#### July CEP feature



A big session gets underway and an alert and anxious group—all eyes and all ears — appears to be taking everything in.

# .... the operators report on SAFETY in air and ammonia plants

In a CEP exclusive, chemical engineers in air and ammonia plants analyze and discuss problems of actual case histories of full-scale operating and maintenance experience involved in the safe operation, maintenance and expansion of all air and ammonia plants.

This is Part 1—Ammonia Plant Safety. Parts 2 and 3 — Air Plant Safety — will appear in August CEP. Chairman H. E. MAUNE (Mississippi River Chemical Co., Crystal City, Mo.): The first item I'd like to start with concerns ammonia plant operation. It has to do with the high pressure systems and I think we should be mainly concerned with metal inspection of vessels, piping, exchangers and miscellaneous equipment. At this time I will call on Mr. King from Sohio Chemical Co. to introduce the subject of metal inspections.

KIŃC, Sohio, Lima, Ohio: Sohio is relatively new in ammonia operation. However, I took this opportunity to introduce the metal inspection portion of this program since a new operator might have something novel to review. Of interest may be the things observed in the two shutdowns we have had, one carried out in October 1956, and one we completed in August 1957.

In the converter section of the high pressure synthesis loop we have not had any evidence of nitriding, a subject which we will cover later on in these sessions. We have taken the opportunity to open a synthesis con-. verter at each inspection and have actually performed some repair work, or revamp work, on the converter basket.

External forces have come into play in the synthesis section and have left their mark. The necessity for a good water treating method is an example. We are using exceptionally hard water in high temperature service. There is some evidence of sulfur in our well water also. As a result we lost the high pressure coolers on the converter effluent through external corrosion. In our particular process the converter effluent flows through a fin-fan cooler, utilizing air coolers, as the first step, well water coolers for the second.

The major cause of failures in the high pressure section has been vibration. We have gone into great detail to unitize all appendages to our high pressure piping and vessels. The one fire that we have experienced in the synthesis section resulted from the cracking of a nipple at the pressure gauge connection. In our process we have an externally fired heater in the synthesis loop. The failure occurred on the pressure tap at the inlet to that heater. Since that time we have surveyed the entire high pressure section for points where compressor vibration can contribute to that type of failure. We have unitized all of these small appendages through gusseting and framing, making them a part of the piping or the vessel.

To move back through the rest of our system we have in gas purification an MEA system for  $CO_2$  removal and a copper solution process for CO removal from the synthesis gas. There we are faced with the problem of MEA corrosion in the high temperature portion of the  $CO_2$  stripping equipment. We have gone to the use of Monel at the highest temperature points. At medium temperatures, all have exchangers of 316 stainless steel or 4-6% chrome. Carbon steel is used in the cooler portions of that system. In the CO removal system, we have found that the simplest type of installation is the best for handling the copper solution. Any dead spaces are to be avoided. Remove or weld over anything and everything that might create a dead space. Also, protection should be provided at interfaces in the high temperature portions of the system. The vapor-liquid interface has proven to be a critical point.

The first step of our process is high pressure reforming. We have been faced with some erosion losses in piping. While it is too early to determine the tube life in a high pressure reforming furnace, we have been following the condition closely. Ours is

#### WHO TOOK PART

N. H. WALTON, general foreman, Ammonia Dept., Atlantic Refining Co. An organizer of this session, Walton spent his first seven years after graduating from the U. of Pennsylvania as an operator at Atlantic, gained respect for safety from the grass roots. With the increase in air and ammonia installations, and several recent fires and explosions, Walton felt the industry could gain much from this discussion, found good cooperation.





#### H. E. MAUNE, plant superintendent, Miss. River Chemical Co., Crystal City, Mo.

Also an organizer of this session, Maune spent many years at the TVA ammonia plant at Wilson Dam, has long been known as an expert in the field where he has helped many engineers build and operate other ammonia plants.

- R. Bollen, Dow Chemical Co. of Canada, Sarnia, Ont.
- J. W. Buddenberg, Collier Carbon & Chemical Corp., Brea, Calif.
- R. E. Butikofer, superintendent, Chemical Products Div., Calumet Nitrogen Products Co., Whiting, Ind.
- N. A. Carter, asst. production manager, Grand River Chem. Div., Deere & Co., Pryor, Okla.
- F. E. De Vry, process engineer, Hercules Powder Co., Wilmington, Del.
- A. C. Faatz, process design engineer, Foster Wheeler Corp., New York, N.Y.
- A. J. Gorand, safety coordinator, Sun Oil Co., Philadelphia, Pa.
- L. B. Henderson, Ammonia Dept., Dow Chemical, Midland, Mich.
- **O. A. Holstein**, operating supervisor, Ammonia Dept., Atlantic Refining, Philadelphia, Pa.
- G. E. King, general supt., Sohio Petroleum, Lima, Ohio.
- J. A. Lawrence, USI Division, National Distillers & Chemical Corp., Tuscola, III.
- W. A. Mason, Engineering Dept., Dow, Midland, Mich.
- J. E. Ohlson, project engineer, Pennsalt Chemicals Co., Wyandotte, Mich.
- R. W. Sanders, supv. of chem. operations, The Texas Co., Lockport, III.
- D. L. Stockbridge, Jr., Southern Nitrogen Co., Savannah, Ga.
- W. F. Super, chief chemist, Nitrogen Division, Allied, Hopewell, Va.
- G. Weigers, oxygen supt., American Cyanamid, New Orleans, La.

a single furnace of some 336 tubes of Kellogg design. At the pressure and temperature of our reforming operation, we feel we are going to have some failures. We do not know just what to expect in the furnace itself.

As you can gather, our inspection has been made on more or less of an annual basis. The inspection this year was early as a result of the strike we had during June, July and August. At the end of the strike period, we completed the turnaround work on the ammonia as well as the other operating units. We are trying to develop our own records on this equipment. We have a group of people who are analyzing the data on record. The data taken are being used to set up a system of records.

In metal inspection, we are perhaps on the conservative side. However, we feel that it is a must for us as we go on with this particular venture. It no doubt will add expense to the operation. We have faced that problem and are providing for it by what we call a "fixit" cost in our in-itial investment. In that portion of our budget we are then able to absorb the cost of additional downtime and the cost of the inspection. We have accepted a lower "on stream" time and efficiency. However, in order to have a safe and efficient operation of this facility, we are developing a metal inspection program that will be beneficial to our own operation, as well as providing some data for use in future get-togethers of this nature.

Chairman MAUNE: At this time we would like to have discussion from other representatives working in the ammonia plant field or anyone else who might have something to offer at this time.

HOLSTEIN, Atlantic Refining, Phila., Pa.: We too have experienced some cracking metal failures at nipples for safety valve takeoffs and we have attributed these to vibration. I was wondering if you have any particular program for compensating for this vibration in any planned way. Perhaps methods to follow to eliminate your vibration in a systematic manner.

KING, Sohio: We did some engineering work on vibration, and on how to approach the problem. Our principal point of vibration was in the initial part of the synthesis loop, at the point where the circulator outlet and the purified synthesis gas make-up join to go through an oil trap and a series of exchangers. At this point we had a particularly serious vibration problem that was beyond the scope of our engineering staff. We called in an outside engineering firm who provided us with tiedowns and with piping changes in that area. The problem was eliminated. Our metal inspection group at the plant has gone over every vessel, every compressor and every run of pipe looking for vibration. The appendages have been unitized, whether it be a level controller on letdown, a pressure tap on a compressor, or a drain valve on a snubber. Anything else in the way of an appendage has been tied down to the main piece of equipment.

HOLSTEIN, Atlantic: Our problem is complicated in some respects since we have a varied production rate and with variable production the vibration levels are different. When we get the vibration straightened out at one rate and then switch to another, we have the same problem all over again.

BUTIKOFER, Calumet Nitrogen Prods., Whiting, Ind.: I'm interested in the fire you had. I gather that this was a furnace that was processing "syn" gas and is used in the startup operation of the converter. How serious was the fire and how did you handle it?

KING, Sohio: The fire lasted for exactly 18 minutes. We handled it by blocking in the synthesis loop and by depressuring that portion of the system where the failure occurred. The use of an external heater gives additional valving in the high pressure system. It was through the use of the manifolding in the lines to the external heater, that we were able to block off the system. The pressure was off the furnace loop almost immediately. The damage was slight.

BUTIKOFER, Calumet: Did you use any water to cool the equipment?

KING, Sohio: Yes, we used monitors adjacent to the synthesis structure. They covered the entire structure. The fire burned.out in short order.

LAWRENCE, USI, Tuscola, Ill. You said that you tore out your effluent cooler over there. We've never torn out ours with this clean gas. I was wondering what you found on the inside. I understood you to say that your failure was from the outside-from the water. Is that right? Was there anything on the inside or was the inside in excellent shape? So you feel that with this type of clean gas you don't get any erosion problems on the inside.

KING, Sohio: The failure was due to water side corrosion. The interior of the tubes showed no erosion or attack.

LAWRENCE, USI: I think it is similar to yours-almost the same.

FAATŹ, Foster-Wheeler, New York: I'd like to ask Mr. King two questions. First, I'd like him to enlarge a bit, if possible, on this erosion which he mentioned as having taken place in the high temperature reformer furnace or at least somewhere in that part of the plant. I don't recall just where he said the erosion occurred. Second, if this vibration he speaks of, which occurred in the high pressure system, was a progressively worsening condition with the passing of time, or was evidenced almost immediately after the fire had occurred.

KING, Sohio: In the reforming portion of our plant the erosion occurs in the piping after the secondary reformer. The combustor outlet and the shift converter outlet piping have some evidence of erosion. The vibration problem began on day-1. As soon as we came up on pressure while reducing the synthesis catalyst, we ran into the vibration problem. It was the first serious thing that occurred in the synthesis portion of the plant.

**STOCKBRIDGE**, Southern Nitrogen Co., Savannah, Ga.: I'd like to ask Mr. King, first, whose compressors you have and then would you give me a general description of them?

KING, Sohio: The compression in our plant is accomplished using a two-stage Ingersoll-Rand centrifugal

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with a 4000 hp. electric motor driver on raw "syn" gas. The primary compressors are reciprocating Ingersoll-Rand compressors with three stages of compression. We have two of these machines each capable of 55% capacity for a 300-ton plant. Each stage of compression on each machine has two cylinders. The first stage has an 860 lb./sq. in. gauge discharge pressure; the second stage 2200, third stage up to 5000.

STOCKBRIDGE, South. Nitrogen: Would you mind elaborating a little bit on this inspection program you have? Exactly what do you do and how do you do it?

KING, Śohio: Some eight months or so before the plant was completed, our metal inspection group started to develop a set of prints and forms for the records. These were  $8\frac{1}{2}$  imes 11 prints of all piping and of all vessels. We go into a turnaround with a complete set of working prints on which observations and measurements are entered. This volume becomes the work book. Actually, there are three sections-piping, vessels and safety valves-in the work book. All data in the book are entered on data sheets in the metal inspection files. We progressively plot the changes in metal thickness, in furnace tube elongation or in the physical characteristics of the equipment. We determine areas



Typical pulsation dampener installation on a reciprocating compressor. Shown within the shaded area is a rugged support for the isolation of vibration.

## ... vibration, which causes failures of small connections, is a major problem in most air and ammonia plants, most plants handle it in the same way.

to watch and forecast replacements of equipment using these records.

WALTÔN, Atlantic Refining, Phila .: I'd like to make a few remarks here. A number of us in the ammonia field are now people who have been in the oil business all our lives and we are accustomed to thinking in terms of yearly inspections of pressure vessels. This, in many respects, is not too practical in the ammonia business where you have a converter that you would not like to take apart every year unless there is some really good reason. We discussed this in what we call our Pressure and Corrosion Committee, which in our refinery is the advisor to the Metal Inspection Committee, and we decided we would not inspect our converter for five years. A method of inspection was left in the air temporarily. We're still considering just what sort of tests we want to apply to it. It's a multilayer vessel and that adds some complications. We did come down after about a year and remove the internals of the converter and at that time rigidly inspected the inside and trepanned out a small sample of the inner layer. We're considering what we should do at the end of five years.

On this vibration problem which has been mentioned we are quite conscious of that and we started off the plant on this basis-that on compressor piping up to the first major vessel, all small connections such as pressure gauge connections, samples, vents, etc., would be constructed using Taylor forge nozzles (a heavy, long welding neck nozzle) and a flanged valve. That has proved to be very satisfactory. However, we found that vibration extends beyond the first vessel and we had some-I guess about six failures altogether-of small connections from piping after that point. So we have reviewed that again and have extended the use of the welding neck nozzles beyond that point with gussets and so on, which Mr. King mentioned. We have our metal inspection people go over all the small connections in the plant once every month to look for cracks and we are still somewhat concerned as to whether we have done as good



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Large valve support showing a method of minimizing vibration.

a job as we should.

The vibration problem seemed to be under pretty good control until we added more equipment in expanding the plant. Then we found we had a completely new vibration problem. This vibration is a very nasty thing. You can stop vibration at one point, of course, and then, as you well know, it becomes transmitted to some other point. So it is our feeling that it is really a problem for the experts as Sohio apparently felt in going back to Kellogg.

There are various inspection tools, of course—audiogauging, visual inspection, proof testing and others. I think it would be interesting to know whether anybody has set up any basis in his own mind of how he is going to test the major vessels like the converter and the oil filter, and high pressure separator, and how often.

HENDERSON, Dow, Midland, Mich.: With regard to the comments by Mr. King concerning corrosion and failure of high pressure coolers, our solution to that problem is to use copper-clad, extra heavy seamless steel tubing in the coolers. With no corrosion inside the tubing and with copper cladding on the outside, we have coolers that have been in operation 15 years and are still in excellent condition.

For sample points and pressure gauge connections in high pressure service, we use couplings welded on the piping or equipment with American Instrument Co. high pressure valves for control. The valve outlet is connected by rubber tubing to the sample line which runs to the control station. If the tap is used for obtaining pressures, we use ½ inch capillary tubing between the valve and the pressure gauge.

Commenting on our setup for testing and inspection, all pop valves are changed and tested at least once each year. High pressure equipment such as converters are hydrostatically tested at 5/3 the operating pressure at 2-year intervals. Other high pressure equipment is tested at 5-year intervals.

WALTON, Atlantic: Speaking for Atlantic, the final responsibility is with our Plant Protection people. The way the system works is something like this: The maintenance people request permission to weld in a certain area or location, or to enter a vessel, whichever the case may be. The operator then prepares the area in accordance with what in his opinion is the safe and proper manner insofar as clearing the apparatus of gas, or removing refrigerants from the area and so on. Then the plant protection, or safety people are called in and they inspect the area and ask questions and they must be satisfied that it is safe. They then give the permit and for the permit to be valid they must sign it and the operating supervisor in the area must sign it. That makes it a joint responsibility and welding or other work of that nature cannot be done unless they jointly agree, in writing, on a fire permit or a welding permit to do so.

ANONYMOUS: Are your plant protection people technically trained?

WALTÔN, Atlantic: No, they are not. They are trained mainly in the College of Hard Knocks.

ANONYMOUS: Your safety people then are experienced in operation? Is that right?

WALTON: Yes, to some degree.

GORAND, Sun Oil, Phila.: We differ somewhat. We have our gas testing department but our permits are made out in triplicate. The man doing the hot work keeps one on his person; one is sent to the superintendent's office; the operator keeps the other. The gas tester himself retains the copy in the book. The gas tester's function is to properly interpret the readings of his instruments and to let us know whether or not gas is present and how much. He also has the privilege of making suggestions, based on his experience, if he would like to criticize it. On this form there is a place for him to write down remarks which may contain suggestions. But the end responsibility is placed, we think properly, on the man in charge of the plant, the operator.

HOLSTEIN, Atlantic: I'd like to expand a little on Mr. Walton's statement. He spoke about our major repairs-where we have to enter vessels, etc. We also have the problem of routine maintenance for which we have a resident mechanic group; here we put the responsibility on the operators themselves. If a piece of equipment is to be worked on, there is a work ticket made out and the back of the ticket is stamped with a safety check procedure. Both the shift foreman and the mechanical foreman then go over the work in regard to the safety aspects of the iob.

SUPER, Allied Chemical Corp., Hopewell, Va.: Our procedure is quite similar to that which Mr. Walton mentioned. We call in our chemical

department also for responsibility in making the necessary clearance tests. They issue a memo which goes along with the burning permit and which gives their statement as to whether conditions are safe to perform the burning or for entering the vessel. The chemical clearance takes in a general survey of the area and the possibility of a dangerous condition developing even though the gas tests may be O.K. I might also add that burning permits are only good for one day and must be reissued every day.

BUDDENBERG, Collier Corp., Brea, Calif.: Our safety procedure is quite like that used by Atlantic, with one exception. We have, for any planned work-that is, work that doesn't come up on the spur of the moment-what we call a "hot work" plan sheet. This is set up in triplicate and is signed by a representative of the Fire & Safety Dept., the Operating Dept., and the Maintenance Dept. Most important, the hot work plan sheet is filled out at least 24 hrs. in advance of the job. We find that this permits it to be seen by responsible people in those departments and it permits a little mulling over the situation so we don't have any unusual incidents because of insufficient planning,

BUTIKOFER, Calumet: We're all in accord with this business of trying to clear the area before you do' any work. However, we've had the experience with hydrogen streams at about 125 lb. pressure that even though we close the valves the gas will leak through. We find ourselves trying to change a compressor valve in a hydrogen atmosphere. Now we have taken the stand that if we can change a valve within five minutes, we'll do it. Anything that takes longer we actually blind off. That makes it a good deal more complicated job.

DE VRY, Hercules Powder, Wilmington: In addition to that, we make it a practice in our plants to put a bleed valve between two high pressure block valves so as to stand between high pressure operation and equipment to be worked on so. When the two valves are closed, the bleed is open between them and we can thus be sure that no gas under pressure is leaking past the second valve. This applies to work on equipment where there is a possibility of high pressure leakage past the second valve. Our systems are at approximately 1,000 atm.

WEIGERS, American Cyanamid, Fortier, La.: We feel that the primary responsibility for safety must lie with the operating people because, in our

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opinion, they are the ones most familiar with the process hazards involved in hot work. Consequently we ask that any hot work be signed by the shift foreman and also by his immediate supervisor to give a double check and make sure that all process considerations have been taken care of.

With respect to valving off a particularly dangerous gas we make a blanket ruling never to rely on valves if hot work is involved. Any line must be blanked if on one side you have an explosive or a combustible gas and you are working on the other side. Now if there is an argument between the Maintenance Department and the operating group, it will be referred to the Safety Director of the plant for settlement. In other words, any maintenance mechanic can challenge the wisdom of particular safety permits. In fact this happens very rarely.

ANONYMOUS: I know of several instances where people have tried this double block and bleed system and you'd be surprised that the small valves can get plugged up. In my experience and knowledge of other plants I know of places where several bad accidents occurred because they didn't blind. The bleed didn't work and caused failure.

CHAIRMAN MAUNE: We have about 20 minutes left and we now open this meeting to discussion, so that anyone may bring up any subject he wishes.

OHLSEN, Pennsalt Chemicals, Wyandotte, Mich.: We are a little different, I guess, from most of the recent installations. Our reason for doing that was architectural considerations in squaring off an existing building. At the time, the construction which was used was to louvre the area so that there was plenty of air movement on the back side. We had some discussions among ourselves as to continuous monitors to pick up leaks in advance of the big bang, but we could not get any agreement on the subject so we never did it. I was curious as to whether any other organizations in their confined spaces are using continuous monitors to pick up and warn of final gas concentrations shortly after they occur. Of course that's a tough thing to do-to know what is going to leak but it appeared to me that a monitor of 46 points in our monitor room placed strategically might tell us of incipient fires or something of that sort. However, we've never done it. Has anybody

## ... some companies still favor hoses for loading ammonia, others have gone to pipe.

else? Has anyone gone into continuous monitoring?

HENDERSON, Dow: Our high pressure equipment is located outside the building. The parts of the plant that are housed are the low pressure blowers and the compressors. We have had leakage from the packings of the Connersville Blowers handling hydrogen and have installed Johnson Williams industrial gas detectors over each of the blowers and at various points on compressors. At 40% of the Lower Explosive Limit of Hydrogen in Air an alarm is sounded and a panel board light comes on to alert the operator to shut the plant down and correct the leakage.

We were concerned as to whether flashbacks would occur in the instrument which would ignite a flammable mixture. The complete range of hydrogen in air, up to 100% hydrogen, was checked without flashbacks occurring so it is completely safe to operate in an atmosphere containing hydrogen.

BOLLEN, Dow Chemical of Canada, Sarnia, Ont.: We have the same analyzers at Sarnia as at Midland. They are located over our synthesis gas compressors, hydrogen and hydrocarbon compressors, and also over our circulator compressors. We find them very useful for detecting small packing leaks-leaks that might be missed under normal operating conditions. The analyzers are connected to an alarm which rings on the control panel and alerts the operator to the specific area where we have the leak. By testing with a gas analyzer we can pin down the particular compressor cylinder or valve that is causing us the trouble and if it is serious enough, shut down the machine.

GORAND, Sun: We recently built a new unit which is about 350 ft. high, and the site was in juxtaposition to active gas plants and there were many sources of contamination. The whole construction area was literally crawling with welders and a lot of hot work was being done. We invested something between \$18 and \$20,000 in buying continuous combustible gas alarms. We used the type with rotating selectivity valves at different locations and at different levels. I think we have one or two that were hooked up to a large klaxon that could be heard over and above the hum of the industrial construction noise. These alarms were automatically tested and on one or two occa-

sions when there was an escape of gas the detectors kicked off and all hot work in the area was doused at once. Throughout the construction job we had no serious fires.

So we think the equipment paid for itself because it could be utilized elsewhere not only in permanent locations but as a portable monitor.

For instance-on one occasion when men were working on a spheroid-type tank which was on legs and was some distance above the ground it was placed above a manhole. Of course the valves to the tank sphere were dropped out and all precautionary measures were taken to be safe but we still stuck a "sneak" in there-a nose in there, we call it, and this is what happened (bringing up the theory of the monotony index)-at noontime all men who are welders are supposed to bring their torches out with them-we had some gas welding to do-and they are supposed to disconnect the torch and take their hose out. But you know how men are, especially in the summertime when they are anxious to get out and take advantage of every minute of that noon break. One individual left his torch in there, it had a leaky combustion chamber on it and just before the whistle blew for them to go back to work this monitor picked up and kicked off. Had the men gone into the area and lit their torches, they might have come out of there very rapidly, which proved the value of the equipment.

HOLSTEIN, Atlantic: I'd like to get back to the subject of equipment failures. We've been concerned primarily with the steel equipment in the discussion thus far. However, there is one obvious problem in ammonia plant operation, and in others. You have to sell your product and to ship it, and here you encounter the problem of what types of hoses, the connections, and inspection methods for hoses. Another subject is that of quick couplings; we don't feel that any observed so far are satisfactory and I would like to know if anybody else has experience with quick couplings? Our program is to inspect hoses every six months by a hydrostatic test. As for couplings, we use the "Weco" coarse-threaded union to make the couplings between our hoses and our tank car. Does anybody care to comment on the subject?

CHAIRMAN MAUNE: I might add something to that. In my experience

with hose in ammonia service it hasn't been too good. For that reason we have gotten away from it entirely and we use pipe and Chiksan joints which have worked out very satisfactorily. The joints don't leak if you take care of them and lubricate them once in a while and we think it is a pretty good step in the right direction.

BUDDENBERG, Collier: We also use Chiksan joints. We find they are very acceptable. We use them for tank cars. We haven't used them in the truck loading lines as yet.

DE VRY, Hercules: We use air baths with rubber or neoprene hoses and so far we have not experienced any particular trouble with aging or cracking of the hose. We've used them for years, we've replaced a few of them which began to look bad at the connections.

But we use neoprene and I must say neoprene hose works out. I would like to get an expression of opinion from this group about something we've talked about a great deal and can't seem to come to any agreement, at least within our company, and that is the advisability of using an explosion-proof fitting on a thermocouple connection in an area where it is possible to get gas leaks. We have had both sides of the question argued hotly but I personally do not see why one should have to. However, there are those who say it should be done. It amounts to considerable expense in an installation and I just wonder how much real danger there is. Have any of you any thoughts on this?

WALTON, Atlantic: We argued quite a lot between ourselves when the plant was built about the necessity for complete explosion-proof setup and our conclusion was that we would not use explosion-proof fittings for thermocouples wiring and in the laboratory which was within the plant limits, we used vapor-proof construction. We do not ordinarily bring hydrogen into the lab but we do have ammonia in there and the feeling was that the temperature required according to the best sources to be found in the literature, was so high that it was difficult to justify explosion-proof lighting and electrical outlets in the laboratory.

FAATZ, F-W: Most of Foster-Wheeler's plant construction, in fact all of our plant construction in the ammonia field, has been with Texaco partial oxidation, so that's the only field about which I can speak. But a lot of the ammonia plants that are being built have been built to operate with high pressure reforming-furnaces, which operate in the range of 80, up to, I guess, as tar as 125 lb./sq. in. -nobody cares to say just how far. It would certainly be of interest to me, and perhaps to all of us here, if there could be some discussion on what precautions are taken with regard to tube rupture, tube failures, etc., whether there have been any such experiences, and what has been done about them.

STOCKBRIDGE, Sou. Nitrogen: We have a high pressure reformer and we have had some tube ruptures at the bottom of the catalyst tubes which are about 4 in. in diameter. These ruptures start where the gas comes out of the bottom-I believe it's 1-1/4 in.; at that weld we've had several ruptures. They haven't been serious. As you know, you can't see the light of a flame very well in the daytime so we have them inspected all the time and we particularly note them at night when the flames can be seen. We haven't had any real serious ruptures because the pressure is not that high. If they aren't too bad we go ahead and operate until we have enough other maintenance, to justify a shutdown and then we have to shut down and reweld.

We have had a rather lengthy shutdown in which we went over and rewelded the whole batch of 150 tubes. We had no trouble with ruptures but we considered it advisable to strengthen those welds and since that time we haven't had any trouble with ruptures at that point.

CARTER, Grand River Chem. Co., Pryor, Okla .: I'd like to go back to this point of thermocouples-lead wires into the instrument. I have observed on one occasion where hydrogen at high temperatures was being handled at low pressure and you could almost always find explosive gas tests if we checked the thermocouple well. So I do think there is a potential hazard, if the thermocouple is piped from the thermocouple well directly into the instrument; it was our practice on our recent plant not to run the conduit to the head of the thermocouple. We have a loop exposed there so there is no way in which hydrogen gas could be led into the instrument. We felt we would bring a potential bomb right into the control board in so doing, where we operate at higher temperatures.

CHAIRMAN MAUNE: I think we should move on to another subject now. There is one that is always with us or has been in the past—I think it is here all the time—that is the internals of an ammonia converter or anywhere where you have high temperature and pressure and nitrogen

present. The nitriding problem has been quite serious in the past. There have been some developments--there are still some going on which have greatly helped the situation. I can remember when I first got into the ammonia business, my old boss told me that whenever you pull the catalyst basket out of the converter you're just apt to take it out in pieces. You just can't pull it out in one unit.

Back in those early days there weren't the alloys we are working with now to make the internals, the heaters, the baskets, and so on. Of course in extended runs and operation of an ammonia plant, especially the converter, we like to keep them on as long as we can. My experience has been with one converter which operated  $8\frac{1}{2}$  yrs. without opening; and another for  $7\frac{1}{2}$  yrs., until there was evidence of nitriding and peel.

I have no formal presentation at this time but a gentleman came up and brought some specimens of nitrided steel and I wonder if he would come forward now and say a few words and maybe he would like to identify them or talk about the way they would affect his operation in the safety area when operating an ammonia plant.

BUDDENBERG, Collier: I brought along a tube sheet and a portion of a tube from the methanator-exchanger in our synthesis loop which operates at 3,000 lb. We had a rather severe case of nitriding-at least it was so analyzed by our metallurgist. The tubes involved were 18 gauge, 5% chrome,  $\frac{1}{2}$ % moly in composition. The tube sheets and cross baffles had the worst nitriding and they were 12% chrome. The examples are here on the table if you care to examine them. You will note that the cross baffles are very severely corrodedactually they were completely disintegrated in the top 3 or 4. The

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temperature at this point runs at a maximum of about 850°F. Our converter has internal exchange so that the exit gas is already somewhat cooled.

I have a few comments on the previous subject if they are suitable here too. We also had a nipple failure in a high pressure system, depressuring the system through a  $\frac{1}{4}$  in. nipple. We attributed it to vibration at the compressors (where most of our vibration has occurred). We think we now have the problem solved. We have used the usual methods of unitizing down the nipples as described by others here. In addition, we have done a better job of tieing the piping in the compressor area down to the concrete and we have installed pulsation dampener tubes within the discharge bottles of some of the compressors.

We had one other failure in our synthesis system which might be of note. We have a methanator ahead of our synthesis loop; several depressurings at high rate during various operating difficulties caused the catalyst in the methanator to fragment and some of the fragments went through the support screen. In making the turn in the piping at the bottom of the methanator these fragments severely eroded the piping. A failure occurred at the elbow and enough of the hydrogen-nitrogen mixture leaked to flash and burn. We fought the fire in the same manner as that described by Mr. King-depressured the system and flooded it with nitrogen; the whole affair was over in about five minutes.

To combat this problem we changed to a stainless pipe at that point and also made a wide radius curve from the methanator. We've



Whether taking notes or watching the speaker, the three in front, and everyone else, had a busy session.

### ... a tragic experience involving a <u>broken tail ro</u>d requires drastic measures to prevent a recurrence of the same accident.

had no more trouble and our inspection indicates that the metal is still full thickness.

CHAIRMAN MAUNE: Are there any other comments on nitriding problems? There is one thing I would like to bring up at this time because I've experienced it myself and I think that everyone would like to hear about it. I'd like to pass it on to you because if there is anything you can get out of it, or profit by it, it would be very worthwhile. It is about a serious accident we had in our plant at the Mississippi River Chemical Co. over a year ago. It involved



a syn-gas high pressure compressor. We compress syn gas to 9,000 lb. pressure in four stages. The last stage cylinder is a double-acting piston with a tail rod. Of course the old conventional way of covering tail rods to keep down or vent off the leakage from the packing or catch the oil drip is merely to put a piece of schedule 40 pipe or so over the rod-also for protection against personnel getting into the way of the rod. This particular compressor had been operating I would imagine for about sixty days or perhaps between 45 and 60 days.

The story was this: we were starting the reaction in both of our ammonia converters. We were at a point where we were producing and just lining out the loop. It so happens our control panel was right in a direct line with the end of this compressor cylinder. Conditions were very well stabilized. We had three people in front of the panel-our ammonia superintendent, our shift foreman and an operator, and for no apparent reason at that time, the tail rod broke off inside the cylinder and of course the tail rod came flying out of the end and hitting a building beam about 40 ft. away. We were at 8,000 lb. pressure at that time. There was nothing to prevent the gas from coming out of the cylinder. The compressor ran for about a minute or so before we could shut off the compressor. The gas ignited and filled the whole building-that end of the building-with gas. It literally blew out the end of the building, blew three men out and burned them.

Now there is quite a bit that we have learned about that situation. We investigated every possible angle of "why did it happen." This compressor piston rod is made of a Nitralloy material which is a very high tensile steel. In fact we are told it is one of the best materials made for use as rods of that sort. The rods are actually about 12 ft. long, 3 in. in diameter, and we made various tests and when we got the pieces together the tailend rod was literally full of cracks. There were hairline cracks, others that were halfway through, quarter-way through-just full of them.

We have developed information which we think might have caused the break in this rod. That particular rod was damaged in the earlier stages of operation, we needed to get on stream, the rod was damaged in going through the bushings and the

runout was in excess, so steps were taken to straighten the rod and regrind the surface that had been damaged going through the packing bushings. We found out later that this Nitralloy material is very tricky stuff -you can't grind it-and when you do grind it you produce surface cracks. So that is the information we have developed on why the rod failed. We thought we still had a problem there -we didn't want any more tail rods flying around. So we worked with other people. Our engineering group and Cooper-Bessemer came up with an idea of putting a catcher over the tail rod. It was something I had never heard of before but I understand others have successfully used it in the past. What it involves is taking the schedule 40 pipe off the tail rod, cover, and actually getting a steel forging machined which bolts right next to the packing case. It is designed to withstand the full pressure which is inside the cylinder.

There is a leakoff connection—the normal leakoff from packing can get by. If the leakoff should develop so it gets to 50 lb. pressure, we have a pressure alarm on it which would sound an alarm and we'd know that something was causing gas to get back into the catcher in excess of normal leakage.

Another thing we did was to bore the tail end of the rod. We have a hollow rod. From the end of the rod all the way to the piston there is a 1 in. hole. The reason for that is this: if a crack were to develop, or a break, the pressure would immediately equalize going through the crack down through the bore and back through the catcher. We think that might improve conditions in case the rod were to break. Also, in the end of the catcher on the inside, we have a lead block which will absorb shock when the rod breaks. We don't think it would go back real fast. Also, where the flange of the catcher bolts to the packing case there is a bronze bushing which is of a smaller diameter than the outside diameter of the piston rod. There is about 2 to 3/1000 ths interference there which is another means of slowing the rod down when going through the bushing and before it gets to the end. I think it is worthy of passing this on to you. I don't know if any of you people have tail rods on any of your compressors. You probably do. This is my first occasion of using a rod of this type at this pressure-9,000 lb. I've used them before at 3,000 lb. and at 5,000 lb. Of course those particular rods were larger diameters-say 4 in. Whether this has any effect on it-whether the long rod or the smaller diameter or the higher pressure, I don't know. However, if anyone cares to comment on that I'd be glad to hear him. Possibly somebody else has experience along that line and if so, I'd like to have the benefit of it.

I'd like to get comments from somebody, especially on the rods. If you have a damaged rod which is bent when the runout is too great, has anybody set up any limits on the maximum amounts of runout? Or do you try to reclaim a rod by straightening it, or scrapping it, or just what. HOLSTEIN, Atlantic: Based on your experience (we also have tail rods on some of our compressors) we have actively engaged in plans for tail-rod guards. One of our worries is the alarm system that you mentioned. Have you experienced any difficulty in being able to set the pressure alarm due to the reciprocating action in the compressor?

CHAIRMAN MAUNE: We haven't had any difficulty along these lines. Actually, the way it is arranged is this: on the bottom of the catcher we have a spring-loaded check valve. Should the pressure get to 50 lb. of course it will shut off and once your springloaded valve closes your pressure will increase. You can set the pressure at any spot on the dial you want to. What we have is one we had built which will go to the full 9,000 lb. What we have is a pressure transmitter located right at the face of the cylinder. Many pulsations or fluctuations in pressure at that point wouldn't affect the alarm any. We also have a bleed valve there whereby the operator can go by, say, once an hour or so, and open it up and check it himself. He just opens it up and normally there is very little gas coming out if the packing is all right.

Here is another item I think we should talk about. It concerns high pressure welding. Any welding failures we might have, any welding inspections, magnofluxing, reflectoscope, things of that nature.

CARTER, Grand River: Our experience occurred after approximately one year's operation. We had a high pressure exchanger—this is on a synthesis gas compressor which failed at a weld on the return bend on the exchanger. The result was that it depressured this exchanger into the cooling water stream which went back to the cooling tower. The only damage other

than to the exchanger was to cause the riser on the side of the cooling tower to tear away from the tower and fall to the ground. Visual inspection of the welds of this exchanger did not look good. We elected then to have all of the welds x-rayed. Some 70% of the welds in this exchanger would not pass x-ray inspection. They had voids and slag inclusion in them. Of course the result was rewelding some 70% of the welds and stress relieving them before we could put the exchanger back in service. This exchanger, of course, was built under code and with insurance inspection. The purchase order did not require that all welds be X-rayed, and I thought it might be well to bring this up for discussion to see what the experience of other companies has been with high pressure exchangers. I would say this, without mentioning the name of the exchanger company, that it did not exercise what we consider the proper concern about the failure of a weld or the fact that some 70% of their welds were faulty. ANONYMOUS: I can add a little something to this business of weld failure but a little different kind of an incident. We have in our nitrogen wash operation a very low temperature and in operating upsets some of our steel lines are cooled off rather drastically. In so doing we have had the experience of a weld failing and usually at a slag inclusion. The split in the pipe is usually of a spiral nature. It starts at the weld and then splits right through the virgin metal. **ANONYMOUS:** I'd be very interested to hear if anyone else had an experience like this with low temperature piping. Particularly whether anyone else has had experiences like this with low temperature steel piping. This is something that concerns us very much and it is news particularly to a group of petroleum men.

HOLŠTEÎN, Atlantic: We have experienced the same type of failure in our cold blowdown system where we let down some cold materials into a blowdown header. Carbon steel is not satisfactory material for a very low temperature process.

ANONYMOUS: (Question is asked on compression inspections.)

WEIGERS, Cyanamid: We've been arguing about this subject for the last four years. Right now we are operating eight steam turbine driven centrifugals and we are putting seven more on the line. We've come down to this system now. We make an annual inspection—what we term an "at rest" inspection of only the bearings, the governors, and the various auxiliaries of the machines. Every three years

we are planning to lift the cases on them. Maybe we'll find as we follow the program of three year inspections that this is too frequent or possibly not frequent enough. We are starting off with a one and three program. WALTON, Atlantic: Atlantic checked Cyanamid's practice of three and that is about the frequency with which we inspect turbines. Of course if you have a vibration setup some time which is excessive, why naturally you look then.

There is one topic that I'm not sure everyone is aware of and that is this -that is the question of velocity checks in tank cars. At the moment there is no regulation which requires velocity checks in tank cars. When we started operations one of our customers specifically requested that the velocity check be removed from tank cars to help unload the cars more quickly. I believe that recently there are some proceedings before the ICC which are becoming crystallized now and which will require velocity checks to be used in all tank cars and tank trucks in interstate service. That may be of some interest to you. Also if you have any feelings one way or the other, you might care to get in touch with the regulatory authorities. The customer who requires the velocity check to be removed from tank cars going to him has been talked out of that by ourselves, and we have insisted that all tank cars leaving our plant have velocity checks in them. Does anybody have any comments on that?

MASON, Dow, Midland, Mich.: I would like to ask Mr. Walton at what velocity these check valves are designed to close?

WALTON: I can't answer that. Of course the cars used for ammonia are usually interchangeable between ammonia and LPG, and we have never gone into the matter of just how they are set. The people we lease tank cars from are Union Tank Car Co., and it is whatever their standard installation is.

DE VRY, Hercules: We use a plug valve of that type made by York, that works very well. We have them in all our cars and we have them of such size that a car can be unloaded in an hour which is pretty fast and which is a pretty fair average for a car of anhydrous. All our cars are equipped that way because we have often had to go out and explain what is wrong, to customers, when they can't unload sometimes. However, we wouldn't take them off even though we might lose a sale.

CHAIRMAN MAUNE: We're coming to the end of this session, gentlemen,

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and if there is anyone present who would like to bring up something else we still have a few more minutes. **ANONYMOUS:** We have not considered testing cylinders but it is an interesting problem to consider.

KING, Sohio: On initial construction, Sohio hydrostatically tested the piping and compressor cylinders as a unit. MASON, Dow (Midland): Several years ago, we attempted to apply hydraulic tests to compressors' cylinders in Midland and ran into considerable difficulty. We tested the discharge line, discharge port, and cylinder back to the inlet valves, at 11/2 times the maximum discharge pressure. We also tested the inlet line and inlet port at 11/2 times the maximum inlet pressure. Although we did manage to reach these pressures in spite of leakage at the packings, we recognized that these tests did not simulate actual operating conditions. The operating temperature range and distribution could not be simulated. Therefore, the stress distribution was not representative of operating conditions. Any possible failure of a cylinder in operation would be a fatigue type, which would be progressive rather than sudden. Such failures always give warning by leakage. Consequently, we believe that periodic hydraulic testing of the compressor cylinders is not justified, and we have abandoned any further hydraulic tests.

BUTIKOFER, Calumet: One safety problem gives us quite a bit of concern. It's a matter of oxygen in ammonia. We are not so concerned about the oxygen itself in the ammonia, but as the vapor ammonia and the oxygen pass back through the ammonia recovery system, the tendency is, of course, to condense the ammonia away from the inerts. A mixture like that always contains hydrogen and it is possible to finally have a mixture which is very rich in hydrogen and quite rich in oxygen. An explosion in the ammonia recovery system of the plant can occur. Anybody else thought about this and if you have, what has been done?

WALTON, Atlantic: It looks like there are an awful lot of unanswered questions here that should give us all a lot of food for thought. There is one other thing I'd like to mention which would be of interest to the group. A number of our smaller customers have recently been requesting that we pressure tank cars and tank trucks with nitrogen to help them unload the cars more easily. Presumably they

are not equipped with a compressor system for unloading. It seems like a rather innocuous request but when you stop to consider it you are doing away, in a sense, with the safety valve protection for the tank truck or tank car that's been calculated, considering only the vapor pressure of the ammonia in the car or truck. So, if you add additional pressure from nitrogen it is conceivable that if the car or truck sits around in the sun for a long while and warms up, you may have a serious discharge of ammonia from the safety valve which wouldn't make the railroad people or the population in that vicinity very happy, let alone your company. I don't know whether anybody else has had such requests or has considered the legalities of it. We are doing this right now.

SUPER, Allied: We have such requests but we give them the pressure by giving them warmer ammonia in the car. The higher vapor pressure of the warm ammonia produces a higher pressure in the car.

BOLLEN, Dow (Canada): We too have had several requests from smaller companies to pressure tank cars of ammonia with nitrogen and we have complied with these requests. However, we pressure the tank cars according to a sliding maximum pressure scale which we have developed. This scale is based upon the time of year and the prevailing temperature conditions in the areas of the country to which the cars are shipped. In this way we make sure the total pressure in the tank car is kept well below the setting of the safety valve on the tank car.

HENDERSON, Dow (Midland): We have had requests for nitrogen padding on cars, but only in winter when the vapor pressure in the car is low. Our top padding pressure is 100 lb./ sq. in., so we are away below the pop valve setting of 225.

One question that I have is in regard to tank trucks for ammonia. The question is before the Compressed Gas Association now and concerns the use of tanks of 250-lb. design pressure. Present ICC regulations require a design pressure of 265 lb. ANONYMOUS: I want to address this to the earlier speaker about the oxygen contamination in the car. I'm not certain of the gas stream he is talking about. But if he is not interested in the hydrogen contained in that stream could you not just pass that stream through a deoxo unit? This is actually what you can do if you are using an ammonia dissociator to give you a hydrogen gas for desulfurization

or something like that. You will actually react the oxygen in the stream with the hydrogen.

Does the stream you were talking about contain hydrogen which has to be saved, or can you just go ahead and destroy it?

BUTIKOFER, Calumet: I don't think I made myself too clear. I was thinking about this sort of situation: when a tank car of ammonia is loaded, the car is usually vented back to the sphere. Suppose a new car comes in or one that has been cleaned, you can get air into the sphere. Now that sphere is refrigerated by compression. The air, along with the ammonia, is drawn down to the refrigeration compressor and the ammonia is condensed.

Now all of the ammonia we've ever had in our spheres has had hydrogen with it. We have always found hydrogen above the ammonia in the vapor space. When that ammonia is condensed, then the concentration of hydrogen and oxygen increases and you can get into the explosive limits. Now some plants, and particularly ours, are set with a pressure controller to release the inert gas out into our sweet gas vent header, which in turn goes to the flare. You can see we could release a combustible mixture of gas right into our flare line and that could flash back. We've had to make some changes to avoid putting it into the flare system. This is a problem where you can have a part of the ammonia refrigeration equipment with inerts that are explosive and I wondered whether this had been recognized by others, and if so, what precautionary measures they might have taken.

WALTON, Atlantic: I might mention that at Atlantic- we are fortunate in having a nitrogen plant and we make a practice of purging all new tank cars when they come in. We also make a practice of purging our spheres with nitrogen before we go into them, and when we shop a car, we purge with nitrogen before the car goes into the shop.

CHAIRMAN MAUNE: This is the first session of this Air-Ammonia Plant group. This will end the session of this particular group for this morning. Tomorrow there will be two sessions on air plants, which will probably include air plants as such, liquid nitrogen scrubbing units and even the compressor end of compressing air in air purification systems.

Source of information for illustrations on pages 37 and 38 is the M. W. Kellogg Co. Parts 2 and 3-Air Plant Safety-will appear in August CEP.