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UNIDO/FAI Interregional Meeting on Safety in the
Design and Operation of Ammonia Plants

New Delhi, India
10-12 January 1973

SAFETY CONSIDERATION IN THE OPERATION
AND MAINTENANCE OF AMMONIA PLANTS -

by

P.M. Das*

* Deputy Chief Engineer, The Fertilizer Corporation of India Ltd., Trombay Unit, India

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SUMMARY
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New Delhi, India
20 - 24 January 1976

SAFETY CONSIDERATION IN THE OPERATION
AND MAINTENANCE OF AMMONIA PLANTS 1/

SUMMARY

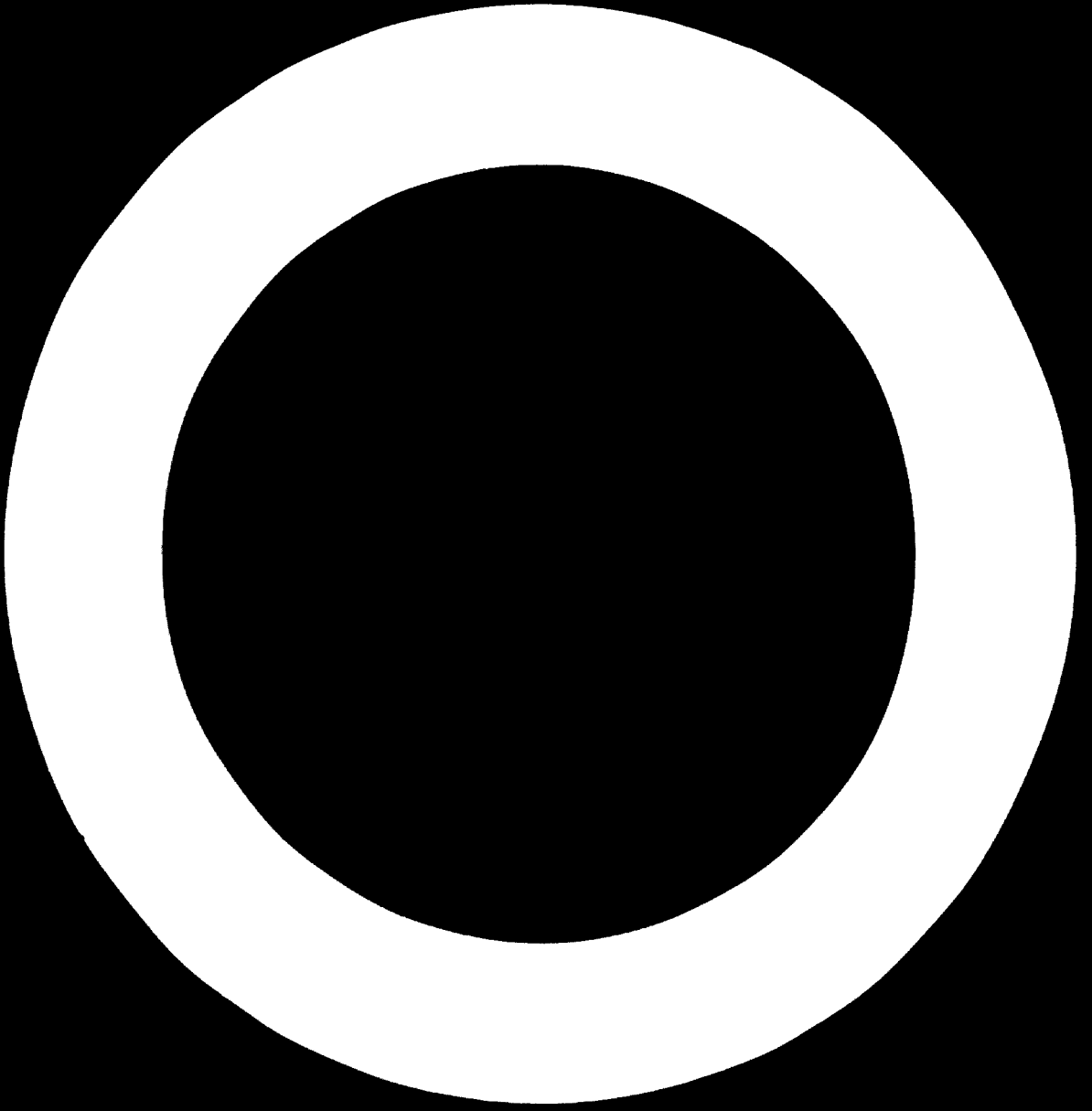
by

T.M. Das*

1.0 Over the past decade or two, there has been a phenomenal increase in Ammonia production. Plant capacities of 1500 M.T./day and above, at one time considered gigantic and utopic, have become a reality. With the increase in capacities, there is an attempt to squeeze all the energy possible out of waste process heat for increasing thermal efficiencies. These have led to intricate plant lay outs with increase in sizes of equipment, pipelines, bends and elbows which in turn have increased the vulnerability of plant to failures and thus affecting safety to both equipment and personnel. Hence the need for safety.

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1.1 Safety in operation and maintenance is normally confused with maintaining continuity of production at the cost of safety to men, material and machines. Certainly they are inseparable. If safety in operation and maintenance of plants is attained, production follows, production continuity is assured and persons, life of men, machines and material will improve. In our paper we have made an attempt to list out some of the major safety hazards inherent in any ammonia plant in its operation and maintenance. An attempt is also made to list out some of the safe practices that will go along way in overcoming these hazardous situations. Stress has been laid particularly on operation and maintenance of Air liquefaction plants in O₂ purification processes, handling of catalysts, their safety, reduction of corrosion and erosion, their control in various situations, predictive and preventive maintenance procedures, safety of plant and personnel. Various personnel protective devices have also been dealt with.

1.2 To date there is no viable wire process that can produce O₂ on commercial scale in hundreds of tonnes/day as required in any ammonia plant. All processes are based on low temperatures. Various hazards due to presence of hydrocarbons, nitrogen oxides entering with process air, their avoidance and necessary action when these are present have been dealt with. Handling cryogenic liquids and other various operational and maintenance problems with due regard to safety have been discussed.

1.3 Various catalysts used in ammonia plants constitute 2 to 3% of total investment. But their performance and life is more important than their cost, due to long downtime required for replacement in case of inactivity or deterioration in performance.

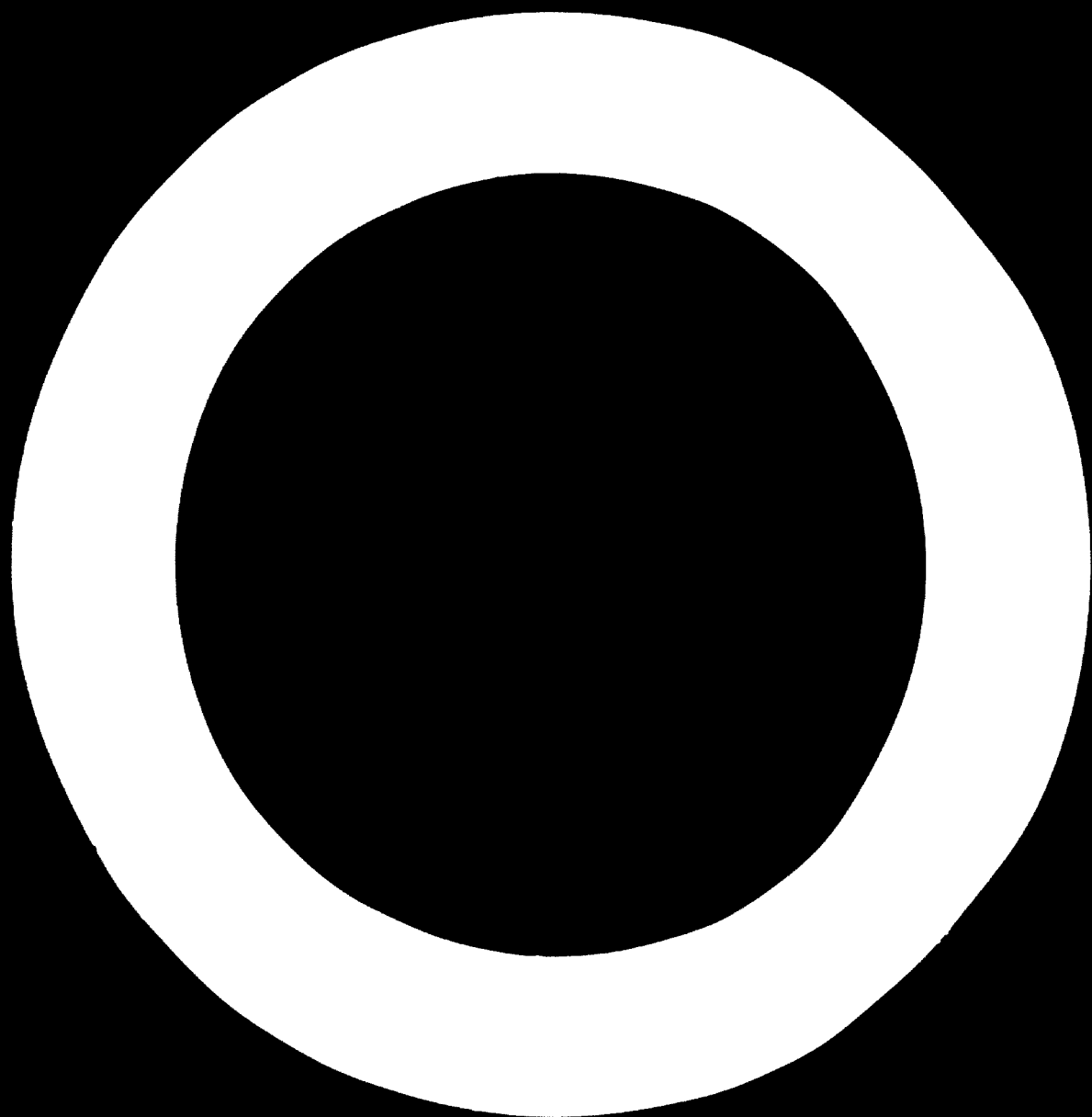
1.4 Various typical corrosion and erosion phenomena encountered and their controls or remedial measures have also been enumerated as far as possible. Despite of retarded innovations and advances in technology, corrosion and erosion do occur and hence the necessity for their control.

1.5 Ammonia plants produce hydro carbons in solid, liquid and gaseous phases and each processing step involves handling of toxic and explosive gases and liquids in a wide range of temperatures and pressures. Pressures from near atmospheric to hundreds of atmospheres and temperatures from -200°C to 1500°C are very common. Process flows vary from several M^3 to thousands of M^3 depending on plant capacities. All these necessitate fine automatic control of all the above variables lest they lead to safety hazards. Human hands and their skill are not adequate to this type of operational control. Moreover, the operations will be time consuming. Hence the need for automatic control. This is achieved by transmitting all the important critical process variables like pressure, temperature, flows, levels, gas composition etc. to a centrally located control room where suitable action can be initiated immediately. Alarms are also provided along with suitable safety shut down devices to avoid human slips, machine and equipment failures. Suitable time allowance is also made in settings of alarms and safety devices to overcome inherent time lag of monitoring instruments and control circuits between sensing the abnormality and the effect of corrective action to be taken. This is the essence of fail safe instrumentation in ammonia plants and is as important for operation from safety point of view as any other conceivable factor.

1.5 The... handling... of...
with...
talent...
devices...
also have...
their...
1.6

1.6 Various... mainly
due to failure... critical areas
in plant and production... alternate arrange-
ments for power... areas have been dis-
cussed and dealt with.

1.8 In spite of all the... and technological advances,
it is not uncommon for... to occur. The
undoubtedly follows... of the
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asserted that... may
a potential danger by...
Competence and... in the
operation of... lead
upon themselves. It is...
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I N T R O D U C T I O N

Depending on the type of Ammonia Plant, operation and maintenance considerations for safety of the plant also differ. But, there are certain factors with regard to safety which are common to all plants.

Safety is from my understanding is simple and straightforward - the protection of human life, their limbs and health. True safety is sometimes confused with the protection of continuity of production and equipment of machine performance. Certainly they are interlinked, because failure of equipments lead to safety hazards. Fundamental difference between the consideration of safety and the consideration of continuity of production and equipment performance is, that with regard to safety we don't gamble, nor do we take calculated risks, even though at times these considerations may be at odd with economic considerations.

If safety is attained, production follows, and production continuity is attained, equipment performances will improve. Both are complementary to each other.

Over the past decade there has been a phenomenal growth of Ammonia production. Plant capacities, gigantic and stupendous at which people used to scoff at a decade ago have become very much a reality. Ammonia tankers are crossing the mythical seven seas carrying ammonia from country to country from coal pit heads or from oil/gas producing centres to consuming areas. Capacities of 1900 MT/day and above have already become a reality particularly in West. In India too we have plants already under advance stages

of completion with capacities over and above 1000 MT/day. With these increasing capacities, there is an attempt to squeeze all the energy possible out of steam and waste process heat in order to increase the onstream thermal efficiencies. These have led to complicated Ammonia Plant layouts, which have in turn increased the vulnerability of plant to failures and hazards and thus affecting safety to both equipment and personnel. When plant lay out is complicated, there are increasing number of bends and elbows. With large capacities piping and equipment get larger, wall thicknesses increase and the net effect is, the whole system get less and less flexible. It is not the pressure and temperature they hold that counts but it is the stresses developed. The larger the equipments, lesser is their flexibility and they are more prone to failures and in turn safety suffers.

Ammonia Process Technology:

From a technological angle, the production of Ammonia from any hydrocarbon feed stock essentially consists of the following operations:

Production of raw gas from feed stock by inter action either with steam and air or steam and oxygen (either high purity O_2 or enriched air); the aim here is to get maximum yields of $(CO+H_2)$.

Using carbon monoxide in this mixture to react with steam in the presence of a catalyst, to yield additional quantities of hydrogen.

Isolation of H_2 (along with N_2 if present in raw gas) from the mixture by removing carbon dioxide by absorption/desorption processes.

Eliminating last traces of carbon oxides and sulfur compounds or converting these objectionable impurities to acceptable impurities (i.e. Methanation).

Correcting the composition of mixture of H_2 & N_2 to a proportion of 3:1.

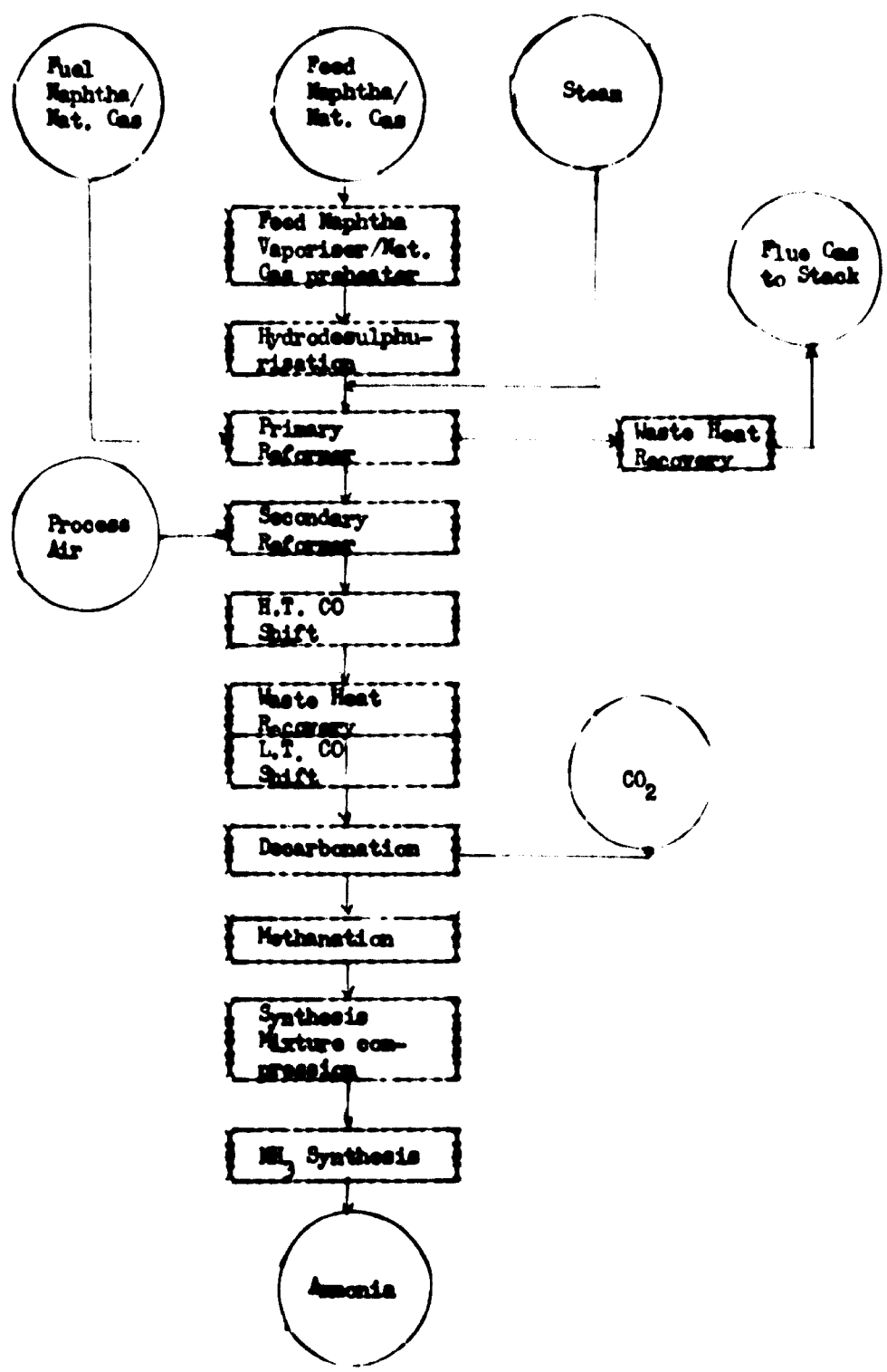
Finally catalytic synthesis of ammonia from the H_2 & N_2 mixture.

Various factors influence the exact process sequence. First and foremost is the hydrocarbon feed stock. Depending on the feed stock two distinct gasification routes have been well established. They are:

1. Steam Reforming
2. Partial oxidation or oxygen gasification.

1) Steam Reforming:-

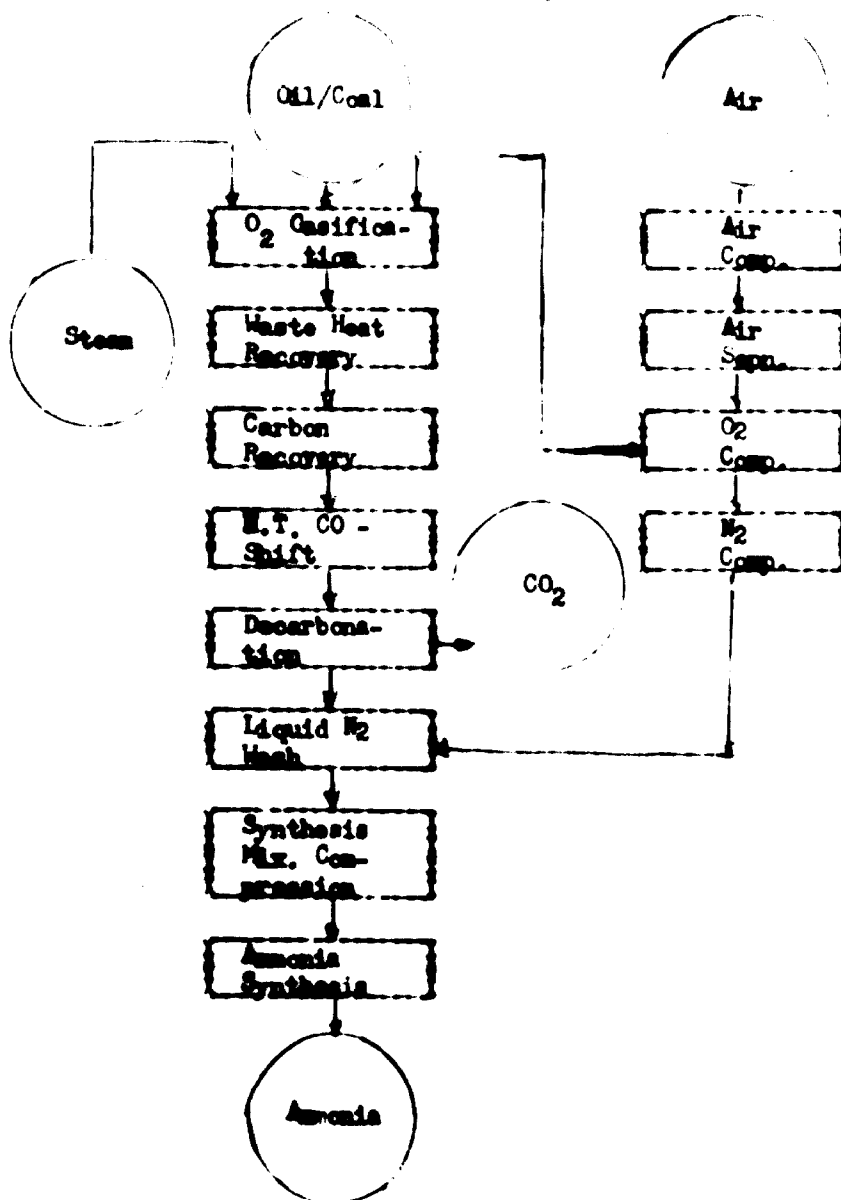
This is more amenable to lighter feedstocks like natural gas and straight run naphtha. The processing sequence is as follows:-



2. O₂ Gasification:

This is best suited to hydrocarbons having higher C/H ratio or which are heavy. This is the only alternative for Gasification of these feed stocks, since these heavy hydrocarbons may crack and form lot of carbon due to heavy coking which will reduce the catalyst activity in the case of steam reforming and also the problem of hydro-desulphurisation of feed stock.

The process steps and sequential arrangement is as follows:



Partial oxidation techniques for gasification of heavy liquids hydrocarbons are well established, as evidenced by Shell & Texaco processes. There is considerable interest in this area towards increasing pressures. Pressures upto 85 kg/cm²g are a reality, gasification pressures upto 160 kg/cm²g are in the pilot plant stage and have every chance of succeeding in the near future. Another area is the avoidance of pure O₂ and use of air/enriched air.

In the field of coal gasification two German firms Lurgi, Koppers & Totzek, have developed processes. Whereas Koppers & Totzek is a gasification at atmospheric pressure, pressure in the Lurgi process is as high as 25.0 kg/cm²g.

Safety Hazards, Personnel Safety & Personnel Protective
Equipments for Personnel Safety in Ammonia Plant,

We have listed down some of the major safety hazards inherent in any Ammonia Plant.

1. Handling of hydrocarbon feed stocks which are highly inflammable.
2. Handling of toxic and inflammable gases at high temperatures and pressures - In many parts of the Ammonia Plant these confined gases are well above their ignition temperatures, which means if mixed with O_2 or air immediate fire or explosion results.
3. Also these inflammable gases are frequently confined in large vessels which contains large quantity of stored energy. A failure or rupture can result in considerable damage and loss of life.
4. Another hazard to safety which is well known and is a subject of long drawn out discussions and studies is corrosion and erosion of pipes and equipments. The results of these two hazards are well known.
5. Metal or material deterioration - This occurs in one form or another, from one end to the other end of an Ammonia Plant. Such phrases as a sigma phase, carbon precipitation, nitriding hydrogen attack, caustic embrittlement, vibration fatigue and low temperature effects are all too familiar.
6. The large volume storages of liquid ammonia is also a potential safety hazards.
7. Handling of catalysts and chemicals which are recurring, events in any ammonia plant is another hazardous area.

8. Another hazard which affects the surrounding community at large in addition to working personnel is the plant effluents let off to the ground and atmosphere. This is most significant when plants are situated near thickly populated urban and metropolitan centres.

9. Higher noise levels are another hazardous area to personnel safety. The grumbling and screeching sounds of compressors and pumps are of much higher frequency than human eardrums can tolerate. Moreover it impedes proper communication.

10. Last but not least are of course human failures, such as ignorance, lack of training, laziness, overwork, poor housekeeping, lack of co-ordination and disregard to safety codes and practices.

All these are part and parcel of the Arconia Plant irrespective of their size and shape. We can only try to minimise these hazards to a great extent, with the technological advances in material and process technology and implementation with fail safe operation and maintenance and strict adherence to safety practices. But human errors, failures and slips will always be with us. The experience of several operating plants is a wealth of knowledge to avoid some of the unknown hazardous areas.

2. Personal Safety In Ammonia Plants:

Great personnel hazards exist in the handling of chemicals and gases in Ammonia Plants. In all cases affected individual should be given effective first aid and prompt medical attention. The following outlines will give the effect of the gases and chemicals that we come across in NH_3 plant.

1. H_2 : As such it is not toxic. However gaseous hydrogen in large quantities is asphyxiating.
2. Carbon Monoxide: This is highly poisonous. The gas has metallic odour and taste and its mixture with air is flammable and explosive. Exposure to CO causes severe asphyxiation. Maximum allowable concentration in air is 100 ppm. 200 ppm i.e. twice the allowable concentration can cause headache in 2 - 3 hours. 400 ppm, four times the allowable concentration will cause nausea and headache in 1 - 2 hours. 800 ppm, 8 times the toxic limit can cause headache dizziness and nausea in $\frac{1}{2}$ an hour. If exposure lasts for 2 hours collapse can occur. Exposure at higher concentration can result in collapse and death immediately. Acute poisoning sometimes causes $\frac{1}{2}$ of sight.
3. Carbon di-oxide : Maximum allowable in air is 5000 ppm. Intoxication from CO_2 causes headache, vertigo, dyspnea, drowsiness, weakness, dizziness and muscular weakness. Following the initial excitement high concentrations may result in coma & death.
4. H_2S : Maximum allowable concentration in air is 20 ppm. This material is dangerously poisonous highly inflammable, corrosive to the eyes and respiratory tissues and tract, and at increasing concentrations more and more harmful and can cause immediate death.

5. NH_3 : Anhydrous NH_3 can cause irritation to the skin, mucous membranes and respiratory organs with possible fatal results. Liquid ammonia can cause severe burns on contact with skin or delicate tissues. Physiological response to various concentrations of ammonia in air are as follows:

- a) Maximum detectable odour 53 ppm
- b) Maximum concentration for prolonged exposure 100 ppm
- c) Maximum concentration for short exposure 300 - 500 ppm
- d) Minimum amount causing immediate irritation to the throat 408 ppm
- e) Minimum amount causing immediate irritation to the eyes 698 ppm
- f) Minimum amount causing coughing 1720 ppm
- g) Dangerous for even short exposure 2500 ppm
- h) Rapidly fatal for even short exposure 5000 - 10000 ppm

Chemicals:

MEA, K_2CO_3 and Caustic : Because of their strong alkaline nature, are injurious to eyes and skin. Any contact with eyes, to any part of body or clothing should therefore be avoided. In case of contact immediately flush skin or eyes or affected part with plenty of water for at least 15 minutes. If eyes are affected immediate medical attention to be obtained.

These have low oral toxicity salt water should be taken to induce vomiting if consumed.

Personnel should not be allowed to enter these vessels contaminated with above chemicals unless these towers are thoroughly flushed with water.

The other chemicals like activators and inhibitors are also injurious to body, skin and eyes. DEA is one such. Arsenic used as inhibitor is highly poisonous and fatal if goes into the body. In cases like this salt water is to be given to induce vomiting and then doctor is to be called. Methanol used in Rectisol process of acid gas removal.

Noise is a hazard:

Human ears are sensitive to frequencies between 20 and 10,000 cycle/sec. But the physical and psychological response to different frequencies vary, the higher audible frequencies, being the more damaging ones. There is no universally agreed upon standard regarding noise levels. But there are noise control safety orders in various states. If the limits are exceed for the specified durations the use of ear protectors is recommended.

The large capacity compressors and pumps and Ammonia Plant are the main culprits as regard to noise levels. Their grumbling and screeching sounds hammer upon human eardrums like sledge hammers. It is always essential to maintain these machines in top working condition, so that noise levels will be minimum. Vibration analysis checks on rotating and moving machines are to be carried out regularly and any corrective measures required should be effected at the earliest opportunity.

Noise levels will be more during start up operations, due to venting of gas or air etc. at downstream of machines and equipments. Normal practice is to provide vent silencers at the venting point.

Higher noise levels are detrimental with regard to communication during operation which if not proper may lead to costly mistakes resulting in loss of life, damage to equipment plant and machine.

Noise Control : Some of the measures necessary for noise control are:

1. Safe operation and better maintenance of machines.
2. Vent silencers at suction and discharge of machines and equipments.
3. Accoustically lagged compressor bays and control rooms.
4. Accoustically lagged piping and equipment work.
5. Long stacks and vents.

3. Personal Protective Equipment for Personnel Safety.

Ammonia Plant is usually a large complex with moving machines and hot equipments. Working personnel should wear tight fit clothes, there should not be loose flaps or strings. Loose, torn or ragged garments shall not be worn near moving parts or machines. In addition shoe laces should be kept tied and shoes also should have a protective toes.

1. Head Protection:

Normally workers in an ammonia plant are exposed to falling objects, since some maintenance job or other continuously goes on. In addition there are valves and lines sometimes almost at head level. Hence operators should be made to wear hard hats or helmets. These helmets should have less than 0.4 kg. wt. and they should be made of non combustible or slow burning material. In addition they should be non-conductors of electricity. These hats should have a brime all around to protect the head, face and back of the neck.

2. Hair protection:

Hair should not be loose and falling on the sides. All persons working near moving machines should put on helmets or caps to cover their hair.

3. Eye Protection:

Suitable eye protection shall be provided for all workers performing any operation which may endanger their eyes. For example handling of caustic, M.A. Carbonate solutions and liquid ammonia. Normally goggles are supplied to workers. Workers whose vision requires the use of corrective lenses in spectacles shall be provided with goggles of one of the following types.

- a) Goggles, the protective lens of which provide the proper optical correction.
- b) Goggles which can be worn over corrective spectacles without disturbing the adjustment of the spectacles or
- c) Goggles which incorporate corrective lenses mounted inside the protective lenses.
- d) Goggle lenses shall be not less than 38 mm in height and 44.5 mm in width. If it is circular it should be of minimum 50 mm diameter and in addition there should have soft eye caps to fit tight so that splashing liquid or vapors should not enter the eyes.
- e) Goggles and shields for welders and grinders and those who are working near furnaces and boilers where their eyes are exposed to glare shall have filter lenses or windows conforming to absorption standards accepted by the competent authority.

4. Ear Protection:

Men working in intense prolonged noisy areas should wear suitable ear plugs or earmuffs. These should be cleaned daily and kept in containers.

5. Protective Suits:

Workers exposed to corrosive or harmful substances shall use liquid and gas proof protective suits and hoods.

Asbestos suits for protection where sudden fire or explosion may occur during emergency operations. This should consist of boots and also helmet.

6. Aprons:

These should not be worn near moving machinery. Usually these are for operations in plant laboratories and for operators handling corrosive liquids. For corrosive liquids such as caustic, acids etc. should be made of natural rubber or synthetic rubber. These are for body protection.

7. Hand and arm protection:

When using gloves consideration should be given to the hazards to which the wearer may be exposed. Gloves for electricians should be made of rubber. Gloves for hot surroundings and equipments should be of asbestos. Gloves for handling chemicals like caustic and carbonate should be of rubber. For other purposes just only for protection cotton gloves may be used.

8. Foot protection:

Foot wear should be rubber or specially treated plastic while handling corrosive liquids. Foot wear shall not have any nail of iron or steel or any other ferrous material since they are liable to create sparks.

9. Respiratory Protection:

In selecting respiratory equipment, the following considerations should be taken into account.

1. Process and conditions that create the exposure.
2. The chemical, physical toxic or other hazardous properties of the substance from which protection is required.
3. The nature of the duties to be performed by the persons who wear the equipment and the encumbrance or restriction of movement

in the working area and

4. The facilities to maintenance, upkeep and supervision of use.

In addition these protective equipment shall be capable of fitting various facial contours without leaking.

Various types of Respiratory equipment and their use are detailed below:-

1. Mechanical filter respirators: These are used as protection against dust and they should not be used against solvent vapours, injurious gases or in atmospheres deficient in O_2 . These filters shall be changed when breathing resistance becomes uncomfortable.

2. Chemical Cartridge Respirators and Canister type masks: These should not be worn in any confined space or in any other place that is poorly ventilated or in atmospheres deficient in O_2 . Minimum O_2 requirement should be 16%. It should not be used where more than one gas is present (for e.g. CO & H_2S). The concentration of the gas against which it is used should not be more than 2%.

While using, before and after, the following checks should be carried out. They are:

1. Check for physical wear and tear
2. Check for leaks.
3. Check for canister whether serviceable or not.
4. Check for area and concentration of gas. This should be used only in open areas.

At the first sign of doubt these canister should be replaced. Various types of canisters that are essential in ammonia plant are:

1. NH_3 Gas Mask : For NH_3 only
2. CO Gas Mask : For CO only
3. H_2S Gas Mask : For H_2S only
4. Universal Gas Mask : Any of the above gases.
5. Fresh air blower mask and hose respirators.

a) These should be used for work in dangerous atmosphere in all cases where the work is of such nature and carried out in such places that the fresh air supply can be safely maintained and (b) Shall be used for non-emergency operations in atmospheres in which the content of dangerous gas or fumes is too high for the safe use of canister or cartridge respirators. (c) This can be used in any place even it is confined or without ventilation provided the blower is kept at a safe distance in free atmosphere.

In the case of hose respirators pressure shall not exceed 21 lb/sq" absolute and there should be a filter for dust and moisture. The hose should be one inch size and shall be of non collapsible type.

Oxygen breathing apparatus or O_2 masks: These masks should be used for.

1. Workers engaged in fire fighting, rescue or repair work in atmospheres containing high concentrations of gases (i.e. more than 2%).
2. Areas deficient in O_2 (i.e. less than 16% O_2).
3. Workers whose respiratory organs must be protected and who are situated at more than 45 metres from the closest possible source of sufficiently pure air, provided however, that in such case the use of filter respirators is not permitted. Care should be taken in the usage of these masks.

(1) These should be worn only by experienced and trained persons

(2) The O_2 cylinders should be charged at a pressure not exceeding 150

atmospheres and a visible gauge which should indicate pressure continuously so that the amount of O_2 in the cylinder remaining can be ascertained.

3. These cylinders should delivery O_2 less than 2 litres/minute. Inspection and maintenance of breathing apparatus:

At intervals not exceeding one month, every breathing apparatus shall be:-

1. Examined by a competent and authorised person with regard to its general condition and with particular attention to any delicate and perishable parts and
2. Tested for leakage
3. All meters and valves on O_2 masks should be checked for operation and accuracy.

These apparatus should be under the direct supervision of a competent and authorised person.

The cartridge and cannister type respirator masks shall be cleaned and thin face pieces sterilised after each use. Face pieces and air lines or hoses should be washed with soap and water, rinsed in clear water and dried before being put away. Breathing apparatus used by one person shall not be used by another before it has been washed with soap and lukewarm water and then sterilised.

It is not sufficient to have equipments for personnel protection or for safety. It is of utmost importance to train the actual users. It is particularly important in large modern ammonia plants, for personal safety is always in danger in cases of hazards and emergencies. So there should be workers training programs in the use of these safety equipments. In addition these safety protection equipment should be located in plant in easy accessible places, in sufficient numbers. The plant personnel should be very well acquainted with the location of these equipments.

UNRELIABLE NATURE OF POWER SUPPLY AND ITS CONSEQUENCES IN AMMONIA PLANT

The main problem in India, every Ammonia Plant faces is unpredictable nature of power supply. The power situation becomes very acute particularly in summer months. We face lot of frequency and voltage dips. Frequent fluctuations of voltages are a normal feature. There are many total power failures too. Normal under voltage instantaneous releases are a normal feature of any synchronous or induction motor for heavy duty. As a result slight fluctuations are sufficient to trip these motors. Power situation has become so unpredictable, these things happen when you least expect it to happen. The net result is frequent shut down of plants. Apart from the production loss it entails many other difficulties stem in, in addition to safety of plant and personnel. Some of the common occurrences due to these emergency shut downs are:

1. Sudden development of leakages in steam & gas lines.
2. Thermal shocks to various equipments.
3. Thermal shocks to various catalysts in converters.
4. Gas & solutions backing into downstream sections due to sudden fluctuations in pressure and their consequent problem.
5. Damage to tower packings, which are made of porcelain or stone ware and are fragile and vulnerable due to frequent shocks. Added to this, there is the problem of plant lighting. Further due to tripping of cooling tower pumps cooling water supply will be discontinued so also instrument air supply. All these will give rise to potential hazards which may give rise to fatalities.

To overcome all these todays plants are becoming more and more self sustained with energy conservation. Almost all compressor drives are steam turbines. Only some critical and small motors are connected

with electric drives.

**SAFETY OF PLANT DURING POWER FAILURES AND EMERGENCY
POWER SUPPLY**

Normal practice in any ammonia plant is to instal a generator set driven by diesel engine which will take over the critical load in the event of a power failure. This critical load consists of (1) Maintaining continuity of cooling water supply to critical areas (2) Maintaining Instrument air supply (3) Maintaining ammonia storage pressures with sufficient refrigeration (4) Emergency lighting and power supply to all instruments working on electrical energy to sound alarms etc. (5) Maintain circulation in boilers.

Continuity of water supply:

In the case of a total power failure, cooling tower circulating pumps will also stop. There are areas in plant which will require continuous water supply even after the plant is shut down. For e.g. shell reactors. At the time of shut down these will be at a temperature of 1400°C at the inside. Combustor nose of these reactors is water cooled. In case the water supply fails the nose will get heated up due to heat backing to the combustor nose from blocked gases. The combustor may get damaged or it may burst. Hence it is necessary to maintain continuity of water supply in these areas. This is done by having a spare pump of small capacity which will take start and work on the emergency power supply and will supply water to critical areas.

As an alternative an overhead tank for water is provided which will take over the supply of water to critical areas in the case of failure of cooling water.

Continuity of Instrument air supply:

Normally there will be an instrument air bottle which will maintain the supply of air to instruments for at least for some time,

till complete plant is shut down in an orderly way in the event of a power failure. It is also not uncommon to have an instrument air compressor running on critical emergency power supply to maintain instrument air pressure.

Boiler circulation:

In the event of power failure a shut down of boiler circulating pump, there is always one spare pump which will be steam driven which will take over or can be put in line. It is necessary that boiler circulation should be maintained otherwise boiler tubes or coils may get damaged. This circulation should continue as long the tube and coils are hot.

Ammonia storage refrigeration:

Pressure in ammonia storages is being maintained by a continuous refrigerating system. In the event of a power failure, these storage vapor pressures will start rising if alternate refrigeration arrangements are not there. Usually an alternate refrigeration system is provided which will run on power supply from emergency feeder. Or else emergency venting systems are to be provided. These vapors are normally vented through flare stacks after burning.

Emergency lighting:

This is most important particularly if power failures occur during night to take proper and complete shut down of plants, and for plant personnel to carry on their work.

Avoidance Explosive Mixtures, Fire Hazards etc.

Operation of any gasification process involves potential hazards of explosion, fire and toxicity. The areas where utmost care is to be taken are listed below:

Hydrocarbons: Care must be exercised in the handling of hydrocarbon feed stocks in both liquid and gaseous states. In opening any of the feed stock lines for changing of blinds in flanges or for maintenance, care should be taken to prevent seepage of the hydrocarbons toward furnaces or other combustion sources. It is particularly important that all hydrocarbons be kept away from any potential leakage of O_2 . With respect to coal, while handling dusting is a problem. This dust is highly flammable and even a minor spark will create an explosion. So it should be emphasized in these areas, there should be no use of naked flame, smoking should be strictly prohibited and lighting in the area should be explosion proof.

The other points, that are of importance in handling of hydrocarbons, particularly naphtha are:

1. Receiving tank and feed supply tank to gasification units should be different.
2. Receiving tank should be drained for water at regular intervals. Proper settling time should be given to received naphtha or hydrocarbons before it can be connected to plant.
3. Pump suction line should be at least one foot above from the tank bottom.

These are the steps to avoid explosive mixtures due to carry over of water along with hydrocarbons to shell reactors or other gasification units.

The other points that are of importance from safety angle are:

- 1) electrical continuity of naphtha hydrocarbon lines and equipments,

at the flanges. (2) Proper earthing of naphtha/oil storage tank and provision of lightning arrester. (3) Use of non sparking tools while doing maintenance jobs on equipment handling hydrocarbons.

Oxygen: O_2 because of its great capacity for reacting with hydrocarbons should be carefully controlled. Oxygen valve gland leaks and packing leaks should be continuously monitored. Great care should be taken to prevent any contact with grease and oily materials in valves and piping or equipment. Any newly installed or repaired equipment in oxygen service must be thoroughly degreased dried and inspected prior to contact with O_2 . Whenever possible these lines and equipments should be purged with N_2 or steam before opening out for inspection and maintenance. During operation of any O_2 gasification process it is essential to admit hydrocarbon feedstock to the reactor or gasifier first and then only oxygen/steam mixture. In cutting reactants out of the system, the order should be reversed i.e. O_2 should be cut off first, to avoid the presence of free oxygen which is likely to cause an explosion.

Product Gases: There are normally mixtures of CO_2 , CO , H_2 , CH_4 and other hydrocarbons along with H_2S in varying compositions. These are highly combustible and they burn with low luminosity. If leaks are there they should be rectified at the earliest opportunity. No risk is worth taking as far as these gases are concerned. Before opening out any equipments for maintenance or while doing any maintenance jobs, the equipment concerned should be isolated by providing blinds and breaking flanges and should be purged free of these gases. There is more so when any hot work, like welding, grinding etc., are involved. Explosives limits of gases handled in ammonia plants are as given below:

1. H_2 - Explosive limits : 4.1 to 74% by volume in air
2. CO - Explosive limits : 12.5 to 74% by volume in air
3. H_2S - Explosive limits : 4.6 to 46% by volume in air
4. Ammonia - " : 15.5 to 27.5% by volume in air

Other points to be kept in mind for avoidance of fire and explosive mixture are

- 1) Hydrocarbon handling system should be as far as possible free of leaks. This is more so, particularly in case of liquid hydrocarbon handling pumps. If leaks are there it should not be allowed to spill and should be collected in a tank.
2. Air/ O_2 should not be allowed to enter any equipment which was under gas or ammonia pressure, earlier, unless it is purged out thoroughly.
3. Gas leaks if any in the plant are to be immediately or at the earliest opportunity rectified so that they may not prove fatal.

AIR SEPARATION PLANTS FOR OXYGEN GASIFICATION PROCESSES.

Uptil now there is no viable warm process that can produce O_2 on commercial scale in hundreds of tonnes/day which is the requirement of Fertilizer Ammonia Plants. All the commercial processes are based on low temperature, basic raw material being air from atmosphere. As is the case with all other technical low temperature plants, air separation plant consists of four constructional units i.e.

- a) Air compressors
- b) Heat Exchangers for air and product streams
- c) Fractionation columns
- d) Refrigeration equipment, such as expansion machines or expansion valves.

Although all these units are of equal importance and are indispensable, the heat exchangers are of particular importance. There are some in which the heat exchanger is coupled with the removal of water and carbon di-oxide and some in which these two processing stages are conducted separately. The latter system required additional equipment. The every-day names of these air separation plants are derived from the differences of these systems. For examples:

- a) Molecular sieve plants or Dehydrier plants.
- b) Regenerator plants
- c) Revex Plants and
- d) A combination of Revex and Molecular sieve or Regenerator and Molecular sieve plants.

In the layout of Molecular sieve plants, after compression the air flows through the molecular sieve adsorbers in which water and carbon di-oxide are removed or carbon dioxide is removed separately by caustic wash. It is then cooled down in the heat exchangers and passes

into the usual fractionation columns.

In the layout for regenerator plants, the condensation of water and carbon di-oxide and in addition sublimation of the latter into one of the product streams take place in the regenerators. Regenerators are containers filled with material of high heat capacity. Air and product gases flow through these alternatively from and to fractionation columns.

The feature of revex plants also, is similar to regenerator plants which combines the function of heat exchange and water plus CO_2 removal before the air is sent to fractionation columns. All these systems or a combination of any of the two are a feature of any Air Separation Plants.

If we take as an example a plant in which only dry and carbon di-oxide free air is to be separate at a power consumption of 100%, the molecular sieve plant would have a power consumption of 108%, the regenerator plant of 104% and revex plant of 102%. It is the regeneration of adsorbers that is responsible for the high additional power consumption of this type of plant. It is mainly the change over losses in the regenerators or revex units that are responsible for the increase in power consumption in the other two plants.

Molecular sieve adsorber plants require the least attention by operating personnel. This applies to start-up, normal operation and also to shut-down. A particular advantage is that these plants may be left in cold state after shut down.

Regenerator plants and revex plants require a certain amount of operational skill. There are strict rules that must be followed when starting up these plants. During the cool down period the cold ends of the Regenerators/Revexes must be kept at a lower temperature than the low temperature section in order to avoid the accumulation of ice

and carbon di-oxide snow. A further requirement in the case of reverb. plants is that not only during normal operation but also during cool down process, it must be possible for the water and carbon dioxide to be completely sublimated, that is care must always be taken to ensure that the permissible temperature difference is maintained. Start-up adsorbers are often used to facilitate start up of Reverb plants.

Even under normal conditions the temperature of the regenerators and Reverbs must be monitored and kept within the prescribed limits of the plants are left in the cold state after shut-down, precautionary measures must be taken when they are started-up again. If, however, the instructions for use are adhered to - and with a certain amount of care - this is easily possible these plants can also be adopted to suit any required conditions at short notice and without any difficulties of operation developing.

A most important problem is that of corrosion of low temperatures. A distinction has to be made in two types of corrosion. One of these arises in connection with a plants that are out of operation and thus at ambient temperature mostly during prolonged shut-downs. The other type of corrosion affects the plants that are actually in operation. Corrosion appears at the point where the plant is cooled down. There are various corrosive compounds in air. The tendency is for these compounds to cause corrosion where the air is cooled, causing its water content to precipitate and from an aqueous solution in combination with the corroding components. The type of plant that is least endangered by corrosion is the regenerator plant. Regenerators can be cheaply manufactured from any required material, such as for example Aluminium,

but it is also possible to use steel. In this way it is possible to eliminate any corrosion problems. Molecular sieve plants are more corrosion prone than the regenerator plants. Molecular sieve is the corrosion sensitive part in that case. Revox plants on the other hand are corrosion sensitive because some kind of solder is used in combination with aluminium to make the revox. Such combinations tend to form local cells. If a revox plant is operated with air containing corrosion components, the air must be passed through a water wash before it is allowed to enter the revox.

Plants of any size that are constructed for the extraction of gaseous products will usually operate at pressures between 4 and 6 bars/atms. It is only in the case of these plants Regenerators/Revoxes are used in practise. Wherever large quantities of liquid air are required, higher pressures are used. Two distinct methods be used for this: either process air is compressed to the higher pressure or else the separation plant operating at a lower pressure is combined with the Air or Nitrogen cycle operating at a higher pressure. Plants of this kind are usually molecular sieve types. If plate type heat exchangers are to be used, 50 atms is the max. permissible pressure. This limit being set by the material strength of the plate type heat exchanger. If the tube type heat exchanger is to be used there is no objection to going up to pressures of even 200 bars/atmosphere.

Reciprocating compressors have been largely replaced by rotating compressors where low pressure plants are concerned. Turbo compressors are used in almost all large plants. Reciprocating compressors are used mostly for compressing product gases.

Screw type compressors are used only in special cases. The reason being that in air separation plants pressures are required

which are not within the efficient operating range of these compressors.

The well established double column system is a common feature all air separation plants. The operational range of the entire plant is determined by the column system in an air plant. A great deal of development has been done on column trays. Modern trays operate at high gas and liquid speeds and have a wide working range.

For making air separation plants Cu has been for the most part replaced by steel, aluminium alloys and Aluminium. Within the plants practically all tube type heat exchangers have been replaced by plate type heat exchangers and flange connections by welding connections etc.

Upto now only the smaller type of plant has been completely automated and with the minimum of expenditure. Usually the automation of large plants has been restricted to remote control. Valves that require constant monitoring such as for example, the control valve for reflux to the pressure column's level of liquid O_2 from column. Sump are automatically controlled. The remaining valves are permanently adjusted. An alarm switch off system is fitted. In the event of any malfunctioning that may result in the impurity of the product and danger in the plant automatic shut down takes place. The plant is started again by hand. If uninterrupted production is essential and this being the case with all plants operators are always available and they take over when an alarm is given.

At present most of the old air separation units in India are essentially of Cu, Aluminium and Brass construction. Recent trend is to make use of S.S., Aluminium and Aluminium alloys. Some of the units use slagwool as insulating material in the cold box. However, S.S. and Aluminium construction - wherever good quality rock is available, expanded perlite may be made use of.

Safety in tonnage plants is of prime importance. Unfortunately there are no Indian Standards until now with regard to installation and testing procedure for tonnage O_2 plants and O_2/N_2 pipe line distribution system.

OPERATIONAL AND MAINTENANCE CONSIDERATIONS
FOR AIR SEPARATION PLANTS.

1. Hazardous Impurities in Air Separation Unit and their control during separation.

The careful monitoring and control of certain contaminants entering with the process air are essential to the continued safe operation of ASU. The principal impurities affecting safe operation are acetylene and oxides of N_2 . Hydrocarbons other than acetylene present additional hazards although less familiarity exists with these substances.

In many plants near the vicinity of Air Separation Plants Nitric Acid and refinery are located. In order to avoid the intake of contaminated air, the air intake location has to be fixed after giving due consideration to the wind direction. If the ground level concentration of gases is high then a tall air intake tower should be built, so that least contaminated air is taken inside the box.

Suitable filters should also be provided to remove the particulate dust. A water washing is also provided in order to scrub the contaminated gases before it enter the air box.

Wherever copper equipment are used inside the cold box special care has to be exercised to see no ammonia gets into the box which otherwise will damage the copper equipment.

Acetylene is the most feared of the hydrocarbons because of its highly explosive nature and because of its low solubility in liquid O_2 . Because of their relative insolubility large amounts of acetylene in solid form can build up. The actual quantity of which cannot be determined by analysis. In practice, the presence of acetylene in the vaporiser liquid in excess of 1 ppm is considered as a dangerous limit. Sufficient to justify shutting down of the plant,

purging all contaminated liquids. This lower figure in the vaporiser has been established as it is important to maintain acetylene in solution at all times.

Oxides of N_2 also introduce hazards if present in the air entering the Air Separation Unit. This is particularly there if any hydrocarbon material is also present at the same time, even in low concentrations. Temporary peaks can be tolerated if they are of short duration (one or two hours), but if concentration in excess of 0.05 ppm persist for longer periods, the contaminated liquid must be purged and the source of contamination removed. The upper limit of oxides of N_2 is 0.1 ppm.

The behaviour of hydrocarbons, other than acetylene, is less predictable, unless the actual impurity can be clearly identified. Certain unsaturated hydrocarbons are known to be dangerous, but the presence of relatively high concentration of some of the unsaturated hydrocarbons can be tolerated. The normal method of analysing for hydrocarbons is to measure the quantity of CO_2 produced on complete combustion of hydrocarbon in a sample of liquid O_2 . The quantity of CO_2 there is therefore employed to establish limits of safe operation. The total concentration of Hydrocarbons other than acetylene in the vaporiser liquid should not be permitted to exceed 150 ppm expressed as moles of CO_2 /mole of O_2 . Limits for individual hydrocarbons are given in table that follows:

When abnormal conditions exist, steps should be taken to eliminate the impurities by changing over of the hydrocarbon filter and if necessary, by generous purge of low pressure columns and vaporisers. If in spite of these steps, the concentration of hazardous contaminants continue to build up over the specified maximum limit, it will be necessary to divert the air feed and to purge

the contaminated liquids.

It should be noted that the only criterion for such action should be the condition of the liquids in the main vaporiser and L.P. columns. Although tolerable limits are listed for the impurities in the entering air, satisfactory conditions at this point do not necessarily indicate safe operation, nor does the lack of these conditions necessarily indicate the presence of immediate danger. This fact can be established by careful analysis of the liquid O_2 in the vaporiser of L.P.Column.

However, if due to a combination of circumstances, the acetylene content of the air feed should greatly exceed 1 ppm, it may be necessary to shut down the air plant until these conditions can be corrected. After shutting down, the liquids in L.P. column and vaporiser should then be carefully analysed during the duration of shut down and if necessary sufficiently purged out.

If the plant has been shut down for a long period, better to drain the liquids. Or else concentration of hazardous impurities should be checked and a decision should be taken to retain or drain the liquid. When the plant is started a considerable amount of liquid will be vaporised during initial stages and thus the concentration of impurities in the retained liquid will increase. Care, therefore, should be taken to ensure that these contaminants are well below normal levels. Otherwise, it would be better and so far that these liquids be purged and fresh liquids produced.

It is always advisable to drain a small quantity of liquid both from vaporiser and low pressure columns at regular intervals so that these impurities will not build up to abnormal limits during operation. These impurities should be checked at least once in an 8 hour shift.

Concentration limits for hazardous contaminants when

operating:

Contaminant	L.P. Column		Main vaporiser	
	Normal	Abnormal	Normal	Abnormal
	(Vol/Vol - in ppm)			
CH ₄	10	100	10	100
Acetylene **		0.2		0.6
Ethane *** Ethylene each	2.5	25	7.5	75
Propane *** Propylene each	1.5	15	5	50
Butane * Butanes each	0.5	5	1.5	15
Pentanes * each	0.1	0.6	0.3	2.0
Hexanes *	0.02	0.05	0.05	0.15
Total HC	5	50	15	150

Note: Total hydr carbons as Mol fraction methane equivalent

* Limited by volatility in O₂ exchanger.

** Limited by solubility in Main vaporiser.

*** Limited by maximum 150 ppm in bulk liquid phase.

Purity of products: Of the products N₂ purity is the most important. O₂ in N₂ product as impurity above 100 ppm is hazardous and it should never be allowed to exceed beyond this while the plant is running. At 150 ppm normally a high O₂ in N₂ alarm will sound in the control room as well as N₂ compressor. Immediately N₂ to NSU should be cut off and N₂ should be vented at the compressor suction till conditions become normal again. Otherwise it may form an explosive mixture inside nitrogen scrubbing unit and rupturing the equipment, endangering both personnel and equipment.

Purity of products is maintained by the adjustment of pure and impure N₂ refluxes to low pressure column. These reflux streams are

to be checked for their O_2 content at least once in every 8 hour shift and accordingly these refluxes adjusted so that purity of N_2 will not be upset.

One other regular analysis which should be carried out is the waste N_2 analysis for its O_2 content. This will give us the extent of O_2 recovery. If the O_2 in waste is more O_2 recovery will be less and this will require again adjustment in refluxes without upsetting N_2 purity.

OTHER OPERATIONAL CONSIDERATIONS

1. Operation of hydrocarbon adsorbers on whose performance hinges the plant safety.

These are mostly situated on the stream going to Low Pressure columns as feed. There are plants which use them in more than one location. Normally silica gel is adsorbent. Molecular sieves are also being extensively used. These are always in pairs.

When the plant is running normal without any disturbances or upsets these adsorbers will be in line for about a weeks duration. After wards it will be cut out of line after putting in line the spare one and then regenerated to a temperature of 250°C with either air or N_2 to get rid of adsorbed hydrocarbons and then cooled and kept as standby.

Annually the material should be inspected and if it is degraded it is to be replaced.

At times in plants situated near petroleum refineries or petrochemical complexes there is high concentration of hydrocarbons in feed air. In such emergencies it is normal practice to put in line the fresh adsorber so that abnormal build up of hydrocarbons will not be there in low pressure columns and vapouriser liquids. This is a safety precaution.

Other criterion are (1) unusually high pressure drops across the adsorbers (2) Oil break through into the box - Oil will contaminate the adsorbent and will reduce their adsorption efficiency.

2. Operation of Air-Oil adsorber, N_2 - Oil adsorbers Air Dryers.

Oil or moisture break through into the plant equipment and exchangers creates all sorts of problems in addition to safety. These will build up unusually high pressure drops across exchangers.

Oil film will reduce the cold or heat exchange between process streams. In addition oil is a hydrocarbon which if breaks through into O_2 rich areas is highly hazardous and is a potential hazard.

Explosions and rupturing of equipments cannot be ruled out. In addition condensing moisture along with few CO_2 etc. in exchangers is highly corrosive to equipments.

So it is essential that we take care that oil and moisture do not break through into the box. This can be accomplished by proper operation of dryers and oil adsorbers. Material used in oil adsorbers and driers is either activated alumina or Molecular sieve. Air driers are normally in pairs or in threes with regular regenerative and adsorption cycles ranging from 8 hours to 16 hour duration. Proper attention should be paid to maintain the regenerator temperature and the cooling rate for better performance of these adsorbers. Material in these driers or dehumidifiers is normally changed during annual turn around. Other criterion for changing is unusual dusting or pressure drop and breakage of the material due to upsets and sudden failures in plant operation. Oil adsorbers are always in pairs. These are normally in line for a duration of 40 days to 60 days depending on the adsorber capacity. Normally material changed after this duration after putting in line the spare one. Other criterion for changing is inefficient oil adsorption and excessive pressure drop and unusual dusting. In the choice of drying agent the material should be such that it does not disintegrate during heating and cooling so that there won't be any dust carry over when it is in line. It is important that for better working of these de-oilers care should be taken in the operation of compressors from where the oil originates. Proper precaution should be taken that

oil drops are checked every day and corrected if required. Check should be kept on oil consumption in the high pressure reciprocating compressors for both N_2 & Air compressors of boxes.

In air separation plants insulation is the most important thing. If boxes are not properly insulated heat will leak into the system and upset the plant condition. Various types of insulating materials that are in use are:

1. Mineral wool or slag wool
2. Perlite or Rock wool
3. Celite
4. Silica & Asbestos, Magnesite
5. Foam glass.

First and foremost requirement is it should not contain oil. Lines and equipments are usually lagged with foam glass. Box is filled with slag wool or mineral wool or perlite. Other type of insulations are also used to lag lines and equipment. Slag wool filling or taking out is a laborious job. Care should be taken while filling and taking out slag wool because it pierces the skin. Personnel working should be provided with Face Shield and goggles and overall to cover the body.

Defrost & Solvent washing:

Operation of any low temperature process very much depends on the efficient heat and cold exchange in exchangers. Regardless of the efficiency of CO_2 and moisture removal. Over a period of time while the plant is in line impurities like moisture oil + CO_2 that have slipped through get accumulated in these exchangers and form a film on exchanger surfaces.

Usually Air Boxes are completely derimed during the annual turn a round. Other occasions when this is done is whenever any maintenance job is done in the box and internal of the equipments get exposed to atmospheric air.

In N_2 wash Units also complete deriming is done during the annual turn around. Partial deriming of affected exchangers is done whenever pressure drop across the exchangers and cold losses at warm end increases.

With deriming only moisture and CO_2 if any are driven out. But oil and heavy hydrocarbons accumulated will remain as such since these requires very high temperatures. The alternative is solvent washing of the affected portions.

Solvents used for solvent washing are:

1. Trichloroethylene (TCE)
2. Methylene Chloride
3. Chloroethene Cl_2
4. Carbon tetrachloride (CCl_4)
5. Acetone.

Of these Trichloroethylene, Chloroethene Cl_2 and Carbonate trachloride are the most widely used solvents. These have got high capacity for oils. These solvents have high solution power and are volatile sufficiently, so that traces remaining can be readily removed at the temperature used in deriming. Solvent should be pumped in the opposite direction to the normal flow in the equipment. Usually all exchangers require solvent washing once in two years. However, it should be remembered that before solvent washing and complete defrosting whole box should be sufficiently warmed up after draining all the liquid.

Other Operational and Maintenance hazards:

In addition to the normal hazards encountered in gas producing plants there are additional hazards associated with the operation of an Air Separation Unit and these require certain additional safety precautions. These arise mainly from the presence of O_2 in gaseous or liquid form and from the use of very low temperatures.

O_2 : Even materials difficult to burn in air, burn easily in O_2 . Parts of the equipment within air separation unit contain almost pure O_2 in liquid state and this may react even explosively in contact with small quantities of flammable substances for these reasons, it is necessary to prevent any flammable material from coming into contact with O_2 and to avoid all possibilities of ignition whenever O_2 is present. Thus no oil or grease deposits should be permitted to accumulate or remain anywhere in the vicinity of the air separation unit or any other oxygen equipment. Any rags which may have been used for wiping up oil spillage should be kept in closed metal containers and in no case should they be left near the cold box or carried in the pockets of clothing.

For their own protection, operators and workmen should not be allowed to wear oily or greasy clothing or wear oil stained gloves, when working on or near oxygen equipment. There are occasions when liquid O_2 is withdrawn from the plant and consequently high local concentration of O_2 can occur. Special care should be taken in these vicinities. In general the area surrounding should be treated as a hazardous location and the same strict caution observed as with highly flammable organic gases.

When maintenance work is conducted on the ASU or any other O_2 equipment, precaution should be taken similar to those employed

on refinery practice in hazardous locations. The vessel should be completely disconnected and all lines blanked out. The vessel should then be carefully and completely purged with air before any major work done or undertaken. Non sparking tools should be used at all times. All pipes and fittings should be installed free of flammable oil and grease and where threaded joints are necessary, no white lead, grease or pipe fitting compounds containing oil should be used. When a compound is necessary compound of latharge and glycerin or Tri chloroethylene may be used.

Should it be found necessary at any time to enter the cold box, care should be taken to provide adequate ventilation as local pockets of O_2 can be present due to minor leaks in the equipment and high concentrations of oxygen can therefore occur. It should always be remembered that the only "safe" O_2 concentration is that existing in atmospheric air.

Low temperature:

In handling all low temperature materials, additional hazards due entirely to the low temperature are present. Injuries such as "cold burns" or actual freezing of the hands or other parts of the body can readily occur unless great care is taken. When withdrawing liquid from the plant for sampling or for other purposes care should be exercised as the introduction of cold liquid into a warm vessel results in the generation of large quantities of very cold gas and the violent boiling which occurs may also produce some splashing of the liquid. Gloves and protective goggles should therefore be worn whenever handling containers of low temperature liquids and direct contact with any low temperature materials should be scrupulously avoided.

MSU: Nitrogen scrubbing unit treats gases which are highly inflammable and in certain concentrations are explosive. When maintenance work is conducted on the N_2 scrubbing unit or associated equipment precautions should be taken similar to those employed in refinery practice for hazardous location. Any vessel on which work is to be performed should be completely disconnected and all lines blanked off. The vessel should be completely purged with N_2 before any major work is done. Non-sparking tools should be used at all times. If it is necessary to enter the cold box, great care and precaution should be exercised. If any leaks have developed within the unit pocket of liquid and hydrocarbons and gases may be held in the insulation for considerable periods. If exposed this liquid will vaporise producing large quantities of flammable gas. Generous ventilation is therefore required any time such work is carried out.

The danger from N_2 should not be ignored. Although N_2 is non-toxic an atmosphere of N_2 will not support human life and can thus be as dangerous to life as the presence of toxic gases and death can result from suffocation. Any vessel or enclosed space which has contained or been purged by N_2 should therefore be purged with air before being entered.

N_2 Bleeding of boxes:

Another point which is very much important and mostly overlooked is the purging of the box with N_2 continuously. Purging should be thorough and evenly distributed in the whole of the box. This is to avoid atmospheric air getting into the box where leakage of hydrogen and hydrocarbon are likely to be present.

Maintenance of plant machinery and equipments:

Mostly maintenance problems arise in rotating machines like O_2 pumps, O_2 compressors, air compressors and N_2 compressors.

Maintenance of air separation and gas separation plants calls for good knowledge of cryogenic engineering because of special materials of construction involved like Cu, Al, brass, bronze and S.S. The physical properties of these materials at very low temperatures differ so drastically from those commonly encountered under normal condition.

For example: 1. Some metals become very brittle at low temperature. This is true of particularly carbon steel. Disastrous failures of engineering structures have been attributed to this cause. On the other hand stainless steels behave very well. Some non-ferrous metals like Al, ore and nickel.

2. Electrical resistances of pure metals are extremely small at low temperatures. Some metals have zero resistance below a certain temperature. This phenomenon is known as super conductivity.

3. Thermal conductivity of pure metals and non-crystalline solids increases greatly at low temperatures.

4. In all low temperature apparatus and equipment proper insulation is of paramount importance and since one of the best insulators is vacuum, high vacuum techniques are of great importance in low temperature technology.

Nearly all the common structural metals can be used. Ordinary carbon steel is not often used for those parts which become cold, because of the low temperature embrittlement. Cu and brass find many places in the low temperature process on account of their high reflectivity for thermal radiation and can be joined very easily, although their softness and rather low mechanical strength are distinct disadvantages. Their malleability at low temperature makes them relatively safe than other metals. SS of types 304, 347 are extensively used for low temperature. Process lines where good tensile strength and good impact

resistance are required. Also these steels have low thermal conductivities, so they are very good for use as insulating supports for inlet and outlet lines communicating between cold and warm equipments. S.S. type 347 is preferred because of its stability - areas adjacent to welds are less apt to be damaged by overheating. Emissivity of S.S. is 5 to 8% which is rather high as compared to 2% for Al and 1.5 to 2.0% for Cu. S.S. are more difficult to soft solder than Cu and Cu alloys. A rather corrosive flux is required to remove the refractory oxides. Such a flux must be used carefully and be completely removed after the joint is finished, because residual flux may corrode through their stainless steel lines and develop leaks inside the box. Present trend is towards the use of Aluminium and its alloys due to the full development of Argon arc welding process.

Gasket materials for flange joints:

Relatively soft gaskets are employed in the hard compression flanges. Although Teflon or PTFE are used successfully at low temperatures. Special precautions must be taken to compensate for the large thermal contraction.

Soft metals like lead, Cu, and aluminium have been successfully used as gasket materials. These can be vacuum tight over a wide range of temperatures. It has been found beneficial to use a film of special type of grease on gaskets to be used at low temperatures. Only approved joining or gland packing materials should be used for O_2 service. Mixture of sodium silicate and china clay is a suitable sealing agent for flanged joints, P.T.F.E. is used for packing the glands of the high pressure liquid O_2 plunger type pumps.

Leaks inside the cold box:

Leakages inside the cold box constitute a major maintenance problem. Removal of mineral or slag wood insulation and detection of exact spot of leaks are laborious jobs. Leaks develop mainly due to vibration of improperly supported pipelines. For example pipe lines connected with expansion engines, liquid oxygen plunger pump or centrifugal pumps transmit a good deal of vibration. The other area where leaks develop are the reversing exchangers where the alternate paths of plates and fin type exchangers are subjected alternately to two different pressures and consequent vibration. Normal type of failures are cracks.

Valves inside the cold box:

We have also some across valves inside the cold box, developing internal leaks or gland leaks and valves becoming inoperable due to defects in the handle. Plants have been shut down on a couple of occasions for replacing or reconditioning of these valves. The non-return type valves in the air exit line from Regenerators and Reversing exchangers also develop leaks sometimes. These are normally of faliper type with hinges and seals which wear out.

High Pressure Liquid Oxygen Pumps:

H.P. Oxygen pump at Trombay is a 20 stage vertical centrifugal cryogenic pump having a mechanical seal. Initial stages of plant operation there were many failures of these pumps mainly due to the problem of mechanical seals and due to vibration of rotating elements. The original coupling design was changed to a more rigid type. Mechanical seal was changed to grease lubricant seal. Since this modification was carried out no other serious problem had been experienced and the pumps are giving satisfactory service. Where gaseous oxygen is produced these are compressed by means of multi

stage compressors which are lubricated by demineralised water. It is essential to ensure that no oil or foreign material enter the compressor which would cause explosion. The quality of demineralised water and the regular flow should be positively ensured to avoid such hazards.

High Pressure Air and N_2 Compressors:

These are used to compress air upto $200 \text{ kg/cm}^2 \text{g}$ and nitrogen upto $30 \text{ kg/cm}^2 \text{g}$ usually. Suction to the compressor on air side is mostly from an air chiller where this air is chilled with water. Due to this saturated air we have the problem of carbon steel suction line getting corroded and the resultant rust fouling the suction strainer also breaking through it, getting carried over to compressor cylinders. This damages cylinder valves and piston rings and the cylinder liners and causing frequent breakdown of equipment. As a remedial measure we have suction catch pot packed with raschig rings to act as an additional strainers. We propose to change over the line to that of S.S. and also providing an additional parallel strainer for cleaning purposes while the machine is in line.

We had two serious failures on the air side 4th stage after cooler air discharge line bursting, resulting in considerable damage to equipments. We use mineral oil as lubricating media on our air compressor cylinders. It has been suspected that cracked carbon from the mineral oils in presence of air has cause the explosion. Now to avoid this we keep a check on oil drops thoroughly and also keep a check on oil consumption. We also clean the after cooler and piping with Trichloroethylene at regular intervals to remove deposited carbon and oil.

Turbo Compressors:

The main problem we have is frequent fouling of intercoolers. This is mainly due to the salt dust and other corrosive particles sucked along with air. This problem is acute. These intercoolers get fouled up within an year. The intercoolers are of admiralty brass tubes and aluminium fins.

Reversing Exchangers:

We have a plate and corrugated fin type of aluminium exchangers where heat transfer, CO_2 and moisture removal from air are carried out by successive condensation and sublimation. Air and waste N_2 path get reversed at regular intervals. Part of the pure N_2 is also taken through this exchanger to maintain the proper cold balance. After 4 years of operation inter path leakage was observed with air leaking to waste N_2 and pure N_2 streams contaminating the pure N_2 product. When conditions became worse, this contaminated part of N_2 was vented immediately down stream of Revex train. In '74 we have changed the whole Revex train. After 4 months of operation in the new exchanger also interpath leakage was observed with bulging of one of the core plates. The possible reasons for the failure of these Revexes could be (1) Water & CO_2 freezing and their subsequent corrosion. (2) Manufacturing defect (3) or faulty material. The leaky core replaced with a salvaged block.

Expansion Engine:

We have three vertical Reciprocating Engines for expanding air from 180 kg/cm² g. The problems experienced are -

1. Inadequate cooling hardly 100 to 110°C as against 142°C
2. Undue vibration of the machine (3) Very wide flow fluctuations.

To overcome these (1) The design of exhaust can profile has been changed (2) Speed has been reduced from the original 360 rpm to 300 rpm.

(3) Automatic capacity control has been changed to manual control. All these have overcome the problems to some extent but performance of the machine as regards to cooling, there is not such improvement. Vibration has been reduced to a considerable extent. We have had several bad experiences of oil breakthrough into A.S.U. N & S because of the wide flow fluctuations of these machine. The filters at the exhaust we clean it once in fifteen days.

Switch warm exchangers:

These are plate and fin type exchangers to exchange cold between product synthesis gas and incoming feed gas. We have some times experienced cracking up of connecting piping, because of caustic carry over from decarbonation system.

Feed Gas De-carbonation Towers:

These are to remove last traces of CO_2 after hot potash and MEA wash with caustic wash. The carbon steel vessel provided for this was found to have corroded and eroded in the region of feed gas inlet. The thickness was found to be considerably reduced. The vessel has been welded with reinforcement plates from outside.

Nitrogen Scrubbing Columns:

We had to replace this column in one of our units since CO was breaking through continuously after liquid N_2 scrubbing. We have cut the original column and have found one or two trays damaged and tilted. The exact reason for this damage is not known.

CATALYSTS : HANDLING, REDUCTION AND CONTROL

Safety in the large ammonia plants not only is concerned with the protection of personnel and equipment but also should be directed towards the protection of catalysts that are used at each step in processing. The catalysts used represent 2 - 3% of the total plant investment. But improper use of them, however can result in an appreciable loss not only in the value of the catalyst itself but, more important, in terms of lost production. It should be appreciated that the designs of the new large ammonia plants impose much more severe operating conditions on the catalysts than do the older low pressure plants. Higher pressures and temperatures greater throughput resulting in higher pressure drops across the catalysts and greater expansion and contraction forces on the catalysts all contribute to this more severe service. In order to realize the maximum utilisation of catalysts, it is essential that the plant operators have a thorough working knowledge of the catalytic units and the operating procedures to be used with the catalysts to ensure the safe and efficient performance of catalysts.

General Handling Procedures:

There are a number of general rules for handling and operating any catalyst. Potential safety hazards not only to the catalysts but also to the personnel associated with the use of handling of the catalysts are outlined here in below.

1. Most catalysts used in modern ammonia plant are quite rugged, but they can be damaged by rough handling. Particular care must be given during charging of the catalysts to avoid damaging them.
2. All catalysts are screened before shipment, however, there will be some dusting and possible breakage in transit and handling. It is recommended that all catalysts be screened before charging to

the reactor. In some cases it is not necessary to screen it all, but certainly the bottom portion of each drum should be screened.

3. In charging catalysts to a converter care must be taken not to drop them from too great a height. Further care must be taken while loading to evenly distribute the material. This can be done by pouring the catalyst directly from the drum or a bucket or by charging through a funnel with a long chute which is flexible enough to move around inside the reactor. If it is felt necessary to have a personnel inside, in which case, he should stand on boards to distribute his or their weight over a large area and prevent damage to the catalyst.

4. The state of the catalysts as delivered should be known i.e. is it pyrophoric or hygroscopic and suitable precautions observed if required.

5. Catalyst used in ammonia plant are mostly in reduced state. Generally speaking, these should be reoxidised before removing them from a converter or before opening the reactor for any maintenance work or catalyst examination.

6. If personnel are to enter a reactor the catalyst should first be oxidised if required. The vessel should then be thoroughly purged and personnel should be provided with sufficient and adequate safety equipment before entering the vessel.

7. The vendors recommendations should be followed in the handling, loading, start up operation and during shut down.

8. If the catalysts are not to be reused again they can be taken out as such after thoroughly purging and cooling down the catalyst bed and also by keeping inside an inert gas atmosphere. The precautions to be taken further depends on the type of catalyst. However it is important that we always have available water hosing facilities nearby.

Production of ammonia from any feed stock involves the following basic steps.

1. Removal of sulfur from feed stock.
2. Raw synthesis gas production (i) by steam naphtha reforming and subsequent air oxidation (ii) by partial oxidation with high purity O_2 .
3. Co-oxidation with steam.
4. CO_2 removal by liquid scrubbing.
5. Removal of residual trace quantities of CO by methanation or by liquid nitrogen scrubbing.
6. Ammonia synthesis.

Each of these steps with the exception of step No.4 is a catalytic process in the case of plants based on steam reforming plants and with exception of step No.1, 2, 4 and 5 in the case of partial oxidation plants.

Desulfurisation of feed stock (Natural gas/Naphtha)

Of all the catalysts that are used in ammonia production, the activated carbon used to desulfurise natural gas is the only one that is combustible. The ignition temperature in air can be as low as $200 - 210^{\circ}C$ for carbons impregnated with iron or cu and this should be kept in mind when storing and handling. Before removing this activated carbon it should first be regenerated. In order to increase the sulfur adsorption of these activated carbon beds, they are usually impregnated with oxides of iron or cu or both.

The second system is the cobalt molybdate and zinc oxide desulfurisation. This presents no real hazard during handling operation. Normal precaution should be taken during loading and unloading to ensure that excessive dusting does not occur. Personnel handling the catalyst should be protected against possible ingestion

of catalyst dust which is very harmful.

During start up zinc oxide catalyst is not reduced but only cobalt molybdate. But cobalt molybdate catalyst has known to cause a dangerous situation. This can arise when heavy feedstocks are being reformed and it is considered necessary for the primary reforming catalyst to be reduced prior to start up of the plant proper. When this operation is to be carried out, the normal technique is to recycle a process gas stream containing H_2 , CO & CO_2 through the desulfurisation system and then into primary reforming catalyst when this is done at reasonably high pressures the cobalt molybdate catalyzes methanation reaction in the gas stream. In one of the plants in Germany where a desulfurizer reactor was severely damaged due to a run away reaction taking place during start up. As a general rule, it is advisable to limit the CO plus CO_2 content of any gas stream passing through cobalt molybdate bed to not more than 5% during the reduction. By this means if conditions are suitable for methanation reaction to take place the total temperature rise through the bed will not be sufficient to cause damage to either catalyst or reactor.

Primary and secondary Reforming Catalyst:

For best operation of primary and secondary reformers certain set procedures to be followed. The weight of catalyst loaded to each tube should be determined and recorded. Tubes should be vibrated to prevent bridging. If bridging does occur hot spots will develop. At best this results in shorter tube life and could also result in tube failures. If furnace temperature is to be controlled by one, or two tubes production losses will occur. The reformer catalyst in commercial plant are also associated with following problems. (1) Coke formation, (2) Sulfur poisoning (3) Arsenic poisoning (4) Poisoning due to Cl_2 , Cu and Pb in feed stock etc.

The greatest single hazard to guard against in the operation of primary reformers is the loss of steam feed. This will result in carbon formation with physical break up of catalyst and plugging of the tube. In extreme cases this will result in rapid over heating of the tube with subsequent rupture and a potential fire hazard. The usual procedure for guarding against steam failure is by interlocking the gas/naphtha and steam flow controllers which will cut off both simultaneously in the event of steam failure.

While putting a reformer back in operation after a steam failure the tubes should be observed for hot spots which will indicate whether catalyst breakage or tube plug up has occurred. If carbon has been formed in the tubes it can usually be removed by steaming for a period of time. In severe cases the carbon can be removed by the addition of air to the steam.

Severe cases of carbon formation are hazards not only to the reformer furnace but to virtually every catalytic unit in the system. Another problem sometimes encountered in primary reformer is break up of catalyst due to carry over of liquid water, to the reformer tube due to thermal stresses developed.

Secondary Reformer:

Operation of secondary reformer catalyst is relatively trouble free. In several cases there have been mechanical failures in air and synthesis gas distribution nozzles which have resulted in the direction of high velocity stream on to the catalyst which has resulted in over heating and fusing of the catalyst.

A potential hazard of secondary reforming is stoppage of synthesis gas flow to reformer which might occur due to upset in primary reformer. Provisions must be made for diverting the air from the secondary reformer in such instances. Another potential problem is

putting too much air into the secondary, which would result in overheating and possible damage to catalyst. Since this catalyst also operates at a high temperature care must be taken to prevent any water carry over to the catalyst.

Shift Conversion catalyst:

High temperature shift:- High temperature shift catalyst, like reformer catalysts present only minor safety problems. There are no known hazards involved in handling the new catalyst during normal initial loading. When placing the iron chrome high temperature shift catalyst on stream, certain precautions should be taken. There have been instances where air is used during the initial heat up of a new plant. The air flows through primary and secondary reformers and the high temperature shift converter when steam becomes available, steam flow is established and the air flow is discontinued if air is in contact with high temperature Co-conversion catalyst at temperatures above 550°F , it is possible for the graphite in the catalyst to react with O_2 . The resulting high temperature will damage the catalyst.

Reduced iron chrome catalysts are pyrophoric and will oxidise when exposed to air. When used catalyst is to be discarded, it can be removed in the reduced state with no difficulty, if certain precautions are taken. The catalyst bed should be purged with N_2 /steam and then cooled to the ambient temperature. The top and bottom manways are then opened and the catalyst removed. As the catalyst leaves the reactor it should be sprayed with water and it should be kept wet until it is removed from the plant area.

Iron chrome Co-conversion catalysts have been damaged while purging with inert gas, when using gas from air inert gas generator analyses should be made to ensure that the N_2 is O_2 free. There have

been several instances of fouling of the high temperature catalyst bed by solid deposition on the top of the bed, but these problems have been relatively minor. They have been corrected by removing the top portion of the catalyst and eliminating the source of solid contamination by vacuuming of the top layer. During this period the bed is kept under inert atmosphere.

Low temperature Co-Conversion Catalyst:

Low temperature Co-Conversion catalysts are more susceptible to poisoning and to damage from overheating than any other catalysts used in ammonia plants. The active ingredients of this type of catalysts are cu and zinc and there are no special precautions required in the handling of these catalysts during loading. The reduction of LTS catalyst is highly exothermic and if proper care is not taken during reduction the catalyst can be easily overheated. Adequate reduction facilities should be available and these should include meters for metering and controlling the flow of both the carrier gas and H_2 . Procedures for removal of used catalyst can be similar to those for removal of HTS catalysts. If the catalyst is to be discarded it can be removed in the reduced state. If the catalyst is to be reused, it must be carefully oxidized out of service.

LTS catalysts are very sensitive to sulfur and chloride poisoning and every precaution should be taken to minimize the possibility of sulfur compounds and halogen compounds from contacting the catalyst. Process steam should also be chloride free. HTS catalysts can also contain sulfur and should be properly desulfurized prior to admitting gas to the LTS converter lubricating oils used in air compressors, also should be sulfur and chloride free.

LTS catalysts are also very sensitive to high temperatures. Those in excess of $350^{\circ}C$ will permanently damage the catalyst.

Several cases of catalyst overheating have resulted in plants which use a direct quench system between HTS & LTS. Hence the catalyst is to be guarded against failure of quench.

Methanation Catalyst:

The operation of methanator present safety problems somewhat unique in an ammonia plant. An upset in CO₂ removal/CO shift system can result in excessive carbon oxides being fed to the reactor. The resulting high temperatures can easily exceed the design and safe working temperature of methanator vessel. Protection against high temperature is obtained by use of a high temperature alarm system that automatically bypasses the bed when a preset temperature is exceeded. It is also necessary to close the block valves to the methanator so that no gas will flow through. At one plant during an upset in the CO₂ removal system, high temperature activated the alarm and automatically opened the bypass valve. The plant operators did not close the block valves in the methanator feed line. As a result a small flow of gas continued to flow through and temperatures in the methanator exceeded 1300°F. Fortunately reactor did not rupture, but only deformed.

Formation of nickel carbonyl is also a possibility in a methanator. Nickel carbonyl is extremely toxic and whenever men are going to enter or open a reactor stringent precautions should be followed to ensure that nickel carbonyl is not present. Even in a closed system carbonyl formation should be prevented since nickel can be removed from the catalyst with the resulting loss in activity.

Conditions favouring nickel carbonyl formation are high pressures, high Co-Concentrations, temperatures in the range of 50°C to 200°C and the presence of a catalytically active nickel. Sulfur compounds if present act as promoters to carbonyl formation. Nickel

carbonyl formation is most likely to occur during start up and shut down. Methanators should not be kept blocked in under process gas that contains CO. All CO should be purged from the system before the reactor is cooled below the normal operating temperature.

With new catalyst, in the oxidised state there is no problem since the nickel is not catalytically active and it does not become reduced until temperatures are reached that are above the maximum temperature which nickel carbonyl can be formed. If a cold reduced methanation catalyst is to be placed back on stream with CO containing feed gas, it must be assumed that some carbonyl will be formed. If the gas is to be heated to temperatures above 400^oF subsequently nickel carbonyl will be decomposed. If the gas is vented during start up, precautions need to be taken to ensure that personnel are not exposed to vented gas. Gas should be burnt in a flare.

Ammonia Synthesis Catalysts:

Ammonia synthesis catalyst can be divided into two general types. The conventional catalyst is supplied as magentite (Fe_3O_4) which has been treated with various promoters. The second type is a pre-reduced catalyst which is supplied as metallic iron with a stabilizing surface covering of Fe_3O_4 . The latter catalyst has advantage of needing little reduction. But there have been occasions when drums of prereduced catalysts on exposure to air have got overheated and oxidised completely with considerable increase in temperature. Generally these drums are sealed. But it is very important that it should be checked. While storing a check should be kept on the drum temperature.

Reduction of conventional catalysts is of a fairly lengthy procedure, but cannot normally be considered a hazardous operation. It is probably more to be considered a problem of avoiding the

deactivation of the catalyst than of any particular hazard being developed.

No serious problems relating to the catalyst have been experienced in the ammonia synthesis converter in the new plants. Although the catalyst is subject to poisoning from oxygen, the normal purity of synthesis gas entering the ammonia loop is such that virtually no hazard to the catalyst is presented from this source. Another problem is the oil from the synthesis compressors containing sulfur in varying degrees. This also is eliminated in the modern plants with the use of centrifugal compressors and sulfur free oil. Other catalyst poisons are oxygen compounds like carbon monoxide, carbon dioxide and water vapor. Among these water vapour and CO₂ are of less consequence and are essentially removed in the secondary condensation of ammonia and hence they will not build up in synthesis loop. But CO builds up in the loop and in concentrations more than 20 ppm reduces the catalytic activity gradually and in higher concentrations converter becomes unstable and to put it back in line it may take a few hours depending on the extent of poisoning. But in modern ammonia plants with respect to these oxygen compounds purification steps are much better and break through of these compounds into synthesis loops is a very rare phenomena.

Phosphorous, halogen compounds and arsenic are other catalyst poisons. Metallic compounds of lead, tin, bismuth, copper and zinc are also catalyst poisons.

The poisoning effects of halogens and halogen compounds particularly Cl₂ is very severe. These halogens deactivate the ammonia catalysts by depleting alkali promoters. Very extensive depletion of potassium promoter has been experienced in many units.

Another problem with ammonia synthesis catalyst in the past has been the gradual build up pressure drop due to attrition of the catalyst during operation. To overcome this the newest development is the removal of edges and corners of the catalyst by polishing. This gives a catalyst that is more uniform in shape and size and which has a higher packing density in the reactor. The initial pressure drop for a given volume of catalyst is higher than that for a bed of irregular shaped particles. But anticipation is that attrition loss and pressure drop build up will be much less and will result in longer catalyst life.

Ammonia synthesis reaction is highly exothermic and hence care must be exercised during the initial reduction and subsequent operation to prevent overheating and damage of the catalyst. Further during reduction build up of water vapor in the gas should not be rapid. This can be controlled by controlling the reduction temperature.

The reduced catalyst is highly pyrophoric and care must be taken when removing for change out. If proper care is taken oxidation step is not necessary.

In a general way these are some aspects relating to catalyst handling, reduction and control in any ammonia plant. The real cost of catalysts used is ultimately determined by the tonnes of ammonia produced and not by initial cost. In the new large, single train, high pressure plants life of the catalyst is more important, since a change out will require long down times of the plant. Operating conditions of the new plants subject the catalyst to more severe service and there is much less or very little margin for error in plant operation.

TABLE - 2
 CATALYSTS USED IN AMMONIA SYNTHESIS 162 WITH HYDROCARBON FEED STOCK

General Name of the Catalyst	F.C.I. Equivalent	Catalyst composition	Form	Typical Operating Conditions					Life expected with careful operation	Salient features
				Space velocity	Temperature	Pressure	Others			
1	2	3	4	5	6	7	8	9	10	
1.a) Hydro Desulfurization	-	Cobalt Oxide 4% Molybdenum 12%	a) Pellets b) Granules		260° - 430° C	Upto 30 kg/cm ²		3 - 5 years		
b) Desulfurization (Adsorbent)	CDZ-6	1. Zinc Oxide plus Binder 2. Activated Iron Oxide 3. Metal Impregnated Carbon.	a) Pellets b) Extrudates c) Crushed particles		350 - 450° C	Upto 30 kg/cm ²		10-12% Pick-up strength. High 2% pick sulfur capacity. up 1 year to 1.3 kg/litre.		
2.a) Primary Reformer	CD-RE-33	Naphtha Reforming: 20% Nickel Oxide on Ca, Mg, Si & Al oxides with potash promoters. Light hydrocarbon Reforming: 20-30% Nickel Oxide on Refractory carrier, plus promoters.	a) Rings b) Pellets c) Irregular lumps	0.9 kg. of Naphtha/hr. (Max.)	750° - 850° C	Upto 30 kg/cm ²	Steam/Carbon Ratio 3 to 4	3 years Resistant to Sintering upto 1200° C. Resistant to Carbon deposition Good sulfur tolerance. Bulk density - 1.0 to 1.1 kg/litre. Crushing strength 80 kg/cm ²		
B) Secondary Reformer	-	13-21% Nickel Oxide on Calcium Aluminate	a) Rings b) Pellets c) Spheres		982° / 1684° C			3 to 5 years The catalysts is more refractory than Primary Reformer Catalyst		

1	2	3	4	5	6	7	8	9	10
2.c) Methane Steam Reforming	CDR-66A	Nickel Catalyst. Alumina based.	1. Bin s. 2. Cylindrical	3500 (Max.)	700° 850° C	Up to 30 kg/cm ²	Steam/Carbon ratio 2.5 to 3.0	Defining color range upto 1% N.T. Bulk density 1 to 1.1. Crushing strength 30 kg/cm ²	
d) Methane Steam Oxygen Reforming	CDR-66B	Nickel Catalyst Alumina based	1. Rings 2. Cylindrical Rods	3500 (Max.)	700° 950° C	Up to 30 kg/cm ²	Hydrocarbon in the gas 2 to 7.10%	Excellent Thermal stability upto 12000 hrs. Bulk density 1.0 to 1.1. Crushing strength 30 kg/cm ²	
3.a) H.T. Shift	CDC-63	Chromia Promoted Iron Oxide CR203 9-11% Fe2O3 77-81% Graphite 3-4%	1. Pellets 2. Tablets 3. Extrudates 4. Grits	2500 (Max.)	300° 575° C	Up to 30 kg/cm ²	-	High Activity. Tolerant to sulfur poisoning. Excellent mechanical strength. Bulk density 0.9 to 1.4. Crushing strength 1 to 14 kg/cm ² .	
3.b) L.T. Shift	CD-LL-21	Copper Oxide Aluminium oxide Zinc Oxide	Pellets	1800 to 2000	190° 270° C	Up to 3 kg/cm ²	- 2 years	High activity. Bulk density 1.4 to 1.6 kg/litre. Crushing strength 6 to 7 kg/cm ² .	
4. Methanation	CDM-15	Nickel Oxide 32% - 68% Remaining Carrier Binder	Tablets Rings	6000 (Max.)	205° 540° C	Up to 30 kg/cm ²	- 2 years	Excellent Thermal stability. High degree of fluidity with resistance to inlet temperature content. Bulk density 1.0 to 1.2. Crushing strength 50 kg/cm ² .	
5. Ammonia Synthesis	-	Doubly promoted Magnetite	Granules	-	305° 500° C	-	-	4 years	

ANNEXURE - I

High temperature shift converter
Catalyst changing.

<u>Sl.No.</u>	<u>Activities</u>	<u>Duration hours</u>
1.	Load reduction, shutting down and isolating from other sections.	2.0
2.	Depressurising the system	6.0
3.	Slip plate removal for making N ₂ connection for purging	2.0
4.	N ₂ purging and cooling down the converter to make the converter free from CO and H ₂	24.0
5.	Slip plating job for isolating the converters	12.0
6.	Removal of Desuperheaters	6.0
7.	Opening of Manholes	4.0
8.	Quenching with water hose (after breaking) the brick lining of manholes	12.0
9.	Catalyst discharging	36.0
10.	Cleaning inside, grate and wire mesh with water	12.0
11.	Charging of catalyst	36.0
12.	Fixing up of Thermocouples	2.0
13.	Putting Refractory brick work and closing the manholes	4.0
14.	Boxing up manholes	8.0
15.	Leak testing and rectification of leaks	8.0
16.	Slip plate removing	12.0
17.	Heating with N ₂ upto 150° C temperature in bottom bed	24.0
18.	Heating with steam	8.0
19.	Introduction of gas for reduction of the catalyst	28.0
20.	Loading upto 50%	8.0
21.	Loading upto 100%	8.0
	Total time taken	<u>262 hours</u>

ANNEXURE-II.SYNTHESIS CONVERTER CATALYST CHANGING

<u>Sl.No.</u>	<u>Activities</u>	<u>Duration hours.</u>
1.	Reduction of Load from maximum to 100 kg/cm ² g pressure.	1.0
2.	Continue circulation for bringing down the temperature from 500 to 250°C	3.0
3.	Depressurising the unit below 25.0 kg/cm ² g and started purging and cooling (Oxidation) is not done nowadays, basket is taken out in Nitrogen atmosphere)	20.0
4.	Slip plating	16.0
5.	Opening the head bottom cover	80.0
6.	Opening the other connecting pipelines	60.0
7.	Taking out the basket	8.0
8.	Cleaning the reactor with Trichloroethylene	24.0
9.	Charging of catalyst in spare basket is done simultaneously while other maintenance jobs are in progress	NH
10.	Introduction of spare basket in the converter	8.0
11.	Boxing up bottom cover	12.0
12.	Gland packing at bottom and top	8.0
13.	Testing of bottom cover gasket and gland packing by N ₂	4.0
14.	Boxing up of top covers.	24.0
15.	Final gland packing	4.0
16.	Thermocouples fixing and boxing up	8.0
17.	Leak testing rectification and final testing (include starting up of compressor)	24.0
18.	Reduction with Heater on (In the case of prereduced catalyst and 192 hrs. in the case of unreduced catalyst)	36.0
19.	Loading gradually upto 100%	24.0
Total time taken 1) Prereduced		<u>364 hrs.</u>
2) Unreduced		520 hrs.

PROCESS CONTROL CONSIDERATIONS FOR LARGE AMMONIA PLANTS

1. Fail safe ammonia instrumentation:

Most of the new Ammonia plants employ the single train concept. This design as applied to the large plants has made many of the older multi train plants economically obsolete. On the other hand the simple train plant has an inherent weakness - that is, failure in a single component the "weak link" as it were, may cause a failure in the entire plant.

It is for this reason that no plant is truly "single train". The object thus is to identify the critical elements and reinforce them. Spare equipments are provided in many instances as for example in Boiler feed pumps, hot potash pumps, MEA pumps, starting turbo generators and several other instances. The general rule can be stated that if a particular item is a machine involving rather complex moving parts and if installed cost of a spare is reasonable, then sparing of an equipment is justified as a form of insurance. Therefore, to some extent, most new large scale plants are, in fact, to some degree multi train plants.

Consider now a typical control circuit. The set of components of almost any automatic control circuit fits the criteria of a complex mechanical device and the cost of a spare control circuit is a reasonable figure. Why then are not plants equipped with spare control circuits?

To some extent of-course spare instrumentation is provided. For example, check thermocouples are installed where demand advisable, various overrides are provided on controllers and cascade control systems and alarms and trips are installed in many circuits. Actually, however, this degree of sparing is minimal. Control circuits in general are not spared.

This practice is not a matter of economy. A spare controller would serve the same purpose as a spare boiler for a purpose, it is certain that designers and industry will be searching for critical services. This is not a matter of engineering, but a matter of need for a new technology. A spare part is not a spare operation when a signal is received via the instrumentation circuit, that the spare is needed. To apply this technique to control systems requires a control system which can recognise a failure within itself and which can distinguish between true or false signals.

Present "state of the art" of instrumentation can do the job, but a prohibitive maintenance and training effort would be required. The system of course would be complex involving multiple logic circuits. An attempt to achieve a higher degree of reliability through "duplication" with "the present state of art" hardware, would be self-defeating. The multiplicity of hardware, with the associated logic, would present an operational and maintenance horror.

Use of Computer Control:

Present day computer technology, particularly in the realm of direct digital control, or DDC can begin to approach this problem. The computer could, to take a particularly simple example, undertake the lean solvent flow control task. It can determine the behaviour of the transmitter, the reasonableness of the measurement high and low measurement alarm limits, measurement compensation, set point limits, deviation limits, control behaviour, determine restricted valve output, filter circuit and fluid dynamic noises. At the same time it could recognise the possibility of instrument tubing failure, pump instability or failure, pipe line plugging or rupture, exchanger bypassing and leakage and a host of similar parameters in this loop. This logic in turn would provide output logic patterns through calculation of this

system and adjust released unit operations accordingly. This is possible today, but only costwise it is prohibitive.

Control circuits must remain, for the present we must anticipate instrumentation failures and attempt to soften the blow of such failure by consideration of "fail safe" philosophy. In its simplest form the "fail safe" philosophy requires the proper choice of the least hazardous valve action in the event of instrument air failure. It involves addition of alarm and trip circuits, it sometimes involves the use of override control circuit.

The main objective of any ammonia plant process control system or safety instrumentation is protection of personnel, mechanical equipment and catalyst inventory locked up in processing units. Activation of plant shut down system for any other reason is unnecessary.

Process Control: Temperature, pressure, flow and level controls,
Alarms and continuous analysers.

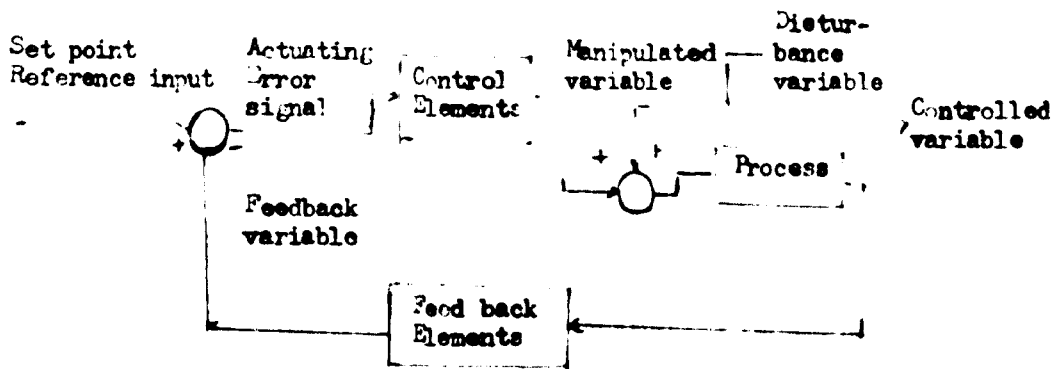
Automatic controllers are widely used in any Ammonia plant to ensure a safe and steady state operation once the plant is put on stream. Most of the operations in Ammonia plant tend to be difficult and tedious hence the need for autocontrol. The three process variables that are to be controlled at every stage of operation of process in ammonia plant are pressure, temperature and flow. Another variable i.e. to be controlled is level. If an hands however skilled they may be are not amenable to this type of operational controls where slight change in one or the other of the variables may upset the whole plant and result in safety hazards both the equipment, material and personnel.

Measurement is a fundamental requisite to automatic, semi-automatic or manual process control. The most commonly used temperature elements in ammonia plants are thermocouples of Platinum-Rhodium, Chromel-Alumel and Copper-Constantan depending on the temperature range. Dial thermometers (mercury filled or gas filled) and Bimetallic (iron-Invar) thermometer are also widely used. As regards to flow measurement most widely used elements are orifices and rotameters. Bourdon gauges Diaphragm elements or certain type of bellows or floats of different types are used as pressure elements. Level measurement is done by floats or level gauges with sight glass.

These measurements at every step is essential for any type of control of any process. This is indicative of what is happening in a closed vessel, a tower, or a reactor etc. They act as guides.

It is to be remembered that while starting, the controllers should always be on manual and only after steady state conditions are

achieved the process should be put on auto. Before taking in line it should be checked thoroughly for proper operation and control. By means of automatic process control system processes can be controlled continuously and precisely to give more uniform and steady conditions without too many upset, thus ensuring continuity of production and also safety of plant equipments and operators at the same time. Some processing units like reformers, amine is converters, Re-converters where reactions are very fast and much too rapid to be controlled manually. They may have run away reactions resulting in unsafe temperatures or they may sink off either due to loss of temperature of catalyst beds. The loss of level in absorber in hot potash system may result in a mishap with high pressure gas backing into the low pressure lines and low pressure Regenerator. The essential idea of any control system is that the process and controller form a closed loop of action and response. There is feed back of information from the output of a process to a controller, which regulates the process in order to hold the output to any desired value. Hence the name "feed back contro"



Block diagram of a feed back control system.

The components of a simple control system can be seen from the above block diagram. Most of the control systems in large ammonia plants are based on mainly pneumatic system. There are some areas where

electric and electronic control systems are used. In the pneumatic system air is supplied to the diaphragm of a control valve. In most control systems this pressure will be in the range of 2 psig to 15 psig range, standardised by agreement between instrument manufacturer and users. There are some special systems which use other ranges.

Mode of control of any control valve whether it is for pressure, temperature, flow or level, in essence is the same. Only difference is in the sensing element or the measuring element.

Analog Display and Process Data logging and working Alarms.

Ammonia plant, for that matter any plant contain hundreds of equipment and machines. It is normal practice as well as a safety measure to transmit almost all the important and critical process variables to a centrally located control room and get the data displayed on panel mounted gauges or get recorded continuously in graphic charts. These charts will give the status and trend of the process variables. This continuity is important not only for post mortem but also for instantaneous changes that may have to be carried out to keep the process variables in check and to prevent unnecessary upsets and hazards. Moreover sitting in the control room the operator will get an overall picture of the plant as a whole rather than an isolated equipment. Normally alarms are also provided for each of the variables for two limits i.e. minimum or maximum, low or high as a safety measure and to avoid human slips. For example: The level of a tower say carbonate absorber, there is a lower safe limit below which a level should not go so that there is a safe liquid seal in the system. As soon as the level comes to this minimum level, the field located switch will transmit signal and sound an alarm. Similarly too high a level will result in carry over of liquids along with gas streams, may be

resulting in problems in downstream DSA system due to carbonate carry over. To avoid such happenings normally panel mounted alarms are provided which will sound alarms and at the same time the source of the abnormality in the control room itself giving sufficient time even to get to the spot and adjust the abnormality. This is an insurance against instrument failures and human slips, which are common to every ammonia plant. Every critical measurement that is under control will have as an example high level alarm (set at 80%) and low level (20%) alarm. Even in places where there are automatic control valves, these act as a check in case of abnormalities. This is one of the most important things in ammonia plant or any chemical plant.

Data logging is another important thing. This is essential for the post mortem of plant conditions over and after a period of time. Now a days with the advent of computers even digital data logging is possible and is being done even in India. This will avoid human error which are very frequent to creep in.

Continuous Analysers in Ammonia Plant:

Continuous Analysis of composition of product gases from a small side stream sample is important for safe operation of ammonia plant, to control at every stage what is happening. For example, CH_4 in shell unit product gases along with reactor temperature, gives an idea of what exactly is happening, inside the reactor. Similarly, purity of O_2 product and N_2 purity the products of air separation gives us an idea as to what is to be done, whether the process is balanced or any further adjustment is required for example in reflux rates. The rate of N_2 to H_2 in synthesis mixture, whether it is in order whether it requires any change and so on. This shows how important is continuous analysis is for any process. The analysers

that are important in ammonia plant operation are:

1. Methane analyser for gasification units.

This works on infrared principle.

2. O₂ product purity analyser in Air separation units.

This works on the uncommon physical property exhibited to a significant extent only by oxygen. O₂ gas displaces other gases from strong magnetic fields permitting their measurement. This property is known as paramagnetism.

3. O₂ in N₂ that is N₂ product purity analyser

These analysers employ chemical fuel cells. Here principle of rise in temperature due to exothermic reaction of unknown O₂ in small traces is employed.

4. N₂ in synthesis mixture (Ratio Analysers)

These are mostly based on thermal conductivity principles.

5. CO & CO₂ Analysers in traces at the outlet of N₂ wash columns or Methanator outlets.

These again work on the infrared principle. Time lag in relay of data and automatic controls.

The purpose of a control system is to hold the process variables within limits so that the process output falls within the range desired from performance criterion.

If there were no disturbances there would be no need for controls. But there always will be some. Like wise if all parts of a process responded immediately, corrective action instituted at once and the error signal brought back to zero with no time passing, the problem of automatic control would be simple. However processes and all parts of their control systems have characteristics that retard changes and responses initiated by the changes. These delaying characteristics shift the time scale of the response of system element to an input change.

They also cause distortion of the form of the change as it passes through the element. This delay in response in relay of data and automatic controls is called time lag. The problem of process control is to overcome these time delays and to hold the value of controlled variable at the desired level.

Time lag is due to three properties of the system - capacitance, resistance and transport or dead time. Capacitance is the property of the system to adjust to new energy level whereas resistance is the property that will resist these capacity changes. The third property found in processes and control systems and which contributes to time lag is the time required to carry a change from one point to another in the system. This is dead time or transportation lag.

AUTOMATIC TRIP SYSTEM FOR SAFETY

The interest in automatic trip systems stems from the fact that at times they can be the source of operating difficulties. They can cause needless plant trips and as a result contribute to such things as a costly production loss and excessive wear and damage to the equipment. Consequently there is a real interest in limiting the use of trip circuits to only those areas having a need of automatic protection. It is generally believed that these are areas where a rapid response to a process upset or equipment malfunction is required in order to avoid

- 1) Endangering personnel
- 2) Costly equipment damage
- 3) Prolonged unit down time.

Both the concept of rapid action and, either personnel safety or major cost damage are important in determining the automatic trip protection. For example many process upsets would, if left unattended, result in unit damage of major proportions. But the unit response is slow enough so that operators can take the necessary action to recover or make the decision to shut down before conditions reach a hazardous situation. Alarms are provided in these cases and operators are trained to respond to deviations in normal operating conditions.

It is interesting to note that virtually all ammonia plants have automatic trip systems on certain process equipment and the use of circuits on compression equipment is virtually universal. However, except for the compressor oil system trip circuits there is little agreement on which process upsets require automatic trip protection.

It is accepted that certain unique design characteristics in a given plant may require special considerations. But, there are enough similarities among ammonia plants.

Reformer Furnace Trips: We find the following sources of danger:

1. Furnace over pressure: Hazard is that when positive pressure occurs, the hot fuel gas will escape from the furnace casing. This is a personnel hazard and could also result in damage to the furnace itself.
2. Momentary fuel loss: Hazard is that an explosion can occur if fuel is reintroduced without reignition of the burners.
3. Feed loss: Hazard is that secondary reformer catalyst can become overheated and fuse if air flow is continued when feed flow is lost.
4. Steam loss: Hazard is that the primary reformer catalyst can be lost due to coking of feed is not shut off when the steam flow is lost.

Process steam generation: Actuating shut down of process equipment due to a failure in the onsite steam generation is usually used to protect the heat exchange equipment from operating dry and overheating.

Usual trips associated with are

1. Steam loss
2. Boiler feed water supply loss
3. Loss of steam drum level
4. Loss of boiler circulation water flow.

In the case of boiler feed water supply loss and loss of boiler circulation water flow the signal is used mostly to cut in the spare pump and not as a plant trip.

But mostly it is left to operating personnel to take effective action in case of failures as stated above, because some of them may be momentary and may cause unusually large number of trips.

Shell safety shut down system for partial oxidation:

This is a unique system. The important aspect of the shut down operation is that the ratio of oxygen to hydrocarbon be kept

under control at all times in order to prevent (1) high temperature damage to the reactor lining and (2) to prevent explosion and resultant damage to equipment and personnel due to the formation of explosive mixtures due to excess oxygen.

The shut down system is of the electro pneumatic type, where in an electric signal from the instrument detecting an abnormal condition de-energises a solenoid 3 way valve in the master shut down air system, blocking the air supply and causing the system to lose air pressure. Thus each reactor unit is shut down in a manner similar to that occurring on loss of instrument air pressure.

A shut down sequence is initiated automatically upon attainment of any of the following abnormal operating conditions:

1. High ratio O₂/hydrocarbon feed stock
2. Low O₂ flow to reactor
3. Low hydrocarbon feed pressure inlet to feed stock preheater
4. Low flow steam to FSPH
5. Low flow steam to reactor.

If any of the above abnormal conditions is reached by any of the 5 variables, the following events take place.

1. Oxygen feed is cut off by the quick closing shut off valve.
2. Naphtha feed supply is stopped by the pneumatically operated time delay switch at the naphtha charge pump. The effect of this delay is to prevent O₂ break through from the reactor.
3. The shut down alarm is sounded by the action of pneumatic switch in the master shut down system.

The loss of air pressure causes corresponding 3 way pneumatic valves to block the air supply and vent air from the diaphragms of the corresponding process control valves. In the case of naphtha charge pump the loss of pressure in the system acts on the time delay switch

directly without an intermediate, diaphragm valve. This pneumatic switch includes an adjustment for delaying the shut off of power for as long as 60 seconds. Thus except for the occurrence of a power failure, naphtha feed to the reactor may be continued for a period of time after the O₂ supply is stopped.

Manually operated 3 way valve is provided on the control panel of each unit for starting that unit. This valve by passes the solenoid operated valves in the pneumatic control circuit. As soon as the unit reaches normal operating condition, the valve is manually switched from bypass to the normal running position. In this latter position the shut down system is in service.

In modern Shell plants there is one more safeguard i.e. shut down due to high temperature reactor bed. This is to safeguard against unusually high temperature and explosive situations that may occur due to excessive water carry over with naphtha both for liquid and vapor phase and also to integrate the shut down system in the case of flame out off FSPs when vapor naphtha is the feed.

Feed stock preheater burner control:

Originally the Honeywell control system was there which provided safeguard for any number of burners. The system provides an adjustable preignition purge period, an adjustable trial for pilot and main flame ignition, alarm and silencing circuit. The system provides cut off of all fuel in the event of flame failure thus safeguarding against possible explosions.

But frequent flame outs occurred due to improper mounting of flame scanners and problems with electronic circuit components.

Original honeywell protectoglow system was replaced with fireye system. Location of amplifier control system changed in order to keep it away from hot surroundings of feed stock preheaters.

Flame scanners were relocated for proper viewing. Now they are working fine.

The other trips used in Ammonia Plant are:

1. Loss of secondary reformer air: This is usually used to shut the air valve, probably in order to prevent a surge of air flow when the air supply is required. Depending on the plant design some units are required to protect an air preheat coil by introducing steam upstream of the coil when air is lost. This also serves to keep a positive flow in order to prevent back up of combustible gases into the air line. This trip is normally used to trip synthesis compressors also.
2. Loss of level in CO_2 absorber is used to actuate a trip circuit. Usually rich solution let down valve is closed. This serves to protect the regenerator system from damage caused by loss of liquid seal in the bottom of the absorber and escape of pressurised synthesis gas. It is also actuating shut off of air injection to regenerator so as to protect against a possible explosive condition.
3. A few plants employ a trip of the methanator on loss of CO_2 solution flow. But usually spare pump is cut in with a signal.
4. There is an automatic trip on high methanator temperature to shut down the reactor, to trip the down stream compressor, or both. This protection against runaway reactions catalyst and equipment damage.
5. The fired start up heater in ammonia synthesis loop when used is frequently protected with automatic trip circuits. In this unit a coil rupture can result in a serious fire due to escaping high pressure synthesis gas. There is trip against loss of gas flow and loss of fuel to protect against possibility of overheating and possibility of an explosion caused by momentary fuel failure.

Centrifugal compressor trip circuits:

1. Lube oil - loss of pressure
2. Loss of seal oil - low level
3. High KO drum level
4. High discharge temperature
5. High oil temperature
6. Vibration/Axial displacement
7. Underspeed.

No.1: Is for machine protection. Rapid response to a loss of oil is critical for minimising damage to machine. Universally employed.

No.2: Also universally employed except in air compressors. Trip protection constitutes personnel as well as equipment protection due to the fire hazard associated with loss of seal oil.

No.3: This is to protect the rotor from excessive liquid carry over due to a flooded KO drum.

No.6: Very rarely used. It is used only as an alarm.

Reciprocating compressor trips

1. Frame lube oil loss of pressure
2. High KO drum level
3. Low (or high) suction pressure
4. Loss of cooling water pressure
5. High discharge temperatures
6. Vibration.

No.1 Universal

No.2 Very rarely

No.3 This is universally employed but on occasions also for high suction. This is generally used to protect red overloads caused by a suction pressure excursion into an unsafe region. Low suction trip is provided as a safeguard (1) against liquid or

solution carry over from towers upstream of compressors (?) If the suction is from a gas holder to prevent the failure or crumbling of gas holders due to vacuum creation. Time delay between detection of trip condition and initiation of the trip is of real importance. This amounts a time period during which the plant can return to a nontrip condition without having the trip occur. This is very useful in order to allow for instrument swings, or to allow for operator recovery of a unit if he can respond in time. Alternatively in cases requiring rapid equipment shut down, this further delays the achievement of a safe condition.

A bypass switch in the control room can be useful in avoiding a trip due to an instrument malfunction (if caught in time) or to prevent a unit shut down if the operator judges he can recover given some more time. The risk is that the operator will decide against the trip and then not be able to recover but will instead extend the "unsafe" condition long enough to cause damage. Hence such thought should also be given in having bypass switches.

Reformer and Furnace

1. Fuel loss - low fuel pressure.
2. Loss of draft - low draft.
3. Induced draft fan shut down
4. Forced draft fan shut down
5. Feed loss - low pressure or flow
6. Steam loss - low flow/low steam/feed ratio
7. BFW supply loss - low pressure/flow
8. Loss steam drum level - low level
9. Loss of boiler circulation flow - low flow

Other process equipment

1. Air loss to secondary reformer - low air flow
2. High secondary reformer temperature - high bed/coil temp.
3. High LTS temperature - High inlet temperature
4. Loss of CO₂ removal solution flow - low flow or low pump discharge pressure
5. Loss of CO₂ tower levels - low level absorber/regenerator
6. Methanator high temperature
7. Ammonia converter start up heater fuel loss - low fuel pressure/flame detector
8. Loss of gas flow to ammonia start up preheater - low flow to process gas
9. High temperature ammonia converter start up preheater - High stack/process outlet temperature.

Instrument air system:

The success of high pressure and high temperature plants is mainly due to the instrumentation and sophisticated methods of measurement of process variables. Without their guide everything will go away. Most of the instruments are operated pneumatically.

While for many applications, i.e. general plant equipments, oil is an aid and a lubricant, it is deleterious for the pneumatically operated process control instruments. So do the moisture. Designers seldom face this music. Any modern plant has an installed instrumentation worth about 3 - 5% of the total plant cost. This is a meagre amount when compared with the disaster that can happen to the equipment and operating personnel due to failure of instruments because of the fouling of oil and moisture in pneumatic system.

The oil normally comes from the cylinders of instrument air compressor where it is used as a lubricant. This oil even affects the working of the dehydrating action of the alumina or molecular

sieves. The adsorbent loses its action by the ingress of oil which forms a coating on these solid particles. The net result is instrument air is wet and oily. This spoils or damages many diaphragms of relays and control valves, in addition adding up tremendously to our maintenance efforts and cost. Indirectly this also adds to unreliable performance of the instrument, since in any one of the thousands and odd of pneumatic relays can misbehave and upset everything.

So it is essential that only oil and moisture free air is supplied to the instrument mains. This can be ensured by adding an oil adsorber in addition to the dehumidifiers already existing in any plant, so that only oil, moisture and dustfree air is supplied to instrument system. There should be a regular check on the performance of this system by checking dew points regularly. Any malfunctioning in this system is to be rectified immediately.

PRESSURE RELIEVING SERVICES

These are safety devices mounted on lines and vessels so as to protect the equipment and operating personnel from failures due to excessive pressures building up inside the system. There are several codes which govern the installation and maintenance of these safety devices, the world over. In India we have got the code for Unfired Pressure vessels by Indian Standards institution. As per this code,

1. Every pressure vessel covered by this code shall be provided with a pressure relieving device in accordance with the provisions of this section except where otherwise states as below:
2. When the source of the pressure external to the vessel and under such positive control that the pressure in the vessel cannot exceed the maximum working pressure for the vessel at the operating temperature, a pressure relief device need not be directly provided on the vessel.
3. Vessels that are to operate completely filled with the liquid shall be equipped with a liquid relief valve unless otherwise protected against over pressure.
4. When a vessel is fitted with a heating coil or element whose failure might increase the normal pressure in the vessel the designed relieving capacity of the protective device shall be adequate to prevent this increase.
5. Vessels intended to operate under vacuum conditions unless designed for full vacuum shall be provided with a vacuum break relief devices.
6. Vessels intended for internal pressure which were likely to be subjected to partial vacuum, say due to the cooling of contents shall be provided with a combined pressure vacuum relief device unless

the vessel is designed for full vacuum.

Designs:

1. The protective device used shall be suitable for the conditions of service and shall be adequate for duty.
2. In general relief valves are preferred for vessel protection, but bursting discs or a combination of relief valves and bursting discs may be preferable in certain circumstances.

Relief valves: 1. Spring loaded relief valves are preferred, but other types like valves fitted with a weight or with lever and weight loading are acceptable, provided that they are equally safe.

2. Pilot valve control or other indirect operation of relief valves is not permitted unless the design is such that the main valve will open automatically at the set pressure and discharge to the full capacity, should the pilot or auxiliary device fail.

3. The relief valves shall be designed that they cannot be inadvertently loaded beyond the set pressure (4) The design of valves shall be such that breakage of any part will not obstruct the free and full discharge of the fluid under pressure.

Bursting discs: The use of a bursting disc as a pressure relieving device is preferred. (a) where pressure rise may be so rapid as to be analogous to combustion or explosion so that inertia of a relief valve would be a disadvantage. (b) where service conditions may involve heavy deposits or gumming up, such as would render a relief valve imperative and (c) where even minute leakage of fluid cannot be tolerated.

Bursting discs may be mounted in series with a relief valve provided that (a) the maximum pressure of the range for which the disc is designed to burst does not exceed the maximum working pressure of the pressure vessel (b) The opening provided through the disc after

breakage is sufficient to prevent interference with the proper functioning of the relief valve (c) In case of bursting disc fitted on the discharge side of a valve, back pressure cannot be built up and so influence the lifting pressure of the valve.

Every bursting disc shall have a specified and certified bursting pressure at a specified temperature. It shall be certified by the manufacturer to burst within $\pm 5\%$ of its certified bursting pressure at the specified temperature.

Relief Valve: Every Relief valve shall incorporate permanent marking as follows:

1. Manufacturers identification
2. Nominal inlet/outlet sizes
3. Design pressure and temperature and
4. Certified capacity in kg. of fluid/mixture

Bursting Disc. It should be stamped with the following information:

1. Manufacturers identification
2. Size
3. Bursting pressure
4. Coincident disc temperature and
5. Capacity of discharge

Unless the size of the disc is insufficient in which case the disc shall be contained in a sealed envelope prior to the installation and envelope should be clearly marked with the above information.

A register of bursting disc data should be kept by the user for each vessel protected by a bursting disc with service conditions.

Inspection and maintenance

Safety and pressure relief devices: The safety valve equipment and other pressure relief devices such as rupture discs safety valves etc. should be inspected and tested as frequently as possible but at least once in a year.

RESIDUES FROM AMMONIA PLANT AND THEIR CONTROL

With development of industry, the problems of pollution have come into prominence. There are now strict government controls as to the effluents that one can discharge to from any plant either through ground or to atmosphere. This is particularly important to plants situated in thickly populated areas or metropolitan centres or near rivers and seas to which these effluents are normally discharged through the drainage system.

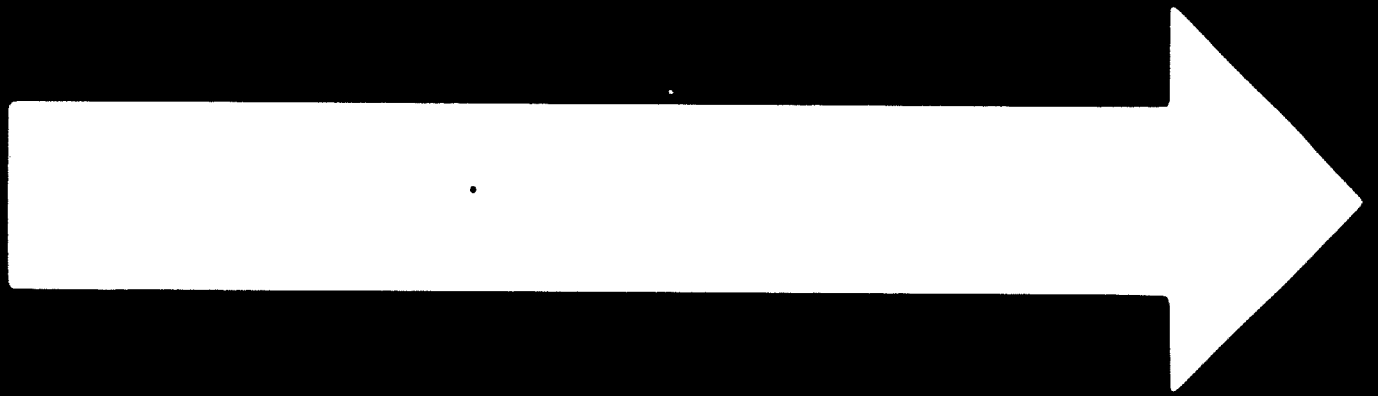
The characteristics of waste water going out and their affect on plant and fishculture and society at large are listed down below and the possible ways of controlling are also enunciated.

Pollution potential of waste waters from Ammonia Plants:

1. pH: The pH of waste waters may vary from acidic to alkaline. This will cause toxicity to fish and other aquatic life and also to plant culture. The toxicity of pH to fish is depends on other factors such as temperature, dissolved O₂ and other anions and cations. The direct lethal effects are not produced within the pH range of 5 to 9.5, but for optimum productivity, the pH should be within 6.5 to 8.3.

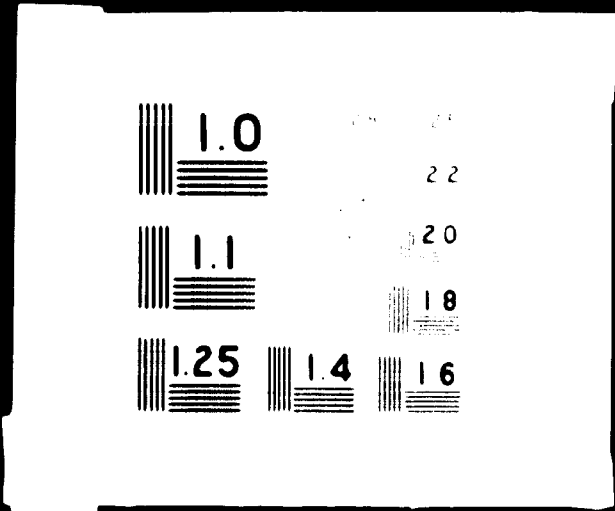
The particular waste waters will, hence, require some neutralisation before discharge to maintain the pH within the permissible range.

2. Ammonia: The toxicity of ammonia to aquatic animals is directly related to the amount of undissociated ammonium hydroxide in the solution which in turn is a function of pH. Thus a high concentration of ammonia ions in water at a low pH may not be as toxic as at a higher pH. The toxicity is due to the reduction in the ability of haemoglobin to combine with oxygen and consequent suffocation of fish. A concentration of 2.5 mg/litre of NH₃ in the



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pH range of 7.4 to 8.5 is considered harmful to fish.

3. Oils: Oils depending upon its nature, affects fish life by interfering with respiration, removing food sources (algae) interfering with spawning, de-oxygenating water and causing direct toxicity.

4. Carbon dioxide: In addition to the above, the waste waters also sometimes contain CO₂. The toxicity of CO₂ to fish depends upon the sensitivity of the fish and other environmental factors. The lethal concentrations vary from 50 to 300 mg/l. Some more delicate marine species may even be more sensitive and may be harmed at concentrations below those mentioned above. On the other hand, toxicity due to ammonia is reduced by the presence of CO₂ presumably due to the lowering of pH.

5. Arsenic: Arsenic is poisonous and toxic to fish in concentrations varying from 1.1 to 2.4 mg/l. The tolerable concentration reported is 0.7 mg/l. The limit for arsenic generally accepted for fish and other aquatic life is 1.0 mg/l.

Treatment Methods:

Preliminary treatment will consist of equalisation for averaging of flow and to even out the variations in the characteristics of the waste waters. This would be followed by neutralisation for pH control.

1. Ammonia: Various methods for the removal of ammonia from liquid wastes are available. These include steam stripping, air stripping, biological treatment, ion exchange, chlorination and simple lagooning.

Biological treatment for waste waters containing ammonia consists of nitrification of ammonia to nitrates by microbial action and followed by denitrification if necessary to NH₃ again also by microbial action.

Arsenic: The treatment for arsenic may be dilution with sufficient quantity of water. The other alternative would be segregation of this particular stream and evaporation to concentrated sludge which is suitably disposed off after filling. It may also be disposed of by burying in sealed containers or by burling out to sea. This is a serious problem for plants having wetcoke process for CO₂ removal.

OIL: Various methods for oil removal are available. These include gravity separation, air floatation, chemical coagulation etc.

Generally simple gravity separation would be sufficient.

CO₂: CO₂ removal may be achieved by aeration or by chemical treatment with lime.

Air Pollution:

In any ammonia plant there are bound to be upsets every now and then. During these periods large quantities of gases containing CO, H₂ are to be vented upstream of equipment and machinery and also during start up and stabilisation of individual units. These have to be burnt in a flare stack before being vented to atmosphere. Unharmful CO₂ & H₂O and usually they are burnt. One other pollutant i.e. possibly present is H₂S, this also to be burnt in flare stack or preferably treated in claus units. So that S may be recovered to avoid sulfur oxides emission.

As can be seen various processes are there for effluent and waste control. As regards to a particular route it depends on the local conditions and will have to be decided case by case basis. However, it is essential that pollution should be controlled so that it will not be hazardous to community at large.

CORROSION AND CONTROLS

Corrosion not only in metals but anywhere else (e.g. non-metals and organic compounds which are used as materials of construction) is a fact of life. As far as industry is concerned it is basically an economic problem which needs a scientific solution.

The characteristic of reversion to its natural state, which is the basic cause of corrosion of any material resulting in damage to the capital equipment and machinery and consequent loss of production, apart from posing safety hazards. To a developing country like ours replacement of machinery and equipment is not only expensive but also difficult, since we have to depend mostly on imports for this purpose, which involve protracted procedural delays through I.C.T.D. Hence the importance and imminent need to control corrosion.

Modern ammonia plants make use of most of the unit process and unit operation and we encounter every type of corrosion to a varying degree. Various forms of corrosion encountered are:

1. General corrosion or uniform thinning down:

This is the most common form and proceeds uniformly over the entire surface by chemical or electrochemical reactions. The metal becomes thinner and ultimately fails. This is encountered in almost every pipe line and equipment.

2. Pitting or local corrosion is the most destructive form of corrosion. In this case most of the surface of the metal shows practically no attack and the corrosion is located in more or less isolated areas. For example the Hot Potash Regenerators and Absorbers of carbon steel, in areas where corrosion inhibitor do not come in contact particularly just above the packing beds. The same can be

overcome by increasing concentration of corrosion inhibitors and also by lining the affected areas with P.V. lining.

3. Inter granular corrosion consists of selective or localised attack at the boundaries of the metallic crystals. The 12-8 Cr. Ni S.S. are particularly susceptible to this type of attack, when they are not properly heat treated or otherwise stabilised.

4. In transgranular or transcrystalline attack cracks due to corrosion proceed through the crystals of metal or alloys not conforming to the boundaries alone. This results in stress corrosion/fatigue conversion.

5. Erosion corrosion/impingement attack occurs when corrosion is supplemented with mechanical or abrasive conditions such as moving fluids or fluid streams. Examples of equipment that suffer this kind of corrosion are pumps, valves, pipelines especially at elbows and tees, heat exchanger and coils for heat transfer. The main cause for accelerated effect due to this type of attack is that the protective film formed as the metal surface is continuously removed due to mechanical factor and is subjected to a fresh corrosion attack. Thus a metal generally resistant in ordinary stagnant condition fails rapidly under these conditions. Eg. Naphtha vaporiser, pipes carrying synthesis mixtures, chrome moly alloy steels are more effective to combat this corrosion.

6. Stress corrosion is the result of corrosion attack accelerated by internal stresses or externally applied stresses (e.g. Temperature pressure etc.). In practically all instances stress corrosion manifests itself in the form of cracks. Almost all the metals are susceptible to this attack. The attack depends in general on three basic factors i.e. stress, concentration and nature of chemical

environment and the temperature. E.G. caustic embrittlement in towers and boilers, failures S.S. lines carrying oxygen and steam mixtures.

7. The damaging effect of simultaneous action of corrosion and cyclic stresses on metals is known as corrosion fatigue, e.g. Turbo compressor impellers, centrifugal pump impellers.

8. Fretting corrosion is caused where slight relative movement occurs between highly loaded surfaces Eg. connecting rods, suspension springs etc.

9. Film corrosion is the type of corrosion which occurs under lacquer paints and metallic coatings in the form of hair like filaments.

10. Weld decay is special type of corrosion of austenitic stainless steel which occur at specific zones away from a weld. In this case the metal adjacent to the welds is subjected to inter granular deterioration or exposure to corrosive medium whereas the metal proper and weld metal are relatively free of the trouble.

11. Sulphur attack: The attack due to sulfur compounds Eg. Fired heaters and gasification reactors etc.

12. Oxidation on exposure to air slowly or in O₂ atmospheres at high temperatures Eg. Rust or scale formation.

13. Nitrogen attack: In surface of pipes tubes and reactors vessels in cracking or reforming plants. The weld is damaged due to intensive N₂ absorption from the mixture of gases. Corrosion of structural materials: & pipelines and equipments: Structural materials are exposed to broadly three types of conditions.

1. Atmosphere (2) Under water (3) Underground.

Corrosiveness of atmosphere: It is observed that that intensity of the corrosion attack in different parts of the country varies considerably, the reasons being the differences in conditions of exposure.

Humidity, temperature fluctuations, rain fall, dew and pollution of a place are the contributing factors for atmospheric corrosion. Corrosion being an electrochemical phenomenon, existence of an electrolyte is essential for the reaction. In case of atmospheric corrosion, the surface of the metal gets an electrolyte through the precipitation process like rain, dew etc. Even a thin film of moisture condensed on the surface due to high ambient humidity and fluctuation in temperature is sufficient to start corrosion. This gets aggravated due to presence of corrosive pollutants in the atmosphere (NH_3 , CO_2 , SO_3 , Cl_2 , HCl , O_2 etc.)

Protective measures:

Common practice is to provide a suitable coating on the surface, plastic laminated steel parts are being increasingly used. Various types of wrappers including plastic tapes laminated with grease and corrosion inhibitors are used for the protection of overhead lines. However, paints and metallic coatings are perhaps the best and widely used for the protection of structures pipelines and equipments. Coating should be impervious continuous and free from pores. The most important single factor is the choice of the primer coat. The primer should contain inhibiting pigments to exercise its influence on the electrolyte, which may diffuse through the finishing coats and under coats. Such pigments include red lead, metallic chromate and metallic lead. The most widely used top coats for steel are aluminium, iron oxide, micaceous iron oxide, graphite, leaded zinc oxide, white lead and titanium dioxide. Where exceptionally corrosive conditions have to be met paints in phenolic resins chlorinated rubber polyester and epoxy resins are used.

Underground Corrosion:

Corrosiveness of the soils vary within vastly wide range from place to place and is generally expressed in terms of its resistivity. Soils having resistance lower than 1000 Ohm/cm are severely corrosive.

Sulphate reducing bacterial in an aerated water logged soil provides highly corrosive conditions.

Corrosion in underwater surface mainly depends on the chemical nature of the water like pH, salt content and accessibility of oxygen. Coatings of bitumin, coal-tar or epoxy coal tar give excellent protection. Now a days cathodic protection is also extensively used for the underground and immersed structures. In the chemical process of corrosion a potential is established i.e. a certain voltage is established between the iron pipe and surrounding soil as a result of this chemical reaction. By application of a suitable reverse voltage, reaction would stop which means corrosion does not take place. This scheme is known as Cathodic protection.

Solving corrosion problems by prevention:

No discussion of corrosion problem would be complete without recognising that by far the best way to solve a corrosion problem is to prevent one from occurring. This can be accomplished best while the new plant is being designed by keeping abreast with technological advances in material specifications, innovation in production processes and from past experience.

- 2) With good cooling tower water treatment.
- 3) With good boiler feed water treatment.
- 4) By providing a larger thickness at bends and tees and joints and at the most fragile points.
- 5) Lining equipments.

- 6) Use of inhibitors during solvent handling processes.
- 7) Avoiding as far as possible the number of bends and tees and elbows. Preventive measures: regular checking of thickness of pipe lines and equipment by non destructive testing methods like ultrasonics and radiography and identifying the critical areas in the plant and keeping a record of measurements and comparing them with the actual thickness. Corrective action wherever is necessary should be effected. Such as replacement of the affected portion of pipe or finding out better material of construction etc. In the case of equipments the affected portion should be either lined or repaired or reinforced wherever is necessary as a stop gap measure/ permanent measure. The causes for the occurrence of these defects are to be identified and possible corrective measures to be devised to overcome the problems.

Stress corrosion: Stress corrosion is the result of corrosion attack accelerated by internal stresses or externally applied stresses. The attack depends in general on the three basic factors, i.e. stress, the concentration and nature of corrosive environment and the temperature. This usually appears in multiples or families of cracks. This also results usually and predominantly as a result of residual stresses. This is of considerable concern because of the possibility that a stress corrosion crack, growing to critical dimension in the wall of pressurised equipment, would propagate in a fast drastic mode with catastrophic consequences.

Stress corrosion in Hot Potash Units:

All the research with Hot Potash Units have shown that cracking only took place when the corrosion potential exceeded certain values depending on the carbonation index. Probes are installed in these units to measure the electrochemical potential.

For example, Air is injected to maintain it at a satisfactory level in vatrocoke units. This air is injected to hold and maintain the pentavalent arsenic level between the arbitrarily set values.

In the Benfield and Catacarb systems mostly Vanadium is used as the corrosion inhibitor. Experience is that there is no in service, stress corrosion cracking in vanadate inhibited carbonate system.

Cracking of towers has been almost invariably associated with welds. There are instances when complete towers have been replaced. This is mainly due to insufficient stress relieving of welds, mainly done in plant sites.

Ammonia system:

Stress corrosion is encountered in units and equipments handling ammonia to varying degrees. Experimentally it has been ascertained that an addition of 0.2% water inhibits cracking of carbon steel and alloy steels. At reduced water levels, there appears to be measurable decrease in ductility showing onset of stress corrosion cracking.

It has been ascertained that stress corrosion cracking in this system is caused by presence of O₂ and NH₃. The presence of CO₂ may aggravate the situation. All the vessels and pipings etc. made for ammonia service should be suitably heat treated. Another area where stress corrosion is encountered in O₂ gasification processes is in O₂ steamlines. We have had several failures of O₂/steam lines. On metallographic examination it has been found that corrosion and cracking was due to chlorides coming with steam. At that time we had only cation exchangers in our demineralisation system. At present we have anion exchangers thus affecting total demineralisation. This problem has not recurred since then.

Quenching and heat recovery units in 24 shift sections is another area where stress corrosion is encountered. This is mainly due to the presence of CO₂ & H₂O which will form carbonic acid and cause stress corrosion of equipment and lines. Modern practice is to go for complete stainless steel equipment in these units.

Aspects of water treatment in corrosion control.

Cooling tower water:

Open recirculating cooling water systems depend for successful operation upon the use of effective water treatment to control corrosion, fouling, microbiological growth and deposition on the heat transfer surfaces. The cost of the treatment versus the cost of repairs and downtime is an important factor in the choice of measures to be adopted. Broadly there are four objectives in treating cooling water each relate to the other and all important to the corrosion engineers. These are:

1. Preventing scale formation on cooling surface.
2. Preventing corrosion of metal in contact with cooling water
3. Preventing fouling of the cooling surfaces
4. Preventing deterioration of the wood.

1) Preventing scale formation: Major scale forming constituents are CaCO₃, Calcium Phosphate calcium sulphate and corrosion products.

Scale formation can be prevented by lime softening or ion exchange.

Scale forming salts decrease in solubility with increasing pH.

Lowering the pH with sulphuric acid helps to prevent scale formation.

2) Prevention of corrosion: Preventive measures can be subdivided into the following (i) Corrosion control by pH and dissolved solid control (ii) corrosion control by the use of inhibitors (iii) Corrosion control by the use of cathodic protection.

Most widely used inhibitor combination is chromate - poly phosphate. Sodium chromate and sodium poly phosphate are used. This furnishes protection against corrosion as well as scale formation. Chromate is a good corrosion inhibitor whereas poly phosphate prevents the precipitation of scale forming salts. The other inhibitors which are in use are sodium nitrite, sodium silicate and sodium benzoate. But experience has shown that chromate poly phosphate combination is very effective in corrosion control. The effective concns. are 40 ppm of poly phosphate and 20 ppm of chromate. pH is controlled by dosing sulphuric acid as and when required. To avoid fungi and algal formation which usually foul the cooler or condenser surfaces sodium hypochlorite or Cl_2 is dosed periodically to give a shock to these organic growth. A residual Cl_2 of 0.5 ppm should always be maintained. Further pH should be maintained slightly above so that water is always alkaline. At pH below 7.0 i.e. in acidic region fungus growth is accelerated. Addition of a quaternary amine also wash out colonies of bacterias.

In spite of best of these treatment occasional fouling of coolers and condenser does exist. Back flushing, aqua blasting roding and chemical cleaning of scales and rust or decayed organisms have to be undertaken from time to time. It is always a good practice to take a side stream and add a coagulating agent and then filter and recycle.

Boiler Feed Water:

Correct design of feed water treatment system and circulating water cycles for steam raising units necessitates careful attention to all variables including materials of construction and temperature and pressure conditions, but especially water quality control.

The boiler feed water should correspond to the following quality.

	<u>Low solids</u>	<u>No solids</u>
Silica, mg/l	0.3 - 0.5	0.3 to 1.5
Silica, max. mg/l	1.0	1.0
pH	5 - 10	10.2
P. Alkalinity mg/l		0
Hardness	0	0
Chlorides	0	0
Phosphates mg/lit.	2 to 4	0
Hydrazine mg/l (max.)	0.1	0.1
Specific conductance	15 to 30	
Specific conductance (Max.)	50	
Dissolved oxygen mg/l	0.02	0.02

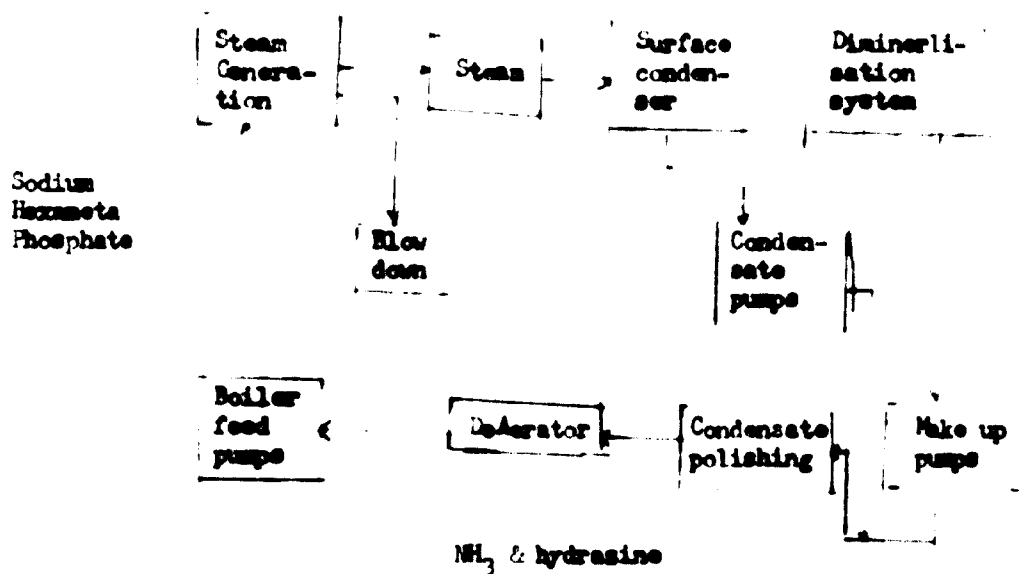
The values under 'low solids' are most frequently applied and represent what now may be considered standard. With proper control of both make up water quality and the effects of leakages if any of undesirable material into the steam cycle through use of condensate polishing. It may be feasible to employ the 'No solids' approach.

In the 'No solids' treatment volatile chemicals NH_3 and hydrazine are fed to the condensate. In the low solids treatment sodium hexameta phosphate is fed to the Boiler water and Ammonia and hydrazine to the condensate.

Of late several condensate polishing system have been developed. One amongst them is a simple sodium cation exchange followed by zeolite softening.

Demineralisation of make up water is normally affected by a primary cation exchanger, a anion exchanger and mixed bed exchanger and a degassifier, in between, before the anion exchanger. The

Degassifier is preferably in two stages and working under vacuum so that traces of dissolved O_2 & CO_2 are negligible. However it is of prime importance that a continuous blow down or bleed is kept in boiler circulating water so that whatever impurities or solids present, they don't accumulate. 5% bleed is a standard for any ammonia plant.



A compact steam cycle in Ammonia Plant.

CHANGING CONCEPTS OF MAINTENANCE

Today increasing pressure is being applied on operation and maintenance people to increase productivity and profitability of course without overriding the safety angle and at as low a cost as possible.

A failure of a single machine or equipment or a breakdown of a critical equipment can shut down the entire plant and it very often does. The loss due to this will run into lacs of rupees. Hence the importance of maintaining the equipment in running condition always. The aim should be to keep the plant going on as far as possible.

A good maintenance organisation should have properly trained people in each trade at all levels. For ones maintenance organisation to be effective, one should have work order system with enough planning, scheduling and paper work to ensure that work is ready as soon as the personnel is available, so that they will be productive for as many hours of the day as possible. With proper methods with the material readily available in hand, the supervisor knowing what to be done, and the worker knowing what to do, the productive working hours could be extended. There should be a history of failure and break downs on each and every machine so that in the long run break downs or failures may be predicted or prevented by predictive or preventive maintenance.

Regardless of how it is done, the preventive maintenance programme must have,

1. Regular inspection and record of plant equipment and facilities. During this, conditions that may cause equipment failure or damage to the machine may be noted. For this plant operation and maintenance men

has one advantage of seeing the machine running.

2. Necessary maintenance either in running condition if it is possible, or scheduled and well planned stoppages whenever it is necessary to correct such conditions. Unscheduled break downs and failures are an operators nightmare and are also hazardous to both plant and personnel. In addition it will be time consuming since it happens when you least expect. The damage also is much more. There is real sense in the age old adage "stitch in time saves nine" as far as maintenance is concerned.

Substantial savings can be made by concentrating maintenance efforts on

1. The machines which have caused the greatest amount of production down time and break downs and which are dangerous.
2. Those which have cost the greatest amount of money to keep repaired.
3. Those which were inoperative the greatest number of times in the past.

Predictive maintenance:

Things have changed substantially with the arrival of single train, high volume plants. Preventive maintenance in those plants no longer means disassembling equipment routinely, for inspection. Now it means diagnosing the exact condition of each and every part of equipment and plant by means ^{of} on-stream, non destructive testing. The objective is the avoidance of unnecessary shut down. This is called predictive maintenance. There are two distinct fields in predictive maintenance. One is the monitoring of vibrations in running machinery or rotating machinery such as large compressors, turbines and pumps, fans and blowers. The other field is checking for crack, corrosion and erosion in pipes and vessels.

Instruments that assist in predictive maintenance are

1. Vibration and frequency analysers (Balancing rotating machinery).
2. Metal thickness measuring instruments (ultrasonics and radiography).
3. Locating cracks in metals (dye penetrates).
4. Noise measuring instruments.

Vibration: Frequency of vibration tells, what is causing the vibration and is therefore the most important measure of vibration. By comparing the frequency of vibration to rotating speed and multiples of rotating speeds the particular part causing the vibration and the trouble with that part can be pinpointed. These vibration meters can be permanently mounted and vibrations monitored in control room at a predetermined vibration amplitude.

Ultrasonics & Radiography: The two most widely used tools for the onstream checking of corrosion, erosion in pipes and vessels, detecting of flaws and cracks in materials and inspecting of welds.

Ultrasonic or Ultrasound testing consists of projection of high frequency sound waves into almost any material. Their behaviour is used as a means of judging the properties of material, measuring thickness and corrosion/erosion rates. This technique has developed to a point where even fatigue can be detected early, before the actual damage occurs.

The entire area of non destructive testing is in its infancy. It is visualised that the plant of future will have permanent ultrasonic test stations at all critical areas wired to a central control room. These will be completely monitored to that operator will be forewarned of the impending danger limits. In this manner, all areas of the plant where integrity of operations and safety are of vital

importance would be under constant surveillance.

Contract Maintenance:

Contract maintenance is becoming more and more a normal phenomenon as compared to permanent staff engaged in maintenance work. Admittedly there are areas like repacking of tower, catalyst charging, cleaning of heat exchangers and cleaning of boilers and its tubes and things like that which are labour intensive and do not require such high technological skill.

But maintenance of machines like compressor and pumps require high technical skill. Our country has not advanced to that stage of development where we can get people skilled in these specialised jobs. At the present stage of development the only solution seems to be to provide for adequate number of personnel for maintenance work.

Machinery & Equipment failure prevention.

Any unscheduled and unanticipated event occurring in the operation of a processing plant constitutes a potential hazard. Additionally such events inevitable represent a potentially significant loss, loss from significant damages, loss resulting from loss of production, loss as represented by resulting increased insurance.

However there are many items which are common regardless of the process or plant, which can significantly improve on stream performance. In a simplified form we can discuss these under five broad areas.

1. Properly detailed purchase specification when ordering for materials and accessories.
2. Design audit to check for performance capability, mechanical integrity and adequacy of critical items.

3. Proper engineering analyses of all the machinery and modifications.
4. Incorporation of monitoring system/instrumentation to warn about incipient hazards and dangers.
5. Preventive and predictive maintenance programs for safety of machines and to prevent hazards and failures from their occurrence.

Monitoring instrumentation: It is very distressing to realise that a large proportion of disasters resulting from mechanical failure, would not have occurred had monitoring instrumentation been installed which would have detected and given warning of incipient failure.

In most instances, a machinery component which is in danger of failing gives obvious warning signs. For example the bearing, oil and piston ring, temperature rises, if either component is overloaded or wearing, pedestal vibration and noise levels increase in a turbine or compressor and in many instances, there is a marked deterioration in operating performance.

To a significant extent, mechanical failures can be traced to a high proportion of failures occurring in a few critical or failure sensitive components; bearing, gears, seals ring, piston etc.

To design the optimum monitoring system it is necessary that we correlate data on the way in which components fail under operating conditions, including failure time and overload tolerances. Fig. 1 shows the correlation of design information with regard to detection systems.

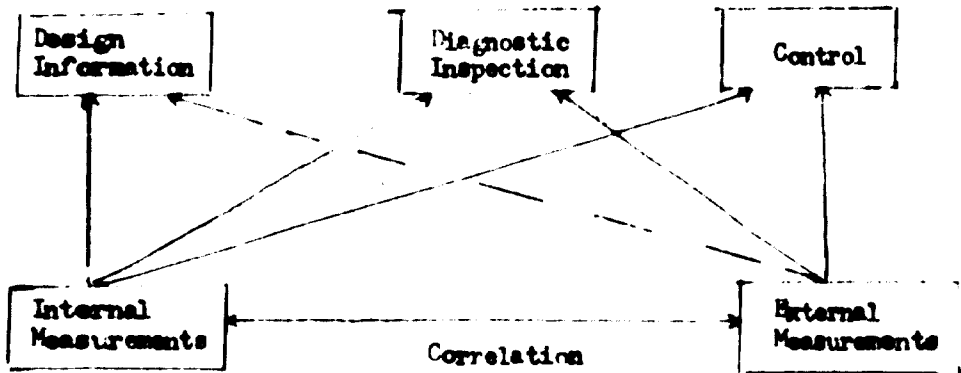


Fig. 1 Detection System.

and Fig. 2 relationship existing between analysis detection and control.

<u>Analysis</u>	<u>Detection</u>	<u>Control</u>
Basic Mechanisms	Component Instrumentation studies	Improved Design Reduced vulnerability
Wear failures		
Vibration & fatigue		Fail safe design
Corrosion & erosion		Materials.
Creep & fracture		
Component failures	Failure transducer	Improved maintenance
Failure Analysis	Development	Diagnostic Instrumentation. Control instrumentation.
Information Analysis		Modify operative conditions. Improved malfunctioning Quality control Test procedures

Fig.2 Analysis, Detection and control.

These components contain certain obvious locations where incipient failure may be monitored in any machine. Research to a very significant extent has provided a basic understanding of the failure process, how deterioration of these components affect system performance, and importantly, what type of instrumentation will detect impending failure. The tools are at hand to develop diagnostic systems which will detect failure and/or deterioration in any given piece of machinery.

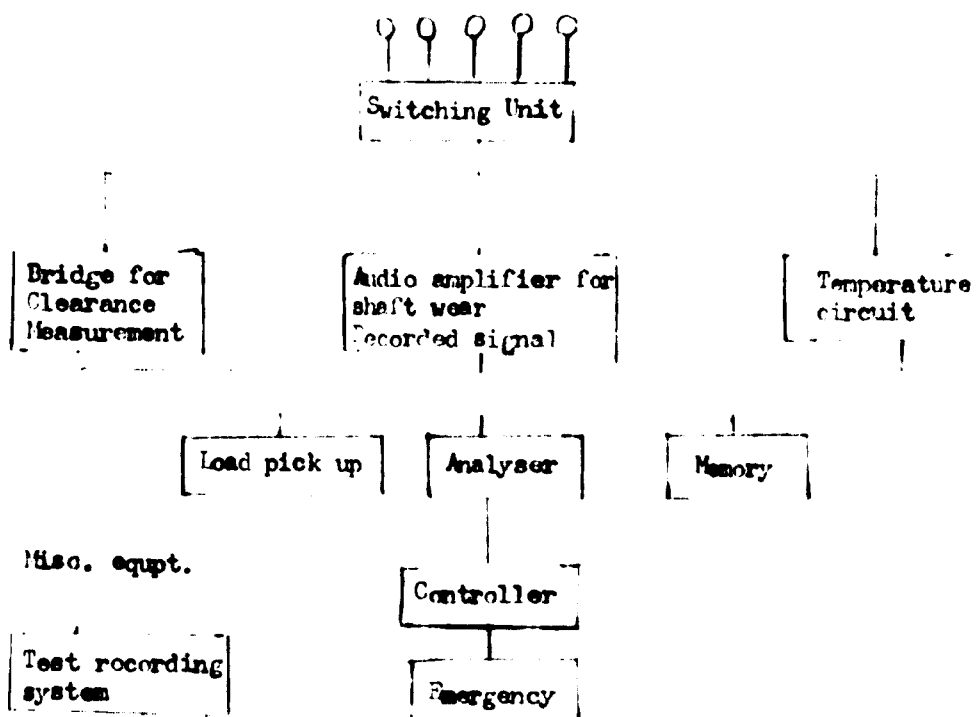
Analysis of an set of failures we can be well aware that it is rare for a failure to occur due to a single cause. In most cases, a complex of interacting circumstances conspire to cause the final catastrophe. It follows that complete answers to the following questions are a fundamental necessity in the development of an adequate monitoring system:

1. What is the sequence of events which lead to failure?
2. What effects would the operating variables have on the failure process?
3. What was the effect of design variables?
4. Which measurements are best able to sense impending failures and what degree of accuracy is required?
5. What steps can be taken to arrest the failure process once it is started?
6. How do impending failures and proceeding failures affect overall machine operation.

Considerable instrumentation is involved in obtaining this information.

Modern instrumentation techniques have advanced to the point where it is practical to measure directly or indirectly a specified number of desired variables. Fig. 3 below gives a diagram

of approach used to measure specified variables for example shaft wear.



Reduction of potential hazards:

In new ammonia units the number of field welded connections is being reduced and welded joints are being relocated to avoid welds in hot, highly stressed regions. Clearances and thermal expansions are being carefully reviewed carefully to reduce the possibility of excessive loading from unexpected restraints or constraints. Metal temperatures at some points where failures have occurred are being reduced in order to increase strength. Use of strainers on inlet lines seems the only answer to avoid damage to large amount of foreign material being found in piping. Vibration monitoring equipment on bearings of high speed centrifugal compressors and drivers though expensive, seems highly desirable. It should be pointed out that this requires good maintenance on the part of the user or it is worthless.

Automatic shut down of high speed centrifugal compressors in the event of rotor axial shift is highly desirable, provided a reliable measuring instrument devices can be installed.

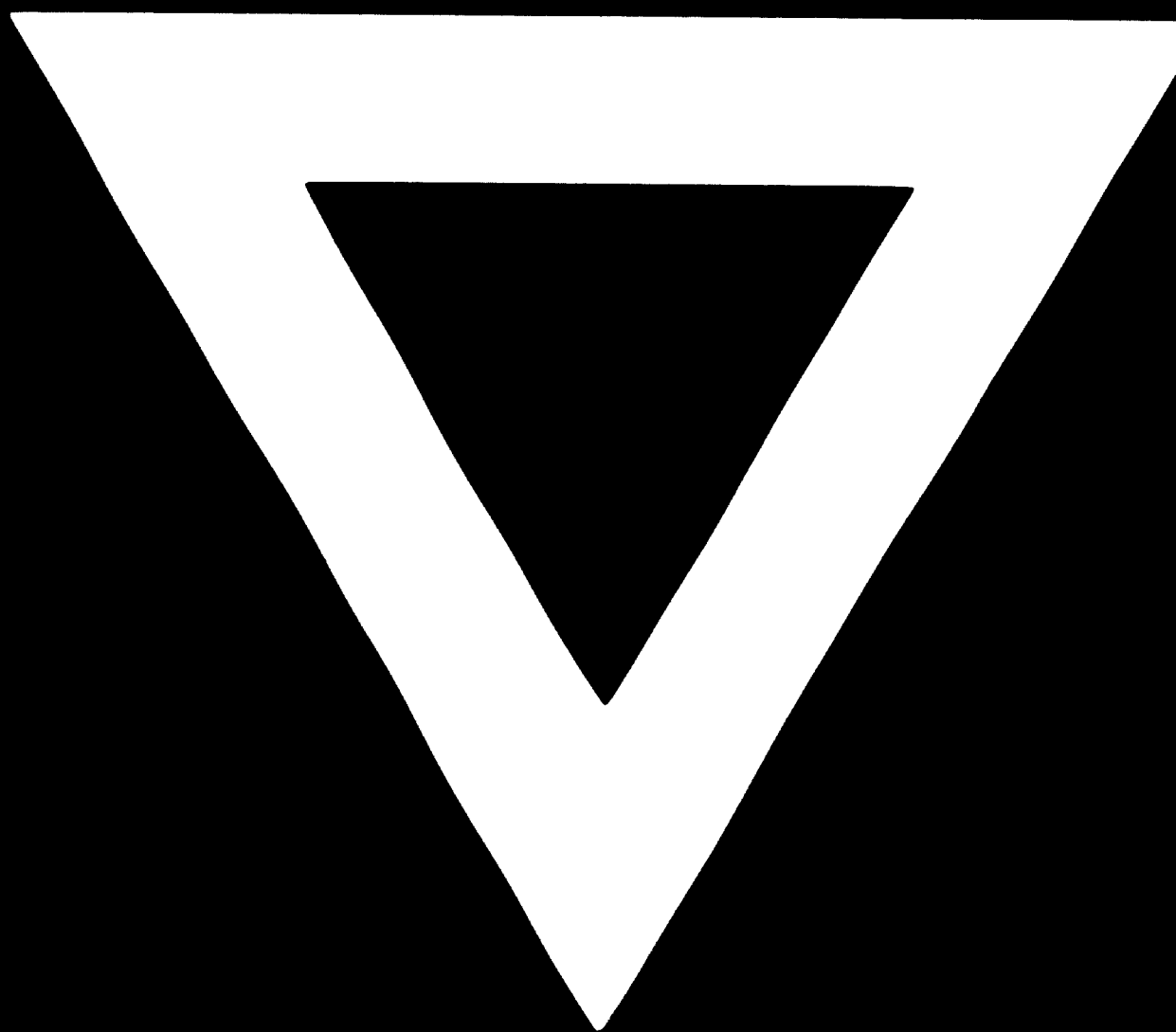
TRAINING AND ITS IMPORTANCE

Failures undoubtedly follow a probability curve determined by the reliability of individual components. It is not possible to say that only good operation will prevent all the problems that have occurred.

Conversely it can be asserted that trained and alert operators can forestall many types of potentially serious failures by early detection of warning symptoms. Competence and experience is of particular importance in the operation of sensitive heat balanced units where events feed upon themselves. A unit operating smoothly can be down in minutes, due to an error with probable damage to equipment or catalyst. On the other hand good operation will often carry a unit through with only machine stoppages and restarts without any major upset.

It is indeed a paradoxical situation that these new and highly automated plants are in a sense more dependant on the human factor for the safe and profitable than the older smaller units. The risks and costs of failures are magnified with increasing plant size and level of operator efficiency should be commensurate.





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