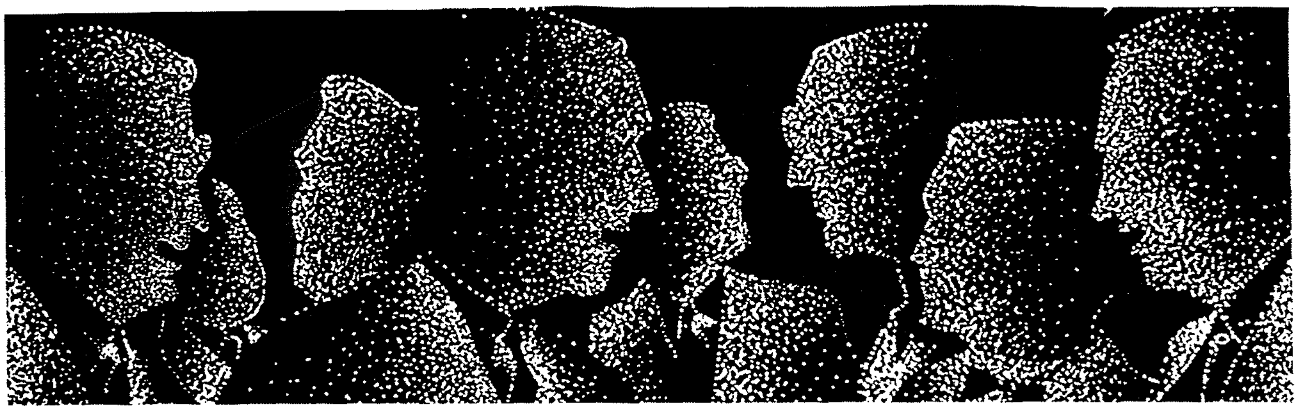

the operators report on
SAFETY in air and ammonia plants

This third section of the exclusive CEP report on the recent informal roundtable session takes up such problems as tankage, repairs, and transportation of liquid air and ammonia.

CHAIRMAN WALTON: Last year Mr. Maune told us about failures of piston tail rods. It has led many of us to adopt additional safety devices.

deVRY, Hercules Powder: The safety record of ammonia-plant synthesis gas compressors has been good. However, there have been two major ammonia-plant accidents in recent years which were the direct result of compressor failure. In each case, a large quantity of synthesis gas escaped directly into the compressor house, resulting in explosion and fire, with heavy loss of life and property. These accidents, plus several others of lesser magnitude, emphasize the need for care. A piece of high-speed, reciprocating machinery, compressing a flammable gas mixture at high pressure and (usually) located in a closed building occupied by operating personnel, has all the ingredients for a major catastrophe, and therefore deserves serious consideration.

There are two common types of failures in which large amounts of gas can suddenly escape from the compressor: (1) the failure of connecting pipe or joints; (2) the breakage of a cylinder, cylinder head, or valve-chamber cover; (3) and a special case, the breakage and subsequent escape of the tail rod from a compressor cylinder (the cause of one of the two major accidents mentioned above).

Connecting pipe failures are usually the result of repeated stresses, caused by pulsation. This pulsation is not basically a vibration of the pipe, but of the gas column inside the pipe. The moving gas column is traversed by pressure waves, moving at the speed of sound and usually at a frequency corresponding to the speed of the compressor. Usually these pressure waves do not build up much amplitude and the resulting vibration is moderate and easily controlled. However, if the compressor piping happens to be of critical length, to allow the system to resonate at the compressor speed, the pressure wave will be reinforced and built up. The actual gas flow from a cylinder is irregular because of the changing piston speed at various stroke positions. This results in a number of harmonics which, if they match the resonant frequency of the piping system, can build up large pulsations, and cause trouble. Because of its complexity, the prediction, in the design stage, of the resonant frequency and resulting vibration amplitude of a piping system is extremely difficult.

A number of methods have been proposed but none give satisfactory results. We usually are faced with the problem of correcting a situation soon after the plant has started up. There are a number of ways to cure resonance troubles. The most direct is to change the natural frequency of the pipe by changing its length, or adding a stub pipe to detune the system. An orifice not only changes the natural frequency of the piping but also damps the pulsation wave. A volume chamber lowers the natural frequency and reduces the pulsation amplitude and resonance. An acoustic filter (pulsation dampener) which passes only waves below a given frequency, will cure the trouble if the frequency is set below that which causes the vibration.

Several basic design practices which should be included in any new system to avoid this trouble are:

1. Use adequately sized suction and discharge surge drums as recommended by the compressor builder.
2. Avoid closely sized interstage piping.
3. Avoid building bourdon tubes into piping layouts.
4. Use machine-mounted intercoolers whenever possible, they avoid connecting pipes.
5. Use long-radius ells in piping.
6. Avoid long, unsupported pipe runs.

One frequent cause of cylinder or head fracture is liquid carryover into the cylinders. Compressors are invariably provided with liquid separators ahead of each stage, with the possible exception of the first stage. However, if the process includes, for example, CO_2 removal between compression stages, it is quite possible to get liquid carry-over from one of the scrubbers which exceeds the holding capacity of the separator. Such systems should be provided with a large separator in the form of an empty vessel between the scrubbing system and the next compression stage. The vessel should be provided with a sight glass, blowdown, and a liquid-level alarm. In certain cases such a vessel should also be installed on the compressor suction line.

Sometimes condensate will carry over from an inner cooler separator. This usually can be traced back to failure of an operator to blow the system down to schedule. But sometimes it results from the use of excess oil, which emulsifies with the water to a mayonnaise-like mixture, difficult to purge from the drum. This is especially true at high pressures.

Where it is necessary to use a cylinder having a tail rod, such rods are ordinarily enclosed in a light steel case which serves to trap packing leakage and to prevent personnel injury. Should the rod break, for example, where it joins a piston, the broken rod will act as a free piston and under the pressure differential be driven with great force against its enclosure, breaking out, followed by a heavy rush of gas. The remedy is to provide a tail-rod case heavy enough to withstand the impact force and also to provide a means of absorbing a portion of the shock load.

These factors are incorporated in a design where the normal packing flange is provided with elongated studs to which the flange of the tail-rod catcher is bolted. To help absorb the shock load, a plug of cushioning material is provided at the end of the catcher.

Walton **Wright** **Bollen**

One basic way to minimize the danger of fire and explosion in and around the compressor is to install it outdoors. Our most recent installation of a large-size compressor was outdoors.

MAUNE, Mississippi River Chemical Co.: The accident at my plant was covered in *CEP* recently. A tail rod broke in a compressor. Apparently, cracks developed and originated in the ring groups. The problem has been solved by newly designed rods which have been operating about eight months with no apparent difficulty.

We experienced difficulty with our high-pressure cylinders. The synthesis gas compressors are four-stage machines, compressing hydrogen and nitrogen from 290 to 9000 lb./sq. in. A crack developed in the main cylinder body. There were three such failures during the past year, fortunately, without serious accidents, because the compressors were taken down in time. Although we and the compressor manufacturer are still working on the problem, to date we don't know what is causing this difficulty.

We have been plagued with vibrations on the synthesis gas compressors, to the extent that the whole unit would move several mills on the foundation. We also experienced breaking of pipes in the piping system. Some vibrations in the piping system were eliminated by redesigning the piping layout, and placing bottles nearer the compressor cylinders. Some were traced back to the engine firing, which indicated that the speed varied 5 to 10 rev./min. Replacing several ignition coils eliminated this problem. Now, the units are doing very well. Our biggest problem is still the high-pressure cylinder breakage.

WRIGHT, Standard Oil (Indiana): At the Calumet Nitrogen Products plant, a rod on one of the synthesis gas compressors failed. Fortunately, the failure was detected when the crack had progressed only three quarters through the rod. The breakage was indicated by leakage of hydrogen down a longitudinal hole in the rod. On checking the rod, we found the steel was not of the proper composition for nitriding.

Another experience was failure of interstage piping around the synthesis gas compressors, due to vibration. The bracing consisted of wood blocking with tie-down straps over the piping. As the wood blocking worked loose, the strap bolts broke and allowed excessive motion. The bolting and blocking were improved, and no more failures have been experienced.

BOLLEN, Dow (Canada): We have had three failures on the third and final stage of our synthesis gas compressor. The rod in question has a suction valve contained in the piston itself. The piston rod has two lands which support the piston at each end with an undercut section of rod between the two lands. On the first occasion the break occurred at the thread root at the extreme end of the piston rod which holds the suction valve to the piston. This rod break was believed caused by excessive tightening of the nut holding the suction valve to the piston rod. Subsequent tightening operations were made with a torque wrench, in accordance with manufacturer's recommendations.

A few months later, the rod failed at the same point as the first break. Careful examination of the break and a review of design stress calculations for the rod indicated that the probable cause of failure was metal fatigue, with the main stress point at the last thread root. To overcome this problem, the manufacturer redesigned the rod and reduced the diameter of the undercut section of the rod from 0.872 to 0.6875 in., to keep the minimum cross-section area of the "business" end of the rod less than the root area of the 1-in. threaded section. This was to keep the operating stresses in the threaded section below those in the undercut section and so improve the fatigue life of the rod.

The third rod failure occurred at the undercut section of the rod and it appeared that the weak point had just been transferred from one place to another. However, examination of this fracture showed that it was not a fatigue failure. Something had jammed between the piston and the cylinder wall causing elongation of the undercut section to the point where the rod broke.

In each of these rod failures, no gas was released into the building and the breakage was confined to the cylinder, piston, valves, and cylinder line.

WHITE, San Jacinto Chemical: We have cracked some 15,000-lb. cylinders. Our supplier has come up with a pretty good answer—which is stress corrosion. We had never paid too much attention to our spare cylinders. When in storage, their outsides would get a little bit rusty and, frankly, the insides too.

WAIBEL, Ingersoll-Rand: It is interesting to note that the cylinder and the cylinder heads that failed at San Jacinto had been in operation at Louisiana-Missouri all during the war. Because the cylinders had been in operation for years, the source of the corrosion is difficult to pin down, but the evidence was there. There is no doubt that stress-corrosion fatigue was the main reason for the failure of the cylinders and heads. We recommended that they heat the inlet gas, and we made a major design change which should solve the problem.

CHAIRMAN WALTON: What was the nature of the atmosphere? Was it wet ammonia?

WHITE, San Jacinto Chemical: It was synthesis gas, nitrogen-hydrogen, and probably traces of NO, but we never found it.

deVRY, Hercules Powder: I can bear out that those cylinders had been in use a long time. They had hundreds of millions of reversals, and it actually is not too surprising that some of them did crack. We had the same experience on sister compressors at other plants; it wasn't a bad problem, but we did help it by heating the gas coming into the cylinder, to keep in the vapor phase little droplets of water that can cause what we think are points of stress corrosion.

CHAIRMAN WALTON: Certainly you always have to be alert to small details such as the need for fillets instead of sharp corners and shoulders. The more you hear about metal problems, the more you realize that the metallurgist is a man who still has a great deal to do with our problems with engines, and piping, and so on.

HENDERSON, Collier Carbon & Chemical: We had a synthesis gas compressor experience. The compressor started knocking severely and was shut down and examined. The third stage of the compressor operates at a discharge pressure of about 3,000 lb./sq. in. gauge. The piston is held onto the rod by a hex nut, threaded onto the rod, and recessed into the piston. The nut is about 4-in. diam. and 4 in. long. The threaded portion of the rod containing the nut had broken off, and in the space of 1/5 sec., it had come out of the recess, reversed itself, and had gone back into the hole. Fortunately it had jammed itself with only a small portion of the broken end of the rod extending past the end of the piston.

WHITE, San Jacinto: We have had trouble with breaking tail rods and overheating packings. At high pressures packing does not flex up and down at all, but just becomes a very tight bushing. We had made the mistake of not starting our circulation until we needed it, usually between 4,000 to 5,000 lb. of pressure after reduction. If the circulator is started at that time, the packing can't align itself in its natural reciprocating motion. But if we start the circulator at zero pressure, and let it run until somewhere around 1,000-lb. pressure, then it can be shut down, and as long as we have pressure on the circulator, we can start any time. That might be one lead on tail-rod breakage.

CONKLIN, Du Pont: For years we have always enclosed our compressors in buildings. Presently, we are building an anhydrous ammonia plant near Atlantic Refining's installation. This new plant is along the river where winters are not too severe, but extremely damp with much rain and some snow. Underscoring Dr. de Vry's remarks concerning outdoor installations, we did believe it necessary to put a wall on the river side of the building to keep out the wind-driven spray. We also added a roof with a full length ridge ventilator. The other three sides of the building are completely open. The operating floor, which is 7 ft. above grade, is of subway grating construction. All of this makes for a rather cool place to work. However, we believe that all possibilities of forming flammable gas pockets within the building have been eliminated. Any additional reduction in weather protection will not make the installation less hazardous, but will increase operator discomfort to an undesirable level.

Are there any here who can relate knowledge or experience they may have concerning the approach that the Southwestern Research Institute has made in investigating compressor pulsations, piping vibrations, and the like by means of their direct-simulation analog computer?

WAIBEL, Ingersoll Rand: A conference was held in San Antonio earlier this month, financed mainly by all of the major compressor manufacturers and members of the Southern Gas Association. Southwestern has obtained some interesting data and information which will be available in the near future.

CHAIRMAN WALTON: We've attempted to measure vibration with instruments to help us control it, without much success. We have varying rates in our plant because our hydrogen supply is variable. Piping vibration is a continuous problem. We have a boiler-maker who spends almost full time, five days a week, on compressor piping vibration problems, making new braces and tightening up existing ones.

deVRY, Hercules: We found we could do a good bracing job if we planned the piping in the compressor foundation to allow heavy anchor bolts to be buried in the compressor foundation. Such anchors pin the pipes close to the cylinders. It just isn't enough to put piers down. A small pier just breaks loose from its base in a short time. Wherever possible, we provide the anchors in the compressor foundation and wherever possible, we use machine-mounted intercoolers, because they eliminate the pipe run to and from the cylinders.

WHITE, San Jacinto: We have used wood bracing several times. It seems to us that wood has just enough give in it so the anchor bolts don't tear out quickly, and to some exent, it helps absorb vibrations. We use 10 in. x 10 in. timbers.

CHAIRMAN WALTON: We used wood many times for emergency, but we have never left it in for any length of time. It is interesting that you have snubbers, such as Burgess-Manning's, which help but are not by any means the entire answer. On our most recent compressor installation we had such snubbers which did reduce, but not cure, our problem.

During the past year we had a 2-in. connection break about 75 ft. from a compressor. It was a safety valve off the main line which we thought would never break because of fatigue; it cracked. There was no fire, but it caused a shutdown. That connection was replaced with a Taylor forge-type welding nozzle and brace. We originally thought we had to use these heavy weld nozzles in the piping (only) between the compressor and the first major piece of equipment. We have since had failures beyond that point and are expanding our use of heavy welding neck nozzles for such connections.

In every case we had insisted on having flanged valves as the first valve on a small piping connection, from a large line or vessel. These valves, in small piping sizes ½, ¾, or 1 in. are fairly heavy. In the past year, however, we changed to welding a ¾ in. stainless steel Pratt & Cady-type barstock valve into a flange. That has been satisfactory and there's less weight to vibrate.

deVRY, Hercules: Our practice on small gauge connections and sample points on high-pressure lines was to make the connection and then lead the tubing along the main pipe down to some independent point. Of all the piping failures in compressors (high and intermediate pressures), fully 50% of them have come from breakage of cantilevered valve connections at sample points.

CHAIRMAN WALTON: Use of piston-pipe checks has helped in our vibration problems. This is a floating-type check without the vibration that's caused by the swinging check slamming closed after each stroke of the compressor. On equipment procedures for preventing liquid carry-over, one thing which is too easy to do, is to place great dependence on safety devices, liquid-level controls, level alarms, and so on. Routine checks of level alarms and such, by the operators are, I think, quite necessary—something requiring vigilance.

Waibel White Henderson

High pressure equipment

CHAIRMAN WALTON: If a crack develops in a connection off the synthesis loop, what is the practice of handling leaks? Our practice is to shut down and depressurize. If the leak flames, we don't put it out until the pressure has been reduced, feeling that a lot of synthesis gas around the place is more of a hazard than a small fire at the point of the leak, for a short time. Of course, we worry about a leak causing too rapid a drop in pressure, and its effect on the catalyst.

In the history of line failures, has there been any failure in high-pressure synthesis gas systems other than small connections to gauges and sample connections?

WILEY, Standard (Indiana): When our plant was built, the installers **forced** some of our synthesis piping to make it fit. Then we had some failures. We had to take a lot of that piping down and heat it for stress relief to put it back as it should have been. We also had trouble with the interstage piping on our compressors because of poor weld penetration—some as little as 25%. We just completed a turnaround where we had to redo much of this welding.

deVRY, Hercules: We had success with lens joints on our high-pressure piping. We have used copper gaskets between tongue-and-groove-type high-pressure flanges at 1,000 atm. In our recent plants, we have gone over completely to lens joints, as we find they're more easily lined up, stay lined up better, and seem to contain the gas better.

CHAIRMAN WALTON: I can duplicate that experience. We have lens-type joints on our high-pressure vessels which have been extremely satisfactory. Our plant operates at about 4,000-lb. pressure and most of the piping joints are ring-type joints, which have also been satisfactory at that pressure.

WHITE, San Jacinto: Where you have a high-pressure leak, has anyone else noticed that sometimes some finely reduced catalyst (which is usually in your lines) will blow out through the leak and light off the leaking gas. It's a hazard which we now realize.

CHAIRMAN WALTON: That's an ever-present danger. A question was raised as to liquid-level devices in normal and subzero services in high-pressure equipment. Does anyone have problems to bring up? In our high-pressure separator, we have a Fisher-Leveltrol which has been satisfactory. There's always danger that you'll collapse the float, but if its been properly designed, it will be all right.

STOCKBRIDGE, Southern Nitrogen: We have some serious problems with level controls on our high-pressure separators. Would you mind mentioning the ones you use?

CHAIRMAN WALTON: Fisher-Leveltrol. The specifications given to the Fisher people were the operating conditions of the unit. Other than that, I don't believe there's anything special about it.

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We have a spare float for it on the chance that it might collapse, but have never had to use it.

STOCKBRIDGE, Southern Nitrogen: Is it mounted in the vessel, or external to the vessel?

CHAIRMAN WALTON: It's external. One problem that came up with a Magnetrol high-level alarm we installed external to that vessel was that the high-pressure separator in our plant operates at 20°, and in warmer weather, the heat leak into the lower leg to the Magnetrol was sufficient to cause boiling. This made the operation of the alarm rather erratic. It gave peculiar readings. Insulation of the leg corrected the difficulty. The top leg should be of greater diameter than the bottom, because there'll always be some heat leak and evaporation, and if the diameter of the top leg is restricting, it can give a false level.

KELM, Grace Chemical: For a level indicator in the high-pressure separators, we use a DP cell with a purge gas into the dip tube. Of course, the difference in the purge pressure and the vessel vapor pressure represents the level. This is not a controller; it is simply a recorder. To my knowledge we have had no instrument failure up to this time.

Tankage and spheres

KING, Sohio Chemical Co: So far we've heard about the troubles of ammonia manufacture. Of course, there must be good procedures for handling the finished product.

Monitoring vapor space for explosive mixtures occurs when dealing with small amounts of ammonia in air. It occurs more frequently in the user's plant than in an ammonia plant. There are two recorded instances: a Canadian brewery, where small amounts of ammonia in air caused a violent explosion; and a Texas food processing plant, where leakage of an ammonia refrigeration system caused a buildup of ammonia in air, resulting in an explosion. At Sohio, we try to keep equipment in operation for long periods, minimizing this problem.

Water-filling of storage vessels and blending drums and subsequent displacement of the water by gas is good practice for initial startup. In normal operation, where small amounts of air accumulate, an enclosed system can be vented to the atmosphere through an absorber or to a pressure-maintenance system. Normally, small vessels are removed from service by flooding with water after opening to the atmosphere.

Maintaining pressure on storage equipment and relief systems is another problem. To control pressure, Sohio uses vapor recovery systems to an absorber, or to a compression and liquefaction system. The liquid receiver of the vapor recovery system is continuously vented to an absorber system, which recovers any ammonia included in the vent. The vent consists of noncondensables, particularly hydrogen. The absorber system may serve as storage areas, loading racks, blending areas, or processing areas. Ammonia is continuously absorbed in steam condensate and may be sold as aqua-ammonia.

Relief systems on ammonia storage generally blow to the atmosphere where possible. As they age, relief systems must be brought under closer surveillance, and may require changing. Inspection on older storage vessels should be more intense, since stresses, corrosion, hardening, decarbonization, and a variety of things can occur. In a normal situation, a pilot-operated relief may be used.

In cold storage, failures can result from use of improperly processed materials. Cold forms have a high strength and are hard, hot forms are ductile. When shapes are welded together, notch formation can weaken joints. The same thing can occur in storage vessels; a notch can lead to cracking. In pressure-testing these vessels, the temperature should be carefully controlled. Failures can occur at low temperatures even in relatively low-pressure tests.

WHITE, San Jacinto Chem. Co.: One point worries me: putting water into a vessel containing ammonia. There might be some misunderstanding — certainly you'd never want to do that. Several vessels have been collapsed that way. One should always purge the ammonia out before adding water.

CHAIRMAN WALTON: Yes, I saw a tank car "pulled in."

ANONYMOUS: When we put our ammonia spheres into service, we did a lot of soul-searching before we finally said, "Well, we'll put the ammonia in the top and when we get ammonia out the bottom, it's there." I'm wondering just how much of a hazard we've been experiencing.

WRIGHT, Standard Oil Co. (Ind.): On the decision to use this method, the ammonia storage vessels have bottom connections of fair size available. A three-inch line was run from one of these nozzles to a location well away from the vessel, and a flame arrester was installed at the end of the line. Ammonia vapor was introduced at the top of the vessel at a very low rate so that, at most, there would be a very thin interface of flammable mixture.

Data on detonation propagation were examined to assure ourselves that, even if combustion occurred in this thin interface mixture, no damage could result to the vessel. We kept analyzing for ammonia at the base of the vessel and, as soon as it arrived, the vessel was buttoned up. There was practically no air left in the vessel and the residual amount could be safely eliminated through the storage refrigeration system.

CHAIRMAN WALTON: When we prepared our spheres for use the first time, we purged them out with nitrogen and pressured them up to about 20 lb. of nitrogen, released it, pressured again and released, and continued until the oxygen concentration was down to about 6%. Any cars sent to the repair shop are purged with nitrogen until no odor of ammonia is left in the car. And any cars that come in new, whose previous contents are unknown, are purged out with nitrogen. We use nitrogen strictly for that purpose.

We have monitored the spheres a number of times for explosive mixtures and have never found any. We find small (expected) amounts of hydrogen but by consistent use of nitrogen purge we haven't had any difficulties with explosive mixtures. To keep sphere pressures down, because a small amount of inerts always creep in, we maintain a continual bleed, which goes to the aqua-ammonia manufacturing system.

MARTIN, Spencer Chem. Co.: We


Wiley


Conklin


Stockbridge

piped nitrogen to the spheres, which we didn't do originally. We got scared at a safety meeting after learning about static discharges. We used to empty the spheres, then stick a fire hose in the top and let the spray absorb the ammonia vapors. The speaker described some accidents which made us realize that what we were doing could lead to a static discharge. So we pipe nitrogen to the spheres. I think it has taken care of the whole problem.

CHAIRMAN WALTON: Nitrogen is so inert, I recommend its use to any one not using it.

SWOPE, Southern Oxygen Co.: We use nitrogen as a purge gas on hydrogen gasometers and storage vessels as well as acetylene, and other flammable gas containers.

I would like to discuss static electricity. A fire was described, which occurred when someone ran nitrogen into a void space in the top of a cold box through rubber hoses. I'd like to ask the gentleman, if he's present, whether those rubber hoses had any metallic fittings at the free end.

ANONYMOUS: Those were typical air hoses with metal Chicago fittings on the end. The fitttings were not touching the vessel.

SWOPE, Southern Oxygen: If they had been grounded or banded you might not have had the fire. To cite an example of static buildup: we had a line carrying nitrogen which we were using as a drying medium and discharging to the atmosphere through a rubber hose. To build a slight back-pressure on the system, we inserted a small metal ferrule in the end of the rubber hose. The hose extended through the building wall and the ferrule was approximately 6 to 8 in. outside the wall. We found sparks jumping the 6 or 8 in. from the metal ferrule in the end of the hose to the metal surface of the building. There was nothing but nitrogen flowing through the line, at low pressure but at fairly high velocity. The explanation, as I understand it, is that a static potential will build up between two conducting media separated by an insulating medium when a fluid flows from one conductor to the other. Such is the case with a metal fitting on each end.

LARKIN, Monsanto: We use a different method. Our problem has been to purge air out of storage tanks used for hydrogen and nitrogen mixtures. We fill the vessel completely with water and then displace the water with nitrogen.

CHAIRMAN WALTON: We didn't use water because we were trying to keep the ammonia anhydrous; and actually the water left in would not have amounted to much, so we were probably being more careful than necessary.

KELLEY, Standard Oil (Calif.): Our experience in commissioning spheres was almost identical with yours. We pressured with nitrogen, 15 lb. or so, and depressured repeatedly down to about 5% oxygen; then put ammonia vapor in the top. It was interesting to note in analyzing the gas coming out at the bottom, that when the interface hit, we went from about 10% ammonia to better than 80%, rather quickly.

CULP, Grace Chemical: I would like to ask about monitoring the vapor space in aqua storage. We have 5,000,000-gal. tanks for storing 25% aqua. They are uninsulated and breathe heavily during the day and night. We have considered piping nitrogen to them, but to offset the breathing effect, a ten-inch line is necessary. We wonder if anyone has had a successful experience in monitoring this vapor space on a more-or-less continuous basis?

SIMMS, Phillips Chem. Co.: We have 150,000- and 323,000-gal. aqua tanks in which we store anything from 25% to say, 42% aqua. I agree with Mr. Culp that he can get an explosive mixture more readily in an aqua tank than in anhydrous spheres. At about 44^{c}, the 25% aqua, assuming equilibrium conditions, is explosive. At a higher concentration, it may not get explosive until near 0^{c}. In our installations we maintain a natural gas blanket, or use vapor from spheres, for pressure maintenance. If you have it available, your purge gas from synthesis can be used since it is a waste gas. We maintain a constant pressure of between ¼- and ½-in. of water, and breathing out is handled through, essentially, a vacuum absorber for ammonia recovery. It's been successful so far, but we shudder sometimes when we see all these other tanks around the country holding 25% aqua, and everybody thinks they're perfectly safe.

ANONYMOUS: May I ask what you do with the tail gas off the absorption of the vapors?

SIMMS, Phillips Chem.: If it is the purge gas from synthesis, of course we just discharge it into air because normally you would handle your purge gas for ammonia recovery through an absorber anyway. We merely take off what we need for pressure maintenance, bring it back to an absorber, and discharge it there. If it's natural gas, fortunately we're a natural gas producer—it is a loss.

CHAIRMAN WALTON: The "book" says the ignition temperature is 1200^{c} for an explosion of ammonia in air, so people don't worry about it, but maybe we should.

There is some difficulty with corrosion of field tanks of ammonia in agricultural service and the conclusion is that our corrosion problems were confined to the cold formed shapes. We have examined our spheres, after three years' service, by magnaflux and visual examination, and have found no evidence of corrosion. The "orange peels" that went to make up the spheres were hot formed, of course.

Tank cars and trucks

HEDMAN, Shell Chemical: In introducing the subject (from the Compressed Gas Association's viewpoint) "Tank Cars and Trucks" in anhydrous ammonia service, I would like to review some recent developments relative to the increase in use, design, and regulations pertaining to these vessels. The increase in use of transport equipment, of course, goes hand in hand with the increase in the production of ammonia.

Equipment requirements, standards, and regulations, for industrial ammonia have remained more or less constant, while many developments have taken place in the agricultural ammonia industry due to the widespread use of anhydrous ammonia as a nitrogen fertilizer in dry farming areas.

Because of its rapid growth, the agricultural ammonia industry is, unfortunately, going through a trying period during which the lack of nationally recognized uniform standards plays a key part. The average dealer, or farmer, does not have the advantage of technical assistance, and is dependent upon the ammonia producer and the equipment manufacturers to furnish him with equipment which will give him maximum safety and a maximum return for his money. The customer has a right to expect equipment designed for the product he intends to handle and the job he expects to do, as well as protection through uniform standards.

The most widely used regulations have been those issued by the Interstate Commerce Commission applicable to the transportation of anhydrous ammonia in interstate service. Several other organizations such as the Compressed Gas Association, the National Safety Council, the Manufacturing Chemists Association, and the Agricultural Ammonia Institute have published recommendations for the storage, handling, shipment, and use of ammonia. Some 30 associations concerned with safety, compressed gas in general, and anhydrous ammonia in particular, are represented in the American Standards Association, K-61 Committee, presently developing American standards for storage and handling of anhydrous ammonia. We are sure that each of the controversial topics listed in the symposium's agenda will be discussed by the above representative group of experienced ammonia people and that we shall have a workable American standard in the near future.

Some of the most recent changes that have been made or are being recommended at this time in tank car and tank truck standards are:

1. The installation of more 2-in. valves for faster loading and unloading.
2. The recommendation which may make the installation of excess-flow check-valves mandatory.
3. The approval of uninsulated ICC Specification 112A400 tank cars, which will have relief valves set at 300 lb./sq. in. maximum, and a maximum filling density of 57%. However, during the months of November to March inclusive, a filling density of 58.8% may be used if these cars are loaded and shipped directly to the consumers for unloading. Storage in transit is prohibited.
4. A new proposal to allow insulated ICC Specification 105A300 tank cars to be filled to 58.8% from November through March is also being discussed.

Tank trucks for transporting anhydrous ammonia have been one of the controversial pieces of equipment in recent years due not only to the increase in use and dual service, but principally because of the 265 lb./sq. in. minimum design pressure required by the ICC Specification MC-330. Even though anhydrous ammonia has a higher vapor pressure than LPG, and would lower the present safety standards, many attempts have

Martin Kelm King

been made to change the regulations to allow ammonia to be shipped in existing 250 lb./sq. in. design pressure cargo vessels.

The most recent study made by CGA confirmed its earlier recommendations that ICC Specification 51 portable and ICC Specification MC-330 cargo vessels for anhydrous ammonia should be designed for a minimum working pressure of 265 lb./sq. in. gauge.

Velocity checks or excess-flow check valves have always been a controversial subject because of the difficulties they cause while unloading. These valves are necessary safety features and are designed to contain the product within the vessel in which they are installed in case the block valves on the vessel are sheared

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off. However, these valves are not designed to protect the material in the car or truck when a break occurs on the downstream side of an unloading system. These systems and storage tanks should be protected independently with supplementary check valves. Excess-flow check valves are necessary for safe operations even though they are touchy and some improvements in their design are in order.

Unfortunately, I don't have design data for the various types of excess-flow check valves. However, results of studies made of unloading rates by various methods vary considerably. The most accepted rate of maximum flow seems to be somewhere in the neighborhood of 50 gal./min. But tests indicate that with

Kelley Swope Simms

proper systems and operating techniques, there shouldn't be much difficulty in loading at 100 gal./min. In any case, one of the most important features, governing efficient unloading operations, is to train the operator to open valves in such a manner that sudden surges of ammonia will not shut excess-flow valves during initial startup operations.

Inspection and maintenance is a never-ending job, if done properly, to insure safe operations. Tank cars and trucks used interchangeably in LPG and ammonia service should be cleaned and inspected when entering ammonia service as well as when returning to LPG service. The following is a suggested cleaning procedure: 1. Depressurize tank car by pulling vapor to atmospheric pressure at plant. 2. Connect vapor valve to steam neutralizer. Vapors are mixed with steam and discharged to the atmosphere through a pipe extending into the air. 3. Connect steam to liquid valve and steam car until fumes disappear. 4. Dome cover plate with valves and piping is removed. All angle valves, safety valves, and other fittings are removed, inspected, and repaired. Packing glands are inspected, repacked if necessary and lubricated with grease suitable for the commodity for which the car is being cleaned. 5. Water is sprayed into the car at 150 lb. /sq. in., then pumped out and the interior of the car dried with air. (If car has excessive scale, this is removed by sandblasting.) Tank walls are then wiped dry and cleaned with a vacuum cleaner to remove all fine lint or other foreign material. 6. Commodity stenciling must be changed on each side of the car to indicate the product for which the car has been cleaned. 7. Air is purged from the tank at the plant.

Air purging of tanks is very important. Air that is not removed from ammonia tanks prior to being filled with liquid ammonia will raise the total pressure in the tank over and above normal ammonia pressure. As an *extreme example*, if the tank is initially filled without a vapor return, this excess pressure will amount to approximately 83 lb./sq. in. for a tank filled to the 85% level. However, since a tank is generally filled with a vapor return, this excess pressure, for an initial fill, normally will amount to some 15-25 lb./sq. in. if no air is purged from the tank prior to the filling.

Many of the leaking relief valves on tank cars and trucks as well as portable and storage containers, are attributed to the lack of proper air purging practices. Recent investigations into the cause of stress corrosion crackings of ammonia containers indicate that improper air purging may be one of the causes. Every effort, therefore, should be made to eliminate air from ammonia systems.

Specifications for tank car and truck unloading as well as portable container filling hose, have finally reached a point where the industry has accepted a uniform standard. The basic specification for 1-in. and 2-in. tank car and truck unloading hose is wire-reinforced high-pressure hose with a maximum working pressure of 350 lb./sq. in. or greater, a minimum safety factor of 5 to 1, that is, a minimum burst of 1750 lb./sq. in. The cover stock is pin pricked black neoprene. The tube stock is ammonia-resistant GRS (or approved substitute). And the markings shall be permanently marked, that is, etched, cast, or impressed every five feet with the manufacturer's name or brand, the working pressure, year of manufacture, and Anhydrous Ammonia also indicated. The periodic test of hose assemblies, however, is still a very controversial subject. Several states have regulations requiring periodic hose tests, but there is no standard to guide them. The test pressure for new units is generally twice the maximum working pressure and retest pressures vary from ⅔ of the maximum working pressure of the hose to 1000 lb./sq. in.

COCHRANE, Sun Oil Co.: Testing of the loading hoses has been something we've always been concerned about. I would be interested in hearing what other people do, what kind of inspections they have, and how often.

deVRY, Hercules: We don't test them; just change them once a year.

I'd like some comment on this question of quick coupling on the ends of the hoses. We have used what looks like a half union. It's set up for a quick-coupling job. I don't know whether it's going to be adopted as a standard, but we see nothing unsafe about it.

WHITE, San Jacinto: We have standardized on a Weco-type coupling and several producers in our area have also adopted this coupling. It's fairly quick coupling but safe and practical.

CHAIRMAN WALTON: We also use it. It's a coarse-threaded union. We tried a number of the quick-coupling devices on the market and gave up on them. We were afraid of them. They stuck at times, and there were too many features that frightened us.

WHITE, San Jacinto: We tried to find a rubber that will take the cold temperatures you get when you take the pressure off these hoses, because each time you finish loading the truck and relieve the pressure, it gets very cold. We found that quite a few of the so-called ammonia hoses would crack. We do specify that our hoses will not crack, and actually test them. We change them once a year and use those removed for low pressure aqua service around the plant.

CULP, Grace Chemical: We, too, have found that hoses cracked from cold ammonia when disconnected. As a result we have standardized on hydrostatic testing of hoses once a month. I'm not sure of the pressure, but I believe it's 400 lb./sq. in.

DAVIS, Mississippi River: LPG hose operates at about the same pressure as ammonia-loading hose. I remember one plant in which a hose burst. There was an immediate clamor for hydrostatic tests. About 15 hoses were tested at 275 lb./sq. in.—about the maximum allowable working pressure. In no time new leaks began to appear. After some discussion it was decided that all high pressure propane hose would be changed at the end of a year, the old hose to be put in low pressure LPG service.

An inspection of ruptured hoses sometimes revealed the hose connections had been improperly inserted and had injured the hose tube. With most of the small leaks, the outer fabric would swell up and look very dangerous. "Pinholing" of the outer fabric was the solution for this.

MUZZY, Chemstrand: We had four rubber hose failures as we were starting up our ammonia handling facilities. These were ruptures, not cracking failures. Ammonia evidently penetrated the inner layers of the hose structure to collect near the outer surface. The higher temperature there caused a bubble to form and burst the hose covering. We solved our problem, not by changing hoses every year, but by changing to flexible stainlesss steel hoses which have caused us no trouble to date.

CHAIRMAN WALTON: The initial hoses we had developed bubbles also. We are now using U. S. Rubber style P-7130 which has proved satisfactory (no failures, leaks, or bubbles). It meets the standards Mr. Hedman mentioned. It has a wire-braid reinforcement with a neoprene cover that's pinpricked so that if leakage develops from the inner tube it'll show up quickly. Every six months we test at twice working pressure. Usually when the jacket starts to show signs of wear from abrasion against car domes or such, we retire it to aqua service. The company we lease our cars from inspects safety valves every five years, tanks every ten years, and never inspects velocity checks unless the customer asks them to, or somebody reports something wrong with them. The latter is a rotameter-type design with a floating plug.

STOCKBRIDGE, Southern Nitrogen: I wonder how often anyone here inspects velocity checks in his own cars?

CHAIRMAN WALTON: To see if they're dirty or stuck?

HEDMAN, Shell Chemical: That's right. With the rotameter type, there's not much you can do except to see that they are clean, free and properly machined in the first place. We do this as a regular routine. We inspect and/or overhaul all the fittings in the dome.

CHAIRMAN WALTON: Does Shell Chemical inspect theirs regularly?

HEDMAN, Shell Chemical: That's right.

CHAIRMAN WALTON: How often? Every five years?

HEDMAN, Shell Chemical: Of course the relief valves have to be tested every five years by ICC Regulations. The cars go into the shop for full overhaul of fittings at that time. We've had considerable difficulty in the interchange service from LPG to ammonia and also from ammonia to LPG, so it has become necessary for us to run those tank cars through the shops for cleaning prior to customer service. At that time we inspect and/or overhaul all valves, but we do not have a set period under five years.

CHAIRMAN WALTON: I notice in Mr. Hedman's presentation that the Compressed Gas Association is apparently going to recommend complete cleaning of cars in shifting from LPG to ammonia. Am I correct in that, Mr. Hedman? The cars are put into the shops and cleaned?

HEDMAN, Shell Chemical: No,—that has been discussed from time to time in the CGA, but it was one of Shell's recommendations that it be done.

CHAIRMAN WALTON: We have taken some cars which have been in LPG service and purged them with nitrogen until free of hydrocarbons and put them into ammonia service without internal inspection. Has anyone else done that, or are we alone?

WHITE, San Jacinto: We had done that, especially in hauling on trucks, because very often they want to haul a truck one way with LPG and the other way with anhydrous ammonia. We had quite a fight on our hands to insist that they even purge them out, because the truckers say most people don't worry about that.

That brings up one other point while I'm up, if I may. Has there been any consideration given to differences in specs for tank trucks which use the tank itself as a frame for the truck? I've always worried about the extra stress due to rough roads, putting additional load on the tank, in addition to the internal pressure of a load of ammonia. I've often wondered whether there's additional strength designed into the tanks for this extra load.

HEDMAN, Shell Chemical: I don't know whether I can answer this question adequately, but I believe the required structural characteristics are taken care of in the original design to adequately withstand the stresses thereby imposed, in addition to those covered by "the Code." (For further reference, see Para. 78, 336-4 (C), ICC Regulations.)

Preparation for repairs

MARTIN, Spencer Chemical: One of our workers, working on an oxygen line from below, which had been depressurized but not purged, suddenly found a lively fire burning around his neck, ears, and face. He knocked off his mask and extinguished the fire easily, suffering only minor burns and the loss of his shirt collar. Cool oxygen gas from the pipe opening had flowed down over his head and shoulders, saturating his shirt. A welding spark, which would ordinarily have done no more than burn a hole in his shirt, set it afire, and it burned intensely until the supply of free oxygen was exhausted. This, of course, represents insufficient preparation for repairs, but who would have foreseen that accident? The job was safely finished with a small nitrogen purge going through the line. So much for the wrong way!

In Spencer's organization, the primary responsibility for the safe preparation of equipment to be repaired lies with the operating group. This group is most familiar with the process, and therefore, with its hazards. To be sure, maintenance personnel are expected to satisfy themselves that their undertaking is safe and that they observe the common safeguards for the type of work they're doing. Safety personnel are present in an advisory or consulting capacity and while they have the authority to stop a job, this is rarely done. In fact, operations and maintenance rarely disagree with safety section recommendations or suggestions. When this happens the matter is postponed until a solution can be reached which is satisfactory to everyone. Normally, the shift supervisor, or someone in that capacity, has the ultimate responsibility for seeing that the job is carried out safely. In some cases where the hazards justify it, the plant superintendent, the maintenance superintendent, and the highest of the local management may take a direct hand in the proceedings.

Clarity in explaining the exact nature and location of the job to be done is very important. This was illustrated when we had a maintenance crew start work on the wrong one of two identical exchangers. One had been prepared for them and was safe; the second was not. In fact, it contained hot MEA under pressure and this is the one on which they started to work. Fortunately, they were stopped before they got far enough to be in trouble.

Preparation for repair may be said to start with the writing of the maintenance work order. The writing is done by a supervisor as soon as he recognizes the need for maintenance. In addition to space for a proper job description, our maintenance work orders include several lines labelled "safety precautions." This space is not always sufficient to detail all the precautions necessary, but it is sufficient to act as a "flag" for the haz-

ards involved and the writer is expected to list them. The work orders are reviewed by plant and maintenance superintendants before being issued to the people who will actually perform the work, so that several opportunities exist to recognize safety hazards and specify proper precautions. Shutdown work orders are further reviewed in a regular weekly production meeting. This meeting includes representatives of operations, maintenance, engineering, technical or laboratory, and safety. They are considered especially qualified to recognize hazards and to suggest precautions. Detailed, stepwise procedures are written for major, but infrequently accomplished, repair work. This not only expedites the work, but reduces the chance of error by men unfamiliar with the particular job. Many of these procedures are included in our operating manuals.

A daily operating bulletin, which contains production plans for the ensuing 24 hours, is also used to publish safety procedures relating to maintenance occurring during that period.

Occasionally exceptional circumstances will require consideration by the best qualified people in our company. A case in point is our procedure for purging and warming the nitrogen wash equipment. Due to an internal explosion and consequent damage which occurred in the wash tower in early days of operation, and which was never satisfactorily explained, the procedure now followed is very detailed and covers several pages.

The physical preparation often involves first depressuring or venting dangerous gases. (The liquids, such as caustic and MEA, can be taken care of by sufficient water flushing.) At our Vicksburg works, it is possible to burn all vent gases containing a high percentage of carbon monoxide. Other combustibles, such as hydrogen and natural gas, are not burned unless they are tied to the flare stack for convenience. However, they are normally vented at a high point. Regardless of whether the reason for purging the gases is due to their explosibility, toxicity or because they are capable of asphyxiation, purging methods are similar. Purging is preferably done with a once-through sweep—in one end of the pipe or vessel and out the other, at low pressure and high volume. This results in maximum displacement of objectionable gas by the gas being used for purging. Less desirable, but occasionally necessary, is purging by pressuring and depressuring through the same opening. If not done thoroughly, this procedure can leave pockets of toxic or combustible gases within the purged equipment.

Nitrogen from an air separation plant is a very satisfactory general purpose purge material, particularly when the equipment being purged is to be kept

* SAFETY ROUNDTABLE

dry. It's inertness makes it satisfactory for purging explosive and toxic gases. Where separation plant nitrogen is not available, a mixture of nitrogen and carbon dioxide (products of combustion) can be used in the same way, but this gas is rarely dry unless a special dryer is used. If the unit being purged is to be entered during maintenance, it's mandatory that the inert gas be followed by air providing sufficient oxygen for men within the vessel. This may seem fundamental, but in two separate instances we found it necessary to rescue people who had entered the nitrogen wash cold box and become unconscious because it was filled with nitrogen. In some cases steam may be used as a satisfactory purge material, in a shift converter, for instance. It is readily available and will not dry nor damage the catalyst, as would nitrogen. We have occasionally found it desirable to keep a small purge going through the equipment undergoing repair, when it's desirable to exclude air. Again, an example is the shift converter. In some circumstances, a vessel or line can be purged by filling it with water, or carbon dioxide if it's available.

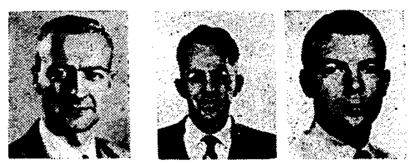
Several analytical methods are available to determine whether purging is complete. The Davis Equipment Co. explosive detector is a satisfactory device in capable hands for determining the presence of an explosive gas mixture and the MSA sniff-type CO detector is convenient and accurate. Orsat analyses may be used to determine overall composition. A German "Drager" tester (Model 19/31 Multigas detector available from Safety Supply Co., 214 King Street East, Toronto 2, Ont., Canada), which we recently put into service, can detect a variety of hazardous gases and vapors, such as oxides of nitrogen, trichloroethylene, and CCl_4. If necessary, gas tests are conducted at frequent intervals while the work is in progress.

The use of blanks at flanges, and tags and locks on valves and switches is determined for each particular job. Blanks are always used to isolate a vessel which will be entered by personnel. They may also be used on piping where the hazard of burning or welding is great. Several times we've stopped someone in the act of breaking the flange on the pressure side of a closed valve to insert a blank. The use of chalk or soapstone marks is helpful in preventing this.

Valve tags are placed and signed by operating supervisors. They have detachable stubs which are also filled out and presented to the maintenance crew (I think these are fairly standard) after the tags are placed. These stubs are held by "maintenance" until the job is finished. No tag is to be removed until the stub has been returned to the operating supervisor who removes the tag, staples the stub to it, and forwards them to the area superintendent to show that this safety feature has been observed.

Electrical switches are usually locked open and tagged. Our electrical tags may be placed by electricians as well as by operating supervisors. This is the only exception to our rule that all safety work permits and tags will be placed and removed by operating supervisors.

When the area containing specific equipment has been properly prepared for maintenance, the operating supervisor fills out and signs the safety work permit in triplicate. This is a lengthy form which serves to state that the equipment is safe and as a check list to make sure certain things have not been forgotten. The original copy goes to the mechanic in charge of the work and is good for only one day. Other copies go to the area superintendant and the safety section. At this time the operating supervisor is expected to explain to maintenance personnel any hazards involved in the work, what precautions have been taken to protect them, and generally satisfy them that the area is safe. An operator or supervisor may be stationed in the immediate area while maintenance is being carried out to guard against unexpected hazards or unsafe practices. The operators are expected to feel and share the responsibility for safety and usually do all the preparatory work under the direction of their supervisor.



Cochrane **Hedman** **Muzzy**

Everyone concerned is challenged to be continuously alert for hazards unique in the particular area where work is being done. We also try not to forget the more common hazards, such as hot water, the absence of necessary oxygen for breathing, and high-pressure air. While we have a number of rules, they are to be considered for guidance only, and not as substitutes for careful thought on each job that's undertaken.

CHAIRMAN WALTON: One other thing I might mention in passing, is that we have had a couple of occasions where we had people working outside a vessel that had a nitrogen purge going through it, and up on a platform. The wind direction and velocity were such that it created an oxygen deficiency and a man got dizzy and nearly fell off the platform. So, you not only have to watch out inside vessels and inside cold boxes, but also outside if you have a nitrogen purge going through a vessel or a line at that point.

LARKIN, Monsanto: Our procedures for tank-entering and fire permits are similar to those at Spencer. I would like to elaborate a little on what Monsanto does.

The "Tank Entering Procedure" includes any equipment which may present the same hazards as a tank. A permit or form is used to make certain that all possible hazards are checked. Permits are usually initiated by the foreman or supervisor of the department where the work is to be done. It provides:

1. general description and location of equipment.
2. last product in tank before wash.
3. cleaning method used, or to be used, to prepare for entry.
4. precautions check list includes:
   - A. agitator motor leads disconnected in the starter or at motor. (No exceptions.)
   - B. all lines to and from vessel banked or disconnected.
   - C. a fresh air supply from a blower or from air bottles is discharged into the tank during the entire course of work.
   - D. watcher is located at opening to the equipment 100% of time work is in progress. (No exceptions.)
   - E. person entering must wear a wrist harness with life line attached, unless there is a bottom entry to the vessel.
5. if for any reason one of the aforementioned items is by-passed it requires approval at the superintendent level, or above. This approval is signed on the form.
6. special precautions to be taken other than those normally included on permit, such as protective equipment or clothing.
7. explosive gas testing result, time and by whom. (Required on all tanks by a trained tester.)
8. field inspection approval by foreman to ascertain if all precautions have been carried out.

We also require a fire permit before any spark-producing equipment can be used. The permit must be displayed on the job at all times, while the work is going on. The permit is generally initiated by the department foreman or supervisor where the work is to be performed, and approved at superintendent level, or higher. It includes:

1. general description of work to be performed, its location, dates, and times.
2. type of equipment or tools to be used.
3. all closed equipment and/or hazardous areas require gas testing for explosive atmosphere.
4. all special precautions required for the particular job are listed.
5. the area is checked for compliance to all precautions and signed for by the field inspector (normally the foreman). He signs the permit and witnesses the startup of the job.

These procedures do cause delays but have, for all practical purposes, completely eliminated accidents caused by unsafe tank conditions, and fires from careless sparks.