DOE/PC/79672--T5

DE93 011457

TECHNICAL PROGRESS REPORT

MHD SEED RECOVERY/REGENERATION - PHASE II
TRW APPLIED TECHNOLOGY DIVISION

SPONSORED BY: U.S. DEPT. OF ENERGY
PITTSBURGH ENERGY TECHNOLOGY CENTER

CONTRACT NO: DE-AC22-87PC79672

REPORT PERIOD - QUARTER ENDING NOVEMBER 27, 1992

TASK 1 - DESIGN, REFURBISH, OPERATE POTASSIUM FORMATE SYSTEM

Task 1 calls for the design, procurement, construction, and installation of the Seed Regeneration Proof-of-Concept Facility (SRPF) (Figure 1) that will produce tonnage quantities of recyclable potassium formate seed at a design rate of 250 lb/hr for testing in the channel at the CDIF while collecting data that will be used to upgrade the design of a 300 MW_t system. Approximately 12 tons of KCOOH (dry basis) as 70-75 wt% solution have been produced. Activities during this reporting period focused on plant securing operations and waste disposal.

TASK 1.3 PRODUCTION OPERATION OF KCOOH POC UNIT

Production operations were completed during April with the production of more than twelve tons of regenerated seed (dry basis) as 70-75 wt% solution for shipment to the CDIF for use in channel tests.

The long-term plant securing operations which were started in May were completed during this reporting period. Securing operations included both the front end of the plant (potassium sulfate reaction and solids separation/washing units) and the evaporator/crystallizer. In addition, weekly preventative maintenance was performed.

Tank T-309, which is a 16,000 gallon tank used to store dilute potassium formate, was cleaned of all residual solids. All remaining calcium sulfate and calcium carbonate solid waste material generated during plant operations was hauled away by Security Environmental for off-site disposal. A roll-off bin had been provided for the solids. Waste disposal is now complete.

TRW is awaiting word from the CDIF in Montana that it is alright to ship the 41 drums of concentrated potassium formate final product to Montana.

TASK 2 - DESIGN, CONSTRUCT, OPERATE THE CALCIUM FORMATE PRODUCTION POC

The objectives of Task 2 are to design and construct a POC unit (Figure 2) that will produce calcium formate from CO gas and lime slurry in a high pressure reactor system and to operate that POC unit to obtain data that will allow for low risk scaleup to larger sized systems. Since over two-thirds of the presently forecasted cost of seed regeneration is associated with this unit operation and the production of CO gas for reactor feed, verification of the process parameters for this operation is crucial to the MHD program.

TASK 2.1 DESIGN/CONSTRUCT CALCIUM FORMATE POC UNIT

All mechanical buildup activities were completed in the calcium formate POC unit. High pressure leak checks were performed on the unit with both gaseous nitrogen and water. All equipment was functionally checked.

The following documents have been written, reviewed by management and signed off:

- Shakedown Test Plan
- Safety Hazard Analysis Report
- Sampling and Analysis Plan
- Operating Procedures

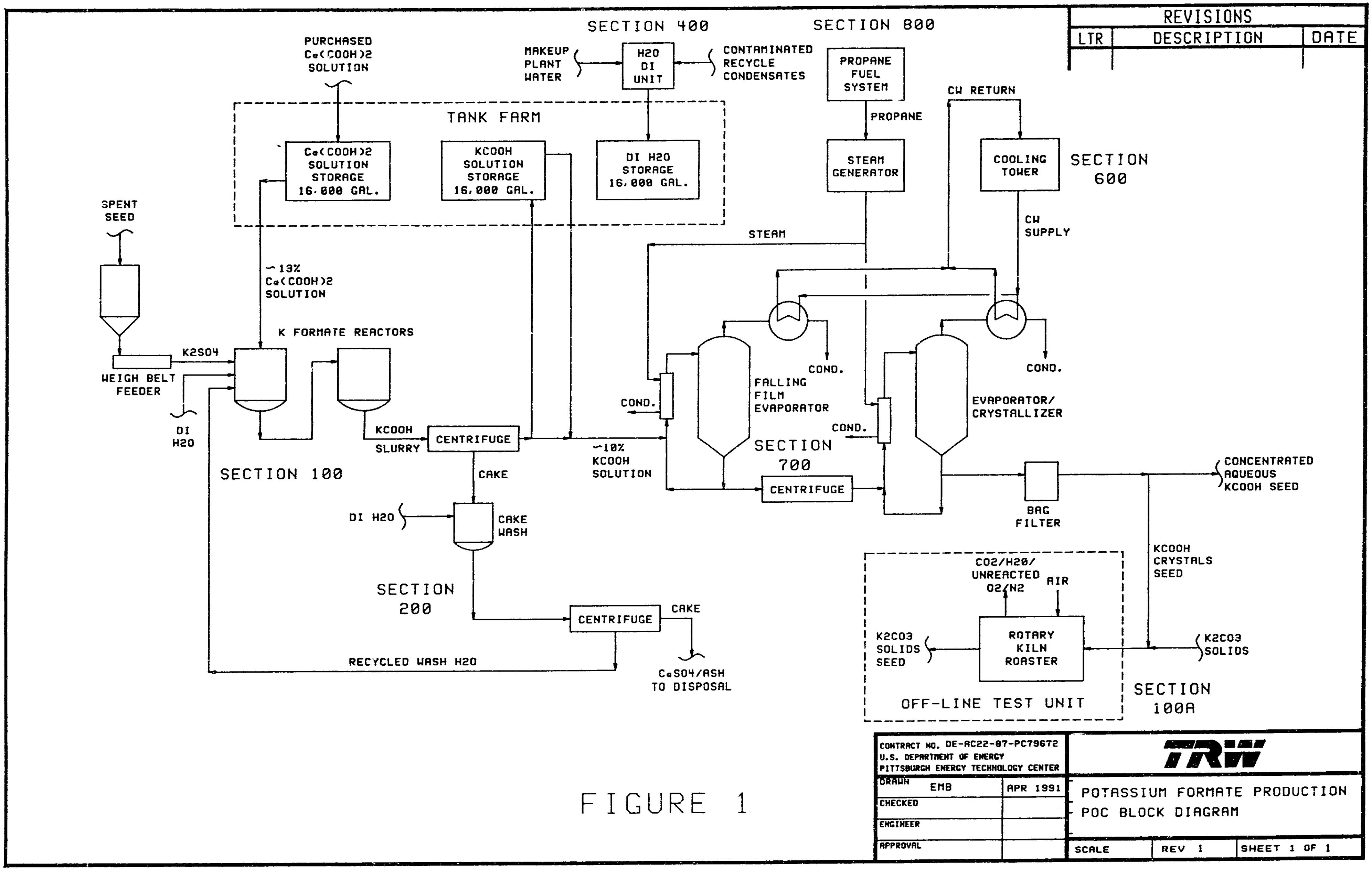

SECTION 400
SECTION 800
PURCHASED Ca(COOH)2 SOLUTION
MAKEUP PLANT WATER
H2O DI UNIT
CONTAMINATED RECYCLE CONDENSATES
PROPANE FUEL SYSTEM
PROPANE
CW RETURN
TANK FARM
Ca(COOH)2 SOLUTION STORAGE 16,000 GAL.
KCOOH SOLUTION STORAGE 16,000 GAL.
DI H2O STORAGE 16,000 GAL.
STEAM GENERATOR
COOLING TOWER
SECTION 600
SPENT SEED
CW SUPPLY
STEAM
~13% Ca(COOH)2 SOLUTION
K FORMATE REACTORS
K2SO4
WEIGH BELT FEEDER
DI H2O
COND.
FALLING FILM EVAPORATOR
COND.
COND.
EVAPORATOR/ CRYSTALLIZER
COND.
KCOOH SLURRY
CENTRIFUGE
SECTION 100
~10% KCOOH SOLUTION
SECTION 700
CAKE
CENTRIFUGE
CONCENTRATED AQUEOUS KCOOH SEED
DI H2O
CAKE WASH
BAG FILTER
KCOOH CRYSTALS SEED
SECTION 200
CO2/H2O/ UNREACTED O2/N2
AIR
CENTRIFUGE
CAKE
K2CO3 SOLIDS SEED
ROTARY KILN ROASTER
K2CO3 SOLIDS
RECYCLED WASH H2O
CaSO4/ASH TO DISPOSAL
SECTION 100A
OFF-LINE TEST UNIT
REVISIONS
LTR
DESCRIPTION
DATE
CONTRACT NO. DE-AC22-87-PC79672
U.S. DEPARTMENT OF ENERGY
PITTSBURGH ENERGY TECHNOLOGY CENTER
DRAWN EMB
APR 1991
CHECKED
ENGINEER
APPROVAL
TRW
POTASSIUM FORMATE PRODUCTION
POC BLOCK DIAGRAM
SCALE
REV 1
SHEET 1 OF 1

FIGURE 1

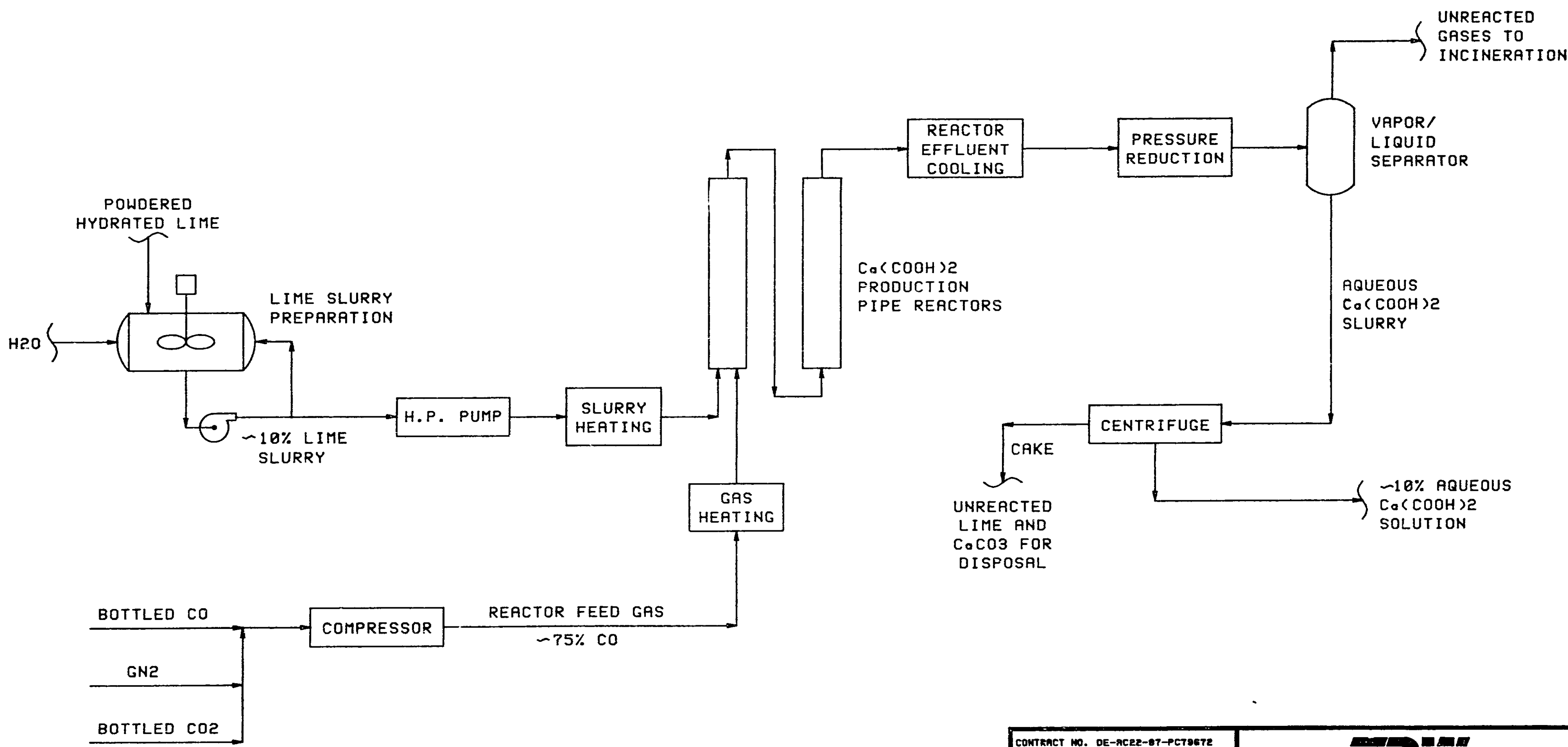


FIGURE 2

REVISIONS		
LTR	DESCRIPTION	DATE

CONTRACT NO. DE-AC22-87-PC79672 U.S. DEPARTMENT OF ENERGY PITTSBURGH ENERGY TECHNOLOGY CENTER		TRW		
DRAWN EMB	APR 1991	CALCIUM FORMATE PRODUCTION POC BLOCK DIAGRAM		
CHECKED				
ENGINEER				
APPROVAL		SCALE	REV	SHEET 1 OF 1

The shakedown test plan is included as Attachment 1.

A Test Readiness Review (TRR) for the calcium formate POC unit was held on October 5. All action items from the TRR have been completed.

TASK 2.2 SHAKEDOWN OF CALCIUM FORMATE POC UNIT

Shakedown operations began immediately after the TRR. Since there were problems with the two pressure letdown valves located downstream of the high pressure calcium formate reactor, a fixed orifice was installed in place of the pressure letdown valves for the first few shakedown tests until the problems with the valves were resolved. One letdown valve had tungsten carbide trim and the other letdown valve had stellite trim. Initially, we were not able to get the tungsten carbide valve to work at all, whereas, the stellite valve worked only marginally well.

The flow characteristic for the tungsten carbide valve is C_v = 0.063:linear and for the stellite valve it is C_v = 0.100:equal percentage. The C_v factor indicates the maximum flow of water which would pass through the valve at a pressure drop of 1 psi. This flow is obtained when the valve is fully open. The flow and pressure drop, and hence C_v, are adjusted by closing the valve. Physically, the valve trim consists of a stem inserted into an orifice (seat). The seat diameters in the valves are 3 mm for stellite trim and 4.5 mm for tungsten carbide trim.

At our test flow conditions, the C_v ranged from 0.02 to 0.03, which meant that the stem was inserted almost completely into the seat (99% closed). The estimated clearance between the stem and seat at this condition was on the order of 100 μm. At high pressure and low velocity, unreacted lime solids no longer remained in suspension and became compacted into particles greater than 100 μm, hence the valves plugged.

It was observed that the valve with the tungsten carbide trim plugged constantly at our flow conditions and the valve with the stellite trim plugged only under particular operating conditions (high pressure > 1400 psig). "Stroking" (fully closing then instantly opening) the valve to buildup pressure in the valve, then suddenly increasing the clearance between the stem and seat in an attempt to dislodge the compacted solids, did not help to unplug the tungsten carbide trim because of the smaller trim size (C_v = 0.063). Valve "stroking" only worked sometimes with the stellite trim, which was larger, thus enlarging the clearance between stem and seat sufficiently to accommodate the compacted lime solids.

We arranged with Kämmer, the valve manufacturer, to provide us with modified stellite trim (with a larger C_v) which would enable the stem to be retracted completely from the valve seat during "stroking" operations. This would provide a larger orifice area for "flushing" out compacted solids.

Until the new stellite trim arrived, we operated the pressure letdown system with fixed orifices or with the existing stellite valve in the manual mode, such that the operator could "head-off" anticipated plugs.

A modified stellite trim set with a larger C_V was received and installed in one of the pressure letdown valves. The valve containing this modified trim set, in fact, performed better than expected; improved "flushing" ability was observed, and more importantly, manual reactor pressure control was improved. Specifically, the operator only had to make one or two valve adjustments during a run to maintain reactor pressure with the new trim, whereas, with the old trim the operator had to constantly adjust the valve position to maintain reactor pressure. Improved performance of the valve may be attributed to the increased clearance between stem and seat, thus preventing solids compaction from occurring.

Improved pressure control of the reactor caused other mechanical problems to surface in the plant. These problems were previously hidden because they were masked by large pressure swings. Large pressure swings caused intermittent flow of the vapor/slurry mixture to T-502 and FE502, the vapor/liquid separator and the vent flowmeter, respectively. With improved pressure control, the rupture disc in T-502 failed more frequently than it had in the past (these failures had been thought to be caused by pressure surges). The rupture disc was originally installed in a Tee-fitting in the feed line to T-502. In this configuration, a continuous flow of slurry past (and in contact with) the disc caused it to erode and corrode (it was aluminum) more frequently than it did before. The rupture disc has since been reinstalled in a different location on T-502, where it does not come in contact with the process slurry.

Good pressure control resulted in a stable, continuous flow of vapor in the vent from T-502. With the steady flow, it was apparent that under certain operating conditions in the plant, the vent flow was out of range of the calibration for the integral flow orifice (in flowmeter FE502) used to measure the vent flow. Specifically, with a high CO content in the feed gas and high CO conversion in the reactor, the vent flow was so low that there was no pressure drop across the orifice, and thus no flow was registered. A smaller orifice has since been installed in FE502 and calibrated. When operating conditions change such that the CO content in the feed is reduced and low CO conversion is achieved, the gas flow rate in the vent will exceed the high end of the range of this orifice. For this case, the orifice will be replaced with one suitable for higher flows.

Other plant improvements include fabrication and installation of a second nitrogen gas manifold to accommodate increased nitrogen flow rates in future test operations. A faulty reactor pressure transducer was repaired by the manufacturer and returned and reinstalled and recalibrated. To aid in material balance calculations and account for water vapor in the gas vented from T-502, a pressure transducer was installed in the vent line from this vessel. The appropriate hardware and software changes were made to the data acquisition and control system to accommodate the altered/new instrumentation. In addition, minor problems with process plant equipment/ instrumentation, control software and the data logging systems were fixed.

A total of six shakedown tests (SD01 through SD06) were conducted during October. The first three tests (SD01, SD02 and SD03) were conducted with pure CO gas feed to the system at reactor residence times varying from six to nine minutes based on slurry feed to the reactor. Reaction temperature was varied between 260^{o}F and 370^{o}F and reaction pressure was varied between

800 and 1400 psia over several subtests. A CO material balance was calculated using measured CO (and N_2) feed rates and measured process vent gas flow rates (corrected for the presence of water vapor). A second CO material balance was calculated using a calculated water flow rate (determined from the metered lime slurry feed flow and the solids analysis of the feed lime slurry) and the measured $Ca(COOH)_2$ concentration in the product. Both methods indicated CO conversions within 15% of each other.

The reaction of carbon monoxide and lime is highly dependent on pressure and temperature. For the test conditions stated above, CO conversion was 26% at 294^{o}F and 1365 psia. At higher temperatures (thought to be 370^{o}F), CO conversions of 62% and 85% are achieved at lower pressures, 818 psia and 1000 psia, respectively.

The next two shakedown tests (SD04 and SD05) were conducted at roughly the same slurry residence time ranges in the reactor; however, the gas feed consisted of a 75% CO/25% N_2 mixture. As expected, CO conversion dropped off due to the reduced partial pressure of CO in the system. Reaction temperatures ranged from 342^{o}F to 369^{o}F, and reaction pressures ranged from 965 psia to 1357 psia. At low pressure (~1000 psia), CO conversion was about 70%. At moderate pressure (~1200 psia), CO conversion increased to approximately 85%, and at high pressure (~1400 psia), CO conversion was in excess of 90%.

The sixth shakedown test (SD06) was conducted at the same temperature, pressure, flow and CO partial pressure conditions as the previous two tests; however, the residence time was cut in half (to four minutes) by bypassing the second reactor. CO conversion dropped to 41%, 53%, and 61% for the low, moderate, and high pressure conditions, respectively.

These results indicate that reactor performance is affected to varying degrees by these variables. To systematically study these effects, a test matrix was generated to study the impacts of the following independent variables on CO conversion in the reactor: 1) reactor feed gas composition, 2) reactor pressure, 3) reactor temperature, 4) reactor residence time, 5) reactant stoichiometry, and 6) lime slurry feed solids loading.

The operating conditions for the eight shakedown tests (SD07 through SD14) conducted during November were culled from this test matrix. The results from these tests are being analyzed and will be reported in the next Technical Progress Report.

Shakedown operations are now complete. Normal test operations have commenced and will continue into the next reporting period.

Diesel fuel for the air compressor was ordered and received. The waste liquid holding tank was pumped out and the contents were hauled away by Security Environmental for off-site disposal.

Normal test operations, plant performance assessment and data analysis (including analysis of shakedown tests SD07 through SD14) will continue during the next reporting period. An on-line, continuous CO Non-Dispersive Infrared Analyzer (NDIR) will be installed (and spanned) in the gas vent from the vapor/liquid separator; this will provide a direct measure of CO conversion during operations, and provide a means of cross-checking the CO material balance. With the reactor pressure transducer now installed and properly functioning, a feedback control loop to maintain reactor pressure automatically will be configured and tuned.

TASK 6 WESTERN SEED STUDIES

Laboratory studies using "simulated" Western seed continued. Spent Western seed is a mixture of potassium carbonate and potassium sulfate plus inerts. Since potassium carbonate is a fresh seed material for the channel, there is no need to regenerate this component. A modification to the current Econoseed process configuration would allow potassium carbonate to be separated from potassium sulfate and inert material via their different solubility characteristics in water. The purpose of these laboratory studies is to determine the amount of water necessary to solubilize only the potassium carbonate component of the seed.

Chemical analysis results from work performed during the previous reporting period indicated inaccuracies in the sampling techniques and experimental procedures. The experimental procedures and sampling techniques were reviewed and revised. The new procedures and techniques were implemented when the laboratory studies resumed in October.

Laboratory scale K_2CO_3/K_2SO_4 separation experiments were performed on simulated spent Western seed prepared from pure reagents only. The spent seed had the following composition (as determined by the latest MHD system model which incorporates a K_2CO_3/K_2SO_4 separation scheme and a different feed coal composition from that used previously):

$K_2CO_3:K_2SO_4$ = 0.59, $Na_2CO_3:Na_2SO_4$ = 0.58, $Na_2SO_4:K_2SO_4$ = 0.007, and 0.38 wt% KCl, all on a weight basis.

Chemical analyses were performed on the solid and liquid phases of the spent seed/water slurry. Material balance and ion closures were within 20%. This is short of the target for closure.

The results of the chemical analyses indicated that further refinements to the revised experimental procedures and sampling techniques were necessary. These refinements were incorporated and used in separation studies performed during November to determine the amount of water necessary to solubilize only the K_2CO_3 component of simulated spent Western seed. Chemical analyses and material balances are currently being performed on the solid and liquid phases of the slurry. The results will be reported in the next Technical Progress Report.

ATTACHMENT 1

SHAKEDOWN TEST PLAN FOR THE CALCIUM FORMATE PRODUCTION UNIT OPERATION

TRW Space & Defense Sector
One Space Park
Redondo Beach, CA 90278

CAGE CODE NO. 11982

TITLE

MHD SEED RECOVERY AND REGENERATION POTASSIUM FORMATE PRODUCTION POC PLANT SHAKEDOWN TEST PLANT FOR THE CALCIUM FORMATE PRODUCTION UNIT OPERATION

DOCUMENT DATE 11 SEPT 1992

NO. K319.2.92-155

REV.

SUPERSEDING:

PREPARED BY: H. Thridandam/E.M.Barrish 9/22/92 DATE

APPROVAL SIGNATURES:

R.A. Meyers DATE Program Manager	J.P. Mix, Manager 10-5-92 DATE CTS Safety & Fire Protection
W.B. Battaile, Manager 9/15/92 DATE Capistrano Chemical Facility	O.W. Clausen, Manager 10/5/92 DATE Capistrano Test Site
J.W. Scott, Manager 9/23/91 DATE Chemistry Technology Dept.	L.C. McClanathan 9/21/92 DATE Assistant Program Manager

ORIGINAL CADM RELEASE: ______ DATE

1.0 INTRODUCTION

This test plan defines the objectives, requirements, and schedule for shakedown of the Calcium Formate Production unit (Section 500) of the Econoseed MHD Seed Regeneration Proof of Concept Facility (SRPF) located at TRW's Capistrano Chemical Facility (CCF). The test program is being conducted for the Department of Energy (DOE), Pittsburgh Energy Technology Center under contract number DE-AC22-87-PC79672.

Aqueous calcium formate [$Ca(COOH)_2$] is used in the Econoseed process to dissolve and recover the potassium content of spent MHD seed material (K_2SO_4 plus mineral matter) as aqueous potassium formate (KCOOH) while precipitating and rejecting its sulfur content as gypsum ($CaSO_4$). Calcium formate for the POC operations conducted at CTS is purchased as a high priced, specialty chemical. On a commercial scale, however, it is critical for the economics of the Econoseed process (and to the MHD system as a whole), that calcium formate be produced at a low cost. "In-house" studies have indicated that calcium formate has the potential to be produced from carbon monoxide and lime at a low cost. A complete description of the Calcium Formate Production POC unit that was built to test the feasibility of commercializing this new process is given in the referenced (Appendix A) Final Design Package for this unit operation.

This document presents the shakedown plan for the Calcium Formate Production unit. It discusses the hardware and instrumentation utilized, the sampling, analysis and data collection requirements, the consumable materials and the safety precautions to be followed during operation of this unit. Finally, the shakedown tests for this unit are described.

2.0 OBJECTIVES

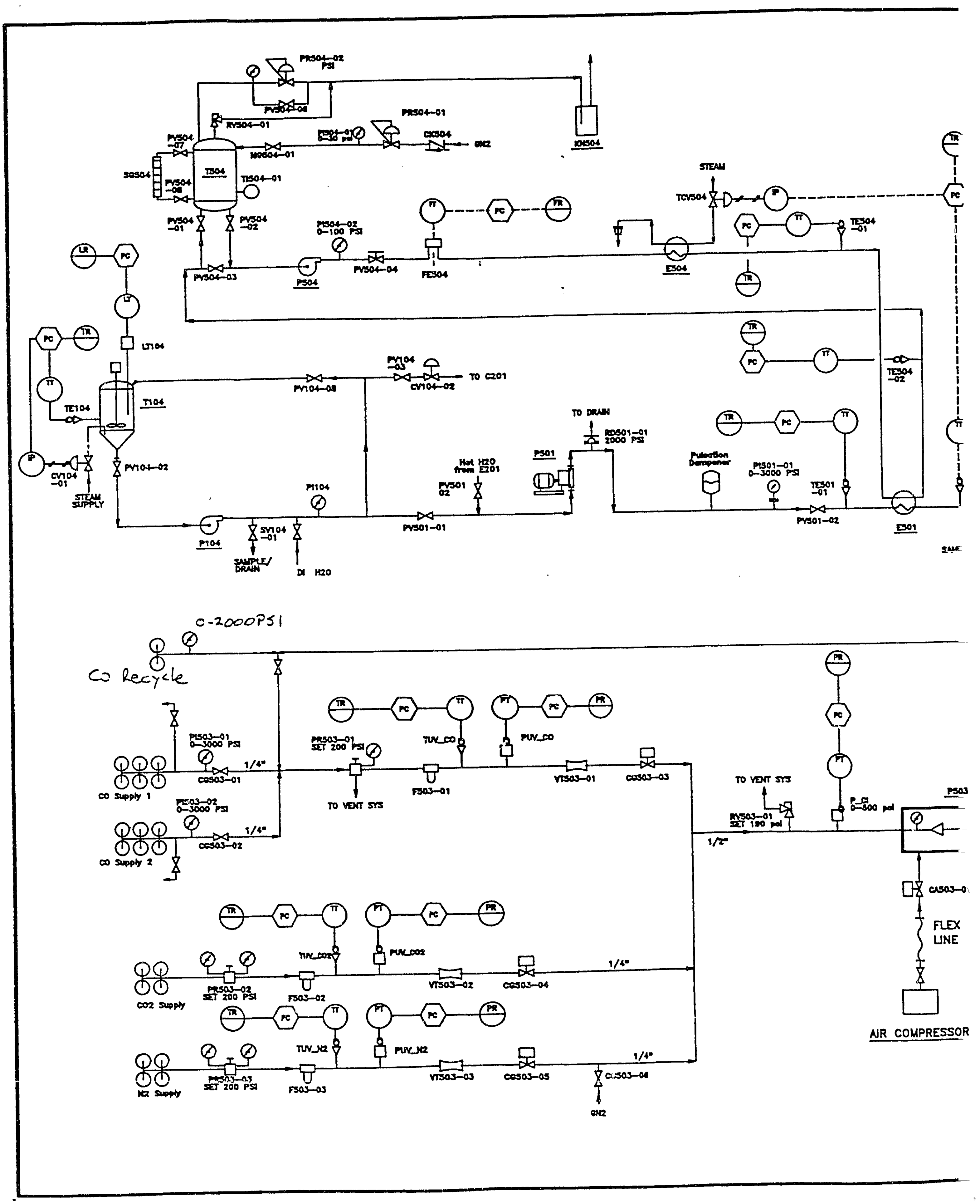
The objective in operating the Calcium Formate Production unit is to demonstrate the feasibility of producing 20 to 50 pounds per hour of calcium formate from carbon monoxide and lime. This unit will be operated on an integrated basis to provide steady state operational information on small scale versions of commercial equipment and data on process parameters such as reaction rates, reactant conversions, and liquid/solid separation rates. The data base generated will provide the necessary information for scale-up to the size calcium formate production unit required to support a 300 MW_t Econoseed MHD SRPF plant design.

During shakedown, the Calcium Formate Production unit will be operated with process feeds - hydrated lime powder, deionized water, and a mixture of carbon monoxide, nitrogen, and carbon dioxide gases - at the expected reaction temperature and pressure for the first time. One objective of these shakedown tests is to demonstrate the capability of this unit to elevate slurry to high pressure, compress gas, heat and transport reactor feeds, react the feeds at the expected process conditions, and to separate and collect products. A second objective of these shakedown tests is to verify and validate the planned sampling and analysis techniques used to assess the performance of the Calcium Formate Production unit.

3.0 TEST HARDWARE CONFIGURATION

The Calcium Formate Production unit Reactor Feed System is depicted in Figure 1. This system is used to prepare the reactor feed streams and elevate them to operating pressure and temperature. Lime slurry feed is prepared in an off-line batch operation; in a continuously stirred tank (sized to support at least four hours of POC testing), hydrated lime powder is added to and is mixed with deionized water heated with steam in a shell and tube exchanger. A circulation loop on this tank maintains the lime solids in suspension. During reactor operation, lime slurry feed is continuously pumped by the tank circulation pump to a metering pump which then, elevates the slurry to reaction pressure. Pressurized slurry is heated to reaction temperature in a countercurrent, coiled heat exchanger with Syltherm heat transfer fluid on the shell side (Syltherm is previously heated to the required temperature in a steam heated shell and tube exchanger). Bottled carbon monoxide, carbon dioxide, and nitrogen gases are combined to form a feed gas mixture. The feed gas mixture is continuously compressed to reaction pressure by a gas booster. Pressurized feed gas is heated to reaction temperature in a steam heated double pipe exchanger.

The pressurized, preheated, feed streams are continuously fed to the Calcium Formate Production unit Reaction/Pressure Letdown/Product Separation/Centrifugation System depicted in Figure 1. These streams are contacted at elevated temperature and pressure in a two-stage pipe reactor. Each stage contains tower packing to ensure intimate contact between the reacting lime slurry and carbon monoxide gas streams. A steam jacket maintains the reactor stages isothermal. Pressurized reactor effluent is cooled in a coiled heat exchanger with cooling water on the shell side and is then reduced to ambient pressure across control valves. The reactor effluent mixture is separated in a knock-out tank. Gas from the tank is incinerated to destroy unreacted carbon monoxide. Liquid from the tank is pumped to a storage tank. In an off-line operation, the storage tank contents are centrifuged to remove unreacted lime solids and any calcium carbonate solids formed in the reaction. The clarified product, aqueous calcium formate (centrate), is collected and stored in drums. The centrifuge cake is collected and stored in drums.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MG504-01
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P504
PV504-04
FE504
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TCV504
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P104
SV104 -01
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DI H20
PV501 02
Hot H20 from E201
PV501-01
TO DRAIN
RD501-01 2000 PSI
P501
Pulsation Dampener
PI501-01 0-3000 PSI
TE501 -01
PV501-02
E501
TE504 -02
0-2000PSI
CO Recycle
PI503-01 0-3000 PSI
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CG503-01
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PUV_CO
VT503-01
CG503-03
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CG503-02
TO VENT SYS
RV503-01 SET 190 psi
P_CI 0-500 psi
1/2"
P503
CA503-0
FLEX LINE
AIR COMPRESSOR
CO2 Supply
PR503-02 SET 200 PSI
F503-02
TUV_CO2
PUV_CO2
VT503-02
CG503-04
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F503-03
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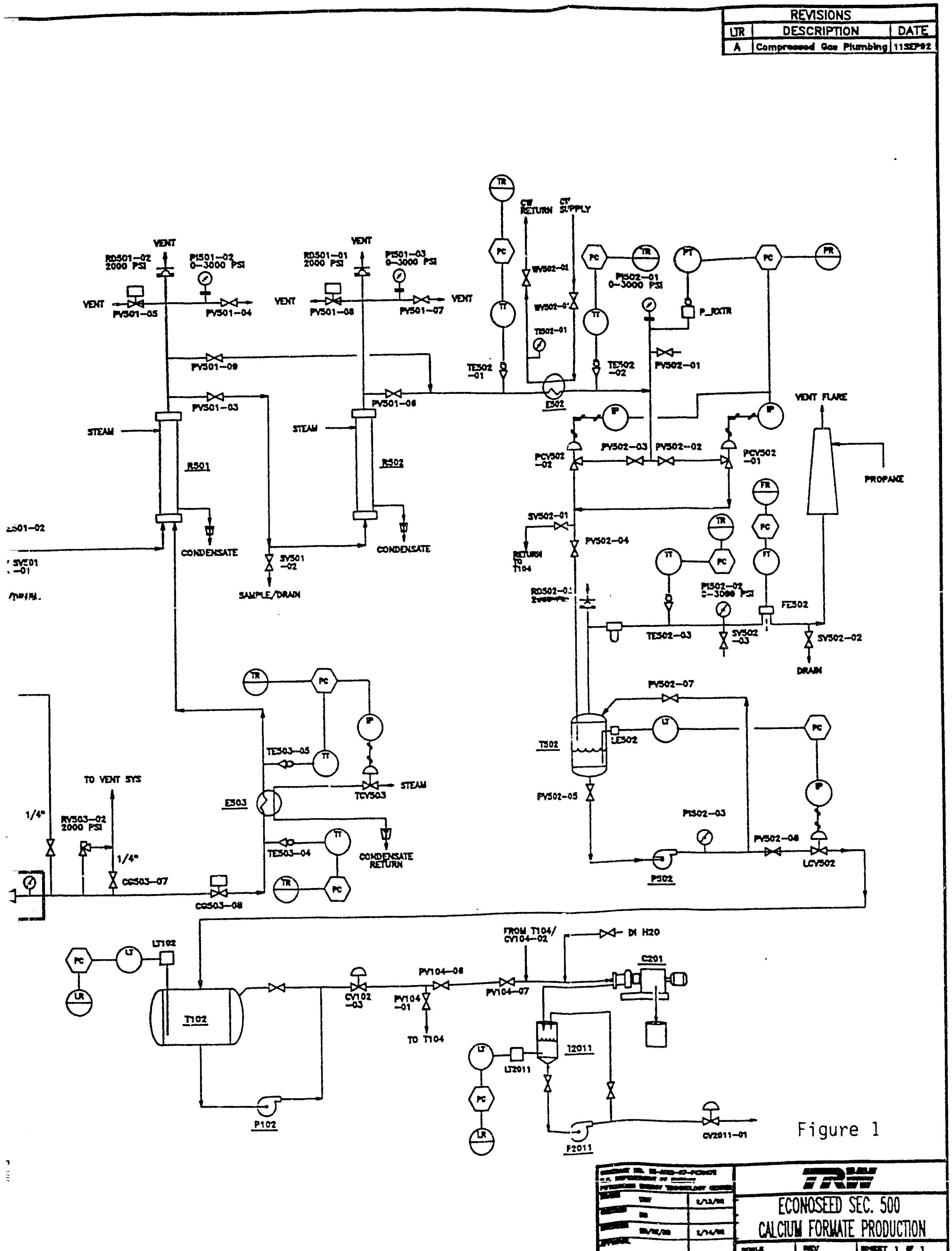
PUV_N2
VT503-03
CG503-05
CU503-08
GN2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CW RETURN
CW SUPPLY
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VENT FLARE
PROPANE
PCV502 -02
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PV502-02
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TCV503
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TO VENT SYS
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CG503-07
CG503-08
T102
P102
LT102
CV102 -03
PV104-06
PV104-07
PV104 -01
TO T104
FROM T104/ CV104-02
DI H2O
C201
T2011
LT2011
F2011
CV2011-01
ECONOSEED SEC. 500
CALCIUM FORMATE PRODUCTION

Figure 1

4.0 REQUIREMENTS

This section summarizes requirements detailed in the referenced (Appendix A) POC plant Engineering Requirements Document.

The Calcium Formate Production unit is required, as a minimum, to demonstrate the ability to:

- Produce 20 to 50 pounds per hour of dilute (10 - 15 weight %) calcium formate aqueous solution from carbon monoxide gas and lime slurry.
- Provide for the measurement of process conditions (flow, pressure, temperature), and for the sampling of all feed, product and process streams in order to assess plant performance.
- Segregate, store, remove, and dispose of all waste material (solid or liquid) in a lawful and safe manner.

4.1 CONSUMABLE MATERIALS

The consumable materials required for shakedown testing are listed in Table 1 (along with an estimate of the maximum usage).

4.2 INSTRUMENTATION/CONTROLS/DATA ACQUISITION

The data acquisition/process control system which interfaces with the plant instruments is required to perform the following tasks:

- measure process and operating condition data for this unit operation and record this information on magnetic media.
- continuously control and monitor operation of the calcium formate production unit over a wide range of operating conditions.

The data acquisition/process control system for this unit operation has the following on-line capabilities:

- Instant operator access to all information necessary to operate and troubleshoot process subsystems including; plant overview displays, group displays, individual control loop displays, trend displays, and alarm displays.
- Operator control, either remotely or locally of the following functions: control loop set point adjustments, alarm set point adjustment, mode switching, manual output adjustment.

The data acquisition/process control system provdes for automated data acquisition and allows for safe, reliable operation of this unit operation. When warranted, the control system allows for shut down of the remote high pressure diaphragm pump and gas compressor as well as remote venting of each reactor.

4.3 SAMPLING AND ANALYSIS REQUIREMENTS

The Calcium Formate Production unit process has provisions for taking representative samples of lime slurry feed, carbon monoxide gas feed mixture, calcium formate product slurry (prior to centrifugation), product gases (prior to incineration), centrifuge cake, and calcium formate product solution for chemical analysis. Feed, product liquids, and centrifuge cake shall be analyzed for calcium, formate, and carbonate content. The moisture content of centrifuge cake will also be analyzed.

4.4 SPECIAL TECHNICAL AND ENGINEERING SUPPORT REQUIREMENTS

In addition to the normal operating crew, chemical facility technicians and engineers will be called upon (as needed) to support off-line analysis of process operating data and to perform chemical analyses on the various solid, liquid, and gas streams sampled during operation.

TABLE 1. CONSUMABLE MATERIALS, SHAKEDOWN ECONOSEED MHD SEED RECOVERY AND REGENERATION POTASSIUMM FORMATE PRODUCTION POC PLANT CALCIUM FORMATE PRODUCTION UNIT

ITEM	GRADE/SPECIFICATION	MAXIMUM* QUANTITY REQUIRED FOR SHAKEDOWN
Carbon Monoxide	Commercial, gas	32,928 SCF
Carbon Dioxide	Commercial, gas	1,344 SCF
Nitrogen	Commercial, gas	9,696 SCF
Hydrated Lime	Commercial, powder	3,552 pounds
Propane Fuel	Commercial, fuel grade liquid	1,056 gallons
Process Water	Deionized water prepared at CTS from site water	21,600 gallons
Cooling Water	Recirculated, deionized water prepared at CTS from site water	92,160 gallons (total circulation)
Steam	Generated from deionized water prepared at CTS from site water	36,624 pounds

* Basis: 50 lb/hr calcium formate production over 96 hours total operation.

4.5 DATA ANALYSIS REQUIREMENTS

All process operating data and process sample data collected per Sections 4.2 and 4.3, above, shall be summarized in a data base which will include physical and chemical characteristics of the process samples as well as plant equipment, piping and instrument wear and performance data. Raw data shall be recorded on magnetic media, in plant operating and maintenance logs, and on space provided in Engineering Test Directives.

The data base shall include, but not be limited to, the following:

- Heat and material balances
- Waste stream composition
- Carbon monoxide and lime conversion efficiencies as a function of the key process variables.

4.6 QUALITY ASSURANCE REQUIREMENTS

This program has no requirements for quality assurance verifications.

4.7 SPECIAL HEALTH/SAFETY REQUIREMENTS

All of the process chemicals utilized are commonly used in industry, as are the types of operations and the hardware to be used. All process chemicals must be used with caution; especially the carbon monoxide. All operating personnel are required to be familiar with the physical properties and health hazards associated with carbon monoxide. The plant was designed and constructed to capture all automatic and manual vents of carbon monoxide into a common vent line heading to a flare for the destruction of carbon monoxide. The flare is required to be operational before the carbon monoxide cylinder isolation valves are opened. Personnel when working in the plant will carry portable gas detectors for carbon monoxide.

Handling and storage requirements for all chemicals (including waste streams) are listed in Table 2. See the Safety Hazard Analysis Report (K319.2.92-xxx) for a complete description of chemical handling and storage requirements and for all Material Safety Data Sheets (MSDS).

4.8 HAZARD COMMUNICATION/ENVIRONMENTAL AFFAIRS

With the exception of carbon monoxide, none of the chemical feeds, products, or in-process streams for the calcium formate POC unit under discussion are considered to be hazardous materials. The hazards associated with carbon monoxide (and any other operational hazards) are discussed in the referenced (Appendix A) Safety Hazard Analysis Report.

The Calcium Formate Production unit will be operated in a manner compliant with local, state, and Federal environmental regulations.

5.0 TEST PROCEDURE REQUIREMENTS

5.1 OPERATING PROCEDURES

The Calcium Formate Production unit utilizes the following unit operations of the Econoseed MHD SRPF:

- Reactor Feed System (Section 500)
- Reaction/Pressure Letdown/Product Separation/ Centrifugation System (Section 500)
- Potassium Formate Reaction (Section 100)*
- Potassium Formate Separation/Washing (Section 200)**

  * Section 100 equipment is used to prepare lime slurry feed.

  ** Section 200 equipment is used to separate unreacted lime solids and solid reaction byproducts from the product formate solution.

- Support Utilities:
  - DI Water System (Section 400)
  - Cooling Water System (Section 600)
  - Steam and Condensate System (Section 800)

Operating procedures have been written for these unit operations. Personnel conducting the shakedown tests must have a working knowledge of the components and function of each of the unit operations within the overall Econoseed MHD SRPF Production POC plant, have read and be familiar with all plant operating procedures, with the Safety Hazard Analysis Report, and all other pertinent documents (see list of references, Appendix A).

5.2 TEST SCHEDULE

Shakedown of the Calcium Formate Production unit is expected to last for approximately 24 days of 8-hour (single shift) work days. The operating crew will consist of ≈ 3 men (a lead man, 2 technicians, and a part time steam plant operator). Chemical and data analysis will be performed off-line by the appropriate personnel.

5.3 TEST CONDITIONS

The shakedown tests will be performed with feeds consisting of aqueous hydrated lime slurry and a carbon monoxide gas mixture. The tests will be conducted in the composition, flow, pressure, temperature and residence time regimes specified in the POC unit Engineering Requirements Document (summarized below), and will be described in detail in the Engineering Test Directives.

Range of operating parameters for the Calcium Formate Production unit:

- Gas flow/feed rate: 185 - 460 SCFH
- Lime slurry flow/feed rate: 215 - 535 lb/hr
- Feed/operating pressure: 500 - 1500 psig
- Feed/operating temperature: 140 - 170 °C
- Reactor residence time: 5 min. max.
- Lime content of slurry: 7 - 10 wt. %
- Feed gas composition: 75 - 90 mol. % CO; 10 - 25 mol. % N_2; 1 - 3 mol. % CO_2

6.0 SHAKEDOWN TESTS

6.1 PRE-SHAKEDOWN TESTS

Prior to the start of shakedown testing, a checkout will be conducted to confirm that the processing equipment has been installed properly. A Test Readiness Review (TRR) will confirm that all necessary operating procedures have been prepared and approved, and that all safety precautions necessary to protect personnel and equipment have been implemented.

During the equipment checkout preceding the start of shakedown testing of the calcium formate production unit, the reactors will have been checked for hydrostatic water tightness. Deionized water and nitrogen gas will have been continuously metered into the reactors and then processed through the pressure letdown and separation systems. This will allow the system equipment and equipment interconnect plumbing to be checked for leaks, and will demonstrate the operability of the circulation loops and pumps, the centrifuge, the high pressure liquid feed metering pump, the feed gas compressor, and the pressure, level, and temperature control valves and intrumentation. A water heating test will be conducted on the lime slurry heater, and a water cooling test will be conducted on the reactor effluent cooler during this check out. These tests will provide information on baseline heat transfer coefficients for clean tube surfaces.

6.2 SHAKEDOWN TESTS

The shakedown tests will be conducted under the range of test conditions specified in Section 5.3, above. The duration of each test will be 4 to 6 hours. A 200 gallon batch of lime slurry must be prepared before the start of each test. A Test Directive will be provided for each shakedown test. The directives will give the specific operating conditions to be used in a particular test. Process and waste streams will be sampled in certain quantities at intervals and locations to be specified in the Test Directive.

6.3 SHAKEDOWN TEST EVALUATION

During process operations, operators will monitor and assess the performance and reliability of process equipment, instruments, and controls in calcium formate service. These observations and assessments will be recorded in the operations log.

At the conclusion of a test, material balances will be performed to verify sampling and chemical analysis procedures. These balances will also be used to determine whether operating data acquired during the test is sufficient and adequate to assess the capability of the calcium formate production process to meet the design and test requirements. Any deficiences in process data, sampling, and analysis methods will be noted and corrected.

The operating log for each test will be reviewed to determine whether the information presented in the Test Directives and on the data acquisition/process control computer display screens is sufficient to operate and troubleshoot the process. Any deficiencies noted will be corrected.

APPENDIX A

REFERENCE DOCUMENTS

- Final Design Package, Econoseed MHD Seed Regeneration POC Plant, Calcium Formate Production Unit, 5 April 1991, No. K319.2.91-060

- Engineering Requirements Document for the Calcium Formate Production POC Plant, April, 1991, No. K319.3.90-040, Rev. A.

- Operating Procedures for MHD Seed Recovery and Regeneration POC plant:

  - Dilute Potassium Formate Production, Procedure No. YS-28P-51.

  - Concentrated Potassium Formate Production, Procedure No. YS-28P-52.

  - Steam Generation System, Procedure No. YS-28P-53.

  - Data Acquisition and Control, Procedure No. YS-28P-54.

  - Calcium Formate POC Unit, Procedure No. YS-28P-XX.

- Safety Hazard Analysis Report, K319.2.92-156.

- Sampling and Analysis Plan, K319.2.92-xxx.

END

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