

GENERAL DISCUSSION

AIR INTAKES

ANON—Peter Hepp of Sun Oil Co. has done quite a lot of work in this regard on dual intake and its effect on hydrocarbon patterns in the air separation boxes. This year we undertook to do much the same thing, only in order to do it economically we first had to find where best to locate our air intake. Our particular plant is surrounded, you might say, by an industrial area, refineries, ethylene plants, polyethylene plants, in such a manner that it becomes very difficult at first sight to determine where best to locate a second air intake. We are at present trying to solve this problem by running a temporary air intake.

This consists of a vinyl-coated fabric made up in a chute form, suspended on a series of rings which actually are the rings that go around these barrels and this looks like a dragon, and as a matter of fact, the operators have affectionately called it "The Dragon." We run this for some 300 or 400 feet away from the plant and we can move it in practically any direction and by charting the various hydrocarbon patterns, we hope to be able to determine the best location for our air intake.

It worked out quite cheap. It's not going to last too long, but at least it will determine where best to locate a permanent air intake.

JENKINS—U. S. Industrial Chemical. We think we've found our solution to the air plant air intake but we have a problem with corrosion—that is, sulfuric acid fumes in the air—and we're trying to find a way to eliminate sulfates and other corrosion products. Our present tack is to try and put some kind of a water scrub on the front end of the suction. This may increase the humidity in the air and cut down the capacity of our compressor by the fact that you have higher absolute humidity in the air. Corrosion is a rather serious problem to us. We have air compressors with brass tube inter-coolers and corrosion has been quite a problem.

We'd like to find some way to eliminate corrosive materials from the air. I wonder if anyone has found any way to do this economically.

WELLS—American Oil. In our Hammond plant, we have the same problem and we're looking for the same solution. We have in our air compressor for the nitric acid plant, a nickel coating and this has let us get by with no reduction in capacity from one year to the next. Previously, without the coating, we had been unable to do that without serious loss in capacity.

The scaling that we get is largely the sulfates that you talk about with an appreciable quantity of salt which I guess is an iron ammonium sulfate complex, so we are apparently getting SO_2 with the ammonia that's always in the air around the plant—this gives us that complex.

We've had corrosion problems on our centrifugal intercooler compressor. They have usually been in the latter stages where the air is always at the dew

point and moisture is separated. We've gotten by there by cleaning it up, replacing the rotor after the year's operation and we have suffered a little loss in capacity but it has not been serious at the end of the run. But I think this is, for people in an atmosphere such as ours, one of the pressing problems to get a good solution.

Compressor Lubrication

KEITH—Hydrocarbon Research. Last year we delivered a short paper on the non-lubricated compressor we had installed, which at the time had 5,500 hours of continuous operation on it. I just want to spend a minute and bring the symposium up to date on what has happened to that machine. As you'll recall, that was a nitrogen machine, four stages, six throws, discharging dry nitrogen at about 600 lbs. It was a non-lubricated machine using Teflon compressor rings and Teflon rider rings. At 5,500 hours we replaced the rod packing on all stages and we replaced a compression ring and a rider ring on one piston. The rider ring and compression ring that were replaced at 5,500 hours were not damaged in service but were damaged in removing and so we had to replace them.

The machine now has 13,000 hours operation on it. We changed the rider rings in the 28-inch piston and the 9-inch piston at 11,800 hours. The other stages weren't touched. We also replaced a rod packing at 11,800. In order to determine wear on the rider rings, we measured the distance between the rod and the packing cup and we found something which we thought was quite unusual. We would show a measurable amount of wear but in checking it the following week, no wear was apparent.

That is to say, the wear we had shown earlier had been eliminated and in shutting the machine down and investigating we found that the rider rings actually move around the piston. They don't stay in one location. Either vibration or the way they're dovetailed together, caused the rider rings to move around the piston and we get fairly even wear. Other than that, we have had no problems except once in a while we will find a rod packing will begin to blow a little bit.

We have found that if this packing does blow a little bit, very often if we leave it alone, apparently the action of the rider rings turning on the piston will affect the location of the rod in such a way that this spring-loaded packing will seal up again. And at this point we're very satisfied with the operation of that machine.

Q. What pressure is it operating at?

KEITH—We take suctions at about 16 lbs absolute and discharge at 565 lbs absolute. The packing is a non-lubricated Teflon packing. It is cooled by an indirect type of cup around the packing which is of course cooled by water. No water is dripped or sprayed on the rod.

Q. Do you have any rod wear in the packing area?

KEITH—Yes. As a matter of fact, I don't recall specifically what all the details were. I do know that last year I think Mr. Penrod discussed the fact that in one case we had a rod which appeared to have been undercut by the action of the rubbing of the packing on the rod. There was some talk about turning the rod down and using smaller packing. However, we didn't do that. We replaced that rod.

Q. Did you have glass filled Teflon or carbon?

KEITH—Glass filled Teflon.

Q. The trend is obviously towards non-lubricated compressors. I would like to ask some of the compressor manufacturers whether they have made progress in converting existing compressors to non-lubricating compressors. This would be an interesting subject.

HUNTER—Cooper Bessemer. The biggest problem in converting lubricated machines to non-lubricated construction is with the dog house because of the tendency for the lube oil to migrate from the crankcase into the compressor cylinder, so it becomes necessary to install an extended distance piece. Extended distance pieces amount to quite a bit of money. Some people have been doing some work with the minimal lube approach such as we mentioned in our paper last year. This reduces oil requirements to about 30% of normal and does not require extended distance pieces. Our experience with Teflon indicates that a small amount of lubrication is worse than no lubrication at all.

SOMMERS—Pennsalt Chemicals. There are really two problems—keeping the synthetic lubricant out of the crankcase and in the case of the non-lubricated machines, keeping the cylinder lubricant out of the rod packings and cylinders. We have had the first mentioned trouble and have observed that the heavy paint film on the inside of the crankcase is attacked and comes off, getting into the oil stream and causing filtration problems.

Normal crankcase painting is not designed for synthetic lubricants.

NICHOL—Air Products. We have our crankcase lubricant checked every so often by the lubricant supplier and they examine it for cellulose contamination and once they scared me by telling me I had 20% cellulose in there.

The methods that they had been using to determine the contamination apparently worked out around 1% but don't work when it gets to 6 or 7. So, if you are dealing with percentages around 5 or 6 and having them reported to you, it may be well to ask them to examine their technique and find out whether its really giving you a good figure or not.

HOUSER—Navy Bureau of Ships. This may at first seem to be out of step with the compressor lubrication question. The gentlemen who described the explosion talked about first having an air stream going through the compressor and through the oil separator and then subsequently had a nitrogen stream going through this machine. And then at the end of his talk he spoke about the fact that the concept that the nitrogen may have deoxidized the surface of some of the metal and then subsequently when he had an air stream that this could have caused a re-oxidation of surface and the metal in it caused the explosion. Sometime ago I read an article in which somebody was pumping oxygen with a pump—I believe this was liquid oxygen—with carbon ring pack-

ing and the pump worked fine—there was no trouble with the carbon at all. As I recall, they then used nitrogen in the same pump and observed sparks coming out of the packing of the pump.

The theory was that the nitrogen took the oxide film off the carbon and then as soon as the carbon got out and hit the air that the carbon was sensitive and of course started to burn. And I am wondering if there are any other experiences of people having trouble in which they first pumped nitrogen and then pumped an oxygen containing gas.

MASON—Dow Chemical. I'm not sure whether this incident referred to by Mr. Houser is the same one that was discussed at a previous symposia or not. As I recall, this previous discussion covered the use of a vacuum pump alternately on oxygen and nitrogen gases. In that case, it was believed that finely divided carbon had accumulated in the pump during its operation on nitrogen. The breaking of the carbon-carbon bonds during wear in the absence of oxygen left this carbon in a nascent state which oxidized very rapidly immediately upon exposure to oxygen.

We had a somewhat similar experience at Midland with the packing of a liquid argon pump. The original packing was a "Graflon." packing composed of shredded teflon and graphite. During operation of this pump, sparks were observed at the outer edge of the argon fog due to pump leakage. We concluded that these pyrophoric sparks were due to oxidation of the finely divided graphite made nascent by the breaking of carbon bonds in the absence of oxygen or any other reactive material.

Perhaps the oxidation of the metal in the incident referred to by Mr. Houser may have been similar. Metal produced by reduction of oxides or divided to a fine powder in the absence of oxygen might very likely be pyrophoric.

HEPP—Sun Oil. With regard to this initiator in the air reduction explosion and this compressor—there is a Bureau of Mines paper, RI 4465 2 19 49, which deals with the oxygen carbon complex as a possible initiator of explosions in compressed air systems. Now since the service switches back and forth or has oxygen in the nitrogen stream, this could be a possible initiator.

HUNTER—I wanted to clarify one point. I had a question asked me during the interim period here. In my comments I want to make it clear that Teflon rings and packing will stand lubrication. At the present time, we feel it will stand maybe 30% of what you'd use for metallic rings, or above, but there's somewhere in the region between 30% and 0% lube, that Teflon will not behave very well and you're better off if you're going down into that region to go non-lube absolutely.

Catalytic Oxidation

DJUVIK—Rohm and Haas. We have had the MSA type filters ever since the plant was built. They started at something like 90% efficiency for removing acetylene. We noticed that dropped off and finally ended up around 50% acetylene removal. So during the summer months when the wind is normally from an area that doesn't allow much acetylene contamination, we removed all the cartridges and have sent them back to MSA for reconditioning.

Some of the cartridges were collapsed very slightly and some of them had apparently lost activity as shown by this 50% drop and they are being repaired.

BOLLEN—Dow Chemical of Canada. We have an MSA

catalytic filter on the air feed to our Air Separation plant too. Our problems with this installation have been due to compressor oil in the air stream. The combustion of this oil in the catalytic filter has resulted in much shorter catalyst life than was originally expected.

Our catalytic filter is located immediately downstream of the reciprocating air compressors to take advantage of the heat of compression. The hot air passes through an M.S.A. oil removal prefilter before entering the catalytic filter. This prefilter is supposed to reduce the oil in the air to the catalytic filter to tolerable limits. Several attempts have been made to improve the design of the prefilter to increase its efficiency but short catalyst life is still a problem.

The accumulation of oil in the system immediately ahead of a hot catalytic filter is also of considerable concern to us. We have had one fire in the oil prefilter since the installation was started up. This fire occurred when the installation was being shut down. The installation had been by-passed and isolated from the air stream by means of block valves prior to being depressured. The operator opened the bottom blow-down valve on the prefilter but found it plugged. He had just turned to open the blowdown valve on the adjacent catalytic filter vessel when the prefilter blow-down valve blew clear behind him with explosive force and fire. The heat was intense enough to badly burn the steel valve and the bottom of the steel prefilter vessel.

It was reasoned that when the prefilter blow-down valve blew clear the hot gases from the catalytic filter came back through the prefilter and ignited the oil which had accumulated there.

We have changed our operating procedure to ensure that the catalytic filter is allowed to cool down before depressuring and that it is always depressured before the prefilter. We have also instituted a program of changing out the prefilter inserts on a much more frequent basis to try to prevent a large accumulation of oil in the system.

Silica Gel Absorbers

HEPP—This morning the question was raised about when to discard silica gel absorbers and if anybody had any factual information on the selection of discard time. I have some notes that I made in the Tulsa meeting in the untaped section in which Dr. Karwat of German Linde presented certain of his data. I've talked to Dr. Karwat and he has graciously consented that we read them into the tape portion this time.

Dr. Karwat added oil to silica gel in 1, 2, 3, 4 and 5% by weight concentrations, then placed the gel in liquid oxygen and ignited it, with the following results: 1% oil, nothing happened. 2% oil, nothing happened. 3% oil, scattered burning. 4%, more scattered burning. 5%, even more scattered burning. As a result, we, at Sun Oil, are using a criteria that 1% oil is sufficient to justify discarding the gel. As I mentioned this morning, I think that it's perfectly possible to operate and get 1% in the front end and not have the overall efficiency of the gel impaired in any way.

HEPP—I do have some additional data that I'd like to report, which is a supplement to the paper that was given last year by Mr. Schilly of our company, on the hydrocarbon accumulations in an air box.

At that time, we reported that we had undergone four separate defrosts since the start of the plant and that if the initial couple of defrosts were eliminated because we were still learning how to operate the plant, that the amount of hydrocarbons removed in the last two defrosts showed that about 0.28% of the hydrocarbons

that entered in the feed air were being removed on the annual defrost.

We defrosted again last year and this time we found that 0.33% of the hydrocarbons that entered the plant through the life of one run—one year—were removed on the defrost. So we have here 3 years of information which indicate that the amount of hydrocarbons retained, at least in our plant, is fairly consistent and represents about 0.3 of a percent of the entering hydrocarbons retained inside the plant and we get them out on defrost.

The same paper contained information on the amount of lube oil removed in solvent washing our plant. At that time we had had one good run of a year and solvent washing removed 1.6 pounds of oil—this information is in the paper of last year in Volume 4, Safety in Air & Ammonia Plants—this amounted to .005 pounds of oil accumulated per stream day. It was a one year run.

Now last year we solvent washed after a 3 year run. So we had an opportunity to make an interesting comparison between the amount of lube oil accumulated in 1 year versus the amount of lube oil accumulated in 3 years. And we found that in 3 years we accumulated 3 times as much lube oil.

Frankly, I was of the opinion that we accumulated this oil in a short time and it was simply what wasn't removed in exchanger defrosts and possibly a pound and a half of oil is all you'd ever find in there if you ran for 10 years, but this is not the case. Apparently the oil builds up in a uniform manner and at least in our case we feel that we can predict fairly accurately.

We accumulated a pound and a half of lube oil in the front of the plant each year, year after year.

Q. How about your desiccators and your desiccant removal filter?

HEPP—We have all the usual filters and dryers and we run them at what we consider to be good operating practice. We have reciprocating feed air compressors and have Peerless separators after them. Then we put the air through alumina oil absorbers which we discard before they're saturated. Then the air goes through the driers. So that the oil goes through Peerless separators, alumina oil absorbers, driers, and into the plant, but it is retained in the front end in the exchangers.

Q. Also your caustic and water wash?

HEPP—We have a four stage compression and the caustic wash is in between the second and third stages, so the oil can be coming from the third and fourth stages.

LAWRENCE—Armour & Co. So that could make some difference. The reason I ask you is that we haven't experienced it quite that heavy and the caustic and water wash are after the last stage.

HEPP—Bill Mason asked a question, could any of this oil be coming from expanders. No, absolutely not. The expanders are downstream from where we're picking this up and also we have what we consider to be positive proof that oil from expanders can't get into the box. Also, the expander outlet piping is always oil free when we examine it.

Standards of Cleanliness

WALTON—Walton, Sun Olin. In past symposiums we've had various people speak about standards of

cleanliness in oxygen systems and I had the experience here in the past year of putting into service an oxygen pipe line and a system including control valves and a steam heater and various control devices in an oxygen system which supplied oxygen to a secondary reformer.

I remembered very well a statement that was made in this symposium that no matter how many certificates you got with the valves saying it had been degreased by the manufacturer, just don't believe them. Go ahead and examine it and degrease as if it had never been done. I insisted that this procedure be followed in the case of this system. The contractor's oxygen expert was equally insistent on an extremely thorough inspection and cleaning. We took apart every valve. We removed every packing. We found valves that had certificates attached to them that had packing of graphite impregnated asbestos. The certificate said that it was degreased Teflon. We found a stainless steel check valve also with a certificate on it. It was full of vaseline.

Miscellaneous

DOYLE—Factory Insurance. I don't know just where this comes in. It's not mentioned specifically. I have a personal and probably prejudiced phobia against taking H₂S out of synthesis gas and putting it in the main boiler.

Because if you get an upset and put synthesis gas in the main boiler and blow up the boiler, the plant goes down and we start paying. So if you can find anyway to avoid it, please do so. Thank you.

DJUVIK—I might mention a fire we had at a Texaco generator. It wasn't too serious, but it could have been. We feed all the oxygen and natural gas through separate metering runs—it consists of a flow control valve in the orifice plate and a 3-way valve (in the case of oxygen when you're starting up the 3-way goes to a vent, in the case of the natural gas it goes to flare) and a block valve which is a fairly fast acting thing and about 20-25 feet of run through check valves and then on into the burner itself.

On a start up, we ran into a fire at the oxygen 3-way valve that succeeded in burning out almost the entire valve—it burned out the seat, most of the plug; it burned the stem in half and it burned the vent line off. This is, I believe, a 4-inch valve. It was found to be due to a leaking check valve in the oxygen line up near the burner. You light this generator by first starting the natural gas flow, but it came back through the oxygen check valve, back through the block valve and out the 3-way oxygen valve which acted like a mixing tee. Quick action stopped any further damage, but it could have been a lot worse.

Q. How closely related do you think the preheat temperature would be to an accident like that?

DJUVIK—The generator was hot. The gas that had backed out all the way through the check valve to the 3-way valve must have been hot.

Q. But it would have been the gas directly off the preheater, wouldn't it?

DJUVIK—No. The preheater was cold. But the generator was hot. In other words, the cold gas had gone into the generator, heated up, and gone back out the oxygen line.

Corrosion. Materials of Construction

STOCKBRIDGE—Southern Nitrogen Co. We had an occurrence which could have been serious but which luckily was not, insofar as injury goes, but it shut our plant down for a few days. We have a steam gas reformer. The product gas from this reformer is collected in a manifold system and goes from the primary to the secondary reformer in a ten-inch pipe. This pipe and manifold was constructed of 310 stainless. Our plant has been operating a little over five years and we had had some leaks occur in this line, none very serious, so that we could shut down for a few hours, make repairs and get back on stream.

Recently we had a line blow out, about five feet of a longitudinal weld blew. This line operates at about 160 lbs and you can imagine the explosion and fire which we had when this hydrogen hit the atmosphere. Luckily it happened in an area where there was no one close by, and no one was injured. We were able to fabricate some pipe and get back on stream in a few days. Since that time we have replaced this entire system with a system fabricated from Incoloy which we were told is much better than 310 for this service. And we certainly hope it is because we don't want to see any other occurrences of this type.

Q. When you took the rest of the header out and inspected it, did you find all along it was the failure of the welds or just what did cause it to go?

STOCKBRIDGE—The failure was associated in every case with the weld.

Q. Did the weld just sort of deteriorate or was it cracked, fractured from next to the weld, or how was it?

STOCKBRIDGE—It was analyzed and reported to be associated with fissuring of the weld due to the analysis of the alloy in the weldment, and the plate associated with the weld. We're told by International Nickel on an analysis of this weldment, that due to a wide range of variation allowable in the specifications of 310 stainless, that you can have an alloy in this weldment which is subject to fissuring and this, as I understand it, is an age process and will become worse as time goes on.

Tank Padding

JENKINS—USI. I would like to go back to this padding a second. All of our tanks throughout our ethanol tank farm are padded with nitrogen, with a back up of fuel gas. Actually, for those people who don't have an air plant, fuel gas is the logical thing to pad with. Fuel gas, of course, doesn't contain any oxygen and you're only interested in eliminating oxygen. So that we use fuel gas when necessary. We have a possibility of supplementing the nitrogen system with fuel gas. Fuel gas isn't nearly as expensive as nitrogen for those people who don't have an air plant.

COOPER—Monsanto Chemical. All of our methanol storage tanks in Texas City are blanketed with nitrogen.