

**Figure 1.**  
The Agrico plant  
at Blytheville, Ark.

## The Startup of Two Major Urea Plants

What was learned during startup of these two plants is expected to speed the commissioning of six similar units.

D. F. Bress, Foster Wheeler Energy Corp., Livingston, N.J.  
M. W. Packbier, Hoechst-Uhde Corp., Englewood Cliffs, N.J.

During AIChE's 74th National Meeting in New Orleans, March 12-15, 1973, D. F. Bress and R. K. Fidler presented a paper titled, "New Concepts in Design of Urea Plants." The startup of these two facilities is the subject of this article.

Both installations have a capacity of 1,000 short ton/day and employ the Stamicarbon CO<sub>2</sub> stripping process. Engineering and construction work was carried out by Foster Wheeler Energy Corp. in collaboration with the United States branch of Friedrich Uhde, Hoechst-Uhde. The first plant, built for CF Industries, Inc., at Donaldsonville, La., was started up in November, 1974, and the second, erected in Blytheville, Ark., Figure 1, for Agrico Chemical Co., began operation in November, 1975.

Figures 2 and 3 are simplified flowsheet for the CFI facility. The compressor, a steam turbine-driven centrifugal machine from Dresser Clark, has two casings and four sections. The normal discharge pressure is about 2,000 lb./sq. in. gauge; the power requirement is about 6,500 h.p. The turbine is made by Turbodyne Corp.

The reciprocating ammonia pump and spare installed at CFI are from Peroni of Italy and have three horizontal plungers. The single ammonia pump installed at Agrico was made by Uraca of West Germany and also has three

horizontal plungers. These pumps are electrically-driven in both plants.

The synthesis section of the plant operates at a pressure of about 2,000 lb./sq. in. gauge and consists of a stainless steel-lined reactor, a carbamate condenser, a stripper, and a scrubber. The condenser, stripper and scrubber have stainless steel tubes. In the reactor, about 58% of the carbamate is converted into urea. The reactor mixture descends through an overflow line to the top of the stripper where it is equally distributed over the tubes. CO<sub>2</sub> gas introduced in the bottom causes the partial NH<sub>3</sub> pressure to decrease, resulting in a decomposition of the non-converted carbamate. The required heat for this decomposition is provided by about 325 lb./sq. in. gauge steam admitted around the tubes of the stripper. The urea solution leaving the bottom of the stripper has the following typical composition (in wt. %): urea 58, water 26, NH<sub>3</sub> 7, and CO<sub>2</sub> 9.

The gases leaving the top of the stripper are fed to the carbamate condenser together with the incoming ammonia. In this condenser the NH<sub>3</sub> and CO<sub>2</sub> is almost completely condensed and fed to the bottom of the reactor. The heat of condensation in the condenser is used to raise steam with a pressure of about 50 lb./sq. in. gauge.

## Oxygen aids in preventing corrosion

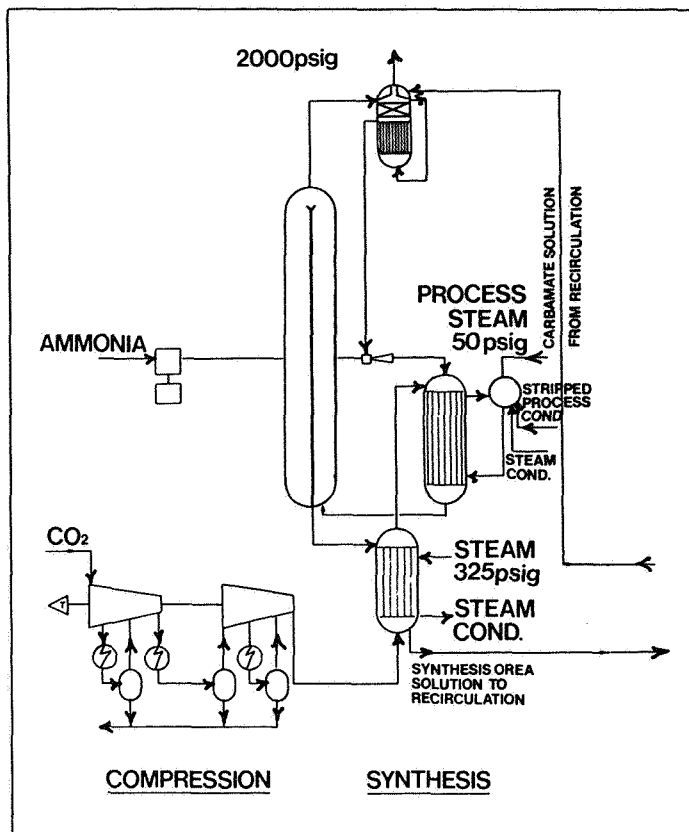
For corrosion prevention some oxygen, in the form of air, is supplied to the suction side of the CO<sub>2</sub> compressor and fed together with the CO<sub>2</sub> to the stripper. This air together with the inerts which are originally present in the CO<sub>2</sub> and NH<sub>3</sub> must be blown off continuously to maintain the desired inert pressure in the synthesis section. The purge passes from the reactor to a scrubber where practically all of the NH<sub>3</sub> and CO<sub>2</sub> are condensed. The non-condensables consist mainly of N<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>.

Depending upon the amount of H<sub>2</sub> originally present in the CO<sub>2</sub> or NH<sub>3</sub>, the scrubber off-gases can be within the flammable range. To make sure that the plant can be operated safely under all conditions, the scrubber is of an explosion-resistant design. If, for some reason, the off-gases should ignite, no damage is done to the scrubber itself and no leakage of carbamate liquid or gases can occur. Only the internals have to be replaced.

The CFI plant uses a monoethanolamine (MEA) solution with a low pressure flash step in the CO<sub>2</sub> removal system of the ammonia plant. Thus the H<sub>2</sub> content in the incoming CO<sub>2</sub> in the CFI plant is normally so low that the scrubber off-gases always are out of the flammable range.

The H<sub>2</sub> content in the CO<sub>2</sub> of the Agrico plant, which employs the Catacarb (potassium carbonate solution) carbon dioxide removal system without a low-pressure flash step, is higher. Therefore, if the NH<sub>3</sub> and CO<sub>2</sub> were condensed completely, the off-gases would be within the flammable limits.

To prevent a time consuming shutdown and draining period of the synthesis section in the unlikely event of an explosion, the NH<sub>3</sub> and CO<sub>2</sub> are not completely condensed in the Agrico scrubber. The NH<sub>3</sub> and CO<sub>2</sub> still left in the off-gases have a diluting effect, resulting in a vent gas com-



▲Figure 2. Simplified flowsheet of urea plant at CF Industries showing the compression and synthesis sections.

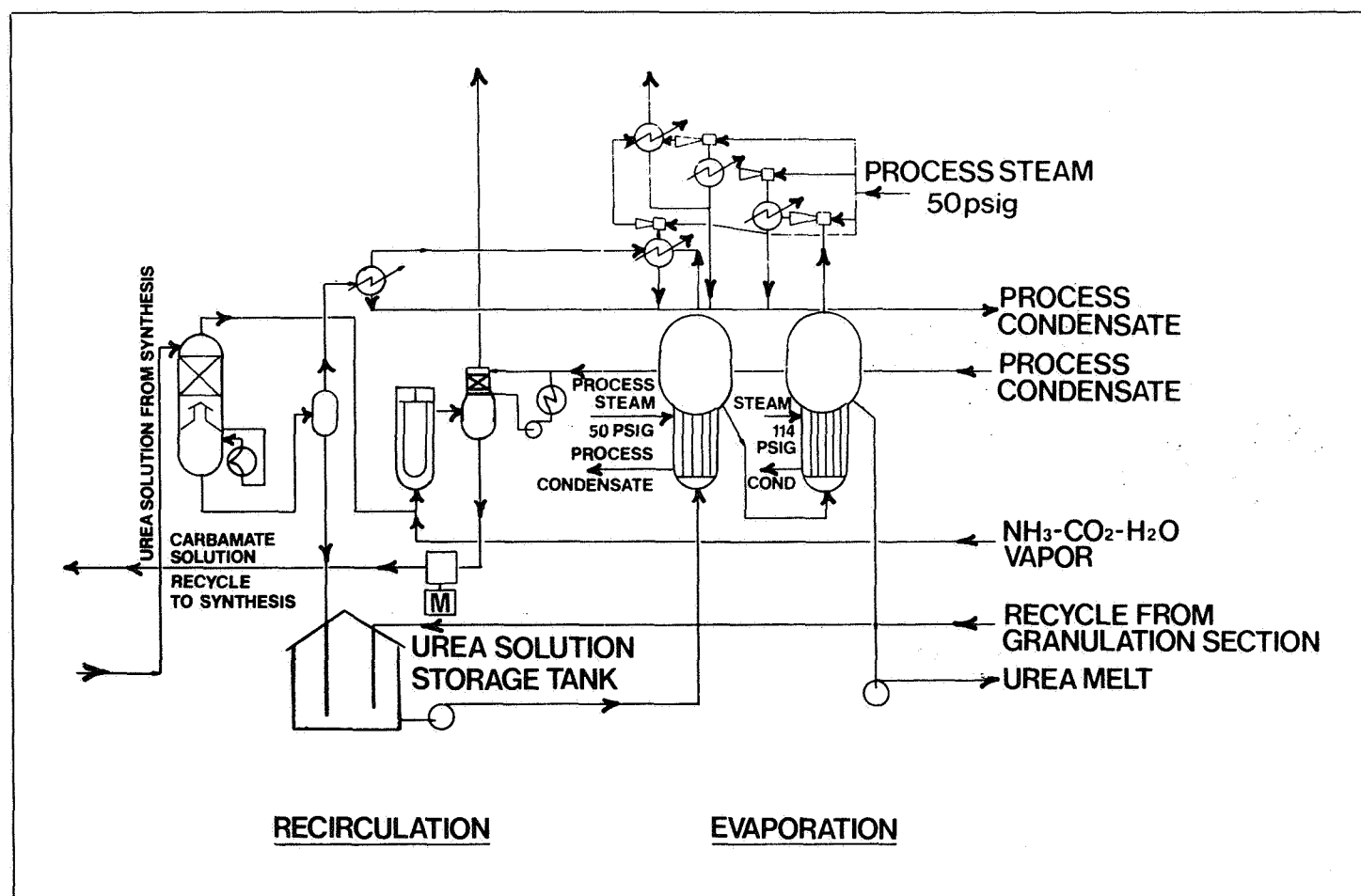


Figure 3. Recirculation and evaporation sections of CF Industries' urea plant.

position outside the flammable range. The vent gas is reduced in pressure to about 85 lb./sq. in. gauge and treated in an absorption column to free them from the  $\text{NH}_3$  and  $\text{CO}_2$ , as shown in Figure 4. At the low pressure in this absorber, the resulting vent gases are saturated with sufficient water vapor to place them outside the flammable limits.

**Recirculation.** As shown in Figure 3, the urea solution leaving the bottom of the stripper is expanded to 30 lb./sq. in. gauge and heated to 275°F. As a result, the carbamate left in the solution will decompose and evaporate from the liquid.

The rectifying column is used to reduce the amount of water passing overhead with the ammonia and carbon dioxide resulting from carbamate decomposition. The urea solution is then further expanded in a flash tank under a vacuum where the last traces of ammonia, as well as some water, will evaporate causing the solution to cool to 200°F. The resulting solution, containing about 75% urea, drains from the flash tank into the urea solution storage tank.

Gases passing overhead from the rectifying column are fed to a low pressure carbamate condenser and condensed. Inerts in these gases are passed to the atmosphere via a low pressure scrubber to minimize the amount of ammonia vented.

The carbamate solution from the low pressure condenser is recycled by a high pressure carbamate pump to the synthesis section. The carbamate pumps installed at CFI are Peroni pumps, while at Agrico Uraca pumps are used. Spare pumps are included for both plants and are electrically-driven.

Gases flashed from the urea solution pass from the flash tank to a condenser for recovery of ammonia. Inerts from this condenser mix with inerts from the evaporation condensing system before final vent to atmosphere.

**Evaporation.** The urea solution in the storage tank originates from two sources. One is the normal 75% urea solution made in the synthesis section. The second is a 45 to 50% urea solution recycled from the granulation section. The amount of urea in this stream is specified by the vendor for the granulation process and amounts to a considerable fraction of the total plant production.

The two-stage evaporation section is designed to handle both urea solution streams. Each evaporation stage includes a heater, a separator on top of the heater, and an indirect condensing, ejector system to maintain the required vacuum in the separators. The urea solution is concentrated to a 99.8% urea melt, having a temperature of 280°F, which is pumped to the granulation section.

Figure 5 is a simplified flowsheet of the process condensate treatment section of the plant. This system was developed by CFI and used at their Donaldsonville plant, as well as at the Agrico unit. Process condensate, with a composition as shown in Figure 4, is pumped via a heat exchanger to the first desorber. The main part of the free  $\text{NH}_3$  and  $\text{CO}_2$  are stripped out of the liquid and passed to the low pressure carbamate condenser. The liquid bottoms are heat exchanged with the feed and pumped via a storage tank to the steam drum of the high-pressure carbamate condenser of the synthesis loop. In the steam drum, urea present in the process condensate is totally hydrolyzed and the  $\text{NH}_3$  and  $\text{CO}_2$  produced is evaporated as components of the 50 lb./sq. in. gauge steam. This so-called "process steam" is used in the heater of the first evaporation stage and in the ejectors of the vacuum system.

Condensate from the process steam contains all the  $\text{NH}_3$  and  $\text{CO}_2$  originally present in the process steam generated. This condensate is pumped via a tank and a heat exchanger to the second desorber. In the second desorber 114 lb./sq. in. gauge stripping steam is used, while the balance of the process steam is also introduced in the middle of the column. The overhead vapors are used as stripping medium in the first desorber. Liquid effluent of the second desorber contains no urea and less than 50 parts/million of  $\text{NH}_3$ .

**Steam balance.** The largest power user in the plant is the  $\text{CO}_2$  compressor, and it is therefore a key item in the overall steam balance. Figure 6 is a simplified flowsheet of the steam balance for the urea plants under discussion. The steam balance is based on production of 134,000 lb./hr. steam at 800 lb./sq. in. gauge and 825°F from the boiler, which is oil-fired at Agrico and gas-fired at CFI. This total quantity is sent to the  $\text{CO}_2$  compressor turbine. The required amount of steam is extracted from the turbine at a pressure of 325 to 350 lb./sq. in. gauge, and this extracted steam is desuperheated and sent directly to the stripper. In  $\text{CO}_2$  stripping plants producing a prilled urea product, all of the 50 lb./sq. in. gauge steam requirements plus an excess is generated by the high-pressure carbamate condenser.

In the CFI and Agrico plants, however, the urea product is granulated, and a considerable amount of 50 lb./sq. in. gauge steam is required to concentrate the 45 to 50% urea solution recycled from the granulation section. This results in a deficiency of 50 lb./sq. in. gauge steam, which is supplied by expanding 325 lb./sq. in. gauge steam through additional turbines that drive the boiler feed water pump and some smaller users.

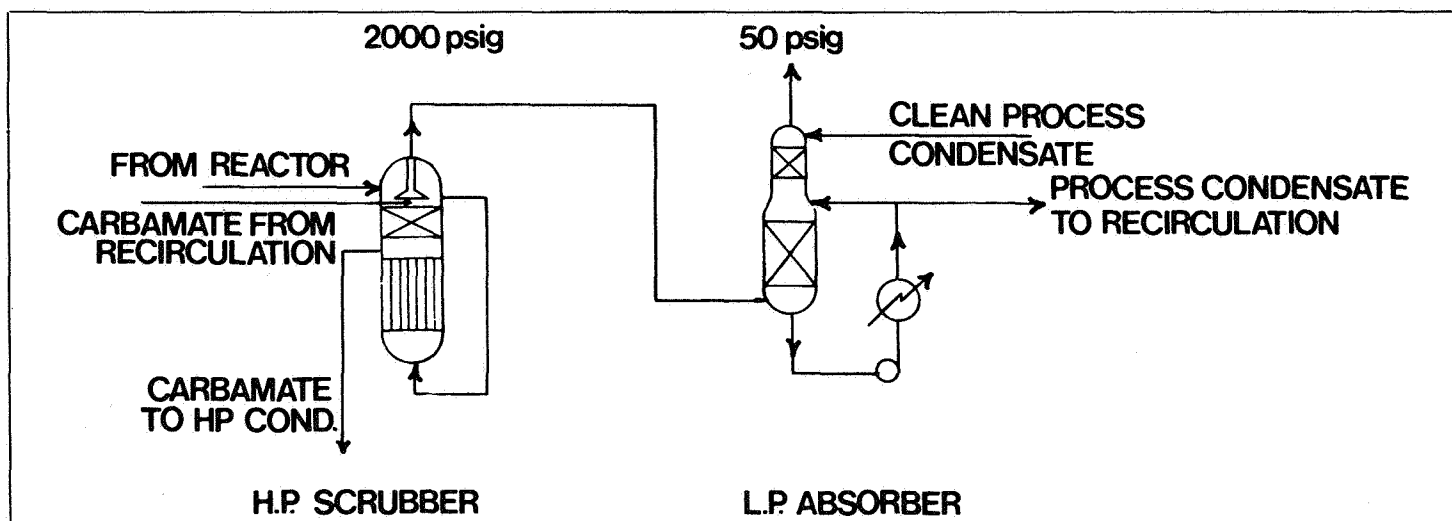


Figure 4. Scrubbing system at the Agrico plant.

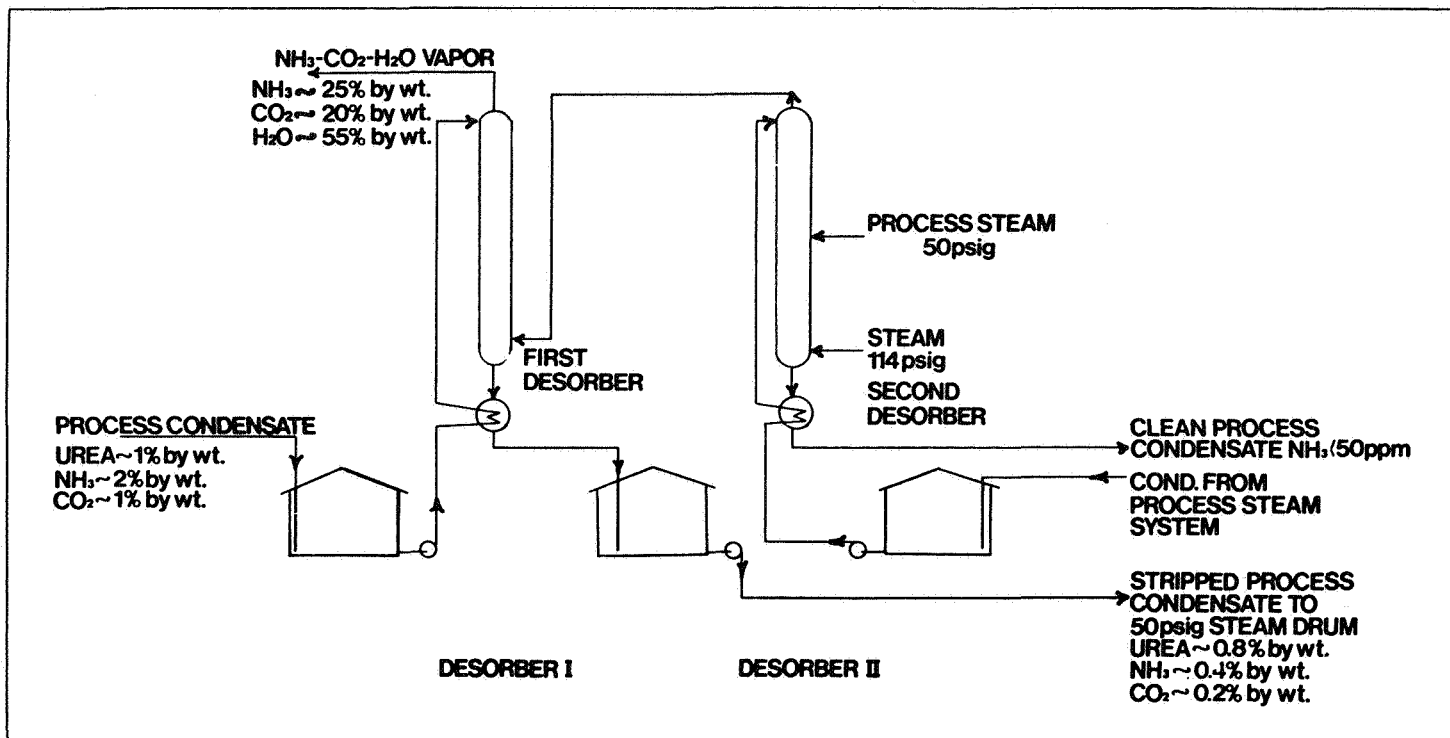


Figure 5. CF Industries' process condensate treatment system.

The 114 lb./sq. in. gauge steam requirement is supplied by flashing the 325 lb./sq. in. gauge condensate drained from the stripper and supplemented by a small amount of steam let down directly from the 325 lb./sq. in. gauge steam system.

### Startup problems

During the first startup of the CFI plant, the rotor of the compressor vibrated at a certain frequency. As a result, the bearings had to be redesigned. In addition, turbine bearings had to be strengthened to accommodate a lateral thrust in the opposite direction. These were the only mechanical problems with the compressor.

Another problem was shock cooling in the first interstage cooler, which resulted in an excessive amount of submicron condensate that was not removed in the first interstage separator. This caused corrosion of the carbon steel discharge volute following this separator and minor damage to the second interstage seal.

The immediate solution was to add a second separator in series so that the original unit acted as a coalescer for the new one. These two were eventually replaced with a single high efficiency separator. Having learned of the problem, high efficiency units were installed at Agrico and no difficulty occurred.

Two problems were experienced during startup in the evaporator section of the plant. The first was a larger urea entrainment from the separators than was expected.

In the CFI condensate treatment system, this extra urea is hydrolyzed in the steam drum and results in increased NH<sub>3</sub> and CO<sub>2</sub> concentration in the process steam. This, in turn, causes an overloading of the second desorber in which the process steam condensate is stripped prior to being sewerred.

By modifying the internals in the two separators, the entrainment was decreased to an acceptable figure. The urea content in the process condensate after the modification is now about 1% at CFI and 0.3% at Agrico. Both systems now produce waste condensate in accordance with the design.

The other problem was an excessive biuret increase over the evaporation section because the heaters were designed for a specific recycle stream recovered from the granulation section scrubbing system. In practice at CFI it was found that the recycle was only 75% of the design value with a urea concentration about 10% higher than expected. These two factors caused a higher than expected retention time in the evaporator tubes and, therefore, increased biuret formation. To decrease the retention time, a certain percentage of the tubes in both evaporators were plugged.

The second reason for the higher biuret increase was internal recirculation in the first stage evaporator. At the Agrico plant, measures were taken to prevent such recirculation, and while this appears to have had a beneficial effect, both plants now make urea with a biuret content of about 0.9%.

It must be recognized that the CFI condensate treatment system was a prototype and not all the difficulties experienced could be anticipated. For example, in spite of the improved performance of the entrainment separators in the urea evaporation section, it was not possible to reach the required low NH<sub>3</sub> level in the first desorber bottoms during normal operation.

At a load of about 80% of the design liquid feed rate, the first column started to flood. The first tower only flooded when the liquid feed rate or the NH<sub>3</sub>, CO<sub>2</sub> and urea concentration was increased, and operation was stable at vapor rates in excess of the maximum design gas flow.

This behavior led to the conclusion that flooding was caused by foaming of the liquid in the column. Originally both desorption columns were equipped with valve trays having a 12-in. tray spacing. The first desorber had 20 trays and the second had 25 trays. To improve the performance of the first column, the trays were removed and replaced by No. 2 Minirings, which are similar to modified Ballast or Pall rings, having a diameter of 1-1/2 in. and a length of 1/2 in. These rings were used in two 13-ft. packed sections.

Originally, process steam was fed to the base of the second desorption column. The NH<sub>3</sub> concentration in the effluent of this column therefore depended on the NH<sub>3</sub>

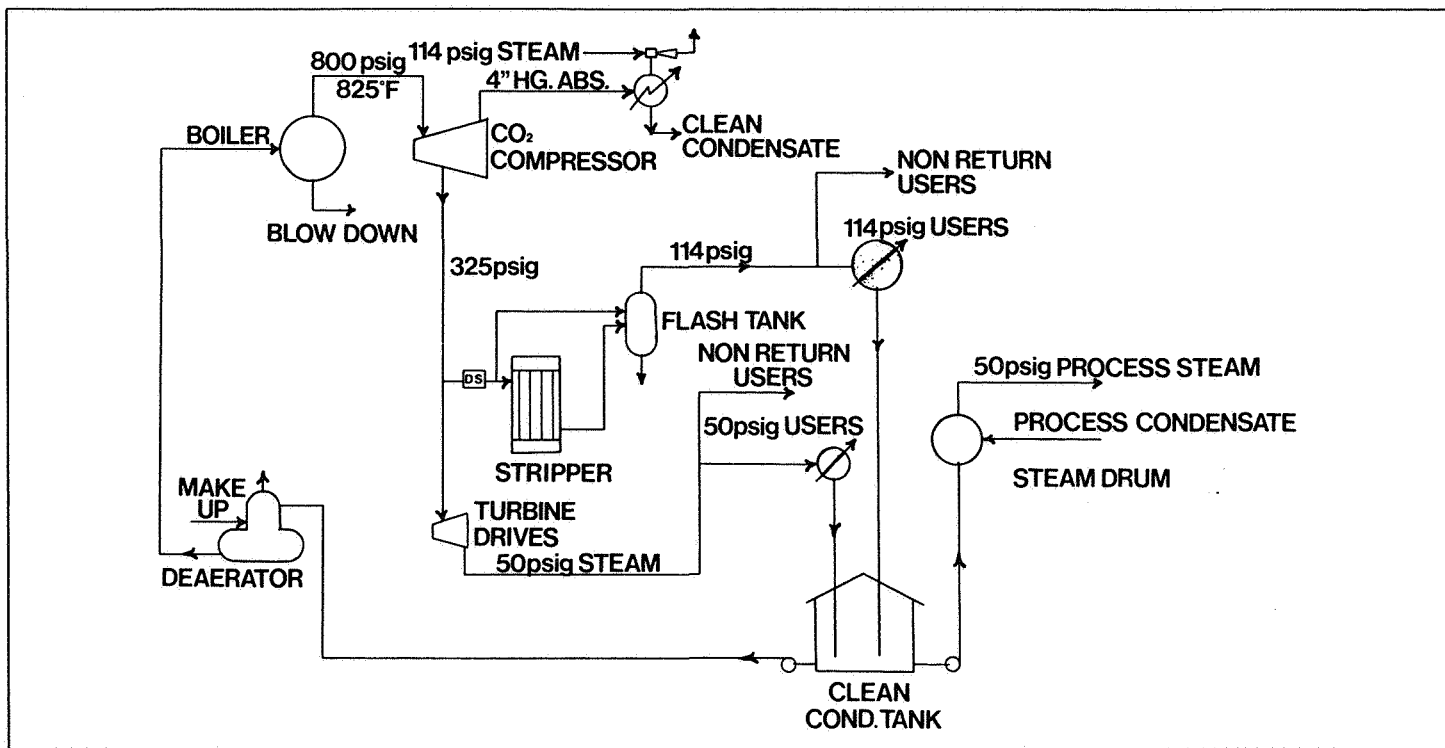


Figure 6. Simplified flowsheet of the steam system.

concentration in the process steam. To improve this situation, clean 114 lb./sq. in. gauge steam was sent to the base of the column, and the excess of process steam was introduced at the midsection. Since these alterations were made the desorption section has worked very satisfactorily with an ammonia concentration in the waste condensate of less than 50 parts/million at design rate.

### Maintenance

The plants have been in operation for too short a period for a complete maintenance record to have been compiled, but some useful observations were made at the CF Industries plant during one year of operation.

After making the initial corrections described, the CO<sub>2</sub> compressor and turbine operated without problems and needed no maintenance. Aside from minor problems with the instrumentation, the Agrico compressor and turbine operation have been completely trouble-free.

The stuffing boxes of the high-pressure ammonia and carbamate pump were changed once.

After one year of operation, the synthesis section was opened for a corrosion check. The reactor lining did not show any corrosion. Some minor corrosion spots, probably caused by slag inclusion, were found in the connection welds of the lining. The stripper tubes were checked by means of a probolog, which can measure the wall thickness very accurately. No decrease in wall thickness was found.

One area of concern with respect to corrosion was the CFI condensate treatment system. The first desorber handling raw process condensate was made from stainless steel, but beyond that vessel, carbon steel was used for most of the equipment, including the steam drum for the high-pressure carbamate condenser. Evidently, deaeration in the first desorber is excellent as no evidence of corrosion of this equipment was found.

### In conclusion

Both plants were started up very satisfactorily because

the relatively small alterations required did not interfere with urea production. At both facilities, production was frequently limited by mechanical difficulties in downstream facilities outside the FWEC-Hoechst-Uhde scope.

The CFI plant is now producing about 1,100 ton/day. The limiting factor for further production increase is the capacity of the high pressure steam boiler.

The Agrico plant is now producing about 1,050 ton/day urea. The limiting factor here is also the steam boiler. Both plants are meeting all the local and federal EPA regulations with respect to air and water pollution from urea plants.

Foster Wheeler, in collaboration with Hoechst-Uhde Corp., now has under engineering and/or construction six more similar plants—two with a capacity of 1,000 short ton/day, one with 1,100, and three with 1,500. What was learned during the engineering and startup of the CFI and Agrico plants is expected to speed the commissioning of these units. #



D. F. Bress, coordinating manager, Commercial Development Dept., Foster Wheeler Energy Corp., is a registered professional engineer who earned his B.S.Ch.E. from the Univ. of Michigan and his M.S.Ch.E. from New York Univ. Prior to his current position, Bress was process design manager, Process Design Dept., where he was responsible for fertilizer and organic chemical plants process design.



M. W. Packbier, who is technical sales manager, Hoechst-Uhde Corp., was a senior process engineer with Stamicarbon, Holland, The Netherlands prior to his present position.

## DISCUSSION

**ED JOHNSON**, Allied Chemical: Did I understand that you use centrifugal compression all the way up to reactor pressure on your CO<sub>2</sub>?

**PACKBIER**: That's correct.

**JOHNSON**: What is the minimum capacity urea plant on which you can use centrifugal CO<sub>2</sub> compression all the way?

**PACKBIER**: We think that today the minimum capacity is about 800 metric tons a day.

**JOHNSON**: About 800 metric

**PACKBIER**: That's about 900 short tons a day.

**JOHNSON**: Right. Further, if we consider your reactor, stripper, and high pressure condenser as a synthesis loop, what percentage of the CO<sub>2</sub> that you feed into that loop is converted into urea?

**PACKBIER**: The urea solution leaving the synthesis loop contains about 9% of CO<sub>2</sub>. That means that the conversion rate in the synthesis loop is about 85%.

**JOHNSON**: 85%. Thank you.

**PAOLO GRAZIANO**, Anic SPA, Milano: We have a Urea plant running on a CO<sub>2</sub> centrifugal compressor operating from 1971. I want to know the speed of your compressor, if you had problems with corrosion in the casing, because we had some, and what kind of seal you use in the compressor.

**PACKBIER**: I would like Jack Stewart of Agrico who is in the audience, to answer this question. Jack is urea plant manager of the Agrico urea plant in Blytheville.

**JACK STEWART**, Agrico: The rpm of our compressor is 9300. We didn't have any corrosion.

**PAOLO GRAZIANO**: What is the material of the casing of the compressor?

**PACKBIER**: It's normal carbon steel, but the intercoolers and all the parts which are in contact with wet CO<sub>2</sub> are from 304 stainless steel.

**PAOLO GRAZIANO**: And what kind of seals do you have in the compressor?

**PACKBIER**: Please Jack, would you also answer this question.

**STEWART**: We have silicon seals on the rotor. The first case of the compressor is coated with urethane resin. At our plant we have had no indication of any corrosion whatsoever. The efficiencies on the compressor are just as good today as they were a year ago. We watch very closely the temperatures on the interstage condensers, the separators, and make sure that we are above 110 degrees on the suction of each case. And so far, good luck.

**Q**: I have three questions. The first is, do you have a water seal on the carbamate pump?

**PACKBIER**: The stuffing box of the carbamate pump is divided in a high pressure and low pressure part. The lower pressure part is flushed with process con-

densation.

**Q**: Any problems?

**PACKBIER**: Due to the low plunger speed and the relatively low temperature and condensation of the carbamate recycle stream in a CO<sub>2</sub> stripping plant, the stuffing boxes have to be changed once in a year. In the conventional type of urea plants this has to be done every four to six weeks. Therefore you see that we have made a big improvement. We are not experiencing any other problems with these carbamate pumps.

**Q**: The second question is - What is the kind of material of the waste water treatment system.

**PACKBIER**: The first desorption column is stainless steel and everything beyond this first desorption column in the desorption section is carbon steel. The shell of the heaters using the process steam are made from stainless steel.

**Q**: The last question is related to your first desorber. You said you had foam formation and you changed the sieve trays in packing.

**PACKBIER**: Yes, for some reason the liquid in the column was foaming. This causes considerable liquid entrainment in the overhead gas stream. We solved this problem by changing the sieve trays in a packing of the described mini rings.

**Q**: Well you didn't use any anti foaming agent.

**PACKBIER**: No this was not necessary.

**BOB OSMAN**, Exxon Chemical Co.: Could you talk a little bit more about the shock cooling effect that you observed in the intercooler? Under what circumstances would it occur—is it a property of CO<sub>2</sub> systems only?

**PACKBIER**: I don't know if this problem is a property of CO<sub>2</sub> systems only. We experienced this phenomena after the first stage intercooler, in the separator. We think it is caused by a too good cooling effect and a pressure drop in the separator. The result was that the condensate was mixed in the form of fine droplets with the CO<sub>2</sub> gas. These droplets were so fine that the separator could not separate them from the CO<sub>2</sub> gas stream.

Since we did not expect this, we did not install high efficiency separators which are able to separate these fine droplets. To solve the problem we installed an extra separator in series.

**OSMAN**: Are you saying that the droplets were much finer than you normally would expect to come out of the intercooler?

**PACKBIER**: Yes, they were much finer and we didn't experience this phenomena in urea plants using reciprocating compressors. It is probably caused by the temperature and pressure relation of the centrifugal compressor.

**OSMAN**: Did you actually measure the sizes of particles

that you had in the stream, take samples?

**PACKBIER:** Yes, we took samples of the gas stream out of that separator to measure the water content. But we could not measure the size of the particles.

**S.H.R. ZAIDI,** Esso Pakistan Fertilizer Co., Ltd.: I have two questions. The first is about the oxygen level in the synthesis section. You mentioned that you are introducing some oxygen, could you tell me how and what kind of levels you are using?

**PACKBIER:** We inject air in the suction line of the CO<sub>2</sub> compressor. The total amount is such that an oxygen concentration in the total CO<sub>2</sub> stream of about .7% by volume is maintained.

**ZAIDI:** I see. The other question is about the evaporators. You mentioned that by plugging some tubes you cut down the retention time. Now could you tell me what effect on temperature it had—did you keep the same temperature.

**PACKBIER:** Yes we maintained the same temperature because there is a relationship between the pressure, the temperature, and the urea concentration. Since we

finally are interested in making a urea melt with a urea concentration of 99.8%, we could not decrease the temperature. The only alternative to decrease the biuret content was to decrease the retention time.

**Q:** Do you have sieve trays in the reactor and what is their purpose?

**PACKBIER:** In the reactor are six sieve trays. Their purpose is to prevent backmixing of the solution in the reactor and therefore to increase the conversion rate.

**Q:** Have you experienced any failure on these sieve trays?

**PACKBIER:** No, the sieve trays are welded to the wall of the reactor by means of a special construction. This is an old procedure and already proven for more than 15 years.

**Q:** The second question—do you have any on-line analyzers in this process?

**PACKBIER:** No. Both urea plants, Agrico and CF Industries have no gas chromatographs installed. Some CO<sub>2</sub> stripping plants have gas chromatographs but this is not a necessity.