QUANTITATIVE X-RAY DIFFRACTION MEASUREMENTS

BY FAST SCANNING

by

R. L. Handy

Project HR-111 of the Iowa Highway Research Board
Project 576-S of the Iowa Engineering Experiment Station
Soil Research Laboratory Contribution No. 65-2

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Two goals were pursued in this research: first, to evaluate statistically some effects of sample preparation and instrument geometry on reproducibility of X-ray diffraction intensity data; and second, to develop a procedure for finding minimum peak and background counting times for a desired level of accuracy. The ratio of calcite to dolomite in limestones was determined in trials.

Ultra-fine wet grinding of the limestone in porcelain impact-type ball mill gave most consistent X-ray results, but caused considerable line broadening, and peaks were best measured on an area count basis. Sample spinning reduced variance about one-third, and a coarse beam-medium detector slit arrangement was found to be best.

An equation is developed relating coefficient of variation of a count ratio to peak and background counts. By use of the equation or graphs the minimum coefficient of variation is predicted from one fast scan, and the number and optimum arrangement of additional counting periods to reduce variation to a desired limit may be obtained. The calculated coefficient is the maximum which may be attributed to the counting statistic but does not include experimental deviations.

INTRODUCTION

The goals of this investigation were (1) to optimize X-ray diffractometer geometry and powder sample preparation procedures for best data reproducibility with fast scanning, and (2) to develop a procedure to find the required number of scans of peaks and background for a given level of accuracy. The research was originally undertaken to determine the calcite : dolomite ratio in limestones, but results should apply equally to other powder systems or to use of an internal standard. Variables evaluated include effects of various kinds of

1 A report from Iowa Highway Research Board Project HR-111, with funds from the Iowa State Highway Commission. Iowa Engineering Experiment Station Project 576-S, Iowa State University of Science and Technology, Soil Research Lab. Contribution No. 65-2.

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grinding, use of a sample spinner, use of different X-ray wavelengths, selection of diffractometer geometry, and counting techniques. The first requirement for accurate analysis is that results should be reproducible, so evaluations are based on multiple runs. The standard deviation from the mean was calculated for each set of experimental conditions; a high standard deviation indicates low reproducibility, and little chance that the mean can be accurately estimated from a few runs.

TEST METHOD

X-ray diffraction peak intensities were measured by cumulative counting across the peaks during the 2θ scan, the count total being automatically printed out at the end of the scan. Direct counting has been found to increase accuracy over recording chart and planimeter methods by several percent (5). The scan was made at a rate of 2 degrees per minute; each counting period was 40 seconds. The counts measure total peak area rather than peak height; use of area is more accurate in the event that fine crystalline size or lattice strains and dislocations cause peak broadening and flattening. For speed in the comparisons, calcite : dolomite count ratios were calculated from the 3.03 Å and 2.89 Å peak counts without correction for background. Consistency of the count ratios was then evaluated for each run by the standard deviation from the mean, $\sigma = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n}}$.[1] Since the average ratio, $\bar{X}$, depends in part on background and σ depends on $\bar{X}$, another useful measure is the coefficient of variation, which is σ expressed as a percent of $\bar{X}$, or $C = 100\sigma/\bar{X}$. Because of the number of trials, n, was small and not held constant, σ and C were used to estimate the universe parameters, $\bar{\sigma}$ and $\bar{C}$, by multiplying by $\frac{n}{n-1}$ (1). Reliability of σ and C is shown by the relative standard deviation of the standard deviation, which depends on number or repeat runs:

$$\sigma_\sigma = \frac{1}{n(n - 1)} \quad (1).$$

DETECTION

In all tests a scintillation counter was used for high sensitivity and short dead time, a filter was used to eliminate K_β , and a pulse height discriminator ("reverter") was used to minimize detection of white radiation and

[1] Symbols are defined in Appendix A.

sample fluorescence. The counter response was found to be linear to counting rates exceeding 10,000 cps.

GRINDING

Fine grinding is recognized as the most important single factor affecting reproducibility of diffraction intensities, a size reduction to 5 microns bringing C to the order of one percent (3, Table 5 - 4). Fine grinding also reduces preferred orientation effects and increases diffraction intensities, the latter by reduction of extincition. Grinding below 0.5 microns contributes to line broadening.

Samples as received in the laboratory had been run through a fine hammermill twice, with a large fraction still retained on the No. 200 sieve. An early comparison of calcite : dolomite count ratio data showed that considerable reduction in σ was effected by further grinding with an automatic mullite mortar and pestle (Table I).

Table I. Effect of grinding on X-ray diffraction count ratio, $\overline{X}$, and coefficient of variation, C.

Rm No.	Grinding Method	Average Calcite/Dolomite Count ratio, $\overline{X}$[a]	$\overline{\sigma}$	$\overline{C}$, %[b]	Av. Dolomite line breadth $^{o}2\theta$
1	Hammermill	0.4536	0.0614	13.5 $\pm$ 1.4	0.23
2	Hammermill plus automatic mortar-pestle 1 hour	0.5383	0.0227	4.22 $\pm$ .45	0.28

[a] 3° beam slit, 0.1° detector slit, sample spinning, background not subtracted.

[b] The $\pm$ entry is C times σ_{σ}.

For better reproducibility Tennant and Berger (4) recommend that limestone sample be ball-milled for 17 hours in the presence of ethyl alcohol. Lemish* and his coworkers have found that a similar size reduction may be obtained in

* Lemish, John. 1963. Personal communication

five minutes with a vibratory impact grinder utilizing steel balls with methyl alcohol grinding aid. Comparative X-ray data from a limestone ground by these and mortar and pestle methods are given in Table II.

In general, methods are listed in the table according to increasing reproducibility of test data (lower σ and C). The ball mills gave better results than the mechanical mortar, and in spite of a much higher background due to fluorescence of the iron contaminant, 5 minutes in the impact mill gave more consistent results than did 17 hours in a standard chert-pebble ball mill. This could undoubtedly be further improved on by use of a porcelain mill, and one sample was so treated. Unfortunately small size porcelain balls were not available, and the data on run 7 are from a comparatively poorly ground sample.

An expected side effect of grinding is to increase line broadening, i.e., decrease sharpness of the diffraction peaks. A convenient measure of broadening is the peak breadth expressed in degrees of two theta and measured from the chart at the half-maximum level. Average dolomite peak breadths for the various grinds are listed in Tables I and II. The hammermill sample gave the sharpest peaks, the average dolomite peak breadth equalling 0.23°, and any subsequent grinding resulted in line broadening. The impact mill gave maximum broadening, peak breadth equalling 0.40°. By use of the Scherrer equation the latter gives an effective crystallite size of about 440 Å, or 0.044 microns, indicating very appreciable internal shattering and fracturing of grains into mosaic clusters.

DIFFRACTION GEOMETRY

Starting at the X-ray tube, useful variables in any diffractometer geometry are the shape of the target image (line or spot), angle of view, beam slit (γ), soller slits, detector slit (ν), scan rate (ω), and time constant. Of particular interest are the beam slit and detector slit, which compromise intensity against resolution. Various slit systems were investigated, the other conditions being as follows:

Table II. Further Effects of Grinding: Automatic Mortar vs. Ball and Vibratory Mills

Run No.	Method of fine grinding[a]	Av. Calcite/Dolomite count ratio, $\overline{X}$[b]	$\overline{\sigma}$	$\overline{C}$, %[c]	Ave. Background counts[d]	Ave. Dolomite counts[e]	Ave. Dolomite line breadth °2θ
3	Mortar 1 hr.	.3463	.01515	4.38 ± .46	6,000	42,370	0.29
4	Mortar 4 hr.	.3933	.00957	2.43 ± .26	8,000	25,210	0.35
5	Ball mill 17 hr.	.5084	.00894	1.67 ± .18	6,000	27,450	0.33
6	Steel impact mill 5 min.	.6555	.00867	1.33 ± .14	14,000	17,350	0.40
7	Porcelain impact mill 5 min.	.4100	.00857	2.09 ± .38	2,800	36,840	0.29

[a] All samples were previously hammermilled.

[b] 1° beam, 0.2° detector slit, 3 sec. time constant, sample spinning, background not subtracted.

[c] The ± entry is C times σ_{σ}.

[d] 40 second scan, estimated from strip chart.

[e] 40 second scan, background subtracted.

Target image ....line
Viewing angle......................... 4^o
Soller slits...........medium resolution
Scan rate,................. $2\theta = 2^o$ /min.
Time constant, sec. $= 30\, \nu/\omega = 15$

(The time constant is equal to one-half the time width of the detector slit.)

Results. Effects of diffraction geometry on C are shown in Table III for two different grinding methods. In both grinds, optimum conditions are 3^o beam and 0.1^o detector slit, the finer ground material being most sensitive to differences in diffractometer arrangement.

Counting statistic. X-ray diffraction intensities are measured in counts occurring randomly in time, contributing a standard deviation (3):

$$\sigma_N = \sqrt{N} \qquad (1)$$

$$C_N = \frac{\sqrt{N}}{N} = \frac{1}{\sqrt{N}} \qquad (2)$$

For a ratio, N_1/N_2, the coefficient of variation is approximated by

$$C_{N_1/N_2}^2 = C_{N_1}^2 + C_{N_2}^2 - 2r_{N_1N_2}C_{N_1}C_{N_2} \qquad (3)$$

where C_{N_1} and C_{N_2} are the individual counting variation of N_1 and N_2, and $r_{N_1N_2}$ is the correlation coefficient between N_1 and N_2. Combining (2) and (3).

$$C_{N_1/N_2} = \sqrt{\frac{N_1 + N_2 - 2\, rN_1 N_2}{N_1 N_2}} \qquad (4)$$

The $\underline{r}$ may vary from zero to one: with r = 0,

$$C_{N_1N_2} < \sqrt{\frac{N_1 + N_2}{N_1 N_2}} \qquad (5)$$

The right hand side of this equation is plotted at the bottom of Fig. 1, and represents the maximum C which may be attributed to the counting statistic. The abscissa for Fig. 1 is a function of slit widths over scan rate, ideally proportional to N. The lowest $\overline{C}$ is for run 13, 0.88%, compared to $C_{N_1N_2}$ 0.75%. However, r was found to be 0.992, and the actual $C_{N_1 N_2}$ calculates to be 0.118%, indicating that much of the variation is experimental.

Table III. Effect of Diffraction Geometry on Reproducibiligy of Calicite/Dolomite Ratio

Run No.	Method of Fine Grinding [a]	Beam, $\gamma°$	Detector, $\nu°$	$\frac{w}{\gamma\nu}$	$\bar{X}$	σ	$\bar{C}$, %
8	Mortar 1 hr.	3	0.2	1.826	.4624	.0240	5.19 ± .55
9	"	3	0.1	2.582	.5383	.0227	4.22 ± .44
10	"	1	0.2	3.162	.3735	.0189	5.06 ± .53
11	"	1	0.1	4.472	.3795	.0245	6.46 ± 1.44
12	Steel impact mill 5 min.	3	0.2	1.826	.7479	.0115	1.54 ± .24
13	"	3	0.1	2.582	.7444	.0066	0.88 ± .14
14	"	1	0.2	3.162	.6552	.0117	1.79 ± .19
15	"	3	0.05	3.651	.8399	.0179	2.13 ± .33
16	"	1	0.1	4.472	.6660	.0135	2.03 ± .32

[a] All samples were previously hammermilled

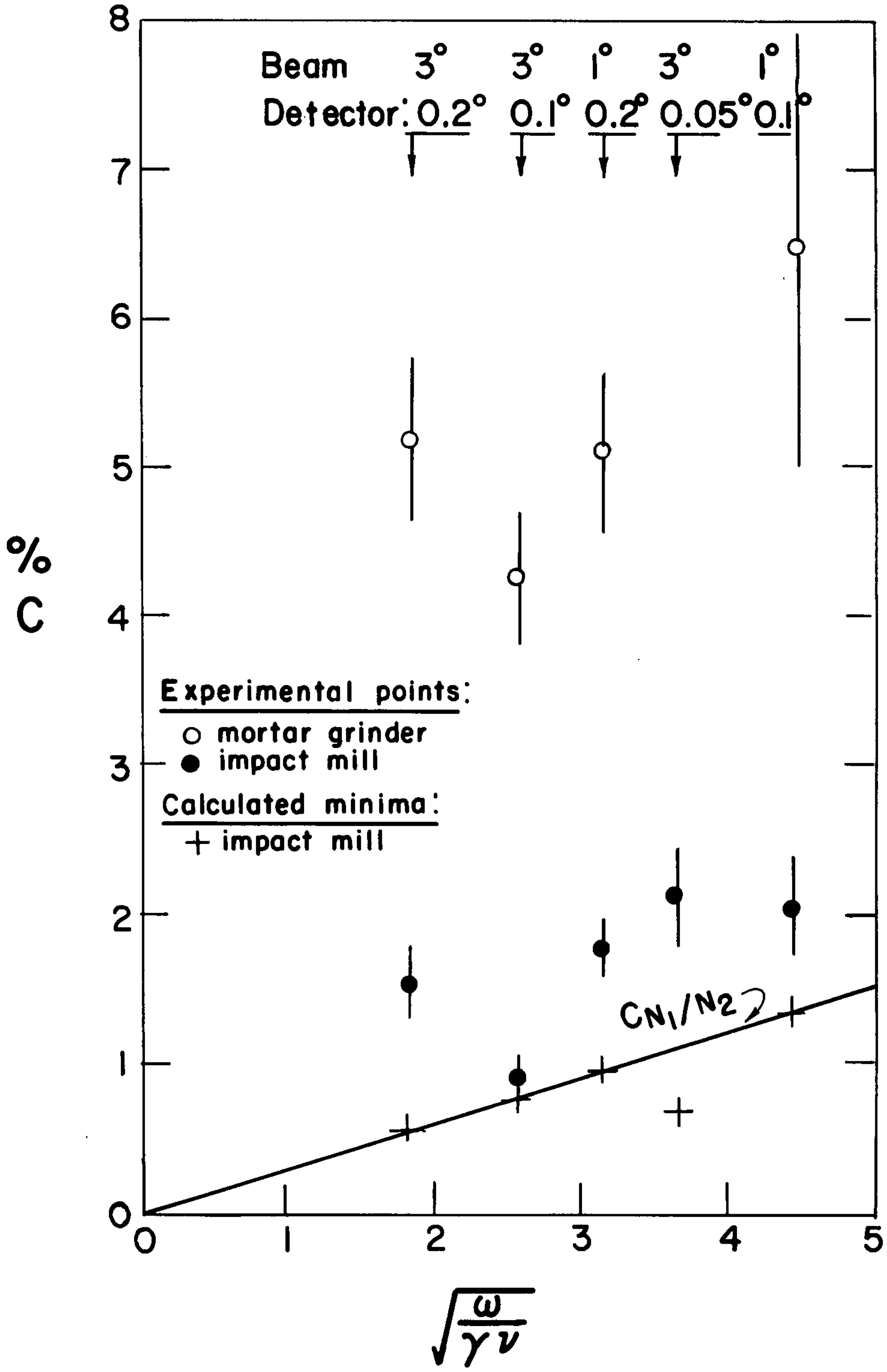

Fig. 1. Experimental coefficients of variation with different grinding methods and diffraction geometry. C_{N_1/N_2} is the maximum coefficient attributable to the counting statistic. The 3° beam and 0.1° detector slit arrangement appear to be best.

X-RAY GENERATION

Variation related to total counts is an inverse function of N, so one means for better accuracy is to increase the intensity of the X-rays, for example by use of a constant potential generator. Another possibility is to use X-ray wavelengths having lower absorption coefficients. Unfortunately, use of shorter wavelengths also decreases 2θ angles and angular separation of the peaks so that a finer diffraction geometry must be used, cutting down intensities. With a Mo tube the angular separation of the two strongest calcite-dolomite peaks is 0.66° 2θ, compared to 1.45° for the Cu tube. For comparison, the pure diffraction line breadth at half intensity was calculated to be 0.21° for material of run 13. With Mo radiation a reduction in beam slit to 0.4° was found necessary to reduce instrument broadening to separate the peaks, with a considerable loss in intensity.

Another possibility is that longer wavelengths would allow a coarser diffraction system due to greater angular separation of peaks. Tests with a Cr tube showed this advantage was outweighted by the higher absorption coefficients and lower counting efficiency, leaving copper the best choice.

SPINNING AND SAMPLE CHANGING

Several experiments were made to ascertain the sources of remaining experimental variation. In all previous runs samples were rotated in the X-ray beam at approximately 70 rpm for two purposes: to expose a greater number of propoerly oriented crystals to the beam and thus improve sample statistics, a single well oriented large calcite grain could completely disrupt the count totals, but if the sample is rotated such a grain will superimpose a tell-tale sine wave on the diffractometer trace.

Lowest deviations were expected from repeated testing of single sample, which should minimize experimental variations. A sample tested without rotation gave $\bar{C} = 0.75\%$ (Table IV); tested with rotaion it gave $\bar{C} = 0.630\%$, which compares favorably with the maximum of 0.620% contributed by the counting statistic.

Differences of $\bar{C}^2$, which are arithmetically comparable, show that spinning reduced $\bar{C}^2$ variance one-third to one-half, 1.09 to 0.63 for 10 samples and 0.56 to 0.40 for one sample. Not changing samples reduced variance one-third, 1.09 to 0.63 without spinning, 0.63 to 0.40 with spinning, and therefore can be expected to give a poorer estimate of the population. It will be noted that run 21, with $\bar{C}$ = 0.79%, is a repeat of earlier run 13, which gave $\bar{C}$ = 0.88%.

Table IV. Sources of Experimental Deviation

Run No.	Condition	$\bar{C}$, %	$\bar{C}^2$
17	C_{N_1/N_2} (counting statistic)	0.620	0.384
17	One sample, no spin	0.745	0.555
18	" " , spinning	0.630	0.397
19	" " , intermittant rotation	0.796	0.634
20	10 samples, no spin	1.045	1.092
21	" " , spinning	0.792	0.627

Virtues of spinning were also shown by static repeats of runs 10 with a mortar-ground sample, and run 14, which was an impact-milled sample tested with a less effective diffraction geometry. Not spinning raised $\bar{C}$ for run 10 from 5.1 to 6.5%, about a fifty percent increase in variance, indicating the gain from spinning is not dependent on grind. Not spinning raised $\bar{C}$ for run 14 from 1.8 to 2.6%, indicating that spinning is even more advantagious with finer diffraction geometry and fewer crystals in the beam.

ACCURACY IN RELATION TO COUNT RATIO

The sample chosen for these studies is a calcareous dolomite with a calcite-dolomite count ratio (less background) of 0.61. From equation (4), C_{N_1/N_2} is a minimum when the count product N_1N_2 is maximum, i.e., when $N_1 = N_2$, or $N_1/N_2 = 1$. A graph of equation (5) solved for $N_1 + N2 = 40,000$ counts is shown in Fig. 2; as the N_1/N_2 ratio (or its reciprocal) goes up, so does variation attributable to counting. This particular solution gives $C_{N_1/N_2} = 1$ when $N_1 = N_2$,

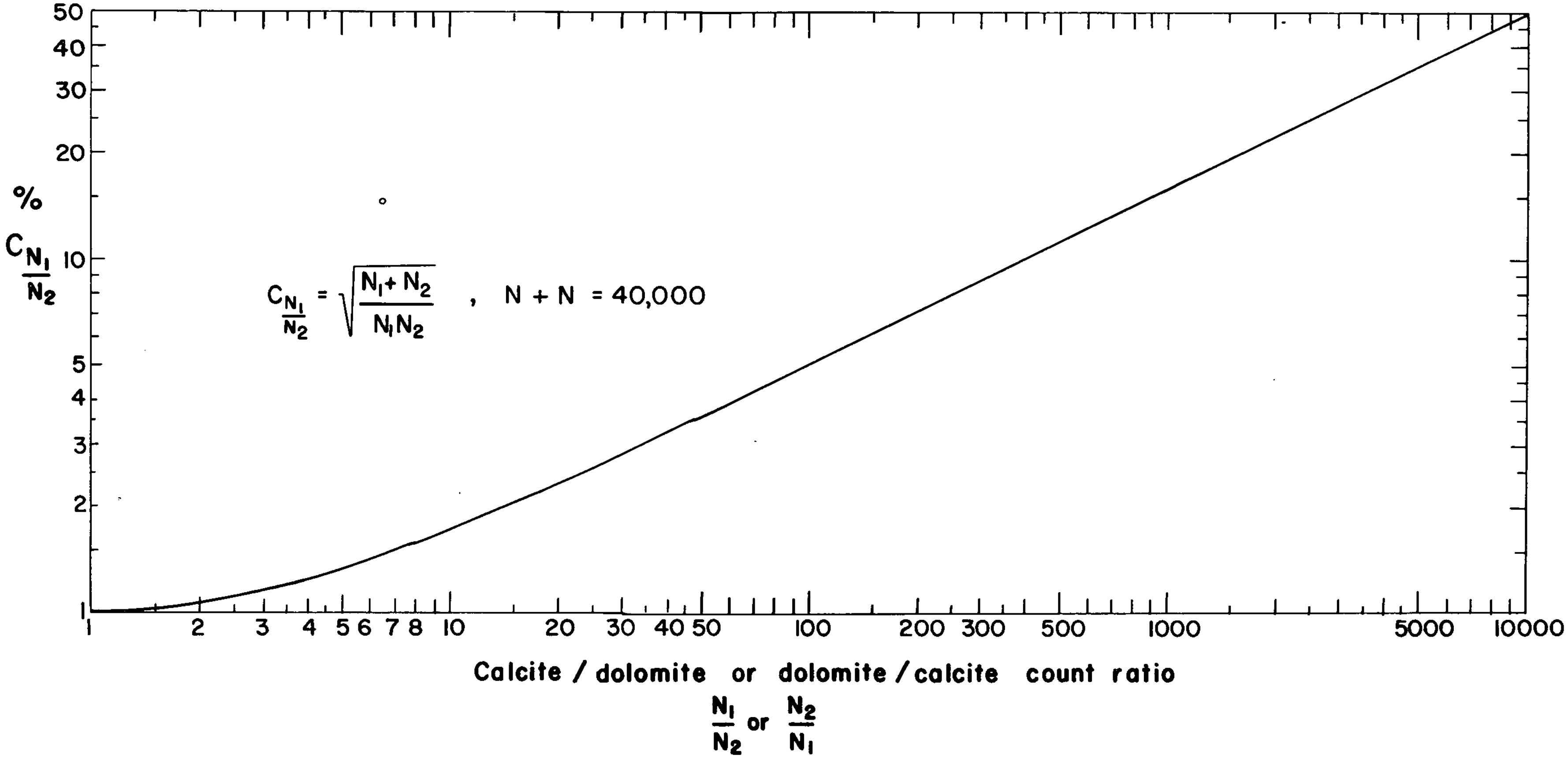

Fig. 2. Relation between coefficient of variation and count ratio; background not corrected.

so the graph shows the ratio of actual to minimum $C_{N1/N2}$ at different count ratios. For example, when the count ratio is 100 to 1, C_{N1N2} will be five times greater than with a count ratio of 1 to 1 tested under the same conditions.

BACKGROUND

In the preceding discussions background counts were ignored. Background B may be counted and subtracted from the count total N to give the difference:

$$D = N - B \tag{6}$$

The standard deviation of D is

$$\sqrt{\sigma_D = \sigma_N^2 + \sigma_B^2} \tag{7}$$

Substituting from equations (1) and (6),

$$\sigma_D = \sqrt{N + B} \tag{8}$$

$$= \sqrt{D + 2B}$$

For low σ_D, background with its double indemnity must be minimized.

By means of equations (8) and (3) and the definition of C, if $r_{D1/D2}$ is small it can be shown that

$$C_D = \frac{\sqrt{N + B}}{N - B} = \frac{\sqrt{D + 2B}}{D}$$

$$C_{D1/D2}^2 \leq \frac{N_1}{D_1^2} + \frac{N2}{D_2^2} + \frac{B}{D_1^2} + \frac{B}{D_2^2} \tag{9}$$

Examples of C_{D_1D2} calculated from actual count data are given in Table V. Counting statistic $C_{D1/D2}$'s are over twice as high for the sample with iron contaminant and high background as for the sample without iron.

The $3^o/0.1^o$ system, although giving the lowest $C_{N1/N2}$ based on total counts, gave a higher background than a $1^o/0.2^o$ system now selected for comparison. As seen in Table V, with the high background steel-milled sample the two systems are about on a par, but with the lower background sample, the $3^o/0.1^o$ system remain better.

Table V. Background Deviation

	Steel-milled Sample		Porcelain-milled Sample	
Beam slit	3°	1°	3°	1°
Detector slit	0.1°	0.2°	0.1°	0.2°
N_1	42,440	19,900	29,640	14,520
N_2	55.220	28,320	53,500	31,630
B	33,200	13,800	14,300	4,810
Max. C_{D_1/D_2}	3.3%	3.3%	1.5%	1.6%
Max. C_{N_1/N_2}	0.65%	0.92%	0.72%	1.0%
D_1/D_2	0.420	0.420	*	*

*Sample inadequate for accurate D_1/D_2 ratio determination.

In the above, background and both peaks were counted for equal time intervals. The question now arises whether the counting time could be more judiciously spent more on the peak and less on the background, or more on one peak than on the other. If $\underline{n}$ is a counting period multiplier, σ's and C's are changed by a factor of $\frac{1}{n}$ (equation 2). Following a similar derivation to (9).

$$C_{D_1/D_2} \leq \left[\frac{N_1}{n_1 D_1^2} + \frac{N_2}{n_2 D_2^2} + \frac{B}{n_B D_1^2} + \frac{B}{n_B D_2^2}\right]^{1/2} \qquad (10)$$

The first two terms in the parentheses relate to respective peak counts; the second two relate to background. Use of this equation requires a fast-scan run with equal counting intervals to measure N_1, N_2, and B. Evaluation of the four terms in the equation (all n's = 1) or an estimation of terms from the graphs of Figures 3a and 3b, will indicate where and how much more time should be spent for desired accuracy. One more counting period (n = 2) then divides the appropriate term by two, etc. From Fig. 3b may be seen that the last two terms of the equation become very small when the peak-to-background ratio excees about 20.

Example $N_1 = 10,000$; $N_2 = 2,000$; $B = 1,000$. By use of the graphs, the solution for (10) is

$$C_{D_1/D_2}^2 = \left(\frac{1.23}{n_1} + \frac{20}{n_2} + \frac{0.12}{n_B} + \frac{10}{n_B}\right) 10^{-4}$$

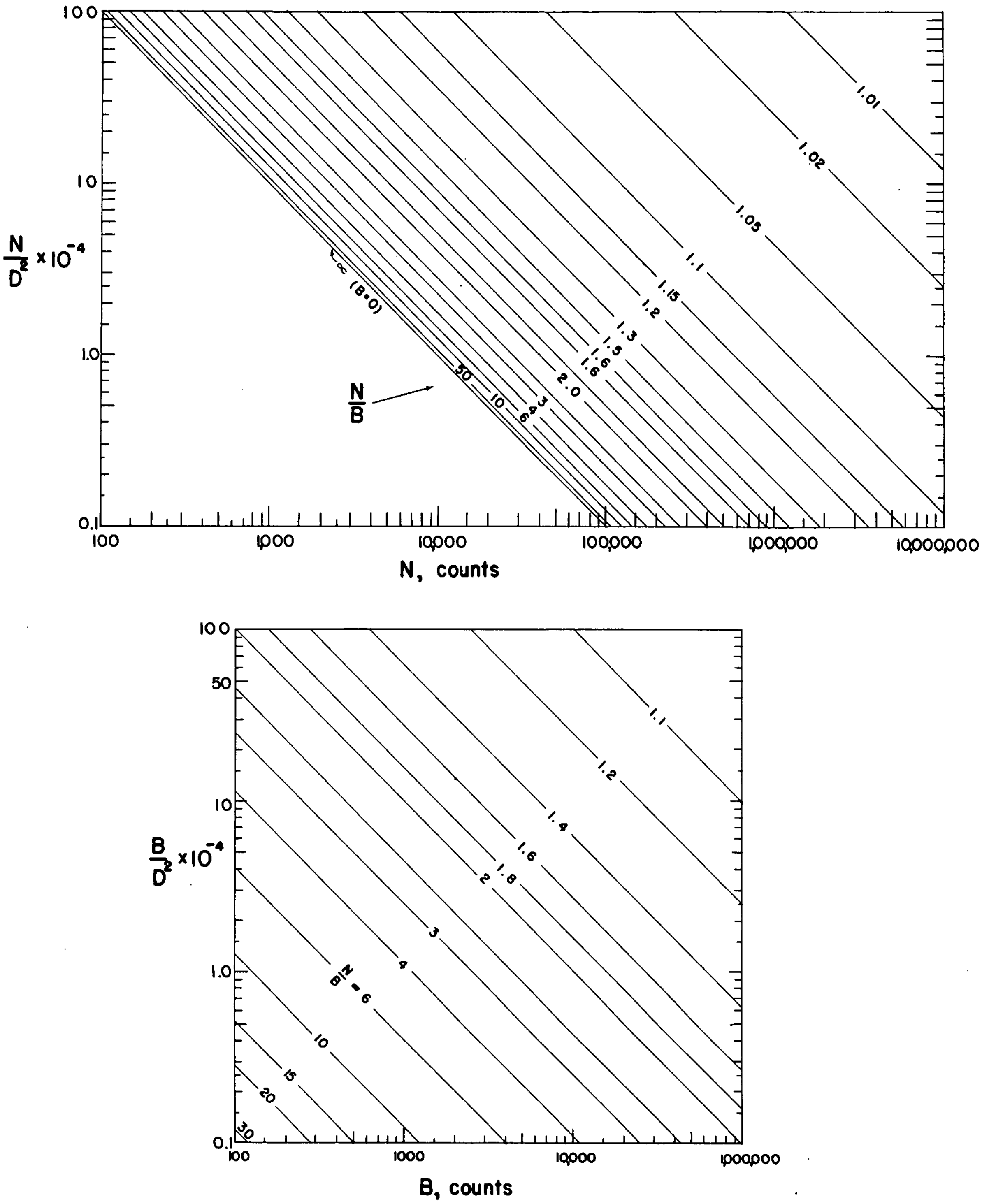

Fig. 3. Graphical solution of equation [9]. Coefficient of variation is obtained by reading upper graph for each N and N/B, and lower graph for B and each N/B, summing and taking the square root.

With all n's = 1,

$$C_{D_1/D_2}^{\;2} = 31.35\ (10)^{-4}$$

$$C_{D_1/D_2} = 5.60\%$$

For a desired accuracy of 2%, $C_{D_1/D_2}^{\;2} = 4\ (10)^{-4}$.

Individual terms should be one-third of this or less.* Let $n_1 = 1$, $n_2 = 15$, $n_B = 8$:

$$C_{D_1/D_2}^{\;2} = (1.23 + 1.33 + 0.01 + 1.25)\ 10^{-4}$$

$$C_{D_1/D_2} = 1.95\%$$

Therefore for 2% accuracy N_2 should be counted 14 more times, the background seven more times.

INTERPRETATION OF C_{D_1/D_2}

To summarize, the counting coefficient of variation, C_{D_1/D_2}, is predicted from a single fast-scan run and may be doctored to any desired level by use of the n counting period multipliers. Ninety-nine percent of the area under the normal distribution curve lies with $\pm$ 2.575σ or $\pm$ 2.575 C% of the mean, which means that ninety-nine times out of one hundred a single run will give results within $\pm$ 2.575 C of the true value. The measured value will be within one $\pm$ C interval about two-thirds of the time, 68.3%.

Near the ends of the binary mix series accuracy becomes very costly, and in the interests of economy a larger C may become tolerable. In this case equation (10) or the graphs of Fig. 3 may be used to estimate C_{D_1/D_2} for inclusion with the data. The above relationships to predict counting variations should apply equally well to any count ratio measurement, whether calcite-dolomite, quartz-cristobolite, or mineral-internal standard, and whether diffraction peaks are counted at maxima or during continuous or step scanning. C_{D_1/D_2} is the maximum coefficient of variation attributable to the counting statistic and does not include experimental variation, which of course must be minimized.

*Usually one of the background terms is very small; hence the use of one-third instead of one-fourth.

CONCLUSIONS

1. Experimental deviations of calcite-dolomite diffraction peak count ratios were minimized with the following test conditions:

   a. The sample was very finely ground, as in a vibratory impact type porcelain ball mill.

   b. The sample was pressed into a holder and rotated during the test, both to improve reproducibility and reveal sample error.

   c. A medium wavelength tube, such as copper, and a diffraction peak area direct counting technique were utilized.

   d. A relatively coarse beam slit (3°) and medium detector slit (0.1°) were used.

2. The counting coefficient of variation is higher at high and low count ratios (equation 5).

3. The counting coefficient of variation of a count ratio is higher with high background (equation 9).

4. The effectiveness and need for additional counting periods of either peak or background may be readily evaluated from equation (10) and graphs of Fig. 3.

REFERENCES

1. Ezekiel, Mordecai. Methods of correlation analysis. John Wiley and Sons. 1941.

2. Birks, L. S. X-ray spectrochemical analysis. Interscience Publishers, Inc. 1959.

3. Klug, H. P., and Alexander, L. E. X-ray diffraction procedures. John Wiley and Sons, Inc. 1954.

4. Tennant, C. B., and Berger, R. W. X-ray determination of dolomite-calcite ratio of a carbonate rock. Am. Mineral., Vol. 42, 23-29. 1957.

5. Copeland, L. E., and Bragg, R. L. Quantitative X-ray diffraction analysis. Anal. Chem. 39: 196-209. 1958.

6. Hald, A. Statistical theory with engineering applications. John Wiley and Sons, Inc. 1952.

APPENDIX A

Symbols Used

Å	Angstrom, 1×10^{-8} cm.
B	Background count
C	Coefficient of variation, $\sigma/\bar{X}$ expressed as percent
$\bar{C}$	C corrected to infinite sample size
C_D	C of a peak count less background (D)
C_{D_1/D_2}	C of ratio of peaks less background (D_1/D_2)
C_N	C of count total N
C_{N_1/N_2}	C of count ratio N_1/N_2
D	Peak count less background count = N - B
n	Number of runs in a test
n_1, n_2, n_B	Number of counting periods of N_1, N_2, and B
N	Peak count
N_1	Peak count for mineral 1 (calcite)
N_2	Peak count for mineral 2 (dolomite)
r_{N_1/N_2}	Correlation coefficient between N_1 and N_2
X	Peak count ratio, N_1/N_2
$\bar{X}$	Mean X from n runs
γ	Beam slit width, °
θ	Bragg angle
ν	Detector slit width, °
σ	Standard deviation from the mean
$\bar{\sigma}$	σ corrected to infinite sample size
σ_D	σ of a peak count less background (D)
σ_σ	Relative standard deviation of a standard deviation
ω	Scan rate, °/min.

APPENDIX B

A Sample Holder, Molder, and Spinner

Sample holders suitable for compacting and spinning powder samples may be made by drilling out U. S. standard 7/16" (1/2" i.d. x 1 1/4" o.d. x 5/64") brass washers to a hole size of 51/64", giving a hole area nominally 0.50 sq. in. A piston force against the sample of 500 lb. thus gives a compactive pressure of 1000 psi. A mechanical hand-operated rack-and-pinion type press with a 10:1 advantage was found to be quite adequate for compaction, and gave a decided advantage of speed over hydraulic or motor-driven presses. Samples may be molded in approximately 30 seconds.

Samples are molded against a polished steel surface (Fig. 4) with a 3/4" polished piston. The steel anvil may be covered with paper to decrease preferred orientation, but after fine grinding this was found to be unnecessary. A cover plate acts as a guide for the piston and prevents puffing out of the sample during the sudden compaction.

The sample spinner (Fig. 5) fits a General Electric XRD-5 goniometer and is a rebuilt commercial spinner. Front faces of the sample holders bear at top and along the bottom to insure precise alignment regardless of changes in sample holder thickness. Rotation is accomplished by a conical rubber friction drive against the top back edge of the holder.

Fig. 4. Disc sample holders (at left and under piston). Piston guide is at rear.

Fig. 5. Rebuilt motor-driven spinner for disc sample holders. Speeds are 70 rpm and 7 rpm for fast and slow scan, respectively.