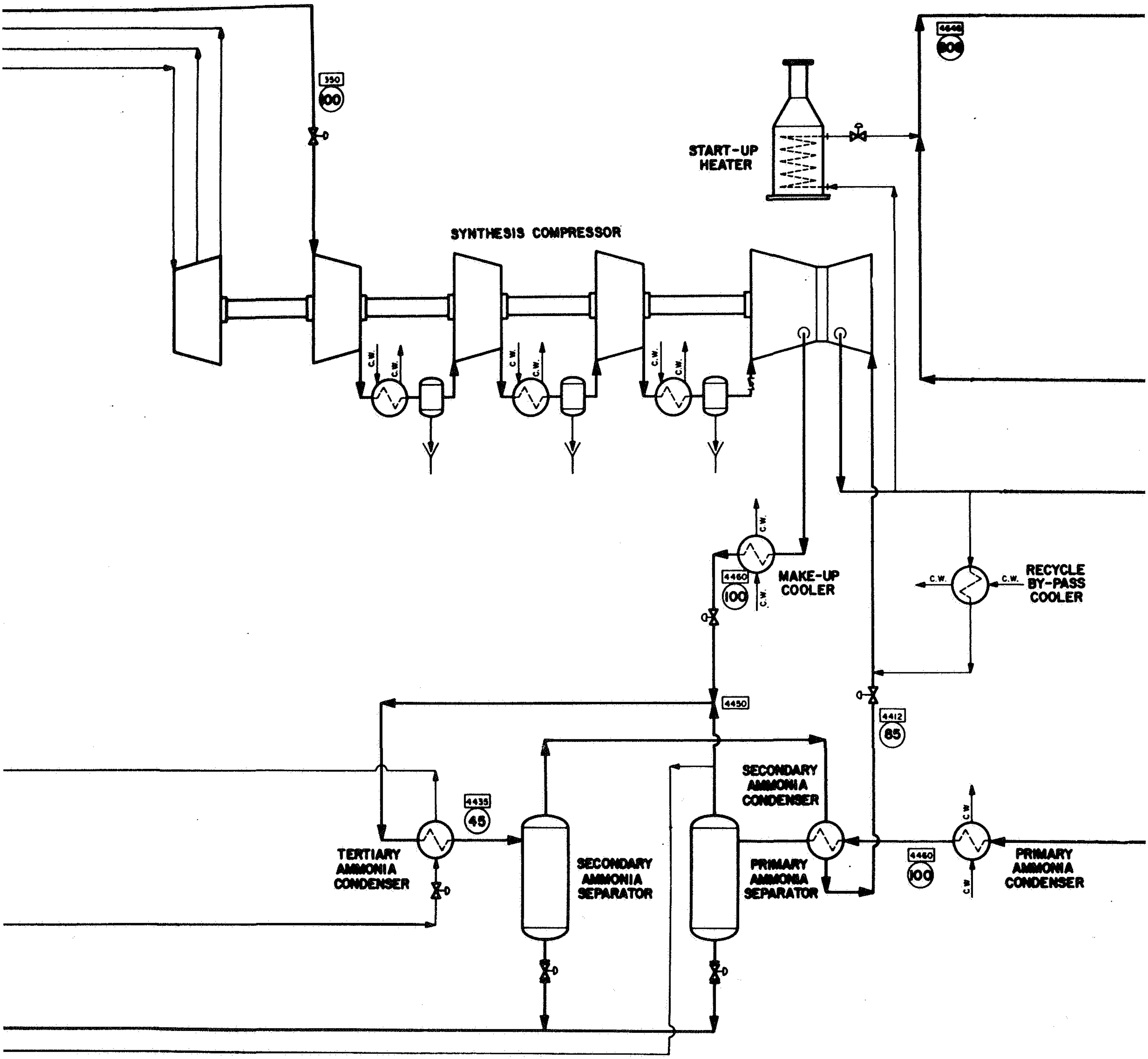

The High Pressure Centrifugal

One of the design requirements of the loop was that it be kept as simple as possible, but in accomplishing this the synthesis gas compressor became quite complicated.

J. D. Stafford, Jr., Farmers Chemical Assoc., Inc., Harrison, Tenn.

IN 1967 FARMERS CHEMICAL MET WITH REPRESENTATIVES of Norsk Hydro of Oslo, Norway to discuss the testing and application of a high pressure centrifugal compressor. At that time Norsk Hydro was building a high pressure ammonia synthesis loop utilizing a four barrelled centrifugal compressor.

Since F.C.A.I.'s ammonia production normally goes to conversion plants with only the excess production going to storage, the high pressure loop (4,700 lb./sq. in.) does have some energy balance advantages. In

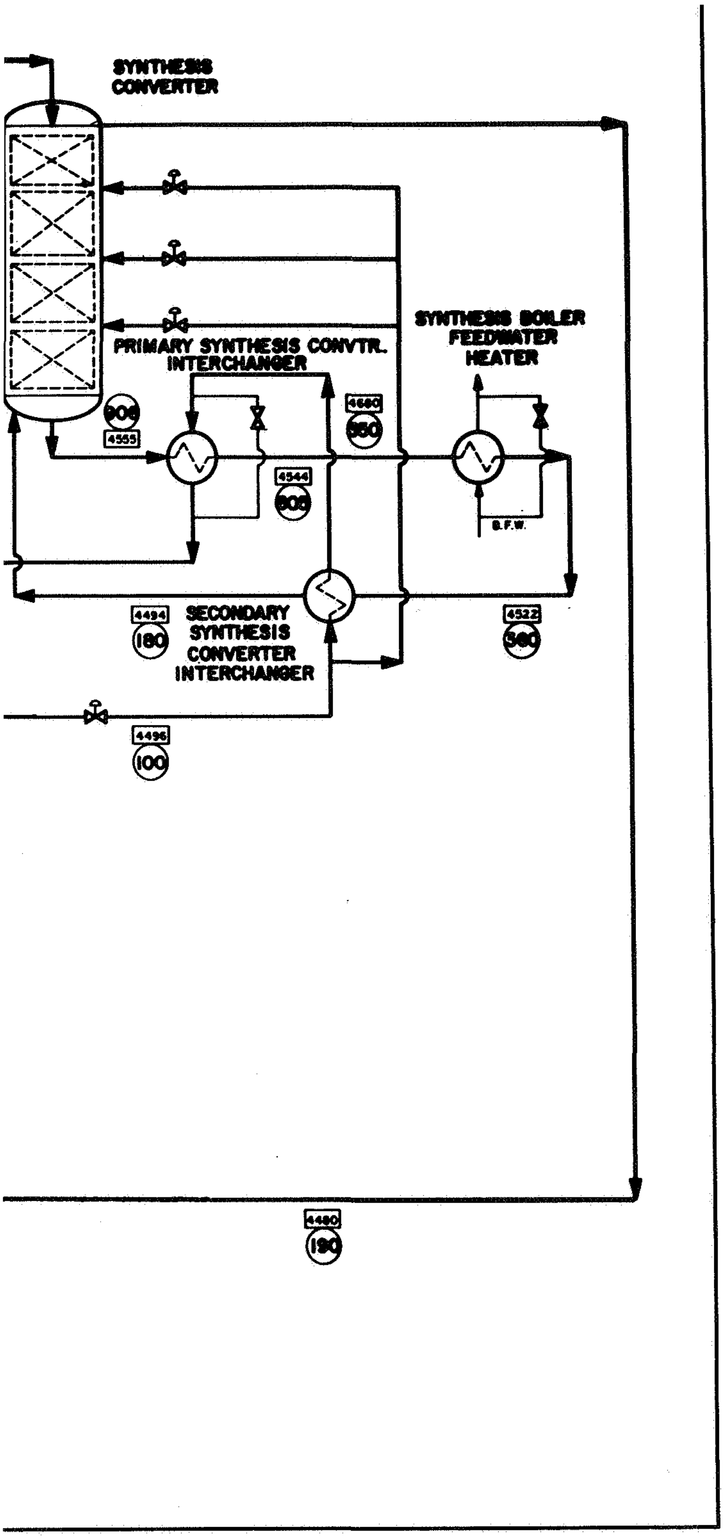

Figure 1. Schematic flow diagram of synthesis loop and compressor.

Compressor Loop

addition, the high pressure loop provides operating flexibility and good economics at reduced throughput.

The purpose of this article is to describe some of the more critical equipment and materials of construction contained in the loop. In addition, Farmers Chemical's thoughts on piping, connections, and operations are included. One of the design criteria was that the loop be kept as simple as possible. As it turned out this was accomplished, but the design of the synthesis gas compressor became quite complicated as a result.

Besides a schematic flowsheet, with design temperatures and pressures, Figure 1, there are detailed sketches of the equipment discussed. The scope of this article, however, does not allow comments on the relative merits of a low, medium, or high pressure loop.

The operations of the loop have been very stable, and satisfactory control is possible at all times. The loop is easily started up, although more than usual care must be exercised in this step. The bottom forging containing the exit nozzle and the hot converter feed exchanger must not be allowed to heat up too fast because of differential expansion between the laminated shell and the head forgings. The switch-over from the start-up heater must be done with care so that the large temperature cycles are not experienced within the converter or the hot feed exchanger. The refrigeration compressor is usually put in service at 1,400 lb./sq. in. loop pressure. The refrigeration compressor suction pressure is reduced slowly from approximately 60- to 35 lb./sq. in. to insure against too rapid a temperature rise in the converter.

Start-up heater

The synthesis converter start-up heater used in the synthesis loop has a design criterion of 2,600 lb./sq. in. at 1,025°F. The duty of this heater is 12 million B.t.u./hr. One continuous pipe coil of 2½ chrome, ½ moly was used in this fabrication. The coil is 4.5 in. dia., 0.48 in. wall thickness, with 1/16 in. corrosion allowance. With the corrosion allowance, a 5.7 yr. life would be experienced at the design conditions. The design criterion with the 4.5 in. dia., together with the material selection, was for a 50,000 hr. creep stress rupture failure.

This heater was designed and supplied by Petro-Chem Development Corp. The start-up heater, as incorporated in the loop, operates under loop pressure at all times; in other words, it can not be isolated from the system. Instrumentation associated with the heater is as follows: pressure indication of the fuel gas between control valve and burners; process gas temperature exit furnace; and fuel gas temperature from the furnace. These temperatures are registered on a multipoint recorder. In addition, each of these temperature recorders is connected to an alarm should high temperatures be experienced. Initially, each of these alarms was also interconnected to the fuel gas control valve which, in turn, would be shut off should the high temperature setting be reached during the use of the start-up heater. This has since been revised to give alarm only. Process gas flow to the start-up heater is indicated on the control panel, and a locally mounted flow indicator is prominently displayed in the vicinity of the start-up heater. The start-up heater is placed in service with the flow control valve located downstream of the start-up heater in the open position. In fact, the normal procedure is to have an operator check the stem position on the valve to be assured that this control valve is performing satisfactorily. The loop isolation valve is closed, and the 3 in. by-pass valve is only partially opened until the flow is indicated in the control room. Temperature rise in the system is supervised to insure against too rapid a start up which could set up stresses as a result of differential expansion.

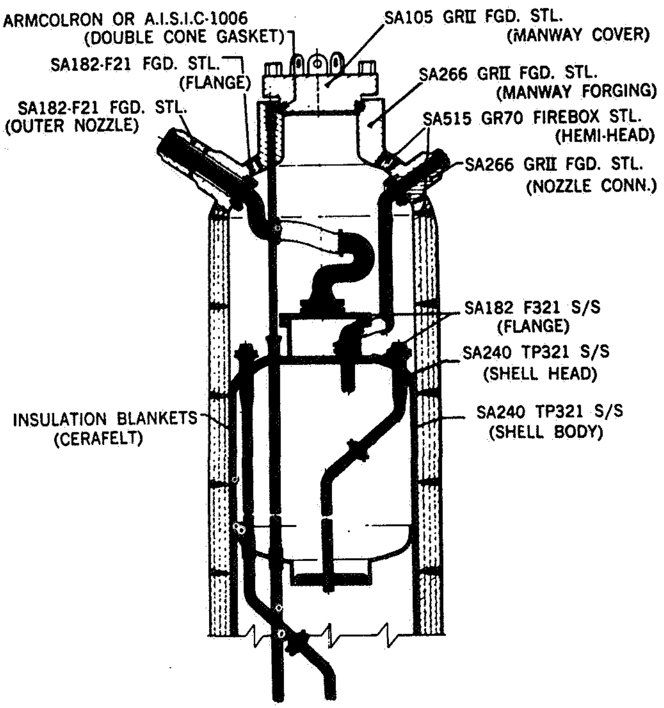

Figure 2. The top of a 600 ton/day ammonia converter.

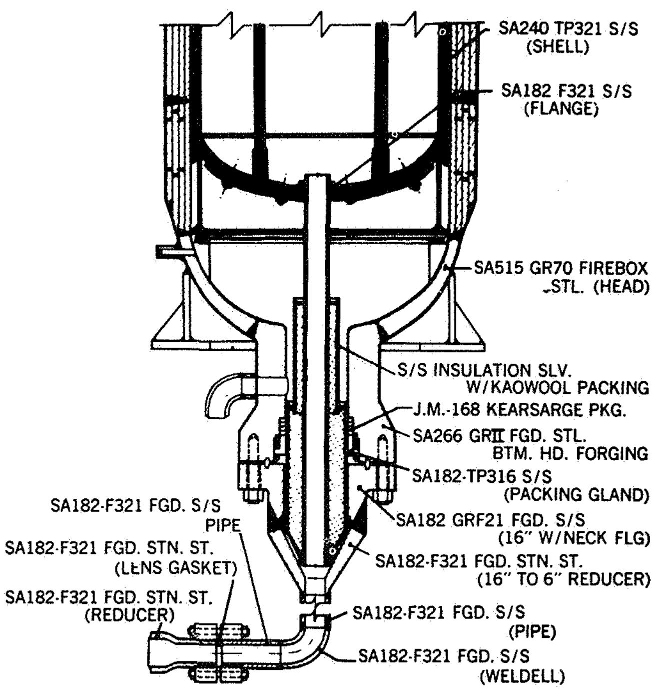

Figure 3. The bottom of a 600 ton/day ammonia converter.

Synthesis converter

The synthesis converter, Figures 2, and 3, used in the high pressure loop was fabricated by Struthers Wells. This vessel's design pressure and temperatures are 4,700 lb./sq. in. and 430°F. It has a 1/16 in. corrosion allowance, 100% x-ray for all longitudinal joints, girth seams, head forging, and all connections. This vessel was hydrostatically tested at 7,500 lb./sq. in. The inside diameter is 63 in. and the length is 52 ft. tangent to tangent.The man-way in the top head is 24 in. dia. Three thermowells are located in this vessel, each containing eight thermocouples which are strategically placed to monitor the inlet and exit temperatures of each bed. The wall thickness of the converter is 6-13/16 in. which is made up of five plates 1-11/16 in. thick. The exit nozzle is 16 in. I.D. A 6 in. I.D. insulated line covered with a stainless steel shroud extends through this exit nozzle. Cool gas enters the annular space in the insulated area to cool the exit nozzle forging, head, and insulated exit line.

The internal vessel containing the catalyst is fabricated, both heads and walls, of 321 stainless steel. The internal vessel is designed as follows: 208 lb./sq. in. @ 1,000°F at bottom, and 208 lb./sq. in. @ 850°F at top. The first bed seam to seam dimension is 9 ft. 1 in., the second bed is 10 ft. 3 in., the third 11 ft. 2 in., and the fourth bed is 15 ft. 10 in. All support members and flanges are 321 stainless steel. Internal flanges are made up using B-6 studs and 8-T nuts. The operation of the loop has, unfortunately, been erratic, although none of this behavior has been a result of the performance of the synthesis converter. The top head contains the three quench lines, feed to converter, and an annular space exit. The gas leaves the recycle compression wheel, goes through two synthesis converter feed heat exchangers, through the converter back through the high temperature heat exchanger, through the boiler feed water heat exchanger to the converter No. 1 heat exchanger, the No. 2 feed synthesis converter feed heat exchanger, through the annular space in the converter, then through the primary ammonia condenser followed by exchange with process gas from the secondary separator, and then through the primary ammonia separator. From there it is mixed with fresh make-up gas and goes through the refrigerated cooled condenser, into the ammonia separator, and back through the primary condenser effluent cooler exchanger. From this heat exchanger, it enters the suction of the recycle wheel of the compressor.

Other critical equipment

The synthesis converter feed interchanger, referred to as the primary synthesis converter interchanger, Figures 4, and 5, is the most critical heat exchanger in the complex. It is designed for an inlet temperature of the converter effluent of 975°F. The design temperature of the interchanged converter feed gas is 850°F. The hot effluent gases are on the tube side and the converter feed is on the shell side. The design pressure of this exchanger is 4,700 lb./sq. in. with a differential pressure of 400 lb./sq. in. The starting up and shutting down of the syn loop must revolve around this exchanger and the synthesis converter. The shell and heads are 3% Cr.–1 moly–SA 182-F21. The tube sheets are 5% Cr.–½ moly 182-F5, the tubes are 5% Cr.–½ moly SA 213-T5C and miscellaneous internals are 5% Cr.–½ moly SA 357.

Protective shields of Inconel have been used in the inlet nozzle of the bottom access nozzle diaphragm, and all internal gaskets. Inconel is used in the expansion joint in the top of the internal connecting system.

This is a floating head type heat exchanger so that expansion can be handled. The spring loading system plus the weight of the internals provides the necessary pressure to seal the base of the exchanger. Assistance is given by the studs in the support located at the bottom of the exchanger.

The loop boiler feed water heat exchanger is a U tube, two-pass shell side exchanger. The tube side of this exchanger was designed for 4,700 lb./sq. in. at 650°F, and the shell side was designed for 1,800 lb./sq. in. at 500°F. This exchanger is designed for full pressure and not differential pressure.

The hot gas inlet nozzle of this exchanger is protected by a lining of SA 387-B–1% chrome–½ moly. The tube sheet on the inlet hot gas side is protected by a baffle plate of SA 204-B. This protective material is carbon–½ moly. The first pass of the tube side hot gas is completely jacketed with 1% Cr.–½ moly material. All internal baffling is also made of this material. The tubes in this exchanger are SA 209-TI, a carbon ½ moly.

The channel tube sheet forging of the exchanger is SA 182-F1 carbon–½ moly, and the shell is SA 515-70. The hot inlet nozzle is SA 182-F22, 2¼ Cr.–½ moly and the exit nozzle is SA 182-F1. The diaphragm closure is SA 204-A, ½ moly and the overlay that this diaphragm is welded to is low Cr., Ni moly.

The failure of seven tubes in this exchanger has been attributed to improperly rolled tubes and improper warm up of the 12 in. thick tube sheet. This exchanger was by-passed, and the loop operated at low pressure and temperature to protect piping, heat exchanger, and the shell of the synthesis converter.

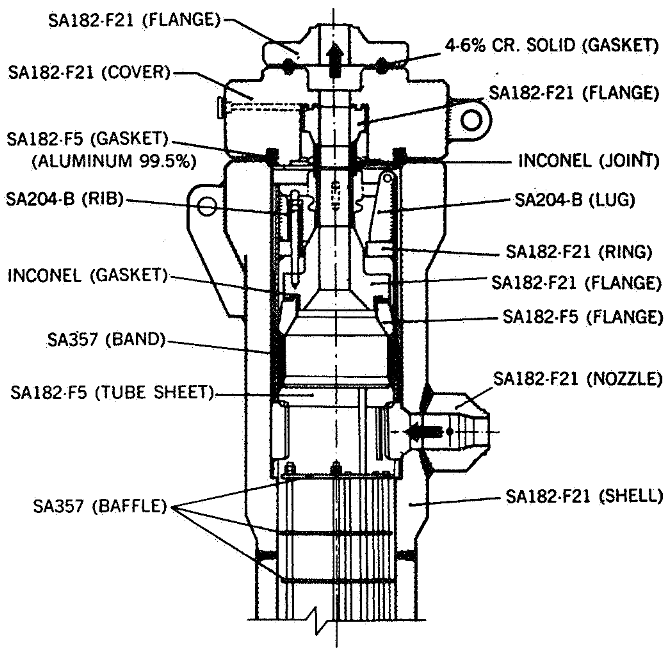

Figure 4. The top of a primary synthesis converter interchanger.

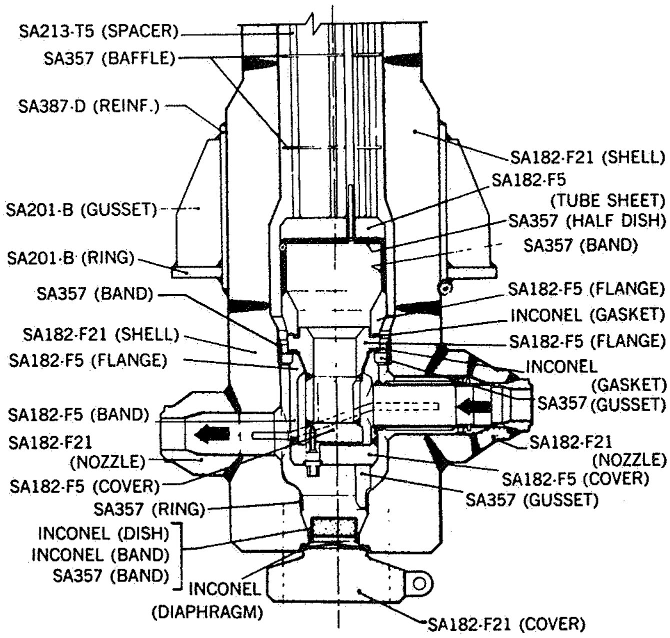

Figure 5. The bottom of a primary synthesis converter interchanger.

Another of the critical loop exchangers is the secondary synthesis converter interchanger. This exchanger, designed for a pressure of 4,750 lb./sq. in. at 425°F, is a two-pass exchanger, both shell and tube. The differential design pressure is 400 lb./sq. in. The hot gas inlet, located on the shell side, has a semi-circular shroud of 304 L which completely encloses the inlet pass of the two-pass exchanger. The partition baffle is also fabricated from 304 L stainless steel.

The tubes are A 210-A1, and the tube sheets are A 105 II. The fixed tube sheets are attached to the shell by 304 L expansion joints. At the point of welding of the expansion joint to the shell, a band of Inconel was overlayed to provide the desired characteristics. The channel and channel cover are 304 L. The shell of this exchanger is A 105 II as are the heads. The studs on this exchanger are made up by using a hydraulic device to insure that the stressing of the studs is done in a uniform tensile manner rather than the twisting effect type stress caused by making up with hammer or impact wrenches. The closure on this vessel is the welded diaphragm type fabricated from 304 L.

Loop design piping specifications

All piping in the ammonia synthesis loop is designed for maximum pressure of 5,000 lb./sq. in. Materials were selected based on operating temperature. pressure, and the resistance to hydrogen attack. Carbon steel SA-106, Grade B was used for all service up to 400°F. For operating service between 400- and 750°F, 2¼Cr.–1 Mo., SA 335, Grade P-22 was used; from 750- to 800°F, 5 Cr.–½ Mo., SA 369, Grade FP5 was used; and for temperatures above 800°F, and limited to 950°F and 5,000 lb./sq. in., 321 stainless steel, SA 213 TP 321 was used.

All welding valves and fittings within the temperature boundaries of each of these specified materials conformed to the ASTM specification related to the piping. All flanges in the ammonia synthesis loop are weldneck type. Lens type gaskets are used exclusively in the loop piping.

Very few flanges were used in the synthesis loop piping. Not counting vessel closures, only three flanges were incorporated in the ammonia synthesis piping. All instruments except level controller gauges contained welded connections. Loop flow meters are welded venturi tubes. F.C.A.I. was insistent, as far as practical, upon a loop free of flanges. All plug and thermowells are back welded.

All vessel closures, with the exception of the ammonia synthesis converter and the primary ammonia separator, have welded diaphragm closures where possible. Snuffing steam rings were incorporated in the original design at the two hot flanges, one in the converter exit head and one in the line between the converter and the converter feed interchanger.

The synthesis gas compressor will "fail safe" insofar as the loop isolation, balancing, vent, and recycle bypass valves are concerned. These valves are pneumatically operated with booster relays which have been set to give the desired response to a trip condition. In addition, limit switches have been installed on all valves to insure that they are positioned properly prior to starting the machine. All control valves are welded into the loop. A check valve is installed between the third and fourth stage cases to prevent reverse rotation of the compressor, should gas leak through one of the isolation valves.

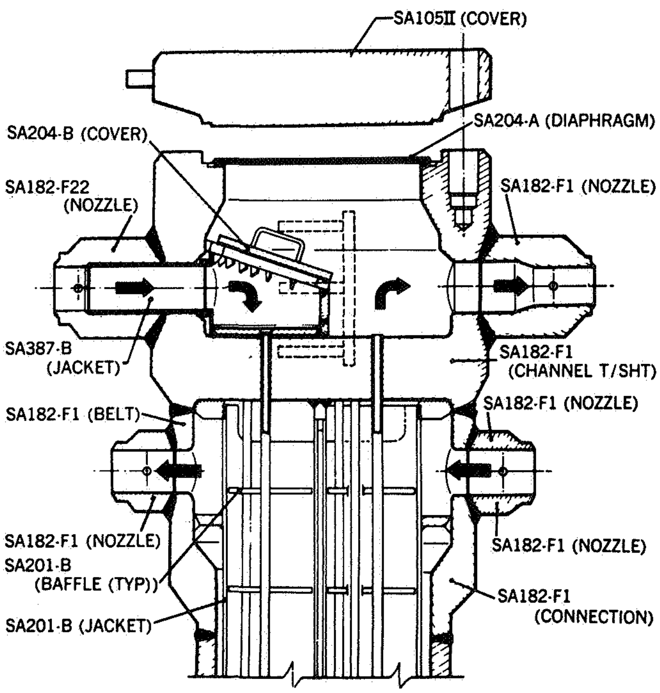

Figure 6. Synthesis boiler feed water heater.

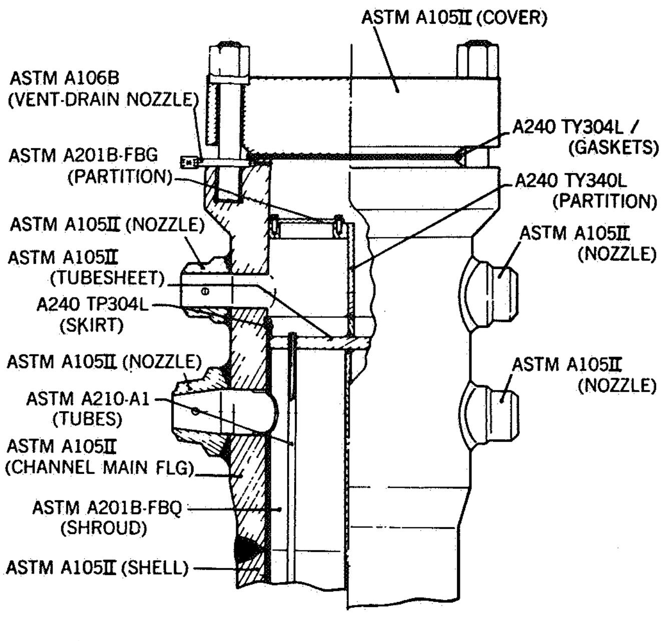

Figure 7. Secondary synthesis converter interchanger.

In summary

I am convinced that the much desired built-in safety of a plant is a result of:

1. Fabricator selection based on previous experience and one which places emphasis on quality control.
2. Engineered simplicity.
3. Complete review by engineers with operating background.

The following equipment failures have been experienced and/or we are living with them at the present time.

1. Synthesis loop boiler feed exchanger—leak at tube-tube sheet weld, Figure 6.
2. Secondary synthesis converter interchanger—internal leak, Figure 7.
3. Third stage blading failure of the synthesis gas compressor turbine low pressure stage—blade vibration together with pitting at the root are the suspected causes. #

J. D. Stafford, Jr. is manager of research and development of Farmers Chemical Assn. He is a graduate of the University of Michigan. Stafford has been associated with Farmers Chemical eight years, and has over twenty years of experience in the fertilizer industry.

DISCUSSION

Q. You mentioned that during startup you were careful about bringing on your refrigeration compressor and you brought it on when the loop was about 1,400 pounds. I was wondering why you didn't start the refrigeration compressor before you pressured the loop?

STAFFORD: There are several reasons. First is, we have a lot more refrigeration capacity built into this machine than we need. We do have it warming up with complete recycle and very low speed (5,000 rev./min.), but not pulling vapors off of the loop. This insures a better control over the converter during the switchover operation.

Q. What I'm wondering if you perhaps cannot eliminate any kind of switchover operation. If the refrigeration machine is in service beforehand, there's just no switchover.

STAFFORD: This is true. But at the same time, we do not have a recycle cooler on the refrigeration system. Our system is equipped with a quench ammonia stream into the suction and we find the sooner we can put a load on it, that we're much better off.

Q. I understand that you have had difficulties with the boiler feedwater heater in this syn loop. Could you explain how you repaired the leaking tubes?

STAFFORD: We repaired this exchanger on two occasions. The first time we tried to weld it with the same material that was used in the original weld. Since this was a field operation, the cleanliness that we strived to maintain was not possible and leaks were again experienced in approximately six weeks. The same tubes leaked plus some additional ones. We welded with Incoloy 800 on the second try and so far it's been satisfactory. Prior to welding we cut the tubes loose and pulled them out approximately 3/4-in. and cleaned them quite well before trying to re-weld.

PETER AMBROSE, Imperial Oil: With respect to your low number of flanges in the synthesis loop, how does this affect your ability to get into the high pressure vessels? We have a Department of Labor regulation in Ontario, at least, that requires that the vessel be blanked at the first flange, which is generally taken to be right at the vessel, before you can achieve vessel entry. Do you have a regulation like this, and if so, how do you manage to get around it?

STAFFORD: We're able to blank the loop at the converter, and at the top, or the inlet to the primary condenser. Or we can also put a blank in on the exit of the hot heat exchanger. Those are the 3 flanges contained in the loop.

AMBROSE: This allows you enough flexibility for purging and gas freeing?

STAFFORD: Yes. So far, the only vessel that we've entered has been the boilerfeed water heat exchanger, and we've been able to clear it. Also, we do have some - some sample points, and thermo couples or pressure gauge connections that we're able to purge various pieces of equipment within the loop itself from these small connections.

Q. I'd like to ask you a question that has to do with the all-welded pipe in the loop as well. I'm interested in what procedure you use to test your welds, after you've made them all. Because you don't have flanges, this sort of makes it impossible to do a hydrostatic test on all your welds.

STAFFORD: On the original startup of the plant, we pressure tested it at 5,000 pounds with nitrogen. Any welding that we do in - in the loop is stress relieved, and 100% X-rayed, and put in service. We do not hydrostat after a repair job or a welding job within the loop piping, unless it falls between these flanges.

Q. But what sort of logic do you use to fix this in your mind, that it's not necessary to hydrostatic test?

STAFFORD: Our experience in both of our plants has been that we're able to maintain the quality control on our welding within the field and with X-ray and stress relieving we are not worried about it.

W.D. CLARK, ICI, Billingham: As far as I can make out from the flowsheet, you're taking the gas away from the ammonia converter catalyst at about 400 Centigrade and it contains about 20% ammonia. I think the piping was either 2¼ or 5-chrome moly. Is that right?

STAFFORD: No, it's 321 stainless.

CLARK: 321 stainess. I see. Because one of the troubles you've got there is that that piping will nitride - even the 321 stainless is likely to nitride, and you get a brittle layer on the inside, and you might get into trouble. We also would be a little worried about that sort of piping. We'd be very careful to keep the rain off it. Otherwise, you're liable to get stress-corrosion cracking from the outside. We have got two systems where we take the hot gas away from the converter, and we were very considerably exercised over the second one, as to what the piping should be.

The first one was thrust upon us, because it was a conversion of a plant originally made to hydrogenate coal to petrol, and it was 3% chrome, half molybdenum piping. That nitrides under the conditions in question, and you get 30, 40 thous of embrittled and cracked layer on the bore and we take a slice out of the pipe every 18 months, and retire the piping at a total depth of nitriding of about 30 thou.

On the second plant, we went to the same material - 3% chrome molybdenum - and well fortunately there, we've never needed to retire anything, because the converter has succeeded in running at about 370° Centigrade, where nitriding is very slow, in fact. But if you were running 400° and 321 piping, I would think it very desirable to have a look after a year or two, to see how much nitriding there was.

STAFFORD: Our normal operations of the converter exit is about 900 degrees, although it's designed for 975.

PETER HUSBAND, ICI Billingham, England: In connection with the synthesis loop boiler feedwater heat exchanger - we also have trouble with tube to tube-plate leaks. In your paper I think you said that you did by-pass this exchanger - at one stage. Could you please provide a little more details. Do you still by-pass this exchanger - or do you by-pass it on every start up?

STAFFORD: That was only during the period of start up. It was 4 to 6 weeks. We operated the loop at 1900 pounds - the maximum temperature on this line was between 550 and 600 degrees.

HUSBAND: I see, the exchanger was physically removed from the loop.

STAFFORD: Right.

HUSBAND: It isn't, therefore, an operation that you can still do at every start up.

STAFFORD: We do not bypass it during the startup. We have since learned to start this exchanger up very slowly with respect to temperature increase, so that excessive stresses are not created in the 12-in. thick tube sheet. I tink that's one of the reasons we had the failure to begin with.

HUSBAND: The second point: you were talking about the failures that you had. I think you said that they failed, you went back on line, and then the same ones failed again in six weeks' time, and by paying attention to cleanliness of the tubes prior to welding - you had made a satisfactory repair.

Am I right in thinking, though, that this is a U-tube exchanger?

STAFFORD: This is a U-tube exchanger.

HUSBAND: I find it a little difficult to understand how you managed to pull the tubes out of the tube sheet for cleaning prior to welding.

STAFFORD: We have a wrench - or a tool - that we're able to stick inside the tube and pull.

Q. What size of tube do you have in the boiler feedwater exchanger?

STAFFORD: They are 5/8ths, and are rolled and welded.

Q. I'd like to ask Willie Clark a question concerning nitriding, which he says progresses to a depth of 30-40 thousandths. Seems to me most of the information I've obtained indicates that this is the extent of the nitriding depth in this type of service. I wonder on what basis you changed your line and whether if we had this depth in a spindle pipe, whether it would be necessary to change the syn loop at this depth?

CLARK: It's a fair question. What we know is that over 2, 3 years, it builds up to 30 or 40 thou, and we believe that it's going to keep on going, and so we play safe and change the pipe. We have been doing some work recently which shows that the rate of nitriding - the increase in depth is dropping off.

But we don't like the look of the cracked and very brittle layer that you get in the pipe. It's nitrided, and the nitrided layer is fissured and flaking off, and so we play safe, and we change the pipe. We could be bolder, and let it run, and see whether it gets as far as 50 thou, and then never gets any further. But we've not risked that so far.

Q. I wonder if in your field repair of your synthesis loop heat exchanger you suspected the welding procedure is what caused your future failures?

STAFFORD: In the boiler feedwater exchanger?

Q. Right.

STAFFORD: Yes, I think so. I might add that while we were shutdown we rolled all the tubes in the exchanger before starting back up, whether they were leaking or not.