DETERMINATION OF THE CAPABILITY OF PITCH VISCOSITY AND SOFTENING POINT MEASUREMENT SYSTEMS

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Introduction

Some of the common tests used to characterize coal-tar binder pitch are: softening point (SP), quinoline insolubles (QI), toluene insolubles (TI), ash, coking value (CV), distillate content to 360°C, and viscosity. It is well known that all these tests exhibit variability which is due to variations in the instrument, analyst, laboratory, sample, etc. This paper discusses the determination of the capability of the viscosity and SP measurement systems used by 3 laboratories.

Experimental Work

The protocol for the study specified determining the viscosity (160°C) and SP, in duplicate, of binder pitches from 20 different but sequential production runs. The samples were obtained weekly over a 4.5 month period. A total of 40 samples (2 from each production run) were coded and sent to each of the participants in random order to minimize bias. All participating laboratories used Brookfield RVT viscometers (Br) and shear rates of 4.7, 9.3, and 18.6 sec^{-1} (5, 10, and 20 rpm). In addition, one laboratory used a Haake RV 20 viscometer (H). To determine the effects of constant shearing on viscosity, readings were taken at given time intervals as the pitch was being sheared at a constant shear rate.

The average properties of the 20 production pitches as determined by ASTM methods are: SP-100°C, QI-16.4 wt%, TI- 6.4 wt%, ash-0.19 wt%, and CV-60.1 wt%. The viscosity was determined by ASTM D5018 which was modified as discussed above.

Determination of Capability

The capability of the viscosity and SP systems was determined by comparing the variability of the viscosity and SP measurements to the variability in the measurements of the 20 sequentially produced pitches. The calculated capabilities for the viscosity and SP measuring systems are summarized in Table 1. A capability ratio of <0.3 is usually an indication that the measurement variation is small enough to detect significant changes in the process.

	Viscosity, cps			SP, °C		
Laboratory	A	B	C	A	B	C
Average	1488	1594	1718	110.2	110.3	111.4
R - Duplicate	66	114	117	0.35	0.74	0.41
R - Moving	129	150	155	0.95	0.97	0.85
Capability	0.26	0.58	0.57	0.14	0.58	0.23

Table 1. Summary of capabilities

Plots of viscosity vs. SP data for Laboratories A and C are given in Figure 1. The plots reflect the data presented in Table 1; i.e., the data with higher ratios show more scatter. In Figures 2 and 3, plots of viscosity vs. reference number and SP vs. reference number, respectively, are presented; reference numbers represent samples from sequential production runs. The lines drawn on each plot are the upper control limit, average, and lower control limit. The more capable the measuring system is, the narrower will be the band formed by the upper and lower control limits. Approximately 2/3 of the points will be *outside* the control limits of a capable measuring system; therefore, 13 points should be outside the limits [1].

Effect of Shear Stress and Shearing Time

It was observed that in almost all cases, the viscosity decreased when the sample was sheared at a steady rate; also, the viscosity decreased as the shear rate increased (Figure 4). The amount of reduction in viscosity with increasing shear time or increasing shear rate varied with the sample and the measuring system; in general, the effect was less noticeable with the Haake viscometer.

Conclusions

This preliminary study has shown that statistical techniques can be used to estimate the total variability in pitch viscosity and softening point and of the viscosity and softening point measuring systems. The capability of each measuring system can be calculated; various measuring systems can then be meaningfully compared.

Reference

1. Hart, R. F. and Hart, M., "Is That Measurement Valid?" *Chemical Processing*, October, 1988, pp 28-32.

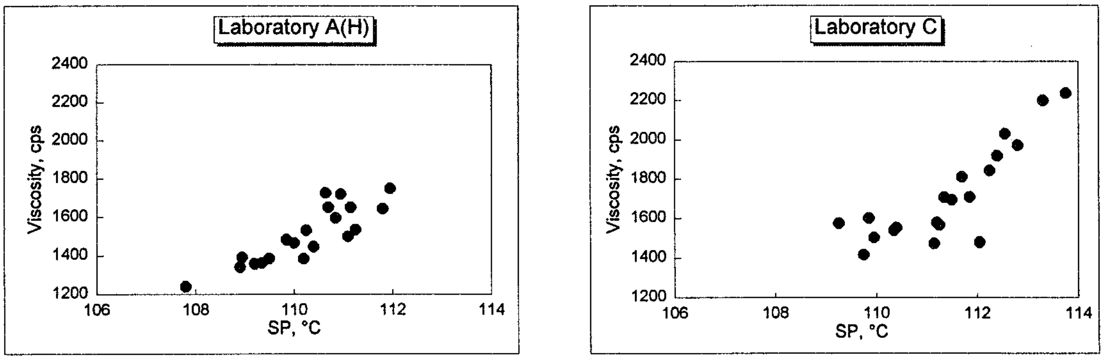

Figure 1. Plots of viscosity vs softening point.

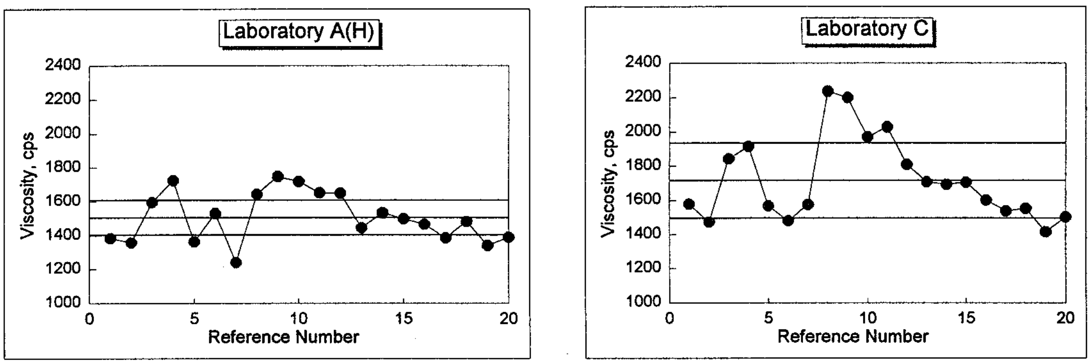

Figure 2. Viscosity of sequential samples.

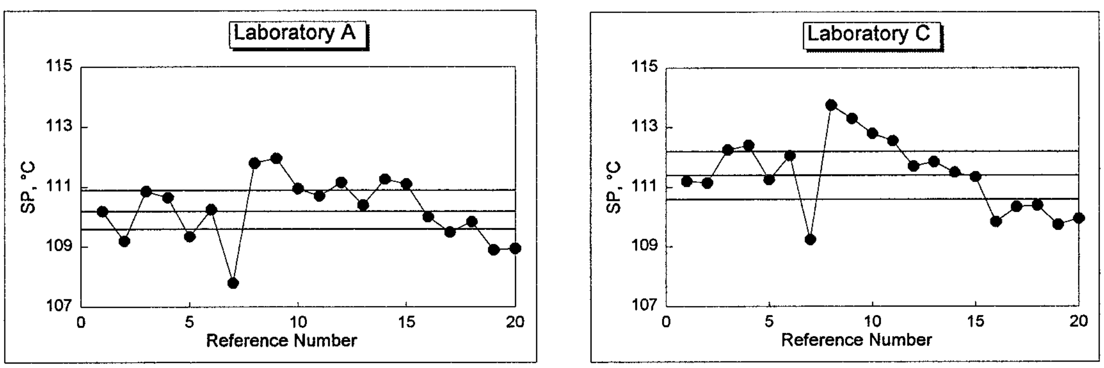

Figure 3. Softening point of sequential samples.

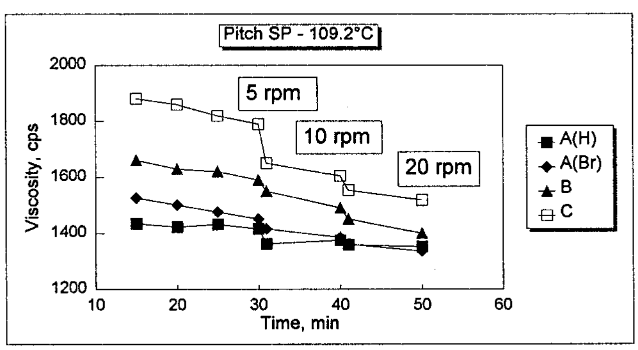

Figure 4. Effect of shear rate and shearing time on viscosity @ 160°C