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**Quarterly Summary Report
July-September 1977
Process for Cleaning and
Removal of Sulfur Compounds
From Low Btu Gases**

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October 1977

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Battelle

Pacific Northwest Laboratories

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I. OBJECTIVE AND SCOPE OF WORK PHASE IV

In this phase of work the Process Development Unit (PDU) is to be remodeled by incorporation of appropriate subsystems to permit operation in continuous process mode. The PDU will be operated for a period of time sufficient to demonstrate process viability.

II. SUMMARY

1) The subcontract to Alon Processing Inc. was completed some six weeks behind schedule but all "Alonized" pipe, fittings, and vessels have now been returned to us. This material has been assembled and mounted on the support structure.

2) A major part of the electrical work including assembly of relay and circuit breaker enclosures, installation of screwcover wireway, and installation of large transformers under subcontract to Lord Electric Company has been completed.

3) Installation of thermocouples and electrical heating elements by Battelle-Northwest's crafts personnel is completed and final wiring is nearly complete.

4) Installation of high-temperature insulation (Kaowool) and a final waterproof outer covering is 90 percent complete.

5) Test operation of individual equipment items is proceeding as rapidly as these become operational.

6) Preliminary calibration of all gas chromatographs is complete and the equipment will be moved to the Chemical Engineering Laboratory and Process Demonstration Unit site within a few days. Installation of sample lines can then be completed.

III. TECHNICAL PROGRESS

The final shipment of alonized pipe and fittings returned to us by Alon Processing Inc. arrived about 7/15/77 and final fitting of pipe and process vessels was completed shortly thereafter. Completion of the pipefitting and equipment mountings by fitters and millwrights allowed the instrument and electrical crafts to move in and install thermocouples and trace heating elements. In addition, a subcontractor, The Lord Electric Company, assisted us by installing heavy electrical gear including a 275 KVA transformer, large conduit, relay and circuit breaker enclosures, and screwcover wireway. With completion of thermocouple and trace heater installation, application of high temperature insulation could begin and personnel from E. J. Bartell Company (Spokane, WA) have this work close to completion (90%). Numerous small pipe and tubing runs for steam, CO₂, N₂, and water have been installed in conjunction with needs for purge gas, cooling gas or water, steam requirements, instrument requirements, and sampling lines.

Thermocouples have been installed so as to register the pipe wall or equipment wall temperature. The thermocouple junction was inserted into a small hole drilled in the wall. The edges of the hole were then peened with a punch against the junction to secure it in place. The junction was then covered with an 1/8 in. thick layer of Kaowool paper taped in place with glass tape to reduce exposure to direct radiation from the heaters. Thermocouples have been installed in pairs, e.g., on opposite sides of a pipe with a beaded section emerging through the insulation. Only one will be connected to the lead wire (the other is a spare). About 90 pairs of thermocouples have been installed.

Great care has been exercised during installation of trace heaters to protect against accidental shorting to pipe or equipment surfaces. The heaters are furnace elements and consist of coils of nichrome wire wound on

ceramic grid. A ceramic grout covers the wire and secures it in place. When new the ceramic grout is very fragile and crumbles away easily if the lead wires are flexed. In addition it is very vulnerable to moisture. We discovered this when we were caught by a surprisingly heavy rain with some of these exposed. The grout was actually washed away in some cases and the elements had to be regouted.

Once the elements have been heated to a red heat, the grout becomes very strong and water insoluble. Once heated, however, the nichrome wire, including the lead wire, becomes brittle and increasingly fragile.

To protect against accidental shorting to the metal walls, the elements were held away from the wall about 1/4 in. by two layers of 1/8 in. thick Kaowool paper placed near the ends of the elements. Small pieces of MgO brick were used as separators between elements and to stand off the element from flanges or support tabs on vertical runs.

All lead wires were beaded with 1/4 in. diameter ceramic beads, about 1/4 in. long. Application of these was a tedious operation. Finally, all of the lead wires had to be fitted with a clamp, which produced the desired series--parallel wiring combination, and labeled (because when covered by insulation there is no way to determine which heater the wire connects to). Our experience following the first rainstorm also demanded that we cover all heaters with plastic to protect against possible damage. This plastic remains in place until the final insulation and weatherproofing is completed.

Figure 1 shows the plastic covered 4 in. line on which trace heaters have been installed. This line is visible coming down the outside of the support structure. Also clearly visible on the left is the extraction column and packed bed de-entrainer encased in trace heaters covered by plastic.

Figure 2 shows the extraction column and in the upper left the regeneration column is clearly visible. The screwcover wireway is illustrated in this photo.

Figure 3 is a photo showing the 275 KVA transformer (extreme rear center). The curious pattern is caused by shadows. Magnetic reactors and circuit breakers are at right. The cabinet in the right foreground (not installed) contains

FIGURE 1. Trace Heater Installation on Extraction Column,
Packed Bed De-entrainer and 4 in. Line

31 solid state proportioning controllers. The Progressive Equipment Company controllers at left foreground were part of the original PDU and provide control for the large pot furnaces.

Figure 4 shows some of the numerous small pipe and tubing runs. The 1.5 in. line with the spiral heater is the regenerator off-gas line. This

FIGURE 2. The Regeneration Column at Upper Left and the Screwcover Wireway Are Visible in This Photo

beaded nichrome wire heater will heat the line sufficiently to prevent steam condensation. Numerous small tubing runs connect pressure gauges to points in the extraction and regeneration columns.

Figure 5 shows a view of equipment on level 4. Mr. Mitchell is stringing beads on trace heater lead wires. Air and steam-CO₂ preheaters, the salt

FIGURE 3. Transformer, Switchgear, and Wireway Installation

cooler and (barely visible behind Mitchell) the regenerator are all visible here. Stringing beads proves to be quite a task when it involves thousands of beads.

Insulation sufficient to reduce surface temperatures to no more than 140°F has been installed with the job now 90 percent complete. Figure 6 taken from the same vantage point as that of Figure 1 shows how bulky in

FIGURE 4. Numerous Small Pipe and Tubing Runs Are Required

appearance things become when fully insulated. Figure 7 is from the same vantage point as Figure 4.

In addition to pressing hard with new construction, considerable work has focussed on bringing long idle equipment back into top operating condition. Both large Progressive Equipment Company pot furnaces and the Gas Preheater (General Electric Co.) have had to be carefully brought back-on line. These

FIGURE 5. Equipment on Level 4 Including Air and Steam-CO₂ Preheaters, Salt Cooler, and Regeneration Column. The 4 in. line and 2.5 in. lines also are seen here.

480 volt, 3 phase, AC powered units have to be carefully dried before being fully powered.

The Battelle gasifier has been repaired to correct a few minor leaks, but a major leak at the lock hopper slide valves has posed a serious problem. These 24 in. pneumatically actuated slide valves at the top and bottom of

FIGURE 6. Insulation Now Covers the Trace Heaters
Shown in Figure 1

a 24 in. dia. x 36 in. long spool piece comprise a lock hopper assembly which sits atop the gasifier. This type of valve is really not designed to be leak proof. Unfortunately, the newly designed PDU will operate with a total pressure drop of from 3.5 to 4 psig, at least twice that of the earlier version. Under such conditions, the leak rate at the lock hopper is such as to preclude the possibility even of operating at all.

FIGURE 7. Insulation Changes the Appearance of Figure 4

To ensure that we can operate, a plate of 3/8 in. thick carbon steel and a 1/4 in. thick silicone rubber gasket has been fabricated as a cover for the opening over the top slide valve. This is clamped tightly in place with easily removable clamps. This seals the lock hopper and permits operation. A continuous CO₂ purge of the lock hopper will prevent in leakage of gasifier products through the lower slide valve.

This is an expedient forced by the program schedule. In time, this balky lock hopper problem could be solved by complete redesign and construction. This expedient will permit us to startup by about 10/21/77. Most sub-systems will be operating before then.

Safety considerations are of major concern. At steady-state, the gasifier makes a gas containing 30 percent CO_2 and small amounts of H_2S . We plan occasional additions of supplemental H_2S to obtain a wider range on this variable.

This is an extremely dangerous gas. At 1.0 percent, CO has almost instant knock-down potential as does H_2S . For safety reasons we are installing three H_2S sensors which will detect, indicate, and alarm. In addition, one portable H_2S monitor will be carried by personnel as needs indicate it should be. A stationary CO monitor will also be available with "alarm" features. Consideration is being given procurement of gas masks which give 30 minutes protection in 1.0 percent CO.

With coal, the gasifier will also produce tars and oils. The former are of concern due to their potential carcinogenicity. Some of these may accumulate in the tar trap and equipment has been provided to drain this trap into 55 gal drums which can be sealed and disposed of. The procedure and equipment provide for tar disposal with minimal exposure of personnel.

Safe operating procedures are now in preparation and these will have to be reviewed by the Safety Engineer before this plant can be operated. The writer has had frequent exchanges with Mr. Gordon Halseth, Safety Engineer, to ensure good rapport.

IV. PROPOSED WORK DURING THE NEXT QUARTER

By the end of the next quarter the PDU demonstration runs should have been completed. There may be some dismantling and damage or corrosion assessment work to complete and final reporting. The next quarter's report may well exceed all others together in size---if all goes as planned.

V. OVERALL PROJECT STATUS

PDU completion is anticipated by 10/21/77, but many systems are completed and operating (or operable) now. The experimental portion of this program should be completed by the end of the September-December 1977 quarter.

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