CHAPTER 7

SAMPLING STOCKPILE MATERIALS

PART 1

INTRODUCTION

7-1 PURPOSE.

To provide instructions and procedures for sampling stockpile materials.

7-2 SCOPE.

This section is applicable to all types of sampling performed for the disposal of stockpile materials.

7-3 GENERAL.

This section 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 in this section are intended to standardize, insofar as possible, the sampling to be performed by DNSC Specialists. Also, more detailed contract provisions or instructions may supersede contractors performing this type of service for the Defense National Stockpile Center.

PART 2

SAMPLING: IMPORTANCE AND DEFINITIONS

7-4 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.

7-5 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.

7-6 IMPORTANCE OF SAMPLING.

a. 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.

b. 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.

7-7 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 sub sample 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.

7-8 SELECTION AND CONTROL SAMPLES.

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.

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.

7-9 PHYSICAL TESTS AND CHEMICAL ANALYSES.

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

PART 3

BASIC SAMPLING PROCEDURES

7-10 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.

7-11 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.

7-12 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 par. 7-15. 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.

7-13 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.

7-14 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.

7-15 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.

7-16 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 DNSC 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. See below for instructions for preparing the Record of Samples Transmitted and Request for Analyses.

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.

7-17 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 outloading. These documents as well as the certificates of moisture shall be forwarded to:

Directorate of Stockpile Operations and Logistics
Defense National Stockpile Center
8725 John J. Kingman Road
Suite 3229
Fort Belvoir, VA 22060-6223
FAX: 703/767-7608

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 outloaded 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 outloading, 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 outloading will be recorded as dry weight for payment purposes.

DNSC Form 34 Sample Identification Label
Instructions for Preparing DNSC Form 34 Sample Identification Label

INSTRUCTIONS FOR PREPARATION. The DNSC Specialist must furnish, in the spaces provided, information required by the following titles on the Sample Identification Label.

1. Commodity. Enter the commodity name.

2. Contract P.O. Enter sales contract number or the purchase specification number whichever is applicable.

3. Location of Material. Enter the location that the material was sampled.

4. Lot Number. Enter the lot number that the sample was drawn from.

5. Quantity Sampled. Enter the total weight/quantity of the lot sampled.

6. Sample Number. Enter the number assigned to the sample.

7. Date Sampled. Enter the date the sample was sent to the laboratory or taken by the sampler.

8. Signature of Specialist. The Specialist that took the sample or witnessed the sampling signs in this block.

DNSC Form 35 Record of Samples Transmitted and Request for Analyses
Instructions for Preparing DNSC Form 35 Record of Samples Transmitted and Request for Analyses

INSTRUCTIONS FOR PREPARATION. The DNSC Specialist must furnish, in the spaces provided, information required by the following titles on the Record of Samples Transmitted and Request for Analyses.

Item 1. Enter date samples are transmitted.

Item 2 Enter applicable purchase or sale contract number.

Item 3. Enter name of the vessel, if the item is imported.

Item 4. Enter symbol for the applicable program.

Item 5. Enter a brief description of the material, including grade and type applicable.

Item 7. Enter applicable symbol of the region requesting the analysis.

Item 8. Enter name and address of analyst to whom the sample is to be sent.

Item 9. Enter name and address of the material supplier or purchaser.

Item 10. Enter analyst's sample number (if known).

Item 11. Enter Government's sample number.

Item 12. Enter lot number and marks.

Item 13. Enter number and type of units.

Item 14. Enter quantity or weight of each lot or batch sampled. In cases where there is no lot or batch, enter the quantity of the shipment, identifying it plainly as such.

Item 15. Check applicable box to indicate method of shipment.

Item 16. Check applicable box to indicate tests described.

Item 17. Check applicable box to indicate the nature of the document under which the tests will be performed. Enter service contractor's contract, etc., number.

Item 18. Check applicable box.

Item 19. Check applicable box or boxes or leave blank.

Item 20. Enter date the sample was taken (the starting and final dates, if more than one).

Item 21. Enter location where the material was sampled.

Item 22. Indicate disposition to be made of the remainder of the sample(s) after analysis.

Item 23. List the government sample number and net weight of the sample and any other pertinent comments concerning the sample.

Item 24. Check the appropriate box for the distribution of analysis certificates.

Item 25. The Specialist signs in this block.

Item 26. Enter when analysis certificate is due to DNSC Operations (this information is available in the service contract).

Item 27. When the contract sampler takes the sample, the sampler must sign and date in these blocks. If the sample is sent to the analyst, then request in Block 23 that the analyst sign one copy and send that copy to DNSC Operations.