Designation: E877 – 21

Standard Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition and Physical Properties[1]

This standard is issued under the fixed designation E877; 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 concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E276 Test Method for Particle Size or Screen Analysis at 4.75 mm (No. 4) Sieve and Finer for Metal-Bearing Ores and Related Materials

E279 Test Method for Determination of Abrasion Resistance of Iron Ore Pellets, Lump, and Sinter by the Tumbler Test

E389 Test Method for Particle Size or Screen Analysis at 4.75 mm (No.4) Sieve and Coarser for Metal-Bearing Ores and Related Materials

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1072 Test Method for Low Temperature Breakdown of Iron Ores (Withdrawn 1995)[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

3.1.1 *nominal topsize, n—in sampling,* the opening of the screen of the standard series that would pass 95 % of a representative sample.

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

4. Summary of Practice

4.1 The precision required for the sampling and sample preparation steps is calculated based on the objectives of the testing, resulting in a sampling plan specifying the minimum masses 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 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

[1] This practice is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide E882.

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 mass 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, adjacent chutes discharging in opposite directions. For use with this practice, there must be a minimum of twelve chutes with an opening width of at least 3 times the nominal topsize.

NOTE 1—For fine ores (< 3 mm), the 3 times nominal topsize 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 topsize 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 the nominal topsize 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 loss of mass (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 loss of total mass 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 mass 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 Test Methods E276 and E389), at 95 % confidence in absolute percentages are as in Table 1.

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

TABLE 1 Overall Precision

Consignment, tons	Iron and Moisture Content, %	Specification Size, Cumulative Percent Passing			
		< 10 %	10 % – 50 %[A]	> 50 % – 90 %[A]	> 90 %
> 100 000	± 0.3	± 0.75 %	± 0.075*C*	± 0.075 (100-*C*)	± 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] In the formulae for calculating the precision estimates within this column, *C* = cumulative percent passing.

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:

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

or

$$\beta_{SDM} = 2\sqrt{\frac{\sigma_w^2}{n}\left(1+\frac{1}{\sigma}\right)+\frac{\sigma_D^2}{v}+\frac{\sigma_{M^2}}{vm}} \quad (2)$$

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,
v = 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, v,* 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 Mass of Increment*—The minimum mass of an increment is calculated by the following formula to ensure that a particle the shape of a cube of the nominal topsize shall not represent more than 10 % of its mass, to avoid bias by larger particles:

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

where:

W = minimum mass 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 mass 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 7.4 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 6.3 mm (¼ in.) sieve and the minimum mass of samples used should conform with Eq 4 (8.6.1). 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 7.3.1.

8.5 At this stage, individual test samples are obtained by a combination of division (mass 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 mass of the total divided sample must be greater than:

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

where:

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

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

8.6.2 Divide the sample by one of the following procedures:

8.6.2.1 A mechanical sampler operated in accordance with the guidelines in Annex A1.

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

8.6.2.3 *Manual Increment Division* (Note 5)—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 mass in accordance with Eq 3. Combine the scoopfuls to form the divided sample.

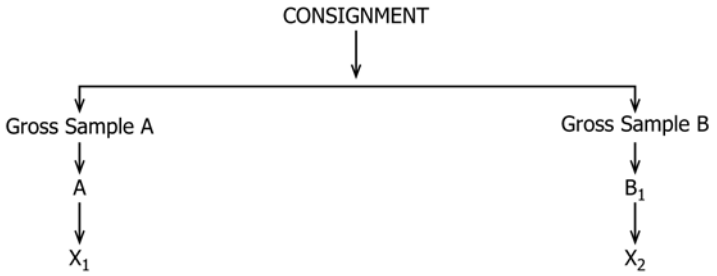
NOTE 5—Manual increment division, although very efficient for moist or cohesive ores, or both, is not recommended for dry ores, sinter, or pellets.

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

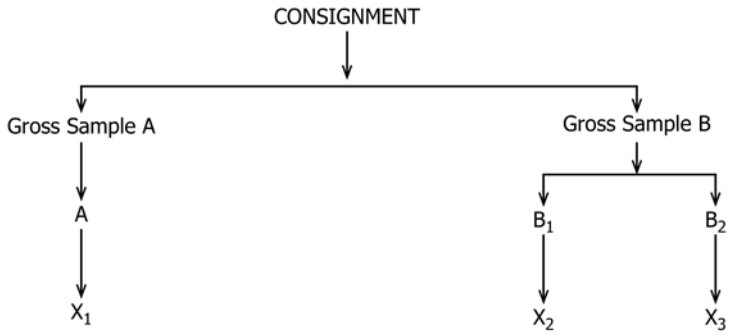
8.7.1 Always dry samples before sample preparation, if possible, to limit contamination from moist ore sticking to surfaces of sample preparation equipment.

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 mass to the extent that the mass of the divided sample exceeds that obtained by Eq 4. **Warning—**Use proper dust collection to protect the operator from fine respirable dust particles.

1.1 - PROCEDURE TO CALCULATE σ_{SDM}



1.2 - PROCEDURE TO CALCULATE σ_{SDM}, σ_w & σ_{DM}



1.3 - PROCEDURE TO DETERMINE
σ_{SDM}, σ_w, σ_D, & σ_M

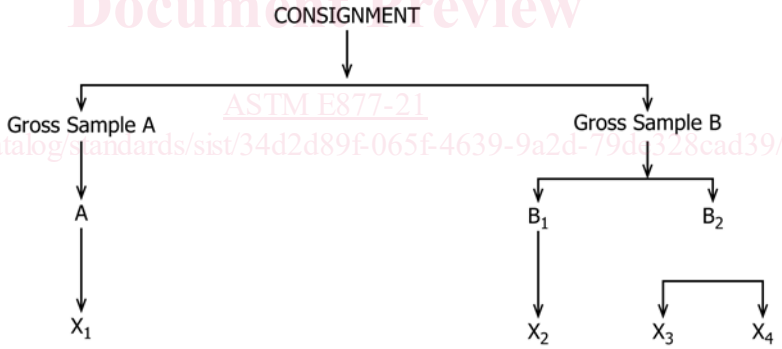


FIG. 1 Procedures for Calculating Standard Deviation

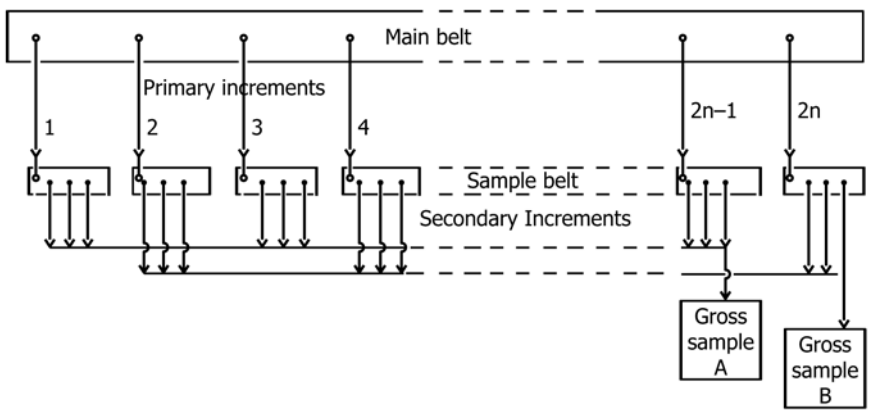


FIG. 2 Sampling Plan for Determination of Precision of Sampling and Quality Variation

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:

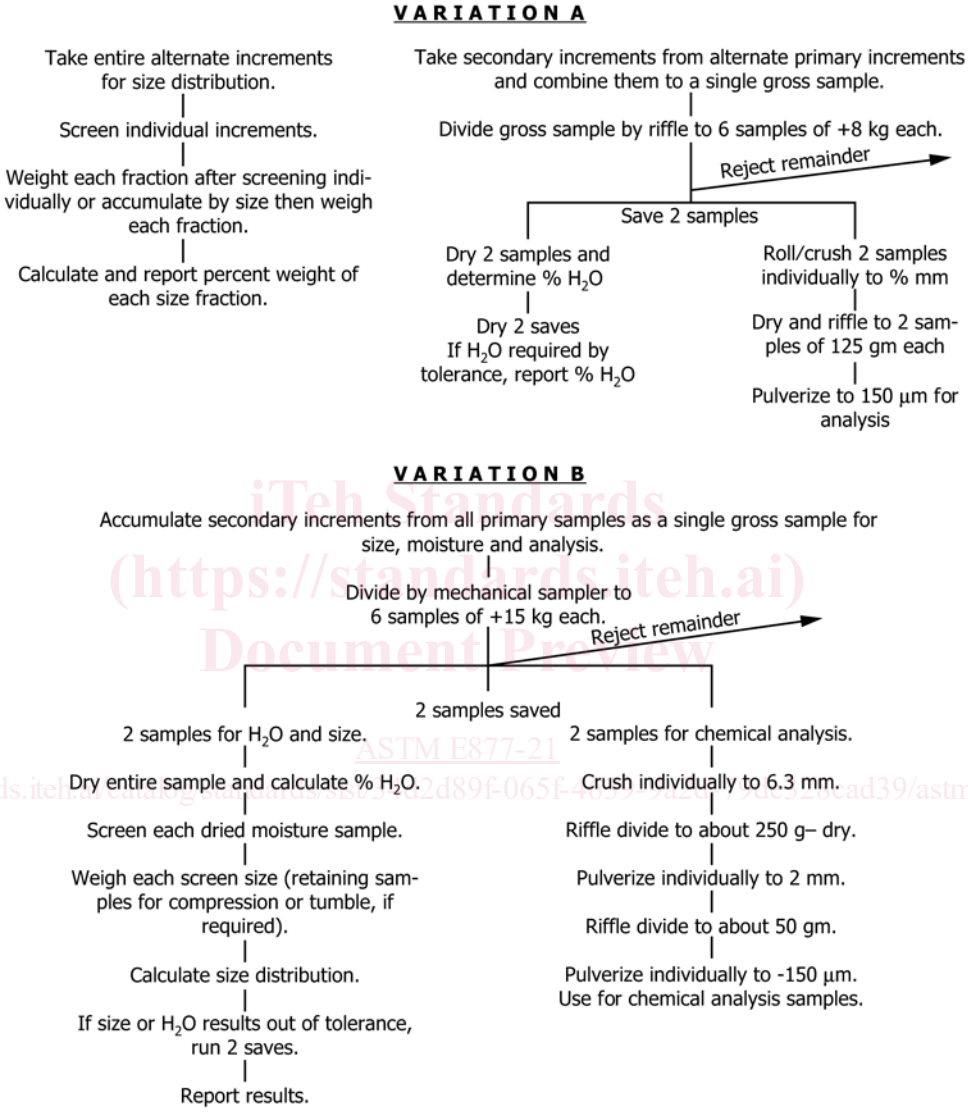


FIG. 3 Flowsheet Examples

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 mass in an oven capable of maintaining a temperature of 105 °C ± 5 °C. Constant mass is obtained when an additional hour drying at 105 °C ± 5 °C does not cause a change greater than 0.05 % mass.

NOTE 6—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 6.3 mm (¼ in.) sieve in