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KN-77-11

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PIGMENT COLOR RESEARCH REPORT

TITLE: "AFFLAIR" - PARTICLE SIZE EFFECTS

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PERIOD COVERED: APRIL, 1976 TO JULY, 1977

PREVIOUS REPORTS: KN-77-11

PROJECT CODE: 81-102

NOTEBOOKS: E-8370, E-8437, E-12474, E-12478, E-12485, E-12937

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ABSTRACT

Smaller mica particle diameter leads to a more opaque, smoother looking "Afflair". Two different "Afflair" samples can be blended together to give a product whose luster and opacity are nearly linearly dependent on the properties of the two components.

Mica particles of diameter smaller than $\sim 70\mu$ are nearly constant in thickness. Particles larger than $\sim 70\mu$ are thicker in proportion to their diameter. The large, thicker particles reduce the luster and opacity of "Afflair" by effectively, diluting the product with excess mica.

OBJECTIVE

The purpose of this work is to determine the effect on "Afflair" properties of the mica particle size and of mica wet classification.

SUMMARY AND CONCLUSIONS

- The diameter of a mica flake determines the amount of surface area to be coated with TiO_2. Smaller diameter particles have more TiO_2 resulting in a more opaque product. Opacity and luster are inter-related according to the equations TSR + 10 TDR = 1200 for unclassified mica and TSR + 10 TDR = 1600 for classified mica.

- Large diameter mica particles are thicker than small diameter particles, which have a nearly constant thickness below diameters of 70 microns. The excess thickness of the large particles does not improve its scattering ability. Large particles then are diluted with excess mica and are responsible for the lower light scattering ability of unclassified "Afflair".

PATENT STATUS

There are no patents or patent filings related to this report.

PROGRAM

No further work is planned.

PUBLICATION STATUS

The material in this report is not suitable for publication.

SPECIAL SAFETY PRECAUTIONS

None.

ENVIRONMENTAL CONSIDERATIONS

None.

TABLE OF CONTENTS

INTRODUCTION

Like a white pigment, it is the function of "Afflair" to scatter light. "Afflair" has the ability to concentrate a portion of this light into the specular reflection angle. This produces a "shininess" at the mirror angle.

It will be shown in this report, that, for a given capacity to scatter light, the effective diameter of the particles determine how much light is scattered diffusely and how much is scattered at the specular angle.

Our means of controlling particle size is wet classification using hydroclones and a centrifuge. Details of the development of our wet classification system are given in KN-68-9 by B. Cetnar and KN-72-8 by D. Malizia. An explanation for the improvement in luster by wet classification of the mica is wanting. It is tempting to say that wet classification "cleans up" the mica. But, particle size analysis has shown virtually no foreign matter, such as clay. It is likewise tempting to say that there is a best particle size for optimum luster. Such an explanation must be accompanied by a reason why one particle size results in higher luster than another. We will show that wet classification removes large, thick mica particles which effectively dilute the product with excess mica.

PARTICLE DIAMETER AND OPACITY

Reducing the particle size of a mica increases its surface area by exposing new particle edges not otherwise available. Additional TiO_2 is required to coat these edges. Because the additional TiO_2 is not on the surface of the mica, it will scatter light diffusely, adding little additional scattering at the specular angle. Luster is reduced by the additional TiO_2 because the TiO_2 will block the path of light that would in its absence be cleanly reflected at the mirror angle.

Figure 1 is a plot of opacity, as TDR, versus particle diameter. A straight line is indicated to approximate the trend. NF-155-D is included in the plot as an exception. This code is made from partially pre-calcined mica. It is probable that pre-calcination opens large pores in the edges of the mica. The TiO_2 demand, and, consequently, the opacity, are increased without reducing the observed diameter of the flake.

The particularity, regardless of opacity, is of some importance in the market place. Modern cosmetics applications favor the smoother product obtained from small particle size "Afflair". Also, tests in automotive finishes show that large particle size "Afflair" reduces gloss by protruding from the finish.

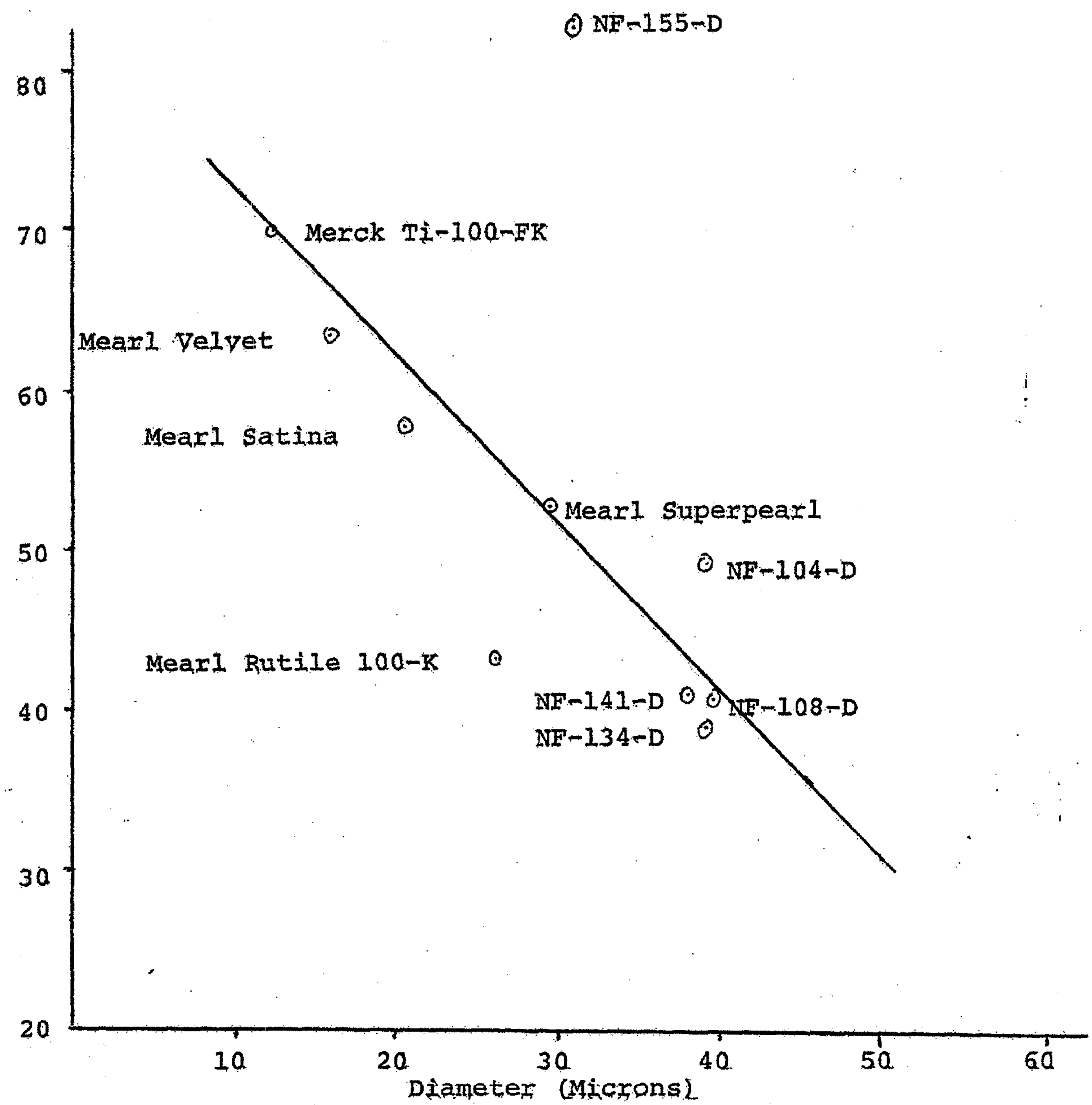

FIGURE 1 - OPACITY VERSUS DIAMETER FOR "AFFLAIR" PRODUCTS

BLENDING

Blending two unlike "Afflair" lots is now used in our manufacturing process to readjust the quality of a lot to standard. Wider application of blending might be used to greater advantage. It is possible that only a few codes need be kept in inventory. All others necessary for sales could be obtained by blending.

Blending of two unlike "Afflair" samples is essentially linear. The resultant luster (and opacity) of a blend is the weighted average of the luster (and opacity) of the components. This rule holds for more than two component systems. An example of blending application in shown in Figure 2. These blends between experimental 12485-28-18 and Merck's Ti-100-FK were made on a small scale (0.75 gms.). Some irregularities would be smoothed out on a larger scale.

Blending with mica is non-linear in both luster and opacity. Figure 3 is an opacity versus luster plot of experimental 12485-28-18 and several of its blends with mica. The non-linearity observed is caused by the drawdown method. At the loading level used (0.75 gms/10 gms acrylic lacquer] the luster is on a plateau so that reducing the loading by 10% decreases the luster only slightly. Similarly, diluting the sample by 10% with mica has little effect on the luster.

THE EFFECT OF WET CLASSIFICATION

To demonstrate the effect of wet classification, we will compare some pigments prepared from Concord mica. A sample was prepared from a portion of the unclassified mica. Another sample was prepared from a classified portion of the mica. The luster and opacity of the samples are compared on the graph in Figure 4. Their particle size distributions are compared on Figure 5. By removing the largest and smallest diameter fractions from the mica, the product becomes more transparent and more lustrous. We see that the sample from wet classified mica is beyond the TSR + 10 TDR "iso-quality" line, indicating a greater ability to scatter light than the sample from unclassified mica.

The sample prepared from unclassified mica was screened, after hydrolysis, into three fractions: -10 μ, +10 μ-400 mesh and +400 mesh. The luster and opacity of these three samples are plotted on Figure 6. Also plotted are samples prepared from the heavy and fine fractions from wet classification. Comparison of the similar fractions, fine, middle and heavy, shows little difference. It therefore has little effect on quality whether the product is classified before or after hydrolysis.

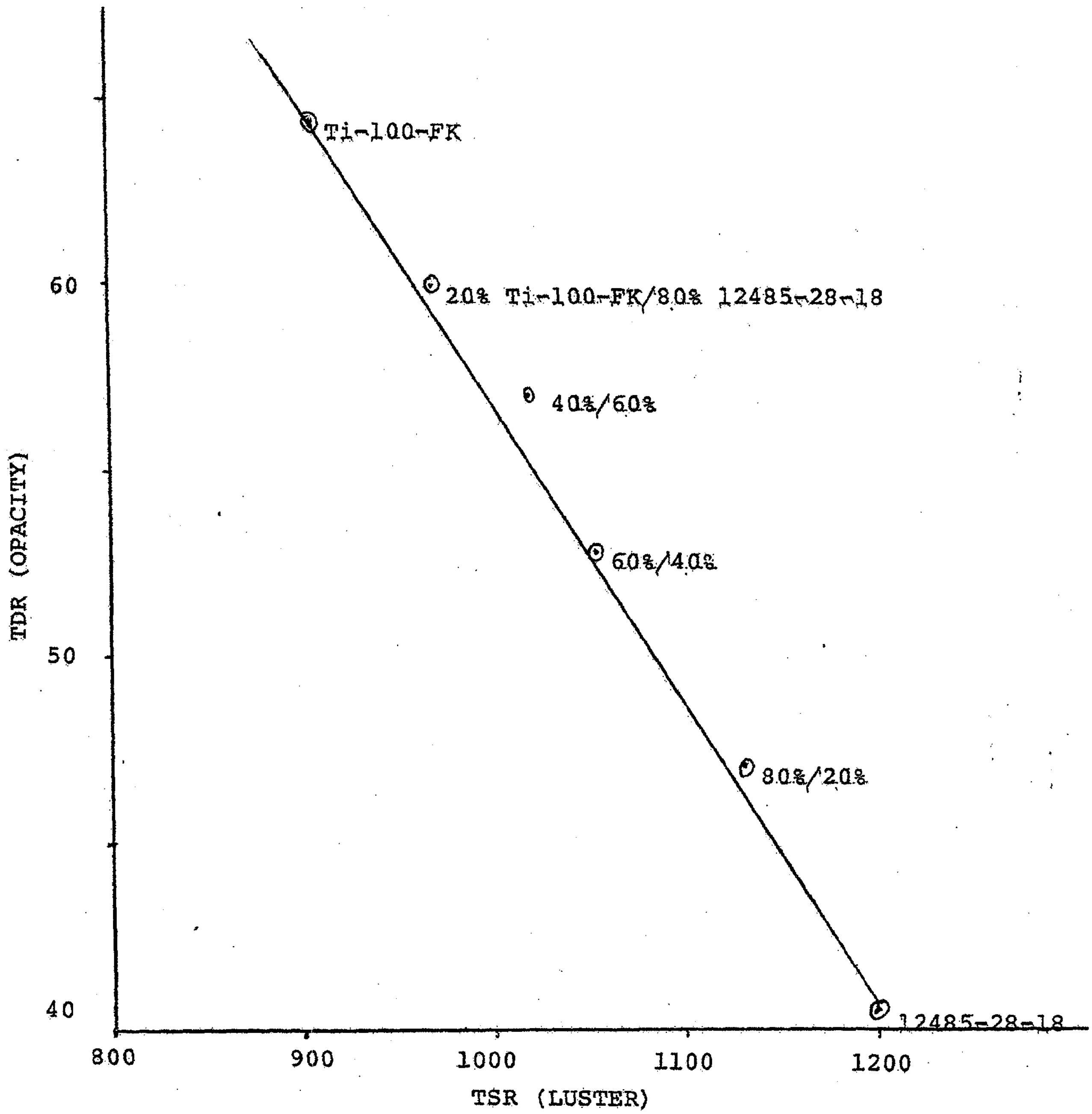

FIGURE 2 - LUSTER VERSUS OPACITY OF TWO SAMPLES AND THEIR BLENDS

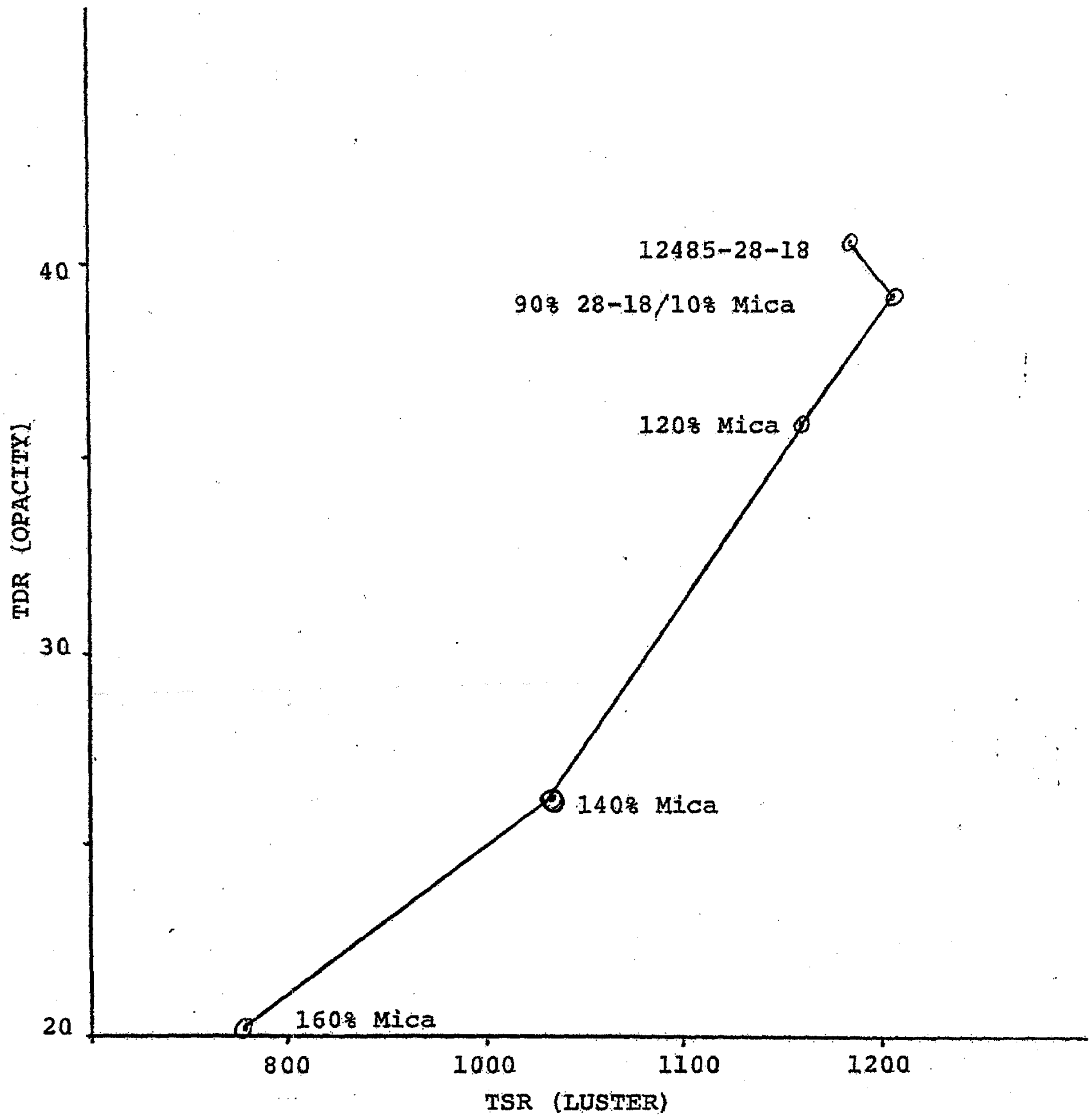

FIGURE 3 - LUSTER AND OPACITY OF EXPERIMENTAL 12485-28-18 AND ITS BLEND WITH MICA

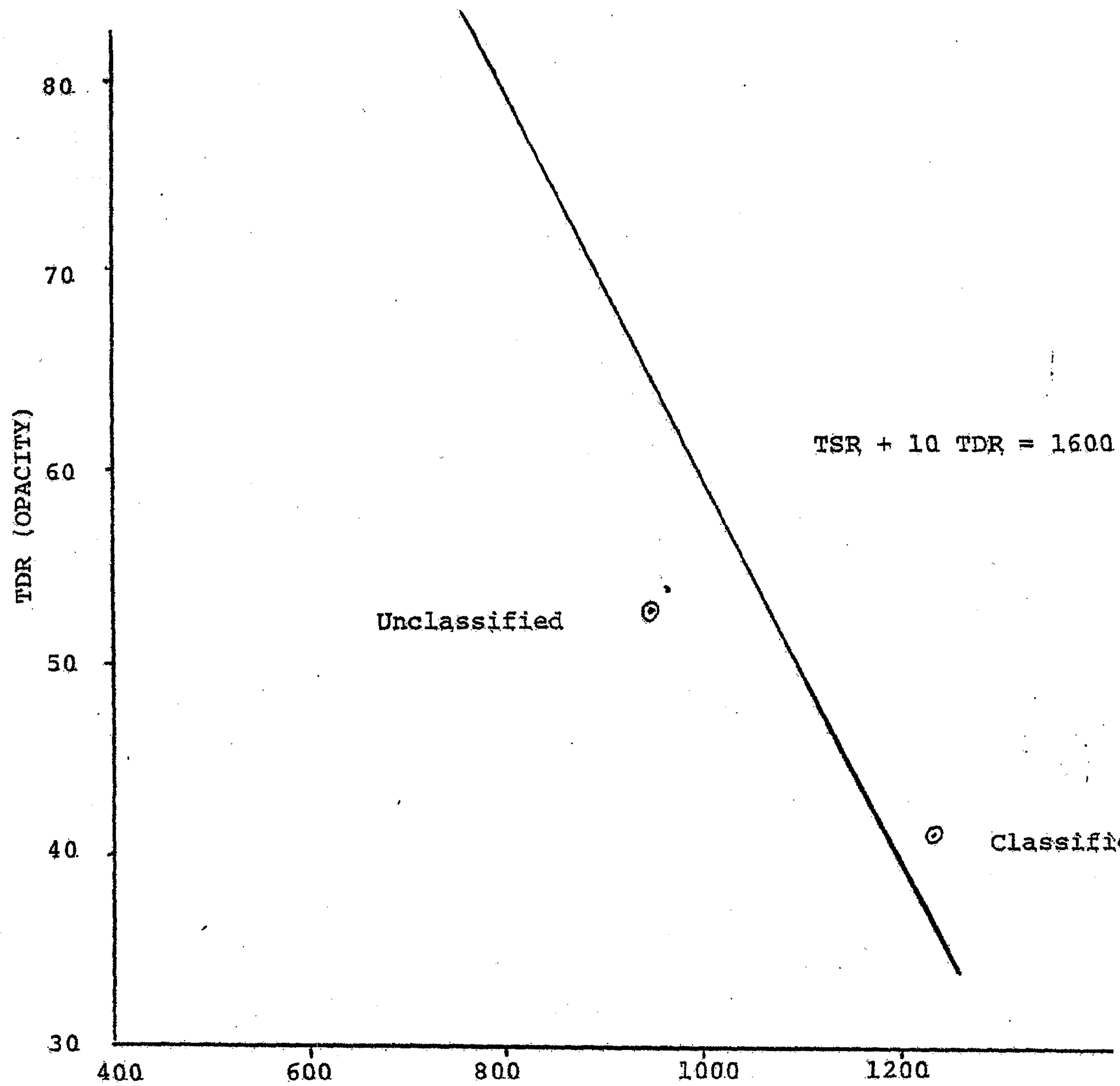

FIGURE 4 - COMPARISON OF LUSTER AND OPACITY OF "AFFLAIR" FROM CLASSIFIED AND UNCLASSIFIED MICA

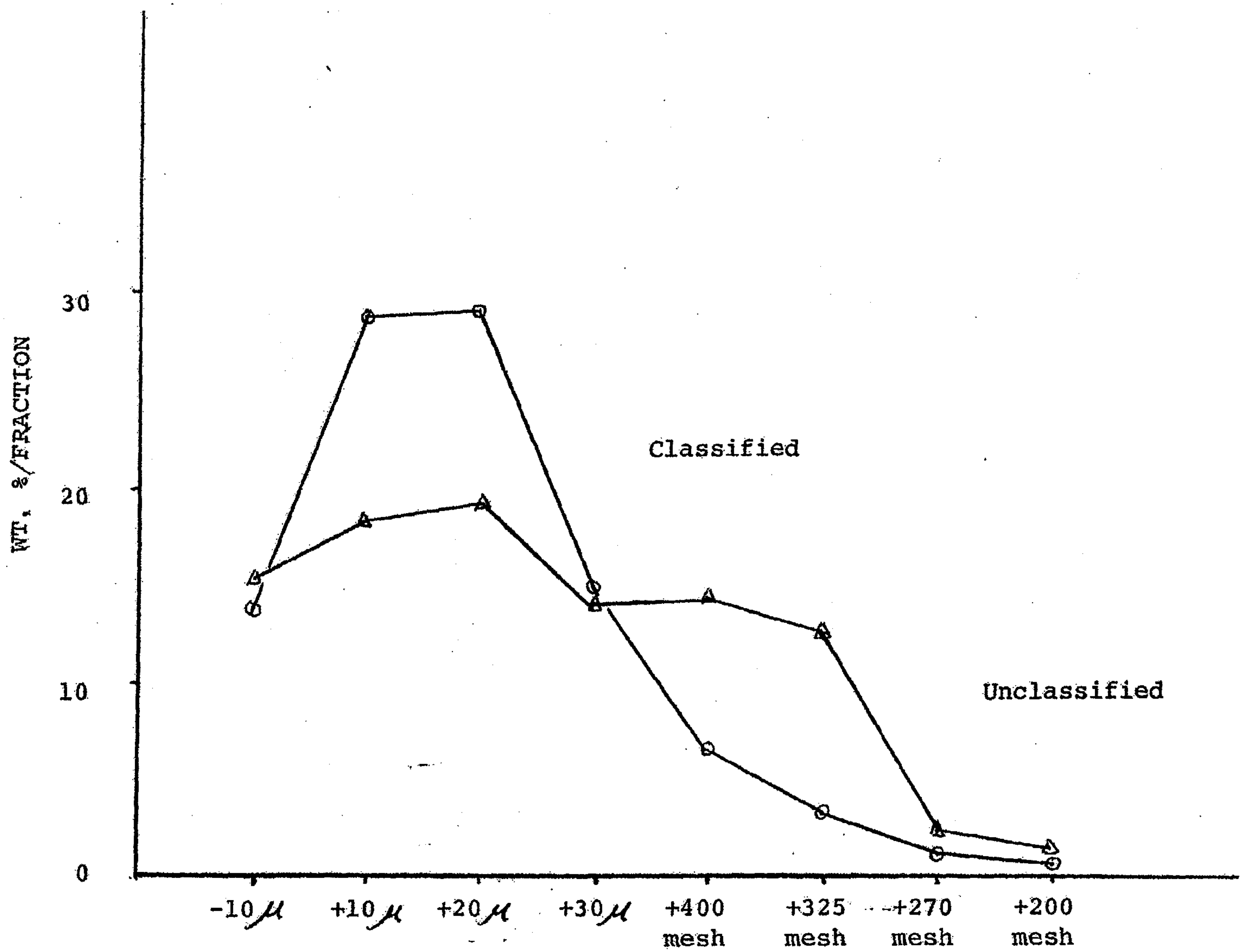

FIGURE 5 - SCREEN ANALYSIS OF CONCORD M ICA, NW-3438, Lot 6, CLASSIFIED AND UNCLASSIFIED

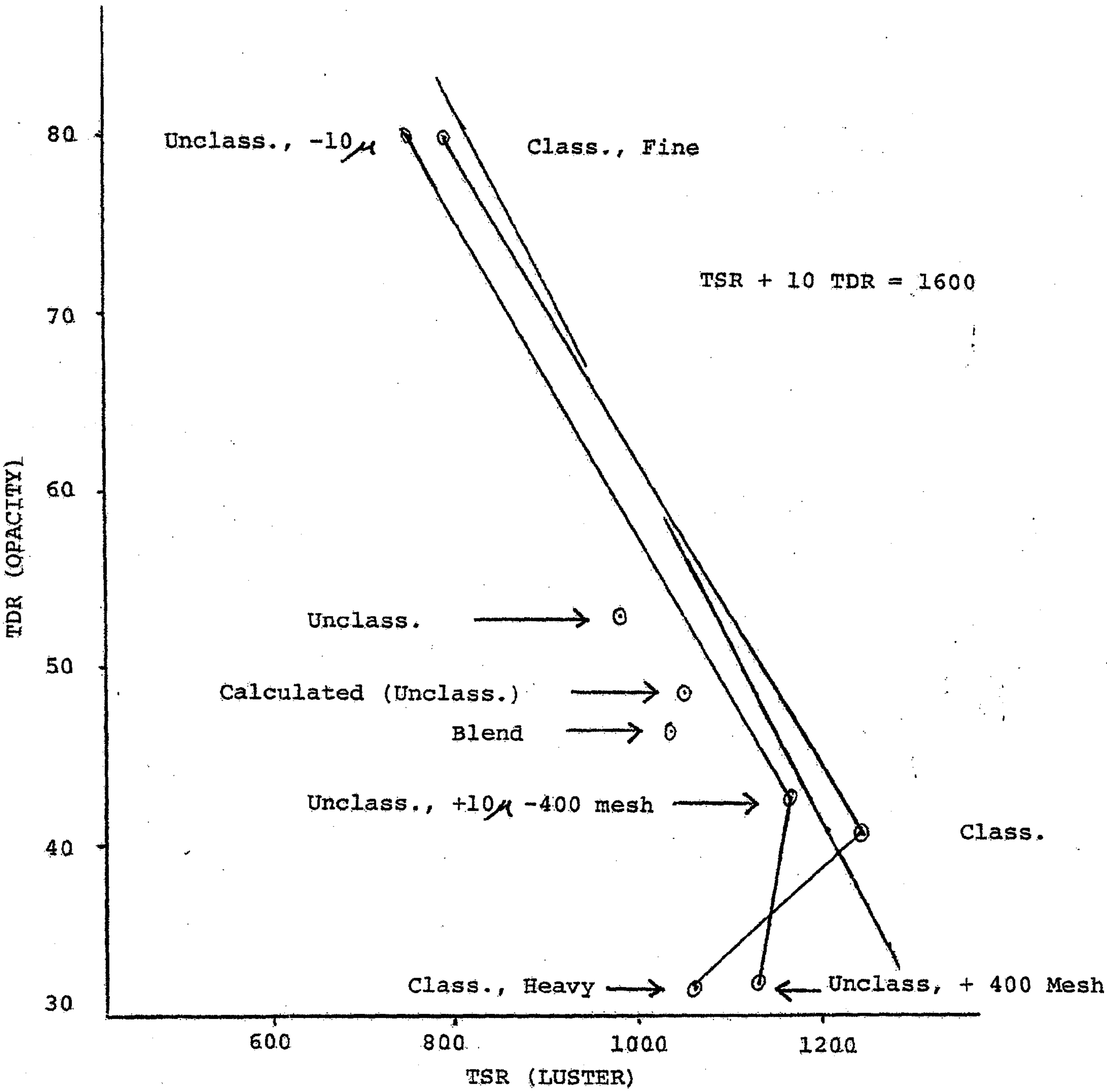

FIGURE 6 - COMPARISON OF LUSTER AND OPACITY FOR CLASSIFICATION BEFORE AND AFTER HYDROLYSIS

It is shown in KN-77-12 that the luster and opacity of a composite of "Afflair" samples are in weighted proportion to the luster and opacity of the components. On Figure 6 is plotted the luster and opacity of the unclassified sample calculated from the components, the classified sample, class., heavy and class., fine. Also shown is the luster and opacity of an actual blend the classified sample, class., heavy and class., fine in proportion to the composition of the unclassified sample. Both the calculation and blend have similar luster and opacity to the unclassified sample. From inspection of Figure 6, it is clear that it is inclusion of the heavy fraction in the calculation and the blend that causes the unclassified sample to have lower luster. The heavy fractions are the only fractions, whether classified before or after hydrolysis that deviate substantially from the TSR + 10 TDR = 1600 "iso-quality" line.

Comparison of Figures 3 and 6 provides a clue to why the heavy fraction scatters light less efficiently that the lighter fractions. The luster and opacity of the heavy fractions are similar to sample 28-18, a middle fraction, with ~30% mica added to it.

The heavy fractions act like lighter fractions diluted with excess mica. The plot of thickness versus diameter in Figure 7 shows that this is actually the case. The thickness of mica particles below 70 microns is fairly constant. Above 70 microns, the thickness increases in proportion to the diameter. The larger flakes are thicker. The additional thickness in no way improves the ability of a flake to scatter light, but effectively dilutes the product. The thickness versus diameter plot in Figure 7 is probably a natural consequence of the grinding process. There is apparently a minimum in thickness that is reached before a minimum in diameter. As the flakes become thinner, they become more fragile and their diameters are more easily reduced.

IMPROVEMENTS IN THE WET CLASSIFICATION SYSTEM

Prior to this work, the wet classification system (as described in KN-68-9) suffered from two disadvantages. It required constant operator attention to prevent the hold drums from overfilling and it was difficult to adjust the overflow to underflow ratios of the hydroclones.

Much of the design of this wet classification system was dictated by the speed of the centrifuge. The centrifuge runs at a feed rate of about 7 GPM, whereas the hydroclones run at about 21 GPM. The hydroclone would not perform effectively at lower flow rates. It was then necessary to recycle two-thirds of the output from the hydroclones to allow the centrifuge to keep pace.

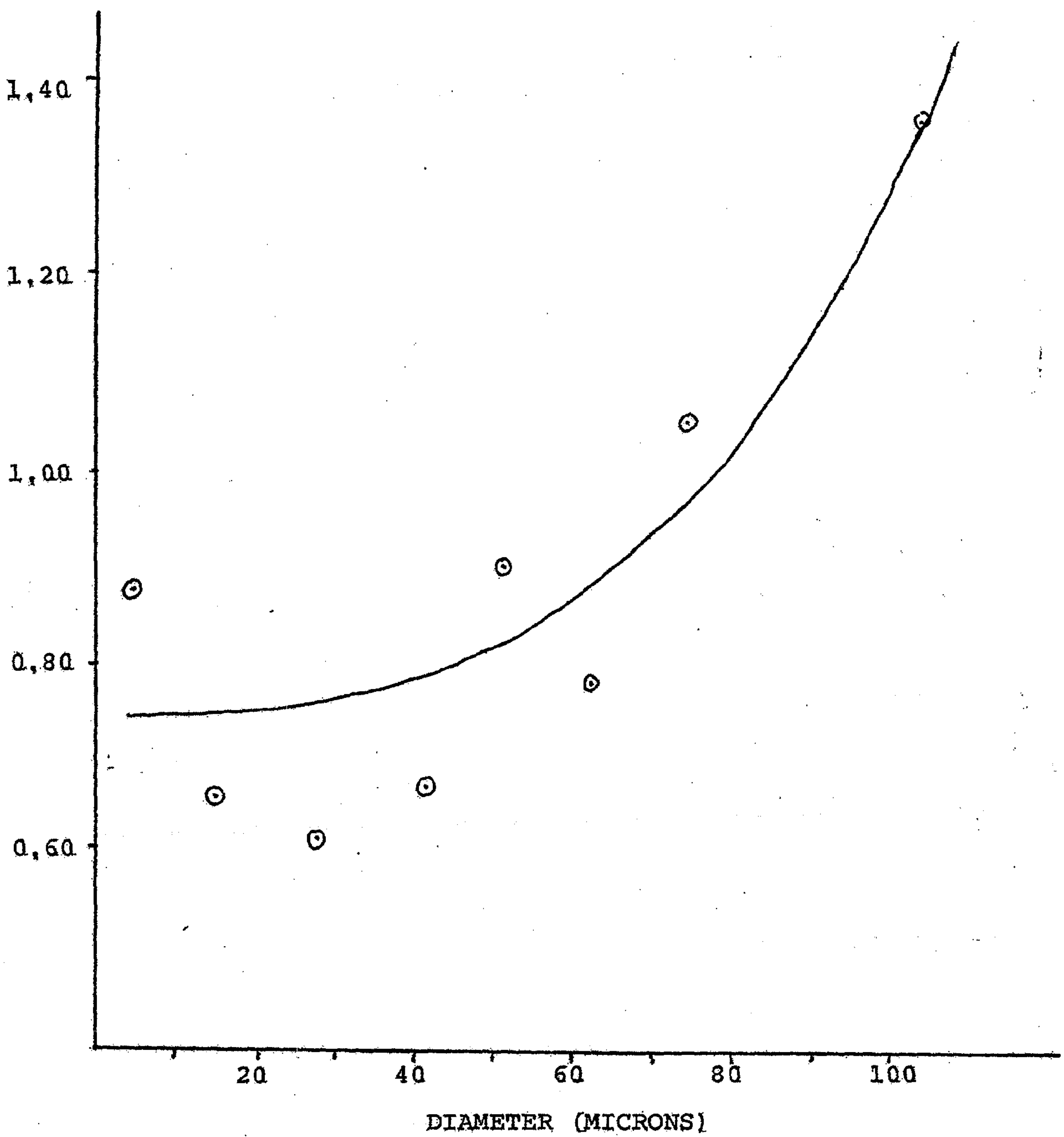

FIGURE 7 - THICKNESS VERSUS DIAMETER FOR FRACTIONS OF ALSIBRONZ MICA, LOT 156

IMPROVEMENTS IN THE WET CLASSIFICATION SYSTEM (Contd.)

The older system recycles two-thirds of the output from each hydroclone by means of tees located in the overflow and overflow lines of the hydroclones. Diaphragms of the proper diameter are located in each side of the tee to allow one-third of the total flow to pass to the next step and recycle two-thirds of the flow.

Unless the total flow into each hold drum exactly matches that drawn from the drum by the pump, the drum will gradually overfill or run dry. Also, in order to adjust the proper overflow to underflow ratios of the hydroclones, the flow rates of every line must be adjusted. Because the division of flow by the tees is approximate and the number of lines is large, the ratio adjustment is a difficult and tedious process.

Figure 9 describes a system implemented during 4Q76. The design was made by R. D. Nelson. In this design, only the outflow from the last hydroclone has a tee. The amount recycled, as dictated by the centrifuge flow rate, is adjusted by valves in the tee. Each hold drum has a 3" line near the top of the drum to allow flow to the preceding hold drum. The level in each drum is then maintained at a constant level regardless of the recycle rate. The water feed to drum #2 is adjusted by a bubbler valve. A baffle was installed in drum #1 to prevent mica from the feeder from flowing directly to drum #2. Without the baffle, the separation of the heavy fraction is less sharp.

This design requires operator attention to fill the Combin Feeder and to change drums on the centrifuge and underflow line. The smaller number of lines simplifies adjustment of overflow to underflow ratios.

CALCULATIONS FOR WET CLASSIFICATION

Methods for calculating the particle size distribution expected from wet classification are described in KN-72-8. In this work a BASIC computer program was written which includes two refinements on the orignal calculation method. In the newer method, the flow ratios of the hydroclones may be varied independently. Also, the amount of fines removed by the centrifuge is included in the calculation.

In the equation

$$X = \frac{(1-Y_3x_4)(x_1x_2)}{(1-Y_3X_4)(X_1X_2) + (Y_1Y_2Y_3)} \qquad (1)$$

X is the fraction of the mica in each particle size range reporting to the underflow. The terms x and y are defined as:

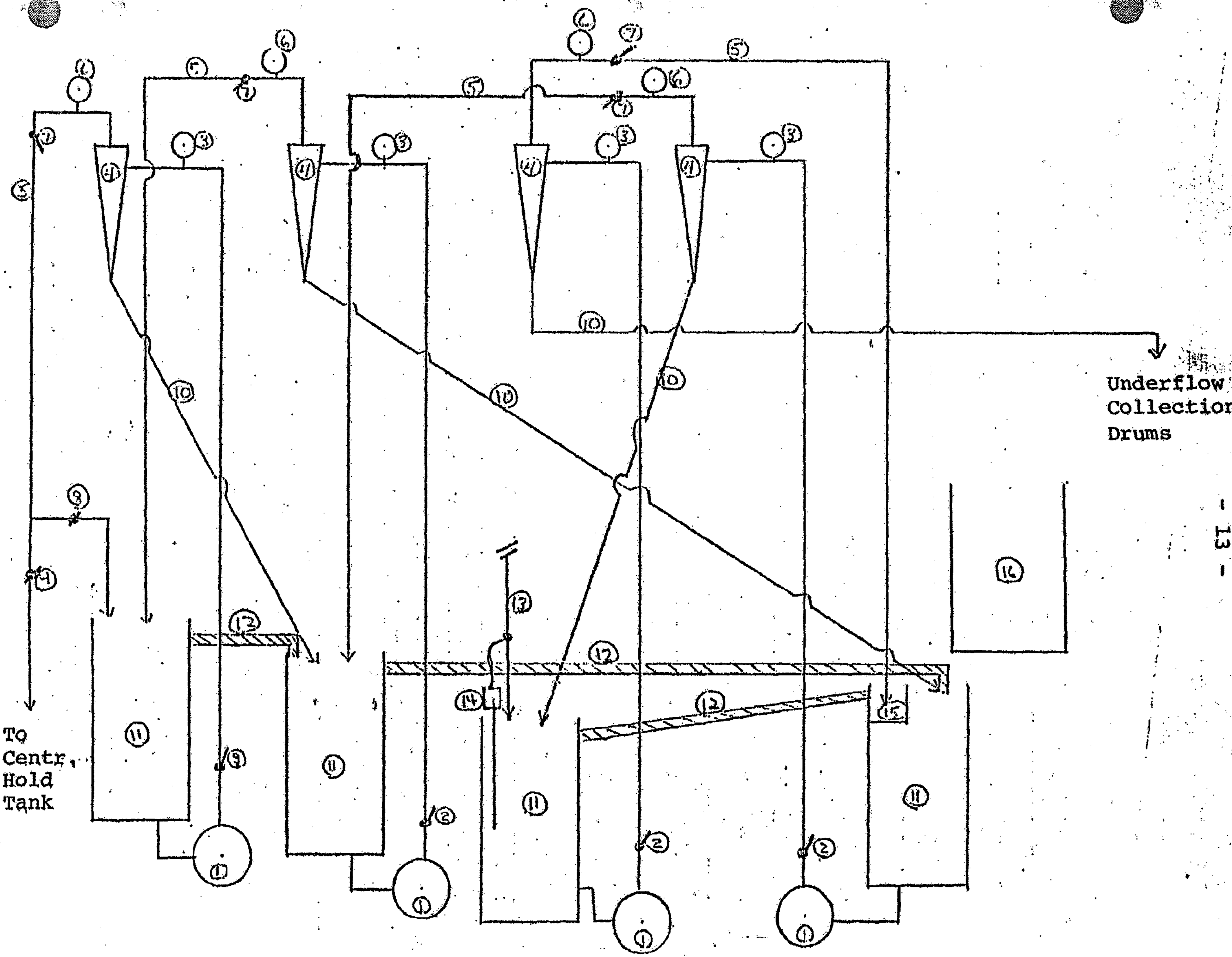

FIGURE 9 - SCHEMATIC OF THE NEW CLASSIFICATION SYSTEM

KEY

(1) Pump
(2) Feed Pressure Valve
(3) Feed Pressure Gauge
(4) Overflow Line
(5) Overflow Pressure Gauge
(6) Overflow Pressure Valve
(9) Hold Tank Valve
(10) Underflow Line
(11) Hold Drum
(13) Water Feed Line
(14) Level Controller
(15) Recycle Baffle

CALCULATIONS FOR WET CLASSIFICATION (Contd.)

$$y = \frac{SnV}{1 + SnV} \qquad (2)$$

$$x = 1 - y \qquad (3)$$

where Sn is the separation factor and V is the overflow/underflow ratio of the hydroclone. The subscripts in equation (1) indicate the hydroclone as shown in Figure 10. The separation factors, Sn, were determined empirically for the 3" hydroclones at 44 PSI and are shown in a plot in KN-68-9. A sample calculation from KN-68-9 is included in the Table.

Data from a single run was used to determine separation factors for the centrifuge. The feed slurry contained 4 wt. % mica, the feed rate was 7 GPM and the wash water rate was 2 GPM. The separation factor was varied by changing the speed of the centrifuge. Figure 11 is a plot of the fraction of mica removed versus the centrifuge speed setting. It is assumed that all of the mica removed is from the -10 micron range. The separation factors are used like x in the hydroclone calculation.

Figure 12 is a sample output from the computer program. In practice, we find that the best conditions for Concord micas are overflow/underflow ratios = 25 and centrifuge speed = 9. For Alsibronz mica, the best conditions are overflow/underflow = 25 and centrifuge speed = 7.

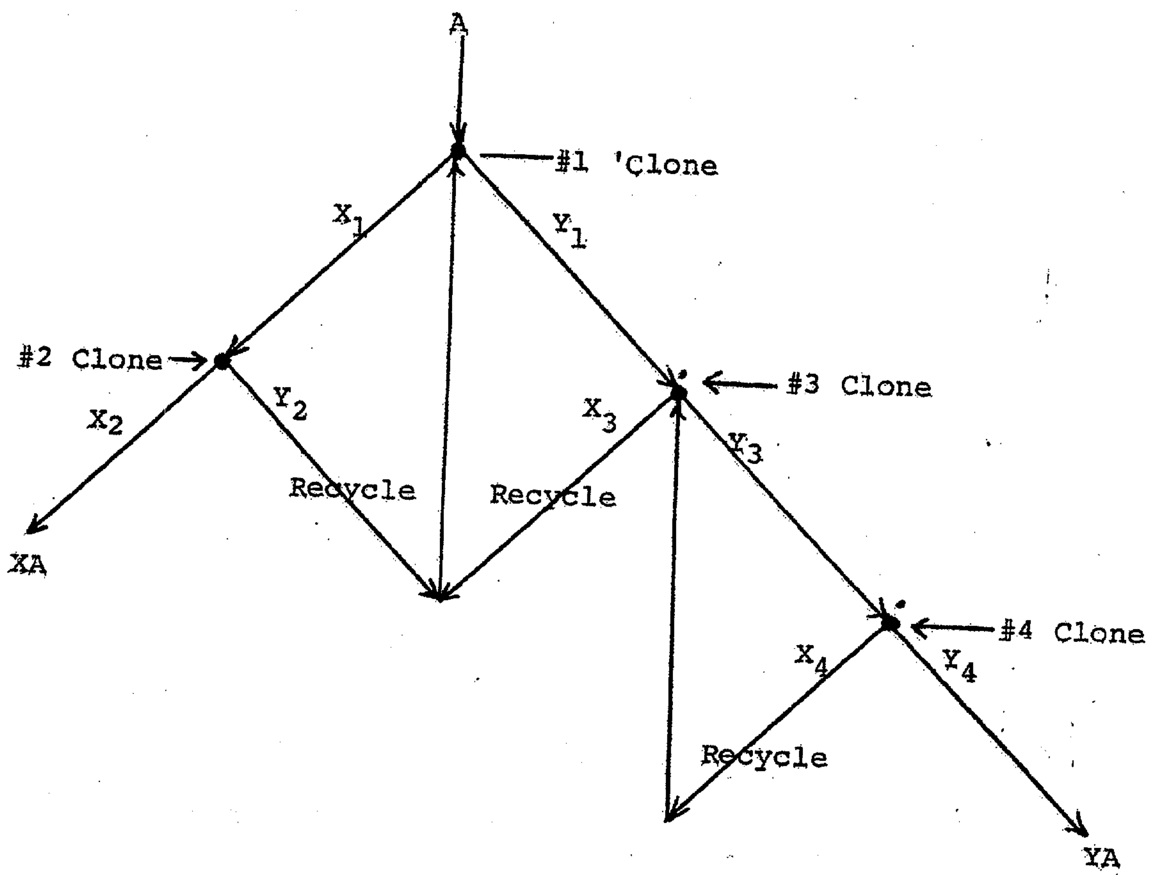

FIGURE 10 - SCHEMATIC OF HYDROCLONE SYSTEM

TABLE
SAMPLE CALCULATION

HYDROCLONE PERFORMANCE FORECAST

Particle Size Range	S_N	Y	x	X	Feed %=#	UF #	OF #
+200 Mesh	.001	.0244	.9756	1.0000	.56	.56	-
-200 +270	.00235	.0555	.9445	.9998	2.48	2.48	-
-270 +325	.01	.2000	.8000	.9853	5.76	5.68	.08
-325 +400	.0195	.3277	.6723	.9092	7.84	7.13	.71
-400 +30	.038	.4872	.5128	.6304	17.13	10.80	6.33
-30 + 20	.09	.6923	.3077	.1835	20.14	3.70	16.44
-20 +10	.235	.8545	.1455	.0289	24.34	.70	23.64
-10	.62	.9394	.0606	.0042	21.27	.09	21.63
					99.97#	31.14#	68.83#

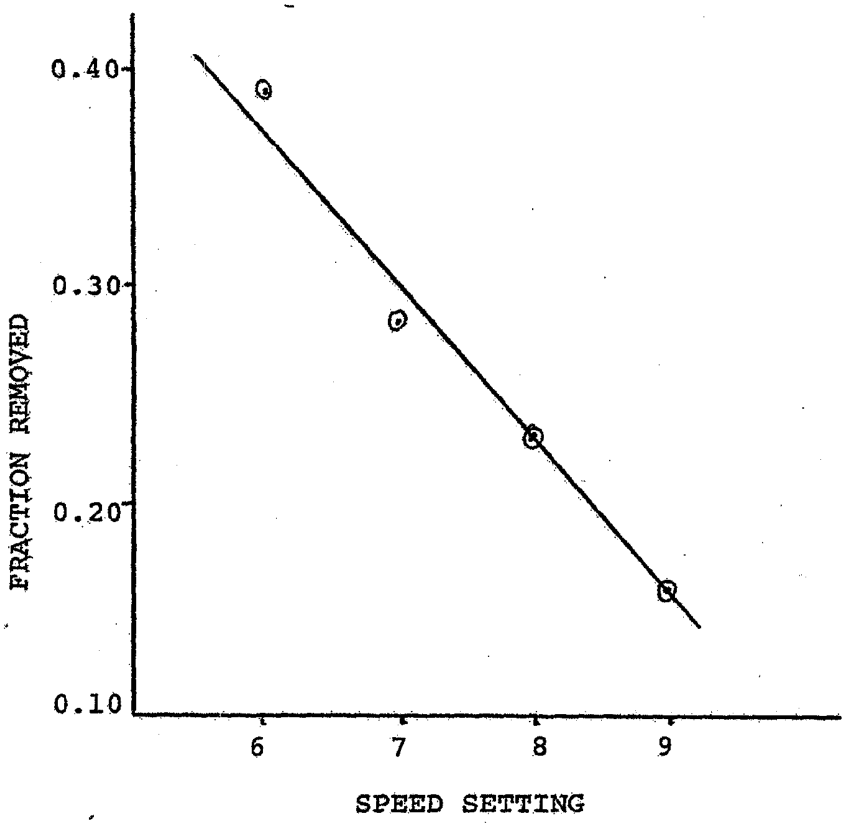

FIGURE 11 - FRACTION MICA REMOVED BY CENTRIFUGE VERSUS SPEED SETTING

HYDROCYCLONE CLASSIFICATION

CONDITIONS: PRESSURE = 44 PSI
MAKEUP WATER RATE = 7 GPM
MICA FEED RATE = 3.5 #PM
OVERFLOW/UNDERFLOW CLONE 1 = 25
OVERFLOW/UNDERFLOW CLONE 2 = 25
OVERFLOW/UNDERFLOW CLONE 3 = 25
OVERFLOW/UNDERFLOW CLONE 4 = 25
CENTRIFUGE SPEED = 6
FEED RATE TO CENTRIFUGE = 7 GPM
WASH WATER RATE TO CENTRIFUGE = 2 GPM

FRACTIONS:	FEED:	PRODUCT:	UNDEPFLOW:
+200	4.25	1.35094E-4	10.1443
+270	10.25	4.21815E-3	24.4611
+325	8.22	0.245442	19.3328
+400	7.62	1.40934	16.5364
+30	14.75	11.0994	22.1972
+20	13.64	22.7835	5.85236
+10	18.1	35.7959	1.24571
-10	23.17	28.662	0.230137

% YIELD PRODUCT = 49.1064
% YIELD UNDERFLOW = 41.8949
% YIELD FINES = 8.9987

FIGURE 12 - SAMPLE CALCULATION FROM THE PROGRAM "CLONE"

LIST

CLONE 10:45 25-JUL-77

```
10 REM [illegible] TO CALCULATE EFFECT OF RATIO AND CENTRIFUGE SPEED
20 REM ON THE CLASSIFICATION OF MICA USING THE PLANT 4-CLONE SYSTEM
30 REM INPUT: MICA TYPE
40 REM SCREEN ANALYSIS OF FEED
50 REM RATIOS OF CLONES
60 REM CENTRIFUGE SPEED
70 PRINT "MICA TYPE AND LOT NO.";
80 PRINT
90 INPUT A$
100 PRINT"FEED SCREEN ANALYSIS" +20,+0[illegible],+325,+500,+30,+[illegible],+[illegible],-[illegible]
110 PRINT
120 DIM F(8)
130 INPUT F(1),F(2),F(3),F(4),F(5),F(6),F(7),F(8)
140 DIM S1(8)
150 FOR I=1 TO 8
160 READ S1(I)
170 NEXT I
180 PRINT
190 PRINT "OVERFLOW/UNDERFLOW CLONE 1,2,3 AND 4";
200 DIM R(4)
210 INPUT R(1),R(2),R(3),R(4)
220 PRINT
230 PRINT "CENTRIFUGE SPEED (6 TO 9)";
240 INPUT C
250 DIM S(4,8)
260 DIM Y(4,8)
270 DIM X(4,8)
280 FOR I=1 TO 4
290 FOR J=1 TO 8
300 LET S(I,J)=R(I)*S1(J)
310 LET Y(I,J)= S(I,J)/(1+S(I,J))
320 LET X(I,J)=1-Y(I,J)
330 NEXT J
340 NEXT I
350 LET C1=0.[illegible]-(0.[illegible]*C)
360 DIM D(8)
370 FOR J = 1 TO 8
380 DIM X1(8)
390 LET A=(1-(Y(3,J)*X(4,J)))*(Y(1,J)*X(2,J))
400 LET C3=(Y(1,J)*Y(3,J)*Y(4,J))
410 LET X1(J)=A/(A+C3)
420 LET D(J)=X1(J)*F(J)
430 NEXT J
440 LET T2=D(1)+D(2)+D(3)+D(4)+D(5)+D(6)+D(7)+D(8)
450 DIM U(8)
460 FOR J = 1 TO 8
470 LET U(J)=F(J)-D(J)
480 NEXT J
490 LET F8=C1*U(8)
500 LET U(8)=U(8)-F8
510 LET T1=U(1)+U(2)+U(3)+U(4)+U(5)+U(6)+U(7)+U(8)
520 FOR J=1 TO 8
530 LET D(J)=(D(J)/T2)*100
```

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```
540 NEXT J
550 FOR J = 1 TO 9
560 LFTU(J)=(U(J)/T1)*100
570 NEXT J
580 PRINT
590 PRINT
600 PRINT TAB(21);"** HYDROCLONE CLASSIFICATION***"
610 PRINT
620 PRINT
630 PRINT TAB(31); A$
640 PRINT
650 PRINT
660 PRINT TAB(11);"CONDITIONS: PRESSURE                        = 40 PSI"
670 PRINT TAB(23);"MAKEUP WATER RATE                = 7   GPM"
680 PRINT TAB(23);"MICA FEED RATE                   = 3.5 GPM"
690 PRINT TAB(23);"OVERFLOW/UNDERFLOW CLONE 1       =";R(1)
700 PRINT TAB(23);"OVERFLOW/UNDERFLOW CLONE 2       =";R(2)
710 PRINT TAB(23);"OVERFLOW/UNDERFLOW CLONE 3       =";R(3)
720 PRINT TAB(23);"OVERFLOW/UNDERFLOW CLONE 4       =";R(4)
730 PRINT TAB(23);"CENTRIFUGE SPEED                 =";C
740 PRINT TAB(23);"FEED RATE TO CENTRIFUGE          = 7   GPM"
750 PRINT TAB(23);"WASH WATER RATE TO CENTRIFUGE = 2 GPM"
760 PRINT
770 PRINT
780 PRINT TAB(16);"FRACTION   FEED       PRODUCT    UNDERFLOW"
790 PRINT
800 PRINT TAB(18);"+20";TAB(25);F(1);TAB(36);U(1);TAB(46);D(1)
810 PRINT TAB(18);"+40";TAB(25);F(2);TAB(36);U(2);TAB(46);D(2)
820 PRINT TAB(18);"+50";TAB(25);F(3);TAB(36);U(3);TAB(46);D(3)
830 PRINT TAB(18);"+60";TAB(25);F(4);TAB(36);U(4);TAB(46);D(4)
840 PRINT TAB(18);"+70";TAB(25);F(5);TAB(36);U(5);TAB(46);D(5)
850 PRINT TAB(18);"+80";TAB(25);F(6);TAB(36);U(6);TAB(46);D(6)
860 PRINT TAB(18);"+100";TAB(25);F(7);TAB(36);U(7);TAB(46);D(7)
870 PRINT TAB(18);"-100";TAB(25);F(8);TAB(36);U(8);TAB(46);D(8)
880 PRINT
890 PRINT TAB(24);"% YIELD PRODUCT    =";T1
900 PRINT TAB(24);"% YIELD UNDERFLOW =";T2
910 PRINT TAB(24);"% YIELD FINES      =";T3
920 PRINT
930 PRINT
940 PRINT
950 PRINT "WANT A PLOT?";
960 INPUT B$
970   IF B$="YES" THEN 990
980 GO TO 1590
990 FOR I= 1TO 9
1000 X9(I)=U(I)
1010 IF INT(X9(I))>=1 THEN 1030
1020 X9(I)=1.0
1030 NEXT I
1040 PRINT TAB(34);"WT. %"
1050 PRINT TAB(7);"0";TAB(17);"10";TAB(27);"20";TAB(37);"30";TAB(47);"40
";TAB(57);"50"
1060 PRINT TAB(7);"------------------------------------------------------------"
1070 PRINT TAB(7);"I"
1080 PRINT TAB(7);"I"
1090 PRINT TAB(7);"I"
1100 PRINT " -10   I";
1110 PRINT LEFT$("XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX",X9
(1))
```

DUP050108608

```
1120 PRINT TAB(7);"*"
1130 PRINT TAB(7);"*"
1140 PRINT TAB(7);"*"
1150 PRINT "  +10  *";
1160 PRINT LEFT$("X[illegible]",X0
(7));
1170 PRINT TAB(7);"*"
1180 PRINT TAB(7);"*"
1190 PRINT TAB(7);"*"
1200 PRINT "E +20  *";
1210 PRINT LEFT$("X[illegible]",X0
(6));
1220 PRINT "R";TAB(7);"*"
1230 PRINT "A";TAB(7);"*"
1240 PRINT "C";TAB(7);"*"
1250 PRINT "T +30  *";
1260 PRINT LEFT$("XXX[illegible]",X0
(5));
1270 PRINT "I";TAB(7);"*"
1280 PRINT "O";TAB(7);"*"
1290 PRINT "N";TAB(7);"*"
1300 PRINT "  +40 *";
1310 PRINT LEFT$("X[illegible]",X0
(4));
1320 PRINT TAB(7);"*"
1330 PRINT TAB(7);"*"
1340 PRINT TAB(7);"*"
1350 PRINT "  +325 *";
1360 PRINT LEFT$("X[illegible]",X0
(3));
1370 PRINT TAB(7);"*"
1380 PRINT TAB(7);"*"
1390 PRINT TAB(7);"*"
1400 PRINT "  +270 *";
1410 PRINT LEFT$("XX[illegible]",X0
(2));
1420 PRINT TAB(7);"*"
1430 PRINT TAB(7);"*"
1440 PRINT TAB(7);"*"
1450 PRINT "  +290 *";
1460 PRINT LEFT$("X[illegible]",X0
(1));
1470 PRINT TAB(7);"*"
1480 PRINT TAB(7);"*"
1490 PRINT TAB(7);"*"
1500 PRINT "ANOTHER COPY?";
1510 INPUT M$
1520 IF M$="YES" THEN 890
1530 PRINT "CHANGE CONDITIONS?";
1540 INPUT K$
1550 IF K$="YES" THEN 130
1560 DATA [illegible],.0235,1,[illegible],.0199,1,.78,1.001,1.035,0.600
1570 END

READY
```

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