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DEVELOPMENT OF SUSPENDED SOLIDS QUALITY CONTROL AND PERFORMANCE EVALUATION SAMPLES

by

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FOREWORD

Environmental measurements are required to determine the quality of *ambient waters and the character of waste effluents. The Environmental* Monitoring and Support Laboratory-Cincinnati:

1. Develops and evaluates techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.

2. Investigates methods for the concentration, recovery, and identification of viruses, bacteria, and other microbiological organisms in water. Conducts studies to determine the responses of aquatic organisms to water quality.

3. Conducts an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.

Commensurate with an Agency-wide quality assurance program, the latest report on the development of synthetic suspended solids samples contains the results of a feasibility study to determine compounds that exhibit the optimum physical and chemical properties for production of large number of samples. Consideration of such factors as solubility, wettability, dispersion, flocculation, abrasion, biodegradability, stability, and sub-sampling recovery resulted in the selection of anthracite coal, Fuller's earth, and rayon fibers as solids materials for suspended solids quality control samples. A total of 10,000 quality control samples, each consisting of one of three specified weights of one of these three compounds, were packaged in individual containers.

Dwight G. Ballinger, Director
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ABSTRACT

A two-phase study was conducted to develop a synthetic suspended solids sample for use in quality control checks and performance evaluation in environmental monitoring laboratories. The first phase consisted of a feasibility study to determine compounds that exhibit the optimum physical and chemical properties for synthetic suspended solids samples; the second phase involved production of suspended solids samples in individual containers.

Compounds investigated that met all the design criteria included rayon fibers and Fuller's earth. A total of 10,000 quality control samples consisting of rayon fibers, Fuller's earth, and anthracite coal were packaged for completion of the project. Anthracite coal was packaged to demonstrate the variability shown by certain types of solids in the sub-sampling step of the suspended solids test method.

This report was submitted in fulfillment of Contract Number 68-03-2333 by Roy F. Weston, Inc. under the sponsorship of the Office of Research and Development, U.S. Environmental Protection Agency. Work was completed in April, 1976.

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ACKNOWLEDGEMENTS

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SECTION I

INTRODUCTION

The success of the environmental protection efforts to a large degree rests on the reliability of the information provided by the data collection activities. Quality control and performance evaluation samples become a requirement to insure confidence in the precision and accuracy of selected methods used by participating laboratories; an evaluation of the results provides a sound basis for judgment of the relative capabilities of those laboratories performing the analyses. The Environmental Monitoring Support Laboratory has prepared various water quality parameters as a quality control service to laboratories and analysts.

To date, no water quality control sample for measuring suspended solids has been developed to assist in gathering water quality data, to aid in determining compliance with established environmental standards, or to aid in the determination of the effectiveness of pollution abatement methods and procedures. A research effort and a feasibility study were required to determine the optimum physical and chemical properties for such a synthetic suspended solid or series of suspended solid samples for use in quality control checks and performance evaluation in environmental monitoring laboratories, because information pertaining to this subject was not available.

BACKGROUND

Industrial and municipal effluents contain suspended solids that vary widely in both physical and chemical composition, including size and specific gravity. Information on solids characteristics has not been researched to an appreciable degree, especially for the various types of industrial wastewaters.

The results of a literature review conducted to characterize sanitary sewage, combined sewer overflows, and stormwater runoff in terms of their suspended solids content and physical and chemical characteristics were reported by Beak Consultants Limited of Rexdale, Ontario, Canada.[1] These results indicated that these wastewaters could not be characterized by single average concentrations of suspended solids or by a single particle size distribution. A wide range of individual chemical and physical parameters would be required to characterize the suspended solids contained in sanitary sewage, combined sewer overflows, and stormwater runoff.

The solids properties of sanitary sewage are influenced by factors such as range of flow rate, time of day, and contribution of industrial wastewaters to the total flow. Industrial wastewaters can add suspended solids

with a variety of particle sizes, specific gravities, and chemical characteristics, depending on the types of wastewaters added. In addition to those solids normally found in sanitary sewage, combined sewer overflows contain solids washed into the sewer systems from land areas and roadways. There also is wide variation in the solids characteristics of separate stormwater runoff, because of land use and varying soil and topographical features.

The characteristics of the grit in wastewater depend on many factors, including: soil; type of ground cover; urban street conditions; age and condition of the sewer pipe and its joints, pipe slope, and catch basins; street cleaning practices; and whether the collection system consists of separate or combined sewers. Available data from existing wastewater treatment plants concerning grit removed were compared to establish criteria for grit characterization.[2,3,4] Based on these data, a "typical grit" for the purpose of investigation ranges in size from 0.2 millimeter (mm) to 2.0 millimeter, with a gradation corresponding to a straight line on a mechanical analysis graph. The specific gravity of the typical grit is assumed to be 2.65. Normal grit concentrations in sewage have been defined as those between 20 and 360 milligrams per liter (mg/l).[2]

Both the velocity and the concentration of suspended solids in sewers vary with position in the sewer cross-section.[5] Suspended solids heavier than water have their lowest concentration near the surface, and the concentration increases with depth below the surface. Suspended solids lighter than water float on the surface of the water. The manner in which the velocity is distributed in the sewer section will affect the distribution of the suspended solids in the flowing water. Therefore, the distribution of suspended solids in the sewer may affect the accuracy of the suspended solids results because of inadequacies in the sample collection or in the methods themselves. However, evaluation of the effects of sampling equipment and sampling procedures on the determination of suspended solids in water and wastewater streams is beyond the scope of this project for the development and packaging of quality control samples of synthetic suspended solids.

OBJECTIVES

The overall objectives of this two-phase contract were: 1) the development of a synthetic suspended solids sample(s) for use as quality control and performance evaluation samples (Phase I); and 2) production and delivery to the U.S. Environmental Protection Agency (EPA) of 10,000 containers (vials) of suspended solids samples (Phase II). This report presents the results of the development program and feasibility study, along with the packaging information and requirements.

DEVELOPMENT CRITERIA

The synthetic suspended solid samples selected from the research effort should be representative of the types of suspended solids in industrial and municipal

wastewaters. The suspended solids present in these types of wastewaters can generally be characterized as falling within the following specific gravity and particle size ranges:

Specific Gravity - 0.8 to 2.65

Particle Size - 0.01 to 4.5 mm

The following design criteria were established for consideration during the feasibility study for the development of the synthetic suspended solids:

1. The weight of synthetic material shall not change during the analytical procedure. Therefore, the material shall be non-volatile and shall not have any other property which will adversely affect the weight during any step of the analytical procedure.

2. Based upon the standard glass fiber filter, 100 percent of the solids should be retained on the filter.

3. The synthetic solids material shall be relatively non-hygroscopic and non-clinging to the sides of the container.

4. The synthetic solids shall be non-flocculating, thereby providing the flexibility to mix several homogenous materials to produce a heterogeneous synthetic solids sample containing fractions of varying size and with varying specific gravities. In addition, the charge on the solid particles shall be delineated. The synthetic solid must be easily wettable and dispersable in water.

5. The synthetic solid samples shall remain constant in weight and character over a long period of time. As such, the solids shall be:

   a. Non-biodegradable
   b. Non-adsorbent
   c. Insoluble in water

6. The synthetic suspended solids shall have high abrasion and impact resistance, to minimize breaking up into smaller particles which would subsequently affect the percent retention on the fiber-glass filter.

SECTION II

CONCLUSIONS

1. During the research investigation, Fuller's earth and rayon fibers were found to be acceptable compounds for packaging as synthetic suspended solids quality control and performance evaluation samples.

2. The precision and accuracy obtainable in the suspended solids test depend within certain limits on the specific gravity and particle size of the suspended solids. These characteristics can affect the accuracy and precision of sub-sampling in the analytical method.

3. Anthracite coal was also selected for packaging primarily to demonstrate error associated with sub-sampling in the analytical method for certain types of suspended solids.

4. Fuller's earth, rayon fibers, and anthracite coal were selected for packaging to obtain the 10,000 vials of quality control samples.

SECTION III

RECOMMENDATIONS

1. Preparation of the suspended solids samples and execution of the analytical method should be in accordance with the instructions specified in Section VI of this report, to minimize variability in the suspended solids quality assurance data obtained.

2. The sub-sampling step of the suspended solids analytical test method should be evaluated, to minimize error in the results. Results from this study indicated that the accuracy of the sub-sampling step is dependent to some degree upon the dispersion characteristics of the suspended solids in the sample.

SECTION IV

DEVELOPMENT AND FEASIBILITY STUDY - PHASE I

LITERATURE SURVEY

Scope and Methodology

The first step in the developmental and feasibility portion of the study consisted of a literature survey to develop a list of synthetic and natural compounds with the desired specific gravity and solubility specifications. Particle size of the compounds was not considered as an initial selection criterion. (For most of the compounds, the particle size could be changed to meet the particle size specifications.) Additional information was also solicited by personal communications (both verbal and written) from manufacturers and chemical processors as to the availability of various types of compounds and their recommendations for additional compounds meeting the initial developmental criteria.

The second step was a literature survey to gather information which would characterize natural, municipal, and industrial wastewaters with respect to specific gravities and particle sizes of the solids in these types of wastewaters. The information characterizing sanitary wastewaters, stormwater, and combined sewer overflow was presented in Section I of this report. However, no information pertaining to the characterization of suspended solids in the various types of industrial wastewaters was found.

The third step was a literature survey to define acceptable developmental test methods for the proposed compounds with respect to the previously mentioned design criteria. There were no acceptable test methods found in the literature for testing the proposed compounds. Therefore, developmental testing procedures were developed for the feasibility investigation of the selected compounds.

Specific tests for each of the following investigation requirements were developed and will be presented in later sections of this report:

Solubility Testing
Wettability and Dispersion Testing
Abrasion Testing
Flocculation Testing
Sub-sampling Recovery Tests
Biodegradability Testing
Stability Testing

Particle size measurements were conducted according to the ASTM Test Method No. E-20 Analysis by Microscopical Methods for Particle Substances of Subsieve Sizes.[6] The analytical procedure employed for suspended solids testing was the method stated in the current EPA manual "Methods for Chemical Analysis of Water and Wastes."[7]

Compounds Meeting Initial Design Criteria

A list of compounds which have specific gravities within the range specified (0.8 - 2.65) and which possibly meet all the other requirements for synthetic suspended solids quality control samples was developed, and is presented in Table 1.[1,2,3,4,5,8]

From this initial list, the compounds listed in Table 2 were selected for the developmental studies, based on established design criteria, availability of compounds, and manufacturers' recommendations.

The compounds listed in Table 2 were categorized into four groups, by specific gravity:

Group	Specific Gravities
I	0.80 - 1.00
II	1.01 - 1.50
III	1.51 - 1.99
IV	2.00 - 2.65

Two compounds (wood fibers and gilsonite) were eliminated before the initial developmental testing. Research and Development personnel in the wood pulp industry recommended elimination of wood fibers for the following reasons:

1. Variability of fiber size. Even after fractionating and passing a pulp slurry through various mesh screens, the thickness of fibers is extremely variable.

2. Even if it were possible to pulp a single tree, the fibers would not be uniform, because of great difference in wood growth during the spring and growth during the summer.

Gilsonite is a very soft hydrocarbon compound that is subject to considerable abrasion and subsequent change in particle size. Gilsonite is extremely friable; it has very little impact resistance and is easily reduced to a fine powder.

TECHNICAL APPROACH

Statistical Treatment of Data

When a sample or a finite number of observations from a population are selected appropriately, one is able to make precise statements concerning

Table 1

Compounds meeting Specific Gravity and Solubility Requirements

Name	Specific Gravity
Aluminum diethyl malonate	1.084
Aluminum Oxide (gibbsite, hydra-argillite, bayerite)	2.42 - 2.53
Aluminum orthophosphate	2.566
Bismuth tartrate	2.595
Boron (tetra) carbide	2.5
Calcium boride	2.3 - 2.45
Cobalt orthophosphate	2.587
Carbon	1.8 - 2.25
Iron orthophosphate (vivianite)	2.58
Lanthanum hexaboride	2.61
Magnesium ortho-arsenate	1.788
Silicon	2.00 - 2.42
Silicon dioxide (cristobalite, lechatelierite, quartz, tridymite, amorphous-opal)	2.1 - 2.66
Sulfur	1.92 - 2.07
2, 4, 6, tribromoaniline	2.35
Anthracene	1.25
Hexachlorobenzene	2.044
4, 4' -dibromo-biphenyl	1.897
4, 4' -dichloro-biphenyl	1.439
Cellulose	1.27 - 1.6

Table 1
(Continued)

Name	Specific Gravity
Methyl Cellulose	1.02
2-Naphthylamine	1.061
Triphenyl carbinol	1.188
2, 2' -dithiobisbenzothiozole	1.50
2-Benzothiozolethiol	1.00
p-Benzotoluide	1.202
Zinc salt of carbamic acid	1.24
Ethyl ether cellulose	--
Crystopine	1.315
Benzene-cis-hexachloride	1.89
Indigotin (Indigo Blue)	1.35
di-1-naphthyl-mercury	2.318
Tetraphenyl	1.49
Triphenylamine	0.774
Dinaphthylmercury (α)	1.929
Tetraphenyl urea	1.222
Wood Fibers	0.4 - 1.0
Natural Clays	
Kaolinite	2.60 - 2.63
Bentonite	2.13 - 2.18

Table 1
(Continued)

Name	Specific Gravity
Plastics Nylon Acetyl Polyethylene Teflon Lucite	0.9 - 1.45
Dowex Anion Exchange Resin 21K	1.06
Gilsonite (natural hydrocarbon)	1.06
Polystyrene	1.05
Petrothene	1.01
Polythene particles	0.92
Alathon	0.96
Bakelite	1.42
Arizona Road Dust	2.65
Amberlite Anion Exchange Resin IRA-93 (Based on polystyrene)	1.04
Non-ionic Resin XAD-2	1.03
Infusorial Earth	2.33
Pumice	1.35
Bituminous Coal	1.12 - 1.35
Anthracite Coal	1.6
Fuller's Earth	2.2 - 2.4
Rayon Fibers	1.52
Styrene Divinyl Benzene Copolymer Latexes	1.14

Table 2

Compounds Selected for the Initial Developmental Investigations

Group I - Specific Gravity 0.80 - 1.00

Name	Specific Gravity
Triphenylamine	0.774
Polythene	0.92
Alathon	0.96

Group II - Specific Gravity 1.01 - 1.50

Petrothene	1.01
Methyl Cellulose	1.02
Non-ionic Resin XAD-2	1.03
Amberlite Exchange Resin IRC-50	1.04
Amberlite Anion Exchange Resin IRA-93	1.04
Polystyrene	1.05
Styrene Divinyl Benzene Copolymer Latexes	1.05
Gilsonite	1.06
Bituminous Coal	1.12 - 1.35
Nylon Fibers	1.14
Anthracene	1.25
Pumice	1.35
Tetraphenyl	1.49

Group III - Specific Gravity 1.51 - 1.99

Rayon Fibers	1.52
Anthracite Coal	1.6
Magnesium arsenate	1.788
4, 4' - dibromo-biphenyl	1.897

Group IV - Specific Gravity 2.00 - 2.65

Hexachlorobenzene	2.044
Silicon dioxide (Sand, Glass Beads)	2.1 - 2.66
Fuller's Earth	2.2 - 2.4
Infusorial Earth	2.33
Aluminum Oxide	2.42 - 2.53
Arizona Road Dust	2.65

the population. Statements concerning the mean, variance, and confidence limits of the population can be made from the sample by including a sufficient number of observations. The minimum acceptable for such calculations is 30 individual observations.[9,10]

Variance and/or standard deviation can be calculated to represent the measures of variability in the samples from the populations investigated. The sample variance is generally denoted by S^2, and its defining formula is:

$$S^2 = \sum_{i=1}^{n} \frac{(X_i - \overline{X})^2}{n-1}$$

where $X_i = X_1, X_2, \ldots, Xn$

$$\overline{X} = \text{Arithmetic Mean} = \frac{\sum_{i=1}^{n} X_i}{n}$$

n = The number of sets of values reported in each study.

The standard deviation, S, is defined to be the positive square root of the variance, S^2. Its defining formula is:

$$S = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \overline{X})^2}{n-1}}$$

To calculate the confidence interval for the mean of a normal distribution with unknown variance, the sample variance S^2 must be used as an estimation of the population variance.

The quantity $t = (\overline{X} - \mu)/(S/\sqrt{n})$, which is not the standard normal distribution, but is known as "Student's t" or "t" distribution, is used. Since the "t" distribution is symmetric about zero, a $1-\alpha$ level confidence interval for μ (the population mean) can be constructed as shown:

$$P\left[-t_{1-\alpha/2} < \frac{\overline{X}-\mu}{S/\sqrt{n}} < t_{1-\alpha/2}\right] = 1-\alpha$$

or

$$P\left[\overline{X} - t_{1-\alpha/2}\frac{S}{\sqrt{n}} < \mu < \overline{X} + t_{1-\alpha/2}\frac{S}{\sqrt{n}}\right] = 1-\alpha$$

where P = Probability

Analytical Method for Suspended Solids

The analytical method used in this study for the determination of suspended solids was the method described in the current EPA manual, Methods for Chemical Analysis of Water and Wastes, 1974, specifying a drying temperature of 103-105°C and requiring a vacuum during filtration to remove excess water.[7] Reeve Angel Type 934-A, 2.4-cm glass fiber filters and 40-ml Coors Number 27007 Gooch crucibles were used for the solids analyses. Sample sizes were 100 ml and 200 ml, depending on the concentration of suspended solids in the samples.

Solubility Testing

The first test procedure applied to the compounds being evaluated involved percent recovery of the compounds after dilution in distilled water and completion of the analytical procedure. Results included solubility, retention of the compounds on the standard glass fiber filters, and change of weight during the analytical procedure. The testing procedure involved the following steps:

1. Specific amounts of each of the test compounds were weighed on a Mettler analytical balance, which was certified on a periodic basis during the project.

2. The specific weights of compounds were diluted to volume in a 250-ml volumetric flask with distilled water.

3. The volumetric flask was shaken vigorously by hand for at least 30 seconds.

4. The total volume of sample (250 ml) was then filtered through a previously dried and tared Gooch crucible with a fiber-glass filter pad in place. The flask was thoroughly rinsed with distilled water to make certain that all of the test compound was removed from the flask.

5. The Gooch crucible containing the sample was dried at 103°C to 105°C for one hour, and then desiccated and re-weighed to determine the percent recovery of the test compound.

Results from the initial solubility testing (six repetitions) are presented as percent recovery in Tables 3 and 4. A 97 percent recovery value was selected for determining acceptance or rejection for the solubility criterion. Percent recovery characteristics of the compounds which passed the initial solubility tests are shown in Tables 5 and 6; Table 5 presents the results from 30 repetitions at 250 mg/l suspended solids, and Table 6 the results from six repetitions at 25 mg/l suspended solids.

Table 3

Compounds Failing Initial Developmental Tests

Compound	Specific Gravity	Wettability	Dispersion	% Recovery	Comments
Triphenylamine	0.774	1	1	58.7*	Floats & clings to sides of container.
Petrothene	1.01	1	1	--	Particles agglomerate in groups and do not wet or disperse well.
Methyl Cellulose	1.02	1	1	--	Methyl cellulose dissolves in water.
Amberlite Exchange Resin IRA-93	1.04	5	5	92.7	Unacceptable recovery.
Amberlite Exchange Resin IRC-50	1.04	5	5	79.2	Unacceptable recovery.
Nylon Fibers	1.14	1	1	103.1	Nylon fibers swell significantly when wetted. Some fibers agglomerate and cannot be separated by vigorous shaking. These agglomerations pour out with the sub-sample and yield percent recoveries greater than 100.
Anthracene	1.25	1	1	67.3*	Floats and clings to sides of container.
Tetraphenyl	1.49	2	2	91.6*	Floats and settles in clumps.
Magnesium Arsenate	1.788	5	5	90.3	Unacceptable recovery, clogs filter pad.
4, 4' - dibromo-biphenyl	1.897	2	2	82.2*	Particles agglomerate into large clumps.
Hexachlorobenzene	2.044	2	2	88.3*	Some floats and clings to sides of container.
Aluminum oxide	2.42-2.53	5	1	93.5	Unacceptable recovery. Difficult to disperse because of high specific gravity.

*Minimum of one or two percent recovery observations.

Table 4

Compounds Passing Initial Developmental Tests

Compound	Specific Gravity	Wettability	Dispersion	% Recovery	Comments
Rayon	1.52	5	5	97.1	Looks excellent.
Non-ionic Resin XAD-2	1.04	3	4	98.1	Floats, but will disperse with vigorous shaking, some clingage.
Bituminous Coal	1.12-1.35	5	3	98.7	Dispersion characteristics depend on particle size.
Anthracite Coal	1.6	4	3	99.9	Dispersion characteristics depend on particle size.
Infusorial Earth	2.33	5	5	100.5	Looks excellent.
Sand	2.1-2.66	5	3	99.9	Dispersion characteristics depend on particle size.
Glass Beads	2.1-2.66	5	3	99.6	Dispersion characteristics depend on particle size.
Polythene	0.92	4	3	99.4	Floats, but will disperse with vigorous shaking, some clingage.
Alathon	0.96	3	3	--	Floats in groups but will disperse with vigorous shaking.
Polystyrene	1.05	4	4	100.1	Particles settle and disperse with vigorous shaking.
Pumice	1.35	5	5		Particles settle and disperse with vigorous shaking.
Fuller's Earth	2.2-2.4	5	5	99.3	Looks excellent.
Arizona Road Dust	2.65	5	4	100.5	Looks excellent.

Table 5

Percent Recovery Tests for Compounds Passing the Initial Screening Tests (250 mg/l) at 30 Repetitions

Compound	Specific Gravity	% Recovered
Polythene	0.92	99.4
XAD-2	1.03	98.3
Polystyrene	1.05	100.1
Bituminous Coal	1.12 - 1.35	98.3
Pumice	1.35	99.5
Rayon	1.52	98.6
Anthracite Coal	1.6	99.9
Infusorial Earth	2.33	99.7
Sand	2.1 - 2.66	100.1
Glass Beads	2.1 - 2.66	99.4
Arizona Road Dust	2.65	100.5
Fuller's Earth*	2.2 - 2.4	99.3

*6 repetitions at 250 mg/l

Table 6

Percent Recovery Tests for Compounds Passing the Initial Screening Tests (25 mg/l) at 6 Repetitions

Compound	Specific Gravity	% Recovered
Polythene	0.92	102.9
XAD-2	1.03	98.8
Polystyrene	1.05	99.4
Bituminous Coal	1.12 - 1.35	99.1
Pumice	1.35	100.6
Rayon	1.52	95.7
Anthracite Coal	1.6	99.0
Infusorial Earth	2.33	99.8
Sand	2.1 - 2.66	102.0
Glass Beads	2.1 - 2.66	98.7
Arizona Road Dust	2.65	100.8

Wettability and Dispersion Testing

The second test procedure applied to each compound involved wettability and dispersion tests to determine if the compounds were hygroscopic, showed any tendency to cling to the sides of the sample containers, or floated or settled to the extent that good dispersion could not be obtained with vigorous shaking in distilled water. Visual observations using empirical scales of measurement were employed to check the degree of clinging of the compounds to the sides of the containers, the degree of wettability, and the degree of dispersion of the compounds in water. The scales of one through five in the following tabulation were used to determine the degree to which the compounds were wetted and dispersed in water:

1 - Poor Wettability	1 - Poor Dispersion
2 - Fair Wettability	2 - Fair Dispersion
3 - Acceptable Wettability	3 - Acceptable Dispersion
4 - Good Wettability	4 - Good Dispersion
5 - Excellent Wettability	5 - Excellent Dispersion

Results of the wettability and dispersion observations are presented in Tables 3 and 4. A scale measurement of 3 (acceptable wettability and dispersion) or better was selected for acceptance. Scale measurements of 1 and 2 indicated rejection of the test compounds.

Flocculation Testing

Compounds meeting the previously-defined design criteria were subjected to mixing tests with other compounds to evaluate the flocculation characteristics of various combinations of the compounds under investigation. Visual observations using the following empirical scales of measurement were used to check the flocculation characteristics of the various mixtures:

1. POOR. Particles agglomerate readily and remain in groups.
2. FAIR. Particles agglomerate readily and tend to remain in groups.
3. ACCEPTABLE. Particles agglomerate to a small degree but break apart with shaking.
4. GOOD. Particles agglomerate to a very small degree but break apart very easily.
5. EXCELLENT. No agglomeration of particles.

All compounds passed the flocculation testing when mixed with other compounds, with the exception of XAD-2 and polythene, which flocculated when mixed (rating 2).

Rayon fibers tended to entrap other particles, especially XAD-2, coal, and pumice. Rayon entrapped air bubbles and floated when vigorously mixed, but would settle with gentle agitation.

Abrasion Testing

No acceptable method for measuring the abrasion and impact resistance of the compounds to breaking up into smaller particles was found in the literature. However, a test procedure involving a Burrell Wrist-Action Shaker (made by Burrell Corporation of Pittsburgh, PA) seemed appropriate for abrasion and impact resistance testing. The procedure for simulation of a "worst case" was as follows:

1. Initial particle size was determined by the previously-mentioned ASTM method.

2. For each compound, three 10-gram samples and six 500-mg samples were weighed out on the balance.

3. The weighted compounds were transferred to nine 250-ml volumetric flasks.

4. The nine volumetric flasks were placed on the Burrell Wrist-Action Shaker for a 24-hour period at a scale setting of 4 (on a scale of 0-10, with 10 being the most vigorous shaking obtainable).

5. The six flasks containing 500-mg samples were diluted to volume with distilled water and filtered to determine the percent recovery of the compounds under investigation.

6. Particle size was again determined for the compounds in the three flasks with the 10-gram samples, for comparison with the initial particle size.

The test compounds were vigorously shaken in the dry state to simulate conditions of storage and shipping, because it was anticipated that the compounds would be packaged in the dry state and that the impact and abrasion forces would be greater in that condition. The results of the abrasion testing are presented in Table 7. No compounds subjected to abrasion testing were rejected on the basis of abrasion and impact resistance.

Evaluation of Sub-Sampling

Mixing Technique--

Mixing techniques for sub-sampling stated in the contract to be investigated included the following:

1. Shaking the samples vigorously by hand.
2. Magnetic stirring of samples.
3. Blade agitation stirring of samples.
4. Blending of samples.

However, the last two mixing techniques were eliminated because they would introduce physical constraints in the subsequent requirement to dilute the sample to volume in a volumetric flask.

Table 7

Abrasion Testing Results

Compound	Percent Recovery After Abrasion Testing	Uniformity Coefficient (Cu) Before	After
Arizona Road Dust	99.8	1.50	1.45 1.45 1.45
Glass Beads	99.9	1.53	1.44 1.53 1.62
Sand	99.9	1.72	1.76 1.72 1.75
Anthracite Coal	98.4	2.95	2.83 3.66 3.46
Bituminous Coal	99.0	4.04	3.53 4.00 4.16
Pumice	99.5	1.88	2.02 2.11 2.06
Rayon	98.9	Particle Size Before Mean = 1.63 mm St. Dev. - 0.573 Var. = 0.328	

Rayon Particle Size After Shaking

#1 Sample

Mean = 1.19
St. Dev. = 0.430
Var. = 0.185

#2 Sample

Mean = 1.214
St. Dev. = 0.458
Var. = 0.210

Rayon fibers form small tight balls when shaken in the abrasion test. These balls are extremely difficult to re-suspend by shaking. A blender was used to re-suspend the rayon fibers.

Rayon Particle Size After Shaking and Blending Approx. 60 sec.

Mean = 0.963
St. Dev. = 0.468
Var. = 0.219

Random samples were taken for particle sizing with a microscope. 100 particles were counted.

The evaluation of mixing techniques and subsequent recovery involved withdrawing 100 ml sub-samples from one-liter volumetric flasks. The compounds in the flasks were mixed vigorously by hand and by magnetic stirrer for comparison. The sub-sample volumes of 100 ml were poured into a graduated cylinder or withdrawn with a volumetric pipet. The results of the mixing tests are presented in Table 8.

Recovery Tests (Analytical Method)--

Precision and accuracy data for the analytical method were determined by withdrawing appropriate sample volumes (100 ml or 200 ml) from the full-volume diluted solids samples into one-liter volumetric flasks in conformance with the EPA method for determining suspended solids.[7] The volumetric flasks were shaken vigorously by hand, and the subsequent sub-samples were poured into graduated cylinders as contrasted to mixing with magnetic stirring bars and withdrawing the sample with a pipet. The average percent recovery was higher, and the range of percent recovery and therefore the standard deviation were less for shake-and-pour with graduated cylinders than for magnetic stirring and pipeting.

Extensive tests were performed on the compounds that appeared to be acceptable for packaging (pumice, rayon fibers, and Fuller's earth). The results of the mixing tests and sub-sample recovery for these compounds and for anthracite coal are presented in Table 9. The percent recovery of sub-sampling appeared to be a function, to some extent, of particle size because the dispersion characteristics were somewhat dependent on particle size as exhibited in Tables 8 and 9.

An experiment was conducted to determine the effects of particle size on the percent recoveries obtainable on Fuller's earth. Fuller's earth was screened at specific particle sizes, weighed, and diluted with distilled water to one liter in volumetric flasks, and the sub-samples were removed by pouring into graduated cylinders for percent recovery analyses. The results of these particle-size investigations are presented graphically in Figure 1.

Selection of Compounds for Packaging

The results of the feasibility testing and the observations of the specific compounds indicated two possible alternative methods available for sample handling without modifying the 1974 EPA solids analysis procedure. One alternative was to package compounds over the full range of specific gravities (0.8-2.65) and particle sizes (0.01-4.5 mm) and to filter the total sample for the solids analysis. Compounds suitable for this procedure included the following:

1. Pumice
2. Rayon Fibers
3. Infusorial Earth
4. Fuller's Earth
5. Sand
6. Glass Beads
7. Arizona Road Dust

Table 8

Evaluation of Mixing Techniques Using One-Liter Volumetric Flasks
(Vigorous Shaking and Mixing with Magnetic Stirring Bars) 250 mg/l

Compound	Mixing	Repetitions	Specific Gravity	Particle Size	Average % Recovery	Range % Recovery
XAD-2	Shake & Pour (Graduated Cylinder)	6	1.03	100-200 microns	102.1	79.7 - 145.7
	Magnetic Stirring (Graduated Cylinder)	6			91.0	28.5 - 106.7
Polystyrene	Shake & Pour (Graduated Cylinder)	6	1.05	1.7 mm	24.3	0 - 80.8
Bituminous Coal	Shake & Pour (Graduated Cylinder)	6	1.12 - 1.35	<150 microns	83.4	77.6 - 90.5
	Magnetic Stirring (Graduated Cylinder)	6			96.2	85.5 - 107.5
Pumice	Shake & Pour (Graduated Cylinder)	6	1.35	<75 microns	90.8	88.6 - 92.0
	Magnetic Stirring (Graduated Cylinder)	3			92.2	89.7 - 95.7
	Pipet	3			82.3	76.8 - 91.9
Rayon Fibers	Shake & Pour (Graduated Cylinder)	6	1.52	1.6 mm	93.6	91.4 - 95.0
	Magnetic Stirring (Graduated Cylinder)	6			92.3	89.9 - 93.5
Anthracite Coal	Shake & Pour (Graduated Cylinder)	6	1.6	<150 microns	60.8	48.8 - 78.5
	Magnetic Stirring (Graduated Cylinder)	6			163.1	68.3 - 290.5

Table 8
(continued)

Compound	Mixing	Repetitions	Specific Gravity	Particle Size	Average % Recovery	Range % Recovery
Fuller's Earth	Shake & Pour (Graduated Cylinder)	6	2.2 - 2.4	<150 microns	81.9	78.2 - 84.4
	Magnetic Stirring (Graduated Cylinder)	6			87.3	83.9 - 90.6
Fuller's Earth	Shake & Pour (Graduated Cylinder)	3	2.2 - 2.4	<45 microns	92.9	92.6 - 93.3
	Magnetic Stirring (Graduated Cylinder	3			91.8	89.4 - 94.7
	Pipet	3			94.3	92.7 - 97.3
Infusorial Earth	Shake & Pour (Graduated Cylinder)	6	2.33	8 microns	93.1	88.5 - 97.4
	Magnetic Stirring (Graduated Cylinder)	6			102.3	99.7 - 103.7
	Pipet	6			91.7	89.1 - 95.2
Glass Beads	Shake & Pour (Graduated Cylinder)	6	2.61 - 2.65	0.9 - 1.23 mm	0	0
	Magnetic Stirring	6			1.0	0 - 2.0
Arizona Road Dust	Shake & Pour (Graduated Cylinder)	6	2.65	100-200 microns	7.1	5.2 - 9.9
Arizona Road Dust	Shake & Pour (Graduated Cylinder)	6	2.65	20-40 microns	77.9	73.9 - 79.6

All sub-samples were 100 ml samples.

Table 9

Mixing Test Results (Shake & Pour) with Graduated Cylinder

Compound	Sub-Sample Size (ml)	Concentration mg/L Proposed	Concentration mg/L Actual Average	Particle Size	Average % Recovery	Range % Recovery	Standard Deviation (Percent)	Variance	Standard Deviation (mg)	Variance	Standard Deviation (mg/L)	Variance
Pumice*	100	100	104.8	< 45 microns	90.7	88.8 - 92.6	1.90	3.61	0.23	0.05	1.99	3.96
Pumice*	100	250	257.3	< 45 microns	92.4	91.3 - 93.1	0.96	0.93	0.26	0.07	2.47	6.10
Pumice*	100	1000	1007.8	< 45 microns	90.3	89.3 - 90.3	1.05	1.10	1.06	1.11	10.58	111.98
Rayon**	200	27	27.4	0.5 mm	98.3	85.3 -107.5	4.69	22.01	0.18	0.03	1.29	1.65
Rayon**	200	50	52.1	0.5 mm	97.3	90.0 -102.5	3.47	12.05	0.32	0.10	1.81	3.27
Rayon**	100	100	102.0	0.5 mm	97.4	93.9 -101.4	2.12	4.51	0.18	0.03	2.16	4.68
Rayon**	100	250	251.9	0.5 mm	97.5	95.1 -100.0	1.23	1.52	0.29	0.08	3.10	9.60
Rayon**	100	855	854.9	0.5 mm	99.1	97.1 -101.5	1.31	1.71	0.67	0.46	11.20	125.42
Rayon*	100	1000	1011.8	0.5 mm	98.8	97.8 - 99.7	0.95	0.91	1.00	1.00	9.61	92.39
Fuller's Earth**	200	27	27.5	< 45 microns	99.6	93.8 -105.6	3.80	14.48	0.10	0.01	1.05	1.09
Fuller's Earth**	200	50	52.6	< 45 microns	94.5	88.4 -101.4	2.91	8.47	0.28	0.08	1.53	2.34
Fuller's Earth**	100	100	111.7	< 45 microns	96.4	92.3 - 99.1	1.88	3.55	0.22	0.05	2.10	4.41
Fuller's Earth**	100	250	254.2	< 45 microns	96.7	93.4 - 99.2	1.63	2.65	0.46	0.21	4.14	17.17
Fuller's Earth**	100	855	855.5	< 45 microns	96.0	93.0 - 98.5	1.67	2.80	1.39	1.93	14.29	204.10
Fuller's Earth*	100	1000	1007.9	< 45 microns	95.3	94.8 - 95.6	0.46	0.21	0.51	0.26	4.67	21.50
Nylon Fibers***	100	250	250.8	0.5 mm	103.1	98.7 -109.0	2.57	6.61	0.56	0.32	6.45	41.55
Anthracite Coal**	200	27	28.5	< 150 microns	92.3	76.6 - 106.0	7.88	62.03	0.42	0.18	2.25	5.04
Anthracite Coal**	100	250	253.4	< 150 microns	77.4	66.9 - 85.1	4.21	17.74	2.94	8.66	10.67	113.81
Anthracite Coal**	100	855	861.5	< 150 microns	74.4	65.7 - 81.9	4.26	18.11	3.77	14.25	36.70	1346.88
Anthracite Coal****	100	250	251.3	44 - 150 microns	69.1	63.9 - 72.5	3.65	13.29	1.07	1.13	9.17	84.13

* 3 repetitions

** 30 repetitions

*** 12 repetitions

**** 6 repetitions

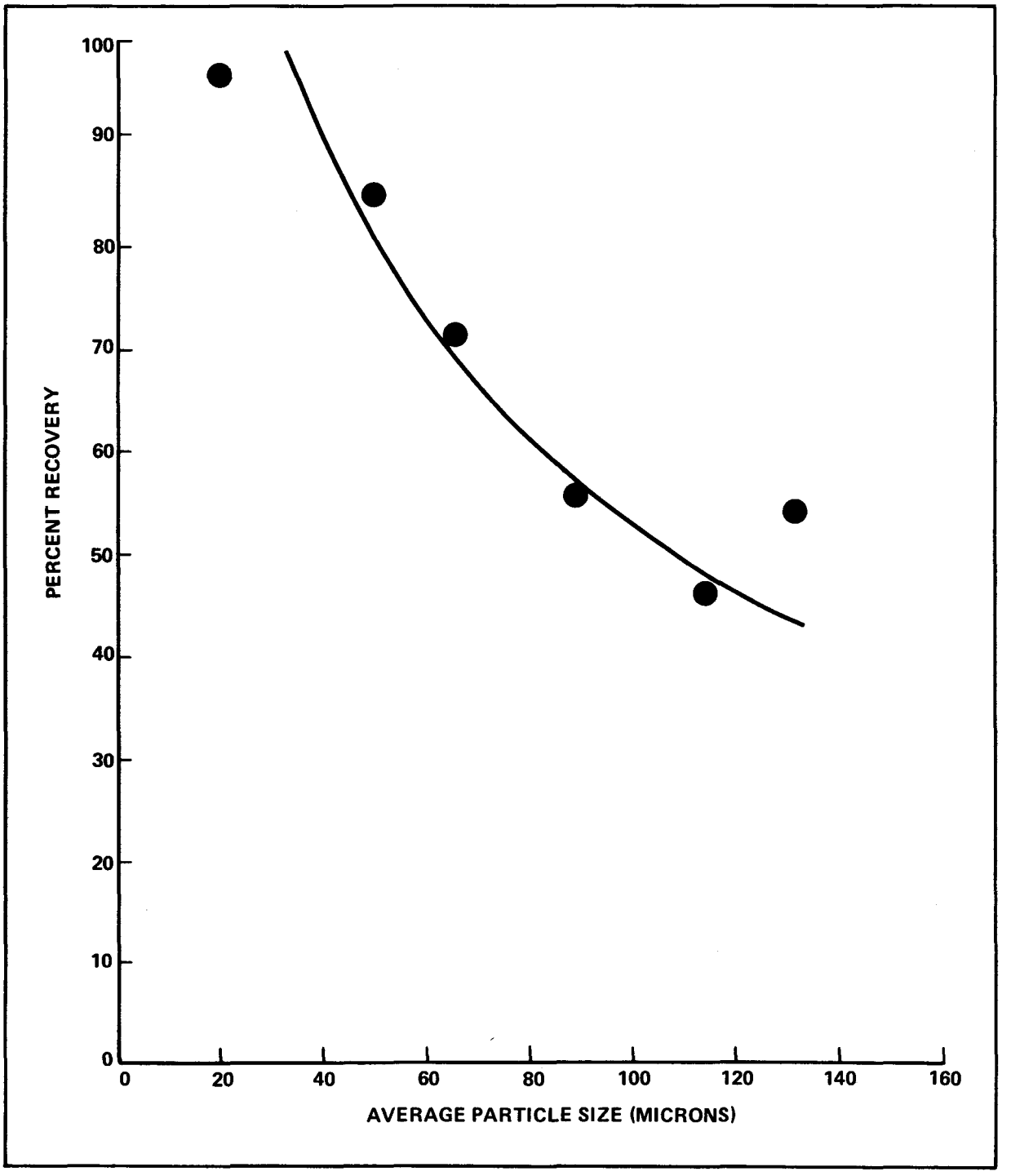

Figure 1. Percent recovery versus particle size for Fuller's earth.

However, this procedure would yield information only on the analytical technique, with no regard to sub-sampling technique and procedure. The second and best alternative would be to package one or a few compounds in the very small particle-size fractions that would allow accurate sub-sampling.

The compounds indicated as acceptable for the second alternative were:

1. Pumice
2. Rayon Fibers
3. Fuller's Earth

The second alternative was chosen for selecting the compounds to be packaged as quality control and performance evaluation samples. Since pumice and rayon fibers were close in specific gravity and since the particle size of each can be varied, the rayon fibers were chosen because of higher percent recoveries during sub-sampling. Consequently, the following compounds shown as compounds passing the initial developmental tests in Table 4 were rejected for packaging:

	Compound	Comments
1.	Non-ionic Resin, XAD-2	Erratic sub-sampling recovery.
2.	Bituminous Coal	Low sub-sampling recovery.
3.	Infusorial Earth	Low sub-sampling recovery. Same specific gravity as Fuller's Earth.
4.	Sand	Low sub-sampling recovery.
5.	Glass Beads	Low sub-sampling recovery.
6.	Polythene	Floats, yielding erratic sub-sampling.
7.	Alathon	Floats, yielding erratic sub-sampling.
8.	Polystyrene	Low sub-sampling recovery.
9.	Pumice	Approximately same specific gravity as rayon fibers.
10.	Arizona Road Dust	Low sub-sampling recovery.

Styrene divinyl benzene (specific gravity - 1.05) was also rejected for packaging because of initial cost (approximately $45 per 15 ml) and handling problems. It is packaged in liquid form and cannot be dried without changing properties.

The compounds selected for packaging included the following:

Fuller's Earth - particle size <45 microns

Rayon Fibers - 1.5 denier*, 0.5 mm long

Anthracite Coal- particle size <150 microns

*A measure of the fineness of rayon yarn. 1.5 denier yarn weighs 1.5 grams per 9,000 meters.

Fuller's earth and rayon fibers met all the design criteria established for the synthetic suspended solids quality control and performance evaluation samples. The reason for packaging anthracite coal as a suspended solids quality control sample was to show some of the problems involved in the suspended solids determination procedure.

Biodegradability Testing

The biodegradability tests were conducted by measuring biochemical oxygen demand (BOD) in BOD bottles according to the procedure described in the 13th Edition of Standard Methods for the Examination of Water and Wastewater.[11] The distilled water, phosphate buffer solution, magnesium sulfate solution, calcium chloride solution, ferric chloride solution, sodium sulfite solution, and dilution water were prepared according to the instructions. Therefore, adequate nutrients were supplied by the prepared solutions added to the dilution water.

Each bottle was seeded with microorganisms from a standard house seed taken from the West Chester, PA municipal sewage treatment plant. The BOD bottles were incubated at 20°C for a five-day period. The dissolved oxygen (DO) was determined with a membrane electrode DO meter, which was checked and standardized before use.

Each of the three compounds selected for packaging was tested at three concentrations for the five-day BOD analyses. Standards of glucose/glutamic acid were also incubated for the five-day period. Five milliliters of the standard were placed in each bottle yielding five-day BOD's of 180 to 204 mg/l. Each condition of compounds and standard was repeated six times.

Fuller's earth and anthracite coal showed no biodegradation in the BOD_5 test procedures, but the rayon fibers showed slight biodegradation at the two highest concentrations. The results of the biodegradability investigations are presented in Table 10.

Stability Testing

Fuller's earth and anthracite coal were packaged mechanically at three weights for the stability testing; the rayon fibers were hand weighed and packaged at the same three weights.

The weights of the packaged compounds were then checked at 30, 60, and 90 days for comparison with the packaged weights at zero time to evaluate any possible problems associated with biodegradability, volatility, or any change of any other characteristics during storage of the packaged compounds.

Comparison of the 30-, 60-, and 90-day data to time zero weights disclosed no significant evidence of increasing weight loss with time. Weight loss of rayon fibers during both the 60-day and 90-day investigation periods was less than the weight loss during the 30-day period. Weight loss of Fuller's earth during the 90-day period was less than the weight loss during

Table 10

Results of Biodegradability Tests
(Five-Day BOD Tests) for Rayon, Fuller's Earth, and Anthracite Coal

Sample Description	Date	Avg. D.O. Depletion (mg/L)	Range D.O. Depletion (mg/L)	Avg. BOD_5 (mg/L)	Range BOD_5 (mg/L)
Glucose/Glutamic Acid Standard (5 ml/bottle)	11/6/75	3.2	3.0 - 3.3	189	180 - 198
Rayon-Low Concentration		< 0.1	-	-	-
Rayon-Medium Concentration		0.8	0.7 - 1.0	2.4	2.1 - 3.0
Rayon-High Concentration		3.1	2.9 - 3.5	9.3	8.7 - 10.5
Fuller's Earth-Low Concentration		< 0.1	-	-	-
Fuller's Earth-Medium Concentration		< 0.1	-	-	-
Fuller's Earth-High Concentration		< 0.1	-	-	-
Glucose/Glutamic Acid Standard (5 ml/bottle)	11/7/75	3.3	3.2 - 3.4	196	192 - 204
Anthracite-Low Concentration		< 0.1	-	-	-
Anthracite-Medium Concentration		< 0.1	-	-	-
Anthracite-High Concentration		0.2	0.2	0.6	0.6

All Tests conducted at 6 Repetitions.

the 60-day period. Weight loss of anthracite coal was random over the 30-, 60-, and 90-day investigation periods and exhibited no trends or patterns in any of the data.

Statistical analyses were performed to test the significance of the difference of the weights observed at 90 days compared to weights at zero time by the t test.[12] The difference in the weights observed did not prove to be significant at $\alpha = 0.01$, with one exception, Fuller's earth at the lowest weight packaged.

Percent Recovery Tests on Mixtures of Fuller's Earth, Anthracite Coal, and Rayon Fibers

Rayon fibers and Fuller's Earth, rayon fibers and anthracite coal, and Fuller's earth and anthracite coal were each mixed at a 50:50 weight ratio at a total concentration of 250 mg/l per sample. In addition, rayon fibers, Fuller's earth, and anthracite coal were mixed at a 33.3:33.3:33.3 weight ratio at a total concentration of 250 mg/l per sample. Each of these mixtures was added to a one-liter volumetric flask and brought to volume with distilled water. The samples were then shaken vigorously by hand, and a sub-sample of 100 ml was poured into a graduated cylinder for suspended solids analysis to determine the percent recovery. The results of these mixing and percent recovery investigations are presented in Table 11.

The percent recovery obtainable by the analytical method for the mixture of rayon fibers and Fuller's earth was excellent (average 99.5 percent). However, the percent recoveries for the other mixtures containing anthracite coal were all less than 90 percent. The percent recoveries obtained were as would be expected without flocculation reactions. Very good percent recoveries were obtainable with rayon fibers and Fuller's earth alone, whereas very poor recoveries were obtainable with anthracite coal alone.

Table 11

Compound Mixing Tests Results

Compound	Ratio	Sub-Sample Size (ml)	Total Concentration (mg/L)	Average % Recovery	Range % Recovery	Standard Deviation (Percent)	Variance	Standard Deviation (mg)	Variance	Standard Deviation (mg/L)	Variance
Rayon Fibers+ Fuller's Earth	50:50	100	250	99.5	99.0 - 100.1	0.50	0.25	0.12	0.015	1.20	1.44
Rayon Fibers+ Anthracite Coal	50:50	100	250	87.4	85.5 - 91.7	2.28	5.19	0.55	0.31	5.50	30.25
Fuller's Earth+ Anthracite Coal	50:50	100	250	79.2	77.3 - 82.1	1.67	2.80	0.47	0.22	4.70	22.09
Rayon Fibers+ Fuller's Earth+ Anthracite Coal	33.3:33.3:33.3	100	250	84.1	82.8 - 85.2	0.79	0.62	0.45	0.20	4.50	20.25

Rayon Fibers at 0.5 mm particle size
Fuller's Earth at < 45 micron particle size
Anthracite Coal at < 150 micron particle size
6 repetitions

SECTION V

PACKAGING OF COMPOUNDS - PHASE II

PREPARATION OF COMPOUNDS FOR PACKAGING

Fuller's earth, rayon fibers, and anthracite coal were carefully prepared for packaging. These compounds were purchased in a pure state and washed several times in distilled water to remove any soluble impurities that might have been present. These compounds were then dried overnight at 103-105°C to remove moisture. The rayon fibers were then ready for packaging, because they were previously sized. The Fuller's earth and anthracite coal were ground or milled and sized to the correct sizes. These compounds were then dried again to remove any moisture that might have been picked up during the sizing operations. These compounds were then ready for packaging.

PACKAGING REQUIREMENTS

Number and Type of Samples

The numbers and types of packages of the compounds (rayon fibers, anthracite coal, and Fuller's earth) chosen for packaging by EPA are presented below:

Compound	Weight (mg)	No. of Vials
Rayon Fibers	Low	1,111
	Medium	1,111
	High	1,111
	Sub-Total	3,333
Fuller's Earth	Low	2,711
	Medium	1,111
	High	1,111
	Sub-Total	4,933
Anthracite Coal	Low	1,111
	Medium	311
	High	311
	Sub-Total	1,733

This combination of weights and number of vials at each weight was chosen for two principal reasons:

1. To obtain variety in the packaged weights.

2. To package fewer vials of anthracite coal, because the main purpose of this compound is simply to indicate some of the problems of the test procedure.

Packaging Technique

Weston used a semi-automatic packaging instrument (Perry Model LM-14 Accofil Portable Powder Filling Machine) to package the anthracite coal and Fuller's earth. However, the instrument could not be used to package the rayon fibers accurately, because of their fluffy physical nature. Therefore, the rayon fibers were hand weighed and packaged. The actual packaged weights of rayon fibers were recorded for each vial.

Strict quality control procedures were employed during preparation of the compounds and the packaging vials and caps, and during the actual packaging operations. Rigid laboratory standards concerning equipment, vials and caps, and housekeeping practices were enforced. Vials were thoroughly washed in dilute hydrochloric acid solution, rinsed, dried at 103-105°C, and desiccated before being used for packaging. All the packaging operations were conducted in a temperature- and humidity-controlled balance room. Calibration of the packaging instrument and quality control checks on the instrument and hand-packaged vials were performed on a routine basis, as described in the sub-section on quality control.

Instrument Packaging Precision

Anthracite coal and Fuller's earth were employed in packaging operations with the packaging instrument to determine the precision of packaging at specified weights. As the Fuller's earth and anthracite coal were final packaged, approximately five percent of the packaged vials were checked for packaging precision. Quality control spot checks of each type of package combination were made, and these quality control checks were included in the precision measurements of the instrument. Packaging precision was shown to be within ±3 percent of the average packaged weights.

Quality Control

Rigid quality control procedures were utilized throughout the packaging operations to insure confidence in the precision and accuracy of the packaged compounds weights. The packaging instrument was calibrated on a daily basis, and approximately five percent of the packaged vials were checked for packaging precision at the time of packaging. The hand-packaged weights were also checked by re-drying approximately five percent of the packaged vials and determining the packaged weights for comparison with the initial recorded weights.

Additional vials of rayon fibers, anthracite coal, and Fuller's earth were packaged at each weight combination for quality control spot checks of the packaged weights by a senior chemist who had not been associated with the packaging of the vials. The samples for quality control checks were randomly selected from each packaged combination of compounds and weights. These quality control checks again established that the packaged weights were within ± 3 percent of the average packaged weights.

SECTION VI

DISCUSSION

STATISTICAL EVALUATION OF DATA

Calculations were conducted to determine the precision of the analytical results that could be expected by an analyst performing the suspended solids test from the packaged compounds (rayon fibers, Fuller's earth, and anthracite coal).

With the concurrence of the EPA statistician, the packaging precision data for Fuller's earth and anthracite coal, and the analytical method precision data were combined into a single precision statement. To develop this precision statement for each compound weight, it was necessary to compare the results of the packaging and the analytical method at the same weights; however, the packaged weights and those used in the analytical method evaluation were different. To put the data on the same basis, a plot of the standard deviations of the analytical method for each compound versus the corresponding weights was made. These plots (Figure 2) indicated straight-line relationships. Consequently, the standard deviations for analytical method at the packaged weights of the compounds were read directly from these graphs.

The combined standard deviation of the analytical method and packaging was then calculated as follows:

$$S = \sqrt{S_1^2 + S_2^2}$$

where S = Combined standard deviation

S_1 = Standard deviation of analytical method

S_2 = Standard deviation of packaging

The percent recovery of the analytical method was also determined for the actual packaged weights of compounds by plotting the average percent recovery versus average concentration. The combined precision of analytical method and packaging was then determined around the sample mean, which was taken to be the average packaged weight multiplied by its respective average percent recovery.

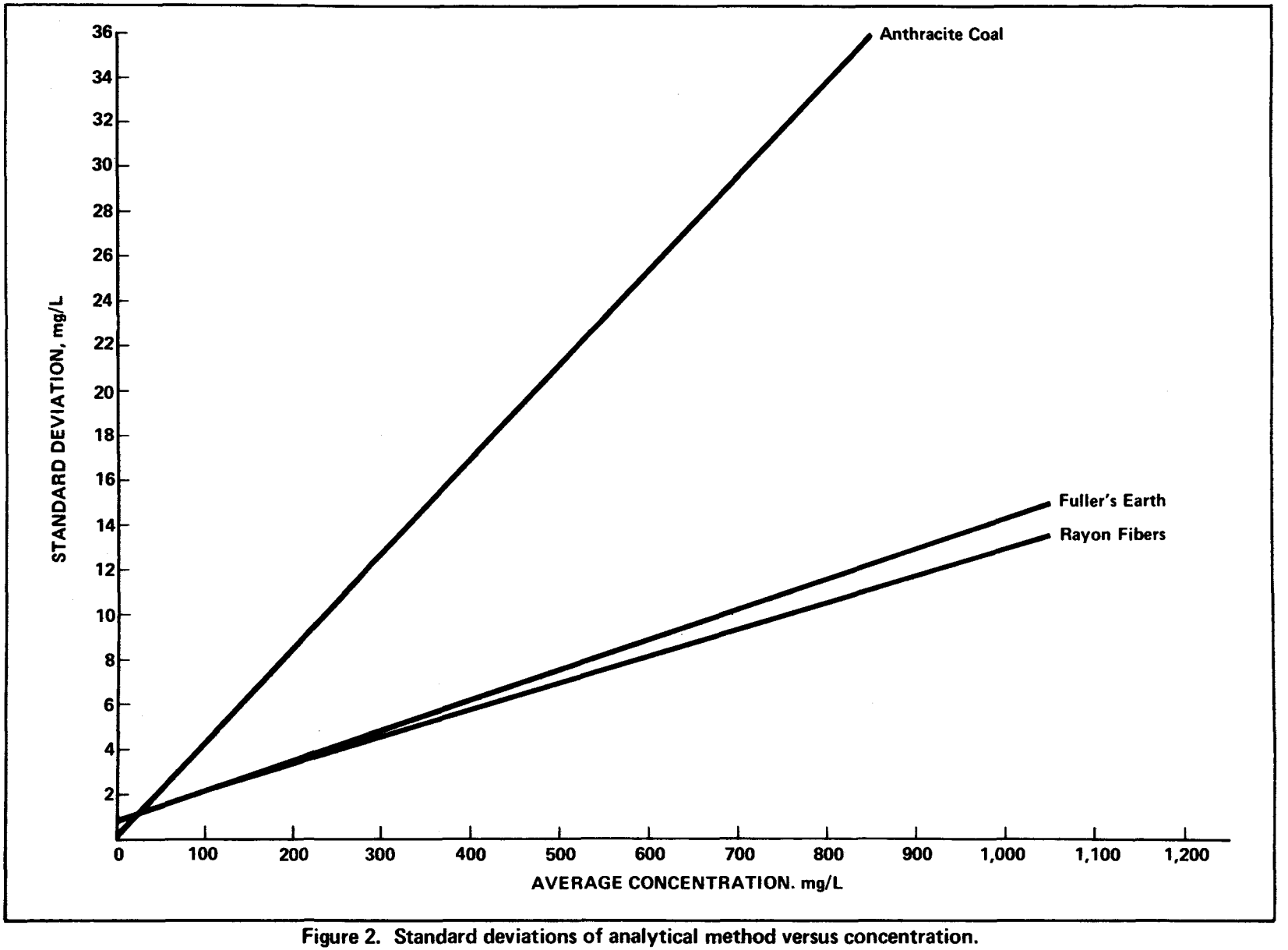

Figure 2. Standard deviations of analytical method versus concentration.

SYNTHETIC SUSPENDED SOLIDS ANALYSIS

Recent investigations by EPA's Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, OH, and an independent study by the National Council of the Paper Industry for Air and Stream Improvement, Inc. were undertaken to examine the effects of procedural differences on measured presence of non-filterable residue (suspended solids).[13] Significant variations in non-filterable solids capture were found to result from the following: type of filtering medium used; type of filter holder apparatus used to support the medium; volume of sample filtered (volume to filter area); and post-washing procedure.

These findings led to the recommendation that a uniform test procedure be developed and employed for measuring non-filterable residue or suspended solids.

Standard procedures for transferring the pckaged compounds to one-liter sample containers (volumetric flasks) and for performing the suspended solids analysis are recommended in the following paragraphs.

Preparation of Quality Assurance Samples for Suspended Solids Analysis

The following procedure is recommended for preparation of the quality assurance samples for suspended solids analysis:

1. Tap contents (compounds) to bottom of vial.

2. Remove rubber-lined seal from vial (being careful to avoid losing any particles that may be clinging to the rubber lining).

3. Clean the rubber lining by flushing thoroughly with distilled water into a one-liter volumetric flask (Class A glassware); with anthracite coal, rubbing the lining with a glass rod may be required for complete removal. Clean until no particles remain attached to lining.

4. Pour contents of the vial into the volumetric flask through a glass funnel.

5. Continually rinse and transfer remaining contents of the vial into the volumetric flask until no particles remain in vial.

6. Dilute to one-liter mark.

Analytical Method for Quality Assurance Suspended Solids (Non-Filterable Residue)

The procedure and the apparatus recommended for performing the suspended solids analysis are as stated in the EPA Manual of Methods for Chemical

Analysis of Water and Wastes.[7] The procedure for determining suspended solids concentrations is as follows:

1. Insert recommended glass fiber filter disc into bottom of suitable Gooch crucible (4.7 cm or 2.2 cm), with the wrinkled surface of the disc facing upward.[14]

2. Apply vacuum to the assembled filtration Gooch crucible in the filter apparatus.

3. While vacuum is applied, wash the disc with three successive 20-ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.

4. Disconnect the vacuum, remove the Gooch crucible with the filter paper in place, and dry it in an oven at 103-105°C for one hour.

5. Remove the Gooch crucible from the oven, and place it in a desiccator until cooled to room temperature. The Gooch crucible can be stored in the desiccator until needed, but should be weighted immediately before use.

6. Place the previously dried, desiccated, and tared Gooch crucible with the glass fiber filter disc into the filtering apparatus, and begin suction.

7. Shake the sample diluted to 1-liter volume vigorously by hand for at least 30 seconds.

8. Rapidly transfer the 100-ml or 200-ml subsample by means of a graduated cylinder to the Gooch crucible.

9. Rinse the graduated cylinder thoroughly with distilled water, pouring the water through the Gooch crucible (minimum of three successive 20-ml volumes of distilled water).

10. Carefully remove the Gooch crucible from the crucible adaptor.

11. Dry in the drying oven at 103-105°C for one hour.

12. Cool in a desiccator for 30 minutes.

13. Weigh the Gooch crucible after the 30-minute desiccation period.

The suspended solids (non-filterable residue) concentration of the quality assurance sample can then be calculated as follows:

$$\text{Suspended Solids, mg/l} = \frac{(A-B) \times 1{,}000}{C}$$

where A = Weight of Gooch crucible plus solids (residue)
B = Weight of Gooch crucible
C = ml of sample filtered

SECTION VII

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16. ABSTRACT

A two phase study was conducted to develop a synthetic suspended solids sample for use as quality control check and performance evaluation within environmental monitoring laboratories. The first phase consisted of a feasibility study to determine compounds that exhibited the optimum physical and chemical properties for synthetic suspended solids samples, and the second phase involved production of suspended solids samples in individual containers.

Compounds investigated that met all the design criteria included rayon fibers and Fuller's earth. A total of 10,000 quality control samples consisting of rayon fibers, Fuller's earth and anthracite coal were packaged for completion of the project. Anthracite coal was packaged to demonstrate the variability in the sub-sampling step of the suspended solids test method with certain types of solids.

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