

EXPERIENCE IN FEEDING COAL INTO A
LIQUEFACTION PROCESS
DEVELOPMENT UNIT

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ABSTRACT

Thirty years of experience in coal liquefaction at PERC provides considerable background technology on feeding coal into pressurized units. Performance of the preferred feed system as now used serves as a basis to compare new developments. Flowable coal-oil slurry is fed by positive displacement plunger pumps into high-pressure liquefaction units. Records of over 600 hours of continuous unit operations without repairs to the feed system attest to its reliability. SYNTHOIL process development operations with slurries of various concentrations, coal, and recycle vehicle oils will be reviewed. Methods for minimizing settling, plugging and erosion will be discussed.

"Experience in Feeding Coal into a Liquefaction Process Development Unit"

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INTRODUCTION

A system for preparing coal slurry and feeding it into a high-pressure liquefaction plant has been in use at the Pittsburgh Energy Research Center (PERC) for over thirty years. The system was developed for the liquefaction units operated at the Center during the 1940's to provide supporting research and development for the Bureau of Mines coal liquefaction pilot plant (60 TPD) at Louisiana, Missouri. Although operation of the Missouri pilot plant was discontinued in 1953, research and development on coal liquefaction has continued almost uninterrupted at PERC and the coal feeding system is still essentially the same with minor materials modifications. The experimental units currently in use for research on production of low-sulfur, low-ash utility fuel oils by SYNTHOIL and other processes are equipped with this coal feeding system. It is the purpose of this presentation to describe the system which has been used for long continuous runs and to review the experience of operating it.

DESCRIPTION OF THE SYSTEM

The system for preparing coal slurry and pumping it into a high-pressure liquefaction plant is shown in figure 1. It is based on positive displacement pumping of coal slurried in a recycle vehicle oil. It consists of three parts: A slurry mix tank where the coal slurry is prepared, a slurry feed tank that maintains the suspension of slurry during feeding, and a slurry feed pump which pressurizes the slurry into the pressurized vessels of the plant. Of paramount importance is the circuit used to keep slurry moving closely by the intake valves of the high-pressure pump to prevent any solids settling in transfer lines to the pump. The slurry mix tank, shown to the left in the figure, is mounted on a scale and is equipped with an agitator and a circulating pump. The tank is connected to an inert gas supply so that the slurry preparation may be conducted under an inert gas blanket. All connections to the tank have flexible hose couplings to enable accurate transmission of weight changes to the scale.

The circulating pump is a commercial* progressing cavity Moyno pump¹. The convoluted rotor and stator are made of tool steel and the rotor has a

*Reference to commercial items does not constitute endorsement by the U.S. Government but is for identification purposes only.

chrome finish. Neither part is plastic lined; there is metal-metal contact. The capacity of the pump is 3gpm which gives a velocity of 1.2 ft/sec to the slurry in the circulation loop of 1-inch ID pipe. This is adequate pipeline velocity to assure suspension of the minus 100-mesh coal in the oil.

The slurry feed tank, shown to the right of the slurry mix tank, is also mounted on a scale and is equipped with an agitator and a circulating Mcyno pump. Like the mix tank, the feed tank also is connected to an inert gas supply and has flexible hose endings for all connections.

The slurry feed pump, shown to the right in figure 1, is a modified Milton Roy plunger-type metering pump². The modification of the head, along with other details of the pump assembly, is shown in figure 2. The pump is a duplex unit with a common drive. The plunger is attached to the crosshead by a floating connection which permits lateral movement and assures alignment of the plunger with the axis of the stuffing box. A metal gland and a lantern ring provide close guidance of the plunger to prevent excessive wear of the packing. The plunger is made of low-carbon steel and is plated with chromium, 0.002-inch thick. Both the stuffing box and the plunger have surface finishes of 60 μ -inch or better. The plunger withstands slurry wear very well as seen by the good condition of the one photographed in figure 3 after 6000 hours of use.

The packing is chevron or "V" type, self sealing, and fabricated from teflon. Lubrication of the packing is essential to reduce friction. Grease lubricant is injected with a grease gun at least once every day.

The suction and discharge valve assemblies are our own modifications of the pump. The ball valves were designed at PERC. The ball and seat, assembled as a unit, are screwed into the pumphead and seated against a gasket. The arrangement permits easy replacement of the valve, when necessary. The ball is made of a hardened chromium steel and the seat of stainless steel 410, hardened to about 350 Brinell. The pump is provided with pressure relief valves, one directly in the pumphead on the discharge side and one in the piping on the suction side. The relief valve on the suction side is necessary to protect the low-pressure piping from over pressurization if the ball valves on both the suction and discharging sides become faulty.

The pump is provided with a gas vent in the cylinder body to bleed out air when priming the pump. The same vent is also used to bleed out organic vapors or steam if the pump is vapor locked by volatiles from the vehicle oil or moisture from coal.

The capacity of the pump has been varied from 2 lb/hr to 70 lb/hr by using one or both sides of the pump, interchanging plungers of 1/4-inch to 5/8-inch diameter, and adjusting the length of the stroke from 1/2-inch to 4-inches. As an additional means of varying the capacity, the pump is equipped with a variable speed drive but it has been used at only one speed, corresponding to a frequency of 28 strokes/minute.

The piping for the feed system and the safety features in it are shown in figure 1. The high-pressure line from the feed pump to the plant is provided with a reverse flow check valve to prevent the plant pressure from blowing back if the ball valve on the outlet of the pump fails to

seat properly. The high-pressure line is also provided with a pressure sensitive cut-off switch to interrupt supply of power to the feed pump if the line, or any point downstream of it, develops a constriction. A manually operated oil pump, shown on the extreme right in figure 1, is then used to hydraulically break open the constriction by applying pressures up to 10,000 psig. To minimize the risk of constriction developing in the slurry line due to settling of coal, horizontal piping is avoided and the length of the line is kept as short as practicable. To facilitate preparation and feeding of very viscous slurries, the system can be steam heated. The mix and feed tanks are steam-jacketed and all pipings involved in slurry transfer are steam-traced. The head assembly of the feed pump is provided with 1/4-inch holes for inlet and outlet of steam.

OPERATION OF THE FEED SYSTEM

The vehicle oil is transferred to the mix tank and the agitator and circulating pump started. Pulverized coal is added, manually in small quantities through the open top while the tank is blanketed with inert gas (nitrogen) by maintaining an outward flow of the gas. After the addition of coal is complete, the tank is covered and pressurized with 5 psig of the inert gas. Agitation and circulation of the slurry is continued for 2 hours to ensure thorough mixing, after which the slurry is transferred to the feed tank by diverting the circulation stream to the tank. Agitation and circulation of the slurry is continued non-stop in the feed tank also under 5 psig of the inert gas. The slurry is supplied to the feed pump from the circulation loop as shown in figure 1. The slurry is supplied at about 6 psig or higher to ensure an adequate flow of the slurry to the suction side of the feed pump. A gauge in the circulation loop measures the pressure of the circulating slurry. A pressure of less than 6 psig on the gauge indicates that the rotor and stator in the circulating pump are eroded and should be replaced. The erosion rates of the rotor and stator depend on the type and amount of mineral matter in coal. With a West Virginia coal containing 3 percent ash, the rotor and stator had a service life of 4,000 hours while with a Kentucky coal containing 16 percent ash, the service life was reduced to 1600 - 1800 hours.

This system has been used with a wide variety of coals and vehicle oils: coals ranging in rank from low-volatile bituminous to lignite and vehicle oils from anthracene oils and coke oven tars to process-derived recycle oils. The coal is dried and pulverized to 70 percent thru 200-mesh, U. S. standard sieve, and 99 percent thru 100-mesh. Immediately before use, the pulverized coal is screened through a 50-mesh sieve to reject particles retained by this sieve and to break up agglomerated clusters of pulverized coal.

Drying of coal is of no fundamental significance to liquefaction per se. However, wet coals are difficult to grind, and pulverized coal containing more than 10 percent moisture generates foam when added to recycle oil at 200 ° F or more. The nuisance is most pronounced with subbituminous coals and lignites, which usually retain about 20 percent moisture after conventional drying and grinding. If foaming is excessive, a good portion of the slurry in the mix tank can spill over to the floor. Excessive foaming is controlled by adding the coal slowly and in small quantities to the recycle oil. It is important to add the coal in small quantities for another reason also. If added too rapidly, the coal particles agglomerate into internally dry lumps

which do not break up easily. These lumps usually float on the oil but occasionally are sucked into the circulating loop and plug up the valve at the bottom of the tank or freeze the rotor of the circulation pump. A suitable rate for adding coal to oil without foaming or lumping must be determined empirically since the wetting characteristics of coals and oils differ. Frequently, some pulverized coal agglomerates stick to the walls of the tank. As a routine practice, therefore, when the addition of coal is complete, the walls are scraped to break up the coal agglomerates and the scrapings added to the slurry.

The slurry must be homogenized thoroughly in the mix tank before its transfer to the feed tank. An improperly mixed slurry can easily cause malfunction of the ball valves on the feed pump. Two hours of mixing time is liberal allowance to ensure thorough mixing, although, with many coal-oil combinations, a far shorter duration may be sufficient.

The slurry preparation and feeding are conducted in an inert atmosphere. This precaution is desirable since the viscosity of coal-derived oils, and therefore of slurries of coals in such oils, increases by exposure to air. An investigation³ of the aging characteristics of product oils from the SYNTHOIL process has shown that the rate of increase of viscosity under different gases varies in the order nitrogen < air < oxygen, and at different temperatures in the order 86° F < 113° F < 141° F. Furthermore, the higher the initial viscosity of the oil, the larger the rate of increase of its viscosity. Since slurry preparation and feeding are frequently conducted at about 200° F and the initial viscosity of the slurry is high, the rate of increase of the viscosity will be substantial if the slurry is prepared and fed exposed to air. It may also be noted that the agitation and circulation of the slurry in the mix and feed tanks are extremely vigorous, and if slurry preparation and feeding were conducted in air, the air-slurry contact would be far more intimate than under the experimental conditions of the aging study. Consequently, the rate of increase of the viscosity of the slurry would be larger than the results of the aging study might suggest.

CONCENTRATION OF COAL IN FEED SLURRY

Maximization of coal concentration in the slurry is a desirable objective since the higher the concentration of coal, the higher the plant throughput for a given slurry feed rate. However, the slurry must be pumpable, a quality determined by its viscosity and stability. If the viscosity is too high, the slurry cannot be circulated or introduced into the feed pump, while if the viscosity is too low, coal will segregate from the slurry and deposit in the inlet and outlet valves of the feed pump and/or downstream of the pump in the high-pressure line to the plant, before hydrogen meets the slurry and coal starts dissolving. Any of these occurrences will disrupt the feeding system.

The viscosity and stability of a coal slurry depend on the nature of the vehicle oil and the concentration of coal in the slurry. Although all coal liquefaction processes are based on process-derived oils, the latter are not all identical. The vehicle oils in some processes are distillate fractions of the liquefaction product, while in others, the distillation bottoms or the whole product oil itself. The vehicle oils in various coal liquefaction processes, their viscosities and the concentration of

coal in pumpable slurries are given in table 1. The vehicle oils consisting of distillate materials are low-viscosity liquids that give pumpable slurries with up to 33 percent coal. At higher coal concentrations, the slurries are too unstable although viscosity is no problem. The vehicle oils consisting of heavy oils, or whole product oil, are viscous liquids containing asphaltenes and some benzene insolubles. These liquids give pumpable slurries with up to 45 to 50 percent coal. At higher concentrations of coal the slurries are excessively viscous. Indeed, if the viscosity of the vehicle oil is more than 100 SSF (at 130° F) and slurries containing more than 35 percent coal are to be prepared, the slurry preparation and feeding system must be heated. The practical upper limit of temperature for our feeding system is about 230° F, at which volatilization of the low-boiling hydrocarbons in the vehicle oil causes vapor-locking of the feed pump.

OPERATING EXPERIENCE

The experience of operating the coal slurry preparation and feeding system with the 1/2-TPD SYNTHOIL unit^{4, 5, 6} over the past three and a half years is summarized in table 2. Every run made with this unit is listed. The pump was always newly overhauled prior to each run. (See footnote to table 2 for details of overhauling). During this history of operation, slurries of five different coals in indigenous recycle oil, a solution of SRC in SRC solvent, and the SYNTHOIL product oil without any coal were processed through the SYNTHOIL unit in the 26 separate runs of table 2. Seven of these runs were of 500 to 700 hours duration, ten of 200 to 425 hours, and nine of 8 to 169 hours. None of these runs was terminated because of feed pump failure. One side of the duplex pump was always ready to run whenever the other side needed overhaul.

The pumping time with each head during the runs is tabulated in column 6 of table 2. When one side of the duplex pump falters as observed by any decrease of feed rate, the other side is put into service and the defective side is overhauled by changing the pumphead ball valves and packing assembly (see figure 2) and cleaning the inlet manifold with light oil. The overhauled side of the pump is then available on standby. The ball valves and the pressure relief valve from the defective pumphead are later cleaned and examined. If not scarred, they are ready for reuse; if scarred they are replaced with new balls and valve seats. Most of the time, only valve cleaning is needed.

In conclusion, note from table 2 that runs of 500 hours and 616 hours duration have been accomplished with a single side of the pump assembly, requiring no overhaul or switchover to the other side of the pump in that time. Also the switchover and overhaul technique, without interruption of a run can be practiced indefinitely for very long continuous runs.

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**TABLE 1.- Vehicle oils and concentrations of coal
in feed slurries in various liquefaction
processes**

<u>Vehicle oil*</u>	<u>Viscosity</u>	<u>Approx. max. concentration of coal in pumpable slurry</u>	<u>Processes</u>
Distillate of boiling range 400°-700° F	10-15 centi- stokes at 140° F	33	SRC, H-coal
Hydrogenated distillate of boiling range 400°-700° F	Unknown	33	Pott- Broche, CSF, Exxon
Heavy oils (distillation bottoms), deashed	10-50 SSF at 180° F	50	Bergius, U. S. Bureau of Mines coal lique- faction R & D
Whole product oil, deashed	5-400 SSF at 180° F	45 ⁷	SYNTHOIL

*All vehicle oils are process-derived materials.

TABLE 2. -Coal slurry pumping history for all runs of the 1/2-TPD SYNTHOIL Unit

Coal	Concentration of coal in feed paste, wt pct	Slurry feed rate, lb/hr	Plant pressure, psig	Total Pumping time, both heads, hr	Pumping time between overhauls*
Hvab, Homestead Mine, Kentucky	35	25	4,000	500	0-249 hrs right side, 249-259 hrs left side, 259-456 hrs right side, 456-500 hrs left side.
do do	35	25	2,000	500	0-130 hrs right side, 130-500 hrs left side.
Mvb, Clearfield County, PA	35	25	4,000	36	0-36 hrs right side, no overhaul.
Hvab, Homestead Mine, Kentucky	35	25	4,000	400	0-53 hrs right side, 53-114 hrs left side, 114-500 hrs right side.
Hvbb, Herron Mine, Illinois	35	30	4,000	30	0-30 hrs left side, no overhaul.
Hvab, Homestead Mine, Kentucky	35	50 (both sides of pump)	4,000	100	0-100 hrs both sides on, no overhaul.
do do	35	30	4,000	200	0-151 hrs left side, 151-200 hrs right side.
Hvbb, Spencer County, Indiana	35	25	4,000	20	0-20 hrs right side, no overhaul.
do do	20	25	4,000	8	0-8 hrs right side, no overhaul.
Hvab, Ireland Mine, W. VA.	35	25	4,000	500	0-1 hr left side, 1-500 hrs right side.
do do	35	25	2,000	300	0-300 hrs right side, no overhaul.
do do	35	25	4,000	500	0-500 hrs right side, no overhaul.
do do	35	25	2,000	169	0-281 hrs right side
do do	35	18	2,000	112	no overhaul.
do do	35	25	4,000	375	0-375 hrs right side, no overhaul.

TABLE 2.-Coal slurry pumping history for all runs of the 1/2-TPD SYNTHOIL Unit-
Continued

No coal. Rehydrogenated product oil from a previous run	35	4,000	34	0-34 hrs right side, no overhaul.	
Hvab, Ireland Mine, W. VA.	35	25	4,000	200	0-200 hrs right side, no overhaul.
50 wt pct SRC in SRC-Solvent	25	4,000	96	0-128 hrs right side, no overhaul.	
Pure SRC-Solvent	25	4,000	32		
Hvab, Ireland Mine, W. VA.	35	25	4,000	712	0-320 hrs left side, 320-712 hrs right side.
Hvab, Sinclair Mine, Kentucky	35	25	4,000	279	0-279 hrs right side, no overhaul.
do do	35	25	4,000	169	0-39 hrs left side, 39-169 hrs right side.
do do	35	25	4,000	425	0-78 hrs right side, 78-94 hrs left side, 94-425 hrs right side.
do do	35	25	4,000	365	0-365 hrs right side, no overhaul.
do do	35	25	4,000	621	0-616 hrs right side, 616-621 hrs left side.
do do	35	25	4,000	98	0-98 hrs left side, no overhaul.
do do	35	25	4,000	226	0-226 hrs left side, no overhaul.
do do	35	25	4,000	564	0-12 hrs right side, 12-158 hrs left side, 158-302 hrs right side, 302-564 hrs left side.

*The overhaul consists of changing the pump head that includes the valves and changing the packing; also cleaning the feed inlet manifold with light oil. The ball valves and the pressure relief valve of the removed pumphead are thoroughly cleaned for reuse. If valves are scarred, balls and seats are replaced.

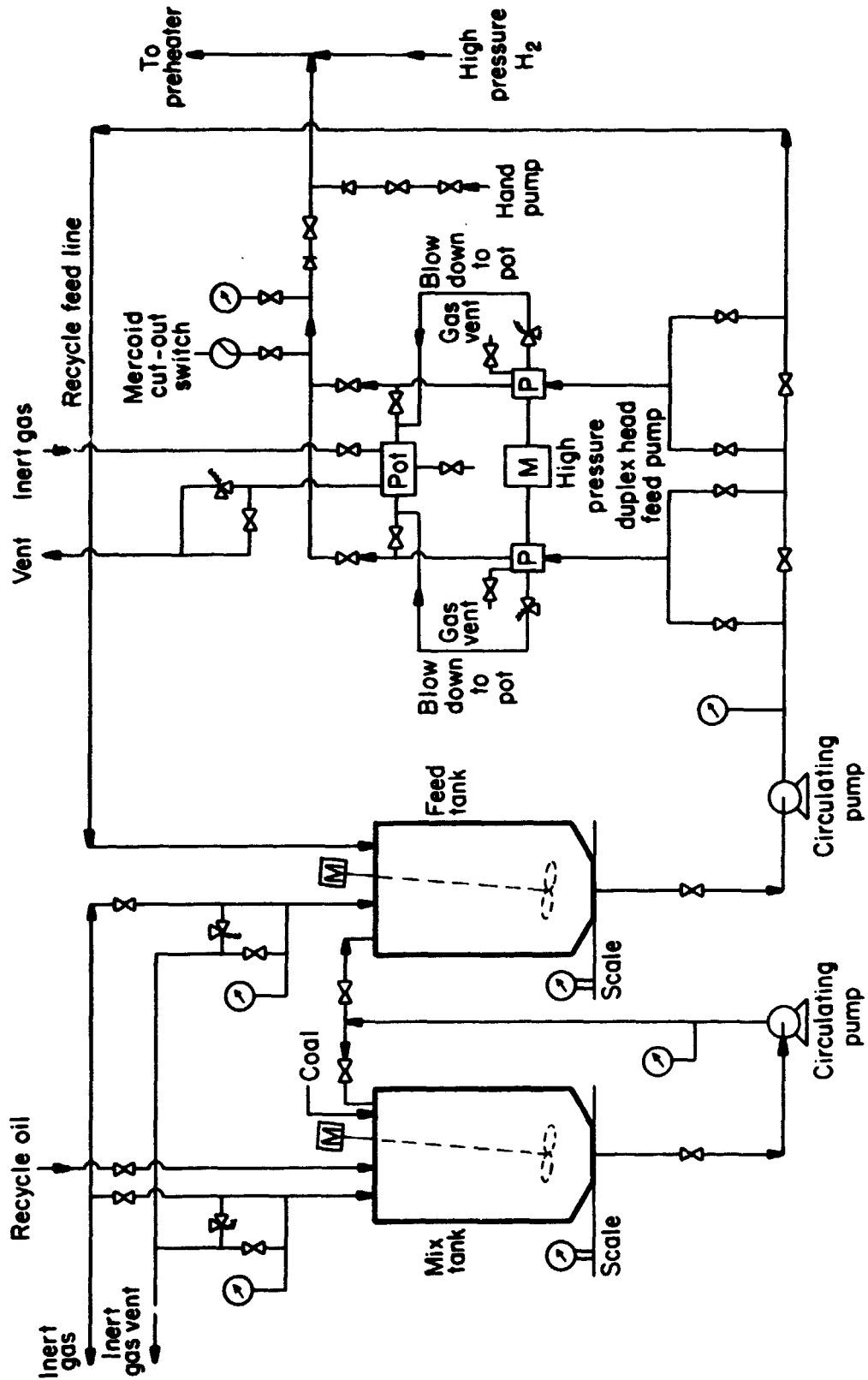


Figure 1 - Coal slurry preparation and feeding system.

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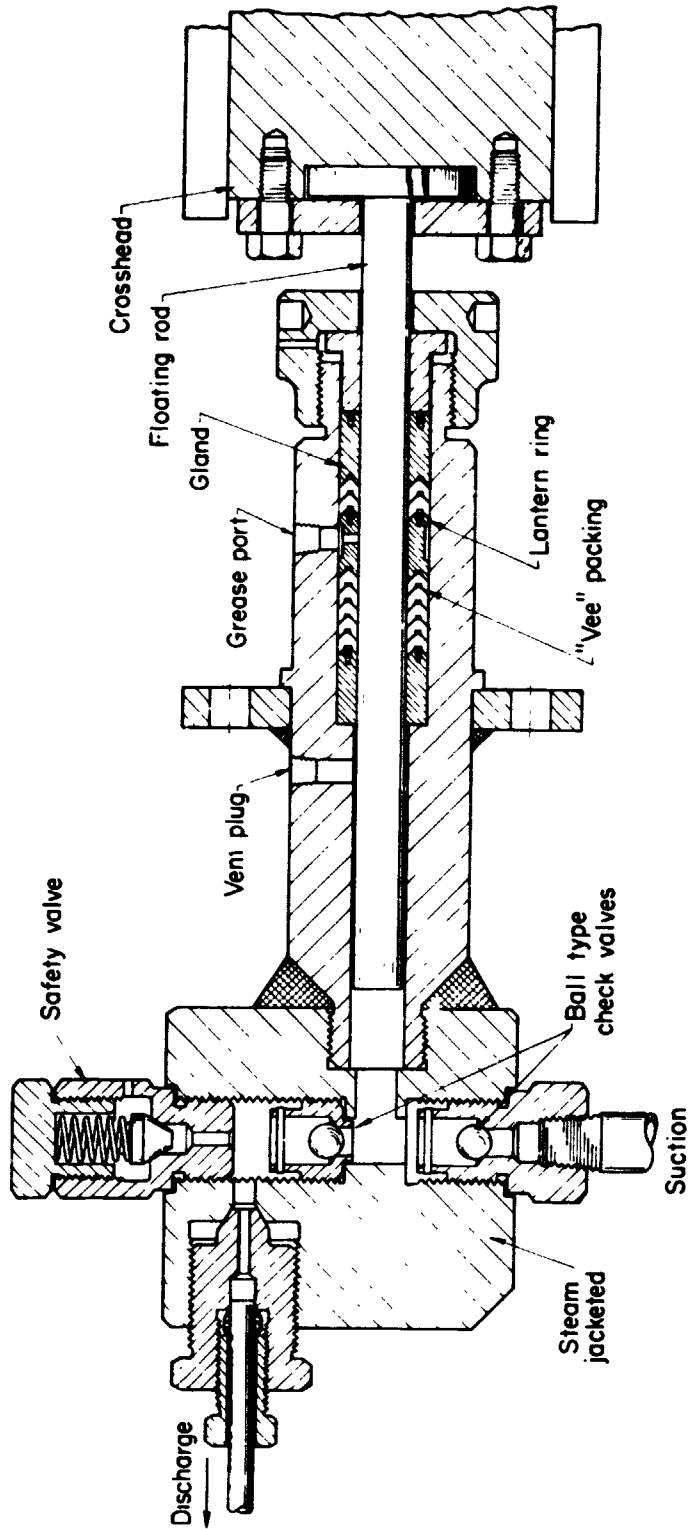


Figure 2 - High pressure feed pump assembly



Figure 3 - Photograph of the pump plunger after 6,000 hours of use.