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A STATIC SYSTEM FOR MEASURING THE SIZE DISTRIBUTION OF VARIFORM PARTICULATES

by

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SUMMARY OF RESULTS

In a previous publication a semi-automatic instrument, the Zeiss Particle Analyzer TGZ3, was used to determine length distribution of asbestos fibres (1). A further study was made of fibre orientation in suspension before sizing. (2) In the interim, two new commercial developments took place which increased the feasibility of a relatively rapid system of measuring odd-shaped particles. This system was developed, standard samples of asbestos were measured, and the precision of the results obtained were compared.

It should be possible to utilize this system for almost any type of particulates, but it is especially useful for difficult systems such as fibres.

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INTRODUCTION

The Mines Branch has initiated a project on asbestos that includes measurement of fibre length. The Zeiss Analyzer and a technique developed at the Mines Branch (1) were found satisfactory for the measurement of lengths of chrysotile asbestos fibres. A description of this instrument was included in a published report (1). Two new commercial developments suggested a much better approach to measuring fibre length and diameter of various-shaped particulates. This report describes the development of a system incorporating these two developments (techniques) and includes the results obtained.

EXPERIMENTAL PROCEDURES

Equipment and Experimental Technique

The two mentioned commercial developments were:

(1) Image-splitting devices that could be used to determine the diameter of fibres and other particulates. One of the units included an adapter that caused the image to oscillate. This oscillation simplified and increased the speed of particle sizing.

(2) A rapid photography unit that permitted a relatively dry print to be obtained in a matter of minutes after a negative was prepared. In this case, the negative was an oil suspension of iodine-stained fibres. This was first described in a Mines Branch report (1).

A more detailed description of the individual parts of the system and their function is given below, in sequence of operation.

1. Iodine Staining

A few grains of iodine are placed in a tubular, 1 x 3-in. plastic bottle, and a fine stainless screen is placed on top of the iodine. A representative sample of asbestos is placed on the screen and then the bottle is stoppered. The sample is jiggled for a few minutes. This is sufficient to stain the fibre uniformly.

2. Dispersion Apparatus

The apparatus for dispersing the fibre, shown in Figure 1, consists of:

(a) Randolph or similar pump;
(b) High cylindrical glass or plastic container;
(c) Magnetic stirrer;
(d) Heavy mineral oil; obtainable from any pharmaceutical wholesaler.

3. Photographic Apparatus

(a) An enlarger, 2.25 x 3.25 or 4 x 5-inch; an ordinary lens will suffice;
(b) enlarging easel;
(c) commercial (Accurapid) processor for rapidly producing a photoprint;
(d) No. 4 extra hard single-weight enlarging paper;
(e) 2 gallons of activator chemical;
(f) 2 gallons of stabilizer chemicals.

The enlarger was modified to accommodate a special mask used with the asbestos suspension.

4. Orientation Apparatus*

The apparatus for orientation of the fibre is shown in Figure 2 and consists of:

(a) High-voltage transformer, 10,000 volts - this can be bought locally;

(b) plastic cell (plastic Petri dishes) - these cells are easily modified with electrodes;

(c) a 10-watt autotransformer.

The wiring is very simple, but should be safe because high voltages are used.

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*The orientation technique has been patented.

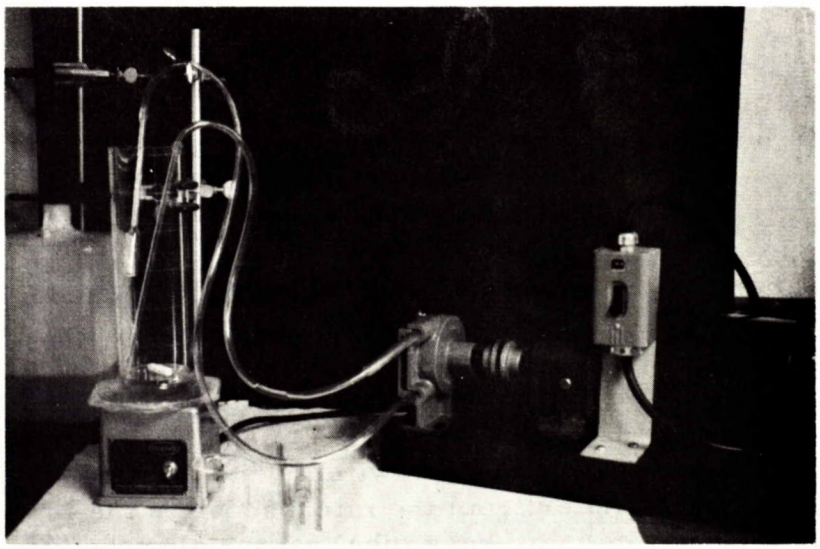


Figure 1. Dispersing Apparatus.

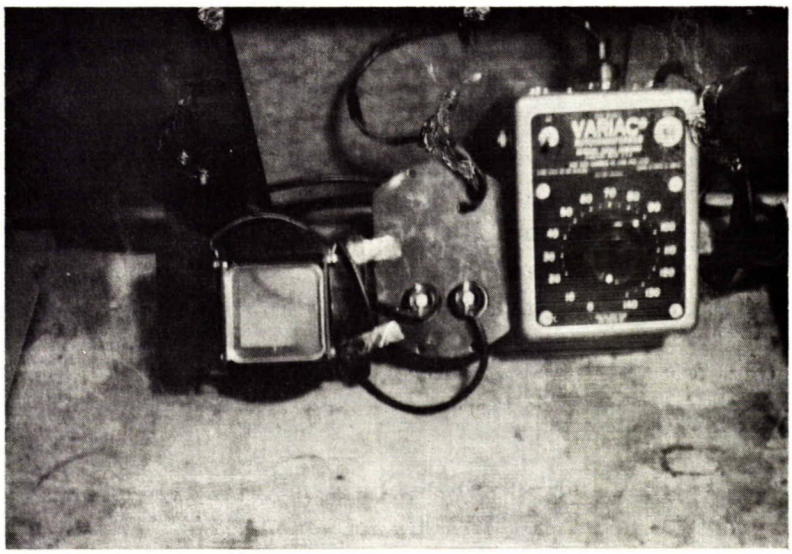


Figure 2. Orientation Apparatus.

The autotransformer regulates the voltage from the high-voltage transformer. The leads from the high-voltage transformer are attached permanently to the mask; and the electrodes from the plastic cell, in place, contact the mask terminals (Figure 2).

5. Sizing Apparatus

(a) Zeiss particle analyzer - this instrument is used here for measuring the length of the fibre;

(b) microscope and adapter, image-splitting device and "go-no-go" adapter; this group is used to measure fibre diameter - any microscope with a wide field of view will serve; the adapter from the microscope to the image-splitting device is a brass tube that couples the two units;

(c) microscope gauge sub-divided in 0.01 and 0.1-mm divisions; this is used simply to obtain a standardizing factor;

(d) base to move photograph in the X and Y directions for known fixed distances; this is home-made.

Figure 3 shows the Zeiss analyzer with a photograph in place and Figure 4 shows the image-splitting eyepiece, which has been adapted to the microscope. A photograph is shown in place on a platform movable in both X and Y directions. Figure 5 shows the detail of the adapter that couples the image-splitting eyepiece to the microscope. The adapter is made of brass and machined to obtain a good fit.

The white spot in the photograph is obtained by placing a machined 1/4 x 1/8-in. round brass rod in the square plastic petri-dish and focussing the enlarger on the top of the brass metal. This facilitates photographing the oriented fibres in a narrow plane. Total enlargement of the photograph can be calculated by measuring the diameter of the white spot.

Samples

A sample of milled chrysotile asbestos was used as a standard. This was obtained from a Canadian source.

Figure 3. Zeiss Particle-Size Analyzer.

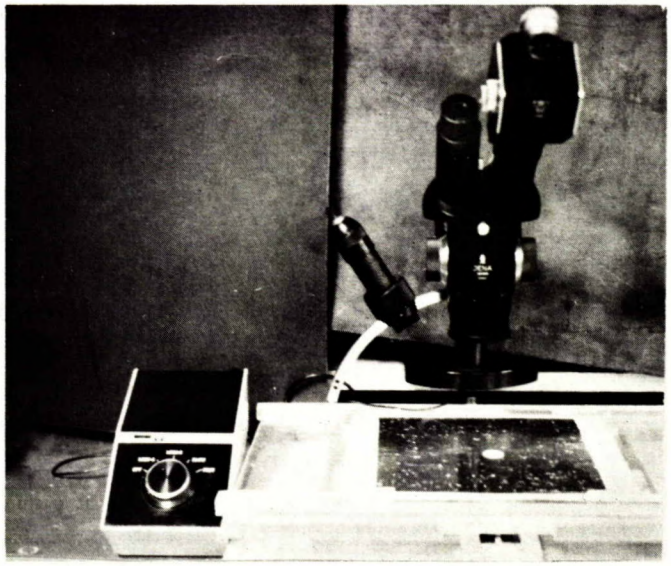


Figure 4. Image-Splitting Eye Piece and Movable Platform.

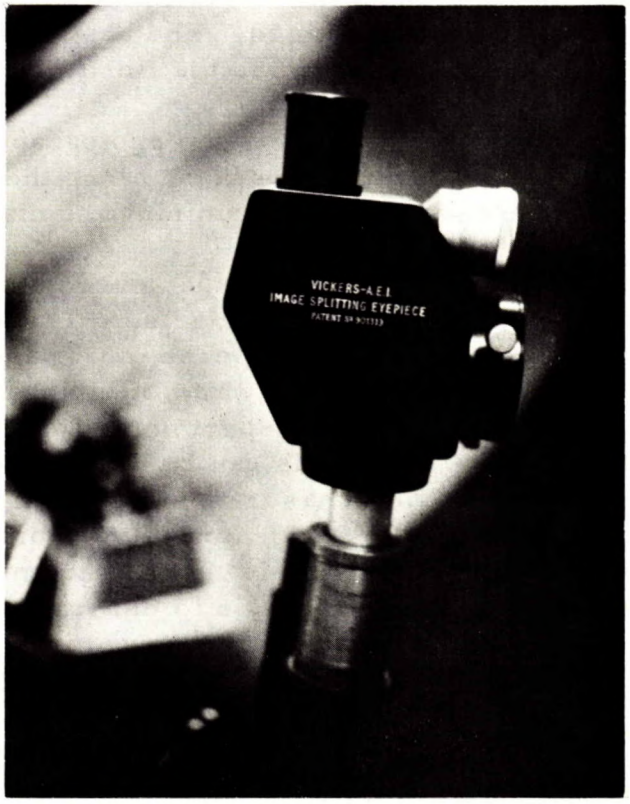


Figure 5. Details of Adapter to Couple the Eye Piece to the Microscope.

Preliminary Experiments

A sample of asbestos was stained as noted on page 1. The fibre was then dispersed by placing 0.2 g in 1 litre of mineral oil and the suspension passed through the pumping system shown in Figure 1.

After dispersion of the fibre, a representative sample of the suspension was taken, during pumping, by slowly moving the intake tube vertically in the suspension from the bottom to the top. The delivery tube discharges the sample into a 100-ml beaker. An aliquot was then taken from the beaker with a pipette, and the sample was then discharged into the sample cell. The cell was filled only halfway to avoid spillage. The 1/4 x 1/8-in. brass rod was placed in the cell to facilitate focussing during photo enlarging.

The cell was placed in the photographic enlarger which was then focussed on the top of the brass plug. Orientation of the fibres was accomplished next, using the apparatus shown in Figure 2. Finally the special photographic paper was exposed for approximately 20 seconds and then passed through the Accurapid processor.

The photograph was placed on the Zeiss Particle-Size Analyzer and the fibre length distribution determined. The photograph was then placed on the movable platform to determine the fibre diameter distribution.

After some preliminary experiments the photograph was divided into five sections and only four horizontal scans, along the lines separating the sections, were found necessary for determining the diameter distribution.

Normally, very little difficulty was experienced using the asbestos fibres; however, difficulty of dispersion may arise. There are numerous surface active agents available that will disperse most particulates, and those suitable for silicates will be best for chrysotile asbestos. Some fibres or particulates will require more experimentation to find a suitable dispersant. Freeze-dry methods can be used to keep the material in dispersed form and ready for the measurement system. Since we were not concerned with deterioration of the strength of the fibre, acids could be used, if necessary, to aid dispersion. However, care must be taken that the fibre does not change in diameter due to the various dispersing agents, especially acids. The same precautions would be required for all kinds of materials.

The problem of dispersion has never been considered the limiting factor in this study at this time.

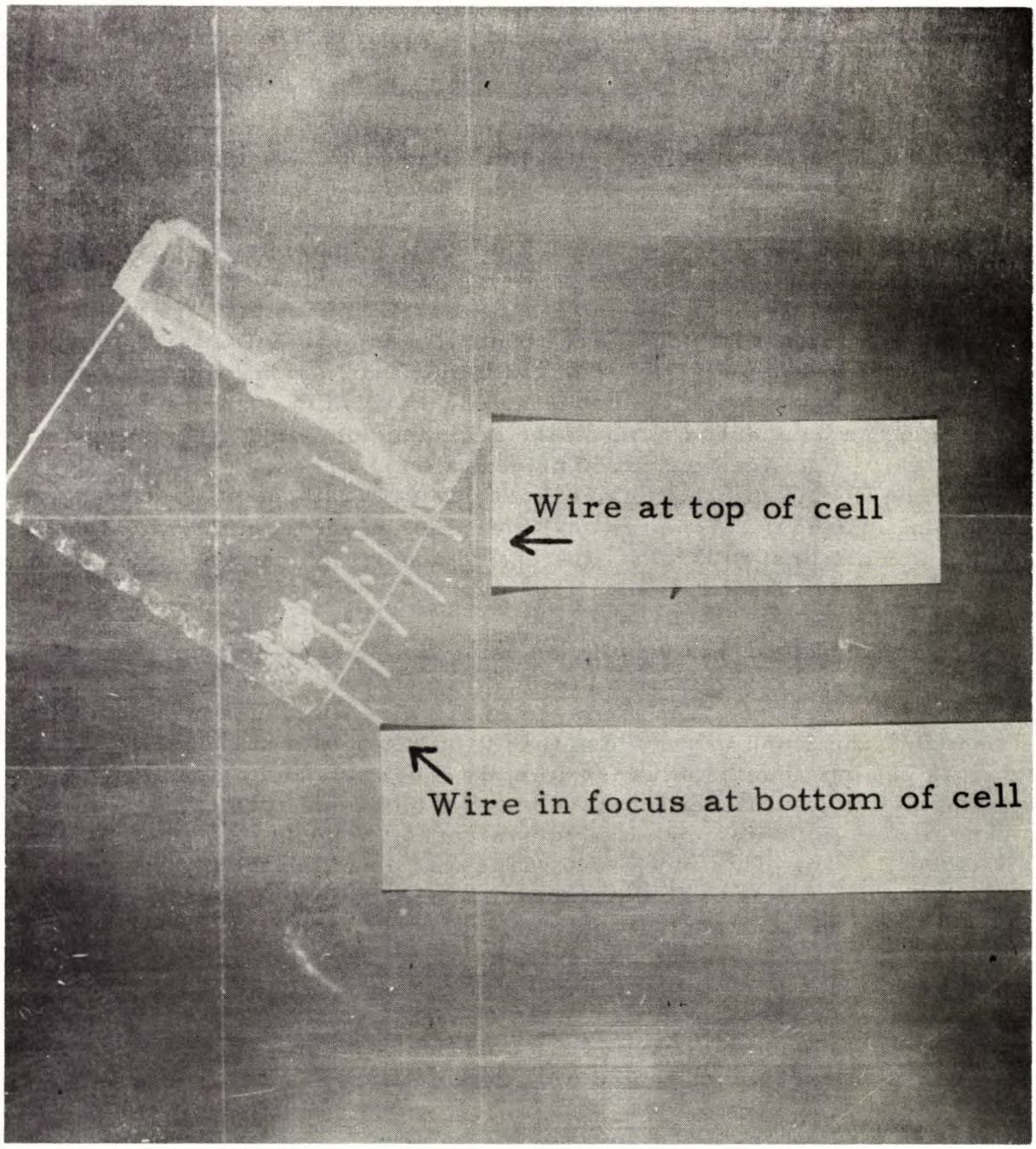


Figure 6. Experiment to Determine the Variation of Wire Diameter at Different Heights in the Cell.

Additional Notes on Measurement Techniques

After the apparatus has been set up, a single photograph ready for sizing can easily be obtained in about 10 minutes. Several photographs can, of course, be obtained at a faster rate.

It is not necessary to align the fibres for measurement, but the count is faster when this is done, particularly when the image-splitting device is used for diameter-of-fibre measurement.

Using the Vickers procedure, the range of diameter desired is set. The oscillation of the image is started and the photograph is moved from right to left on the platform (Figure 4). All fibres that are within the range (the images coincide) are counted, using the Zeiss instrument as the counter. The platform is moved in the Y direction exposing a new area of the photograph and the process repeated. In this way, the photograph can be scanned to count the required number of fibres to give a statistically significant count. The next step is to change the size range of the image-splitting device and to count the fibres of this size range. In sequence, starting from either the largest to smallest diameter or from the smallest to largest, the entire photograph can be scanned and the fibre diameter distribution can be recorded.

An experiment was performed using fine wire to determine whether there was any variation in fibre diameter due to the variation in distance from the lens. The diameter of the wire was measured with a micrometer and found to vary less than 0.001 inch over its length. This wire was cut into lengths of approximately equal size and placed in plastic material at different heights with the clean ends protruding. The thickness of the plastic block was equivalent to the depth of the plastic cell, about 1/2 in. This block with wires was placed in the plastic cell, with mineral oil filling the balance of the space, then placed in the enlarger and a "rapid" photographic enlargement made (Figure 6). The diameters of the wires in the photograph, as measured by microscope, varied by only 1.5 per cent.

Since the lens is always focused on the same spot (brass plug) the error of measurement should be very small.

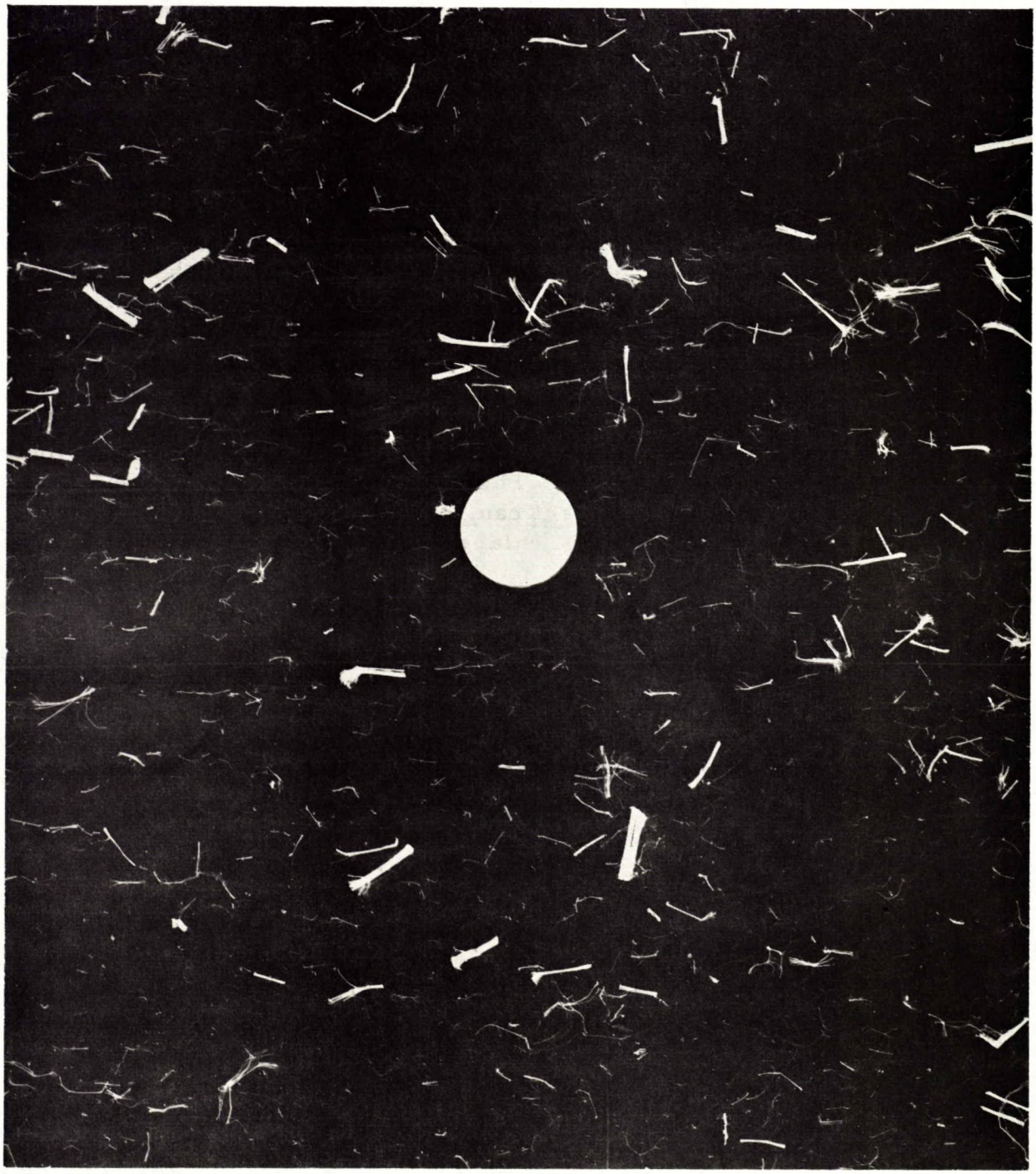


Figure 7. Enlargement made by the Rapid Photographic Process.

RESULTS AND DISCUSSION OF LABORATORY EXPERIMENTS

Reasonably sharp photographs were obtained within less than 10 minutes. The enlargement size could readily be measured from the enlarged spot on the photograph as shown in Figure 7. From previous experience, the decision had been made to count approximately 500 fibres. The fibres to be counted were judged to be "single" fibres, if adherence of the bundle was distinct. This is the area where judgement is subjective. Experience however would show validity of the count.

Figure 7 shows a photograph, obtained by the rapid system, of a fibre suspension. The majority of the fibres have been aligned but alignment is not necessary if time is not of paramount importance. Dispersion for this sample was relatively good. The diameter of the white spot in the centre is approximately 3/4 in. The original diameter of the brass stock was 1/4 in. Therefore, enlargement is very close to 3 X. The exact enlargement can, of course, be measured more accurately if required. Total enlargement was standardized at 3 X.

Figures 1 and 2 show plots of the length and diameter distribution, respectively, of the fibre sample used in this study. The same operator made all measurements. Though operator bias is inherent in this study, this was not considered an inhibiting factor at this stage because of the development aspect of the program.

Steps of equal length were used in this study and both cumulative and distribution curves were plotted. In literature issued by Zeiss (3) a plot of cumulative per cent as well as the distribution is shown. Approximately 400 particles were counted. The cumulative curve is smooth, indicating that the results are reliable, though the distribution curve is jagged. When the count was increased to 5000 particles, the distribution curve was smoothed.

In our counts, 400-500 fibres were sized and counted. The cumulative curves are very smooth, inferring a high degree of reliability. The distribution curve however has irregular jags. This would indicate that the number of fibres to be counted could be increased. For our purpose, i.e., the sizing of the fibre, the maxima and minima, are reasonably good, but the count could perhaps be increased to 600-700 fibres for increased accuracy.

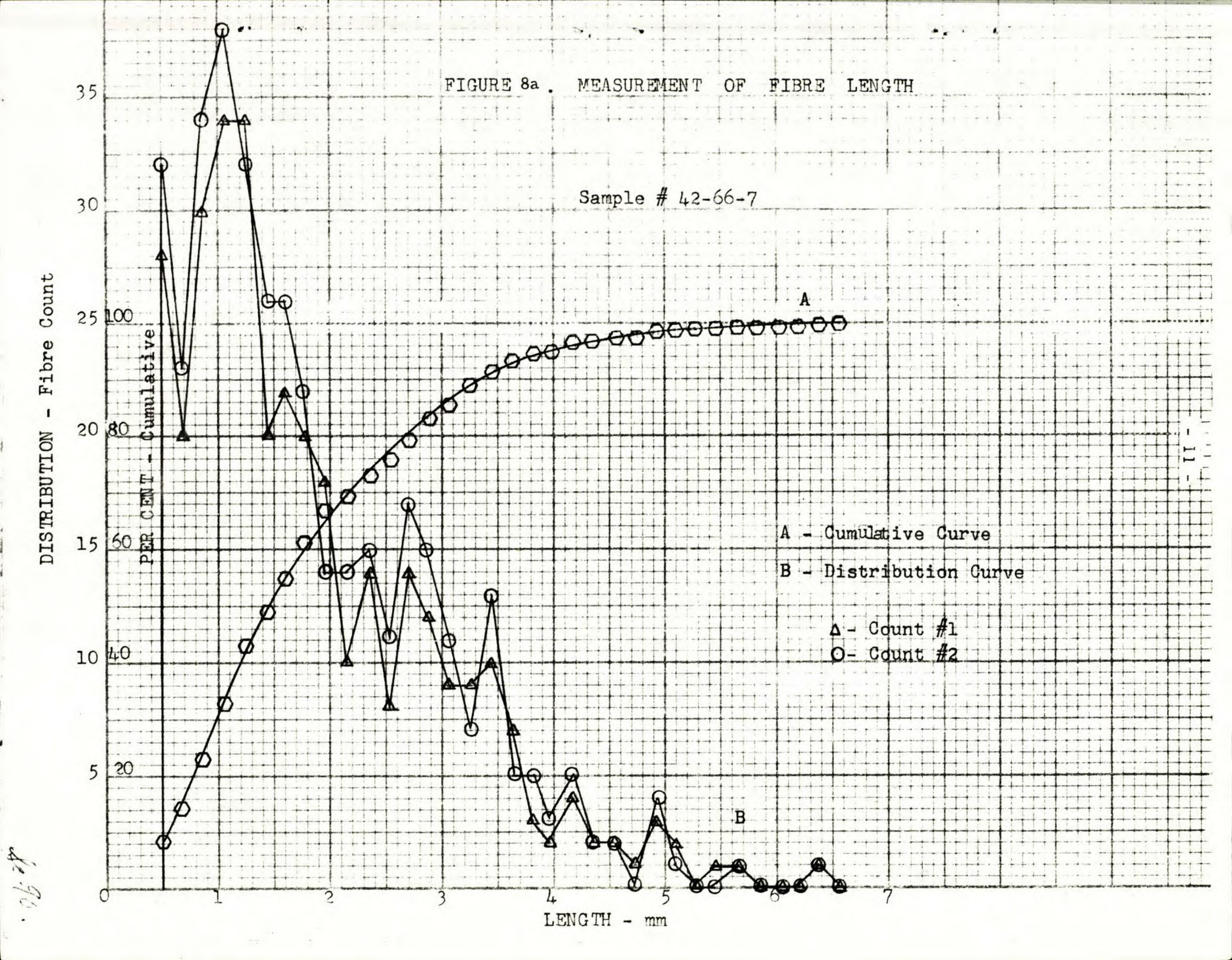
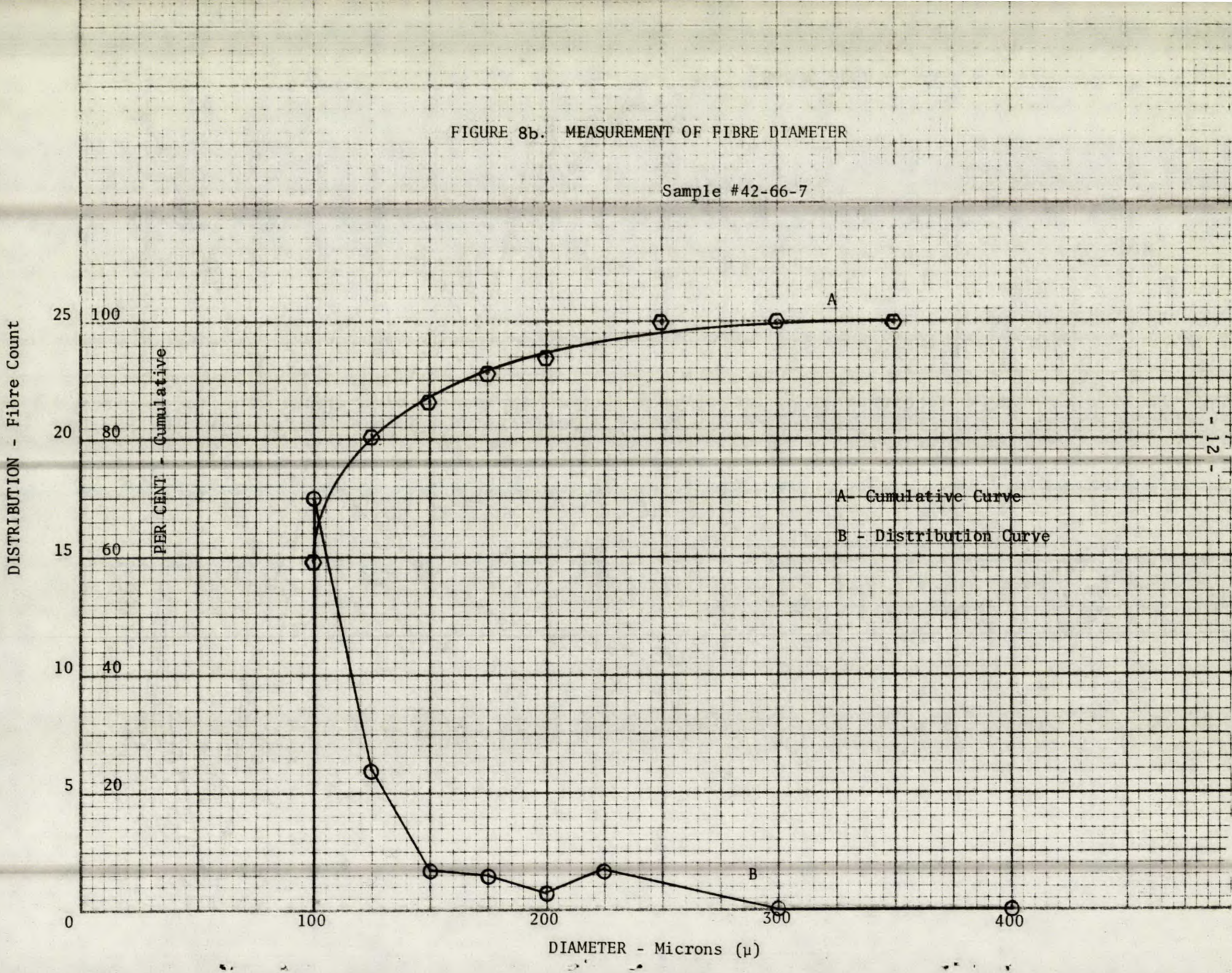


FIGURE 8a. MEASUREMENT OF FIBRE LENGTH
Sample # 42-66-7
DISTRIBUTION - Fibre Count
PER CENT - Cumulative
LENGTH - mm
A
B
A - Cumulative Curve
B - Distribution Curve
Δ - Count #1
O - Count #2
35
30
25
20
15
10
5
0
100
80
60
40
20
1
2
3
4
5
6
7

FIGURE 8b. MEASUREMENT OF FIBRE DIAMETER

Sample #42-66-7



The precision of the count for measurement of fibre length can be seen in curves B, Figure 8a. Despite the arbitrariness of the method of measurement, the curves coincide relatively well. It is possible, of course, to average the count and smooth the curve.

The distribution curve B for fibre diameter, in Figure 8b, appears to be relatively free of the jaggedness displayed in the curves for fibre length. This would appear to indicate that reducing the count of fibres for sizing the diameter is a valid operation and does not reduce the accuracy substantially. Another inference would be that the fibres follow the normal distribution.

CONCLUSIONS

This system offers good potential for measuring odd-shaped particles of any material. It is relatively rapid and appears to have the necessary degree of precision.

This system yields a good appreciation of both the length distribution and the diameter distribution of asbestos fibre.

At all times this system permits the operator to make decisions, i.e., to determine which particles to count and whether a particle should be considered as two. The operator is an integral part of the measuring system.

This system of measurement should be evaluated independently by others so that operator bias can be eliminated. This is being done presently and will be reported upon in the future.

REFERENCES

1. A.A. Winer, Length Distribution of Selected Samples of Chrysotile Asbestos using a Semi-Automatic Counting and Sizing Device, Mines Branch Investigation Report IR 64-73, Department of Energy, Mines and Resources, Ottawa (1964).

2. A.A. Winer - Unpublished results.

3. Particle Size Analyzer after Endter-Carl Zeiss.

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