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SOLVENT REFINED COAL (SRC) PROCESS

Molecular Sieve Drier Tests on PDU P-99—Interim Report for the
Period April—August 1981

Vol. VI: Process Development Unit Work
Part 3: Engineering Studies

By
J. A. Gray
J. G. Iantorno
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February 1982

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The Pittsburg & Midway Coal Mining Company
Englewood, Colorado

U. S. DEPARTMENT OF ENERGY

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Interim Report for the Period
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Vol. VI: Process Development Unit Work
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ABSTRACT

A molecular sieve (Type 3A) drier system was installed on PDU P-99 for confirmatory testing. During 70 hr of continuous operation, three drying and two regeneration cycles were completed. The drier processed bleed off-gas at unit pressure and ambient temperature in tandem with the butane scrubber. High pressure hydrogen was used to regenerate the molecular sieve adsorbent at 500°F. Although the tests should be considered preliminary because of uncertainties in the moisture and gas rate measurements, the results were in good agreement with the predicted performance, and the apparatus operated with minimum difficulty.

I. SUMMARY

According to present designs for a large-scale SRC-II plant, the high pressure recycle gas exiting the lean oil and amine scrubbers will be passed through a guard chamber before entering the cold box of the cryogenic hydrogen purification plant. The guard chamber will consist of a series of activated carbon and molecular sieve adsorption beds. A molecular sieve (Type 3A) drier system was installed on PDU P-99 for confirmatory testing, and operated continuously from June 23 until the scheduled summer turnaround on June 26 (Run P99-85). During the 70 hr of continuous operation, three drying and two regeneration cycles were completed. The drier processed bleed-off gas at unit pressure and ambient temperature in tandem with the butane scrubber. High pressure hydrogen was used to regenerate the molecular sieve adsorbent at 500°F.

Although the tests should be considered preliminary because of uncertainties in the moisture and gas rate measurements, the results were in good agreement with the predicted performance. Bleed-off gas entering the drier contained 170-180 ppm moisture, more than twice as much as originally estimated assuming saturated conditions at 50°F in the butane scrubber. The outlet gas contained 3.5-7.5 ppm moisture until breakthrough started after 6 hr into the adsorption cycle, at which time the moisture content increased rapidly. Regeneration and cooling of the drier generally required about 12 hr. The values for total water adsorbed, calculated from the experimental data, were in good agreement with the sieve capacity calculated from available equilibrium data.

Emission spectroscopy analyses for metals in fresh and unregenerated spent molecular sieve adsorbent show nothing unusual. However, carbon, sulfur, and nitrogen analyses indicated some build-up of organic nitrogen and sulfur compounds. It is not known whether these can be desorbed during regeneration since regenerated spent molecular sieve adsorbent was not available for analysis.

II. INTRODUCTION

According to the proposed design for a large-scale SRC-II plant (Figure 1), the high pressure recycle gas exiting the cold separator will be scrubbed with a wash oil to reduce the content of pentane and heavier hydrocarbons, and then with diethanolamine solution to remove hydrogen sulfide and carbon dioxide. A cryogenic process will be used to remove C_1 - C_4 hydrocarbons from the hydrogen to produce a stream of 88-91% purity. Before being fed to the cold box, the recycle gas will be cooled to 60°F or less and passed through a guard system, composed of activated carbon and molecular sieve beds, to remove traces of C_5 and heavier hydrocarbons, water, carbon dioxide, hydrogen sulfide and other sulfur compounds, and nitrogen compounds. The removal of nitric oxide (NO) is particularly important, because very low levels of this compound can combine with trace olefins to form explosive gums which can accumulate in the cold box. The other trace compounds tend to cause corrosion or to freeze out and cause plugging of the cold box.

The primary function of the Type 3A molecular sieve bed in the guard system preceding the cold box is to remove trace quantities of water vapor and, to a lesser extent, trace quantities of ammonia that may pass through the upstream scrubbing units. This type of sieve has been commercially proven for dehydration of streams containing unsaturated hydrocarbons, such as cracked gas and ethylene plant feed gas.⁽³⁾

Because SRC-II Process Development Unit P-99, located at the Gulf Research Center in Hamarville, PA, closely approximates the proposed flow sheet of a large-scale plant, a molecular sieve (Type 3A) guard chamber was installed on PDU P-99 in June 1981 to obtain design and confirmatory data.

This report describes the molecular sieve drier and the preliminary tests performed during the last few periods of Run P99-85. The test objectives included the following:

1. To confirm the dehydration performance predicted by the vendor's adsorption isotherms.
2. To maintain dehydration performance through a number of adsorption/regeneration cycles.
3. To detect any unanticipated side reactions that might lead to significant fouling.

III. EXPERIMENTAL

A. Process Development Unit P-99 Overview

The high pressure recycle gas system for PDU P-99 is shown on the process flow sheet in Figure 2. The recycle gas exiting the cold separator passed through the lean oil scrubber, which partially removed light hydrocarbons and about half of the hydrogen sulfide, and then through the diethanolamine scrubber, which removed the remaining hydrogen sulfide and carbon dioxide.* Since PDU P-99 does not have a cryogenic purification system to upgrade the purity of the recycle hydrogen, it is necessary to bleed off part of the recycle gas to remove light hydrocarbons from the system. The remainder of the recycle gas is routed to the suction side of the recycle gas compressor. Sufficient pure hydrogen from the laboratory distribution system is added to the recycle gas ahead of the recycle compressor to make up for that chemically consumed and that lost through dissolution and bleed off. Bleed-off rate is set to control the purity of the recycle hydrogen stream entering the coal slurry preheater and dissolver at the desired level.

During operation of the liquid butane scrubber, the bleed portion of the recycle gas was obtained upstream of the lean oil scrubber instead of downstream of the amine scrubber, as shown in Figure 2. This flow configuration not only provided a representative feed to the butane scrubber but also allowed operation of the butane scrubber to be decoupled from the PDU operation so that material balances were not unduly upset. Had the butane scrubber been installed to replace the lean oil scrubber, butane scrubber operation would have greatly affected PDU operation because of the large quantity of liquid butane vaporized in the scrubber. A detailed description of the butane scrubber and the test results are reported in Reference 1 and are beyond the scope of this report.

* As Figure 2 indicates, the reverse order of scrubbing is possible, if desired, and was used for runs preceding Run P99-59. The lean oil scrubber preceded the amine scrubber in all later runs.

The molecular sieve drier system was installed on PDU P-99 during June 1981 in Location A as shown in Figure 2. The flow scheme allowed the butane scrubber outlet gas to be passed through the drier system and then returned to the bleed gas metering system. The drier system could also be completely bypassed during regeneration of the molecular sieve bed. The molecular sieve drier was only operated in tandem with the butane scrubber.

B. Description of Molecular Sieve Drier

A schematic of the molecular sieve dryer is shown in Figure 3. The vessel was constructed from a 10 ft 6-3/16 in. long piece of 316 stainless steel tubing having an internal diameter of 11/16 in. and an outside diameter of 1 in. The end fittings were 1 in., medium pressure, cone-and-threaded-type crosses, also 316 stainless steel. The bottom tee held the molecular sieve support screen (20 mesh) in place approximately 4 in. above the tee center line. A thermowell, constructed from 1/8 in. OD x 1/16 in. ID 304 stainless steel tubing, was inserted through the top tee and extended half way down the vessel. The outside vessel wall was wrapped with one layer of asbestos tape, two sections of nichrome heating wire (2000 Watts per section), and several additional layers of asbestos tape and ceramic-fiber insulation. Two skin thermocouples were installed at the midpoint of each heated section. One thermocouple in each section was used with a West controller to control the skin temperature of each heated zone during the regeneration of the molecular sieves. The duplicate thermocouple was used for temperature indication and recording in the control room of PDU P-99. Two additional skin thermocouples were placed near the top and bottom of the drier column and were also used for temperature indication and recording. The internal thermocouple was used for setting the final temperature during the regeneration cycle.

C. Molecular Sieve Drier System Process Flow

The process flow for the molecular sieve drier system is illustrated in Figure 4. Approximately 10 sm³/h of bleed-off gas is taken from the recycle gas before the lean oil and amine scrubbers and is passed through the butane scrubber. The off gas from the butane scrubber is fed to the molecular

sieve drier at approximately 1950 psig and room temperature (~80°F) and flows downward through a bed of Type 3A molecular sieves. The gas exiting the drier is routed back to the bleed-off gas metering system. A slip-stream of the inlet or outlet gas, whichever is desired, is reduced in pressure to 80 psig, sent to a DuPont 510 moisture analyzer, and then routed back to the bleed-off gas metering system. Part of the sample gas is completely dried in another molecular sieve bed and is used as the analyzer reference gas. The analyzer is capable of measuring moisture contents as low as 1-2 ppm (vol). When bed saturation has occurred, as indicated by the analyzer readings, the drier is bypassed by sending the bleed-off gas from the butane scrubber directly to the bleed-off gas metering system, and the regeneration cycle is begun.

The molecular sieves are regenerated using high pressure hydrogen (2000 psig). Approximately 12 sm³/h of hydrogen is heated to 500°F in a helical tube (1/2 in. OD x 3/8 in. ID x 14 ft 10 in.) traced with two 3000 Watt heating rods and passed upflow through the molecular sieve bed. This flow direction is opposite to that used during the drying cycle. The controllers on the drier skin heaters are also set to yield an internal temperature of 500°F. The initial heatup was usually done in approximately 100°F steps and required 1-2 hr to equilibrate at 500°F. The hydrogen exiting the top of the drier column is cooled as it passes through a section of tubing that is traced with cold water; it is then metered before venting. Moisture analyzer readings of the inlet and outlet hydrogen indicate when the adsorbent has been fully regenerated. A more detailed operating procedure is given in Appendix A.

Although bleed-off gas could be fed to the molecular sieve drier from other locations besides the outlet from the butane scrubber, none of these other options were utilized in the preliminary testing discussed in this report.

D. Molecular Sieve Adsorbent

The molecular sieve adsorbent used in the bleed-off gas drying tests was Linde Type 3A purchased from Applied Science and manufactured by the Linde Molecular Sieve Division of Union Carbide Corporation. The adsorbent was obtained in an activated state as 1/16 in. diameter pellets in 1 lb dessicated containers.

Type 3A molecular sieve is a sodium aluminosilicate crystalline compound belonging to the class of zeolite minerals. The unit cell structure is a sodalite cage formed by atoms of oxygen, silicon, aluminum, and alkali metal (sodium in this case). The cages have a symmetrical, truncated octahedron shape and are stacked in a cubic fashion. The pores formed by the stacking of the sodalite cages constitute the "sieve", and in the case of Type 3A, have an opening of about 3Å. Since the crystals are small, the material is in powdered form and is generally difficult to mold. Therefore, an inert binder is added to enable forming of the mixture into pellets, usually 1/16 or 1/8 in. in diameter. The surface area is generally in the range of 500-750 m²/g and resides nearly completely in the internal cavities. The adsorbent is activated by heating to drive off water of hydration. Unlike most other hydrated minerals, molecular sieves can give up water of hydration without collapse of the crystalline structure. Thus, the remaining structure is capable of readsorbing water or other molecules and can withstand repeated adsorption-desorption cycles.

The pore size of Type 3A molecular sieve makes it selective for adsorption of water and ammonia, since most other molecules are excluded. It has been used for drying polar liquids and gases, such as methanol, ethanol, propylene, butene, and cracked gas, and for drying natural gas.

Using the data of Schoofs,⁽³⁾ it was roughly estimated that 55 g of adsorbent were needed to yield a 12 hr breakthrough time. These calculations are listed in Appendix B. This weight of adsorbent corresponded to a bed depth of 12 in. above the support screen. The adsorbent was held in place by inserting a plug of glass wool on top of the bed. The thermowell did not protrude into the bed.

Fresh and spent molecular sieves were analyzed for carbon (by a combustion method), sulfur (Leco analyzer), nitrogen (Kjeldahl method), and ash. Emission spectroscopy was used to determine metals content.

E. Data Acquisition

Data manually recorded during the drier adsorption cycle included the top and bottom skin temperatures, the internal temperature, the moisture content of the bleed-off gas before and after the drier, and the gas rate leaving the drier. All of these data were recorded once an hour, except for the moisture content of the feed to the drier, which was usually measured at the end of the cycle. The inlet moisture concentration was the more uncertain of the two concentration measurements, because repetitive hourly readings were not obtained, and this value tended to vary significantly during the tests. The bleed-off gas rate was uncertain because there were difficulties in the operation of a new McAfee gauge installed (see Reference 1 for further details) upstream of the butane scrubber. This meter was used during the first two tests and indicated a very low bleed-off gas rate during the second test. During the third drier test, the original bleed-off gas meter, downstream of the butane scrubber and drier, was used. Therefore, the measured gas rate during this test should be reasonably accurate. The rates were recorded by the PDU P-99 data logger every hour.

During the regeneration cycle, all molecular sieve column temperatures were manually recorded, and the outlet moisture content was periodically noted until complete regeneration had occurred based on a constant low moisture level. The hydrogen flow rate was set at about $12 \text{ sm}^3/\text{h}$ (450 SCFH). This was a very rough measurement performed only once during the beginning of the drying cycle before the regeneration gas became too hot to meter.

The details of operating DuPont Model 510 moisture analyzer are discussed in Appendix C. Although the analyzer compared the reference and sample gases every 30 sec, it generally required a long equilibration time before repeatable readings were obtained that could be averaged and used for the recorded reading. For this reason, it was not desirable to switch the

analyzer back and forth between the drier inlet and outlet gases more frequently than necessary. Usually when a switch was made, the analyzer was fed the same gas stream for at least 1 hr.

F. PDU P-99 Run Conditions During the Molecular Sieve Drier Tests

The molecular sieve drier tests were started during Period P99-85-38 and extended into Period P99-85-41. During Run P99-85, a 13.7% ash Ireland Mine coal was processed under standard SRC-II conditions, as shown in Table I. This run was made with the 6 in. ID x 10 ft long dissolver, having an average operating temperature of 455°C and a nominal slurry residence time of 1 hr. The important results from this run and the effect of the operating conditions on the product yields are reported in Reference 2.

The molecular sieve drier was operated in tandem with the butane scrubber during all the drier tests. The bleed off-gas exiting the butane scrubber was simply routed to the drier and then back into the low pressure bleed-off gas system. In transit to the drier, the gas warmed up to ambient temperature. The butane scrubber operating conditions during Run P99-85 are summarized in Table II and indicate that the bleed-off gas temperature near the top of the packing in the scrubber was in the range of 55-63°F during the drier testing. The liquid butane feed rate and scrubber temperature were varied as part of a scrubber test program, also conducted during Run P99-85. The butane scrubber tests are reported in Reference 1.

Although the inlet gas rate shown in Table II for Period P99-85-40 is believed to be accurate, the rates for Periods P99-38 and -39 are very uncertain because of measurement difficulties. The regular bleed-off gas meter was used during Period P99-85-40. This meter is downstream of the drier system. During Periods P99-38 and -39, a high pressure McAfee gas meter, located upstream of the butane scrubber, was used, and considerable difficulties were encountered as discussed in Reference 1. The indicated rate during Period P99-85-39 was much lower than it should have been. Thus, the values in Table II for Period P99-85-39 are adjusted results and are 20-40%

higher than the values used in this report. When possible, the gas rates used in the molecular sieve drier performance calculations were taken directly from the PDU data logger sheets without adjustment.

IV. RESULTS AND DISCUSSION

A. Moisture Breakthrough Tests

Operation of the molecular sieve drier was begun on June 23 at 19:15 and proceeded continuously in tandem with the butane scrubber for 70 hr until PDU P-99 was shutdown on June 26 for the scheduled turnaround. During the 70 hr period, data were obtained for three drying and two regeneration cycles. The drier performance data are summarized in Figure 5, in which bleed-off gas moisture content at the drier outlet is plotted versus the hours on-stream. Moisture analyzer meter readings were taken every hour at either the column inlet or outlet during both the drying and regeneration cycles. As indicated in Table III, the average moisture contents of the bleed off-gas for the three drying cycles were 180, 170, and 142 ppm at the drier inlet. Before breakthrough, moisture levels at the drier outlet were 4.2, 7.5, and 3.5 ppm.

Another important quantity obtainable from the analyzer data in Figure 5 is the breakthrough time, or the elapsed time from the beginning of a drying cycle to the point when the moisture level in the outlet gas increases significantly. Estimated breakthrough times for the three cycles, based on the operating data replotted on a common time scale in Figure 6, were all 6 hr. This time is less than half that estimated in Appendix B because the moisture content used in the estimate was half of the actual water content. The two regeneration cycles, including drier cool down, were completed in 12-13 hr. The complete drier operating log is listed in Table IV.

In order to compare the actual equilibrium data, it is necessary to integrate the data in Figure 5 to obtain the total amount of water adsorbed. The integration is performed using the following equation:

$$W = \rho \int_0^{\theta} (Q_i y_i - Q_o y_o) dt \quad (1)$$

where Q_i and Q_o are the bleed-off gas flow rates into and out of the drier, y_i and y_o are the mole fractions of water vapor in the bleed-off gas into and out of the drier, t is the adsorption time, θ is the total adsorption cycle time,

ρ is the density of water vapor at 60°F and 1 atm, and W is the total water adsorbed. Since the water vapor content is less than 200 ppm, Q_i and Q_0 are essentially the same. To further simplify the integration, the total gas fed to the drier during the adsorption cycle was determined and divided by the cycle time to yield an average gas rate. Thus, Equation 1 reduces to:

$$W = \rho Q \int_0^{\theta} (y_i - y_0) dt \quad (2)$$

where y_i was constant for these tests.

The areas for the flat portions of the plots in Figure 6 were determined by simply using the minimum values in Table III for y_0 and multiplying by the time. Since the data points were reasonably closely spaced, the remaining area was calculated by simply drawing straight line segments between the points, calculating the area for each segment and summing the segments. These results are summarized in Table V. The areas under the adsorption curves listed in Table V represent the areas under the curves in Figure 6 down to the baseline moisture level. This area is proportional to the moisture remaining in the gas exiting the drier relative to the inlet gas. The difference in moisture content between the inlet gas and outlet gas is multiplied by the hours on-stream, and the area under the curve is subtracted to obtain the area that is proportional to the water adsorbed from the inlet gas, that is, the area above the adsorption curve in Table V.

The theoretical loading capacity of Type 3A molecular sieves was calculated using the water vapor adsorption isobar data reported by Schoofs.⁽³⁾ At the drier operating temperatures of 88 to 97°F, the equilibrium water content is 22.0 lb/100 lb of sieves, while at the regeneration temperature of 500°F, the equilibrium water content is 2.5 lb/ 100 lb of sieves. Thus, the available capacity is 22.0-2.5 or 19.5 lb of water/100 lb of sieves. Since 55 g of adsorbent was charged to the drier, the water capacity was 10.7 g or 0.024 lb. A comparison of this capacity with the loadings calculated from the experimental data in Table V shows good agreement (considering the uncertainty in the inlet moisture content and the bleed-off gas rate), indicating the adsorbent performed as predicted.

Several minor operating difficulties were encountered which may have influenced the test results. The temperature of the moisture analyzer crystal oven was not controlled at 130°F during the first adsorption cycle and drifted somewhat. In addition, the oven did not always cycle during the first adsorption cycle so as to admit a fresh sample every 30 sec. Both the above difficulties could have introduced errors in the moisture readings during the first cycle.

Another problem that developed during the regeneration cycle was the failure of the cold water tracing to adequately cool the hot hydrogen regeneration gas exiting the drier. As a result, the regeneration gas meter could not be used continuously. This did not affect the test results, but is a problem that should be corrected before the drier system is started up again.

During the third drying cycle, the butane scrubber was taken out of operation for 1 hr in order to install a fresh cylinder of liquid butane. Although the higher temperature in the scrubber during this period would be expected to increase the moisture entering the drier and slightly decrease the breakthrough time, the experimental data seem to indicate little, if any, effect.

B. Spent Adsorbent Analyses

Analyses of the Linde Type 3A molecular sieves, used in the bleed-off gas drier study during the last few periods of Run P99-85, are listed in Table VI. After the third and final adsorption cycle, the drier packing was not regenerated. Therefore, the analytical data represent the condition of this molecular sieve packing immediately after water vapor breakthrough. Using the analyses of the new material as a benchmark, several important observations can be made. First, the weight loss on ashing is very low (6-7%) compared to the literature value of 18.0%* for the equilibrium water loading at 90°F. This would indicate that less than half of the theoretical capacity

* 22.0 lb water/(100 lb sieve + 22.0 lb water)

of the sieve is available. The experimental capacity data do not agree with this finding and suggest errors in the ash analysis, possibly due to over-exposure of the samples to air. Second, the large increase in sulfur and nitrogen content of the spent sieves compared to the fresh sieves suggests that significant quantities of sulfur and nitrogen compounds were adsorbed in addition to the water. The carbon content also increased a significant amount above that initially present in the binder. Since the sieves were not regenerated and sampled after the last breakthrough, it is possible that these compounds would desorb and may only have been present in the macropore structure of the binding material. Also, it is not clear why the bottom of the bed was more contaminated than the top, since the gas flow direction was downward during drying and upward during regeneration. This may be related to the adsorption dynamics of these particular compounds. Further testing is needed to evaluate the life of the adsorbent and to determine whether or not the carbon, nitrogen, and sulfur compounds can be desorbed during regeneration or whether they would lead to any damage to the sieves.

V. CONCLUSIONS

The molecular sieve drier test results should be considered preliminary, because the tests were limited in scope due to the short time available before PDU P-99 was shut down, and because there were uncertainties in the bleed-off gas rates and moisture contents at the inlet to the drier. However, the results that were obtained were in good agreement with predicted performance, and the unit operated with a minimum of difficulty.

Based upon the experience with these drier tests, the following recommendations are made for the improvement of the drier system:

1. Install a new system to cool the regeneration hydrogen leaving the drier. The present cold water tracing is not adequate and precludes continuous measurement of the regeneration gas rate.
2. Add a strip chart recorder to the moisture analyzer and install a separate analyzer for the drier inlet gas.

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Table I

**PDU P-99 OPERATING CONDITIONS AND
YIELDS DURING THE MOLECULAR SIEVE DRIER TESTS**

<u>Period No., P99-</u>	<u>85-38</u>	<u>85-39</u>	<u>85-40</u>
Operating Conditions			
Temperature,* °F	855	855	856
Pressure, psig	2002	2000	2000
Residence Time, hr	0.99	0.99	0.98
Coal** in Feed Slurry, wt%	30.3	30.3	31.3
Recycled Solids in Feed Slurry, wt%	14.5	14.4	14.3
Gas Rate, 10 ³ SCF/ton of Coal	53.7	55.4	50.8
Outlet H ₂ Part. Press., psia	1410	1410	1410
Yields, wt% Moisture Free Coal			
Hydrogen	-3.38	-3.41	-3.38
C ₁ -C ₄	14.39	14.11	13.97
NH ₃ , H ₂ S, CO, CO ₂	4.63	4.62	4.66
Water	4.99	5.06	5.04
C ₅ -380°F	6.67	6.69	6.38
380-550°F	12.38	13.62	13.23
550-900°F	<u>10.56</u>	<u>9.47</u>	<u>11.08</u>
Total C ₅ -900°F	29.60	29.78	30.69
900°F+ Pyridine Solubles	28.91	29.03	28.34
Insoluble Organic Matter	6.63	6.60	6.46
Ash	<u>14.21</u>	<u>14.21</u>	<u>14.21</u>
Total 900°F+	49.76	49.84	49.02

* Average dissolver temperature.

** Pittsburgh Seam Coal, Ireland Mine (LR-29243).

Table II

BUTANE SCRUBBER OPERATING CONDITIONS DURING THE MOLECULAR SIEVE DRIER TESTS

Period No., P99-	Liquid Butane Rate, lb/hr	Gas Inlet Rate, sm ³ /h	Feed Ratio lb C ₄ H ₁₀ /1,000 SCF	Temperatures, °F							
				Gas		Liquid		Bottom ¹	Upper ² Bottom ²	Lower ³ Top ³	Top ⁴
				In	Out	In	Out				
85-23	2.00	11.278	5.02	54	57	55	64	41	43	45	46
85-24	2.15	12.557	4.85	50	55	52	64	41	43	46	48
85-25	2.50	10.151	6.97	50	55	50	59	39	43	46	48
85-31	2.00	13.762	4.12	50	73	57	72	45	52	54	55
85-32	1.97	8.725	6.39	57	66	52	72	45	46	48	50
85-33	2.08	9.629	6.12	59	70	59	77	48	50	54	54
85-34	2.46	10.478	6.65	61	72	57	72	48	50	54	55
85-37	1.75	9.480	5.23	55	70	54	73	50	50	54	54
85-38	1.95	9.585	5.76	64	70	64	73	52	52	54	55
85-39	2.55	10.520	6.86	61	70	64	73	48	50	52	54
85-40	2.42	8.521	8.04	72	79	68	79	57	59	61	63

¹Bottom thermocouple is located 6 in. above bottom of the packing

²Upper bottom thermocouple is located 16 in. above bottom of the packing

³Lower top thermocouple is located 16 in. below top of the packing

⁴Top thermocouple is located 6 in. below top of the packing

Table III
SUMMARY OF MOLECULAR SIEVE DRIER TEST RESULTS

	<u>Run No.</u>		
	<u>1</u>	<u>2</u>	<u>3</u>
On-Stream Time, hr	16	13	15
Average Drier Temperature, °C	31	36	33
Moisture Content, ppm			
Gas In	180	170	170*
Gas Out	4.2	7.5	3.5
Approximate Breakthrough Time, hr	6	6	6

* Although 142 ppm was measured during this run for the inlet gas, the final outlet value was 150 ppm. Therefore, the value for Run 2 was assumed for Run 3.

Table IV
MOLECULAR SIEVE DRIER OPERATING LOG

Date	Time	Temperatures, °C						Moisture		Analyzer Scale
		H ₂ Preheater		Drier Skin		Drier Internal	Gas Meter	Content, ppm		
		Top	Bottom	Top	Bottom			Gas In	Gas Out	
6/23/81	19:20 ¹	-	-	33	36	33	-	-	10.3	25
"	20:15	-	-	30	31	30	-	-	7.9	25
"	21:15	-	-	30	30	29	-	-	5.0	25
"	22:15	-	-	29	29	29	-	-	4.3	25
"	23:15	-	-	32	33	32	-	-	4.2	5
6/24/81	00:15	-	-	31	31	31	-	-	4.2	5
"	01:15	-	-	30	30	30	-	-	22	25
"	02:15	-	-	29	29	29	-	-	38	250
"	03:15	-	-	29	29	29	-	-	58	250
"	04:15	-	-	29	29	29	-	-	69	250
"	05:15	-	-	33	32	33	-	-	80	250
"	06:15	-	-	33	33	33	-	-	92	250
"	07:15	-	-	33	33	33	-	-	110	250
"	08:15	-	-	30	30	30	-	-	112	250
"	09:15	-	-	31	31	31	-	180	-	250
"	10:15	-	-	33	33	33	-	-	170	250
"	11:45 ²	190	212	156	143	153	66 ³	30	-	250
"	12:45	226	264	257	248	260	43	-	380	2500
"	13:45	229	257	258	248	264	38	-	-	-
"	14:45	227	259	257	248	261	39	-	-	-
"	15:45	226	260	257	248	259	39	-	72	250
"	16:45	229	257	258	248	264	40	22	-	250
"	17:45	229	257	258	248	264	40	21.1	-	25
"	18:45	228	257	258	248	264	41	17.1	-	25
"	19:45	226	260	257	248	259	41	-	19.8	25
"	20:45	226	260	257	249	259	40	-	16.8	25
"	21:45	39	42	76	62	63	38	-	6.4	25
"	22:45 ⁴	39	38	41	38	27	37	-	-	25
"	23:45	-	-	38	38	38	-	-	11	25

¹ Drier on-stream at 19:15

² Drier off-stream at 11:15 and regeneration started (2000 psig hydrogen)

³ Meter bypassed because it became too hot; 1 hr rate 470.0 ft³

⁴ Drier on-stream at 22:45

Table IV (Continued)
MOLECULAR SIEVE DRIER OPERATING LOG

Date	Time	Temperatures, °C					Gas Meter	Moisture Content, ppm		Analyzer Scale
		H ₂ Preheater		Drier Skin		Drier Internal		Gas In	Gas Out	
		Top	Bottom	Top	Bottom					
6/25/81	00:45	-	-	37	37	36	-	-	8.5	25
"	01:45	-	-	37	36	36	-	-	8.0	25
"	02:45	-	-	37	36	36	-	-	7.5	25
"	03:45	-	-	36	36	36	-	-	15.0	25
"	04:45	-	-	36	36	36	-	-	27.0	250
"	05:45	-	-	36	36	36	-	-	50.0	250
"	06:45	-	-	36	36	36	-	-	75.0	250
"	07:45	-	-	35	35	35	-	-	85.5	250
"	08:45	-	-	35	35	35	-	150 ⁵	115	250
"	09:45	-	-	36	35	35	-	-	132.5	250
"	10:45	-	-	37	36	36	-	170	-	250
"	11:45 ⁶	-	-	38	37	37	-	-	165	250
"	12:45	149	166	159	152	157	39 ⁷	29	-	250
"	13:45	237	270	262	256	269	40	23.6	-	25
"	14:45	234	265	263	255	268	39	-	23.5	25
"	15:45	245	270	263	255	274	36	13.0	-	25
"	16:45	242	271	263	254	268	37	12.0	-	25
"	17:45	248	283	262	258	262	38	-	20.5	25
"	18:45	248	282	262	257	262	39	-	19.0	25
"	19:45	248	282	262	257	262	38	-	16.3	25
"	20:45	249	283	262	257	262	37	-	13.5	25
"	21:45	248	282	262	257	262	36	-	13.2	25
"	22:45	248	282	262	257	262	36	-	13.0	25
"	23:45	247	282	261	257	262	35	-	10.8	25

⁵ Read at 08:30
⁶ Drier off-stream at 11:45
⁷ Meter bypassed

Table IV (Continued)
MOLECULAR SIEVE DRIER OPERATING LOG

Date	Time	Temperatures, °C						Moisture Content, ppm		Analyzer Scale
		H ₂ Preheater		Drier Skin		Drier	Gas	Gas In	Gas Out	
		Top	Bottom	Top	Bottom	Internal	Meter			
6/26/81	00:45	34	35	43	50	43	32 ¹¹	-	7.0	25
"	01:45	32	32	34	33	33	31 ¹¹	-	5.1	25
"	02:45 ⁸	31	31	32	31	31	33	-	3.5	5
"	03:45	-	-	31	31	31	-	-	4.5	5
"	04:45	-	-	33	33	33	-	-	4.2	5
"	05:45	-	-	33	33	32	-	-	3.9	5
"	06:45	-	-	32	32	32	-	-	5.5	25
"	07:45	-	-	32	32	32	-	-	10.3	25
"	08:45 ⁹	-	-	30	31	30	-	-	25	250
"	09:45 ⁹	-	-	30	30	30	-	-	58	250
"	10:45	-	-	31	31	31	-	-	76.5	250
"	11:45	-	-	32	31	31	-	-	87.5	250
"	12:45	-	-	32	32	32	-	-	100.5	250
"	13:45	-	-	33	32	32	-	-	110	250
"	14:45	-	-	33	33	33	-	138	-	250
"	15:45	-	-	34	34	34	-	142	-	250
"	16:45	-	-	34	34	34	-	-	135	250
"	17:45 ¹⁰	-	-	35	34	35	-	-	150	250

- ⁸ Drier on-stream at 02:50
⁹ Liquid butane feed empty at 08:15, recharged and restarted at 09:20
¹⁰ Drier off-line at 17:45
¹¹ Gas meter rate after cool down - 797.4 ft³ for 1 hr

Table V
COMPARISON OF CALCULATED AND EXPERIMENTAL WATER CAPACITY

	<u>Run No.</u>					
	<u>1</u>	<u>2</u>	<u>2⁵</u>	<u>3</u>	<u>3⁵</u>	<u>3⁵</u>
Area Under Adsorption Curve, ppm-hr	1000.1	667.5	667.5	887.75	887.75	887.75
Area Above Adsorption Curve, ppm-hr	1812.7	1445.0	1575.0	1759.75	1609.75	1309.75
Moisture Content of Inlet Gas, ppm	180	170	180 ³	170 ⁴	180 ³	150 ³
Average Gas Rate, sm ³ /hr	7.091	8.545 ²	8.545 ²	8.545	8.545	8.545
Total Gas Processed, sm ³	106.4	111.1	111.1	128.2	128.2	128.2
Water Adsorbed, g	9.770	9.390	10.233	10.455	11.431	8.510
Percent of Calculated Capacity ¹	91.1	87.6	95.4	97.5	106.6	79.3

- ¹ Calculated capacity: 55 g sieves x 19.5 g water/100 g sieve = 10.7 g.
- ² Used value from Run 3, since the bleed-off gas McAfee gauge malfunctioned.
- ³ Assumed for estimation purposes.
- ⁴ The inlet value of 142 ppm was below the final reading of 150 ppm; therefore, the previous inlet reading was used.
- ⁵ Alternate estimate using a different moisture content for the inlet gas.

Table VI

**ANALYSIS OF LINDE TYPE 3A MOLECULAR
SIEVES USED IN THE PDU P-99 BLEED-OFF GAS DRIER**

<u>Sample</u>	<u>Composition</u>		
	<u>New</u>	<u>Used Top of Bed</u>	<u>Used Bottom of Bed</u>
Ash, wt%	100.0	94.15	92.91
Metals, wt% of Ash			
Aluminum	25	25	20
Barium	Tr	Tr	Tr
Calcium	1	1	1
Chromium	0.05	0.03	0.03
Copper	0.006	-	-
Iron	0.6	0.5	0.6
Magnesium	0.09	0.4	0.06
Manganese	-	-	0.02
Nickel	0.03	0.02	0.02
Potassium	6.9	6.4	6.2
Silicon	15	15	20
Sodium	3.5	3.6	3.6
Strontium	0.09	0.07	0.05
Vanadium	Tr	0.06	-
Carbon, wt%	0.25	0.34	0.53
Nitrogen, wt%	0.006	0.48	0.65
Sulfur, wt%	<0.04	0.45	0.53

Figure 1
HYDROGEN TREATMENT AND RECOVERY
SYSTEM FOR A LARGE-SCALE SRC-II PLANT

Figure 3
SCHEMATIC OF MOLECULAR
SIEVE DRIER USED ON
PDU P-99

Figure 4
MOLECULAR SIEVE DRIER PROCESS FLOWSHEET

Figure 5
MOLECULAR SIEVE DRIER OPERATING CHRONOLOGY

Figure 6
MOLECULAR SIEVE DRIER OUTLET GAS MOISTURE
CONTENT VERSUS ON STREAM TIME

APPENDIX A

PDU P-99 MOLECULAR SIEVE DRIER COLUMN OPERATING PROCEDURE

Unit Description

A flow diagram of the Molecular Sieve Drier System is shown in Drawing No. PD# P099-23. The system consists of the following equipment:

- a. Molecular Sieve Drier Column - Drawing No. PD30142-01
- b. Hydrogen Regeneration Gas Heater - Drawing No. FA00-C-3843
- c. Regeneration Off-Gas Meter - 450 SCFM Capacity
- d. DuPont 510 Moisture Analyzer - The cell is located near the column, and the indicator is installed in one of the control panels. The drier off-gas is fed to both the sample and reference side of the instrument. The reference gas passes through a molecular sieve drier first. The exhaust gas from the analyzer is vented into the bleed-off gas stream ahead of the meter.

Purpose of Operation

The purpose of operating the Molecular Sieve Drier is to obtain information on the water removal capacity of Type 3A molecular sieves and the regenerability of these sieves. The water breakthrough point is to be determined, as well as the number of cycles the sieves can be regenerated.

Description of Operation

The butane scrubber off-gas or the bleed off-gas stream after the amine scrubber can be fed to the column. The drier column will operate at

2000 psi and ambient temperature. The feed gas enters the top of the column and exits at the bottom to the bleed-off gas system. The gas flow rate is controlled by the existing metering valve.

Whenever it is desired, a gas sample from the outlet of the column can be fed to the moisture analyzer. The moisture analyzer will automatically withdraw a sample every 30 sec and display a value on the indicator dial.

When moisture breakthrough occurs, the column will be bypassed. Pure hydrogen will then be fed through the bottom of the column and out the top and measured by the regeneration gas meter. This gas rate is regulated by a metering valve. The hydrogen is heated to 500°F by an electric heater, and the drier is heated to 500°F by the heater windings. A sample of the outlet gas is sent to the moisture analyzer, and the moisture content is monitored. When the moisture content reaches the desired level, the column will be cooled and the cycle can then be repeated.

Safety

Since there is no chemical reaction in the drier, safety precautions involve handling hydrocarbon vapors under pressure. Normal pressure testing and safety precautions recommended for hydrogen should be followed.

Unit Preparation

The following preparation steps are required:

- a. Isolate and pressure test the drier and high pressure lines at 2000 psi nitrogen and 3000 psi hydrogen.
- b. Check the hydrogen heater circuit, drier heaters, and thermocouples.
- c. Prepare moisture analyzer for operation.

Start-up Procedure

This start-up procedure can be followed after the above unit preparation has been completed.

Normal Feed Gas Operation

- a. Set the column valves so that the specified feed gas will flow down the column and out the bottom to the existing bleed-off gas metering valve.
- b. Open the gas inlet valve to the column, and bring the column to the supply pressure.
- c. Close the system by-pass valve, and open the system outlet valve just ahead of the bleed-off gas metering valve. The bleed-off gas should now be flowing through the column.
- d. Open the supply valve to the moisture analyzer, and set the regulator at 80 psi. The analyzer should now be ready for operation.
- e. Measure moisture content in feed gas before sampling bottom outlet gas to determine breakthrough moisture value.
- f. Whenever the moisture breakthrough point is observed (see analyzer instructions), the column should be prepared for regeneration.

Regeneration Cycle for Drier

- a. Shut-off column inlet and outlet valves, and open system bypass valve. Stop gas flow to moisture analyzer.

- b. Set valves so 2000 psi hydrogen can enter the bottom of the column, exit at the top, and flow through the regeneration gas meter.
- c. Set the hydrogen Tescom regulator at 2000 psi outlet, and start hydrogen through the drier at the prescribed rate by adjusting the metering valve ahead of the gas meter.
- d. Turn on the cold water to the tracing on the outlet line from the drier to the gas meter.
- e. Turn on the heaters on the hydrogen gas preheater and drier to attain 500°F inside the drier. Be careful not to heat the molecular sieve packing above 550°F, as this will cause deactivation.
- f. Turn on the gas flow from the top of the drier to the moisture analyzer.
- g. When the moisture level reaches a prescribed low concentration, the drier is ready for normal operation. The low concentration is determined by sampling the regeneration gas at the bottom of the column.
- h. Shut-off the hydrogen gas and drier heaters, and maintain gas flow to cool down the drier. When the column reaches about the ambient temperature, stop the regeneration cycle, and prepare for normal operation, as described in "A" above.

Shut-down Procedure

Whenever the experiment is completed, this shut down procedure can be followed:

- a. Stop the gas flow through the drier, and divert the gas through the bypass valve.
- b. Shut off all heaters and water tracing.
- c. Depressure the drier into the blowdown, and nitrogen purge.
- d. Shut off the moisture analyzer.

Emergency Shutdown Procedure

In the event an emergency occurs in the drier system, such as a high pressure leak or fire, the following procedure should be followed:

- a. Shut-off the feed gas supply.
- b. Divert the bleed-off gas to its normal flow path.
- c. Shut off all heaters and hydrogen gas feed, if you are in the "regeneration cycle."
- d. Depressure the drier into the blowdown.

Table A-I

MOLECULAR SIEVE DRIER THERMOCOUPLE AND HEATER LIST

<u>Item</u>	<u>Thermocouple No.</u>	<u>West Controller No.</u>	<u>No. 1 Recorder Point</u>
H ₂ Preheater - Top	364	64	1
H ₂ Preheater - Bottom	365	65	2
Molecular Sieve Column - Top	367	66	3
Molecular Sieve Column - Bottom	368	67	4
Molecular Sieve Column - Internal	369		5
Regeneration Gas Meter	370		19

Figure A-1
MOLECULAR SIEVE COLUMN
THERMOCOUPLE LOCATIONS

APPENDIX B

ESTIMATION OF ADSORBENT CHARGE WEIGHT

1. Calculate Moisture Content of Feed

Since the gas is cooled to 50°F in the butane scrubber, the water content of the gas should approximate saturated conditions at 50°F.

Assume ideal gas conditions:

$$\text{at } 50^{\circ}\text{F}, P_{\text{H}_2\text{O}}^{\circ} = 0.178 \text{ psia}$$

$$\begin{aligned} \text{Mole \% Water} &= \left(\frac{0.178 \text{ psia}}{1965 \text{ psia}} \right) \times 100 = 0.00906\% \\ &= 90.6 \text{ ppm (vol)} \end{aligned}$$

$$\text{Gas Flow} = 390 \text{ SCFH}$$

$$\begin{aligned} \text{Water Feed} &= \frac{390 \text{ SCFH}}{379.6 \text{ lb mole/SCF}} \times \frac{0.00906}{100} \times \frac{18.016 \text{ lb}}{\text{lb mole}} \\ &= 0.00168 \text{ lb/h} \end{aligned}$$

2. Calculate Water Capacity of Sieve Between Regeneration Cycles

$$\begin{array}{ll} \text{Equilibrium Water @ } 90^{\circ}\text{F} & 22.0 \text{ lb/100 lb dry sieves} \\ \text{Equilibrium Water @ } 500^{\circ}\text{F} & 2.5 \text{ lb/100 lb dry sieves} \\ \text{Capacity} & = 19.5 \text{ lb/100 lb dry sieves} \end{array}$$

3. Calculate Quantity of Sieves Required for 12 hr Adsorption Cycle

$$\text{Sieves Required} = \frac{0.00168 \text{ lb H}_2\text{O}}{\text{h}} \times \frac{100 \text{ lb sieve}}{19.5 \text{ lb H}_2\text{O}} \times 12 \text{ h}$$

$$= 0.103 \text{ lb sieves} = 46.8 \text{ g}$$

4. Calculate Bed Depth (0.6875 in. ID)

1/16 in. pellets, bulk density = 47 lb/ft³

$$\text{Bed Volume} = \frac{0.103 \text{ lb}}{47 \text{ lb/ft}^3} = 0.0022 \text{ ft}^3$$

$$\text{Bed Depth} = \frac{(0.0022 \text{ ft}^3)}{\frac{\pi}{4} \left(\frac{0.6875}{12} \text{ ft} \right)^2} = 0.85 \text{ ft} = 10.2 \text{ in.}$$

6. Weight and Depth Selected

Use a 12 in. bed depth

$$\text{This corresponds to } \frac{12}{10.2} \times 46.8 \text{ g} = 55.0 \text{ g}$$

and 14.1 hr breakthrough time

APPENDIX C

DUPONT 510 MOISTURE ANALYZER DESCRIPTION AND OPERATING INSTRUCTIONS

General Principle or Operation

The DuPont 510 Moisture Analyzer is used to measure the moisture content of the process gas entering or exiting the molecular sieve dryer. Sample and dry (reference) process gases entering the analyzer are passed through two quartz crystal oscillators. The crystals are coated with a thin film of hygroscopic material, allowing them to readily adsorb moisture. Every 30 sec, the gas flow is automatically switched, so that each crystal is exposed alternately to sample and reference gas. Changes in crystal weight due to moisture sorption vary the frequencies at which the crystals oscillate. The moisture content of the sample gas is determined by comparing the oscillatory frequencies of the two crystals. The frequency difference is ultimately converted to moisture content in ppm (volume/volume) which is read directly off the instrument meter.

General Operating Instructions

1. Start-up: When power is applied to the unit, both the "power" and "oven" lights should come on. Set the range selector to the 0-25,000 position and allow the tubes to warm up and the crystal oven to come up to temperature (the oven operates at about 130°F). The instrument is ready for use when the "oven" light begins to cycle.
2. Set the range selector to bring the moisture reading on scale. The indicator needle should move continuously between 0 (zero) ppm* and some maximum value. This maximum value is the

* Parts per million, volume/volume

moisture content of the sample gas in ppm. The approximate time for one "cycle" (the time it takes for the indicator to move from 0 to the maximum value) is about 30 sec.

3. If consecutive meter readings are not consistent, use the balance knob to adjust the indicator so that consecutive readings are about the same. Alternatively, consecutive readings may be averaged to obtain a fair approximation.

Low ppm Measurements (0-5 Range)

For very low moisture levels (0-5 ppm), the frequency difference of the crystal oscillators is very small, and at around 1 or 2 ppm, they tend to lock in at the same frequency. As a result, no meter reading is obtained. To obtain a meter readout in the case of low moisture content, the following procedure should be used:

- a. Set the range selector to the 0-5 ppm position.
- b. Adjust the balance slowly until the indicator needle oscillates equally above and below the mid-scale zero of the 0-5-0 ppm off-set scale.
- c. Read moisture content in ppm directly on the 0-5 off-set scale.