Defense Logistics Agency
STANDARD OPERATING PROCEDURE

DLAStratMat-MO-0000.02
Effective Date November 19, 2014

DLA Strategic Materials 4145.1

SUBJECT: CHAPTER 7 SAMPLING STOCKPILE MATERIALS

References:

1. PURPOSE: This Standard Operating Procedure (SOP) establishes and implements instructions and procedures for sampling stockpile materials. It explains the importance of sampling, defines basic sampling terminology, and states the role of the Specialist in the selection and control of samples. It provides procedures for the preparation for sampling, obtaining representative samples, care of the samples and equipment, forwarding of samples for analyses and tests, and a description of factors affecting the sampling method and sample size.

2. APPLICABILITY:

   This SOP applies to all DLA Strategic Materials personnel assigned to material handling duties at DLA Strategic Materials depots.

3. DEFINITIONS: See Enclosure 3

4. RESPONSIBILITIES: See Enclosure 1

5. PROCEDURES:

   a. OVERVIEW: This section is applicable to all types of sampling performed for the disposal or acquisition of stockpile materials. This encompasses the importance of sampling, defines basic sampling terminology, and outlines basic sampling procedures and general sampling methods for the physical form and type of material. A table listing the sampling method, sample size and type container for the sample for each stockpile commodity and a section on sales contract sampling is also included. The sampling procedures and methods presented are intended to standardize, insofar as possible, the sampling to be performed by DLA Strategic Materials' Specialists. Also, more detailed contract provisions or instructions may supersede contractors performing this type of service for DLA Strategic Materials. This describes the importance of the sampling function; defines terms applicable to sampling; and outlines steps to be taken in the

collection of samples to determine the value and quality of materials offered for inspection.

b. DESCRIPTION: See Enclosure 2

6. INFORMATION REQUIREMENTS:

a. DLA Strategic Materials designated personnel will review and update processes as needed.

7. INTERNAL CONTROLS:

a. Samples of material for analysis, test, or appraisal must be selected by or under the supervision of a Specialist. When sampling is performed under a service contract, the Specialist must be present when sampling is started and spot checks performed frequently thereafter. All necessary measures must be taken to assure that sampling is performed in accordance with terms of the contract or specified standard.

b. Samples required to be sent to a laboratory must be sent by the Specialist and not by the producer. When sampling is performed under a service contract, the service contractor will forward the samples. The selection and preparation of representative samples for shipments or lots are often laborious and expensive; therefore, a close check or control must be maintained on all samples to be submitted to laboratories for testing.

c. The Specialist will conduct, supervise, or witness required tests in accordance with methods prescribed in the contract or applicable specifications.

8. RELEASEABILITY: Unlimited. This SOP is approved for public release and is available on the Internet.

9. EFFECTIVE DATE: This SOP is effective on January 1, 2014 and remains in effect until either superseded or cancelled.

GARY D. PORTER
DIRECTOR OF MATERIAL MANAGEMENT

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ENCLOSURE 1

ROLES/RESPONSIBILITIES

1. CHIEF OF OPERATIONS AND LOGISTICS will:

   a. Assign Commodity Logistics Specialist or General Supply Specialist to oversee sampling.

   b. Review results outcome and provide final approval.

   c. Provide direction to the Program Manager to disseminate sampling results.

2. COMMODITY LOGISTICS SPECIALIST/PHYSICAL SCIENTIST will:

   a. Create statement of work.

   b. Will act as COR on sampling contract.

   c. Compile results for review and approval.

3. PROGRAM MANAGER will:

   a. Disseminate results to applicable parties.

   b. File results in evidential matter folder.

ENCLOSURE 2

PROCEDURES

BASIC SAMPLING

PURPOSE:

This part prescribes procedures for the preparation for sampling, obtaining representative samples, care of the samples and equipment, forwarding of samples for analyses and tests, and a description of factors affecting the sampling method and sample size.

PREPARATION FOR SAMPLING:

a. Weight Check. A statistical sample of containers in each lot of packaged materials will be selected for weight checks. The sample will be selected according to ANSI/ASQC Z1.4, Sampling Procedures and Tables - Special Inspection Level S2. Each container selected will be emptied and the exact gross, tare, and net weight determined. Compensation must be made in calculating the weight of containers from which stockpile material has been removed.

b. Equipment. The maintenance of all sampling equipment in good working condition is essential for accuracy in sampling. The use of worn or battered equipment on bulk ores, for example, affects not only the size of the sample portion but also the distribution of coarse and fine material, with the result that the sample is not representative of the lot. In coning and quartering, the relative distribution of coarse and fine material can be appreciably altered by an unevenly worn edge on the shovel. Also, a bent dividing partition in a riffle changes the relative size of the sample discharges. All sampling equipment and the containers in which the samples are placed must be inspected for use and carefully cleaned both before and after use to avoid contaminating the sample with dust or dirt or with particles of the material on which the equipment was last used.

METHODS FOR OBTAINING A REPRESENTATIVE SAMPLE:

The method for obtaining a representative sample varies according to the physical characteristics of the commodity; i.e., whether the material is liquid or solid, free flowing or viscous, homogeneous or heterogeneous, and according to other factors dealt with in FACTORS AFFECTING THE SAMPLING METHOD AND SAMPLE SIZE. Contractual requirements for inspection, sampling, and testing, when specified or referenced, will be followed.

a. Random Sampling. Samples must be taken either from scattered locations or at points uniformly distributed throughout the lot. A random sample is drawn so that each item or portion in the lot has the same chance of being the first item in the sample, regardless of its position, quality, or appearance. After the sample is drawn, each of the remaining items in the lot should have the same chance of being the second item in the sample. The Specialist may use either a random sampling table or random numbers generated by a computer or calculator with statistical functions for this type sampling.

b. Formation of a Composite Sample. When the material being sampled varies appreciably in size and composition. The Specialist must make sure that individual sample portions forming the gross or composite sample are representative of those parts of the material from which they were drawn, rather than try to make each portion representative of the entire lot.

c. Biased Sampling. The Specialist's must avoid biased sampling procedures such as preference for easily accessible units or following routine selection patterns that are easily recognized and involve frequent choice of units in the same sequence. Examples of these are: taking items from the same position in containers, stacks, or piles in every inspection; taking items from the top of a container; not taking items from the top of a container; or taking items from the output of certain identical production elements and not from others.

CARE OF SAMPLES:

During the entire sampling process, from the time gross sample is taken until the laboratory sample is packed and sealed in a container for shipment. The sample must not be subjected to any conditions that could alter the quality or composition of the material, or allowed to be contaminated with foreign matter from any outside source. Sample containers must be clean (free of dirt, oil, grease, etc.) and be of the appropriate type in order to prevent contamination or loss of the sample material. Samples not adequately protected or exposed to any condition that may affect a volatile or vital property of the material are no longer representative of the shipment or lots from which they were drawn.

VALIDITY OF SAMPLES:

The Specialist must be able to vouch for the validity of a sample. That is from the time of sampling until delivery to the analyst. The laboratory sample must be placed in proper containers immediately after sampling. These containers must be sealed so that tampering can be detected and delivery initiated at once. It is the responsibility of the Specialist to procure sufficient sets of security type seals for sealing and identifying samples, and, where necessary and appropriate, for packages and vehicles.

FACTORS AFFECTING THE SAMPLING METHOD AND SAMPLE SIZE:

The factors affecting the method used in sampling a shipment of any particular commodity and the size of the sample to be taken may include any or all of the following:

a. Physical characteristics of the material. The difficulty in securing a representative sample and sample size increases as the character of the commodity advances from free-flowing to a viscous liquid, a semisolid, and a solid.

b. Bulk or packaged material. Bulk shipment is used for large lots of moderately coarse material. Sampling can best be accomplished by mechanical means while the material is moving into or from the carrier's conveyance. As the value of the material and the fineness of particle size increases, material must be packed to prevent loss or contamination during shipment and handling.

c. Size of lot delivered. The size of the sampling should always be based on the size of the lot as well as failure rates and other historical data.

d. Accuracy of analytical methods. A sample should represent the original material to within the same degree of accuracy that can be obtained from the analytical methods used in evaluating the sample.

e. Use of samples. The method of sampling, the amount required, and the treatment of the sample varies to some extent with the character of the test to be performed or the use which will be made of the sample.

f. Conditions under which sampling must be done. A poor location with insufficient room for proper handling, unfavorable weather in an outside location, shortage of labor for handling, or the lack of a particular type of sampling equipment may prevent the use of the most desirable sampling procedure. Other important factors are whether sampling can be done while loading or unloading, or from a railroad car.

TRANSMISSION AND DISPOSITION OF SAMPLES:

The following procedures must be used in the identification, distribution, transmission, and disposition of samples under Defense National Stockpile programs when samples are sent to a Government or Government contracted laboratory for analyses or test:

a. Identification of samples. The DNSC Form 34 Sample Identification Label is applicable for medium or large size sample containers but not for small size containers such as vials or small bottles. Sample ID labels for vials or small bottles should be of a size large enough to identify the sample by at least the "sample number" and other pertinent information that will fit on the label. See instructions below for preparing the Sample Identification Label.

b. Distribution of samples. The required number of identical portions which were obtained from the sub-sample or gross sample, each of which represents the lot, is distributed as follows:

    1. If required, one must be sent or given to the prime contractor, or their designee, who, in accordance with contract terms or at their own discretion, may have tests and analyses performed.

    2. One, hereafter referred to as the "Government's laboratory sample", must be sent to the laboratory specified for analyses and/or tests by the Chief, Operations Division.

    3. Unless otherwise directed, two representative portions (one held in reserve and one held in case an umpire analysis is required) will be retained by the designated DLA Strategic Materials field office.

c. DNSC Form 35, Record of Samples Transmitted and Request for Analyses. Record of Samples Transmitted and Request for Analyses must be used when forwarding samples for tests and/or analyses to a Government contracted or Government laboratory. The distribution of the form, analysis certificates, and invoice is printed thereon.

d. Disposition of samples. Discarded portions (those portions not used in the sample reduction) of the gross samples or of partially reduced gross samples may be returned immediately to the material or placed with the shipment. The reserve portions of the gross must be held until the analysis certificate is received and it appears that there will be no further need for it. The rejects from the portion sent to the laboratory are normally held by the laboratory for 60 days, except those for moisture determination which need not be kept, and the chemical pulp and umpire samples are retained for 6 months. Unused portions of samples of precious metals, such as palladium, must be returned to the Government when their weight was included in that of the metal bought by the Government or not included in that sold by the Government.

SALES CONTRACT MOISTURE DETERMINATION:

A solicitation that includes moisture determination shall be made in accordance with standard commercial procedures by an independent sampler/analyst. The independent sampler/analyst shall furnish to the Government for review and approval the proposed sampling methods and moisture analysis procedures prior to out loading. These documents as well as the certificates of moisture shall be forwarded to:

Directorate of Material Management
DLA Strategic Materials
8725 John J. Kingman Road
Suite 3229
Fort Belvoir, VA 22060-6223
FAX: 703/767-7611

For offers submitted, moisture determination shall be made in accordance with standard commercial procedures (drying to a constant weight at 105 degrees centigrade) by an independent sampler/analyst. To be designated by the Contractor and acceptable to the Government. The results of the analysis is final and binding. Certificates of moisture shall be furnished simultaneously to both parties by the independent sampler/analyst.

A lot is defined as the quantity of ore out loaded for which a weight, moisture and analysis certificate is applicable. The lot size shall be mutually agreed upon by the Government and the Contractor but shall not exceed 5,000 SWT.

The sample portions of each lot shall be taken at the rate of one pound per ton, during out loading, from the face of the pile where the material is being removed. The sample will be taken from every third truckload or every 50 SWT if being loaded into railcars. A sample shall not be accumulated for more than five days.

Should the Contractor waive moisture determination by an independent sampler/analyst, the net weight at the time of out loading will be recorded as dry weight for payment purposes.

SAMPLING METHODS

SCOPE:

a. This section prescribes sampling methods to be followed by DLA Strategic Materials' Specialists and by contractors performing services for DLA Strategic Materials.

b. The methods are based upon recognized and accepted industrial practices and must be applied as standards in the sampling of commodities listed herein. These methods are general in scope since they must be adaptable to varying conditions encountered in sampling operations.

c. Specific sampling and analysis procedures that are stated in inspection and sampling plans prepared by DLA Strategic Materials or stated in purchase, disposal, sampler/analyst, and analysis contracts shall always have priority to the general sampling plans listed below.

SAMPLING METHOD NO. 1:

This method is applicable to free flowing powders, granules, small crystals, and other finely divided materials that tend to segregate or stratify by gravity into layers of different compositions. It covers material received in boxes, bags, barrels, drums, and other containers too large to be sent to the laboratory.

a. Apparatus: The apparatus, called a sample trier or thief, to be used on materials in this category consists essentially of two slotted tubes, one of which fits within the other. It can be taken apart readily and cleaned by brushing. The original Minnesota State grain trier or the Grain Sampler recommended by the Association of Official Agricultural Chemists (AOAC) can be used.

b. Gross sample:

1. For commodities received in containers other than bags, 10 percent of the containers in any shipment or inspection lot must be opened for inspection and sampling. The percentage of containers sampled may be increased if the Specialist determines that the character of the material requires additional sampling.

2. For commodities received in bags, the rates of sampling will be as follows:

a. For lots containing 10 tons net or less, a sample will be taken from 10 percent of the bags, or from 20 bags, whichever is the greater number.

b. For lots ranging in size from 10 tons to 100 tons, samples will be taken from 20 bags, plus one additional bag for each additional ton of material in excess of the first 100 tons.

c. For lots exceeding 100 tons net, samples shall be drawn from 100 containers, plus one additional container for each additional 2 tons of material in excess of the first 100 tons.

d. The sampling procedure described in (a), (b), and (c), above, applies to material in bags having a net content of approximately 100 pounds or less. For greater net weights, the number of containers sampled may be reduced in proportion to the percentage of increase in weight over 100 pounds net per container. Containers to be sampled must be selected at random, as nearly as possible, from different parts of the lot. The actual sampling operations will differ according to containers and a condition under which sampling is accomplished. But a cross section of the material sampled must be obtained from the top to bottom of the containers, to eliminate poor representation due to settling or stratification of mixtures or powders of different degrees of fineness or specific gravity. Therefore, the special tier described above should be inserted from either end of the container through to the opposite end and, if possible, diagonally.

3. The accumulated samples from 10 percent of bags or other containers in a 1,000-unit lot will amount to about 20 pounds. The total sample taken from one lot will be thoroughly mixed and then riffled down through an approved riffler to an amount that will provide the required number of samples.

c. Laboratory sample: Three samples are normally required, one for the seller, and two for the Government, one of which is called the umpire. If the vendor desires more than one sample, it must be prepared along with the others. After riffling the composite sample down to at least the minimum amount required for the final samples. The material is again thoroughly mixed and placed on a large sheet of heavy wrapping paper or plastic sheeting, spread out in a layer about 1 inch thick on the mixing surface by coning and flattening, and then divided into the required number of portions.

SAMPLING METHOD NO. 2:

This method of sampling is applicable to solids of known uniform chemical composition, such as sebacic acid, quebracho, or similar loose solids in the form of lumps, flakes, crystals, cubes, sheets, powder, etc. of uniform chemical composition and contained in ships' holds, railroad cars, bags, drums, barrels, boxes, or other containers.

a. Apparatus: A trier or thief (so-called butter trier) measuring 14 inches overall length, with a half-cylinder stainless steel blade approximately 13 inches long with greatest diameter eleven-sixteenths of an inch near the handle and tapering uniformly to nine-sixteenths of an inch near the rounded, sharp digging end, may be used. This trier is used in the case of material in barrels, by first boring 1-inch holes through top or side of the barrel, inserting the trier, removing a portion for sample, and closing the hole in the barrel with a cork stopper or wooden plug. The trier may also be pushed through the walls of a jute or cloth bag, a portion of the contents removed, and the hole closed by sewing. Shovels, spoons, and hands may be used, where applicable. Hammers may be necessary to reduce lumps and, in conjunction with chisels, to chip or break solid masses. Jones'

samplers will be found convenient in the operation for reducing the gross samples to laboratory size.

b. Gross sample: This method requires judgment (based on the character of the materials being sampled). Lumps should be selected to truly represent the material. It is most important to secure a proper ratio between the larger pieces and the finer powder that is practically always present. Uniformly fine materials present much less difficulty in sampling, but due to various causes, the condition of the outside and surface portions may differ from that of the interior. Therefore, it is always advisable to use a trier. In all cases, the sampler must be satisfied that the sample is typical of the whole and not merely of a portion. If individual containers of the same lot appear to differ in any way, samples of the differing material must also be sent to the laboratory. Material packed in barrels must be sampled by removing the heads and taking three trier samples. One near the center, and one radially on each side of the center halfway between the center and side; or holes may be bored through the side of the barrels, the trier portion removed, and the holes closed with cork stopper or wooden plugs. Bags must be pierced with the 14-inch trier, in places equally distant from each other, portions removed, and holes sewed up. Knitting can close the hole with a sharp pointed instrument. Ten percent of barrels, bags, and similar containers in each lot must be sampled. The gross samples should be equal to 1/10 percent of the lot, but never less than 10 pounds.

d. Laboratory sample: The gross sample, which must be collected in bags or buckets, is transferred to a smooth surface, preferably steel, and the lumps or other large pieces broken up. After crushing, mixing, coning, and quartering, the sample is placed on a clean cloth or paper and rolled. The rolled material is spread out with a spatula and small amounts selected from points all over the spread material so that the final sample will be representative. If the spread material is fine, the portion for the laboratory sample may be selected by means of an approved riffler.

SAMPLING METHOD NO. 3:

This method covers hand sampling of heterogeneous solids in various forms. It is recommended only where sampling machinery is not available. The sampler, bearing in mind the particular conditions occurring at the place of sampling must work out each problem. The methods will vary depending upon the type of material such as coarse, fine, or mixtures of both, as well as the containers.

a. Apparatus: Short and long-handled shovels; coal forks with suitable rounded points and others with square digging edges. Wheelbarrows; light and heavy hammers and mauls; gross sample buckets and bags; spatulas; triers as described in method 2. Pipe samplers 6 to 8 feet long and 2 inches in diameter, having a narrow slot lengthwise starting a foot or more from handle end and ending within a few inches of the opposite, sharpened, circular end; and crushers and grinders.

b. Gross sample:

1. Mechanical sampling, the most efficient and economical method, must be used whenever possible. This process produces approximately 2 to 3 pounds of sample for

each net ton of original solids. At remote stockpile sites, where there already is an analysis of record, the gross sample rate could be set at approximately 1 to 2 pounds per ton - the most important element being the care with which the sample is taken and how representative it is of the material sampled. The sample produced in this manner will pass through an eight-mesh screen. The amount of sample at this stage from 100-ton lot solids would be 320 pounds. This is mixed, coned, and quartered to 50 pounds (the gross sample).

2. Hand sampling of coarse and fine solids loaded in bulk into railroad cars is to be accomplished from 12 spots in the material in each car. One in each corner of the car near the bottom of the pile, and one in each corner near the top, and four of the material near the bottom of the pile. A total of 50 to 100 pounds of material is to be obtained from each car. When material is sampled in trucks, a similar procedure must be followed, with a proportionate number of spots selected and quantity taken. These samples can be taken with shovel or, if the material is fine or soft, with pipes driven into the material. The sample portions from all of the car or truck loads of material comprising the lot are to be crushed with a crusher, or broken by hammers and mauls if no crusher is available, so that no lumps exceed 2 inches in size. The quantity is then reduced to approximately 200 to 500 pounds, depending on the size of the lot, by mixing, coning, and quartering. When a power crusher is not used a hard, clean surface, free from cracks and protected from rain, snow, wind, and sun, must be used for breaking up lumps. Cinders, sand, chips, or other contaminating material must be avoided. This additional crushing of large lumps may be done with hammers or mauls. The 200 to 500 pounds portion can be crushed in stages to about one-fourth of an inch size, and the material coned and quartered or riffled to about 100 pounds.

3. Materials in barrels, bags or similar containers must be sampled by removing about 5 pounds from below the surface of every 10th container, with a shovel, trier, or pipe, making sure that the 5-pound sample is representative of the entire contents of the container. If this method is not practicable, every 10th container must be dumped on a clean, hard surface, and by means of shoveling, coning, and quartering, reduced to approximately 5 to 10 pounds in weight. The gross sample thus consists of a combination of these portions. It may be necessary to reduce the size of the particles as outlined above. If this routine cannot be carried out for sampling, representative pieces must be sent to the laboratory as a sample. If a belt conveyor is transporting solids, the belt can be stopped every hour, all of the material between two idlers taken, and the belt then swept clean. If equipment exists for cutting the stream as it passes over the end of the belt, samples of the material, in the amount of at least 2 pounds for each ton, must be taken at regular intervals. Whenever possible, bulk shipments must be sampled while being unloaded from or loaded into the carrying vehicle because samples taken in this manner are generally more representative than those taken from loaded material in a car or truck.

4. Moisture sample. As most ores and concentrates are sold on the equivalent dry-weight basis, it is necessary to determine their moisture content. Because it would be impractical to dry the whole shipment of material before weighing, a sample must be taken as close to the time of weighing as possible. There must be a minimum amount of handling to prevent drying of the sample. If there is only a brief interval between

weighing and the loading or unloading of the conveyance, the sample may be taken during this time. Two or more holes must be dug into the material in the truck, rail car, or pile after the surface layer has been scrapped off. The sample thus obtained (approximately 100 to 200 pounds) must be a representative portion of the material, with emphasis on having the proper distribution of fine and coarse particles. These portions must then be placed in a tight container until the gross sample has been accumulated for the lot. If the sampling of a lot of material continues beyond a normal work shift, the material obtained during each work shift must be combined and mixed to form the gross sample. The gross sample for each moisture-lot will be mixed three times, coned and quartered or riffled down for a net sample of approximately 20 to 30 pounds, which must be placed inside a sealed or tied plastic bag that has been inserted in a tight container.

5. Ferroalloys:

    a. Ferrochromium, Ferromanganese and Silicomanganese. For lump alloy in bulk a representative gross sample from the lot shall be taken at the rate of approximately three pounds per ton. If possible the sample should be taken during loading or unloading operations. For containerized or crushed alloy, one container out of every 10 containers in the lot must be opened and dumped on a clean surface, then sampled at the rate of 1 pound for each 500 to 1,000 pounds in the lot. The samples shall be taken from numerous locations, which assure the obtaining of pieces comprising the gross sample from uniformly distributed points throughout the lot. From each of the large lumps (approximately 5 to 10 inches in size) in the samples selected there shall be broken one piece approximately 3/4 inch to 2 inches in size. These pieces shall be combined with representative amounts of small lumps (2 inches), and fines. It may be necessary to move many surface pieces to sample lumps underneath. After the entire lot has been sampled, the gross sample shall be crushed to 3/4 inch and down, mixed and divided. The gross sample shall be reduced to approximately 25% by splitting two times, then crushing to 1/4 inch and smaller followed by riffling and/or coning and quartering to approximately 2 to 4 lbs. Unused remaining portions of the gross sample shall return to the lot.

    b. Ferrovanadium, ferromolybdenum, and ferrotungsten. These alloys are all high-priced materials. Therefore, it is important that the sample be thoroughly representative, regardless of the amount of material involved. Ten percent of the containers in the lot must be dumped on a clean surface. A shovel scoop can be used to take the portions from the dumped material; and sample, representing 1 to 2 pound for each 1,000 pound in the lots, must be taken. After the entire lot has been sampled, the gross sample shall be crushed, mixed, and divided as stated in (a) above.

c. Laboratory sample: When chemical analyses are to be performed, the 2 to 4 pound sample shall then be alternatively pulverized in a mortar, or suitably designed crusher, and sieved until it all passes a 100-mesh sieve. The minus 100-mesh sample shall be mixed, coned and quartered or divided in a suitable manner into the required number

of equal portions. If the approximately 2 to 4 pound sample cannot be pulverized to minus 100-mesh, the sample shall be mixed, coned and quartered or divided in a suitable manner into the required number of equal portions. Unused remaining portions of the final sample shall not be returned to the lot but disposed of in the proper manner.

SAMPLING METHOD NO. 4:

This method of securing samples for laboratory analysis cover metals, solders, and other similar materials received in the form of ingots, pigs, slabs, rondelles, bars, castings, and scrap.

a. Apparatus. Apparatus will consist of power drill presses using drills of varying diameter, usually five-sixteenths of an inch. And power metals saws or miller; and a Jones or more modern approved sample riffles that divide a sample into two parts by one passing of the material, each part representative of the original material.

b. Gross sample:

1. The gross sample is best taken during plant production at the time of final forming or casting, and simultaneously with manufacturer's sampling. If this is not possible, then sampling must be done either by sawing, drilling, or milling a representative group of castings or sample specimens, and represent the average cross section of the commodity.

2. In the sampling of castings or sample specimens, select samples at random from the lot. Considering three ingots as a rectangular unit, drill three holes entirely through the unit, one at the center and one at each end on a diagonal of the rectangle, starting from the bottom. Use no lubricant on the drill. Remove any oil or grease on the sample with either. Start the drill on the surface to remove all oxide and clean the surface before starting to take the sample. Control the drill speed to prevent overheating and oxidation of the chips. Collect the drillings for the sample. Discard all drillings carrying oxide from the "set" or burned by the drill. Keep drillings in an airtight bottle.

3. Pigs and bars of antimony, bismuth, and cadmium may be sampled by selecting at random three pigs or bars from each lot. The gross sample will be obtained by sawing through the sample in sufficient places to obtain representative sawdust for the required laboratory samples. No lubricants may be used for sawing. Saw cuts must be made approximately five-eighths of an inch deep on samples one and one-fourth inches or more wide, and spaced so that metal from the entire sample is adequately represented. Saw cuts approximately five-eighths of an inch deep must be made on a sample one and one-fourth inches wide by eight and one-half inches long as follows: one longitudinal cut on each and approximately five-eighths of an inch from the edge on a bar one and one-fourth inches wide. Transverse cuts must be spaced with marks on one side at two and one-fourth inches, four and one-fourth inches, six and one-fourth inches one end. While the opposite side must be spaced with marks at one and one-fourth inches, three and one-fourth inches, five and one-fourth inches, and seven and one-fourth inches in

order that the transverse sawing will not segregate the sample bar into more than one piece. Keep sawings in an airtight bottle.

4. For certain metals, gross samples must be obtained by the following special procedures:

    a. Nickel - From 5 percent of the electrolytic cathodes of a lot up to a maximum lot size of 50 LT, using template prescribed by ASTM - B39.

    b. Tin - From 10 percent of the pigs of a melt or lot up to a maximum lot size of 50 LT.

    c. Precious metals - Sampling of precious metals must be done in a clean, isolated place, free of dust, grease, or any other contaminating substances. Each lot must be sampled by cutting out a chip on each of a bar, ingot, or plate of the lot, or by sawing three strikes across each of the four corners of a bar or ingot of the lot. Approximately 3 grams must be taken of each lot of material sampled. In sawing, the depth of the cut and speed of cutting must be regulated to prevent excessive heating or oxidation of the sample. Lubricants must not be used in sampling precious metals. In the case of sampling sponge, granules, or powder, the material must be poured from the container and mixed thoroughly. After mixing, the material must be sampled by cutting the flow stream as it is transferred from one container to another. The sample must be thoroughly mixed and divided into a laboratory sample, an umpire sample, and a reserve sample and distributed as required. After the sample has been taken, the lot must be re-weighed and marked with the new weight.

    d. Cobalt in the form of granules or rondelles must be sampled by selecting representative portions from 25 percent of the containers in each lot in the same manner as the gross sample. The sample is prepared from the gross by collection in bags or buckets. The accumulated samples from 25 percent of containers in a 100-unit lot will amount to approximately 15 pounds. The total or gross sample taken from one lot must be thoroughly mixed and then riffled down through an approved riffler to an amount that will provide the required number of samples.

c. Laboratory sample: Gross sample of drillings, milling and sawings must be reduced by approved methods for the laboratory sample. The receiving laboratory must sift all sample drillings submitted on a screen with 250 meshes per square centimeter to remove material ground between the drill and sides of the hole, and extract with a strong magnet any iron which may come from the drill.

SAMPLING METHOD NO. 8:

This method (known as a "Grab Sample") covers sampling in a random manner of a very limited nature to give an indication of the quality of the material.

a. Grab Sample. A grab sample is one taken in a random manner of a very limited nature to give an indication of the quality of the material. A grab sample is not necessarily representative of the material sampled, depending on the homogeneity of the material. Although it is a very limited sample it must be reasonably representative of that portion of the material sampled. Judgment on the part of the sampler is necessary in the taking of the grab sample because the difference in the character of the material sampled will require different techniques. For lumpy material, care must be taken to secure the proper ratio between the larger pieces and fines. In taking a grab sample of material in a pile, the surface must be cleared away to a depth of about 6 inches to avoid surface-altered material and contamination due to pile coverings or windblown foreign matter. Generally, about three such samples would be taken about one-third of the way up from the toe of the pile and be distributed along the length of the pile. For grab samples of materials in containers, several inches of the surface must be avoided. In grab sampling of finer materials, such as granules or powders, a trier must be used. If a grab sample is to be taken during a continuous loading or unloading operation, the sample may be taken on a less frequent basis than would be necessary for a fully representative sample. The sample of such material may be at one or two intervals, or from one of a number of trucks or rail cars. If a larger quantity of gross sample is taken, it must be reduced by the cone and quarter method or by riffling down to a net sample of 20 to 30 pounds. There are occasions when only a few pounds need to be taken, particularly of packaged material; in this case the whole sample may be submitted.

b. Laboratory Sample. When chemical analyses are to be performed, the sample shall be prepared in accordance with one of the previously listed laboratory sample preparation methods. Use the method that is closest to the type of material sampled.

SALES CONTRACT SAMPLING

SAMPLES FOR PROSPECTIVE BUYERS:

When proper authorization is given usually in the sales solicitation, samples may be either provided by the Specialist or by allowing prospective buyers to draw samples under the direct supervision of the Specialist.

a. The solicitation will usually state the quantity of samples that may be taken by the prospective purchaser

SALES CONTRACT SAMPLING:

Sales contract sampling is defined as sampling permitted under a Sales contract by an independent sampler/analyst for the purchaser. The Government is not the requester of the sampling or analysis. A business letter document is to be used to transmit any samples taken by the purchasers' sampler. A copy should be given to the sampler and an attached copy included with the DNSC Form 32.

Sampling in conjunction with weighing:

On material that has been sold the sampling procedures will normally be stated in the sales or sampler/analyst contract and shall be followed. Normally, commercial sampling procedures based on the standards of the American Society for Testing and Materials ***(ASTM***) is adhered. On sales, the purchaser will usually be required to obtain an independent sampler/ analyst that has been approved by the government. The Specialist will be required to witness all sampling and sample preparation. When the procedures are not stated, the following procedures should be used:

a. The minimum quantity of representative sample portions to be taken should be one pound per ton of material. Sampling should be done immediately before or after weighing. In general, the samples are to be taken from the area where the loading units are working, but if impractical, proportionate amounts of samples may be taken from each truck or railroad car.

b. Samples representative of the material delivered are to be taken daily on a routine and consistent basis for moisture and chemical determinations. A composite analytical sample comprised of representative increments of material from deliveries each day will be made to coincide with each lot, but not more than a weekly interval. The gross analytical samples are to be crushed to appropriate particle size, thoroughly mixed and reduced in quantity by coning and quartering or by the use of sample splitters to arrive at the laboratory samples, including reserve or umpire portions.

c. All remaining unused gross samples should be returned to the original pile, if taken before weighing is performed. If samples are taken after weighing, rejects from gross sample are to be returned to carrier's conveyances after weighing.

CONSIDERATIONS FOR SELECTION OF SAMPLES:

Proper selection of samples is an integral part of any successful quality assurance program. Without proper sampling, there can be no confidence in the estimate of lot quality; therefore, the sample is of little or no value. Procedures for proper sampling are described in a through e, below:

a. Homogeneous sampling. Each inspection lot must, as far as practicable, consist of a homogeneous product. Grouping dissimilar products into one inspection lot must be avoided to reduce the likelihood of considerable quality variation in the lot and a wrong inspection decision.

b. Proportional sampling. A proportional number of sample units must be taken from each sublet to form the required sample size. Proportional sampling must also apply to the lots presented for inspection in several containers such as bins, racks, pallets, or boxes.

c. Impartial sampling. Samples must be drawn without regard to their quality. No deliberate efforts must be made to include good or bad units in the sample.

d. Representative sampling. Samples must be drawn from as many parts of the lot as practicable to provide maximum representation of lot quality.

e. Random sampling. Random sampling provides a proven method for obtaining representative samples. It allows each item in the lot an equal chance of being included in the sample. A simple instruction or haphazard arrangement cannot accomplish effective randomization; it is a deliberate accomplishment and must be planned. Although other methods for obtaining representative samples may be satisfactory, tables of random numbers, a computer generated random numbers system, or the "hat method" is recommended for use, where practicable.

Responsibility for selection of sample:

The Specialist is responsible for determining which units in the lot must be included in the sample. If possible, the Specialist must personally withdraw samples from a lot. If such withdrawal is too laborious, withdrawal of samples by the contractor under the immediate supervision of the Specialist must be arranged.

Functions of a sampling plan:

A sampling plan is a device for deciding whether a lot must be accepted or rejected in regard to given characteristics. Basically, a sampling plan contains two things:

a. How many units to inspect from a lot, i.e., what the sample size must be; and

b. How good or bad the quality of a sample must be in order to accept or reject the lot, i.e., what the acceptance and rejection number must be.

Sample size:

The sample size must be as indicated in the applicable specification or purchase document.

ENCLOSURE 3

DEFINITIONS

SAMPLING: IMPORTANCE AND DEFINITIONS

PURPOSE:

To explain the importance of sampling, define basic sampling terminology, and state the role of the Specialist in the selection and control of samples.

GENERAL:

This part describes the importance of the sampling function; defines terms applicable to sampling; and outlines steps to be taken in the collection of samples to determine the value and quality of materials offered for inspection.

IMPORTANCE OF SAMPLING:

1. Sampling is usually the first and most important step taken in the actual quality assurance inspection of material. Inspection, analysis, and testing, regardless of accuracy, give the composition and quality of only the sample itself. The most accurate analysis is of little value if the sample taken is not representative of the lot inspected. Regardless of the accuracy of the analysis of the final sample or the care with which the examination or appraisal is made, inaccurate or careless sampling may lead to improper classification or evaluation, to improper acceptance or rejection of material, and often to litigation. The work of the sampler is just as important as that of the analyst or examiner.

2. The degree which a sample may be representative of a total shipment or lot depends not only on the sampling method used, but even more on the care exercised by the sampler. The sampler's knowledge, experience, judgment and ability are of greater value because instructions cannot cover every point or combination of circumstances encountered on each inspection.

DEFINITIONS:

a. SAMPLING. Sampling is the process of securing a representative portion of materials for the purpose of gaining information regarding the composition of the whole by investigation of the part. The correct sampling of a lot of material is the process of obtaining from it a smaller quantity containing unchanged percentages of all constituents of the lot sampled. The object of sampling is to obtain this small representative portion for inspection, test, or analysis to determine the type, quality, or composition, and therefore the acceptability and unit value of the total lot inspected.

b. LOT. A discrete quantity of material that contains a single batch or several batches or is the product of a continuous process broken into units on the basis of time or shipment. Individual batches in a lot must be specifically identified so that they may become individual or stratified units for inspection.

c. INCREMENTS. Portions of material selected from various parts of a lot that is treated individually or combined and tested as a unit.

d. GROSS SAMPLE. A gross sample is the total quantity or composite of material withdrawn by means of various types of sampling equipment such as mechanical or hand-tool samplers, from the material tendered for inspection, using appropriate sampling methods and techniques. It is essential that the gross sample be thoroughly mixed before reduction in order to obtain a representative sub sample or laboratory samples. Whenever available, various types of mechanical apparatus are preferable to obtain uniform particle size and reduction of the gross sample volume.

e. SUBSAMPLE. A smaller size sample produced in a specified manner by the reduction in volume and mass of a large quantity of gross sample. The sub sample is usually divided equally into the required number of portions, which are not only representative of the gross sample but of the lot. These portions comprise the contractor, government, reserve, and umpire laboratory samples.

f. LABORATORY SAMPLE. The sub sample or a gross sample (usually 25 lbs. or less) submitted to a laboratory for testing the physical properties and/or electrical characteristics and for the analysis of the constituents (elements or compounds) contained in the lot. (In an infrequent case where no sample reduction equipment is available, the subsample is sent to the laboratory for reduction to a laboratory sample and other required types of sample - reserve, umpire, etc.).

g. CONING AND QUARTERING. Coning and quartering is the method of reducing a gross sample to manageable size while still obtaining a representative sample. After thorough mixing, the material is formed into a cone that is then leveled into a flat circular heap. The heap is then divided diametrically into four equal quarters. Two opposite quarters are discarded and the remaining two opposite quarters are retained and formed into a second cone. The process is repeated until the four equal quarters contain the desired amount of sub sample.

h. RIFFLE. A device consisting of a series of chutes that are directed alternatively to opposite sides with the width of the chutes being varied according to the largest particle size. Equal portions of material passing through the chutes are deposited into rectangular pans.

i. RIFFLING OR SPLITTING. A method of reducing the gross (composite) sample material by mechanical means to the required amount of sub sample. Material is passed through the riffle and deposited into the separate pans. One pan containing material is randomly selected and the material is discarded. This process is continued until the required amount of sub sample is attained. Volume reduction is rapid for dry material of suitable fineness. Using the riffle prior to splitting blends the gross sample. The riffle may be used to divide the sub sample into the required number of laboratory samples.

j. SCREEN TEST. A method for determining the physical size of material sampled. No crushing or reduction of particle size is permitted under any circumstances. All material sampled is to be passed over a square (aperture) screen with the proper size openings. The sample material is to be weighed prior to the screening operation. After the screening procedure, the material that passed through the screen is to be weighed and the percentage that passed will be calculated for each test. Results are to be reported in percentage by weight to the nearest second decimal. The screen test weighing will be accomplished on an approved scale.

k. CRUSHING. A method of reducing the particle size of the gross sample to a uniform particle size before coning and quartering or riffling and splitting. A strongly built jaw, gyrator crusher, or roll mill capable of crushing lumps of material larger than ½-inch in size to ½-inch and down is used. The crushing surfaces with which the sample comes into physical contact shall be made of a hard abrasion-resistant steel or other suitable alloy.

TABLES

TABLE OF NATIONAL DEFENSE STOCKPILE COMMODITIES AND SAMPLING METHODS

GENERAL:

This part prescribes sampling methods explained in Part 3 that are generally applicable for the various commodities inspected in the National Defense Stockpile. Different conditions will make it impossible to follow exactly the methods prescribed in these procedures; however, they will serve as guides to the Specialists and samplers in the many conditions where they apply and provide the basis for developing variations whenever necessary.

TABLES OF COMMODITIES AND SAMPLING METHODS

Method of Sampling	Commodity	Minimum Amount of Laboratory Sample	Acceptable Containers
1 or 3	Aluminum Oxide	4 oz	A-B-C-D-E
4	Antimony	4 oz	A-B-D-E
3	Bauxite	8 oz	A-B-C-D-E
3	Beryl	16 oz	A-B-C-D-E
4	Cadmium	10 oz	A-B-D-E
3	Celestite	8 oz	A-B-C-D-E
3	Chromite	4 oz	A-B-C-D-E
3	Cobalt	4 oz	A-B-D-E
3	Ferroalloys	8 oz	A-B-C-D-E
3	Fluorspar	8 oz	A-B-C-D-E
3	Graphite		
	Crucible Grade	2 lbs	A-B-C-D-E
	Lubricant Grade	1 lb	A-B-C-D-E
	Amorphous Lump	8 oz	A-B-C-D-E
2	Iodine	4 oz	A
4	Lead		
	Corroding	16 oz	A-B-D-E
	Others	4 oz	A-B-D-E
3	Manganese Ore	4 oz	A-B-C-D-E
5	Mercury	8 oz	A-B
4	Precious metals	1 gr	A-B
2	Quebracho	8 oz	A-B-C-D-E-F
2	Quinidine	½ oz	A
3	Tantalite	4 oz	A-B-C-D-E
4	Tin	8 oz	A-B-D-E
3	Titanium	8 oz	A-B-C
3 or 4	Tungsten	4 oz	A-B-C-D-E
4	Zinc	6 oz	A-B-C-D-E
	Special high grade	32 oz	A-B-D-E

	Other grades	16 oz	A-B-D-E
3	Zirconium ores	4 oz	E-F

EXPLANATION OF TABLE

a. First column. This column refers to the suitable sampling procedure for each listed commodity, which is determined primarily by the physical character.

b. Second column. This column is an alphabetical list of the commodities involved.

c. Third column. This column designates the minimum weight of each of the three sample portions finally prepared for laboratory analysis. In cases where the sampler cannot prepare the laboratory sample from the gross sample, consult the applicable sampling method for the amount to be submitted to the laboratory. Special instructions concerning the size of the sample may also be given from time to time.

d. Fourth column.

1. This column indicates the types of acceptable containers into which the prepared laboratory samples must be placed for shipment and storage. Where only one type of container is permissible, this type has been specified. The symbol in column 4 indicates the following containers:

    a. Glass.
    b. Plastic containers
    c. Can with a tight cover
    d. Cardboard cylinder or carton
    e. Manila envelope
    f. Cloth bag

2. When containers D, E, and F are used, care must be taken that they are well sealed to prevent loss of portions of the sample. These containers are not suitable for samples for moisture determination. Airtight containers must be used where moisture is to be determined.

3. Glass containers for samples must be heavy duty, wide mouth jars or bottles of appropriate size, fitted with screw caps of plastic or synthetic resin and chemically resistant liners. Because of the susceptibility to corrosion, metal caps must be avoided. Square jars are preferable to round. These containers must be properly packed to prevent breakage during shipment and marked "Glass - Handle with Care". Glass containers used for such commodities as drugs or iodine must be opaque or dark to prevent deterioration by light.