1. INTRODUCTION AND SUMMARY

The objective of this project is to construct and operate an integrated test circuit for the Molten-Caustic-Leaching (Gravimelt) process for desulfurization and demineralization of coal to prove process economics assumptions, deliver product coal and to test process conditions aimed at significantly lowering costs.

The test circuit consists of six unit operations which together provide a continuous system for leaching coal and regenerating the reactant. These units are a) a kiln for reacting molten caustic with coal, b) a seven stage water washing section for recovering caustic from the coal, c) a three-stage acid washing section for removing the last traces of metals and alkali and providing an ultra pure coal product, d) a water treatment section to provide either dischargable or recycleable water, e) a regeneration section to provide purified aqueous caustic and f) an evaporator section to provide molten-caustic for recycle to the kiln reactor.

The integrated test circuit facility contains more than 160 pieces of equipment including filters, centrifuges, tanks, reactors, feeders and the kiln and rising film evaporator. It occupies 3700 square feet and is fitted with more than 6000 feet of piping, 425 valves, 80 instruments and controls as well as a control room with computer control and data acquisition and reduction system.

The highlight of the quarter is completion of a week of around-the-clock shakedown of the integrated test circuit. The results are presented in the next section.
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2. SHAKEDOWN OPERATION AND DATA

The MCL (Gravimelt) Integrated Test Circuit was operated in a shakedown mode for approximately 100 hours with the kiln reactor temperature at 330 C and at a residence time of approximately 1.5 hours utilizing only one weight of caustic per weight of coal. The caustic feed composition was varied from 50/50 sodium hydroxide to potassium hydroxide, to 100% sodium hydroxide. The coal caustic pellets exiting the kiln were washed with water in a countercurrent mode utilizing five centrifuge repulp tank stages. The water washed coal was next contacted with dilute sulfuric acid and washed free of the sulfuric acid utilizing the three stage countercurrent acid wash and recovery section. Product Gravimelt coal was obtained from the last stage of this section as a dewatered cake product. Spent caustic was sent to the caustic regeneration section where mineral matter and sulfur were allowed to precipitate at ambient temperature. The acid wash stream was sent to a single stage lime treatment vessel for precipitation of impurities.

The assessment based on the data obtained to date is as follows: 1) although kiln reactor conditions were short residence time (1.5 to 2.0 hours vs the previous 2.5 to 5.0 hours), low caustic to coal ratio (only one weight of caustic per weight of coal rather than the usual 2 to 2.5 weights) and use of sodium hydroxide rather mixed caustic, for part of the week, both sulfur and ash removals of 40% and 80%, respectively, were near normal for the temperature of 330° C, 2) cake moisture measured in the caustic water wash train was approximately one weight of liquid per weight of coal which was significantly lower than observed in previous non-continuous testing of 3 weights of liquid per weight of coal, 3) the final Gravimelt coal product moisture level, which averaged about 25% moisture and was as low as 10%, is much lower than that found in previous non-continuous testing of one weight of moisture per weight of coal, 4) the product coal had a volatile content which averaged 40% as compared with 38% for untreated feed Pittsburgh No. 8 coal which corresponds to zero loss of volatiles due to Gravimelt treatment - the Gravimelt coal increase can be accounted for by removal of ash from the coal, 5) the acid
wash solution contained less than 26 ppm of sodium and potassium which corresponds to residual alkali on coal of less than 0.1% due to the efficient action of the countercurrent water wash system, 6) the acid wash was sent to a single stage lime treatment vessel which removed the iron, sulfate and acidity from the stream giving regenerated water containing less than 25 ppm of coal derived sodium, potassium, silicon, aluminum and iron and, 7) in contrast to all previous laboratory, benchscale and modular kiln operations, very little carbonate was formed during reaction of caustic with the coal during the run. Only about 5% of the caustic was converted to carbonate and it is felt that even this conversion could be lowered by further tightening any possible exposure of hot coal to air.

The following process conclusions can be drawn from the above information; 1) the combination of continuous operation, rigorous exclusion of air from the system and mild conditions allows production of Gravimelt coal, for the first time, which has no volatile loss and does not have the propensity to form excessively wet cakes in countercurrent washing - if these features can be maintained under more severe conditions it may be possible to produce compliance Gravimelt coal which will have excellent combustion properties because of near zero loss of hydrocarbon volatiles, 2) the low moisture content of the cakes, if maintained while producing compliance coal or near compliance coal, can significantly lower processing costs through elimination of a number of stages of washing and utilization of less water, 3) first indications are that sodium hydroxide is as efficient as mixtures of sodium and potassium hydroxide - this will offer cost savings in purchase of process chemicals, 4) very low alkali retention by Gravimelt coal obtained from the water wash section indicates that alkali losses may be reduced to an insignificant level resulting in a very large cost savings to the process and eliminating a necessity to precipitate sodium and potassium from waste water prior to recycle, 5) since one weight of caustic per weight of coal appears to operate as efficiently as 2 or 2-1/2 weights, it may be possible to further cut the use of caustic to 0.75 or 0.5 per weight of coal which would result in additional savings of wash water, 6) this would be even more likely for
deeply cleaned low ash coals, if not for the lightly cleaned coal used in this operational shakedown sequence and 7) carbonate formation may prove to not be a problem in continuous test circuit operation, thereby saving lime costs and enhancing operation.

3. EQUIPMENT PERFORMANCE

The package Blaw-Knox evaporator was not used during this shakedown test as a previous detailed shakedown and evaluation had shown that the manufacturer has miscalculated heat losses and thereby underpowered the hot oil thermal system by one third (60 kw vs a necessary 90 kw) for plant load following and had also failed to provide adequate heat tracing and insulation of the molten caustic collection and exit system. After an on-site inspection by a Blaw-Knox representative and reengineering of the unit, Blaw-Knox has taken responsibility for adding an additional 30 kw hot oil heating system to the unit. This system will be skid-mounted and delivered to our site for installation in an estimated 12 weeks. The heat tracing band heaters necessary to correct the molten caustic system have been ordered and will be installed for operational testing in January at which time, it is planned to operate the evaporator with the available 60 kw system to produce caustic for recycle. However, it will be necessary to recycle only a fraction of the caustic stream and thus it will not be possible to buildup impurities to a complete steady state until the modifications are delivered and installed.

The first two separators in the caustic water wash system are rotary drum vacuum filters. These units were not operated during this shakedown run as a previous shakedown test had shown that level control instrumentation supplied by the vendor need to be replaced. The vendor, the Bird Company, has taken responsibility for the necessary replacement. This should occur by the end of January and these units will then be utilized in the washing, as necessary.
The caustic wash section has functioned reasonably well, utilizing only the five stages of centrifugation, but with elevated wash water flow rate to caustic ratio, to provide a first stage liquid specific gravity under 1.2 as is required for coal separation. It was noted that interstage centrifuge slurry lines and tank exits occasionally plug. Inspection disclosed that stirring was inefficient allowing coal packing into tank exit lines. The stirrers will be replaced and lengthened as bar stock is obtained. Foaming is frequently observed in the dissolver and centrifuge feed tanks interfering with operation of the caustic wash section. Methods for eliminating or controlling foam are being investigated.

The kiln reactor, newly fitted with an auger side scrapper device, has functioned exceedingly well providing a smooth feed and discharge of product coal-caustic, heated to reaction temperature and at constant residence time. At the conclusion of the week of testing, a drive bearing unit was in need of replacement. The next bearing will be installed in mid-January.

4. PLANS FOR NEXT QUARTER

Complete shakedown and operate the plant under conditions modified to meet the current ability of the equipment with the objective of producing Gravimelt coal product and process data. Institute necessary replacement and repairs during planned alternating down-weeks in January and bring each of the unit operations into compliance with objectives of the test plan to demonstrate Gravimelt coal production meeting New Source Performance Standards. When all unit operations are brought into compliance with specifications, modify operational conditions into the most effective range.