

# SAFETY ASPECTS OF RECONSTRUCTED ICI TONNAGE OXYGEN PLANT

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On 21st April, 1959, a serious explosion occurred in the tonnage oxygen plant at the works of Imperial Chemical Industries Limited at Billingham-on-Tees, England, during the commissioning procedures. The explosion caused three deaths, and extensive damage both to the plant and to nearby facilities, Figure 1. A comprehensive and detailed investigation was undertaken jointly by Air Products Ltd. and ICI, and expert assistance was also provided from independent sources. While the investigation concluded that the constituents of the explosive material were hydrocarbon oil from the lubricating system of the turbo expanders and liquid oxygen from leaking pipe joints in the cold box, a critical reappraisal (in the light of the growing knowledge of the hazards associated with air separation plants) of all aspects of process and engineering design, fabrication and erection, which could be regarded in any way as being potentially hazardous, was undertaken by ICI and APL prior to and during the reconstruction of the plant. At the same time, Air Products Ltd. introduced certain improvements relating to the cycle; this paper, however, is confined to consideration only of those changes which were relevant to safety.

There are several general principles to which all the changes conform. It may be convenient to enunciate these in advance of the discussion of the details:

1. Delineation of the area in which an abnormal oxygen concentration could occur.
2. Exclusion of combustibles from the "oxygen area" at all times (construction and operation).
3. Minimising the possibility of oxygen leakage from the plant.
4. Provision of safe means of disposal of high oxygen concentrations (liquid or gas).
5. Control of concentration of contaminants within air separation system.

## Elimination of turbo expanders

The original plant obtained most of its refrigeration by means of high pressure air let down across an expansion valve, the balance being made up with expansion of 80 lbs./sq. in. gauge nitrogen in a turbo expander. These turbo expanders, which were arranged to drive induction generators through speed reducing gears, projected into the main cold box shell and were oil lubricated, Figure 2. Following the explosion in the original plant, a careful investigation led to the conclusion that the explosion took place external to the plant equip-

ment and that the major combustible component in the explosion was spilt lubricating oil.

The general decision was made that the reconstructed plant should be fenced, with rigid control of hydrocarbons inside the compound. The choice was offered to us by Air Products Ltd. of balance refrigeration coming from either a Freon evaporator system or from turbo expanders, both housed external to the main cold box shell. Either system was acceptable both on technical and safety grounds, but it was considered that the Freon evaporator system offered less chance of oil contamination either inside or outside process equipment. With this in mind, a Freon evaporator system was chosen. The system was placed outside the compound and connected to the cold box by insulated air piping.

The original plant produced both high pressure and medium pressure oxygen gas products by vaporizing pumped liquid oxygen. The low pressure oxygen stream was produced by taking a proportion of high pressure oxygen liquid, letting it down in pressure and vaporizing it in a shell and tube exchanger. It is well known that this type of vaporizer-exchanger has its contamination problems and it was decided to eliminate it in the rebuilt plant. The medium pressure oxygen gas product is now produced by taking a supply of gas from the L.P. column base and compressing this externally.

## Long term analysis of hydrocarbon contamination

The Air Separation Plant site is located in the center of a very large chemical complex. In view of this it was considered important to know the type and level of hydrocarbon contaminants that could occur in the atmosphere of the plant site, and a long term series of tests was undertaken (lasting two to three years) to obtain qualitative and quantitative values for the hydrocarbon contaminants in the atmosphere under all conditions of wind direction and velocity throughout the year. Samples were taken at 200 ft. and 40 ft. levels, the respective heights of our alternative air intake stacks.

Analyses were carried out by drawing a continuous sample of air through soda asbestos towers (to remove carbon dioxide and water) before passing through charcoal packed adsorbers cooled to  $-112^{\circ}\text{F}$ . At the end of each 2-hour sampling period the charcoal tubes were taken to the laboratory and degassed. A concentrated sample of impurities was then analyzed using a mass spectrometer. In general, contamination was found to be lower at the high intake level. Maximum fig-

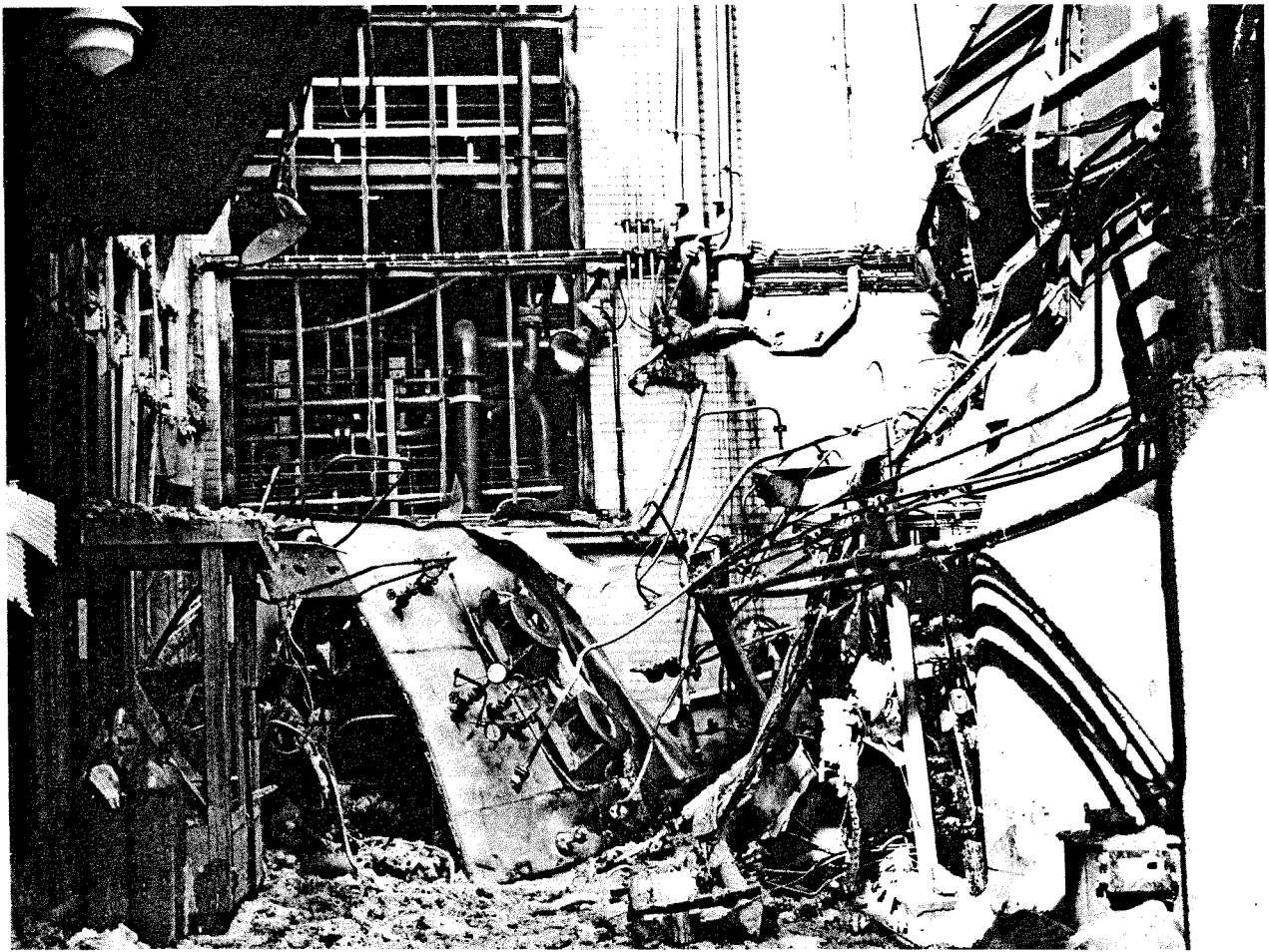


Figure 1. View of explosion area.

ures determined were of the order of :

$C_2H_2$	about 0.1	ppm by volume
$C_2H_4$	about 0.05	ppm by volume
$C_3H_6$	about 3.0	ppm by volume
$C_3H_8$	about 0.15	ppm by volume

Trace quantities of almost all the hydrocarbons were found and a typical figure for total  $C_2-C_5$ 's varied from 0.1 ppm up to 4.0 ppm by volume.

As was to be expected, varying wind directions brought into prominence different hydrocarbon contaminants. In general, contamination at 200 ft. level was a factor of two to three times lower than contamination at the 40 ft. level. The general pattern of contamination did not vary significantly between summer and winter. It showed more variation between night and day. Some measurements taken during still, foggy conditions and during temperature inversion conditions showed an expected overall rise in the total hydrocarbon content.

Coloring all these facts was the proximity of high pressure petrochemical equipment. This equipment may, in an emergency, have to be blown down without prior warning. It is not feasible to flare the vented hydrocarbons, so that considerable quantities of  $C_3$  and  $C_4$  hydrocarbons could be released to atmosphere; the maximum contamination figures mentioned above do not include the effects of such a blow-down.



Figure 2. Turbo expanders after explosion.

## Special instruments to detect contamination

Over the last three years an intensive effort has been made to develop reliable sensitive analytical instruments to measure hydrocarbon contaminants in the process streams to and from the air separation plant. This effort has produced two good analyzers which are worthy of a mention at this point.

Following reports of the success of a flame ionization detector in measuring low hydrocarbon contamination in atmospheric air, it was decided to examine the suitability of such an instrument on hydrocarbon contamination monitoring on the inlet air feed to the rebuilt air separation plant. We were not able to find a commercial instrument that had adequate sensitivity, long term stability and safety. We feel that we have now developed such an instrument and have given it a satisfactory long term field trial. The analyzer consists of a flame ionization detector to which are fed controlled flows (about one liter per hour) of hydrogen and the air to be analyzed. It includes a means of obtaining hydrocarbon-free air for zeroing and a gas mixer for calibration. Low flow controllers were developed so as to achieve adequate long term stability on the most sensitive range of 0-5 ppm of a C<sub>2</sub> hydrocarbon (0-10 ppm equivalent carbon atoms). Response of the analyzer is to carbon-hydrogen bonds present in hydrocarbons and the analyzer is insensitive to water vapour and oxides of carbon, nitrogen and sulphur. This instrument is being developed by ICI for use with chromatographic analyzers and may, in the future, be marketed by W. G. Pye and Co. Ltd.

A chromatograph analyzer has been developed to detect traces of acetylene in the vaporized H.P. oxygen stream from the plant. The chromatograph column is arranged to resolve acetylene from all other likely contaminants. A full chromatogram can be obtained on demand, but not all the contaminant peaks are resolved from one another. The analysis cycle of 30 minutes allows all the possible contaminants to be eluted from the column. This time could certainly be reduced to about 5 minutes by the use of back flushing after receipt of the acetylene peak, but the nature of the plant does not call for this extra complication. Only the acetylene peaks are displayed in the centralized Control Room on a recorder. The instrument has alarms for a high acetylene level. The alarm is given at an acetylene content of 0.5 ppm with an instrument range of 0-3ppm. Since impurities are usually at a very low level and the analyzer is situated some distance from the centralized Control Room, an instrument failure alarm system has had to be incorporated to indicate that zero, or near zero, recording in the Control Room is either due to low contamination levels or to analyzer failure.

This analyzer was developed at our Works and has had very satisfactory field trials for over two years. Similar instruments are now produced by W. G. Pye and Co. Ltd. of Cambridge, England, under licence from ICI, based on this prototype.

## Levels of contamination for shutdown

All operators agree that acetylene is a dangerous contaminant to exist in an air separation plant, but they do not agree on the contamination level at which they should shut the plant down. Shutdown figures appear to vary from 0.5 ppm to 5 ppm of acetylene in liquid oxygen. Some plants, depending on their location, have set danger levels of total hydrocarbons in the liquid oxy-

gen, while one or two have put limits on the concentrations of oxides of nitrogen, although no clear statement could be given of why oxides of nitrogen were considered hazardous in an air separation plant. Only one plant was found which had set definite limits for butadiene contamination.

In view of the risks of heavy contamination of air feed, our plant is being run with the following instruction applying for contamination levels:

(a) 0 to 0.5 ppm Acetylene in the H.P. Oxygen Product

Plant to be operated normally and a changeover of hydrocarbon adsorbers to be carried out every 72 hours.

(b) 0.5 to 1.0 ppm Acetylene in the H.P. Oxygen Product

Changeover of hydrocarbon adsorbers every 48 hours, or more frequently if regeneration cycle permits. Changeover to the alternative air stack (other plant conditions allowing) and see if this reduces the acetylene content of the product.

Take a sample of liquid from the suction to the liquid oxygen pump and have the acetylene concentration determined in the laboratory as a check against the plant analytical instrument.

Check with plants up-wind that they do not have leaks of hydrocarbon or are not venting process gases to the atmosphere for prolonged periods. Inform the plant manager whatever the time of day or night.

It is important under these conditions that the L.P. column level is maintained at or above normal operating level in order to run the reboilers with adequate submergence. If the level is low or tending to fall, check that no excessive refrigeration loss is occurring at the warm ends of regenerators or exchangers.

(c) Above 1.0 ppm Acetylene in the H.P. Oxygen Product

When two successive acetylene peaks on the plant analytical instrument show more than 1.0 ppm and the contamination level in a liquid oxygen sample taken from the pump suction and analyzed in the laboratory confirms this level, then the plant must be shut down and the liquid drained from the columns and vaporized in the dump vaporizer. The columns must not be dumped separately but the liquid in the H.P. column should be all transferred to the L.P. column first so that some dilution of the oxygen content of the L.P. column liquid is achieved before dumping begins and the contamination of the H.P. column liquid is reduced by passage through the hydrocarbon adsorbers.

Before the plant is put back on line a freshly re-activated hydrocarbon adsorber must be in commission. If the shutdown occurs when the spare adsorber is hot it must be cooled with the first air to come through the plant when the plant is restarted.

No other contaminant shutdown limit has been placed on the plant. Despite the fact that the total hydrocarbons in the inlet air analyzer might show a high reading, no change in plant operation will be made apart from changing the air intake stacks, until the acetylene in H.P. oxygen product analyzer shows readings above 0.5 ppm. This has been decided because a number of purification trains exist between the inlet air and the cold box, together with the fact that the solubilities of most hydrocarbons, apart from acetylene and butadiene, are high in liquid oxygen at low pressure (in the range 500 to 10,000 ppm). The operating personnel have been instructed to inform the plant management should any in-

dividual hydrocarbon rise to 10 ppm in the H.P. oxygen product, but no set instructions have been laid down for plant personnel to carry out should this rise occur. Because of our lack of precise knowledge concerning any hazard associated with oxides of nitrogen in an air separation plant, no instruction has been given concerning the presence of oxides of nitrogen in the plant.

During start-up conditions when the plant concentration factor can be many orders of magnitude greater than during normal running, a very close watch is kept on the acetylene content in the liquid burdens, first in the H.P. column base and then later on in the L.P. column base. It is probable that concentrations of impurities will be found in these samples which are much higher than normal when a liquid level first appears, but these should fall off rapidly in a few hours if the cooldown proceeds normally. Higher levels of acetylene contamination can be tolerated in the crude liquid oxygen in the H.P. column as the solubility of acetylene in it is much higher than in pure liquid oxygen in the L.P. column. Up to 5 ppm can be tolerated before action is necessary.

During the cooldown procedure liquid samples are taken initially at two-hourly intervals from the H.P. column until a liquid level shows in the L.P. column. From then on a liquid sample is taken from the H.P. column every four hours and from the L.P. column every four hours, (i.e. alternate columns at two-hourly intervals). This proceeds until the liquid oxygen pumps are in operation, bringing in the plant analytical instrument which samples for acetylene in oxygen product every 30 minutes. From then on routine liquid samples are taken to the laboratory from the L.P. column base once every eight hours.

## Catalytic oxidation unit on feed air

The work carried out on the long term analysis of hydrocarbon contaminants in the inlet air showed that occasional peaks of unsaturated hydrocarbons (notably acetylene and propylene) could occur, together with occasional peaks of propane. With this in mind, together with the fact that acetylene producing and consuming plants and a large Petrochemical Works were situated within half a mile of the air intake, with prevalent wind direction from these plants to the air intake, it was decided to install a catalytic oxidation unit on the inlet air feed to the plant.

An M.S.A. catalytic unit has been chosen, chiefly because commercial experience has been obtained on such units; it will be installed in the autumn of 1962. It will operate at a pressure of 90 lbs./sq. in. gauge on the delivery from the turbo air compressor and is equipped with indirect steam heating facilities to allow its operation at either 338°F or 572°F. No treatment is as yet envisaged for the air which is further compressed to high pressure in an oil lubricated reciprocating compressor, apart from careful reduction of the oil quantities fed to the compressor, use of a Cellulube lubricant or a low vapor pressure Brightstock oil, together with a number of oil filters in the delivery lines.

A prolonged test was carried out on the plant with a small sample of M.S.A. Hopcalite catalyst held at 572°F at 75 lbs./sq. in. gauge. It performed extremely well even with oxidizing trace quantities as low as 0.01 ppm by volume. Virtually all saturated and unsaturated hydrocarbons were oxidized by the catalyst, apart from methane.

## New plant location

The original oxygen plant was built essentially as an integral part of a highly complex centrally controlled facility for the manufacture, by partial oxidation of fuel oil, of ammonia synthesis gas. The air separation plant was located between a workshop, a hydrogen and nitrogen HP compression plant and the centralised control room and sub-station block.

Because design changes in the reconstructed plant would require a larger ground area, (the original site was in any case undesirably congested), and because the hazards can never be entirely eliminated, it was decided that the reconstructed plant should be built on the periphery of the site area, as remote as possible from concentrations of personnel and other equipment. The original and reconstructed plant arrangements, together with the site of the explosion, are shown in Figure 3.

The positions of the LOX pumps and reboilers relative to the rest of the equipment in the new oxygen plant are indicated in Figure 4. It will be observed that the massive screen of the rock-filled regenerators forms a barrier between the cold parts of the plant and the rest of the site. The reboilers, considered the most likely pieces of equipment to suffer a minor explosion, are positioned near the cold box wall facing waste ground outside the plant boundary. The main operating valves have been grouped on the north face of the cold box, screened by the cold box itself from the LOX pumps and the reboiler condensers.

## Philosophy of accessibility

In order that (1) control could be exercised over admission of all materials and potential combustibles into the vicinity of the air separator, (2) only properly trained and authorized personnel should obtain access to the plant, and (3) the immediate plant area should be capable of regular and effective cleaning of combustible material deposited from the atmosphere, it was decided that an enclosed compound, with secure fencing, should be provided around the air separation plant.

This compound, indicated in Figure 4, of necessity in view of its purpose, excluded the regenerator switch valves and their operating gear, and the Freon refrigeration system, since both these sections of plant, by their nature, involved the use of significant quantities of hydrocarbon lubricant.

The only recognized mode of entry into the compound is through a gatehouse which has a multipurpose duty:

1. Control and check of all persons and material entering the compound.
2. Store for clean and oil free protective clothing.
3. Dematching point.
4. Store for degreased small engineering tools.
5. Base for oxygen plant shift operator.

While the plant is in operation, a routine check is made within the compound every four hours and whenever any maintenance work is being carried out for any evidence of oxygen enrichment of the air. Portable oxygen analyzers are used and upon evidence of the existence of an oxygen concentration in excess of 23%, appropriate action is taken.

All operating and maintenance personnel likely to be concerned with the running of the plant have been given comprehensive instruction in the hazards associated with the separation and handling of oxygen, and

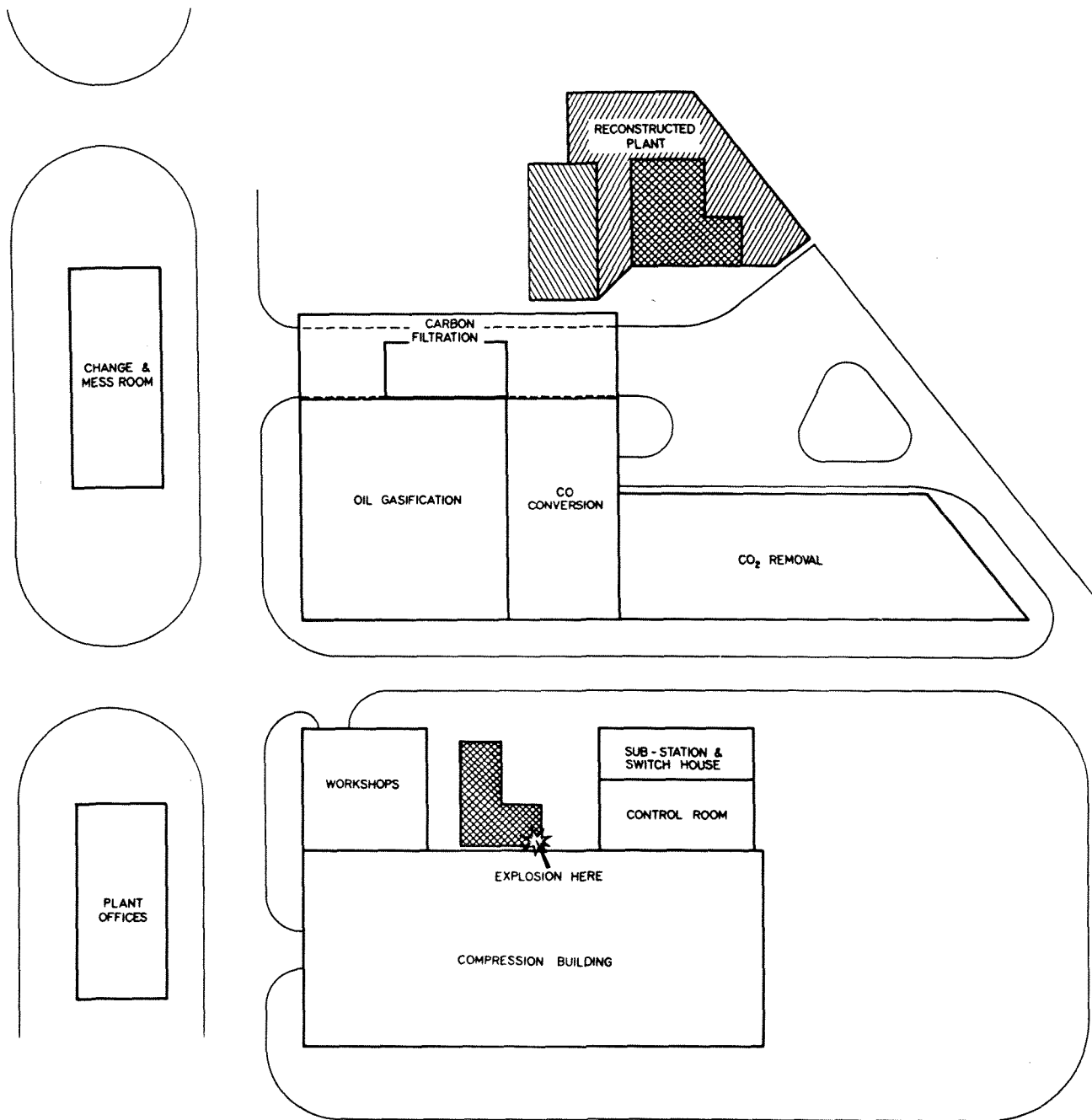


Figure 3. Original and new position of oxygen plant.

in the precautions required to reduce the risks to a minimum.

### Liquid oxygen pumps

In the original plant, the two liquid oxygen pumps delivering at approximately 600 lbs./sq. in. were located together in a "blister" on one face of the cold box; it was leakage from this system which provided the oxidant for the explosion. Because the plant is supplied with one working and one spare pump, some work necessarily must be expected on the standby pump while the plant is still operating. To enable this to be done with minimum risk to personnel, it was decided to mount each pump in a separate insulated box, both being at a distance from the cold box proper.

Piping connections are taken through an insulated trunking. It was decided also that double isolation of the suction and delivery sides of each pump was necessary. In each case one isolation valve is positioned in the main cold box and its partner in the pump box; the intermediate lengths of pipe are each provided with an atmospheric vent and nitrogen purging valves. It is thus possible, after appropriate isolation and purging to work with safety on one of the pump assemblies.

### Cold box foundation design

The original foundation was designed conventionally; Figure 5 shows a typical cross section. The detail design involved many "expansion" joints in the top surface and these had been constructed

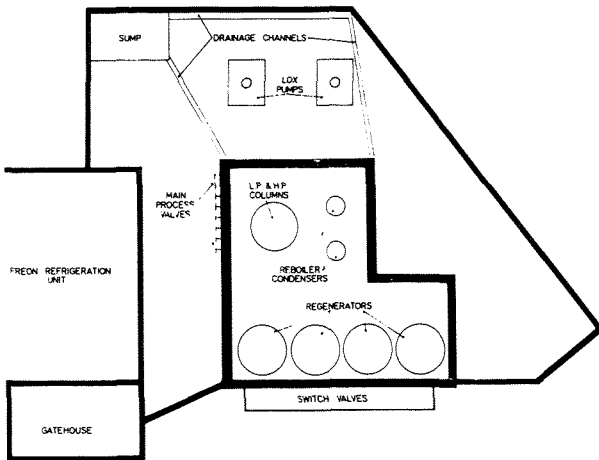


Figure 4. Detailed layout of reconstructed plant.

with a highly combustible bituminous material. Although there was no evidence that this material had contributed to the explosion, nevertheless the presence of any combustible material, or of discontinuities in which combustible material could collect, in this environment, was most undesirable.

For the reconstructed plant, a design was developed which would meet the following requirements:

1. It should be completely self draining.
2. It should have no joints in the top surface.
3. It should have an inherent "tank capacity" sufficient to accommodate the maximum volume of liquid that could operationally be in the air separation plant.
4. It should permit an adequate seal between the cold box wall and the concrete.

Figure 6 shows a typical section of the foundation as designed. The whole of the top section (some 236 cu.yds of concrete) was cast continuously, resulting in a monolithic crevice-free surface for the bottom of the cold box.

A thermal break consisting of self draining 3-in. tubes connected externally to headers and carrying dry nitrogen was cast into the foundation below the distillation section. Provision was also made for the monitoring by thermocouple of the foundation temperature.

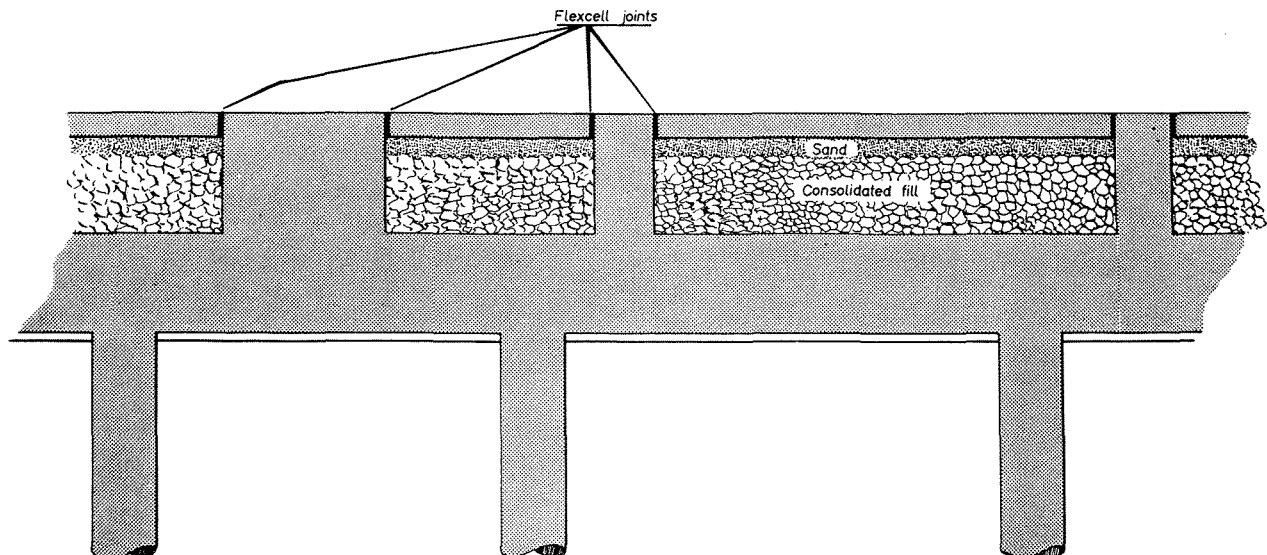


Figure 5. Section of original cold box foundations.

## Disposal facilities

Three problems needed consideration under this heading:

1. Normal disposal of product and waste gases from plant.
2. Disposal of liquid from plant or cold box sump either as normal operation or as emergency.
3. Effects of leakage of cold gas (or liquid) from plant to compound area (e.g. after minor explosion).

The plant is designed to produce 240 T/day of oxygen, two thirds at 520 lbs./sq. in. gauge and one third at essentially atmospheric pressure, together with 370 T/day of pure nitrogen and the corresponding quantity of waste nitrogen. During start-up, at which the consuming plants are temporarily shut down, large volumes of gases of composition which may vary from pure nitrogen to pure oxygen are vented from the plant. While a certain amount of dilution by induction and diffusion from the atmosphere or direct mixing of the effluents can occur, it is still possible in certain atmospheric conditions for the gaseous effluent to form large drifting clouds of air rich in either oxygen or nitrogen. In view of the high nearby concentrations of oil processing equipment and personnel, this possibility could well form a hazard. All vent stacks were therefore taken to a height of at least 10 ft. above the top of the cold box, and so disposed in relation to each other that the maximum mixing of high nitrogen and high oxygen effluents with each other and with induced atmospheric air could be obtained.

Clearly a method for the safe disposal of contaminated liquid from the plant could equally well serve the periodic operational purpose of draining the plant for defrost. The problem, therefore, was that of devising a system for discharging from the H.P. and L.P. column circuits some 20 tons of liquid within one hour, together with the ability to remove safely from the cold box foundation drain any contaminated liquid spillage that may have reached this area. Indirect vaporization could concentrate contaminants, and the vaporized oxygen might leave the apparatus at a temperature well below atmospheric and consequently be liable to flow as a concentrated gas stream at ground level.

The system finally installed was based upon the principle of live steam vaporization, so arranged that

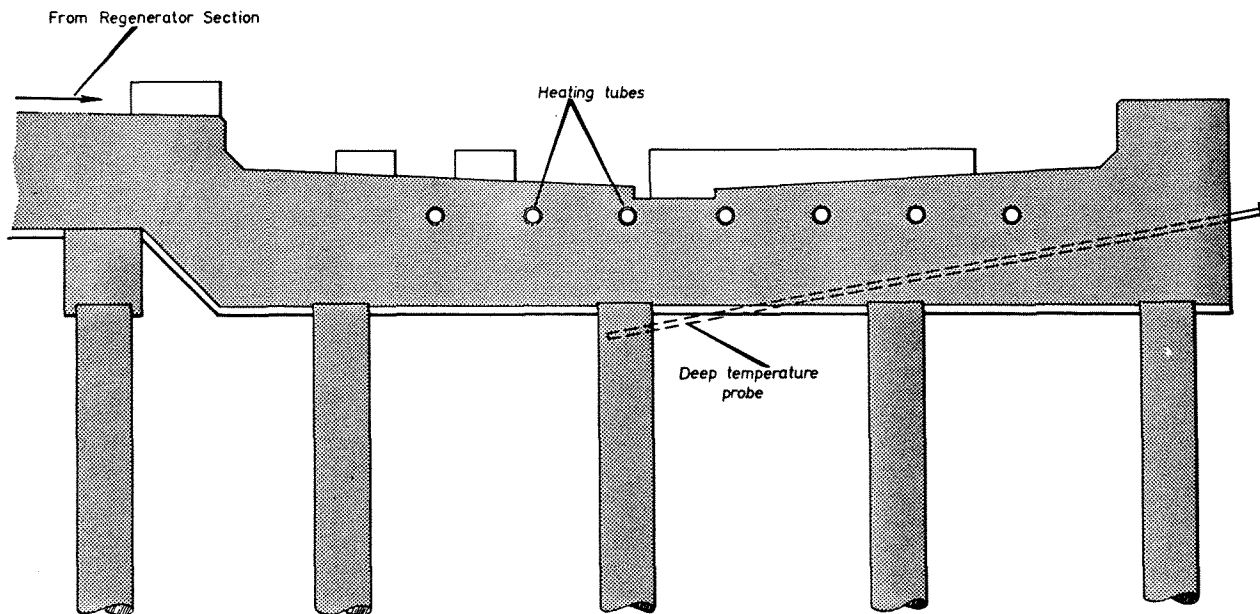


Figure 6. Section of new cold box foundations.

flash evaporation took place in a zone of high steam velocity (thus ensuring simultaneous vaporization of all contaminants with the liquid oxygen) and that the cold liquid feed rate was limited by design to insure that the steam vapor mixture issuing to atmosphere was well above atmospheric temperature and thus dispersed quickly. The apparatus, which was a quite simple adaptation of the steam ejector principle, is illustrated in Figure 7. The apparatus is sited at a level below that of the foundation sump drain, and takes also drain lines for the H.P. and L.P. column circuits; restrictors are provided to limit the flow. Trials have shown that the full liquid burden of the plant can be safely vaporized within one hour.

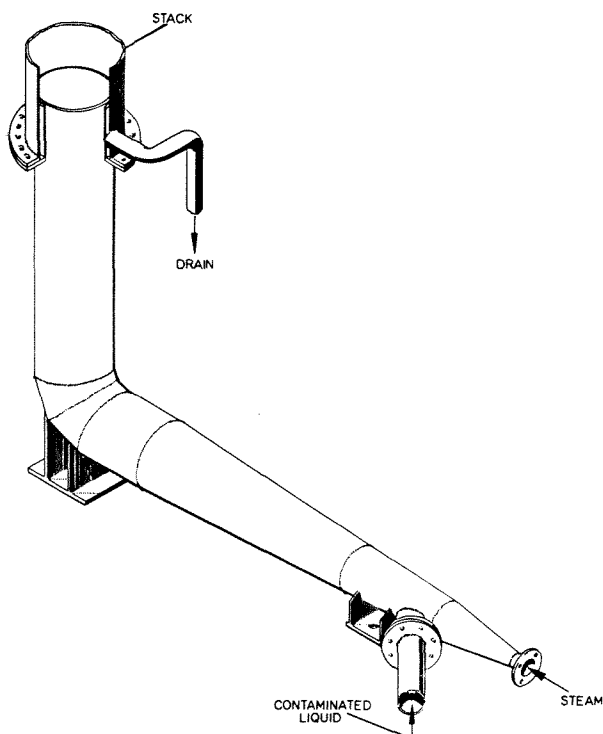


Figure 7. Dump ejector.

Considerable thought was given to the situation which would develop in the event of a serious leak (gas or liquid) of cold oxygen or nitrogen from the plant, as the result of either a mechanical failure or a minor explosion. Even in its new position, the oxygen plant is close to personnel and to a chemical complex using fuel oil and making hydrogen at pressure. It was concluded that the compound should be designed to contain such leakage initially, and to be able to dispose of it safely. The compound was therefore provided with kerbs of sufficient height so that the whole liquid burden of the plant could be contained, a continuous fall to a sump, and a steam system designed to provide a steam curtain at the boundary wall of the compound, Figure 8. It is thus possible to deal moderately safely with even a major spillage of liquid from the plant. Thus, liquid that does not immediately evaporate will drain to the sump, where it can be left; the cold gas from evaporated

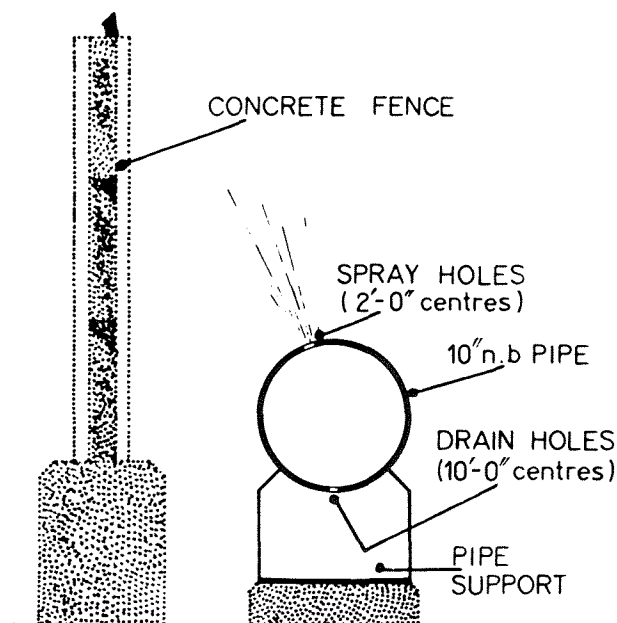


Figure 8. Compound steam fence.

liquid, together with cold gas that escapes from the plant as a vapor, will be contained initially within the compound walls, and then warmed and dispersed at the walls by the steam curtain. By these means, it is expected that the possibility of the flow of cold gas of high nitrogen or oxygen content into areas where there are personnel and combustible materials will be avoided.

## LOX flange joint development

Leakage of conventional flanged joints on LOX service undoubtedly provided the LOX which contributed to the explosion in 1959. Difficulties in maintaining tightness in other flanged joints on cold service had also been experienced, and this behavior was confirmed by discussion with other authorities knowledgeable in this field.

As a matter of policy, therefore, it was decided that wherever possible flanged joints in liquid service lines would be eliminated. However, in a total of 15 cases, many of which were joints between aluminium and austenitic steel, it was found impossible, or impracticable to substitute welding.

It was thus necessary to provide a flanged joint which could be regarded as completely reliable in this particular service environment; as the first step towards this goal, a comprehensive programme of research was implemented on a design of ASA flange modified to provide a means of monitoring leakage across the joint face.

Because of the greater complexity, the mixed joint comprising an 18/8 austenitic steel weld neck flange mating with an aluminium stub and austenitic steel backing flange was selected for the test programme. An insulated test rig was built, so arranged that the joint could be pressurized to 100 lbs./sq. in. and then shock cooled with liquid nitrogen. Facilities were provided for continuous recording of pressure in the system, of temperature and stress of various parts of the assembly (bolts, flanges etc.) and of leakage rate. The initial work concentrated attention upon a number of factors, each of which required particular detailed examination. These included:

1. Behavior of compressed asbestos fibre jointing under load and at low temperatures.
2. Major influence of temperature distribution radially across joint—leading to determination of minimum joint face load necessary to retain tightness.
3. Significance of overall elasticity of the joint and bolt assembly.
4. Phase change occurring in 18/8 steels at relevant temperatures and influence of stress distribution on consequent volume changes.
5. Trials of other types of gasket (e.g. Metaflex, O-rings, U-rings) and of varying thicknesses of compressed asbestos fibre.

The design finally adopted is shown in Figure 9. The bolts of high tensile aluminium alloy are tightened to a predetermined extension, so arranged that the joint face loading during a complete thermal cycle will not be reduced to a value below 2 1/4 times that known to be the critical value for leakage.

The final design was subjected to a rigorous test of 20 full thermal and pressure cycles. The resulting pattern of joint face load variation showed that at no time did it approach the critical value at which leakage could occur. Moreover, the cycle minimum value, which fell by approximately 4% during the first few cycles, levelled out to a limiting value some 2. 1/4 times the critical. This is illustrated in Figure 10, on

which is shown also for comparison the corresponding curve for the first joint design tested.

While the experimental work did not include simulation of the effects of the application of pipe system bending moments to the joint, the quantitative data relating to variation in bolt stress (and therefore joint face loading) enabled the effects of applied bending moments in the joint face load to be simply calculated. In the application of the design to the Billingham plant, the calculated bending moments for the piping systems in no case diminished the joint face minimum loading to a value approaching the critical value. Concurrent experimental and development work by A.P.C.I. at Allentown confirmed the deduction that the pipe bending moments would not invalidate the conclusions.

The whole programme of investigation occupied some 18 months and the results added appreciably to our understanding of the behavior of such joints in the simulated service conditions. This brief outline of the development work has necessarily omitted much interesting information. It is ICI's intention to publish full details of the investigation and its experimental results, and it is hoped that a paper will shortly be presented in the U.K. to the Institution of Mechanical Engineers.

For completeness it is necessary to mention that consideration was given to the use of aluminium/stainless steel couplings made by Project Fabrication Corp. of New York. The manufacturer willingly provided samples to us for examination and destructive testing. The specimens were subjected to the most rigorous set of tests that we could devise; these included pressure tests, leak detector tests, radiography, bond shearing tests, "bond peeling" tests, all of which were performed on the specimens both before and after some 400 shock thermal cycles between -310° F and +212° F. In no case was there any evidence of a weakness in the bond that could have led to failure at the rated operating conditions. However, only because of the limited experience available at that time of commercial operation of the couplings, did we decide not to employ the PFC couplings where applicable.

## Booster air compressors

In our plant cycle approximately one quarter of the air feed needs to be compressed to 1,500 lbs./sq. in. gauge. This implies the use of a reciprocating multi-stage, oil lubricated air compressor, bringing with it the possible hazards of fires and explosions in the compressor delivery lines and the ever present fact that hydrocarbon contamination will be introduced into the air stream being compressed.

The old plant had a single, three stage reciprocating compressor with high stage delivery temperatures and a poor valve plate life. Delivery temperatures of up to 300 F were normal, temperatures of 320° F and higher being achieved when delivery valve failures occurred. A good grade of cylinder and gland lubricating hydrocarbon oil was used and no evidence was detected of carbon deposits on the valve plate assemblies or inside the stage delivery lines to the coolers. Nevertheless theoretical considerations show, and world practical experience bears out, the undesirability of high stage delivery temperatures both from the point of view of air compressor operation and the use of the air as feed to an oxygen plant.

There are four possible mechanisms for transport of compressor lubricant, or volatile materials from the lubricant, into the air stream from the compressor.



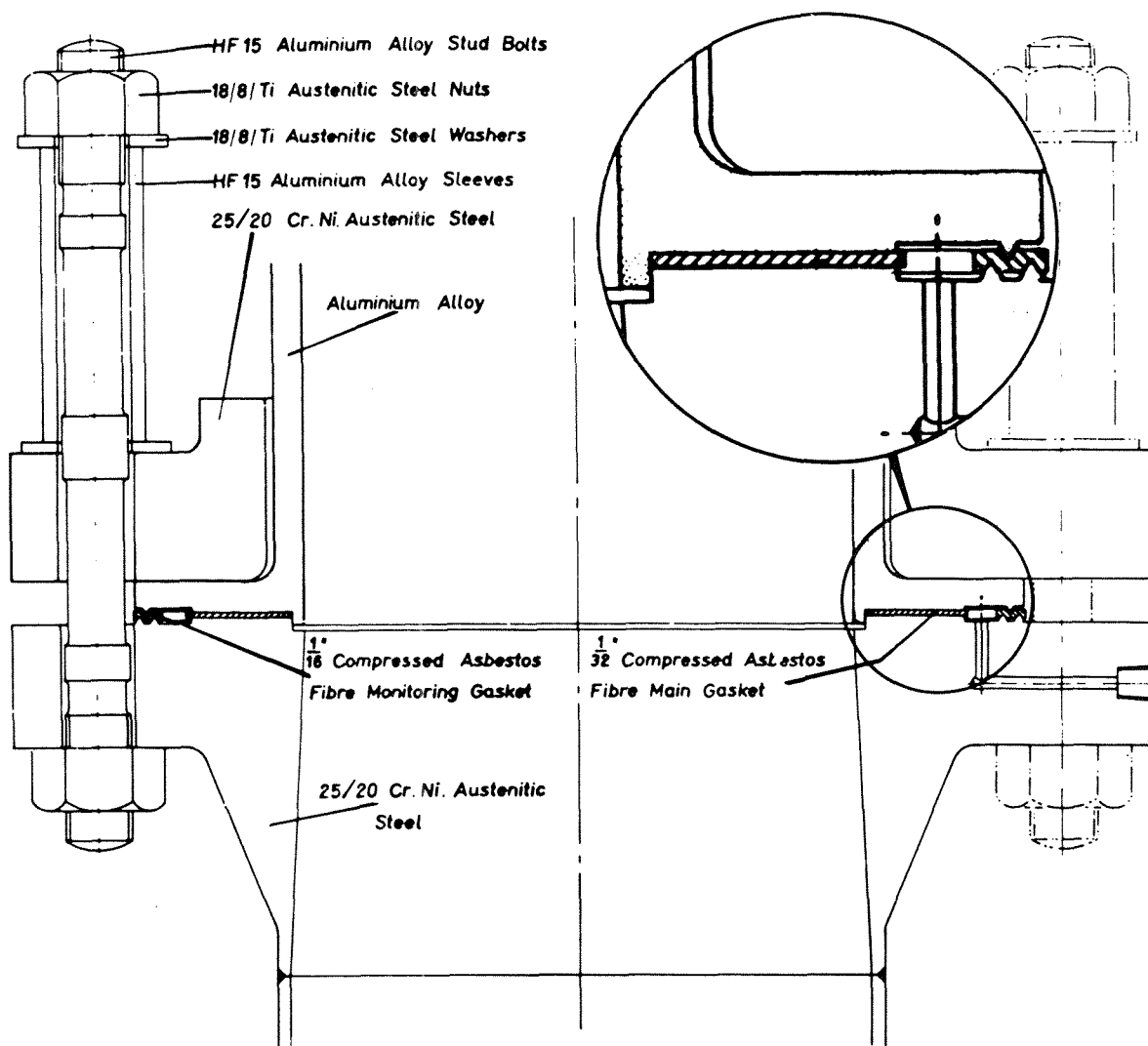


Figure 9. Final design of LOX joint.

1. Stripping all volatile materials (e.g. lower molecular weight hydrocarbons) in solution in the lubricant.
2. Evaporation of the higher molecular weight components of the lubricant at the cylinder temperature and then condensing them to a fog by subsequent cooling.
3. Degradation of the lubricant to form volatile materials.
4. Vapor pressure of the lubricant.

Experiments carried out at our works comparing the relative importance of the first three methods of contamination transfer for a good grade of hydrocarbon oil and for one of the phosphate ester lubricating fluids (Cellulube 220) yielded the following results:

For mechanism one, very little difference was detected between the lubricants, the proportion of dissolved gas being slightly higher for Cellulube. However, no unsaturates were detected with Cellulube. In neither lubricant was the amount high enough to be of direct significance compared with the contamination of the inlet air, being of the order of  $10^{-6}$  gm./M<sup>3</sup> of air when the air and oil under test were both at 356°F.

For mechanism two, it was found that Cellulube was markedly superior, so that at the same operating temperature it gives rise to a smaller carryover of combustible material as fog. Fog transport was of the order of 1 gm./M<sup>3</sup> of air at 356°F for hydrocarbon oil

and less than one quarter of this for cellulube. From this point of view alone it would appear that Cellulube-lubricated compressors are to be preferred for use with oxygen plants.

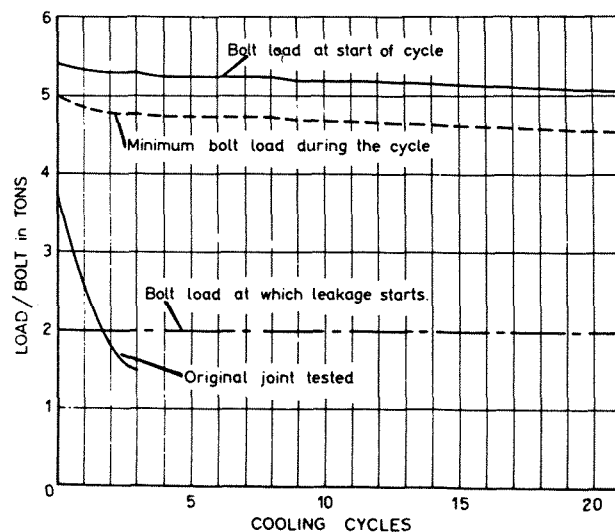


Figure 10. Stress variation in LOX flange bolts during cooling cycles.

For mechanism three, Cellulube was markedly superior to hydrocarbon oil as far as resistance to oxidation and thermal degradation was concerned. It seemed unlikely, under all expected compressor operating conditions, that it would give rise to any volatile decomposition products. For hydrocarbon oil and air at 356°F, decomposition products were of the order of  $10^{-2}$  gm./M<sup>3</sup> of air. No detectable degradation was found for Cellulube and air at 356°F and 482°F. This indicates a marked preference for the use of Cellulube lubrication, both from the point of view of air compressor safe operation and from the reduction of hydrocarbon contaminant in the air fed to an oxygen plant.

In view of these results, together with the good experience reported in the U.S.A. in operating high pressure air compressors with Cellulube, it was decided to convert our three stage air compressor to Cellulube cylinder and gland lubrication. In order to prevent upsets in the oxygen plant caused from compressor valve maintenance, a second H.P. air compressor has been installed. This is a four-stage machine, with low-stage compression ratios and consequent low-stage delivery temperatures varying in the range 176°F to 230°F. Its valve plate life to date is good and it is used as the base load machine for the oxygen plant and is lubricated with a Brightstock cylinder and gland lubricant of low vapor pressure.

Careful tests have been carried out on the amount of lubricant vapor passed forward in the air from the compressor, both at the exit from the compressor and at various points down the line to the oxygen plant after cooling and drying treatments have been carried out. These tests followed a period of reduction of cylinder lubricant feed rate to the minimum required to achieve satisfactory lubrication of the machine. The results of the lubricant vapor test on both the Cellulube lubricated and the Brightstock lubricated compressors show about  $10^{-2}$  milligrams lubricant/R M<sup>3</sup> of air from the machine final deliveries, and less than  $10^{-3}$  milligrams lubricant R M<sup>3</sup> air after the drier filters, before the H.P. air enters the cold box. This is equivalent to less than five ounces of lubricant/year entering the cold box.

Tests were carried out to determine lubricant creep along the air pipeline from the compressors, using degreased stainless steel bobbins. After about 1,000 hours operation no visible liquid film was seen on the bobbin walls (using U.V. lamps) 20 ft. downstream of the compressor delivery isolation valves.

## Test procedure for non-metallic materials

Investigation work immediately following the explosion had shown quite clearly that many apparently inert materials (e.g. asbestos) in their commercial conditions could explode violently in LOX. It was therefore decided that for the reconstructed plant, for each application where a non-metallic material was technically necessary, the available materials would be subjected to rigorous testing so that the apparently least hazardous of those technically suitable could be used in the plant. Samples of every material thus considered (and these totalled about 82) were submitted to the following tests:

### 1. Ignition temperature in oxygen gas at atmospheric pressure British Oxygen Co. method

The temperature of the specimen (0.1–0.2 g) is raised by heat from an external source at 36°F/min. in a flow of two liters/min. of oxygen. Ignition (a bright flash) is observed visually, but can only be identified if combustion is sudden and accompanied by an incandescent flame. It is a disadvantage of the test

that volatile components may be distilled off before the ignition temperature is reached.

### 2. Ignition temperature in oxygen gas at high pressure (600/800 lbs./sq. in. gauge) British Oxygen Co. method

A 0.2 g specimen is heated in a furnace contained in a stainless steel pressure vessel to 930°F in 30 minutes in oxygen at an initial pressure of 600 lbs./sq. in. gauge. Ignition is identified by a kick in the time/temperature curve derived from a thermocouple located close to the specimen. Three classes of result are available from this test:

(a) Ignition at T°F (from temperature record).

(b) No measurable ignition up to 930°F—indicating that subsequent visual examination of the specimen showed evidence of combustion, although rate of generation of heat had not been high enough to register on the temperature record.

(c) No trace of ignition up to 930°F—indicating that subsequent visual examination of the specimen showed no evidence of change.

### 3. Detonation probability in LOX

An apparatus based essentially on that described by the Martin Co., Baltimore Division, (in 'Engineering' in 1958) was constructed for this test, employing an impact energy of 70 ft. lbs. delivered to a sample 0.5-in. dia. and 0.05-in. thick immersed in liquid oxygen. Detonation was observed by flash, sound and odor, evidence of any one of which being regarded as a detonation. Considerable difficulty was met in obtaining reproducibility of results, and it became clear that the test was very sensitive to trivial external factors such as dust, or participation in the reaction of the material of the specimen cup. In this latter connection, the material of the cups was changed from aluminium to lead in later tests.

Results were expressed as X/Y, meaning X detonations out of one test on each of Y specimens of the sample. While this does not give a true indication of the mathematical probability of a detonation, it is sufficiently close for the purpose, in view of the other indeterminate variables in the method. It was concluded that this test should not be used in isolation as a measure of the acceptability of a given material, but should be limited to providing guidance only in conjunction with the results of the other tests.

### 4. Gross Calorific Value (Cals/gm = 1.8 BTU/lb) British Oxygen Co. method

The material is pressurized to 370 lbs./sq. in. gauge in oxygen and touched with a hot platinum wire; the temperature rise is recorded and the heat liberated worked out on this basis. In our experience, materials with calorific values over 2000 cals/gm did ignite completely; where the heat evolved was small, the specimen was mixed with benzoic acid (National Physical Laboratory standard) and the calorific value of the specimen obtained by difference.

### 5. Analysis

Chemical analysis is a useful test for confirming that the materials are what their manufacturers claim. It was clearly impossible to undertake ultimate analyses, and therefore the extent of each analysis was determined after consideration of the probable constituents in the material, and particularly those which might be regarded as potentially hazardous.

Obviously no clear cut levels of acceptability for each of the tests could be defined. The procedure followed, therefore, was to consider the results of the tests on all the technically acceptable materials available for the particular application, and to select the one

which appeared, on overall evaluation, to promise the minimum hazard. In some cases, where no generally acceptable material was available, conditional approval was given for use in a specific location only (e.g. where the risk was of only infrequent and minor enrichment with oxygen). In all, some 30 materials were accepted either entirely or conditionally for use in the oxygen plant.

It is known that manufacturers from time to time deliberately or inadvertently may change the composition of their products, without simultaneously changing the commercial description. In order that such changes should not lead us into the position of unknowingly building into the plant hazardous materials, a system of monitoring was introduced. ICI Inspecting Engineers took samples of all such materials at the point of usage, and these were submitted to the same tests as the original qualifying sample. If the results were not substantially in agreement, then the approval was reviewed. This procedure will be applied to all materials of this kind used in the plant in future maintenance operations.

## Cold box insulation

Initially, all available forms of mass insulation (i.e. fibrous and powder) which would give the required thermal performance in the cold box were evaluated. Comparisons were made of characteristics such as composition, analyses for sulfur and carbon, packing density, thermal conductivity, detonation probability and cost; from these comparisons, the choice was narrowed to one of three:

Slag Wool	-	made by Mineral Wool Products
Rocksil	-	made by Cape Asbestos Ltd.
Banroc 511	-	made by Johns-Mansville

Samples of these were subjected to more detailed tests with the following results:

Test	M. W. P.		
	Slag Wool	Rocksil	Banroc 511
Ignition in O <sub>2</sub> at pressure	None up to 930°F	None up to 930°F	None up to 930°F
Ignition in O <sub>2</sub> at atmospheric pressure	- do -	- do -	- do -
Gross calorific value	110	< 100	< 100
Detonation probability in LOX	2/20	1/20	5/20
Analysis %	C 0.26	C 0.09	C 0.06
	S 1.13	S ND	S 0.54
	Oil 0.25	Oil 0.01	Oil 0.25

It is worth noting that the oil content referred to in the Table arises from the general practice of spraying the hot wool during manufacture with mineral oil to reduce the dust nuisance and risk of fiber breakage.

While the results of the detonation tests in LOX must be treated with some reserve, it was concluded that for the particular purpose intended, Rocksil was marginally superior. This material was therefore used in the plant, but, with the full collaboration of the manufacturers, special precautions were taken in the manufacture, packing and shipment of the material to avoid any potentially hazardous contamination:

1. The mineral oil sprays for dust suppression were isolated.

2. The plant was first washed thoroughly with a solvent and then with water until a check by U.V. light showed no evidence of oil contamination.

3. The manufacturing run was started and samples of the Rocksil were taken and analyzed for oil content. When the analysis showed an "organic content" e. all organic matter extracted with carbon tetrachloride) of only 0.009%, the product was considered acceptable and packing was started.

4. For all samples taken during the run, the organic material varied between 0.004% and 0.012%, the average being 0.006%.

5. The material was packed in new polythene bags, which were closed with wire, and transported to site by road in closed vehicles.

## Miscellaneous design details

### INSPECTION FACILITIES

It is believed to be general practice in the building of oxygen plants not to provide facilities for easy inspection of the internals of the vessels in the plant. The eminently desirable trend towards welding all pipework and vessels eliminates the possibility of gaining access to vessels via flanged branch connections. Since experience of operating oxygen plants within ICI is strictly limited, it was decided that, whenever possible, blanked nozzles of appropriate size would be provided to permit easy access to all vessels or at least to vessels representative of groups. This would facilitate periodic examination of the internal surfaces of the plant to assess both the nature and quantity of non-volatile contaminants deposited, and also the rate of deterioration of the materials of construction during the life of the plant.

### LIQUID DRAINAGE

The whole of the plant was examined in detail during the design stage to identify all places where liquid could remain stagnant, either during operation or during shut down. Appropriate steps were taken (e.g. by provision of small drain holes, self-draining pipe runs, flush tubes on condenser-reboiler top tube plate etc.) to make certain that, during operation, in no part of the plant could contamination be concentrated locally and unobserved, and that, during shut down, all parts of the plant could be drained completely of liquid.

### DEAD LEGS IN LIQUID CIRCUITS

Every effort was made to eliminate "dead legs" in which stagnant liquid could evaporate and lead to contaminant concentration; where such situations could not be avoided, for example in drain lines, the isolation valve was placed as close as possible to the active part of the system.

Where normally closed branch lines (for example, to relief valves or to instruments) could rise from the main pipe or vessel, such lines were arranged to rise continuously to the isolation point. Alternatively, an upward loop was provided of sufficient height to prevent spilling of liquid over the top of the loop as a result of pressure surges.

### PIPE LINE STRESSING

The thermal and pressure stresses in all major pipe lines were checked by computer. Acceptance was based on a fatigue criterion, assuming a life of 7000 stress cycles. In certain difficult cases, this was relaxed, but in no case below 1000 cycles, a linear extrapolation on a logarithm scale being used to derive the equivalent permissible stress.

## STATIC EARTHING

The cold box structure was positively earthed by means of earthing rods. Each major vessel was itself connected electrically to the structure by aluminium strip, following standard British Oxygen engineering practice. It is interesting to note that although it does not appear to be directly applicable to the conditions existing in a conventional air separation plant, the Cryogenic Engineering Laboratory at Boulder had found that in LOX containing solid particles and flowing through an orifice, potentials of up to 1000 volts could be produced.

## TOLERANCE ON FLANGE ALIGNMENT

Quite apart from the cases of the special LOX flanged joints specific tolerances were agreed for the alignment of all flanges in the system. These laid down that:

(a) Flange face should be perpendicular to axis of pipe within an angle of  $0^{\circ} 6'$ , i.e. below 0.002-in. per inch of flange diameter.

(b) The divergence between mating flanges with the bolts loosely inserted in the bolt holes but not hardened up shall not vary across the face of the flange by more than .005-in. per inch with a maximum difference measured at diametrically opposite points on the outside diameter of the flange of  $1/16$ -in.

## LOX PUMP MOTOR BEARINGS

Although it was decided that no hydrocarbons should be admitted into the compound, it was concluded that an exception would have to be made in the case of the LOX pump motor bearings. These are "life-packed" sealed bearings, renewal of which is permitted only when the motor has been removed from the compound; such renewal will be carried out on a routine basis, with a frequency determined by the early experience gained of their performance.

## Quality control

Earlier experience had emphasized the difficulty in attaining an acceptable standard of welding in aluminium alloys, Figure 11.

It was therefore agreed that a specification should be prepared detailing the extent and distribution of defects in a welded joint which could be accepted; these limits were related to those in the ASME Code Clause UW 51 (m) 1, 2, 3 and 4, but relaxed to an extent appropriate to their application to aluminium.

While it was agreed initially with APL that a radiographic check covering some 20% of the welded joints in pipes and 10% of those in vessels, would be applied, subsequent experience proved conclusively that, in the case of pipes, 100% radiography was essential if the specification quality was to be achieved.

Engineering inspection provided by ICI to supplement the various manufacturers' own inspection activities, apart from the normal activities of verification of material and workmanship quality and conformity with drawing dimensions, included the following exceptional controls:

1. Particular care to verify that materials of correct specification were used, particularly in connection with different aluminium alloys and their welding filler wire.

This problem was aggravated by the re-use of salvaged material from the original plant, where alloys of different composition had been used.

2. Close supervision of all welding operations, particularly in aluminium alloy materials, involving

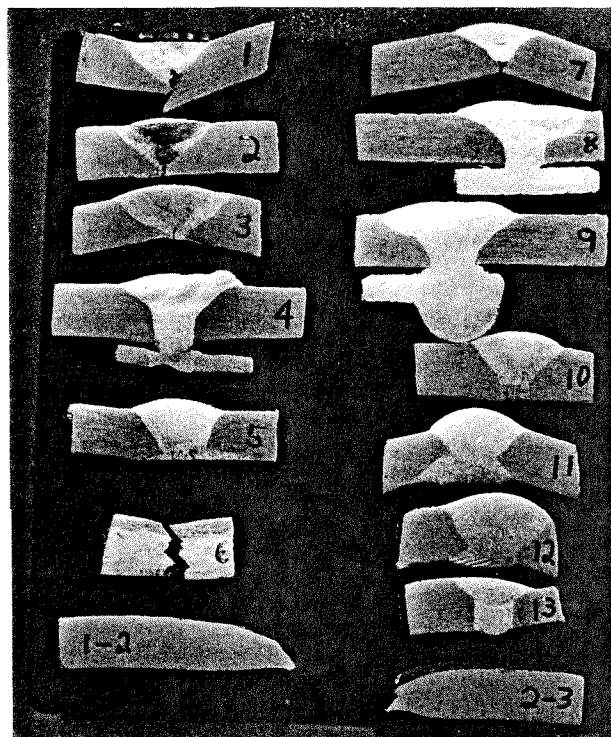


Figure 11. Macrosections of 8-in. n/b  $3/8$ -in. wall aluminium tube welds in original plant.

inspection at several fabrication stages for each welded joint.

3. General cleanliness and degreasing procedures.

## General cleanliness and degreasing

Since it was recognized that an oxygen plant ideally should be in a clinically clean state for commissioning, considerable effort was directed throughout the fabrication and construction stages towards achieving this. Particular emphasis was given to the following points:

1. In those cases where the final degreasing procedure could not be relied upon to achieve the required cleanliness, then at each stage of manufacture the ICI Engineering Inspectors examined those parts continuously throughout fabrication. Such visual examination was supplemented by use of U.V. light. Use of paint etc., for marking was kept to the absolute minimum.

2. Degreasing procedures were discussed in detail with each manufacturer, with particular reference to the type of equipment each was fabricating. Advice was provided by a specialist section of General Chemicals Division of ICI, and specific procedures were agreed for each case. In every case final acceptance was based upon either a direct check on the equipment with U.V. light, a check on the degreasing agent used in the final wash to verify that it was free from hydrocarbon contamination, or in some cases where these were inapplicable, the proper use of a demonstrably satisfactory procedure (e.g. wash of shell side of subcooler with alkaline degreasing agent).

3. As soon as possible after degreasing and dehumidifying, each component was sealed, either at all openings, or by placing in a polythene bag, together with attached certificates of cleaning treatment. Upon

receipt at site, no such item was permitted into the compound before verification of certificate, and confirmation that all seals were intact; in those cases which failed to pass this check, repetition as appropriate of the degreasing procedure was applied.

4. The fabrication of the sieve plate distillation columns provided a unique problem in view of the impossibility of effectively cleaning and degreasing them after manufacture. The following outline of the precautions taken to achieve acceptable cleanliness in each column is recorded, not because of the novelty, but in view of the subsequent events:

a. Before each stage of fabrication within the column shell, the parts to be rendered inaccessible by that stage were thoroughly cleaned and degreased, being checked visually and with U.V. light.

b. Each element to be inserted into the column was independently cleaned and degreased, and handled with appropriate precautions into position.

c. All tools used were degreased frequently and checked, and power tools were used without, or with absolute minimum, lubrication.

d. Personnel wore clean white overalls, overshoes and gloves.

e. The column was covered with a polythene sheet on a framework above the column to prevent adventitious entry of dirt.

f. Continuous ICI and APL inspection was provided.

In spite of all these elaborate precautions, after the L.P. column was completely trayed, the base of the column was inspected again via the inspection manhole, and was found to contain some 12 oz. of mixed aluminium swarf and general debris. This was examined by ICI and found to contain:

Aluminium turnings and powder

Fragments of wood

Crystalline material

Fibers—about 60% cotton, 20% wool and 20% mono-filament.

Application of the test procedures outlined showed:

Ignition in O<sub>2</sub> at 600 lbs./sq. in. 330, 370, 375, 380° F

Ignition in O<sub>2</sub> at atmospheric pressure 520, 480, 455, 492° F

Gross calorific value 4332 cal/gm.

Detonation probability in LOX 6/20

Analysis by weight C 4.0% Oil 0.7%

Less than half the oil could be accounted for by the natural oil content of the several organic constituents.

After the column head had been fitted, the column was blown through with clean air and a further small quantity of debris was collected.

In lifting the column prior to transport, it was allowed to slip so that the supporting cradle hit a building column. The manhole was opened again to ascertain whether the trays had been damaged and a further small quantity of debris was found. After arrival at Billingham, to which it was transported by road horizontally over some 170 miles, it was decided to inspect this column at the three possible points, examining trays particularly at the orientation corre-

sponding with the lowest position in transit. Further small quantities of similar debris were found. (The history of the H.P. column was very similar in terms of nature and quantity of debris.) It was then agreed that an attempt should be made to clean the columns in situ when the plant was completed. This was done by blowing air up through the trays while feeding downwards pure oil-free condensate from our power station condensing turbines, in order to float debris across the trays and down the column, until no further debris was collected. In this way, a further 108 gms (4 ozs) were collected from the L.P. column and 190 gms (7 ozs) from the H.P. column.

While there is no certainty that all debris was removed, no more effective procedure could be devised at the time. This debris was examined thoroughly by optical microscope, and X-ray diffraction analysis, which provided a lot of information, but this was of no direct value in identifying the sources. However, it is fairly clear that, in spite of the extensive precautions and care taken by the manufacturers, a considerable amount of potentially hazardous material had been left in the columns, apparently arising from the protective clothing worn by personnel, from plywood boards used as platforms on the trays and jacks, and from the new hemp rope on a pulley block used for handling components into the columns.

#### 5. Control of admission of contaminants to site

Plant equipment entering the site was examined as indicated at the same time any visible external contamination was removed.

A specification was prepared to define the safeguards to be adopted during the construction period to ensure that the complete plant would be as free as possible from any hazardous materials. This specification postulated three states of construction.

1st stage—To include all site preparation work and construction of foundations and compound. No special precautions except general supervision to ensure that no avoidable contamination occurred.

2nd stage—Complete plant erection including pressure testing and painting, but excluding cold box insulation. For this stage, a fence was erected round the construction site, with limited access only for construction and plant equipment. All equipment coming on to the site was examined, construction plant being checked for excess lubrication or other unnecessary contamination, both at entry and while in use. Protection was provided to prevent spillage of oil etc., on to the concrete of the foundation and compound. (Tests were carried out to determine the rate of penetration and methods of removing such contamination in concrete should this occur.) Only personnel actively concerned with construction were allowed on the site. Only grease-free clothing and degreased hand tools were permitted to be used on the site. Although smoking was permitted, matches, lighters etc., were banned from the site.

3rd stage—Final cleandown of plant and placing insulation. Before cold box insulation was started, the external surfaces of all equipment in the cold box were examined with U.V. light and visually. All contamination thus revealed was cleaned off.

There was then a complete check for other contamination such as timber, paper etc. (When the original plant was dismantled, items such as a timber ladder, scaffold planks, insulation sacks, paper etc., were found buried in the insulation; there was strong evidence to support the view that at least one sack had contributed to the energy of the explosion.) No smoking was permitted and all smoking materials—cigarettes, matches, lighters etc., were strictly prohibited.

## Cold box insulation

The Rocksil insulation was delivered to site in wire-tied polythene bags (each containing about 28 lbs.) in closed lorries. Any damaged bags were rejected and sound bags were taken into the cold box before opening by slitting, in batches of 20. The batches were counted by a senior staff engineer who personally verified that 20 empty bags were removed from the box before the next batch was admitted. APL and ICI Inspectors were stationed inside the cold box to supervise the placing of the insulation and to ensure that no pieces of foreign material were accidentally left in the cold box insulation.

All personnel in the area wore clean grease-free clothing, and smoking was prohibited.

## General observations

While from the foregoing it will be seen that every practicable precaution was taken by APL and ICI during the design, fabrication and construction stages of the reconstruction of the plant, in many respects in excess of what was generally regarded as necessary, in order to ensure that combustible materials were eliminated from the plant both during construction and operation, nevertheless, during modifications following observation of an abnormally high pressure drop in one circuit of the plant, a mild steel and neoprene expanding pipe block was found in a pipe line. This, together with the experience with the two distillation columns, and the finding of a mass of cotton waste and some small pieces of wood in a LOX pump suction filter, can only lead to the conclusion that even more rigorous control of contamination entry into the plant than was exercised in the Billingham reconstruction is needed if maximum safety in operation is to be achieved.

Interest will doubtless be expressed in the financial costs of the precautions outlined in this paper. While it is quite impossible to present a true cost, in view of the particular circumstances in which the reconstruction was carried out, it is the authors' view that such precautions (excluding the cost of the catalytic

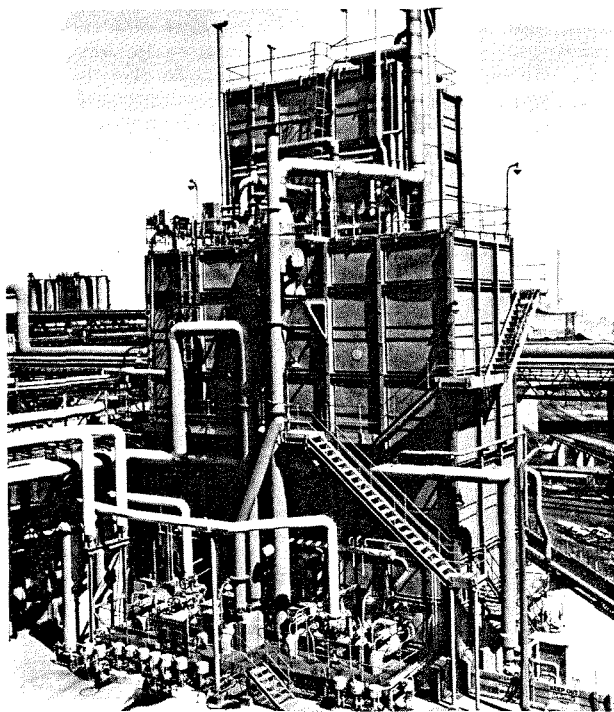


Figure 12. View of reconstructed plant on completion.

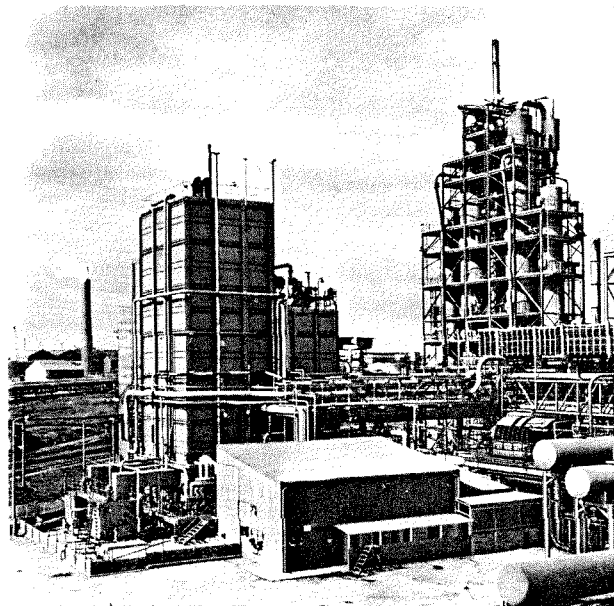


Figure 13. Another view of reconstructed plant.

oxidation unit and of such research and development work as has been necessary) would not have added more than about 2-3% to the cost of a new plant. Whether such additional expenditure is justified must, of course, always remain a matter of opinion: the authors, as will be apparent from this paper, have no doubts on this score.

Figures 12 & 13 show general views of the reconstructed plant at completion; the separate LOX pumps, the dump vaporizer and vent stacks, and the plant compound are clearly seen.

The authors acknowledge gratefully the help and guidance received from their colleagues at Billingham, and from members of the staff of Air Products Limited, in the preparation of this paper; the references to the procedures for the testing of materials for oxygen service are published by kind permission of The British Oxygen Co., and the paper is presented by permission of Imperial Chemical Industries, Ltd.

## Questions and answers

Q. What is your box panelling made of?

OWEN—It's mild steel—zinc sprayed on both sides.

Q. As I recall, it was leakage from your turbo expander oil—external leakage—which ultimately penetrated into your rock wool and then LOX leakage contaminated this and caused the explosion.

OWEN—That is our conclusion. Of course, there is a slight element of doubt but we are satisfied that it is a reasonable and logical explanation which fits the facts.

HEPP—Sun Oil. When your explosion first happened and this explanation came up, we examined our box because the geometrics were very much the same. Our centrifugal turbo expanders are on an overhang. It's trapped in a corner of the box and any oil leakage has a choice of rolling up against the wall of the box, or going out into the roadway and there's always some spillage. We took off the wall of our box and looked in and found, as you have found, the hard way, that our rock wool was saturated with oil. Since we weren't

in a position to put a new floor in, we chose another solution to the problem, which has been satisfactory to us and may be so for somebody else. We built a dam, an all welded steel dam, completely around our turbo expanders and then flooded this area with warm water. We have continuous water running across this box, about three inches deep, and then through an overflow pipe and down out of the plant.

Any drips from the turbo expander are immediately swept away and this system has been in operation since shortly after your explosion. Examination of our box shows that this is keeping the area very clean and no accumulation of oil. It's possibly not as satisfactory but it is a more expedient method if you have an operating plant and you can't rebuild it.

OWEN—I don't think I can make any comment on that. It seems a very sound practical way of getting over the difficulty. We did not throw out expanders as being in themselves inherently dangerous. If we kept expanders, we would have separated them from the cold box, as I think has been done in some plants, and connected them to the cold box through trunking, as opposed to the more conventional method of putting the expander itself within the cold box wall and the load carrying equipment outside.

Q. Our company has an oxygen compressor which has been in service years now with water lubricated bearings. I wonder if these are not regularly available for expanders for these have proven quite satisfactory after the initial difficulties that were encountered on the oxygen compressor unit. My question is, whether or not you've had any experience with a variety of gasketing material? It is made by winding a strip of U-shaped metal with asbestos packing in between these so that in effect you produce a spring which can be compressed and is compressed when making up the joint.

OWEN—We tried Flexatallic as one of the very many gasket types in the experimental rig. It's relatively thick and any increase in thickness increases the relative contractions and expansions where you have a radial thermal gradient through a bolted flange assembly. We deduce from that reason it was not satisfactory in the admittedly rigorous test conditions which we applied.

We worked on the normal compressed asbestos fiber gasket down to 1/32 which gave us the optimum results. We started out with, I think 3/32 and went down to 1/64 and came back to 1/32 as the optimum. So the compressibility of the gasket in itself can contribute adversely to the success of the joint, we found.

Q. This gasket problem is an extremely difficult problem for many of us who are not fortunate enough to have come into the game a little later where all of the new construction is strictly welded construction. We tried Flexatallics in almost every service that you can think of and to us they have not been nearly as successful as an asbestos gasket. You mentioned you go down to a

32nd gasket, and if you go down to 1/32 you have to have perfectly true faces on the two, and I think that this is true that the thinner you get your gasket, the better off you are, but of course the truer you have to have your faces. The opinion that we have come to is that it's nice to weld the place up and this would be the way to go if you could, but if you have to live with gaskets why, asbestos is about the best we've been able to come up with.

We have used a Teflon impregnated gasket with pretty good success. This outfit down in Texas puts out one—I can't think of the name of it right now—it has a very good gasket for liquid oxygen service—Dore Co.—and they do put out one which has been tested pretty thoroughly in our space program.

There was quite an article on that in Chemical Engineering Progress here a while back on the use of this Teflon impregnated gasket.

OWEN—In this connection—I didn't mention it in the talk, although there was a reference to it in the paper—we were aware, in view of the early phases of design of the availability of aluminum stainless steel coupling which is on the market in the United States.

We got some samples of this and subjected these samples to I think about the most rigorous tests that they could ever have been expected to face and as I reported in the paper there, they came out with flying colors—we couldn't fault them. And the only reason why we didn't use them is that their service life under commercially operating conditions was at that stage, short. And against the background of our rather sensitive feelings at that time, we decided to stick to something that we developed ourselves and the performance of which we knew from our own research work. But two years have gone by now since then and I think our decision might well be different if we were faced with the same problem today.

Q. I agree that the use of welded aluminum is better than using couplings or putting yourself in a position where you need a gasket. But I'd like to comment first that a weld is not a one-way street out of trouble. We had an accident caused by a weld failure during the draining of a cold box and the result in our company has been, of course, strict adherence to inspections during welding procedures.

What was the reason for using this type of construction rather than welding when you went back with your lines?

OWEN—We set out to eliminate mechanical joints as far as was practicable. There were 15 left in the plant which, in the main, were connections between steel and aluminum which we decided to retain with an improved form of mechanical joint. The remainder of those 15 were connections to control valves where we wanted to be able to take them out without cutting a line. But I'm entirely with you—a completely welded job is the answer, provided your welding is demonstrably satisfactory and that, in itself, is a problem.