Appendix I
Observations on the Design of the Automated Minimethod Instruments

Observations on the design of automated minimethod instruments

Grabner instrument

On two occasions, Grabner instruments were damaged while the sample exited from the instrument. Three major factors attributed to the failures, 1) the samples analyzed were much too viscous for the designed values of the instrument, 2) the Grabner instrument lacked heated sample lines, and 3) there was no automatic shutdown of the piston during an overpressure situation. Heated sample lines would help improve the flow of the samples in and out of the instrument by decreasing the viscosity of heavy materials. An automatic piston shutdown is critical for the function of this type of instrument while analyzing highly viscous samples. There was no automatic stop feature and the operator had no option to halt the process that ends in the destruction of the pressure transducer.

A very slow plunger speed of 26 was used for all plunger movements, but this slow speed had no advantage on upstrokes during air purging or when there was no risk of damage to the pressure sensor. This consistent and unchangeable setting just added to the runtime of the analysis extending the time necessary to keep the syringe warm and the time needed for the operator to monitor the runs. The operator should be allowed to set plunger speeds for each activity separately to suit to material being tested. This includes drawing the sample in, expelling the sample, expansions, flushing, and general positioning of the piston.

Eravap instrument

The Eravap functions similarly to the Grabner. Therefore, it has some of the same problems expelling a high viscous sample. The Eravap does have an auto stop feature during an overpressure situation. This feature prevented the instrument from damaging itself during the expulsion of spent samples. There was no option to continue after a failed flush injection and a sample injection could not be completed until after a successful flush injection.

The Eravap was equipped with heated sample and exit lines. The temperature was limited to 140°F on these lines. It is recommended that this limit be extended to 300°F for residual fuel oil samples.

While the exit line was heated, there was not an option to set the expulsion temperature of the measurement cell during expulsion. For example, if the sample was injected at 140°F and the vapor pressure was measured at 100°F, the sample would be ejected at 100°F. This would cause the internal piston to impact the highly viscous sample, resulting in an error.

The Eravap included the password for the service screen and the operator was given much more flexibility to make changes to piston speed and make valve

actuations directly from the front panel of the instrument than for the Grabner instruments. However, the plunger rate was restricted to speed ratings of 300 to 2000. It would have been very useful if a lower speed rating was available and if the user could set plunger speeds for each activity (e.g.,, drawing the sample in, expelling the sample, expansions, and flushing) separately to suit to material being tested. The Eravap would draw the sample in at the 300 speed rating, yet expel it at the 2000 rating. This was the cause of many of the instrument faults encountered during this study.

As vapor pressure analyses using the Eralytics instrument were coming to a halt, it was observed that the instrument started the first expansion of the samples before reaching the temperature set point of the desired measurement. The air and vapor above heavy refinery liquids can be slow to reach equilibrium with the liquid, and conducting an expansion at a temperature other than the desired temperature could result in pressure measurements being taken before equilibrium was reached. As shown in Appendix H, even a small error in the pressure reading could significantly impact the vapor pressure results for the fuel oil no. 6 samples. The second and third expansions also took place before the cell temperature was at steady state. As with the Grabner instrument, the Eralytics instrument used a shaker to help dissolved gases escape the sample during and after the expansions. More testing would be required to identify the sequencing and hold time of each expansion to insure that equilibrium was truly reached with the heavy fuel oil samples before a pressure reading was taken. It would be interesting to find out if the vapor pressure values for fuel oil no. 6 would be more sensible if the instrument was instructed to reach each temperature set point, then expand, then shake, then wait for equilibrium to be established. For heavy refinery liquids, it may be necessary to tighten the conditions that determine when equilibrium is reached, as well.

Suggestions for the design of minimethod instruments used to measure the vapor pressure of heavy refinery liquids

Both instruments set unrealistic speeds for moving the fuel oil no. 6 samples through the instrument. If more control over the piston speed was afforded to the operator, damage to the Grabner could have been prevented, allowing for all of the study materials to be analyzed by this instrument, including the fuel oil no. 6 samples. In the case of the Eralytics instrument, operator control of the piston speed would have allowed more samples from each syringe to be analyzed.

There is a lot of unused volume within the instrument chassis of these instruments. This required more transport of viscous materials through narrow tubes than was necessary to conduct the measurements.

Incomplete filling and overfilling of the cell during injections of the samples may have caused poor performance of the instruments in some cases.

Design suggestions include:

- Offering the operator total control of the piston and valves on one page, with the instrument taking control only when it was necessary to stop downward motion of the piston in an overpressure situation. The user could then move the piston down in increments and at different speeds to best clean out the cell between samples. A visual gauge of the pressure that was color-coded in green, yellow, and red could guide the user about the amount of pressure to exert on the internal plunger.
- Reducing the length of the sample and exhaust lines by reducing the overall footprint of the instrument. This would allow for more direct injection and expulsion of samples to and from the cell, which would help during the analysis of heavy refinery liquids.
- Using an automated injector/syringe pump to put pressure on the syringe and inject the sample into the cell. The instrument could be programmed to deliver amounts of sample and monitor internal pressure and sample syringe position with a precision that a human could not replicate. Safeguards against analysis of incomplete samples (e.g., when the syringe runs dry) and injection of headspace if the syringe if improperly loaded could be put in place.
- Implementing a means of controlled heating of the sample syringe by the instrument to aid the analysis of heavy refinery liquids.
- Implementing an automated cleaning cycle once highly viscous materials are removed from the instrument; this would allow the operator to process data or prepare the next sample.