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**STORAGE, TRANSPORTATION, AND ATOMIZATION
OF CWF FOR RESIDENTIAL APPLICATIONS**

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1. INTRODUCTION AND SUMMARY

There are continuing pressures in the United States to reduce SO₂ emissions in order to control acid rain and its damage to the environment. Advanced beneficiation processes are under development to provide low-ash, low-sulfur coals to allow combustion performance approximately equal to that of oil or gas. A major benefit of this development will be the contribution of these technologies to the abatement of acid rain precursors.

In addition to the industrial and utility applications of beneficiated coal products, there is a large market potential for use of these fuels in small-scale applications, including commercial and residential furnaces. Although much work has been done on the development of components and systems for the formulation and use of CWF for industrial and utility applications, the results of these R&D efforts are not directly applicable to small residential combustors. It is therefore essential to develop a data base for future use of CWF for this application.

Under a contract with the Department of Energy, Pittsburgh Energy Technology Center (PETC), Tecogen Inc., is developing a CWF-fired advanced combustor for residential applications. In order to bring the technology to the point of commercial readiness expeditiously, it is important to develop components and systems for storage, transportation, and handling of these fuels for end use. This research and development effort is designed to augment the data base on the use of CWF for residential applications.

A highly qualified team has been assembled to perform the program. The primary responsibilities of the team members are as follows:

1. Tecogen Inc. (A Subsidiary of Thermo Electron Corporation)
Waltham, Massachusetts
Tecogen, as the prime contractor, will manage and direct the program. The CWF storage, handling, and integrated combustion tests will be carried out in their advanced combustor facility for residential applications.
2. AMAX R&D, Golden, Colorado
AMAX R&D will assess formulations of CWF and prepare the fuel DOE in order to conduct tests in the program. AMAX R&D will focus on bulk storage, handling, and transportation prior to distribution for regional residential use.

3. Carnegie-Mellon University, Pittsburgh, Pennsylvania

Carnegie-Mellon University will compare the atomization characteristics of the CWFs prepared in the program. The effects of beneficiation on atomization will be examined, and spray characteristics will be studied by means of high-speed photography and a Malvern Particle Size Analyzer.

The proposed test program will examine the stability and rheology of the CWFs prepared from various beneficiated coal products made from the following coals: Illinois No. 6, Wyodak, and Upper Elkhorn No. 3.

The fuel characterization will include: particle size distribution, ultimate and proximate analyses of coal, ash composition, ash fusion temperature, and solids concentration. For rheological characteristics, data will be obtained at varying shear rates, temperature, and agitation or recirculation in the bulk storage tanks.

The effect of freeze-thaw on the stability and rheology of the CWF will be evaluated. Control of environmental conditions will be examined by means of formulation additives, including the addition of freezing point depressants and biocides.

The bulk storage studies will involve testing CWF stability characteristics with and without agitation or recirculation. The design of the storage tank will be reviewed, and the effectiveness of cone bottom and dish bottom tanks will be tested. The CWF storage and handling tests will be carried out at Tecogen's CWF-fired advanced combustor facility.

Carnegie-Mellon University will evaluate the atomization characteristics of the various atomizers as a function of coal type, formulation, and operating conditions.

Tecogen Inc., and its subcontractors are aware of the work previously carried out and currently under way in the formulation of CWF using beneficiated coal products as well as the data available in the public domain. No attempt will, therefore, be made to duplicate the work funded previously by DOE or through other research organizations such as EPRI. We are strongly confident that the results of this program will significantly augment the data base needed for future development of components and systems for storing, transporting, and handling CWF for residential applications.

During the quarter, work was conducted on Tasks 2, 3, 4, and 5. Under Task 2, two coal water slurry fuels were produced from the baseline coal. Chemical stability tests and mechanical resuspension tests were begun under the Task 3 effort. Residential storage work was also initiated and a bladder tank developed. Residential handling tests were initiated under the Task 5 effort to develop pressure drop characteristics for the slurries at residential flows in residential type components.

2. WORK PERFORMED DURING THE THIRD QUARTER

2.1 TASK 2 - FUEL PREPARATION

The coals used under this program were Upper Elkhorn No. 3, Illinois No. 6, and Wyodak. The Upper Elkhorn No. 3 coal was chosen as the base coal since Tecogen has extensive working experience with that coal. The Illinois No. 6 coal was chosen due to the large reserves of the coal and its high sulfur content. The Wyodak coal was chosen as a representative western subbituminous coal. The sources of these coals are listed in Table 2.1.

Seven slurries will be made from the three coals. The nominal fuel specifications are provided in Table 2.2. The coals will be cleaned by several techniques to assess the relative changes in handling, storage, and atomization of the different fuels. The coals and cleaning techniques used for each slurry fuel are summarized in Table 2.3.

Work during the quarter focussed on making and shipping the base coal, Upper Elkhorn No. 3. Work was done to produce two slurries. The slurries Tecogen produced are: (1) a heavy media separation product and (2) a heavy media separation product water leached. The flow sheet comparing the two processing techniques is provided in Figure 2.1. These two slurries will be used to assess the effect water soluble minerals have on the additive requirements and the overall slurry properties. The properties of the fuels are shown in Table 2.4.

Several preliminary scoping tests were made on a sample of the oil agglomeration coal cleaning product supplied by the Homer City facility. The pellets could be broken easily but could not be fully disintegrated in water, even with high intensity agitation. Full disintegration was readily accomplished in a stirred ball mill, and the rheology of the resulting slurry appears to be acceptable. The ground product from the stirred ball mill was 99.4 percent passing 30 μm in size. The pellets contained 3.4 percent ash and 2.8 percent sulfur. The properties of the test slurry are shown in Table 2.5.

2.2 TASK 3 - BULK STORAGE

Tests under this task looked at slurry resuspension and chemical stabilization. For this task, 250 gallons of each slurry were used. The 250 gallons consisted of four batches ground by differing degrees. Particle size distributions were made on each batch and a spreadsheet algorithm was set up for calculating the amounts of each batch to be included in the final blended slurries to give mass mean diameters of 9 and 13 μm with comparable size distributions.

TABLE 2.1
SOURCES OF COALS

	<u>Rank</u>	<u>Source</u>
Upper Elkhorn No. 3	hvA	Wentz Plant, Virginia Westmoreland Coal Company
Illinois No. 6 (HMS)	hvC	Delta Mine Marion, Illinois AMAX Coal Company
Illinois No. 6 (Agglo)	hvC	PETC Sponsored Testing by Bechtel at CQ, Inc., Homer City, Pennsylvania Mine source not known at this time.
Upper Wyodak (Roland Seam, Power River Basin)	sub C	Eagle Butte Mine Gillette, Wyoming AMAX Coal Company

TABLE 2.2
NOMINAL FUEL SPECIFICATIONS

Loading	Greater than 55%
Heating Value	9000 Btu/lb slurry
Viscosity	200 cp at 100 s⁻¹
Rheology	Pseudoplastic to Newtonian at shear rates to 1000 s⁻¹
Ash	Less than 1%
Sulfur	Less than 0.7%
Top size	98% minus 325 mesh
Mass Mean Size	9 to 11 μm
Stability	No significant settling in 30 days

TABLE 2.3
COAL TYPE AND CLEANING TECHNIQUE COMBINATIONS

<u>Coal</u>	<u>Cleaning Method</u>
1. Upper Elkhorn No. 3	Flotation
2. Upper Elkhorn No. 3	Heavy Media Separation (HMS)
3. Upper Elkhorn No. 3	HMS/Milled and Filtered
4. Upper Elkhorn No. 3	HMS/Chemical Cleaned
5. Illinois No. 6	HMS
6. Illinois No. 6	Oil Agglomeration
7. Wyodak	HMS

Figure 2.1 Comparison of Flowsheets for Preparing Batch C and Batch D Fuels

TABLE 2.4
PROPERTIES OF COAL SLURRY FUELS

	<u>Upper Elkhorn HMS</u>	<u>Upper Elkhorn HMS/Wash</u>	<u>Illinois No. 6 HMS</u>	<u>Upper Wyodak HMS</u>
Fuel Designation	Batch C	Batch D	Batch A	Batch B
Fuel Analysis, %				
Coal	53.90	53.90	54.00	40.60
Water	45.87	45.81	45.55	57.78
Additives	0.23	0.29	0.45	1.62
Dry Coal Composition, %				
Ash	1.52	1.62	5.89	5.75
Volatile Matter	34.60	34.70	37.92	44.86
Additives, % of Dry Coal				
A-23 Dispersant	0.37	0.47	0.99	4.00
Flocon Stabilizer	0.05	0.08	None	None
Fuel Viscosity, cP				
at 100 sec ⁻¹	190	200	200	410
at 500 sec ⁻¹	240	310	275	290
at 1,000 sec ⁻¹	240	260	285	255
Particle Size Distribution, % Passing				
75 Micrometers	99.7	99.7	99.8	95.4
45 Micrometers	98.2	98.3	98.7	93.2
30 Micrometers	93.1	93.9	95.7	87.7
20 Micrometers	83.2	81.6	86.9	74.4
10 Micrometers	55.2	47.9	53.0	51.8
4 Micrometers	28.2	22.6	38.3	29.7
1 Micrometer	2.8	2.7	5.1	5.2
Mass Mean Diameter, Micrometers	12.1	13.2	11.4	17.0

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TABLE 2.5
LABORATORY SLURRY PREPARATION TESTS,
AGGLOMERATION-CLEANED ILLINOIS NO. 6

Fuel Composition, Weight %	
Coal	53.8
Water	45.3
Additives	0.9
Viscosity, cP	200–300
Weight Passing, %	
45 Micrometers	99.7
30 Micrometers	99.4
20 Micrometers	90.7
10 Micrometers	34.6
4 Micrometers	29.9
1 Micrometer	3.6

2.2.1 Chemical Stabilization

For the chemical stabilization tests, a procedure and matrix were developed during the quarter and baseline tests were initiated. The baseline tests consisted of producing the slurries without a stabilizer and allowing them to stand for two weeks. At the end of the two weeks the slurries were modified by the addition of a stabilizer, if required, and the viscosity was adjusted to 200 cp at 100 s^{-1} .

The stability of the slurries is established by the AMAX procedure, given in Table 2.6. The test is similar to the EPRI-recommended ASTM procedure for paints, ASTM standard D869-78. The difference lies in the numerical rating of the observed behavior. The ASTM procedure was used in Task 4 and is presented in Appendix A.

2.2.2 Mechanical Resuspension

Two tanks were produced to test a highly stable slurry and an unstable slurry. One tank has a flat bottom, and the other has a conical bottom. Recirculation and mixing tests will be conducted on this equipment to assess the requirements for bulk storage.

2.3 TASK 4 - RESIDENTIAL STORAGE

2.3.1 Initial Slurry Testing

Four of the seven coal water fuels to be used in this test program were received from AMAX during this period. The fuels, their cleaning methods, and the abbreviations used in this report are presented in Table 2.6. In obtaining a sample to test in the transfer line evaluation, the barrels were stirred to resuspend any settled material. The following observations were made on the bulk properties on opening the barrels.

The stability of the slurries are assigned ratings according to ASTM¹ standard D869-78 (see Appendix A) for determining paint stability. The ASTM standard assigns a rating of "10" to paints which show no signs of settling and "0" to paints which have formed a hard pack that is not resuspendable. A commonly used alternative is the AMAX standard that attributes a "0" to a perfect slurry and a "3" to a slurry that has a hard pack which is not resuspendable. The ASTM standard is used throughout this report to quantify slurry stability.

The Illinois No. 6 slurry (I-HMS), which was cleaned by heavy media separation, was produced 2/16/90. The slurry was opened and stirred 6/27/90 at Tecogen. This slurry exhibited a hard pack at 18 in. from the slurry surface and would be attributed a rating of "2" on the ASTM scale.

TABLE 2.6
COALS AND CLEANING METHODS USED IN THE TEST PROGRAM

<u>Coal</u>	<u>Cleaning Method</u>	<u>Abbreviation</u>
1. Upper Elkhorn No. 3	Flotation	UE-FL
2. Upper Elkhorn No. 3	Heavy Media Separation (HMS)	UE-HMS
3. Upper Elkhorn No. 3	HMS/Milled and Filtered	UE-M&F
4. Upper Elkhorn No. 3	HMS/Chemical Cleaning	UE-CC
5. Illinois No. 6	HMS	I-HMS
6. Illinois No. 6	Oil Agglomeration	I-OA
7. Wyodak	HMS	W-HMS

The Wyodak slurry (W-HMS), which was cleaned by heavy media separation, was produced 4/23/90. The slurry was opened and stirred 6/29/90. This slurry had no hard pack but it did have a very thick feel (like molasses). It would have a stability of "8" on the ASTM scale. The Wyodak slurry formed a skin after 3 hours of exposure to air. The peristaltic pump being used for transfer operations was unable to fill the transfer lines and was replaced with the diaphragm pump, which was successful. The peristaltic pump was reinstalled, but it was found that the lines had plugged while the peristaltic pump was being placed back into the transfer line. It was decided to try further testing at another time.

The Upper Elkhorn slurry (UE-HMS), which was cleaned by heavy media separation, was produced 5/25/90. The slurry was opened and stirred 6/26/90. It seemed very thin with no settling, and would receive a "10" on the ASTM scale.

The Upper Elkhorn slurry (UE-M&F), which was cleaned by heavy media separation then milled and filtered, was prepared 6/5/90. The slurry was opened and stirred 6/28/90. It was similar in consistency to UE-HMS slurry and would also receive a "10" on the ASTM scale.

2.3.2 Mechanical Resuspension

Various equipment designs were considered for residential slurry storage. Of the designs considered, a rigid container with a flexible bladder insert was chosen as the best system for residential application. A schematic showing the storage container with the recirculating pump is illustrated in Figure 2.2. The formation of dried slurry cakes at air/slurry interfaces precluded any design that allows air to come into contact with the fuel. The typical residential application is assumed to store slurry for long periods with the furnace inactive. Even optimal slurry formulations will settle out when stored for periods of 6 months or more. In order to maintain particle suspension necessary for slurry usage a recirculating pump was considered a necessary part of the system. The slurry is withdrawn from the bottom of the tank to ensure complete recirculation of the contents of the container. This flow direction was chosen instead of flow from the top of the container since the configuration must take into account the minimum level of slurry that may be available and the possible leakage of air into the system.

The flexible bladder is shown collapsed in Figure 2.3. The rigid storage container is shown in Figure 2.4. During initial trials with the flexible container, one of the seams ruptured. Modifications are being made to strengthen the bladder before further testing.

Figure 2.2 Storage System Schematic

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Figure 2.3 Flexible Bladder for Slurry Storage

Figure 2.4 Rigid Container for Housing Collapsible Bladder

2.4 TASK 5 – RESIDENTIAL HANDLING

2.4.1 Design

A schematic of the pumping loop configuration is shown in Figure 2.5. Figure 2.6 is a photograph of the pumping loop configuration. The outer loop consists of a 3/8-inch pipe. The measurements on this loop test the pressure drops on a single standard radius elbow, two single long radius elbows in series, straight pipe lengths, and a tee. The two lines issuing from the tee are then sent to a slurry container. The inner loop consists of 3/4-inch pipe lengths with a standard radius elbow, two large radius elbows, a straight pipe length, and a tee. The 3/4-inch tee legs are combined and sent into a 1 1/2-inch straight pipe length which flows into a container.

2.4.2 Testing Procedure

The following testing procedure was established to measure pressure drops on the transfer line.

1. First flush the transfer line with water at the maximum flow rate available until the water appears clear of residual slurry.
2. The pressure transmitters are connected to the transfer line by 1/4-in. polyethylene connecting lines attached to humidifier clamps. In order to make pressure measurements the connecting lines and the transmitter diaphragm must be purged of air. The exit of each transmitter is connected to a manifold which is connected to a purge pump. Before purging the transmitter and line, turn on the purge line pump until the manifold is full and the return line begins recirculating.
3. For each pressure transmitter in the transfer line being evaluated, close the recycle line and purge each side by first opening the Gemini ball valve and then the humidifier clamp until the pressure transmitter and the connecting line are purged of air. The Gemini ball valve and the humidifier clamp are then closed to keep the connecting line full of water.
4. When all the connecting lines are full the purge pump is shut down. The water is drained from the transfer line. High pressure air is blown through the line until water is removed.

Figure 2.5 Transfer Line Flow Loop

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Figure 2.6 Coal Water Fuel Transfer Loop

5. The system is now ready to fill with slurry. The final valve(s) that close the return lines are closed. The humidifier clamp which is located in the top right hand corner is opened and the bleed line placed in a disposal container. Open the opening ball valve and begin pumping slowly. When the bleed line stops purging air close the clamp and shut off the pump. Place the disposal container at the transfer line return.
6. Pump a volume of slurry through the transfer line equal to the volume of the transfer line. Turn off the pump and close the transfer line return. Replace the disposal container with the main slurry container.
7. Open the return line and set the transfer pump to the desired flow rate.
8. After each measurement the flow rate should be set to zero and the static pressure head should be measured. The static pressure head may be different than the offset due to differences in elevation.
9. When slurry begins to diffuse through the connecting lines near the transmitter the flow should be stopped and the connecting lines purged according to steps 2 and 3. An amount of slurry corresponding to the volume of the transfer line should be pumped through the lines to remove any dilute slurry.
10. When testing has finished the transmitter connecting lines should be cleared according to steps 2 and 3. The system will then be purged with water until the rinsing water is clear. The transfer line will be filled with water as in step 6 and the connecting lines closed in order to keep any remaining slurry from forming cakes.

2.4.3 Coal Water Fuel Testing

Experiments were done to determine the effects of coal water slurry properties on the performance characteristics of the transfer line. The peristaltic pump was used for the transfer line evaluation. The slurries were tested in the following order: UE-HMS, I-HMS, and UE-M&F. The transfer loop is shown in Figure 2.6. The mass flow rate was measured for an Upper Elkhorn and an Illinois No. 6 coal water fuel and was divided by their respective densities to get the volumetric flow rates shown in Figure 2.7. It can be seen from these two measurements that the flow rate is directly proportional to pump setting and independent of coal water slurry type.

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Figure 2.7 Volumetric Flow Rate of Two Slurries versus Setting on the Peristaltic Pump Used in the Transfer Line Evaluation

Pressure drops were measured on the various elements of the transfer line simulator. The pressure drops were measured by allowing the connecting line to fill with slurry to the "pigtail." The pressure drop at the desired flow rate was measured, then the zero offset that occurs from the different hydrodynamic head produced by the differing heights of the columns of slurry was subtracted out. This was repeated until the time response of the pressure transducers was observed to begin falling noticeably, which indicates the connecting line is blocked. The response to changes in flow rate produces a characteristic response, similar in shape to voltage versus time for capacitor discharge.

The experimental matrix tested is shown in Table 2.7. The slurries were tested in the following order: UE-HMS, I-HMS, and UE-M&F. The long radius elbow was not measured on the 3/4-in. pipe for UE-HMS due to the range being set too low. The pressure drop for the 1 1/2-in. pipe in the tests on I-HMS were randomly fluctuating and did not respond to the variations in volumetric flow rate. The pressure drops on the long radius elbow and the tee on the 3/4-in. pipe and the 1 1/2-in. pipe were not measured due to problems in the data recording system. W-HMS was not measured at all due to blockage of the line.

2.4.4 Data Analysis

The pressure drop of slurries in the pipe flow has been shown to be estimated reasonably from rotational viscometer data.^{2,3,4} The pressure drop in the pipe flow for three of the slurries has been measured on the transfer line system. An example of the data recorded for each pipe element is shown in Figure 2.8. Appendix B contains plots of the pressure drop versus setting data for each of the tests performed. In Figure 2.8, measured pressure drop is plotted against pump setting. The volumetric flow rate produced in response to the pump setting is shown in Figure 2.7. The theoretical treatment of the effects on the shear rate of non-Newtonian fluid properties has been derived elsewhere.⁵ The pressure drop in straight pipe sections is the result of the shear stress exerted on the fluid. The shear stress can be found from equation 1 to be:

$$\tau_w = \frac{-R}{2} \left(\frac{dP}{dx} \right) \quad (1)$$

where:

- τ_w = shear stress
- R = tube internal diameter
- dP/dx = pressure drop per unit length of pipe

which is independent of fluid properties.

TABLE 2.7
COMPLETED EXPERIMENTAL MATRIX
ON THE TRANSFER LINE EVALUATION APPARATUS

TUBE D.	3/8"				3/4"				1 1/2"
SLURRY	ELBOW	L.R. ELBOW	STRAIGHT	TEE	ELBOW	L.R. ELBOW	STRAIGHT	TEE	STRAIGHT
I-HMS	Y	Y	Y	Y	Y	Y	Y	Y	N
W-HMS	N	N	N	N	N	N	N	N	N
UE-HMS	Y	Y	Y	Y	Y	N	Y	Y	Y
UE-M&F	Y	Y	Y	Y	Y	N	Y	N	N

Figure 2.8 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/8-in. Pipe for Slurry UE-HMS

The shear rate must be modified to take into account effects of non-Newtonian fluid properties. The Rabinowitsch equation modifies the apparent shear rate to correct for the deviation:

$$-\dot{\gamma}_w = \frac{4Q}{\pi R^3} \left[\frac{3}{4} + \frac{1}{4} \frac{d \ln Q}{d \ln \tau_w} \right] \quad (2)$$

where:

- $-\dot{\gamma}_w$ = shear rate at the tube wall
- Q = volumetric flow rate
- R = tube internal diameter
- τ_w = shear stress at the tube wall

For a fluid that obeys a power-law equation the differential part of the equation can be reduced to:

$$\frac{d \ln Q}{d \ln \tau_w} = \frac{1}{n} \quad (3)$$

To obtain the power-law index the pressure drop versus flow rate data was regressed to a power-law over the range of flow rates measured. The regression of the data given in Figure 2.8 gives an R-squared value of 0.999 and a power-law index of 0.925 for the 10 data points. The results of the power-law regression for all the transfer line elements are included in Appendix B.

The viscosity was obtained from the ratio of shear stress to shear rate. The plot of viscosity for the Upper Elkhorn (HMS) is shown in Figure 2.9 for all three size tubes. Plots of the viscosity versus shear rate values obtained from the straight sections are located in Appendix C. The values for the three pipe diameters are found to lie on a continuous curve as shown in Figure 2.9. The data are about a third in magnitude from those obtained from AMAX on the rotational viscometer. Further tests are planned at AMAX and Tecogen to obtain rotational viscometer data at lower shear rates.

Previous work^{2,3} has used the rotational viscometer data to determine a modified Reynolds number. The Fanning friction factor relationship, $f = 16/Re$, is then used to determine the pressure drop. This approach will be tried in future analyses.

K-factors were calculated from the measurements using the straight length measurements as the representative equivalent length. The values obtained are shown in Table 2.8. The values obtained can be seen to have a wide range of values.

Figure 2.9 Measurements of Viscosity versus Shear Rate for 3/8-, 3/4-, and 1 1/2-in. Pipes for UE-HMS

TABLE 2.8
CALCULATED K FACTORS

Slurry	Measurement	Tube D.	K
UE-HMS	Standard Radius Elbow	3/8"	0.6351
	Long Radius Elbow	3/8"	0.6854
	Tee	3/8"	0.1528
	Standard Radius Elbow	3/4"	0.1169
	Tee	3/4"	0.2070
I-HMS	Standard Radius Elbow	3/8"	0.6104
	Long Radius Elbow	3/8"	0.5796
	Tee	3/8"	0.4625
	Standard Radius Elbow	3/4"	0.2948
	Long Radius Elbow	3/4"	2.2442
	Tee	3/4"	0.5827
UE-M&F	Standard Radius Elbow	3/8"	0.6202
	Long Radius Elbow	3/8"	0.6517
	Tee	3/8"	0.5229
	Long Radius Elbow	3/4"	0.1983

3. SUMMARY STATUS ASSESSMENT AND FORECAST

During the quarter, work was conducted on Tasks 2, 3, 4, and 5. Under Task 2, two coal water slurry fuels were produced from the baseline coal. Chemical stability tests and mechanical resuspension tests were begun under the Task 3 effort. Residential storage work was also initiated and a bladder tank developed. Residential handling tests were initiated under the Task 5 effort to develop pressure drop characteristics for the slurries at residential flows in residential type components.

During the next quarter, the remainder of the fuels will be received and tests will continue on them. Atomization tests will also be initiated at Carnegie-Mellon.

4. REFERENCES

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APPENDIX A
STANDARD METHOD OF EVALUATING
DEGREE OF SETTLING OF PAINT

Procedure 17
-- Alternative 1 --

EVALUATING DEGREE OF SETTLING OF PAINT

Designation: D 869 - 78

An American National Standard

Standard Method of
EVALUATING DEGREE OF SETTLING
OF PAINT¹

This standard is issued under the fixed designation D 869; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

This method has been approved for use by agencies of the Department of Defense to replace Method 4208 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This method covers the determination of the degree of pigment suspension and ease of remixing a shelf-aged sample of paint to a homogeneous condition suitable for the intended use.

2. Apparatus

2.1 *Container*—Standard 1-pt (500-ml) friction-top can paint container, $3\frac{1}{8} \pm \frac{1}{16}$ in. (8.6 ± 1.6 mm) in diameter, and $3\frac{7}{8} \pm \frac{1}{16}$ in. (98.4 ± 1.6 mm) in height.

2.2 *Spatula*, weighing 45 ± 1 g with square-end blade $4\frac{1}{4}$ in. (123 mm) in length and approximately $\frac{1}{16}$ in. (20.6 mm) in width, shall be used to examine the paint for pigment settling and reincorporation characteristics.

3. Procedure

3.1 Place the sample to be tested for pigment suspension in a 1-pt (500-ml) container, filling the can to within $\frac{1}{2}$ in. (13 mm) of the top. Close the can tightly and hold undisturbed for shelf aging for 6 months or for such other periods of time agreed upon by the purchaser and the seller.

3.2 Open the can holding the shelf-aged sample without shaking or agitation, and examine the sample without removal of supernatant vehicle. Use the spatula to determine the extent and character of portions of the paint that may have separated during storage. Prepare a suitable spatula for this purpose by

cutting the tip from an ordinary 5-in. (127-mm) flexible steel laboratory spatula to the specified length. Hold the spatula perpendicular to and in the center area of the paint at a height whereby the bottom edge of the spatula is level with the top of the can. Drop the spatula from that position. Rate the condition of the sample in accordance with 3.4.

3.3 After examination of the entire sample as described in 3.2, if a portion of the pigment has separated out to form a firm cake at the bottom of the container pour the supernatant portion of the liquid off into a clean container and hold for subsequent use. Reincorporate the separate cake by hand stirring with the spatula, adding back the liquid in small amounts until the pigment has been reincorporated to form a homogeneous paint suitable for the intended use or until it is determined that the pigment cake cannot be reincorporated by hand stirring. Rate the condition of the sample in accordance with 3.4.

3.4 Rate the sample for degree of settling on a scale from 10 to 0 in accordance with the following. Give intermediate conditions the appropriate odd number.

Rating	Description of Paint Condition
10	Perfect suspension. No change from the original condition of the paint.

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings and Materials and is the direct responsibility of Subcommittee D 01.44 on Traffic Paints.

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Rating	Description of Paint Condition	Rating	Description of Paint Condition
8	A definite feel of settling and a slight deposit brought up on spatula. No significant resistance to sidewise movement of spatula.	2	When spatula has been forced through the settled layer it is very difficult to move spatula sidewise. Definite edgewise resistance to movement of spatula. Paint can be remixed to a homogeneous state.
6	Definite cake of settled pigment. Spatula drops through cake to bottom of container under its own weight. Define resistance to sidewise motion of spatula. Coherent portions of cake may be removed on spatula.	0	Very firm cake that cannot be reincorporated with the liquid to form a smooth paint by stirring manually.
4	Spatula does not fall to bottom of container under its own weight. Difficult to move spatula through cake sidewise and slight edgewise re-		

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

APPENDIX B

PRESSURE DROP VERSUS PERISTALTIC PUMP FIGURES

TABLE B.1
REGRESSION STATISTICS FOR PIPE ELEMENTS

Slurry	Element	Tube Diameter	R ²	n	N
UE-HMS	Std. Radius Elbow	3/8"	0.999	0.927	10
UE-HMS	Long Radius Elbow	3/8"	0.998	0.970	10
UE-HMS	Straight Section	3/8"	0.999	0.925	10
UE-HMS	Tee	3/8"	0.984	1.070	10
UE-HMS	Std. Radius Elbow	3/4"	0.979	1.242	10
UE-HMS	Straight Section	3/4"	0.999	0.644	6
UE-HMS	Tee	3/4"	0.982	1.197	1
UE-HMS	Straight Section	1 1/2"	0.987	0.527	9
I-HMS	Std. Radius Elbow	3/8"	0.996	0.764	12
I-HMS	Std. Radius Elbow	3/8"	0.991	0.795	12
I-HMS	Long Radius Elbow	3/8"	0.998	0.800	12
I-HMS	Long Radius Elbow	3/8"	0.984	0.861	12
I-HMS	Straight Section	3/8"	0.998	0.749	12
I-HMS	Straight Section	3/8"	0.997	0.750	12
I-HMS	Tee	3/8"	0.986	0.806	11
I-HMS	Std. Radius Elbow	3/4"	0.966	0.942	4
I-HMS	Long Radius Elbow	3/4"	0.908	0.370	8
I-HMS	Straight Section	3/4"	0.975	0.828	8
I-HMS	Tee	3/4"	0.763	0.831	4
UE-M&F	Std. Radius Elbow	3/8"	0.999	0.860	12
UE-M&F	Std. Radius Elbow	3/8"	0.999	0.856	12
UE-M&F	Std. Radius Elbow	3/8"	0.999	0.880	10
UE-M&F	Long Radius Elbow	3/8"	1.000	0.924	12
UE-M&F	Long Radius Elbow	3/8"	0.996	0.902	13
UE-M&F	Long Radius Elbow	3/8"	1.000	0.935	11
UE-M&F	Straight Section	3/8"	1.000	0.873	12
UE-M&F	Straight Section	3/8"	1.000	0.875	12
UE-M&F	Straight Section	3/8"	0.999	0.881	10
UE-M&F	Tee	3/8"	0.799	0.502	12
UE-M&F	Tee	3/8"	0.909	1.344	12
UE-M&F	Long Radius Elbow	3/4"	0.812	0.673	6
UE-M&F	Straight Section	3/4"	0.972	0.554	8

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Figure B.1 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Standard Radius Elbow of the 3/8-in. Pipe for the UE-HMS Slurry

Figure B.2 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Long Radius Elbows of the 3/8-in. Pipe for the UE-HMS Slurry

Figure B.3 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/8-in. Pipe for the UE-HMS Slurry

Figure B.4 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Tee of the 3/8-in. Pipe for the UE-HMS Slurry

Figure B.5 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Standard Radius Elbow of the 3/4-in. Pipe for the UE-HMS Slurry

Figure B.6 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/4-in. Pipe for the UE-HMS Slurry

Figure B.7 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 1 1/2-in. Pipe for the UE-HMS Slurry

Figure B.8 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Tee of the 3/4-in. Pipe for the UE-HMS Slurry

Figure B.9 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Standard Radius Elbow of the 3/8-in. Pipe for the I-HMS Slurry

Figure B.10 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Long Radius Elbows of the 3/8-in. Pipe for the I-HMS Slurry

Figure B.11 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/8-in. Pipe for the I-HMS Slurry

Figure B.12 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Tee of the 3/8-in. Pipe for the I-HMS Slurry

Figure B.13 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Standard Radius Elbow of the 3/4-in. Pipe for the I-HMS Slurry

Figure B.14 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Long Radius Elbows of the 3/4-in. Pipe for the I-HMS Slurry

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Figure B.15 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/4-in. Pipe for the I-HMS Slurry

Figure B.16 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Tee of the 3/4-in. Pipe for the I-HMS Slurry

Figure B.17 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Standard Radius Elbow of the 3/8-in. Pipe for the UE-M&F Slurry

Figure B.18 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Long Radius Elbows of the 3/8-in. Pipe for the UE-M&F Slurry

Figure B.19 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/8-in. Pipe for the UE-M&F Slurry

Figure B.20 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Tee of the 3/8-in. Pipe for the UE-M&F Slurry

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Figure B.21 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Long Radius Elbows of the 3/4-in. Pipe for the UE-M&F Slurry

Figure B.22 Measurements of Pressure Drop versus Setting on the Peristaltic Pump for the Straight Section of the 3/4-in. Pipe for the UF-M&F Slurry

APPENDIX C
VISCOSITY VERSUS SHEAR RATE FIGURES

Figure C.1 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/8-in. Pipe for the UE-HMS Slurry

Figure C.2 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/4-in. Pipe for the UE-HMS Slurry

Figure C.3 Measurements of Viscosity versus Shear Rate for the Straight Section of the 1 1/2-in. Pipe for the UE-HMS Slurry

Figure C.4 Measurements of Viscosity versus Shear Rate for the Straight Sections of the 3/8-, 3/4-, and 1 1/2-in. Pipes for the UE-HMS Slurry

Figure C.5 Measurements of Viscosity versus Shear Rate for the Straight Sections of the 3/8- and 3/4-in. Pipes for the UE-HMS Slurry

Figure C.6 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/8-in. Pipe for the I-HMS Slurry

Figure C.7 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/4-in. Pipe for the 1-HMS Slurry

Figure C.8 Measurements of Viscosity versus Shear Rate for the Straight Sections of the 3/4- and 3/8-in. Pipes for the I-HMS Slurry

Figure C.9 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/8-in. Pipe for the UE-M&F Slurry

Figure C.10 Measurements of Viscosity versus Shear Rate for the Straight Section of the 3/4-in. Pipe for the UE-M&F Slurry

Figure C.11 Measurements of Viscosity versus Shear Rate for the Straight Sections of the 3/8- and 3/4-in. Pipes for the UE-M&F Slurry

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