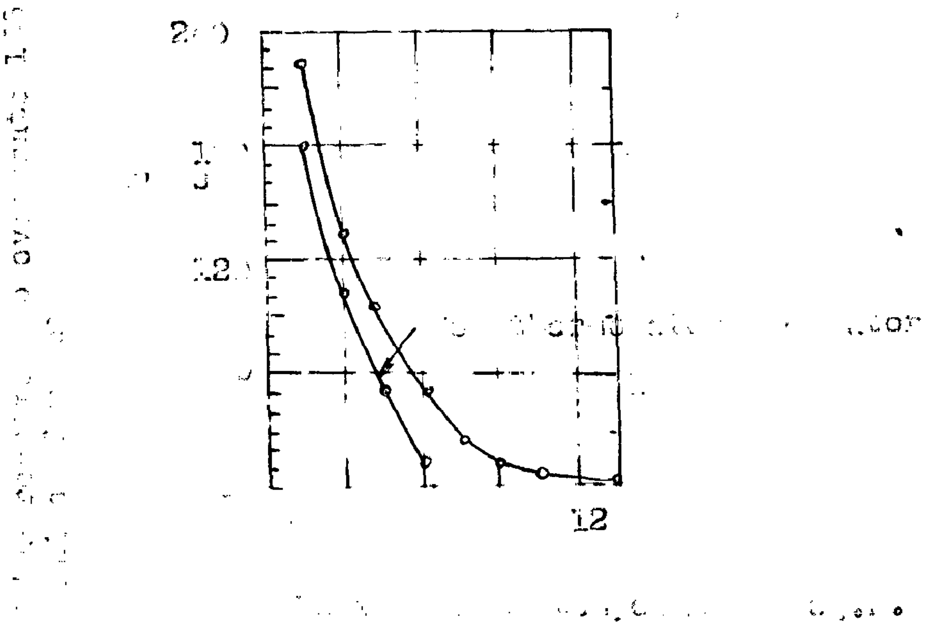
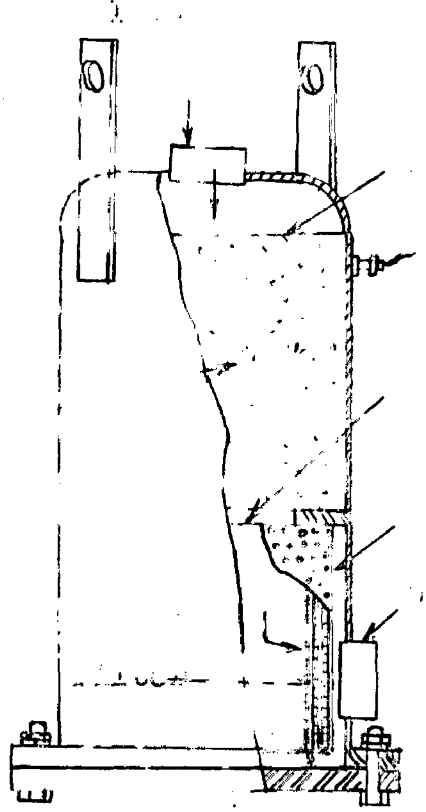


[illegible] 36. [illegible]

[illegible]



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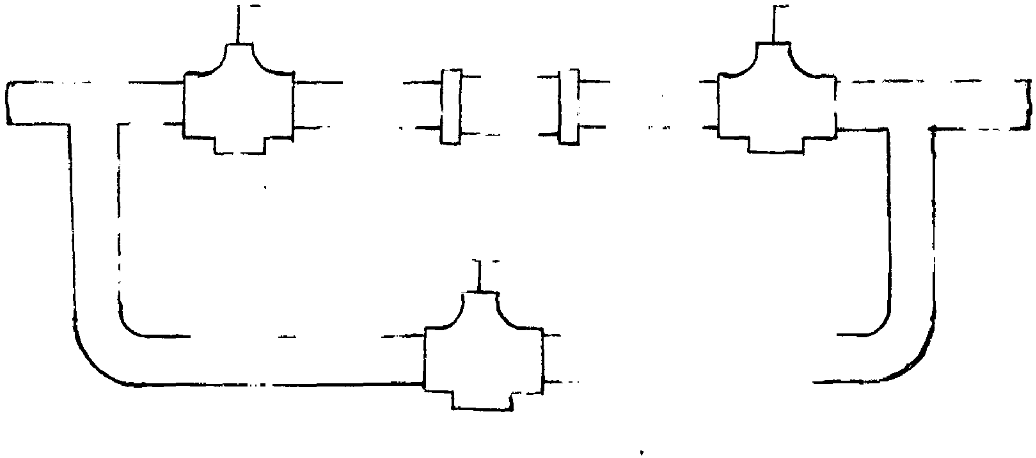
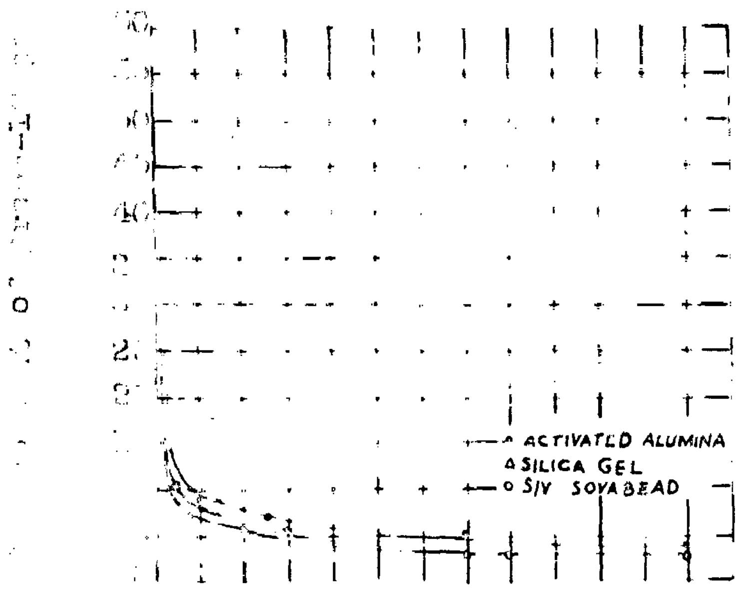


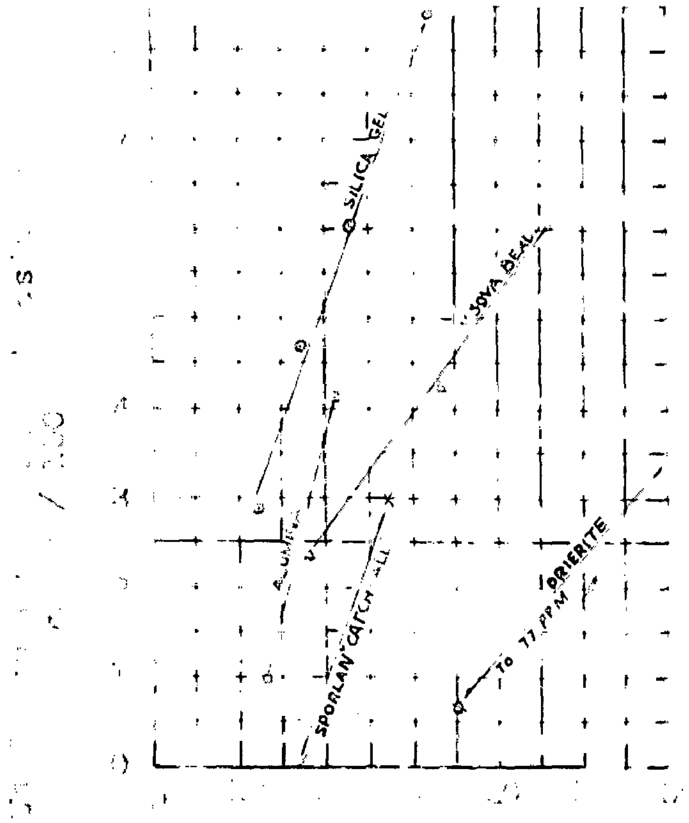
[illegible]

[illegible] shut [illegible] valve
[illegible]

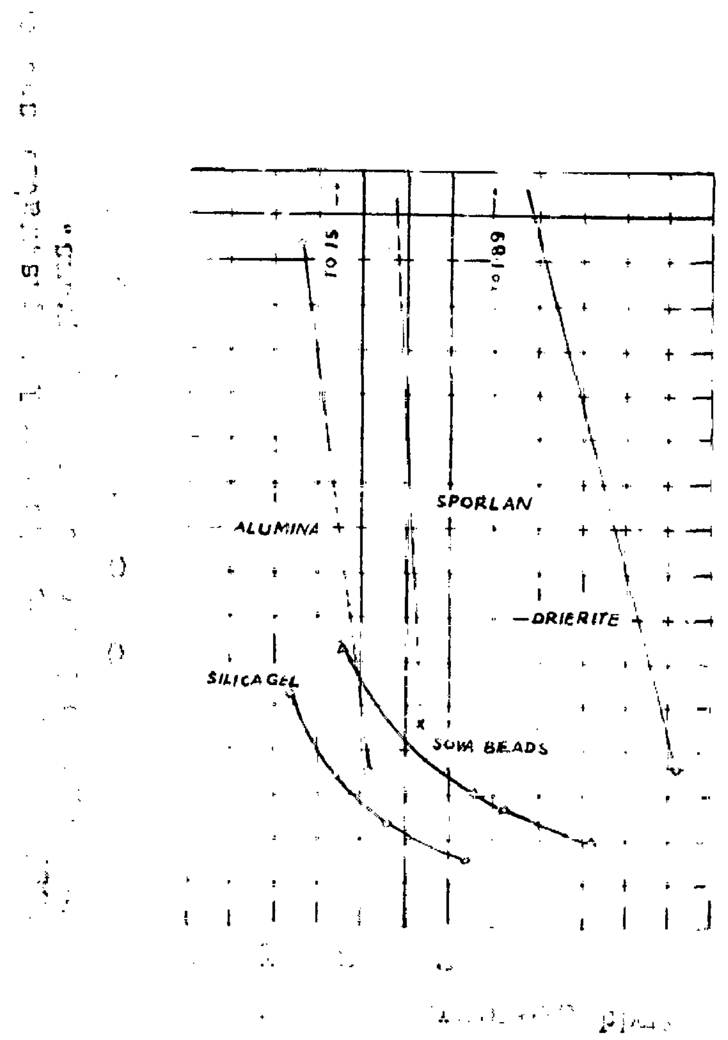

ACTIVATED ALUMINA
SILICA GEL
S/V SOYABEAD

26

[illegible]

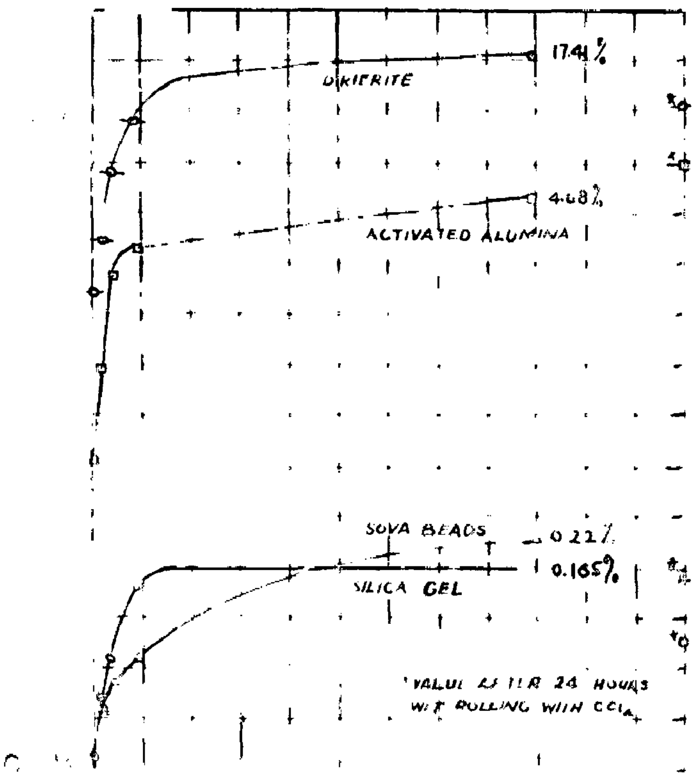

SILICA GEL
SOYA BEAD
ALUMINA
SPORLAN CATCH ALL
DRIERITE
TO 77 PPM

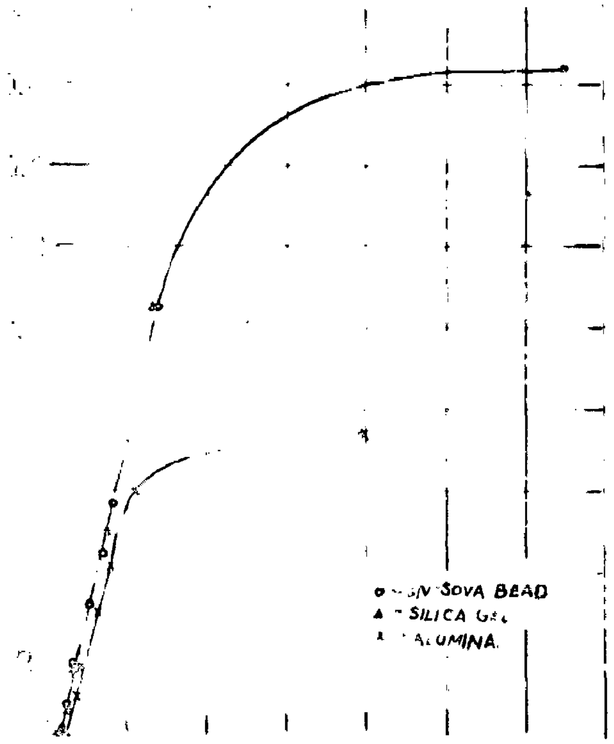
[illegible]

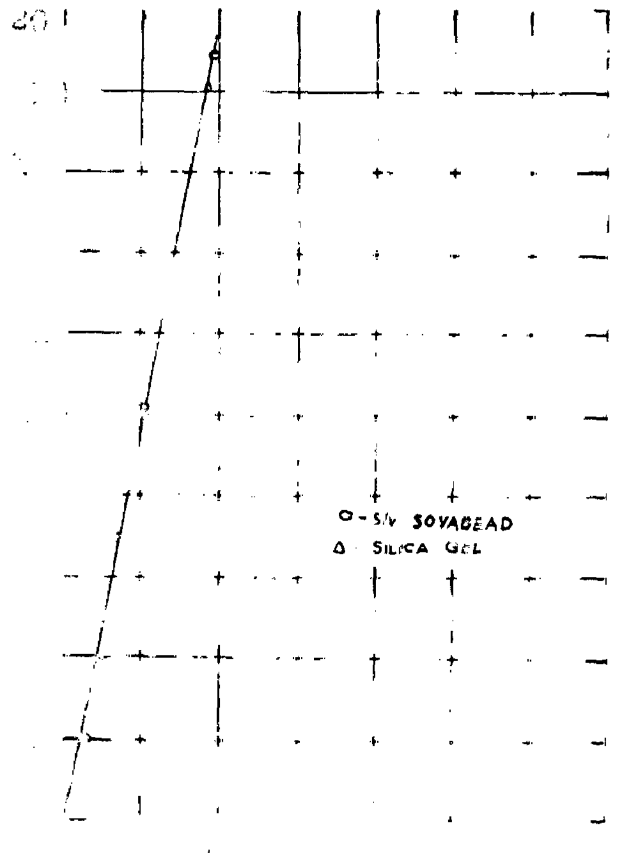

TO 15
TO 189
SPORLAN
ALUMINA
DRIERITE
SILICAGEL
SOVA BEADS

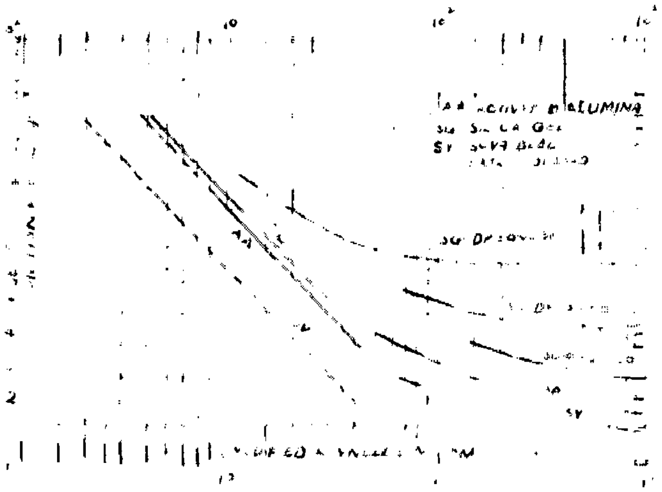
[illegible]

[illegible]


17.41%
DRIERITE
4.68%
ACTIVATED ALUMINA
SOVA BEADS
0.22%
0.165%
SILICA GEL
*VALUE AFTER 24 HOURS WITH ROLLING WITH CCl4


o - SOVA BEAD
▲ - SILICA GEL
x - ALUMINA.


40
O – S/V SOVABEAD
Δ – SILICA GEL


10
AA ACTIVE ALUMINA
SG SILICA GEL
SV SOVABEAD
AA
SG
SV
4
2

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REFERENCES

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