PLAINTIFF'S EXHIBIT

API-87

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May 29, 1973

TO: Committee on Medicine and Environmental Health

SUBJECT: "Collaborative Study of the Microscopic Counting of Asbestos Samples."

Gentlemen:

Enclosed is a copy of a report prepared by R. S. Brief, Esso Research and Engineering Company, on a collaborative study by seven COMEH laboratories on a procedure for counting asbestos particles which was specified by NIOSH and endorsed by OSHA.

In his cover letter to our office, Mr. Brief summarized: "As you will read, the method produces widely variant results on asbestos samples evaluated by either the same laboratory or different laboratories. As expected, the variation between laboratories is greater than within a laboratory. The results suggest that efforts to control work environments to below 5 fibers greater than 5 μm/cc (and 2 fibers greater than 5 μm/cc after July 1, 1976) will be fraught with difficulty because of the analytical procedure.

The laboratories who participated have been sent a copy of this report with their own laboratory identified. None of the laboratories have been told of the number identification of the others, except laboratory number I which is indicated in the report to be our own, the Medical Research Division of Esso Research and Engineering Company.

Although I have individually thanked the collaborators, I would again wish to express my appreciation to all those who helped bring this project to a successful, albeit disheartening, conclusion."

Mr. Brief and the participants in this study are to be commended for this valuable service they have performed for the industry.

Sincerely yours,

James M. McNerney

HUT-113903

ABS-058003

SC-API-4565

# COLLABORATIVE STUDY OF THE MICROSCOPIC COUNTING OF ASBESTOS SAMPLES

By

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HUT-113904

[illegible] OF THE MICROSCOPIC [illegible]

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## Background Information[1]

The term "asbestos" refers to any of six naturally occurring crystalline [illegible] actinolite, [illegible] anthophyllite, [illegible] the deposits of [illegible]. These minerals display a wide range of chemical compositions, as is indicated in Table 1.

The several types of asbestos were formed by the metamorphosis of serpentine and amphibole minerals, both classes of which contain silica. Chrysotile, which is a hydrated silicate of magnesia, is the principal crystalline form of serpentine. Over 90% of the world's asbestos production comes from chrysotile and it is the asbestos material used almost exclusively in the United States. The remaining five types of asbestos are crystalline forms of amphibole [illegible] is associated with riebeckite. Amosite is the only asbestos of grunerite [illegible] than 2 percent.

Electron microscopy reveals that the smallest fibrous subdivision of a chrysotile fiber, called a fibril, has an average outside diameter of 0.034 micrometer (μm). Further, it has been shown that the chrysotile fibril is a hollow tube, rather than a solid cylinder, with an average inside diameter of 0.018 μm. A suggested model views the chrysotile fiber as a tightly packed collection of fibrils, the interiors and interstices of which are filled with [illegible] the majority of dry-milled asbestos fibers each contain many fibrils, smaller numbers of fibers composed of only one or two fibrils are always present; a considerable number of these fibers of smaller diameter are found in asbestos dust.

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TABLE [illegible]

[illegible] of Asbestos [illegible]

| Property | [illegible] | Crocidolite | [illegible]site | [illegible] | Tremolite | [illegible]tinolite |
|---|---|---|---|---|---|---|
| Chemical Formula | [illegible] | $Na_2Fe_3Si_8O_{22}(OH)_2$ | [illegible] $_{22}(OH)_2$ | [illegible] | $Ca_2Mg_5Si_8O_{22}(OH)_2$ | [illegible] $_5Si_8O_{22}$ |
| Essential Composition | [illegible] | Silicate of sodium and iron with some water | [illegible] of iron [illegible] nesium; [illegible] iron than [illegible] yllite | [illegible] | Calcium and magnesium silicate with some water | [illegible] magnesium [illegible] silicate, [illegible] up to [illegible] |
| Percentage Chemical Composition | | | | | | |
| $SiO_2$ | [illegible] | 49. to 53. | [illegible] | [illegible] to 5[illegible] | 51. to 62. | -- |
| $MgO$ | [illegible] | 0. to 3. | [illegible] | [illegible] to 3[illegible] | 0. to 30. | -- |
| $FeO$ | [illegible] | 13. to 20. | [illegible] | [illegible] to [illegible] | 1.5 to 5.0 | -- |
| $Fe_2O_3$ | [illegible] | 17. to 20. | -- | -- | -- | -- |
| $Al_2O_3$ | [illegible] | -- | [illegible] | [illegible] to [illegible] | 1.0 to 4.0 | -- |
| $H_2O$ | [illegible] | 2.5 to 4.5 | [illegible] | [illegible] to [illegible] | 0. to 5.0 | -- |
| $CaO$ | [illegible] | -- | -- | -- | 0. to 18. | -- |
| $Na_2O$ | [illegible] | 4.0 to 8.5 | -- | -- | 0. to 9. | -- |
| $CaO$ and $Na_2O$ | | -- | [illegible] | -- | -- | -- |
| Color | [illegible] | Blue | Gray, yellow to dark brown | Yellowish, [illegible], grayish [illegible] | Gray-white, greenish, yellowish, bluish | [illegible] |
| Texture | [illegible] | Soft to harsh | [illegible] some-[illegible] liable | [illegible] | Generally harsh, sometimes soft | [illegible] |
| Luster | [illegible] | Silky to dull | [illegible] some-[illegible] pearly | [illegible]treous to pearly | Silky | [illegible] |
| Refractive Index | [illegible] | 1.7 pleochroic | [illegible] | 1.61± | 1.61± | 1.63± [illegible] pleochroic |

Workmen handling or working with asbestos and asbestos-containing materials have experienced adverse health effects. Inhalation of the asbestos fibers has been found to be [illegible] of disease. Thus, to minimize the effects [illegible] the [illegible] airborne concentration of asbestos fibers [illegible]

Of the various ways to sample and analyze suspended asbestos fibers in air, with particular relation to the respiratory hazard they create, the generally recognized best method today is collection on membrane filters and analysis by microscopic counting at 400-450X magnifications under phase-contrast illumination. This procedure was collaboratively tested and is the subject of this report.

[illegible]

has arisen from studies of asbestos miners in South Africa and from asbestos fabricators in Great Britain. Epidemiologic studies of unionized [illegible] also reveal an increased incidence of these diseases among these workers. It is important to recognize, however, that working with asbestos-containing materials per se does not necessarily produce the conditions noted. For example, in the petroleum industry a medical evaluation of over one hundred insulators, with 20 or more years experience, revealed that they showed no lung impairment of any [illegible] 3000 [illegible] of exposure [illegible]

[illegible] States, the present limit established by the Occupational Safety and Health Administration (OSHA) is related to fibers longer than 5 μm (a fiber [illegible] greater than 3:1). According to OSHA, the permissible exposure to airborne concentration of asbestos fibers are shown in Table 2.

TABLE 2

| [illegible] | [illegible] Concentration of Air [illegible] | [illegible] |
|---|---|---|
| [illegible] | 5 | 10 |
| [illegible] | 2 | 10 |

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The method of measurement requires that air samples be collected on membrane filters and that the fiber counts be done at 400-450X magnifications [illegible]

[illegible]

Evaluation of the Method

Early efforts to standardize this method [illegible] in different laboratories[illegible] This was disquieting since the OSHA standard could be violated by the variability of the measurement scheme. This could create a health problem if the asbestos count was in error on the low side and an excessive requirement for controls if the counts erred on the high side. Methodology which is required as part of regulations [illegible] to be expected so that [illegible]

[illegible] the possible reasons [illegible] with mic[illegible] counting procedures. In addition, different mic[illegible] and [illegible] the filters and [illegible] fibers to be included in the count. The rules of counting are not clearly defined by the regulations; but we offer some below, based on comments made by Scheinbaum[7].

1. The fibers must fit inside or pass through two predesignated sides of the viewing area formed by a counting reticle. If the fibers pass through [illegible]

[illegible] length so that they appear to be "L", "T" or "V" shaped are counted [illegible] However, [illegible]

3. A clump or mass from which several fibers emanate are counted as one fiber.

During counting, when it is possible to separate nonasbestos fibers [illegible] especially, [illegible]

example, fiberglass fibers are long and straight with a uniform diameter, typically larger than asbestos, and with ends which appear to be fire- [illegible] their length and often have a bulbous center from which numerous fibers emanate.

Because the regulations refer to 5 μm or greater in size, the selection of this length requires another judgment. For the Porton [illegible] number 6 [illegible] μm.

Background counts must be considered because the membrane filters can contain fibers which may be subsequently counted. However, this is usually a minor problem.

[illegible] should [illegible] mounting material, and the like. Sometimes it is difficult to make prompt [illegible] was indicated.

Finally, the calculation relating the count to the air concentration involves definitive measurements of the counting area related to the filtering area and a knowledge of the total air volume of the sample. A stage micrometer is usually used to accurately define the optical counting area and the filter area must be carefully measured. The accuracy of the rotometer used during sampling must also be checked by suitable calibration methods.

[illegible] laboratories who were actively involved in assessment of asbestos fiber [illegible] samples plus one plane membrane filter to slides and distributing them to the participants on a random schedule. The mounting media contained 0.65 [illegible]

The samples were prepared by cutting a wedge-shaped piece of the membrane filter (Millipore Filter Paper Type AA) while in the filter holder (a three-piece plastic container obtained from the Millipore Corporation, Bedford, [illegible]. The piece was removed from the filter with a scalpel [illegible] was placed over the filter wedge. Slight pressure on the cover slip achieved contact between it and the mounting media. The specimen was rendered transparent by absorption and transfer of the mounting media into the filter. The ten specimens were prepared and counted in our laboratory within one day after preparation. The technique used was that described in the Appendix.

The specimens were then sent by mail to each of six other participants for their count of these samples. A repeat count was made in our [illegible] was provided and each laboratory was requested to calculate the respective number of [illegible] per cubic centimeter [illegible]. Details on [illegible] measurements was obtained. The results were statistically analyzed at the Esso Research and Engineering Company.

## STATISTICAL EVALUATION

The fiber concentrations obtained by the seven laboratories on the ten samples are shown in Table 3 in terms of the number of the fibers greater than 5 μm per cubic centimeter of air. [illegible] the standard deviation appears to be a function of the [illegible]. A [illegible] analysis of the data would, therefore, be overwhelmed [illegible] the higher concentrations. [illegible] the data was made with the following adjustments: 1) data from sample 2 were eliminated (this was the blank sample); and 2) the data for sample number 3 from laboratory number 5 [illegible] logarithms were used).

TABLE 3

[illegible]

| | | Sample Number | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| Laboratory Number | 1 | 53.7 | .43 | .95 | 23.7 | .95 | 43.2 | 8.6 | 21.9 | 28.5 | 50.7 |
| | 2 | 29.3 | .07 | .66 | 19.3 | .84 | 17.6 | 3.5 | [illegible] | 19.4 | 25.8 |
| | 3 | [illegible] | 2.34 | 3.0 | 17.9 | 3.0 | 33.3 | 5.1 | 13.5 | 19.2 | 26.4 |
| | 4 | 28.0 | .00 | 2.1 | 19.0 | 1.6 | 39.0 | 4.7 | 9.5 | 19.0 | 28.0 |
| | 5 | 43.2 | .00 | .00 | 23.1 | .95 | 22.8 | 2.4 | 20.4 | 18.1 | 22.3 |
| | 6 | 45.9 | [illegible] | [illegible] | 18.7 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| Standard Deviation | | 10.2 | .90 | 1.1 | 6.2 | [illegible] | 12.7 | 2.7 | 5.5 | [illegible] | 12.9 |

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## TABLE 4

### Log Transformed Data

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Laboratory Number | 1 | [illegible] | -.05129 | 3.51750 | -.05129 | 3.76584 | 2.15176 | 1.08549 | 3.34990 | 3.92591 |
| | 2 | 3.37759 | -.41552 | 2.96011 | -.17435 | 2.86790 | 1.25276 | 2.10413 | 2.91235 | 3.25037 |
| | 3 | 3.34639 | 1.09861 | 2.88480 | 1.09861 | 3.50556 | 1.62924 | [illegible] | 2.95491 | [illegible] |
| | 4 | [illegible] | [illegible] | 2.94444 | .47000 | 3.66356 | 1.54756 | 2.25129 | 2.94444 | [illegible] |
| | 5 | 3.76584 | -.73397 | 3.13983 | -.05129 | 3.12676 | .87547 | 2.01553 | 2.89591 | [illegible] |
| | 6 | [illegible] | -.94161 | [illegible] | -1.60944 | 3.27714 | .47000 | [illegible] | 3.16125 | [illegible] |
| | 7 | 3.74715 | -.96768 | 3.49651 | -.41552 | 3.99083 | 1.52606 | 2.26176 | 3.39115 | 3.97218 |
| Mean | | 3.62558 | -.18135 | [illegible] | -.10475 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |

In Table 4, the standard deviation is now less affected by the value of the mean so that the analysis of variance can properly include all the data tabulated.

Table 5 shows the analysis of variance (2-way classification) for the data in Table 4. Note that the total degrees of freedom were diminished by one to account for the substitution of 0.48 fibers/cc in place of 0.00 for sample 3, laboratory 5. The conclusions that can be drawn from Table 5 are [illegible]

1. [illegible] results on the same sample.

2. Gross differences in sample concentration are observed with high probability.

[illegible] for fiber concentrations can be derived from the error mean square (.20655) as follows:

$$1.96\sqrt{.20655} = .89078$$

The 95% confidence interval for results is, therefore,

$$\text{Lower Limit} = \frac{X}{2.437}$$

[illegible]

For example, a fiber concentration of 5 fibers > 5 um/cc measured [illegible] could be reported (95% of the time) as 2.1 to 12.2 fibers [illegible] laboratories; and a fiber count of 2 fibers > 5 um/cc could be reported as 0.8 to 4.8 fibers > 5 um/cc. To assure that the ceiling value of 10 fibers > 5 um/cc is not exceeded, these data also suggest that no value should exceed 10/2.437 or 4.1 fibers > 5 um/cc.

[illegible]

[illegible]

Without Sample 2 and Adjustment of Sample 3 (Laboratory 5)

| [illegible] | Sum of Squares | Degrees of Freedom | Mean Square | F |
|---|---|---|---|---|
| Laboratories | 3.93353 | 6 | .65559 | 3.17[illegible] |
| Samples | 130.29369 | 8 | 16.28671 | 78.8[illegible] |
| Error | 9.70773 | 47 | .20655 | |
| Total | 143.93495 | 61 | -- | |

[illegible]

In an attempt to [illegible] the [illegible] error within [illegible] laboratory, [illegible] on the [illegible] days [illegible] A [illegible] sign-rank test of these results [illegible] that the amount of time between analyses did not influence results. In addition, a parametric analysis of these paired results was done after a logarithmic transformation of the data and rejection of sample number 2.

TABLE 6

Repeat Results in Laboratory 1

Fibers > 5 μm/cc

| | | |
|---|---|---|
| 2 | .48 | .48 |
| 3 | .95 | 1.9 |
| 4 | 33.7 | 31.7 |
| 5 | [illegible] | [illegible] |
| 6 | [illegible] | [illegible] |
| 7 | [illegible] | [illegible] |
| 8 | 21.9 | 20.9 |
| 9 | 28.5 | 35.4 |
| 10 | [illegible] | [illegible] |

[illegible] in Table [illegible] [illegible] [illegible]

[illegible] laboratory and, therefore, [illegible] [illegible] [illegible] for all laboratories. However, using the same interpretive method described previously, the results in this laboratory, at least, are considered to [illegible] [illegible] when results are multiplied and divided by [illegible]. The geometric methodology for determining acceptable limits of fiber concentrations seems reasonable, although disheartening, because of its magnitude.

TABLE 7

[illegible]

| Source of Variation | Sum of Squares | Degrees of Freedom | Mean Square | F |
|---|---|---|---|---|
| | [illegible] | 1 | [illegible] | [illegible] |
| | [illegible] | [illegible] | [illegible] | [illegible] |
| Error | [illegible] | 8 | .12771 | |
| Total | 34.31957 | 17 | | |

**Significant at > 99% Probability

[illegible]

[illegible]

The blank filter reported as sample number 2 (if one excludes the results from laboratory number 3) would be, on average, about 0.2 fibers per cc. It follows, then, that the sealed filters from Millipore do not contribute significantly to the evaluation of asbestos fiber concentrations in air.

In 7 industrial laboratories using mounted specimens of 2 asbestos samples collected on membrane filter paper and one blank filter.

A geometric methodology for determining confidence limits of fiber concentration suggest that 95% of the time a result in one laboratory

The blank filter (Millipore Type AA three-piece filter assembly) contributes little to the overall count, averaging about 0.2 fibers

## REFERENCES


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4. Asbestos Textile Institute. Measurement of Airborne [illegible]

5. National Institute for Occupational Safety and Health. Criteria for a Recommended Standard -- Occupational Exposure to Asbestos. HSM 72-10267. 1972.

6. [illegible] October 31, [illegible]

[illegible] York, N. Y. [illegible] 1, 1972.

## ACKNOWLEDGMENT

The assistance and cooperation of the participating laboratories is acknowledged with sincere appreciation for the voluntary efforts of the staff members who represented the following organizations.

| Name | Organization |
|---|---|
| [illegible] | [illegible] |
| [illegible] | [illegible]<br>Houston, Texas |
| [illegible] | [illegible]<br>New York, New York |
| W. W. Davis | Atlantic-Richfield Company<br>Harvey, Illinois |
| [illegible] | [illegible] |
| [illegible] | [illegible] |
| R. W. Volk | Phillips Petroleum Company<br>Bartlesville, Oklahoma |

Thanks are also due to Messrs. H. T. Oakley, Esso Research and Engineering Company, and R. Spireas, University of North Carolina for their assistance in evaluating the data.

APPENDIX

## APPENDIX

### Sampling and Evaluation Procedure[5]

**Principles of Sampling**

A dust sampling procedure must be designed so that samples of [illegible]

[illegible] a sample representative of airborne dust, which is likely to enter the subject's respiratory system, it is necessary to position a collection apparatus near the nose and mouth of the subject [illegible]

The concentration of dust in the air to which a worker is exposed will vary, depending upon the nature of the operation and upon the type of work performed by the operator and the position of the operator relative to the source of the dust. The amount of dust inhaled by a worker can vary daily, [illegible]

[illegible] will affect the concentration of dust the worker inhales since the different tasks usually result in exposure to different concentrations. The percentage can be determined from work schedules and by observation of work routines.

The daily average weighted exposure can be determined by using the following formula:

[illegible]

[illegible]

[illegible] sampling procedure, [illegible] workers' breathing zones for periods from 4 to 8 hours, [illegible] the evaluation of average exposures for a half or full 8-hour [illegible] and recommended procedure. Furthermore, dust exposures of a more normal work pattern result from the use of personal samplers. In evaluating daily exposures, samples should be collected as near as possible [illegible] breathing zones.

The sample should be collected on a 37-millimeter Millipore Type AA filter mounted in an open-face filter holder. The holder should be fastened to the worker's lapel and air drawn through the filter by means of a battery-powered personal sampler pump. The filters are contained in plastic filter holders and are supported on pads which also aid in controlling the distribution of air through the filter. To yield a more uniform sample deposit, the filter-holder face-caps should be removed. Sampling flow rates from 1.0 liter per minute (lpm) up to the maximum flow [illegible]

[illegible] fibers the statistical error resulting from the random distribution of the fibers must be kept to an acceptably [illegible] a count of 100 fibers in a sample would have a standard deviation of [illegible] or 10 fibers or ± 10%. Thus [illegible] would be approximately [illegible] standard deviations or ± 20%. Since the 37 mm filter has an effective collecting area of [illegible] $m^2$ and if the projected field [illegible]

[illegible] various limits in various sampling times:

| Sampling Time Minutes | Flow Rate lpm | Number of Fields [illegible] Fibers 0.2 fibers/ml | 2.0 fibers/ml | 10 fibers/ml |
|---|---|---|---|---|
| 10 | 2 | 4350 | 435 | 91 |
| 15 | 2 | 2860 | 286 | 58 |
| 30 | 2 | 1430 | 143 | 29 |
| 90 | 1 | 1000 | 100 | 20 |
| 90 | 2 | 500 | 50 | 10 |
| 240 | 1 | 260 | 26 | 7 |
| [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| | [illegible] | [illegible] | [illegible] | [illegible] |

[illegible] interfere with the accuracy of the count.

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[illegible] on these restraints, i.e., number of fields to be counted and maximum number of fibers per field, acceptable [illegible] the various limits are [illegible] in the above table.

The following conclusions may be drawn from this analysis:

(1) The short-term limit should be for a period of at least 15 minutes and preferably [illegible]

[illegible]

over periods of from 90 to 480 minutes.

As many fields as required to yield at least 100 fibers should be counted. In general the minimum number of fields should be 10 and the [illegible]

Mounting Sample

The mounting medium used in this method is prepared by dissolving 0.05 g of membrane filter per ml of 1:1 solution of dimethyl phthalate and [illegible]

[illegible] is [illegible] the filter with a [illegible] and forceps and places dust-side-up on the drop of mounting solution. A No. 1-1/2 coverslip, carefully cleaned with [illegible], is placed over the filter wedge. Slight pressure on the coverslip achieves contact between it and the mounting medium. The sample may be examined as soon as the mount is transparent. The optical homogeneity of the resulting mount is nearly perfect, with only a slight background granularity under phase contrast, which disappears within one day. The sample should be counted within two days after mounting.

[illegible] concentration of asbestos fibers greater than [illegible] with phase-contrast [illegible] "high-dry" [illegible] suitable for this determination. [illegible] [illegible] the [illegible] particle field serves to define the counting area of the field. Twenty fields located at random on the sample are counted and total asbestos fibers longer than [illegible] are recorded. Any particle having an aspect ratio of three or greater is considered a fiber.

(1) $\frac{\text{Filter area (mm}^2\text{)}}{\text{Field area (mm}^2\text{)}} = K$

(2) $\frac{\text{Average net count X K}}{\text{Air volume sampled (ml)}} = \text{fibers/ml}$

For example, assume the following: area of the filter used was 855 mm$^2$, [illegible] area of one field under the Porton [illegible] was [illegible] 30 minutes [illegible]

[illegible]

0.005 mm$^2$

$\frac{10 \text{ fibers} \times 171{,}000}{2{,}000 \text{ ml/min} \times 30 \text{ min}} = 9.5 \text{ fibers/ml}$

Calibration of Personal Sampler

The accuracy of an analysis can be no greater than the accuracy [illegible] instrument, more frequent calibration may be necessary.

[illegible], pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, a 1-liter burette or wet-test meter should be used. [illegible] using the same type of tubing used in the personal sampling unit. Each pump must be calibrated separately for each type of filter used, if, for example, it has been decided to use a filter with a different pore size. The burette should be set up so that the flow is toward the narrow end of the unit.

[illegible] in the assembly procedure to insure adequate seals at the joints and that the length of connecting tubing be kept at a minimum. Calibration should be done under the same condi- [illegible] of pressure, temperature and humidity as will be encountered. The rotameter should be used only in the field as a check if the diaphragm or piston pumps are not equipped with pulsation dampeners. The pulsating flow resulting from these type pumps causes the rotameter to give results which are not as accurate as that obtained with a burette or wet-test meter. Calibration can be accomplished with any of the other standard [illegible]

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