Size Analysis (Cyclone Method)

Particle size of fertilizer raw materials is defined as the particle diameter ranges of the test material. Particle size of fertilizer raw materials affects granulation techniques, handling properties, and beneficiation efficiency. This procedure is useful in determining size distribution of water-insoluble material in the size range of 10-40 μm.

Apparatus

1. **Warman Cyclosizer® (Figures 1 and 2)**—Consisting of a console cabinet, five cyclones mounted in series, sample container assembly, rotameter, pressure gauge, thermometer, electrical control panel, pump, and water tank (Figures 1 and 2).
2. **Oven**—Capable of attaining a temperature of 105°C.
3. **Balance**—Top loading, with a minimum 150-g capacity and a resolution of 0.1 g.
4. **Beakers**—Two 250-mL capacity, one 3-L capacity, and five 1,000-mL capacity.
5. **Wetting Agent**—Commercial detergent or equivalent.
6. **Test Sieves**—Two, 20 cm in diameter by 5 cm high with openings of 53 and 75 μm, a sieve cover, and sieve pan.
7. **Combs Gyratory Type HL Sifting Machine**—Or equivalent wet-sieving apparatus.
8. **Air-Comparison Pycnometer**—Beckman Model 930 or equivalent for determination of true density of solids (see IFDC S-114).

General Description and Method of Operation

The cyclones are mounted with the apex "vertically up" and are arranged in a series such that the vortex outlet of each cyclone is the feed for the next in line. The vortex outlet from the final cyclone discharges to the drain manifold. The apex of each cyclone opens out into a small cylindrical chamber with a discharge valve. This valve is kept closed during the test, and therefore the apex discharge of the cyclone is, in effect, closed. A flow control valve is provided as an integral part of the sample container assembly, and water pumped from the tank through the five cyclones is manually controlled at a specified flowrate as indicated on the rotameter.

To determine the size distribution of a sample, a known weight is slurried with water and transferred to the sample container, which is removed from the holder for this purpose. After the container is loaded with the sample, the sample container valve is closed, effectively sealing the sample within the container, which is then returned to the holder. Water is pumped through the circuit, and the sample is bled into the water stream by slowly opening the sample container valve. This valve is adjusted so that the whole of the sample is released over a period of approximately 5 min, during which time the flowrate is maintained at maximum and there is a preliminary distribution of solids to the cyclones.

The initial distribution of the sample is an approximate size separation, with each cyclone and apex chamber containing an excess of undersize material. Controlled elutriation is then effected by reducing the water flow to a predetermined rate and holding it constant for a specified time while particles smaller than the limiting particle separation size of each cyclone are gradually elutriated to the vortex outlet. Solids smaller than the limiting size of the final cyclone pass out with the waste water.

After the elutriation time has elapsed, the water flow is increased again; as soon as practical thereafter, the solids that have collected in the five cyclones are discharged into separate beakers by opening the cyclone apex valves. The solids are settled, the water is decanted, and the solids are dried and weighed. The weight passing the final cyclone is determined by difference, and the effective separating sizes of the five cyclones are calculated from the specified limiting sizes and the correction factors for the actual levels of the operating variables.

Subdivision of the Gross Sample

For a sizing analysis using the Warman cyclosizer, the amount of subdivision required depends on the size distribution of the sample. The weight of the test sample used for the determination should be such that not more than 15 g collects in any one cyclone. To determine an accurate maximum weight for a certain sample, a preliminary sizing may be run. In every case the final sample should be true "split" weight and not a weight adjusted to a set figure by adding or removing small portions.

In the normal course of determining size distributions, one must deal with gross samples in both the wet and dry states. Where samples are received wet, it is strongly advised that they be reduced to the test sample and sized without being dried, with the dry weight of the test sample being calculated by determining the moisture content of a duplicate sample. For the reduction of a gross sample of dry powdered material to a test sample of suitable size, riffle splitting is usually satisfactory.

Techniques for the subdivision of gross samples of slurry are not so well developed as those for dry powders. There are three methods in general use:

1. Wet riffling.
2. Filtration followed by mixing and splitting the filter cake.
3. Withdrawing samples from the slurry while it is being agitated.

All methods, when carefully applied, will usually give satisfactory results.

Preparation of the Test Sample

The preparation of dry test samples is conveniently handled in a 250-mL beaker. The samples are pulped with 50-150 mL of water to which a little wetting agent has been added. The choice of wetting agent may vary with the type of material, but the usual commercial detergents have been found satisfactory. The pulped sample should be hand-stirred to ensure wetting of the particles.

Wet samples in the form of filter cake, as distinct from slurry, are pulped as described for the dry samples. Slurries need no preparation unless they contain oversize material.

The Warman cyclosizer is designed to operate on material in the subsieve range; all samples should be screened (according to IFDC S-107) on a 75-μm sieve for materials with a true density of 4.0 g/cm^3 or lower and on a 53-μm sieve for materials with a true density higher than 4.0 g/cm^3 (IFDC S-114). After the test sample is pulped, as described above, it is wet screened on the appropriate mesh, and all undersize is washed through into a 3-L beaker (IFDC S-107). The washings are allowed to settle for 1 h, and the sides are occasionally tapped during this time. After settling, the liquor is carefully syphoned or decanted to waste until the volume remaining is approximately 200 mL. (Any solids still in suspension after 1 h will be less than 10 μm and can be safely discarded.) The settled solids can then be transferred to the sample container for the sizing analysis.

Test Procedure

When the sample has been prepared, select an elutriating flowrate and, by referring to the flowrate correction factor chart (see Figure 6), determine the millimeter reading of the rotameter corresponding to this selected value. Then proceed as follows:

1. With the pump "off," remove the sample container from its holder by turning the container until one of the metal sides is facing you and pull straight upward.

2. Open fully the valve on the sample container and empty out any water. Stand it inverted on the handwheel of the valve.
3. Pour the test sample into the container and wash the remaining solids out of the beaker into the container (Figure 3a).
4. Continue to fill the sample container with clean water until the level is about halfway up the outside taper. Screw up the valve of the sample container until it is closed. At this stage the sample should be sealed within the container and all air eliminated.
5. With the sample container valve closed, return the container to the holder on the Warman cyclosizer by a reversal of Step 1 (Figure 3b).
   Note: It is imperative that you ensure that the sample container is correctly fitted in the holder and that a glass side is facing you before proceeding further.
6. Turn on the water supply to the constant head tank and wait until tank is full (i.e., until the float valve has closed).
7. Ensure that the control valve is closed and switch the pump on at the control panel.
8. Open the control valve slowly and allow the air to be expelled from the pipework.
9. Open the control valve fully (Figure 3c).
10. Starting from No. 1 cyclone, bleed the air from the cyclone by opening the apex valve, allowing the air to be expelled, and then closing the apex valve. Repeat this procedure with cyclones No. 2 through No. 5. The last traces of air are sometimes difficult to remove from the No. 3 cyclone and, in such cases, an alternative procedure can be used. Close both the apex valve and the control valve and allow the residual air to collect in the apex chamber; then open both valves fully and the air will be expelled. Since the vortex outlet of the No. 5 cyclone is open to the atmosphere, it is not possible to remove the central air column; thus, a "flash air column" will always be present.
11. With the control valve fully open, set the timer to 5 min and open the sample container valve slowly (Figure 3d).
12. Manually regulate the container valve, so that by the time the alarm sounds, the sample has been completely discharged into the stream. Avoid sudden surges from the sample container.
13. After the 5 min has elapsed, close the control valve until the flowmeter indicates the required elutriating flow (usually 11-12 L/min). Set the timer to the required elutriating time (usually 60 min).
14. When the alarm indicates that the elutriation time has elapsed, cancel the alarm and turn the control valve to full flow.
15. Starting with No. 5 cyclone, pull the plastic tube from the drain manifold, open the apex valve, and discharge the solids from the apex chamber into a 1,000-mL beaker (Figure 3e).
16. Close No. 5 discharge valve and proceed to No. 4 cyclone and so on in turn to the other cyclones. Note the water temperature.
17. Allow the beakers to stand for at least 20 min and then decant the excess water.
18. For final recovery and weighing, filter the sized fractions on a tared paper and dry them, or simply transfer them to evaporating or petri dishes for drying without filtering.
19. Calculate the percentage passing No. 5 cyclone as the difference between the initial weight (calculated by determining the moisture content of a duplicate sample) and the sum of the weights of the separate fractions.

Calculations

After the weight percentages retained in the five cyclones have been determined, it is necessary to calculate the effective particle separation sizes under the conditions of the test.

To do this, a correction factor must be determined for each of the four variables and multiplied with the limiting particle separation size for each cyclone.

Thus for each cyclone:

$$d_e = d_i \cdot f_1 \cdot f_2 \cdot f_3 \cdot f_4$$

where d_e = effective particle separation size.
d_i = limiting particle separation size.
f_1 = temperature correction factor for the water temperature of the test (Figure 4).
f_2 = true density correction factor for the particle true density (Figure 5).
f_3 = flowrate correction factor for the actual flowrate used (Figure 6).
f_4 = time correction factor corresponding to the time of elutriation (Figure 7).

In actual calculations, it is generally convenient to determine first an overall correction factor by multiplying the four separate factors together and using this figure to correct the limiting particle separation sizes.

Example of Calculation Procedure

Under the "standard" conditions, the cyclones are specified to separate as follows:

Cyclone No. 1—44 μm
No. 2—33 μm
No. 3—23 μm
No. 4—15 μm
No. 5—11 μm

These sizes are the limiting particle separation sizes for a water temperature of 20°C, a particle true density of 2.65 g/cm³, a water flowrate of 11.6 L/min, and an infinite elutriation time.

Consider a test sizing of phosphate rock under the following conditions: water temperature, 10°C; particle true density, 3.04 g/cm³; flowrate, 11.95 L/min; and elutriation time, 60 min.

Then, from the graphs, the correction factors are as follows:

Temperature . . . . f_1 = 1.14
Particle true density . . . . f_2 = 0.90
Flowrate. . . . . f_3 = 0.986
Time. . . . . f_4 = 0.985

The overall correction factor is thus 1.14 x 0.90 x 0.986 x 0.985 = 0.996, and therefore the effective separation sizes are as follows:

Cyclone No. 1: 44 x 0.996 = 43.8
2: 33 x 0.996 = 32.9
3: 23 x 0.996 = 22.9
4: 15 x 0.996 = 14.9
5: 11 x 0.996 = 11.0

For improved accuracy, the calculations may be based on the calibration certificate supplied with the equipment. This procedure is demonstrated in the illustration of the Warman Cyclosizer Result Sheet (Figure 8), on which calculations have been made on the basis of the calibration data shown in the lower left-hand corner of the sheet.

Auxiliary Method

1. **True Density of Solids**—IFDC S-114.
2. **Size Analysis (Sieve Method)**—IFDC S-107.

(Reprint from Warman Cyclosizer Manual; Modified)

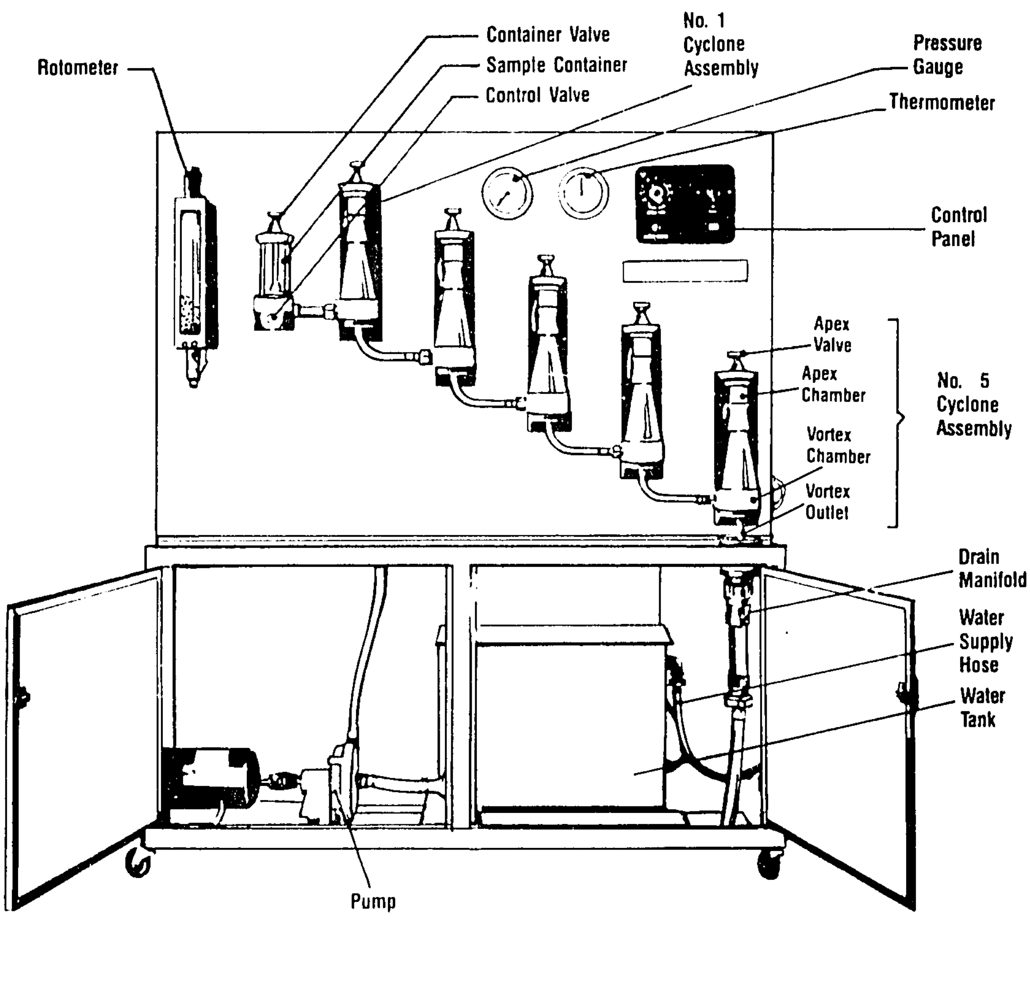

Figure 1. Warman Cyclosizer®.

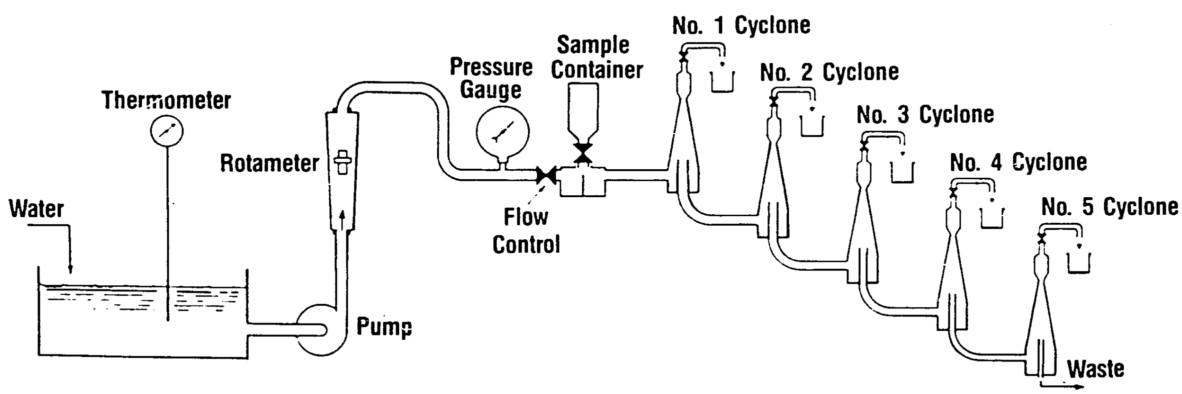

Figure 2. Schematic Diagram of Warman Cyclosizer® .

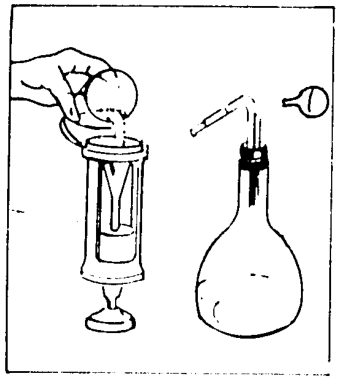

Figure 3a

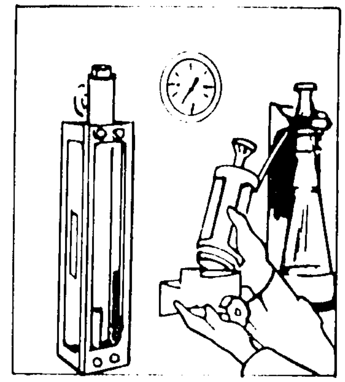

Figure 3b

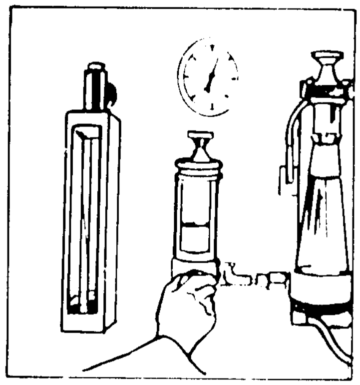

Figure 3c

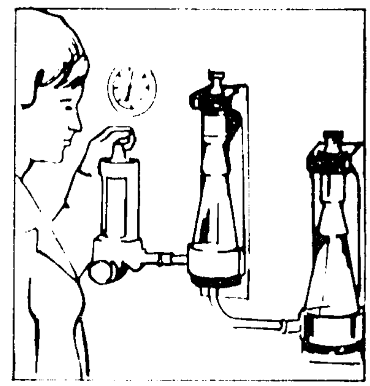

Figure 3d

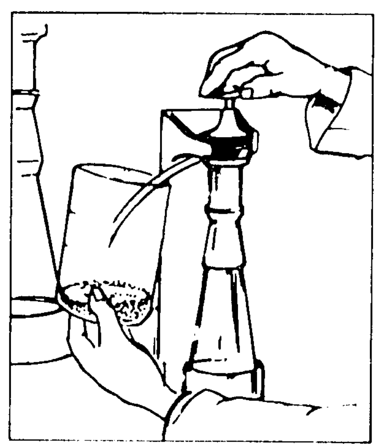

Figure 3e

Figure 3. Step-by-Step Illustration of the Test Procedure.

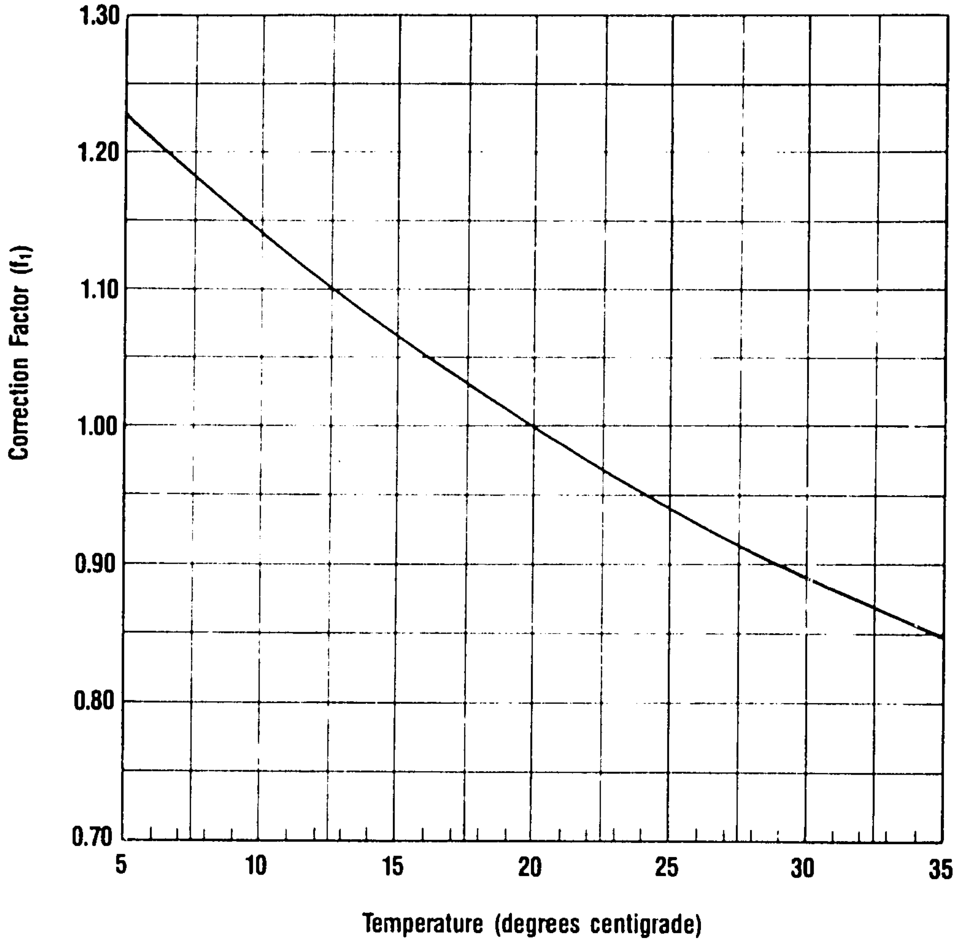

Figure 4. Temperature Correction Factor (f_1) Chart.

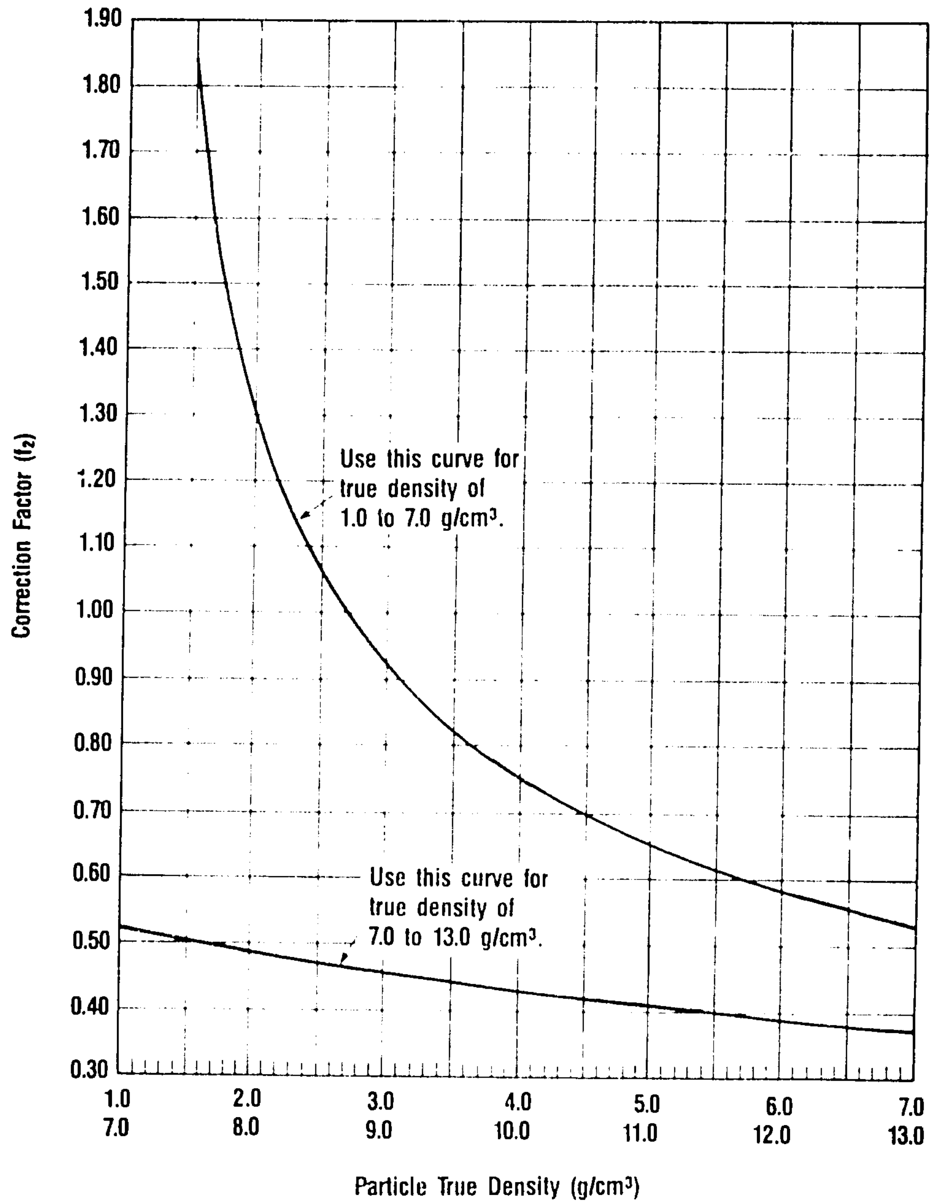

Figure 5. True Density Correction Factor (f_2) Chart.

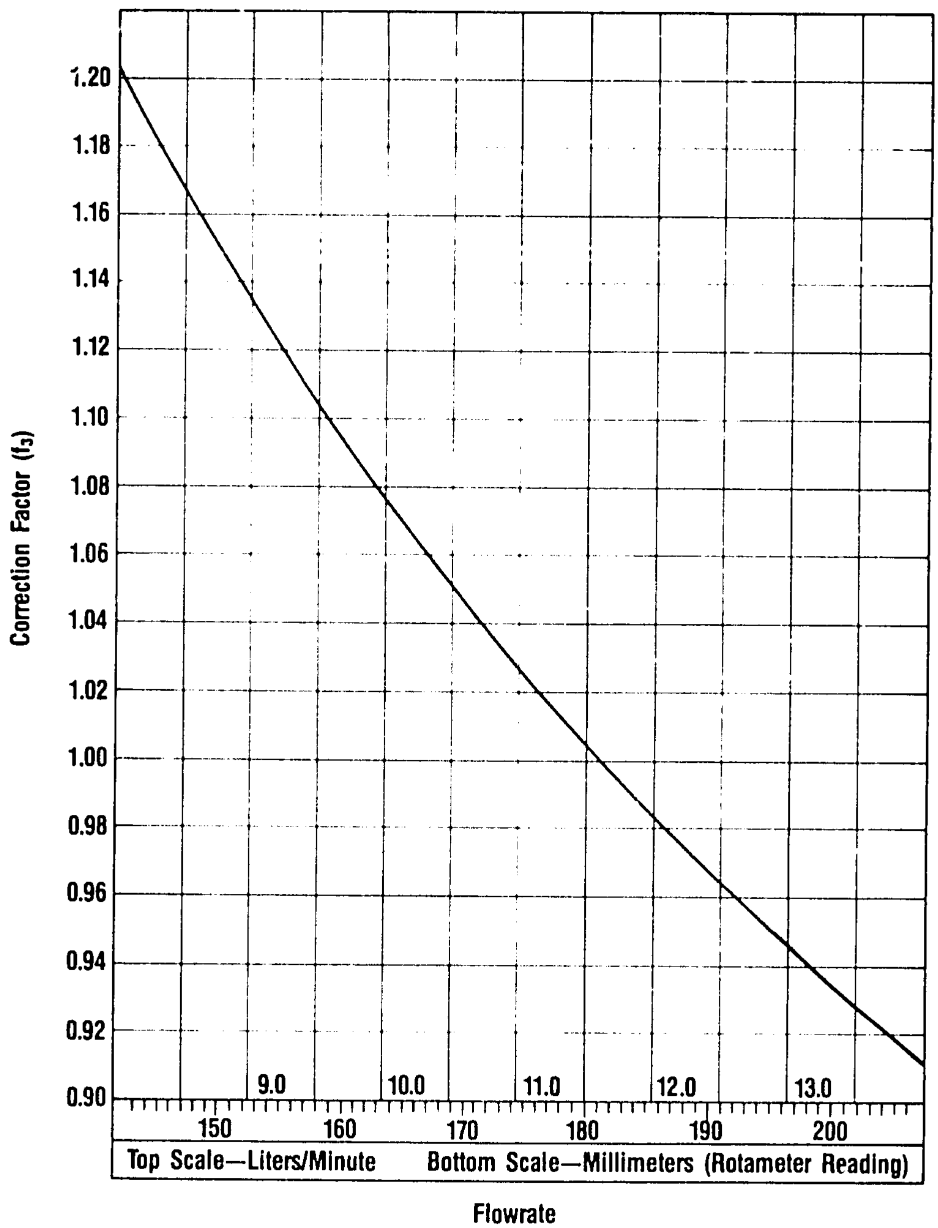

Figure 6. Flowrate Correction Factor (f_3) Chart.

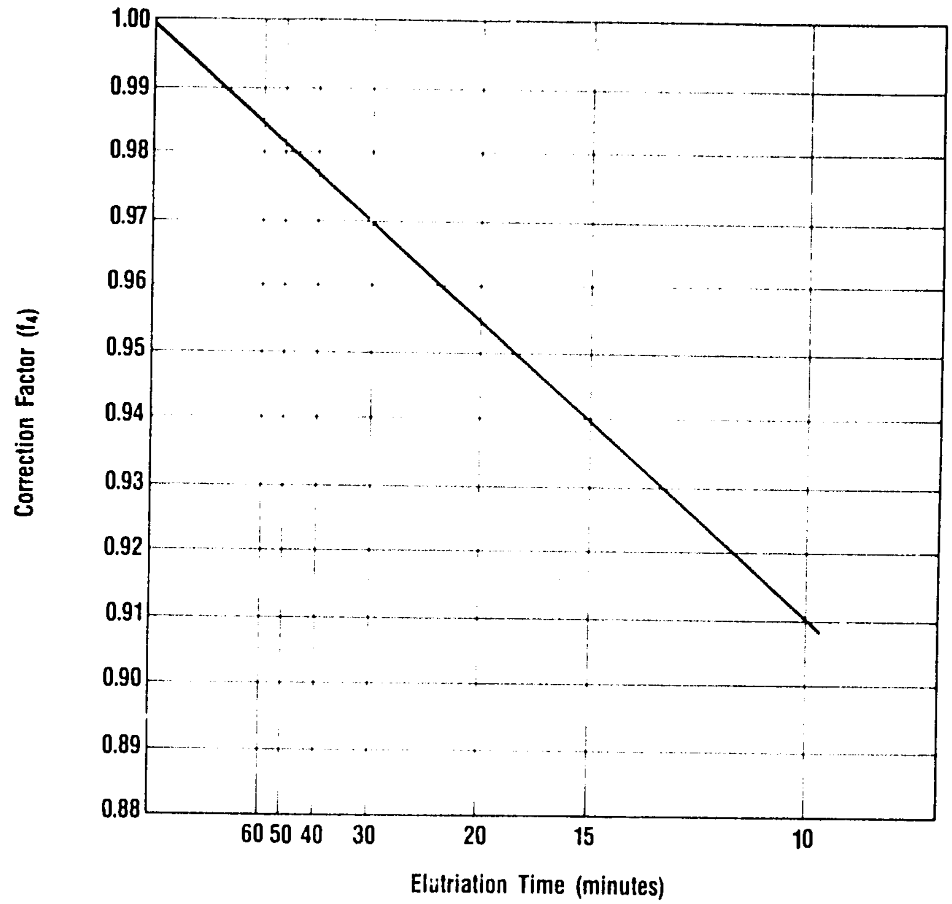

Figure 7. Time Correction Factor (f_4) Chart.

WARMAN CYCLOSIZER RESULT SHEET

SAMPLE No		R231 77
SAMPLE WT	gross	114 6
	tare	50 2
	net	64 4
TEMPERATURE	°C	10
PARTICLE DENSITY		3 04
FLOWRATE	mm	185
ELUTRIATION TIME	min	60
CORRECTION FACTOR	(temp)	1 14
.. ..	(density)	0 90
.. ..	(flow)	0 986
.. ..	(time)	0 985
No 1 SAMPLE WT	gross	51 2
	tare	51 1
	net	0 1
No 2 SAMPLE WT	gross	51 0
	tare	49 8
	net	1 2
No 3 SAMPLE WT	gross	58 8
	tare	50 0
	net	8 8
No 4 SAMPLE WT	gross	63 1
	tare	52 1
	net	11 0
No 5 SAMPLE WT	gross	59 5
	tare	51 6
	net	7 9
% RETAINED No 1 CYCLONE		0 2
.. .. No 2 ..		1 9
.. .. No 3 ..		13 7
.. .. No 4 ..		17 1
.. .. No 5 ..		12 3
% PASSING No 1 CYCLONE		99 8
.. .. No 2 ..		97 9
.. .. No 3 ..		84 2
.. .. No 4 ..		67 1
.. .. No 5 ..		54 8
OVERALL CORR FACTOR		0 996
d_e No 1 CYCLONE		42 3
.. No 2 ..		30 8
.. No 3 ..		21 5
.. No 4 ..		14 2
.. No 5 ..		10 8

CALIBRATION DATA		REMARKS
d_i No 1 CYCLONE =	42 5	USA/Central Florida Phosphate Rock
.. No 2 .. =	30 9	
.. No 3 .. =	21 6	
.. No 4 ..	14 3	
.. No 5 .. =	10 8	

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Figure 8. Warman Cyclosizer® Result Sheet.