Designation: E 877 – 93 (Reapproved 1998)

Standard Practice for Sampling and Sample Preparation of Iron Ores and Related Materials[1]

This standard is issued under the fixed designation E 877; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers procedures for mechanical sampling of iron ores and related materials in a falling stream or stopped-belt sampling and preparing the gross sample to the various test samples required for each characteristic to be measured. Included as Annexes are (*1*) design criteria to prevent bias, (*2*) statistical methods to determine quality variation and precisions of sampling and division, and (*3*) a method for comparing two sampling procedures for possible systematic differences.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials[2]

E 279 Test Method for Determination of Abrasion Resistance of Iron Ore Pellets and Sinter (Tumbler Test)[2]

E 389 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Coarser for Metal Bearing Ores and Related Materials[3]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[3]

E 1072 Test Method for Low Temperature Breakdown of Iron Ores[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *bias*—an error that is consistently positive or negative.

3.1.2 *consignment*—the total quantity of iron ore to be sampled.

3.1.3 *gross sample*—the quantity of an ore consisting of all the increments taken from a consignment for any characteristic or group of characteristics; also, the composite of all these increments or subsamples each individually having been crushed or divided, or both.

3.1.4 *increment*—quantity of ore obtained by a sampling device at one time with a single operation.

3.1.5 *iron ore*—a naturally occurring material whose principal element of economic value is iron, or the products of beneficiation or processing, other than metallization of material, where iron remains or becomes the principal element of economic value.

3.1.6 *nominal size*—the opening of the screen of the standard series that would pass 95 % of a representative sample of the consignment.

3.1.7 *precision*—a measure of reproducibility of test results, using the same equipment and method, statistically derived from multiple data expressed at 95 % confidence level.

3.1.8 *quality variation* (σ_w)—a measure of the variation of a characteristic within the consignment.

3.1.9 *stratum*—quantity of an ore in routine sampling represented by one primary increment.

3.1.10 *subsample*—a quantity of an ore consisting of several increments taken from a part of the consignment; also, a composite of several increments each individually having been crushed or divided, or both.

3.1.11 *test sample*—any sample for determination of size distribution, moisture content, physical properties, or chemical composition that is prepared from each increment, each subsample, or from the gross sample in accordance with the specified method for that type of sample.

4. Summary of Practice

4.1 The precision required for the sampling and sample preparation steps are calculated based on the objectives of the testing, resulting in a sampling plan specifying the minimum weights and number of increments required for each step in the procedure. Samples are then collected, dried, blended, divided, crushed, pulverized, and ground as required by the test

---

[1] This practice is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Slags, Refractories, Cement, and Related Oxide Materials.

Current edition approved July 15, 1993. Published September 1993. Originally published as E 877 – 82. Last previous edition E 877 – 82 (1986).

[2] *Annual Book of ASTM Standards*, Vol 03.05.

[3] *Annual Book of ASTM Standards*, Vol 03.06.

methods to be utilized.

5. Significance and Use

5.1 This practice is to be used for sampling and sample preparation of iron ores and related materials, prior to use of a referee method for testing for compliance with compositional specifications for metal content or physical properties. It is assumed that all who use this procedure will be trained analysts capable of performing common laboratory practices skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide E 882.

5.2 Adequate methods for obtaining representative samples for testing the chemical and physical properties of a consignment of iron ore are essential. The sale and use are dependent on the chemical or physical properties, or both, of an ore.

5.3 The criteria to prevent bias may be used for both design of a sampling system and in checking the design of an existing system.

6. Apparatus

6.1 Any mechanical sampler is acceptable that either by design or comparison, or both (as defined in Annex A1 and Annex A4) can be shown to take nonbiased increments of at least minimum weight and number required and can handle these increments in accordance with the practice.

6.2 *Templates and Related Equipment*, to obtain increments from a stopped belt, with bias protection in accordance with Annex A2, are acceptable.

6.3 *Riffle*—A stationary sampler comprising an even number of equally sized chutes, adjacent chutes discharging in opposite direction. For use with this practice, there must be a minimum of twelve chutes with an opening width of at least 3 times the nominal size.

NOTE 1—For fine ores (<3 mm) the 3 times nominal size should be increased to the point where the plugging of chutes is eliminated. For coarse ores (>12.5 mm) it is recommended not to exceed 3½ times nominal size as it is required that the full width of the riffle be used since the accuracy of the split increases with the number of chutes. For free-flowing ores such as pellets, the 3 times top size may be reduced to 1½ times provided it is ascertained that there is no chute plugging for a particular ore type.

6.4 *Crushers*—Crushers may be jaw, cone, rotary, or other type that can reduce the particle size to the desired level without significant weight loss (less than 0.5 %) and not contaminate the sample.

6.5 *Pulverizers and Grinders*—Pulverizers and grinders may be of plate, cylinder, or other type that can reduce the particle size to the desired level. They should be made of sufficiently hardened material to prevent contamination of the sample. Also, the weight loss during pulverizing should not exceed 2.5 %.

7. Design of Sampling Operations

7.1 *Basic Requirements*:

7.1.1 The characteristics to be determined and precisions desired must be known.

7.1.2 The weight and special requirements for each test sample must be known.

7.2 *Overall Precision* (β_{SDM}):

7.2.1 Overall precision for determining the mean values of the iron content, moisture content, and percentage passing the specified size sieve (in accordance with Methods E 276 and E 389), at 95 % confidence in absolute percentage are as in Table 1.

7.2.2 Overall precisions for other characteristics shall be agreed upon between the parties concerned.

NOTE 2—Nationally or internationally accepted measurement methods should be used to determine the characteristics desired.

7.3 *Equations*:

7.3.1 Calculate overall precision as follows:

$$\beta_{SDM} = 2\sqrt{\frac{\sigma_w^{\,2}}{n}\left(1+\frac{1}{c}\right)+\frac{\sigma^2_{DM}}{\nu}} \quad (1)$$

or

$$\beta_{SDM} = 2\sqrt{\frac{\sigma_w^{\,2}}{n}\left(1+\frac{1}{\sigma}\right)+\frac{\sigma_{D^2}}{\nu}+\frac{\sigma_{M^2}}{\nu m}} \quad (2)$$

where:

β_{SDM} = overall precision for any characteristic,
σ_w = estimated within-strata standard deviation of a characteristic,
σ_D = estimated standard deviation of division,
σ_M = estimated standard deviation of measurement,
σ_{DM} = estimated standard deviation of division and measurement combined,
n = number of primary increments,
ν = number of final samples taken for measurement,
m = number of measurements taken on each final sample, and
c = average number of secondary increments taken per primary increment.

NOTE 3—Factor $(1 + 1/c)$ is omitted from the equation if only primary increments are used.

7.3.2 σ_w and σ_{DM} or σ_w, σ_D, and σ_M are estimated in accordance with Annex A3.

7.3.3 When designing a new sampling installation, refer to Annex A1 for estimating σ_w and σ_{DM}.

7.4 *Selection of Sampling Parameters*—Using the estimated values of σ_w and σ_{DM} or σ_w, σ_D, and σ_M and Eq 1 or Eq 2, choose a combination of *n, c, ν,* and *m* to obtain the required precision. It is recommended in routine sampling to use the same value of *c* used in the determination of σ_w.

7.5 *Minimum Weight of Increment*—The minimum weight of an increment is calculated by the following formula to ensure that a particle the shape of a cube of the nominal size shall not represent more than 10 % of its weight, to avoid bias by larger particles:

TABLE 1 Overall Precision

Consignment, tons	Iron and Moisture Content, %	Specification Size, Cumulative Percent Passing			
		<10 %	10–50 %	>50–90 %	>90 %
>100 000	±0.3	±0.75 %	±0.075*C*	±0.075 (100-*C*)[A]	±0.75 %
20 000 to 100 000	±0.4	±1.0 %	±0.1*C*	±0.1 (100-*C*)	±1.0 %
<20 000	±0.5	±2.0 %	±0.2*C*	±0.2 (100-*C*)	±2.0 %

[A] *C* = cumulative passing, %.

$$W = (S^3/20) \times (\text{sp gr}/5) \quad (3)$$

where:

W = minimum weight of increment, kg,
S = nominal size of the ore, cm, and
sp gr = specific gravity of the iron ore being sampled.

NOTE 4—In practice, the weight of primary increments may be many times greater than that obtained in Eq 3.

7.6 *Treatment of Increments*—Increments will be handled individually or combined to form one or more gross samples or set(s) of subsamples from which test sample(s) for the required characteristics will be taken. Each gross sample must follow the requirements of sampling and preparation. Each gross sample must have, as a minimum number of increments, the largest number (n) calculated from the individual characteristics taken from that gross sample.

7.6.1 *Example*—Assume a gross sample is required for iron analysis and moisture determination and a separate gross sample for size distribution and tumble test. Also assume from 5.4 the number of increments required to obtain precision desired is as follows:

Moisture	30 increments
Iron	20 increments
Size	50 increments
Tumble	25 increments

7.6.2 *Example*—Take 30 increments for iron analysis and moisture determination and 50 increments for size distribution and tumble test, if the sampler has the capability (for example, computer controlled). If, however, alternative increments are used, take 50 increments for *each* gross sample. If one gross sample is to be used for all the determinations, use 50 increments.

7.7 *Special Precautions*:

7.7.1 Samples for size determination or other tests requiring uncrushed particles must be taken prior to crushing.

7.7.2 Samples for moisture determination must be protected from ambient conditions. A subsample should be taken at least every 8 h and the total moisture of the consignment should be the weighted average of these samples. The 8-h period may be extended provided the sample is protected from moisture change (for example, refrigerated). To avoid moisture change, samples must be prepared as quickly as possible, with minimum handling, and must be kept in sealed containers while awaiting any stage of preparation prior to the initial weighing. Moisture samples should not be crushed below ¼-in. sieve (6.3 mm) and the minimum weight of samples used should conform with Eq 4 (7.6.1). Mix sample prior to moisture determination.

8. Sampling and Preparation Procedure (See Fig. 3 for examples)

8.1 Collect throughout the movement of the consignment, in accordance with Annex A1 or Annex A2, the number of primary increments, as determined in 7.4 (with a minimum of 20). Start at random within the first stratum, then sample at equal mass or time intervals. If the ore is handled in such a way that there is a cycle to the variability of a characteristic, it must be ascertained that the sampling cycle is *not* in phase with the handling cycle.

8.2 If the required number of increments is collected prior to completion of the movement of the consignment, additional increments shall be taken at the same interval until ore handling is complete.

8.3 If secondary increments (c) are used, they shall be taken at equal time intervals with a maximum time such that c is 1 or greater.

8.4 Increments are treated individually or combined to form a gross sample(s) or subsamples, or both, in accordance with final test sample requirements in conjunction with precision requirements, as determined in 7.4.1.

8.5 At this stage, individual test samples are obtained by a combination of division (weight reduction) (8.6), crushing and pulverizing (8.7), and drying (8.8), as directed in Section 8.

8.6 Division of gross sample, subsamples, or increment must conform with the following rule:

8.6.1 The minimum weight of the total divided sample must be greater than:

$$W_2 = S^3 \times (\text{sp gr}/5) \quad (4)$$

where:

W_2 = weight of the divided sample, kg
S = nominal size at that division level, cm, and
sp gr = specific gravity of the ore being sampled.

The equation is based on the concept that the weight of the largest piece should be less than 0.5 % of the weight of the divided sample.

8.6.2 Divide the sample by one of the following procedures:

(*a*) A mechanical sampler operated in accordance with the guidelines in Annex A1.

(*b*) *Riffling*—Use a pan the same width as the riffle chutes to feed the ore for division. Add increments of ore to the pan and gently agitate the pan over the center of the chutes, feeding the ore at a constant rate, so that any ore particle has an equal chance of falling to either side of the device. Select the half of the divided sample to be included in subsequent sampling steps, at random. Thoroughly clean the equipment between samples.

NOTE 5—**Warning:** Use proper dust collection to protect the operator from fine respirable dust particles.

(*c*) *Manual Increment Division* (Note 6)—Mix the entire sample and spread on a flat nonmoisture-absorbing surface so that the sample forms a rectangle of uniform thickness. Divide into at least 20 segments of equal area. With a flat bottom, square-nose tool, take scoopfuls of approximate equal size from each segment from the full depth of the bed. These scoopfuls must have a minimum weight in accordance with Eq 3. Combine the scoopfuls to form the divided sample.

NOTE 6—Manual increment division, although very efficient for moist or cohesive ores, or both, is not recommended for dry ores, sinter, or pellets.

8.7 *Drying, Crushing, Pulverizing, and Grinding*:

8.7.1 Always dry samples before sample preparation, if possible, to limit contamination from moist ore sticking to surfaces of sample preparation equipment.

8.7.2 Crush, pulverize, and grind samples to the required maximum size in stages convenient to the equipment available. At each stage, reduce the sample weight to the extent that the weight of the divided sample exceeds that obtained by Eq 4.

NOTE 7—**Warning:** Use proper dust collection to protect the operator from fine respirable dust particles.

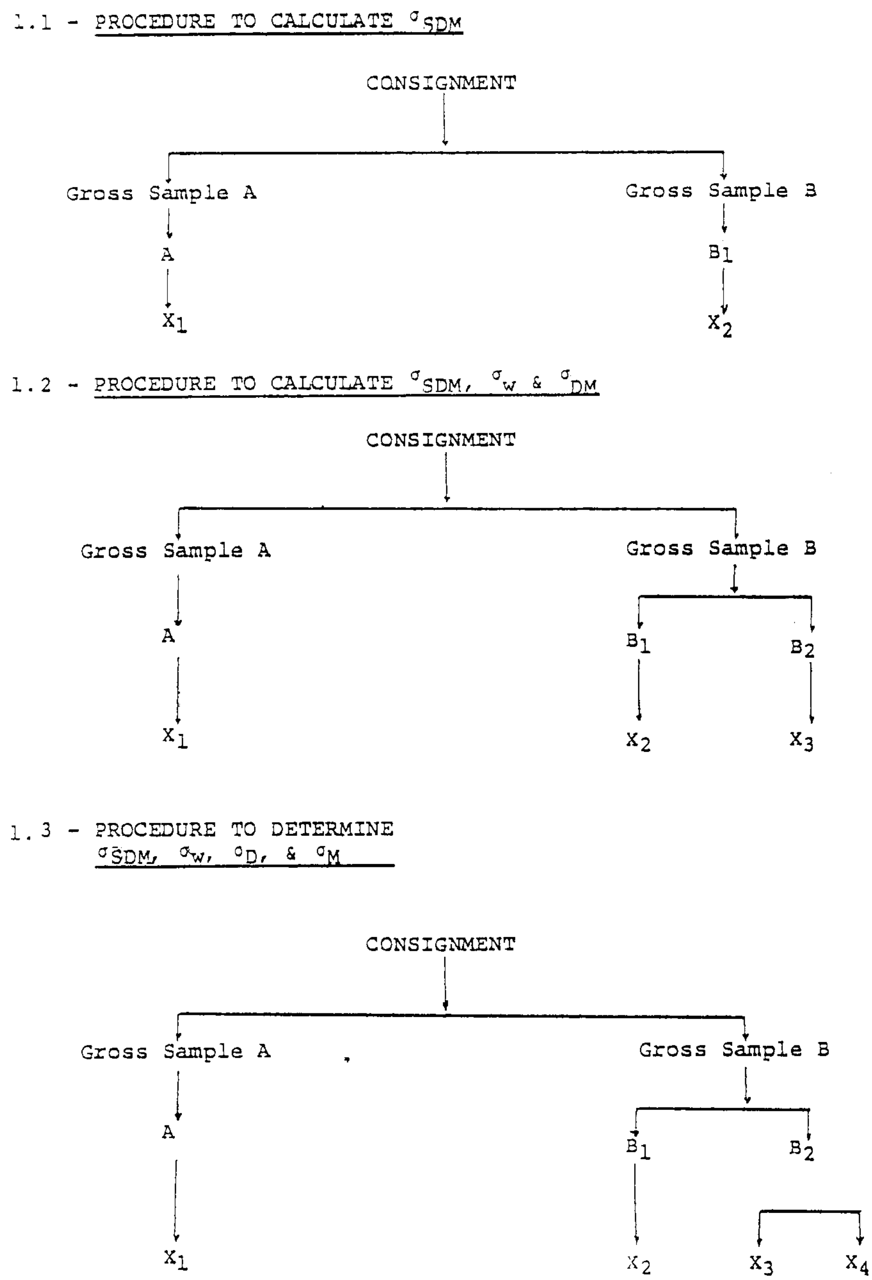

FIG. 1 Procedures for Calculating Standard Deviation

8.8 *Drying*—Drying of any portion of the sample is accomplished in any heating medium as long as the ore temperature does not exceed 110°C. Where specifications call for a dried sample, it must be dried to constant weight in an oven capable of maintaining a temperature of 105 ± 5°C. Constant weight is obtained when an additional hour drying at 105 ± 5°C does not cause a change greater than 0.05 % weight.

NOTE 8—The maximum temperature of 110°C may be exceeded, provided it is ascertained this will have no effect on any of the characteristics to be determined.

8.9 *Crushing*—Clean and preset the crusher(s) to the size required and slowly feed the sample to the crusher so as not to overload it. Ore adhering to the crushing surfaces must be added to the sample by scraping, brushing, or other means. Most ores can be crushed to pass a ¼-in. (6.3-mm) sieve in their natural state; however, pulverizing beyond this size normally requires a dried sample.

8.10 *Pulverizing and Grinding*—Clean and preset disk type pulverizers to required size and feed the sample slowly so as not to overload the pulverizer. Pass all the ore betweenthe plates. Add ore adhering to the surface of the plates to the sample by brushing or with compressed air. Grinding mills are limited to a maximum loading weight and require minimum grinding time to obtain the desired particle size. Determine these parameters experimentally for each ore type. Material adhering to the grinding surfaces must be added to the sample

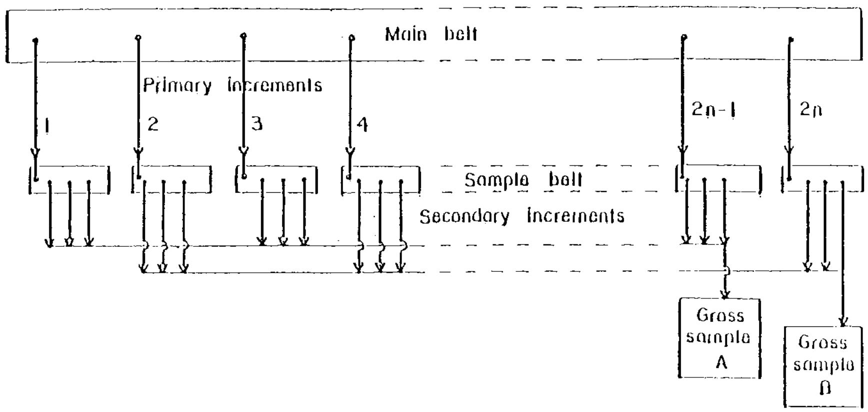

FIG. 2 Sampling Plan for Determination of Precision of Sampling and Quality Variation

by scraping or brushing, or both.

9. Test Samples

NOTE 9—Tumbler test, if required, is done in conjunction with size (see 9.1 and 9.4).

9.1 *Samples for Size Distribution*—Gross, sub, or increment samples are used in their entirety for determination of size distribution in accordance with Test Methods E 276 and E 389 or are divided by the method defined in 8.6 to a weight exceeding that calculated by Eq 4. The size distribution may be specified for samples in the natural or dried state. Dried samples are prepared as described in 8.8.

9.2 *Samples for Moisture Determination*—Gross, sub, or increment samples are used in their entirety for the determination of moisture, or are divided and crushed to the desired weight and maximum size by the methods outlined in 8.6 and 8.7.

NOTE 10—It is generally agreed that some moisture is lost in crushing; therefore, the number of steps should be minimized and the sample should not be crushed below passing a ¼-in. (6.3-mm) sieve.

9.3 *Samples for Chemical Analysis*—Test samples should be dry, weigh about 50 g, and have a maximum particle size passing a No. 100 (150-µm, 100-mesh) sieve, prepared as described in 8.6-8.8 .

9.4 *Samples for Tumbler Test*—The samples should be obtained in conjunction with the size distribution test in accordance with Method E 279.

9.4.1 *Pellets*—In size distribution, both the 1½-in. (37.5-mm) and ¼-in. (6.3-mm) sieves must be included. Sufficient sample must be screened in a unit or in batches to obtain at least 45.4 kg (100 lb) that will pass the 1½-in. sieve and be retained on the 1¼-in. sieve. This +45.4- kg sample of pellets is used for tumbler test. (Weight reduction, if required, is accomplished according to 7.2.6.2 (*a*) or (*b*).)

9.4.2 *Sinter*—In size distribution both the 2-in. (50-mm) and ⅜-in. (9.5-mm) sieves must be included. Sufficient sample must be screened in a unit or in batches to obtain at least 45.4 kg (100 lb) that will pass the 2-in. sieve and be retained on the ⅜-in. sieve. This +45.4-kg sample of sinter is used for the tumbler test. (Weight reduction, if required, is accomplished in accordance with 8.6.2 (*a*) or (*b*).)

9.5 *Sample for Compression Test*—The dry sample is obtained from division without crushing in accordance with 8.6 and 8.8. The sample weight by Eq 4, or greater, shall be such that it shall contain at least 600 pellets in the required size or the size range agreed upon.

9.6 Samples for other tests follow the general guidelines of the method. For tests that require samples with specific size range (for example, Method E 1072), the samples should be obtained by screening a quantity of material determined by Eq 4 (8.6), and taking the material at random from the required size range.

10. Keywords

10.1 crush; division; dry; grind; iron ore; pulverize; sample preparation; sampling

This method is intended to give the operator maximum flexibility in both sampling and preparation of samples, providing restrictions only to eliminate bias (*i, ii*) and to obtain required precision (*iii*):

for example (*i*) Increments must be full cross sections of a flowing stream;

(*ii*) Minimum weight of individual increments and gross samples are related to nominal size, and

(*iii*) Number of increments and samples tested are related to heterogeneity of the ore and precision desired.

Two variations of samples and sample preparation of -20 mm pellets for size distribution, moisture determination and chemical analysis:

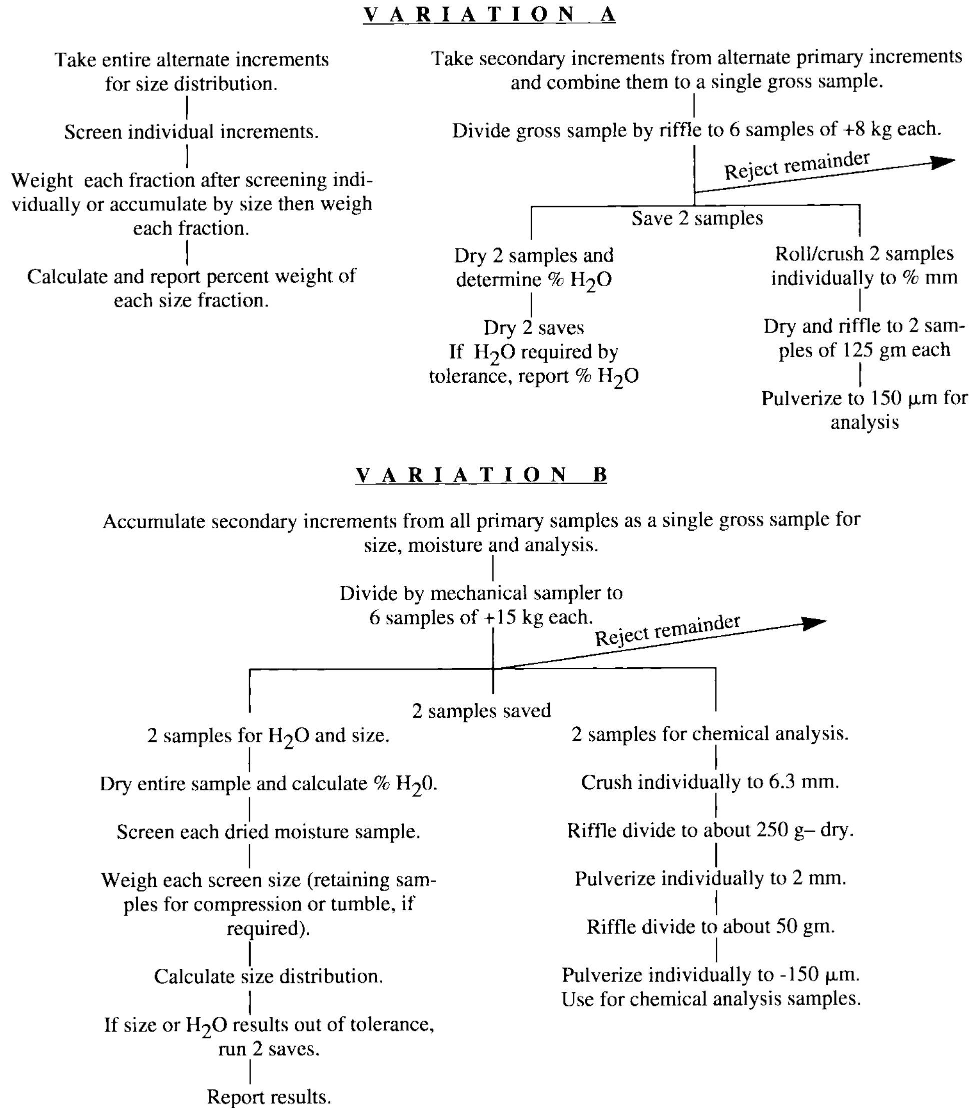

FIG. 3 Flowsheet Examples

ANNEXES

(Mandatory Information)

A1. MECHANICAL SAMPLERS

A1.1 Design Criteria

A1.1.1 The cutter shall obtain a full cross section of the stream with the leading and trailing edges describing the same path.

A1.1.2 The cutter shall have a minimum opening dimension of at least 3 times the nominal size.

A1.1.2.1 In stages of sampling where the flow of ore is low and the chances of plugging are minimal, the opening dimension may be reduced to 1½ times nominal size.

A1.1.2.2 Any ores with a nominal size of less than 3 mm, that are even slightly cohesive, should have a minimum cutter opening of 10 mm.

A1.1.3 If the sampler is bucket type, the dimensions shall be such that it will not overflow and it shall discharge completely. If it is chute type, it shall not restrict the flow of ore.

A1.1.4 There shall be no introduction of materials other than the sample into the sampling system; for example, material from belt scrapers or pulleys, collected *between* increments.

A1.1.5 There shall be no change in the quality of the sample during the taking of increments; for example, degradation of the constituent particles if the sample is taken for size determination, moisture change if the sample is taken for moisture determination, etc.

A1.1.6 When cutting the stream, the cutter shall travel at almost uniform speed ±5 %, perpendicular to the trajectory, or along an arc normal to the trajectory. Acceleration, deceleration, and point of rest must be completely out of the stream.

A1.1.7 The opening of the primary sampler shall be designed so that the cutting time of each point in the stream does not deviate more than ±5 %.

A1.1.8 In a system where the cutter is located after a conveyor, the maximum velocity of the cutter shall be such that the quantity of ore collected is greater than (*1*) the mass of a cross section of ore on the conveyor for a length equal to the effective cutter opening, or (*2*) the mass calculated in accordance with Eq 3 in 7.5, whichever is greater.

A1.1.9 All components of the sampler should be accessible for cleaning and inspection.

A1.1.10 Air flow through crushers and chutes shall be kept to a minimum to prevent moisture and dust loss.

A1.1.11 A secondary cutter, if used, shall conform to all the above characteristics (A1.1 through A1.10) and shall be out of phase with the primary cutter.

A1.1.12 The sampler should be located close to the point of weighing to minimize changes in characteristics, princi-pally moisture.

A1.2 Capability of Sampler

A1.2.1 In designing the system, it should be noted that precision evaluation is based on accumulating separately alternative increments; therefore, the system should be designed so that samples required for Annex A3 can be obtained.

A1.3 Design Factors

A1.3.1 If σ_w is not known in design stage, it may be estimated by the following formula:

$$\sigma_w = (x_{max} - x_{min})/4 \quad \text{(A1.1)}$$

where:

σ_w = estimated standard deviation of a characteristic within strata,
x_{max} = maximum value of a characteristic estimated to be obtained in any increment from consignment, and
x_{min} = minimum estimated value.

A1.3.2 If σ_{DM} for iron and moisture is not known, it is estimated to be ±0.2 %.

A1.3.3 If σ_{DM} for size is not known, the estimated value is taken from the following table:

x	σ_{DM}
$x < 10$ %	0.5 %
$10 < x < 20$	1.0 %
$20 < x < 30$	1.5 %
$30 < x < 40$	2.0 %
$40 < x < 50$	2.5 %

where x = lesser of the estimated percent passing or retained on the specification sieve.

A1.4 Bias Check

A1.4.1 Increments collected by samplers built in accordance with the design criteria of this annex should be without bias. However, the sampler should be inspected periodically to ensure all phases and components of the system continue to operate within the guidelines of this annex.

A1.4.2 Systematic differences may also be checked by comparing the results with those of stopped belt sampling taken in accordance with Annex A2, computed by Annex A4.

A2. METHOD TO OBTAIN STOPPED BELT PRIMARY INCREMENTS

A2.1 Apparatus for Belt Sampling

A2.1.1 Templates shaped to the contour of the belt at average load should be used. Two templates must be parallel and spaced at least 3 times nominal size apart, by one or more spacers placed to give minimum interference with the sample removal.

A2.1.2 Shovel(s), hoe(s), and broom(s) with widths not greater than three quarters of the template spacing are used to remove the sample.

A2.2 Safety

A2.2.1 Safety of operation must be assured by proper lockout procedures of the belt being sampled.

A2.3 Bias Prevention

A2.3.1 *General Requirements*

The ore should be removed while the templates are being lowered through the stream in such a way that:

A2.3.1.1 Negligible amount of ore comes around the template and enters the sample;

A2.3.1.2 The spacing at the bottom of the cut remains as much as possible as wide as the spacing at the top of the cut; and

A2.3.1.3 All moisture and dust accumulated in the area between templates is swept into the sample.

A2.3.2 *Specific Requirements*:

A2.3.2.1 *Lump Ore (Run-of-Mine Ore)*—Any lumps encountered by the downstream template should be placed into the sample while lumps encountered by the upstream template should be rejected. Any fines entering the sample with these lumps should, as much as possible, be rejected.

A2.3.2.2 *Concentrates*—If moisture seeps into the sample area from the other side of the templates, the sample should be removed as quickly as possible and discretion should be used in cleaning out the last of the moisture.

A2.3.2.3 *Pellets*—If, due to varying contours of the belt, pellets roll through the spaces between the templates and the belt, care should be taken to minimize this flow so that the proportion of fines to pellets is not altered. Also ensuring the bottom of the cut is as wide as the top is particularly important here, as the fines will accumulate along the belt.

A3. EXPERIMENTAL METHOD TO EVALUATE QUALITY VARIATION (σ_w), OVERALL PRECISION (β_{SDM}), PRECISION OF DIVISION AND MEASUREMENT (β_{SDM}), PRECISION OF DIVISION (β_D), AND PRECISION OF MEASUREMENT (β_M) OF AN ORE TYPE

A3.1 Plan of Experiment

A3.1.1 *Number of Consignments*—At least 10 consignments of the same ore type of approximately equal tonnage (±20 %) shall be used for this investigation. However, a larger cargo can be divided into parts and each part representing the tonnage range used may be considered as a separate consignment for this experiment.

A3.1.2 *Sampling, Sample Preparation, and Testing*—Additional sampling, sample preparation, and testing for this investigation shall be carried out as in routine practice. Routine sampling for the determination of quality of the consignment should be one of the two final samples required (Fig. 1).

A3.1.3 *Number of Increments*—The minimum number of primary increments required for this investigation is twice the number of primary increments required for routine sampling ($2n$).

NOTE A3.1—In routine sampling, a minimum of 20 increments are required; therefore, $2n$ must be greater than 40.

A3.1.4 Each primary increment is diverted alternatively to gross Samples A and B. The number of secondary increments (c) per primary increment should be the same as those taken for routine sampling. A plan of gross Samples A and B, each consisting of three secondary increments, is shown as an example in Fig. 2.

A3.1.5 *Sampling Division Plan*—Gross Sample A is processed in accordance with routine practice to obtain final Sample A. Gross Sample B is also processed according to routine practice, if only β_{SDM} is required. If β_{DM}, β_D, or β_M are required, gross Sample B is divided into two parts, each part then processed in accordance with routine practice.

A3.1.6 *Measurement Plan*:

A3.1.6.1 *Only β_{SDM} to Be Determined*—The measurement X_1 of final Sample A and the measurement of X_2 of final Sample B_1 are determined in accordance with routine practice (Fig. 1–1.1)

A3.1.6.2 *β_{SDM}, σ_w, β_s, and β_{DM} to Be Determined*—Over the above measurement X_1 and X_2 in A3.1.6.1, measurement X_3 of final Sample B_2 is determined (Fig. 1–1.2).

A3.1.6.3 *β_{SDM}, σ_w, β_s, β_D, and β_M to Be Determined*—Over and above the measurements X_1, X_2, and X_3 from A3.1.6.2, a second measurement X_4 of final Sample B_2 is made (Fig. 1–1.3).

A3.2 Computation of Results

A3.2.1 *Overall Precision* (β_{SDM})*:*

$$\text{Compute } R_{i1} = [X_{i1} - X_{i2}] \quad (A3.1)$$

$$\bar{R}_1 = \sum_i^k R_{i1}/k \quad (A3.2)$$

where k is the number of consignments used;

$$\sigma_{SDM} = \bar{R}_1/d_2 \quad (A3.3)$$

where $1/d_2 = 0.8865$ (for a pair of measurements);

$$\beta_{SDM} = 2\sigma_{SDM} \quad (A3.4)$$

A3.2.2 *Compute* β_{DM}, β_s, and σ_w:

A3.2.2.1 Compute $R_{i2} = [X_{i2} - X_{i3}]$

$$\bar{R}_2 = (\sum_i^k R_{i2}/k) \quad (A3.5)$$

$$\sigma_{DM} = \bar{R}_2/d_2$$

$$\beta_{DM} = 2\sigma_{DM}$$

A3.2.2.2

$$\sigma_s = \sqrt{(\sigma_{SDM})^2 \ (\sigma_{DM})^2} \ (\sigma_{SDM} \text{ from A3.2.1}) \quad (A3.6)$$

$$\beta_s = 2\sigma_s \quad (A3.6)$$

$$\sigma_w = \sigma_s\sqrt{n/(1 + 1/c)} \quad (A3.6)$$

A3.2.3 *Compute* β_M and β_D:

A3.2.3.1 In most cases, if a standard method of measurement is used, σ_M is known and σ_D can be calculated by Eq 2.

A3.2.3.2 In the case where σ_M is not known, it can be calculated as follows:

$$R_{i3} = [X_{i3} - X_{i4}] \quad (A3.7)$$

$$\bar{R}_3 = (\sum_i^k R_{i3}/\ k) \quad (A3.7)$$

$$\sigma_M = \bar{R}_3/d_2 \quad (A3.7)$$

$$\beta_M = 2\sigma_M \quad (A3.7)$$

$$\sigma_D = \sqrt{(\sigma_{DM})^2 - (\sigma_M)^2} \quad (A3.7)$$

$$\beta_D = 2\sigma_D \quad (A3.7)$$

A4. METHOD FOR CHECKING SYSTEMATIC DIFFERENCE BETWEEN TWO SAMPLING PROCEDURES

A4.1 General Conditions

A4.1.1 In this experiment, the results obtained from two sampling methods of the same consignments, both following this practice, are compared for systematic differences.

A4.1.2 In the event that there is no significant difference in a statistical sense between the results obtained from Method 1 and Method 2, both procedures are presumed to be equally accurate.

NOTE A4.1—The no significant difference means the value from one procedure does not depart from the value of the other procedure by more than the difference caused by random fluctuation at the 5 % level of significance.

A4.1.3 The number of consignments on which the differences are based shall be 10 or more.

A4.1.4 Quality characteristics may be iron content, moisture content, particle size distribution, or other, as the case may be.

A4.2 Experimental Methods

A4.2.1 Methods for constituting a pair of gross samples, preparation of samples, and testing shall be as follows:

A4.2.1.1 Increments obtained from one consignment in accordance with Method 1 and Method 2 are grouped respectively, so as to constitute a pair of gross Samples 1 and 2.

A4.2.1.2 The gross Samples 1 and 2 are subjected to sample preparation and testing separately, and a pair of measurements obtained.

A4.3 Analysis of Experimental Data

A4.3.1 The method of analysis of experimental data shall be as follows:

(*1*) Denote measurements obtained in accordance with Method 1 and Method 2 by ^xA_i and ^xB_i respectively.

(*2*) Calculate the difference between ^xA_i and ^xB_i by the following equation:

$$d_i = {}^xB_i - {}^xA_i \qquad i = 1, 2, ..., k \tag{A4.1}$$

where k is the number of consignments.

(*3*) Calculate the mean of the differences to one decimal place further than that used in the measurements themselves:

$$\bar{d} = \frac{1}{k} \Sigma d_i \tag{A4.2}$$

(*4*) Calculate the standard deviation of the difference:

$$s_d = \sqrt{\{[\Sigma(d_i^2) - \Sigma(d_i^2)/k]/(k-1)]\}} \tag{A4.3}$$

(*5*) Calculate the value of t_o to the third decimal place by rounding the fourth decimal place:

$$t_o = \bar{d}(\sqrt{k})/S_d \tag{A4.4}$$

(*6*) When the absolute value of t_o is smaller than the value of t corresponding to k, as given in Table A4.1, conclude that the difference is not significant in a statistical sense.

(*7*) When the value of t_o is larger than that shown in Table A4.1, it can be concluded that systematic difference exists. If the difference of the means of Method 1 and Method 2 is physically or economically significant, both procedures should be checked for bias in accordance with Annex A1 and Annex A2.

TABLE A4.1 Value of *T* at 5 % Significance

Number of Pairs	Student's T
10	2.262
11	2.228
12	2.201
13	2.179
14	2.160
15	2.145
16	2.131
17	2.120
18	2.110
19	2.101
20	2.093
30	2.045