WASTE ANALYSIS PLAN

AlliedSignal Engines
550 Main Street
Stratford, Connecticut 06497

September 1996

TABLE OF CONTENTS

Section	Title	Page No.
1.0	Waste Analysis Plan Purpose	1-1
2.0	Facility Description	2-1
3.0	Parameters Description of Hazardous Waste and Associated Analytical	3-1
4.0	Waste Analytical Methods	4-1
5.0	Waste Sampling Methods	5-1
6.0	Waste Sampling Frequency	6-1
7.0	Record Keeping and Plan Update	7-1

APPENDIX A - SAMPLING PROCEDURES PROTOCOLS A-1

LIST OF TABLES

Figure No.	Title	Page No.
3-1	Waste Parameters and Selection Rationale	3-2
4-1	Waste Parameters and Analytical Methods	4-2
5-1	Waste Sampling Methods	5-2

SECTION 1.0

WASTE ANALYSIS PLAN PURPOSE

The purpose of this Waste Analysis Plan (WAP) is to fulfill the requirements of 40 CFR 265.13(b), "Interim Status Standards for Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities, General Waste Analysis". These regulations require that all owners or operators of hazardous waste facilities with interim status develop and follow a written waste analysis plan which describes the procedures to be used to provide a detailed chemical and physical analysis of a representative sample of various wastes generated at the facility. This WAP must remain in operation until the facility's post-closure requirements are fulfilled. To fulfill the requirements of 40 CFR 265.13(b), this WAP was prepared using the United States Environmental Protection Agency's (EPA) "Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Waste, A Guidance Manual", OSWER 9938.4-03, April, 1994.

To fulfill the requirements of 40 CFR 265.13, this WAP contains the following elements:

1. A description of the purpose of and need for the WAP (Section 1.0).

2. A description of the facility the WAP has been developed for, including processes occurring on-site and the locations of hazardous waste storage (Section 2.0).

3. A description of the hazardous wastes generated at the facility, a list of analytical parameters that apply to each of the wastes, and the rationale for the selection of these parameters (Section 3.0).

4. A description of the EPA approved analytical methods used to test for the parameters present in the hazardous wastes (Section 4.0).

5. A description of the sampling methods used to obtain a representative sample of hazardous waste stored in various containers (Section 5.0 and Appendix A).

6. A description of the frequency of waste sampling (Section 6.0).

7. A description of the record keeping used for the waste analytical results and requirements for the update of the WAP (Section 7.0).

SECTION 2.0

FACILITY DESCRIPTION

This WAP was developed for the Stratford Army Engine Plant located at 550 Main Street in Stratford, Connecticut. The Stratford Army Engine Plant is owned by the U.S. Army and operated by AlliedSignal Engines, a unit of AlliedSignal, Inc. The 75 acre facility is bordered by the Housatonic River to the east, Main Street, various commercial properties, and Sikorsky Memorial Airport to the west, residences to the north, and a tidal marsh marine basin, residences and Long Island Sound to the south.

Stratford Army Engine Plant manufactures and assembles turbine engines for tank, watercraft, and aircraft applications at the Stratford Army Engine Plant. The production process for these engines include: metal working (machining) and treating operations; plating of engine parts with chrome, copper and nickel (including cleaning baths and rinse baths); engine assembly; engine testing; and engine overhauling activities.

All the wastes at the Stratford Army Engine Plant facility are produced on-site as a result of the operations described above. Stratford Army Engine Plant does not accept waste from other manufacturers, nor does Stratford Army Engine Plant treat hazardous Resource Conservation and Recovery Act (RCRA) waste or store hazardous waste for longer than 90 days. The Stratford Army Engine Plant facility does not operate as a Treatment, Storage, or Disposal Facility (TSDF). While the Stratford Army Engine Plant facility has interim TSDF status, in practice, the Stratford Army Engine Plant facility is operated solely as a large quantity generator of hazardous RCRA waste.

Currently, hazardous wastes are stored at three locations within the facility. These locations (and the wastes typically stored at these locations) include:

- Bulk waste oil is stored in a 10,000 gallon above ground storage tank within a tank farm located at the northeast corner of the facility, south of Building 58 and east of Building 15. Bulk waste oil contaminated with solvents is stored in a 10,000 gallon above ground storage tank also located in this tank farm.

- Waste metal hydroxide sludge (produced by the facility's waste water treatment plant) is stored in a covered thirty cubic yard roll-off located on the north side of Building 18 at the southern end of the facility.

- Remaining wastes (i.e. those wastes stored in containers 55 gallons or less in size) are stored at the hazardous waste storage pad located adjacent to the north side of the bulk waste oil and contaminated waste oil tank farm.

Various other wastes (including various non-hazardous wastes and various plating wastes) are produced at the facility. These wastes are treated on-site either at the facility's waste water treatment plant, at the facility's Oil Abatement Treatment System, or at one of two on-site ultra-filtration systems. Wastes treated at these locations are discharged either to the Housatonic River under National Pollution Discharge Elimination System (NPDES) permit number CT002984 or to the municipal sanitary sewer. These non-RCRA wastes are handled under other permits and thus, **ARE NOT** included under this WAP.

In addition to these storage areas, previous operators of the facility operated four surface impoundment's on-site. These impoundment's were used for the disposal of metal hydroxide sludge produced by the facility's waste water treatment plant. These impoundment's were remediated and closed by the former operator in 1991. Currently, these impoundment's are subject to the RCRA Part B Post-Closure Permit Application (Part B Application) prepared for Textron Lycoming (previous operator of the facility) by Wehran EnviroTech, located in Glastonbury, Connecticut. This Part B Post-Closure Application was submitted to the EPA Region I and the Connecticut Department of Environmental Protection (CT DEP) on December 13, 1991. These impoundment's **ARE NOT** included under this WAP.

SECTION 3.0

DESCRIPTION OF HAZARDOUS WASTE AND ASSOCIATED ANALYTICAL PARAMETERS

As required by 40 CFR 265.13(b)(1), Table 3-1 presents the wastes typically generated at the Stratford Army Engine Plant facility. In addition to the name(s) of the wastes, Table 3-1 also includes the parameters these wastes will be analyzed for, and the rationale for the selection of these parameters. For all wastes, the parameters described in Table 3-1 were chosen to:

- identify wastes that may compromise or corrode container integrity;
- identify proper storage conditions; identify potential reactivity and relevant health and safety precautions for waste handling;
- identify constituent(s) for permit compliance; and
- allow waste removal/disposal companies to handle and/or treat wastes.

Wastes may be analyzed for some or all of the parameters listed for the waste in Table 3-1. Analytical parameters given in Table 3-1 are based upon waste type, waste origin, and available information concerning the waste. The wastes listed in Table 3-1 are those wastes which currently, are typically produced at the Stratford Army Engine Plant Facility (i.e. all those wastes produced since January, 1995). Occasionally, wastes not included in this table may be produced on-site. Wastes not included in Table 3-1 will be sampled, analyzed, and included in this WAP under the provisions described in Sections 4, 5, 6 and 7 of this WAP. These new wastes will be added to Table 3-1 as appropriate.

In addition to collecting and analyzing samples of wastes to determine waste characterization, certain wastes will be characterized using other information including existing published or documented data (for example, Material Safety Data Sheets) or previous sampling results in the facility's records. This type of analysis, rather than a physical analysis of a sample of the material, is allowed under 40 CFR 265.13(a)(2). Examples of wastes which may be characterized based upon available information include:

- Discontinued raw product which is being treated as a waste for disposal rather than being used in the manufacturing processes of the facility.
- Metal Hydroxide sludge generated by the treatment plant and stored in the 30 cubic yard roll off.
- Waste oil and waste oil contaminated with solvents stored in the bulk storage tanks.

See Section 5.0 "Waste Sampling Methods" for more information.

Table 3-1

Waste Analysis Plan
Waste Parameters and Rationale for Parameter Selection

Waste Name(s)	Process Generating Waste	Analytical Parameter(s)	EPA (or State) Waste Code	Parameter Selection Rational
Waste Speedi-Dry	Small Spill and Leak Clean-up	TPH, VOCs, TCLP Metals	D001	Generally, this is a non-hazardous state regulated waste. However, this may be either a regulated or a listed waste depending upon the concentrations of contaminants. Based upon the material and origin, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
Fuel oil & Filters (RQ)	Test Cell Operations	TPH, VOCs	D001	State Regulated only. Based upon the material, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
Concrete (with NaOH and Chromic Acid) (RQ)	Excavation and Construction	pH, Hexavalent Chromium, Total Chromium	D007	This may or may not be a listed waste depending upon the concentrations of contaminants. Based upon the origin of the concrete there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
Waste Aerosol Cans	Plant Wide	VOCs, Flammability, Reactivity	D001	This may or may not be a listed or characteristic waste depending upon the concentrations of contaminants. Based upon the material, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
1,1,1-Trichloroethane Waste Solids	Degreasing Operations	1,1,1-Trichoroethane	F001	These are listed wastes. Based upon the origin of the waste, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
1,1,1-Trichloroethane/ Waste Oil/ Water	Plant Wide (from machine maintenance)	1,1,1-Trichoroethane	F001	These are listed wastes. Based upon the origin of the waste, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
Waste TPC Solvent	Welding Operations	VOCs, Flammability	D001	This is a listed waste. Based upon the original material and origin of the waste, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Acetone and Rags	Plant Wide Degreasing Operations	VOCs, Flammability	D001	This is a listed waste. Based upon the original material and origin of the waste, there is no reason to believe that this waste will contain any toxic constituents (other than VOCs) in significant concentrations.
Jet Fuel/Filters (RQ)	Test Cell Operations	VOCs, Flammability	D001	This is a listed waste. Based upon the original material, its origin in the facility and other information, there is no reason to believe that this waste will contain other toxic constituents (other than jet fuel components) in significant concentrations.
Metal Hydroxide Sludge	Waste Water Treatment Operations	TCLP Metals	F006	By definition, this is a wastestream from a non-specific source (F006 wastewater treatment sludge).
Ammoniacal Strip	Plating Operations	TCLP Metals, VOCs, Cyanide	N/A	This is usually not a RCRA waste, however, it could potentially be contaminated with sufficient metals, VOCs or cyanide to constitute a hazardous waste. Based upon the waste's origin, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Dyna Flo Compound	Test Cell Operations	VOCs, Metals, Flammability	D001	State Regulated only as an oily waste. Contaminated batches may be RCRA wastes. Based upon the material, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.

Table 3-1 (Continued)

Waste Analysis Plan
Waste Parameters and Rationale for Parameter Selection

Waste Name(s)	Process Generating Waste	Analytical Parameter(s)	EPA (or State) Waste Code	Parameter Selection Rational
TPC (Hydraulic oil)	Welding Operations	VOCs, Flammability	D001	Identified by available information. This is a state regulated oily waste. Based upon the material, there is no reason to believe these wastes will contain any other toxic constituents in significant concentrations.
Aviation Fuels	Test Cell Operations	VOCs (benzene), Flammability,	D001, D018	Identified by available information. This is a listed toxic waste. Based upon the material and its origin, there is no reason to believe that these wastes will contain any other toxic constituents in significant concentrations.
Nital Etch (etching solutions)	Gear Line Etching	Metals, Flammability, Reactivity	D001, D002	This is a listed waste. Based upon material this is known to be flammable and corrosive. May be contaminated with metals from the plating process. Based upon the origin, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Cooling Tower Corrosion Inhibitor	Plant Wide Cooling	Metals, pesticides	CR04, MA99	Established waste stream, identified by available information. This is a state regulated waste. Based upon the material, there is no reason to believe that these wastes will contain any other toxic constituents in significant concentrations.
Cooling Tower Anti-Slime Solution	Plant Wide Cooling	Metals, pesticides	CR04, MA99	Established waste stream, identified by available information. This is a state regulated waste. Based upon the material, there is no reason to believe that these wastes will contain any other toxic constituents in significant concentrations.
Wood Floor Block	Final Assembly Area (floor repairs)	VOCs, TPH, TCLP Metals	CR05, MA01	State regulated oily waste. Based upon the material and its origin, there is no reason to believe that these wastes will contain any other toxic constituents in significant concentrations.
Ni/Cd Batteries	Plant Wide	TCLP Metals (Ni, Cd)	D006	A listed waste. Established waste stream, identified by available information. Based upon the material, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Lead Acid Batteries	Garage (Cart Maintenance)	N/A	N/A	Established waste stream, identified by available information. These materials are recycled and do not require testing.
Mercury 3000sc	Plating Operations	pH	CR04, MA99	Established waste stream. State regulated material. Identifiable by existing information. Based upon the material and its origin, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Rachem 822	Boiler Operations	pH	CR04, MA99	Established waste stream. This is a state regulated waste (pH above 2.5 and below 12). Based upon the material and its origin, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Waste Paint and Paint Debris (rags, etc).	Paint Shop Operations	VOCs, SVOCs, TCLP Metals	F003, F005, D001, D035	A listed or characteristic waste. Existing information indicates that this may contain more than 5.0 mg/l Cr or Pb. Based upon the material, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Coolant	Plant Wide	N/A	D001	Established waste stream, this is a non-regulated waste as determined by existing information (unless it is suspected that this material is contaminated by solvents). Based upon the material and its origin, coolant does not usually require testing.

Table 3-1 (Continued)

Waste Analysis Plan
Waste Parameters and Rationale for Parameter Selection

Waste Name(s)	Process Generating Waste	Analytical Parameter(s)	EPA (or State) Waste Code	Parameter Selection Rational
Sodium Hydroxide, Sodium Metasilicate, Potassium Hydroxide, Potassium Carbonate, Trisodium Phosphate, Potassium fluoride, Sodium Bisulfate, Sodium Fluoride (Corrosive/Poisonous Solids	Plating Operations	pH	D002, CR05, MA99	Established waste stream. State regulated waste (pH above 2.5 and below 12). Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Plating Wax	Plating Operations	TCLP Metals, VOCs, Cyanide	CR05, MA99	Established waste stream. State regulated waste. Based upon origin and material, occasionally, this material may contain metals causing the material to be listed as a RCRA waste. There is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Freon Contaminated Oil	Plant Wide	VOCs (trichlorofluouroethane)	F002	A listed toxic waste. Based upon origin of material, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Nickel Sulfamate	Plating Operations	pH, metals (Ni)	CR05, MA99	Established waste stream. State regulated waste (pH above 2.5 and below 12). Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Diphenylmethane-4,4' diisocyanate (instapak or instaflex)	Final Assembly	N/A	CR05, MA99	Established waste stream. State regulated waste (foam filler). Based upon origin and material testing is not required for disposal as it can be determined by existing information. Also, based upon the material and its origin, there is no reason to believe that this waste will contain any other toxic constituents in significant concentrations.
Hydroquinone, Sodium Hydroxide	Plating Operations	pH	CR04, MA99	Established waste stream. State regulated waste (pH above 2.5 and below 12). Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Microbiocide	Boiler Operations	pH, pesticides	D002	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Chromic Acid Solution and/or solids	Plating Operations	pH, metals (chromium)	D002, D007	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Hydrochloric Acid, Nickel Chloride, Ammonium Persulfate, Formaldehyde Solution (these are various Enplate products).	Plating Operations	pH, metals (Nickel)	D002, CR04, MA99	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent (other than Nickel)in significant concentrations.

Table 3-1 (Continued)

Waste Analysis Plan
Waste Parameters and Rationale for Parameter Selection

Waste Name(s)	Process Generating Waste	Analytical Parameter(s)	EPA (or State) Waste Code	Parameter Selection Rational
Waste dichloride methane (methylene chloride)	Laboratory Operations	VOCs	F002	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Silicon Fluid	Foundry Operations	N/A	CR04, MA99	Established waste stream. Based upon existing information, origin and material no testing is necessary.
Ethylenediamane, Ethanolamine	Laboratory Operations	Flammability, pH	D001, D002	Established waste stream. Flammable/Corrosive liquid. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Dichloromethane, Isopropanol	Laboratory Operations	VOCs, Flammability	D028, F002	Established waste stream. Flammable liquid and listed waste. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Methyl Cyanide	Plating Operations	Flammability, Reactivity, Cyanide	D001	Established waste stream. Flammable liquid (may have cyanide). Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste gasoline	Plant Wide	N/A	D001, D018	Established waste stream. Identifiable by existing information. Based upon material and origin, this material does not need testing.
Antifoam BB	Waste Water Treatment Plant Operations	N/A	CR04, MA99	Established waste stream. State regulated. Identifiable by existing information. Based upon material and origin, this material does not need testing.
Waste Corrosive Liquids (Phosphoric acid, Gluconic Acid)	Plating Operations	pH	D002	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Caustic Liquids (Sodium Hydroxide)	Plating Operations	pH	D002	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Potassium Permanganate	Plating Operations	pH, Reactivity, Flammability	D001	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Methanol	Plant Wide Cleaning	Flammability	D001, U154	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Waste Barium Compounds (Quicklite A)	Laboratory Operations	TCLP metals (Ba)	D005	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Electrostatic solution	Print Shop Operations	N/A	CR04, MA99	Established waste stream. State regulated material. Based upon material, and origin testing of this material is not required.
Electrostatic black ink	Print Shop Operations	N/A	CR02, MA01	Established waste stream. State regulated oily waste. Based upon material and origin, testing of this material is not required.

Table 3-1 (Continued)

Waste Analysis Plan
Waste Parameters and Rationale for Parameter Selection

Waste Name(s)	Process Generating Waste	Analytical Parameter(s)	EPA (or State) Waste Code	Parameter Selection Rational
Tetrachloroethylene (blankrola solvent)	Print Shop Operations	VOCs	D001	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Petroleum distillates	Plane Wide	Metals	D001	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in
(Copier toner, spray booth liquid, Royco 103)	Plant Wide Office/ Printing	Metals	D001	Significant concentrations (metals analysis will be based upon material origin).
Nickel chloride	Plating Operations	Metals (Ni), pH	CR04, MA99	Established waste stream. State regulated waste. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.
Metal Grindings (Metal Dust)	Plant Wide Machining Operations	TCLP Metals	D006, etc.	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations (i.e. no contaminants other than metals)
Boiler Sludge	Boiler Operations	TCLP Metals	D006, etc	Established waste stream. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations (i.e. no contaminants other than metals)
2-butoxyenthanol, 2-ethanol	Plant Wide Cleaning	Flammability	CR04, MA99	Established waste stream. State regulated waste. Combustible liquid. Based upon origin and material, there is no reason to believe that this waste will contain any other toxic constituent in significant concentrations.

TPH = Total Petroleum Hydrocarbons
VOCs = Volatile Organic Compounds (F001 = 1,1,1-trichloroethane; F003 = Xylene; and F005 = Toluene)
TCLP = Toxicity Characteristics Leachate Procedure
RQ = Reportable Quantity
TPC = trade name
RCRA = Resource Conservation and Recovery Act
pH = Corrosively
flammability = flash point
Ni = Nickel
Cd = Cadmium
Pb = Lead
Cr = Chromium
Cu = Copper

SECTION 4.0

WASTE ANALYTICAL METHODS

As required by 40 CFR 265.13(b)(2), Table 4-1 presents the EPA approved analytical methods for the parameters listed in Table 3-1. Table 4-1 also includes the appropriate reference(s) for each analytical method.

Currently, the majority of wastes generated at the Stratford Army Engine Plant facility will be analyzed by Milford Materials Testing Laboratory (MMTL) in Milford, Connecticut. MMTL is a State of Connecticut certified laboratory. References for test methods described in Table 4-1 are available at the laboratory. As necessary, other state certified laboratories will be used to perform additional or specialized analysis.

Stratford Army Engine Plant will perform sample preservation, storage (if necessary), and appropriate quality assurance/quality control (QA/QC) sampling (including trip blanks, equipment blanks, duplicates, matrix spike, and matrix spike duplicates) under the direction of the analytical laboratory. All samples will be sent to the laboratory under a chain of custody (COC). All laboratory QA/QC presented under “Test methods for Evaluation of Solid Waste. Physical/Chemical Methods”, U.S. EPA SW-846 will be performed by the analytical laboratory.

As described in Section 3.0, Table 4-1 includes parameters for wastes typically produced at the Stratford Army Engine Plant facility. Parameters to be tested for additional or new wastes (wastes not currently included in this WAP) will be determined by the methods discussed in Section 3.0. Wastes of unknown origin or unknown composition will be analyzed for all parameters in the full suite of RCRA tests (i.e. flammability, Corrosivity, Volatile Organic Compounds (VOCs) using EPA Method 8010/8020, Metals using the Toxicity Characteristic Leachate Procedure (TCLP)etc) prior to disposal.

Table 4-1

Waste Analysis Plan
Parameters and Analytical Methods

Parameter	Test Method	Reference
VOCs (solvents such as 1,1,1-Trichloroethane, Acetone, Tetrachloroethylene, and Methylene Chloride	GC/FID (EPA Methods 8010/8020)	Test Methods for the Evaluation of Solid Waste. Physical/Chemical Methods. U.S. EPA SW-846.
TCLP	TCLP Procedure	40 CFR 261 Appendix II
Metals (Cu, Cd, Ni, Pb, Cr)	Atomic Absorption	Methods for chemical analysis of water and wastes. U.S. EPA 600/4-79-020, March 1979.
pH (Corrosivity)	Electromagnetic	Test Methods for the Evaluation of Solid Waste. Physical/Chemical Methods. U.S. EPA SW-846.
Flammability (Flash Point)	Pensky-Martins Closed Cap or Open Cap Tester	ASTM Standard D-93-79 or D-83-90.
Reactivity	Test Method is Material Dependent	Test Methods for the Evaluation of Solid Waste. Physical/Chemical Methods. U.S. EPA SW-846.
Total Petroleum Hydrocarbons	Gravimetric (EPA Method 418.1)	Test Methods for the Evaluation of Solid Waste. Physical/Chemical Methods. U.S. EPA SW-846.
Cyanide	Spectorphotometric	Test Methods for the Evaluation of Solid Waste. Physical/Chemical Methods. U.S. EPA SW-846.

U.S. EPA = United States Environmental Protection Agency
ASTM = American Society of Testing Materials
Ni = Nickel
Cd = Cadmium
Pb = Lead
Cr = Chromium
Cu = Copper

SECTION 5.0

WASTE SAMPLING METHODS

The waste containers used at the Stratford Army Engine Plant facility which contain the various waste streams described in Table 3-1, will be sampled using either the Authoritative Sampling Approach or the Random Sampling Approach, depending upon the waste type. The Authoritative Sampling Approach includes the use of past sampling results, process and waste origin knowledge, and printed or published materials such as Material Safety Data Sheets (MSDS) from the manufacturer, or information from trade or industry publications. This approach will be used when good quality information concerning the waste is available and when process and generation information indicates that the material is not contaminated or does not contain unknown constituents.

For random sampling, a Simple Random Approach including the collection of grab samples from individual containers will be used. However, occasionally, where appropriate, either a Stratified Random Sampling Approach or a Systematic Sampling Approach will be applied depending upon the origin of the container(s). This approach will be used when available information is not considered reliable, when the waste is unknown, or when the waste is suspected to be contaminated.

As required by 40 CFR 265.13(b)(3), Table 5-1 presents the methods used to obtain a representative sample of waste from a specific container to be analyzed. Table 5-1 is organized according to the waste storage container or the type of material to be sampled. All the wastes listed in Table 3-1 are within one of the waste type categories presented in Table 5-1. The appropriate category (and sampling method) should be determined at the time of sampling with the use Table 5-1 and Appendix I (see attached). Table 5-1 includes the waste type, sampling method, description of sampling and a reference for the sampling technique. Appendix I includes copies of the references for the sampling technique. To reduce the possibility of cross-contamination, where ever possible, disposable sampling equipment will be utilized. If it is not possible to use disposable, single use, sampling equipment, the equipment used will be appropriately decontaminated during each sampling event.

Wastes generated at the Stratford Army Engine Plant facility will be stored either in the 30 cubic yard roll-off (waste metal hydroxide sludge); one of two 10,000 gallon above ground storage tanks (waste oil and contaminated waste oil); or in smaller containers (55-gallon drums, lab packs, or smaller containers for the remainder of the waste). It is unlikely that the types of waste storage containers will change; however, should the containers change, appropriate sample techniques will be determined and included in Table 5-1 as described in section 7.0, “Record Keeping and Plan Update”.

Table 5-1

Waste Analysis Plan
Methods for Sampling Hazardous Wastes

Waste Type	Sampling Method	Description of Sampling	Reference for Sampling Method[1]
Waste Solvents and Oils (drums and 10,000 gallon tanks)	Sampling a container (Coliwasa)	Composite sample using a Coliwasa sampler from three grab samples at the top, middle, and bottom of the container or drum.	Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, U.S. EPA SW-846.
Metal Grindings, Boiler Sludge (drums)	ASTM Standard D-140-70, Crushed or Powdered Material	Representative composite samples at top, middle, and bottom of drums using Trier Scoop	Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, U.S. EPA SW-846.
Metal Hydroxide Sludge (30 cy roll-off) and concrete wastes or boiler sludge (drums)	ASTM Standard D-346-75 soil or rock-like material	Composite sample using a Trier Scoop from six grab samples through three different points near the top, of the container to points diagonally opposite the point of entry or representative composite samples at top, middle, and bottom of drums using Trier Scoop	Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, U.S. EPA SW-846.
Paint Skimmings and Paint Wastes (drums)	Sampling a container (Coliwasa)	Composite sample using a Coliwasa sampler from three grab samples at the top, middle, and bottom of the drum.	Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, U.S. EPA SW-846.
Plating Solutions (drums)	Sampling a container (Coliwasa)	Composite sample using a Coliwasa sampler from three grab samples at the top, middle, and bottom of the drum.	Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, U.S. EPA SW-846.

U.S. EPA = United States Environmental Protection Agency
ASTM = American Society for Testing Materials
[1] = See Appendix I for Reference Materials

SECTION 6.0

WASTE SAMPLING FREQUENCY

At a minimum, wastes produced at the Stratford Army Engine Plant facility will be sampled on an annual basis using either Authoritative or Random Sampling approaches as described in "Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Waste, A Guidance Manual", OSWER 9938.4-03, April, 1994 and discussed in Section 5.0 of this WAP. During this annual sampling, wastes will be analyzed for all the parameters associated with the waste as listed in Table 3-1. Therefore, certain wastes (i.e. waste gasoline, lead-acid batteries, etc.) will not require laboratory analysis because the material is sufficiently identifiable using existing source information or because the material is recycled.

When the source or composition of the waste is in question or unknown ,or if contaminants or additional constituents are suspected, sampling and analysis will occur more frequently on an as needed basis to determine all of the waste's constituents. When this occurs, the suspect wastes may be analyzed for some or all of the parameters listed for the waste in Table 3-1. Analytical parameters for more frequent sampling to determine waste constituents will be based upon, Table 3-1, waste type, waste origin, and available information concerning the waste.

For both annual sampling and additional sampling, sample analytical parameters, sample analysis, and sampling methods will be performed according to Sections 3.0, 4.0, and 5.0 of this WAP, respectively.

SECTION 7.0

RECORD KEEPING AND PLAN UPDATE

Original copies of sample analytical results will be filed chronologically and kept within the Health, Safety and Environmental Department of the Stratford Army Engine Plant. At a minimum, the sample results will include: Sample collection date (including sample Chain-Of-Custody), parameters to be analyzed, and the results of the analysis. In addition, sample results will be "matched up" (either by "hard copy" or by electronic format) and kept with the appropriate manifest when the wastes are shipped off-site for disposal.

At a minimum, this WAP will be reviewed, and if necessary, up-dated, on an annual basis. In addition, the WAP will be reviewed and updated when:

- manufacturing processes are added or changed;
- significant new wastes are generated or produced;
- wastes with parameters not included in Tables 3.1 or 4.1 are produced;
- waste storage container types are changed or added; and/or
- the primary analytical laboratory changes.

APPENDIX I

REFERENCES FOR SAMPLING METHODOLOGY

Designation: D 140 – 93

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St Philadelphia, Pa 19103
Reprinted from the Annual Book of ASTM Standards Copyright ASTM
If not listed in the current combined index, will appear in the next edition

Standard Practice for Sampling Bituminous Materials[1]

This standard is issued under the fixed designation D 140, 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.

This Practice has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope*

1.1 This practice applies to the sampling of bituminous materials at points of manufacture, storage, or delivery.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI units in parentheses are provided for information only.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Document

2.1 *ASTM Standard*

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis[2]

3. Significance and Use

3.1 Sampling is as important as testing, and precautions shall be taken to obtain samples to show the true nature and condition of the materials.

3.2 Samples may be taken for either of the following two purposes:

3.2.1 To represent as nearly as possible an average of the bulk of the materials sampled, or

3.2.2 To ascertain the maximum variation in characteristics which the material possesses.

4. Selection of Samples

4.1 Whenever practicable, bituminous materials shall be sampled at the point of manufacture or storage, and at such time as to allow the tests controlling acceptance or rejection to be made in advance of shipment.

4.2 When the samples cannot be taken at the point of manufacture or storage, they shall be taken from the shipment immediately upon delivery.

5. Size of Samples

5.1 The sample size of liquid materials shall be as follows:

5.1.1 For routine laboratory examination, 1 qt (1 L) (emulsions, 1 gal (4 L)),

5.1.2 From bulk storage, 1 gal, or

5.1.3 From barrels or drums, 1 qt.

5.2 The sample size of semisold or solid materials shall be as follows:

5.2.1 From barrels, drums, or cakes, 2 to 3 lb (1 to 1.5 kg), or

5.2.2 From crushed or powdered material in bulk or bags, 2 to 3 lb.

6. Containers

6.1 *Type of Containers:*

6.1.1 Containers for liquid bituminous materials, except emulsions, shall be wide-mouth cans with lined screw caps or triple-seal friction-top cans.

6.1.2 Containers for emulsified bituminous materials shall be wide-mouth jars or bottles made of plastic, or wide-mouth plastic-lined cans with lined screw caps, or plastic-lined triple-seal friction-top cans.

6.1.3 Containers for crushed or powdered bituminous materials shall be triple-seal friction-top cans or plastic sacks placed in other containers suitable for handling.

6.2 *Size of Containers:*

6.2.1 The size of the container shall correspond to the required amount of sample.

7. Protection and Preservation of Samples

7.1 Sample containers shall be new. They shall not be washed or rinsed, or wiped with an oily cloth. If they contain evidence of solder flux, or if they are not clean and dry, they shall not be used. Top and container shall fit together tightly.

7.2 Care shall be taken to prevent the sample from becoming contaminated. Immediately after filling, the container shall be tightly and positively sealed.

7.3 The filled sample container shall not be submerged in solvent, nor shall it be wiped with a solvent saturated cloth. If cleaning is necessary use a clean dry cloth.

7.4 Samples of emulsions shall be protected from freezing by correct packaging.

NOTE 1—When sampling emulsions, care should be taken not to sample material under pressure as such resultant velocities tend to cause air entrapment which could result in erroneous test results. Any visual bubbling of the material is one indication air has been entrapped. In addition, the sampling container should be completely filled to minimize a skin formation at the air-emulsion interface.

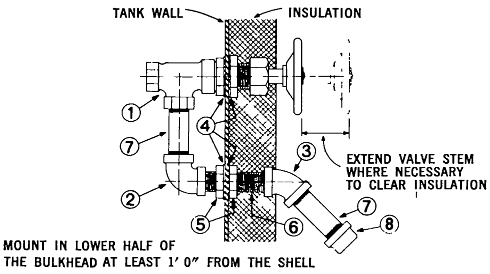
*** A Summary of Changes section appears at the end of practice.**

---

[1] This practice is under the jurisdiction of ASTM Committee D-4 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.30 on Methods of Sampling.

Current edition approved May 15, 1993. Published July 1993. Originally published as D 140 – 22 T. Last previous edition D 140 – 88.

[2] *Annual Book of ASTM Standards,* Vol 05.05.



REF No	DESCRIPTION	No REQ
1	¾″ 'VOGT' P 9844 STEEL ANGLE VALVE OR SIMILAR, PANEL MOUNTED	1
2	¾″ STEEL OR MALL IRON 90° ELBOW	1
3	¾″ STEEL OR MALL IRON 45° ELBOW	1
4	ASBESTOS GASKETS SNUG ON THREAD OR WOUND WITH YARN	4
5	¾″ 150# SCREWED M I LOCKNUT	2
6	¾″ x 3½″ ± PARALLEL THREADED STEEL PIPE NIPPLE (CUT FROM ¾″ STD TANK NIPPLE IF OTHERWISE UNOBTAINABLE)	1
7	¾″ x 3″ THREADED STEEL PIPE NIPPLE	2
8	¾″ MALL IRON PIPE CAP	1

FIG. 1 Typical Submerged Sampling Device

7.5 Samples shall not be transferred from one container to another except where required by the sampling procedure.

7.6 Immediately after filling, sealing, and cleaning, the sampling containers shall be properly marked for identification with a suitable marking pencil on the container itself, not on the lid. Linen tags also may be used for identification if they can be securely fastened to the container in such a manner as to ensure that they will not be lost in transit. Linen tags shall not be attached to containers by using the lids to secure them.

8. Sampling at Place of Manufacture

8.1 *Vertical Tanks Not Capable of Being Agitated (Liquid Materials or Materials Made Liquid by Heating)* (5001 bbl (800 m^3) or more capacity)—Three sampling methods are recommended (Note 2):

8.1.1 *Sampling-Valve Method*—Valves shall be located, with easy and safe access provided, on the side of the tank with the first in the top third of the tank, but no higher than 3 ft (1 m) from the top; the second in the middle third of the tank; and the third in the bottom third of the tank, but no lower than 3½ ft (1.1 m) from the bottom. A recommended design of the sampling valve is shown in Fig. 1.

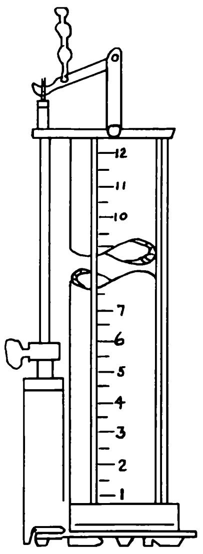
NOTE 2—During sampling by any of the methods described all filling inlets and discharge outlets to the tank shall be closed.

8.1.1.1 A minimum of 1 gal (4 L) of the product shall be drawn from each sample valve and discarded before taking the sample for test, and

8.1.1.2 1 qt (1-L) to 1 gal shall be drawn from each sample valve for test.

8.1.2 *Thief Sampler Method* (not suitable for asphalt cements)—Samples shall be taken in the top, middle, and lower sections at levels indicated in 8.1.1, of the tank by lowering a thief sampler into the material. A satisfactory type with instructions for use is shown in Fig. 2.

NOTE 3—Most bituminous materials in the liquid state are of such viscous and adhesive character that, after normal emptying of a container by pouring, the container retains enough material to cause significant contamination of any second samples secured before the container was thoroughly cleaned. The aforementioned properties of bituminous materials also make container cleaning a difficult, tedious, and time-consuming task.



NOTE—This type sampler is lowered into the tank with bottom valve open (there is no top closure) When the desired depth is reached, the lowering chain is given a snap tug which closes the bottom valve The sampler is then withdrawn from the tank and the contents transferred to the sample container This sampler may be used for repetitive sampling in the same tank

FIG. 2 Thief Sampler

NOTE 4—The thief sampler described in Fig. 2 may be used for repetitive sampling because significant sample contamination by previous contents is avoided by the self-cleaning action provided by the passage of material through the tube-shaped thief sampler which is open at both ends. The practice of raising and lowering the sampler 3 or 4 times through a distance of several feet at the sampling depth is recommended.

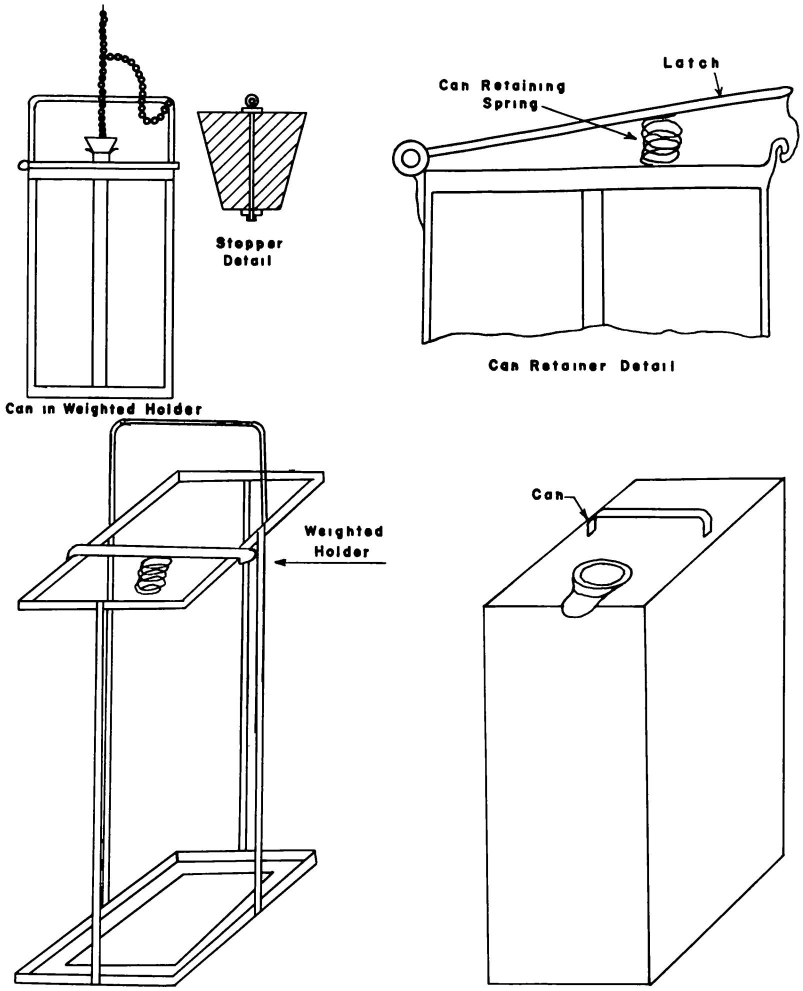
8.1.3 *Throw-Away Container Method*—Samples shall be taken at top, middle, and lower levels by lowering into the material a container in a suitable weighted holding device. The essential feature of the method is to use a clean container to take the sample, pour this sample into another clean container, and throw away the container used to take the sample from the tank. A satisfactory type with instructions on how to use the device is shown in Fig. 3.

8.1.4 The three samples from bulk storage tanks may be tested separately for consistency to detect stratification. They may be combined, thoroughly mixed, and a 1-qt to 1-gal (1 to 4-L) sample may be taken for other tests that may be required for determining average characteristics of the material.

8.2 *Bulk Storage Tanks (Liquid Materials or Materials Made Liquid by Heating) Equipped with Mechanical Agitators*—When the tank is equipped with operating mechanical agitators which, by observation through the sampling or inspection hatch, are performing adequate mixing of the tank contents, a single sample taken by any of the methods described in 8.1.1, 8.1.2, or 8.1.3 will be satisfactory to use for test purposes.

9. Sampling from Tank Cars, Vehicle Tanks, Distributor Trucks or Recirculating Storage Tanks

9.1 The sample may be taken from the sample valve or



NOTE—This type sampler is lowered into the tank with the stopper in place When the desired depth is reached, the stopper is removed by means of the attached wire, cord, or chain and the container allowed to fill Complete filling is indicated by the cessation of bubbles of air from the can at the surface of the liquid The sampler is then withdrawn from the tank and the contents poured into the clean sampler container A clean can (bottle) must be used for each sample taken

FIG. 3 Throw-Away Container Sampler

tap if the tanks are provided with them. When such sampling devices are required, they are to be built into the tank itself. A sampling device of this type is shown in Fig. 1. Before the sample is taken, 1 gal (1 L) shall be drawn from the sample valve and discarded.

9.2 Samples of liquid materials and materials made liquid by heating may be taken by the dip method using a clean wide-mouth or friction-top can in a suitable holder as shown in Fig. 4. A clean container must be used to take each sample, and the material sampled shall then be transferred to another new and clean container for retention or testing sample.

9.3 A detachable fitting, similar in design to the one shown in Fig. 5 may be inserted in the discharge line. Before the sample is taken 1 gal (1 L) shall be drawn from this sampling valve and discarded.

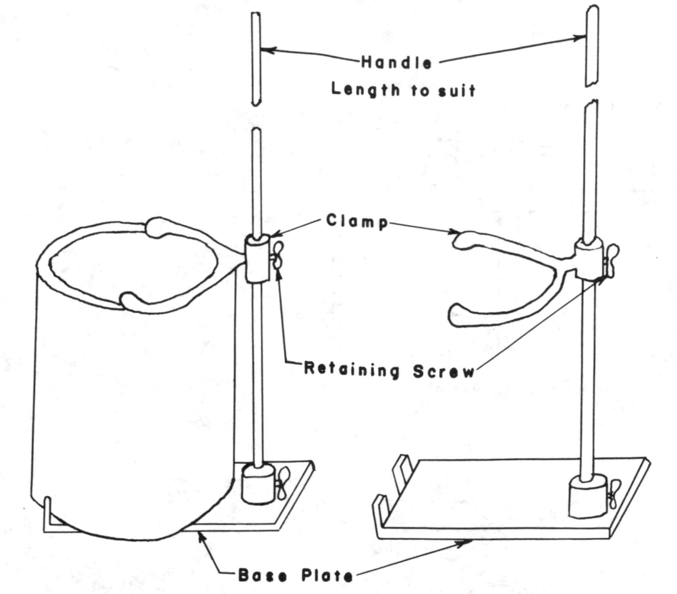
10. Sampling from Tankers and Barges

10.1 For liquid materials (including all cut-backs made liquid by heating) top, middle, and lower samples shall be taken using the methods as described in 8.1.2 and 8.1.3.

10.2 For all other materials made liquid by heating, a top sample only shall be taken by the dip, thief, or throw-away container method.

11. Sampling from Pipe Lines During Loading or Unloading

11.1 When tankers and barges are being loaded or unloaded samples may conveniently be taken from the pipe line through which the material is flowing by insertion of a sampling pipe into a rising section of the pipe line on the discharge side of the pump or in a completely filled line in which gravity creates the materials flow. A suitable in-line sampling pipe is shown in Fig. 5. The sampling pipe shall be not more than one eighth the diameter of the pipe line and its opening should be turned to face the flow of the liquid. This pipe shall be provided with a valve or plug cock and shall discharge into a sample receiver. At least three 1-gal (1-L) samples shall be taken at uniform intervals throughout the entire loading or unloading. At the completion of the loading or unloading, the individual 1-gal samples shall be combined, thoroughly mixed without altering its characteristics, and a 1-gal sample taken therefrom.



NOTE—Sampler with can secured in place is rapidly lowered into tank to desired depth and allowed to fill with sample. The sampler is then withdrawn from the tank and the contents transferred to the sample container. A clean container *must* be used to take each sample.

FIG. 4 Dip Sampler

11.2 When sampling tankers and barges 25 000 bbl (4000 m^3), or less, capacity, at least five 1-gal samples shall be taken at uniform intervals throughout the entire loading or unloading (25 000 bbl, or larger, capacity requires at least ten 1-gal samples). At completion of the loading or unloading the individual 1-gal samples shall be combined, thoroughly mixed in a manner designed to not alter its characteristics, and a 1-gal sample taken therefrom.

12. Sampling from Drums or Barrels

12.1 After thorough mixing, samples of liquid bituminous material from barrels or drums shall be secured by tube or thief sampling by taking 1 qt (1 L) of material from packages selected at random according to Section 13.

13. Sampling Semisolid or Uncrushed Solid Materials

13.1 *Drums, Barrels, Cartons, and Bags*—Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package shall be selected at random and sampled as described below. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single samples selected as described above fails on test to conform to the requirements of the specifications, a number of packages shall be selected at random equivalent to the cube root of the total number of packages in the lot. The following table is given, showing the number of samples to be selected for shipments of various sizes.

Packages in Shipment	Packages Selected
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1000	10
1001 to 1331	11

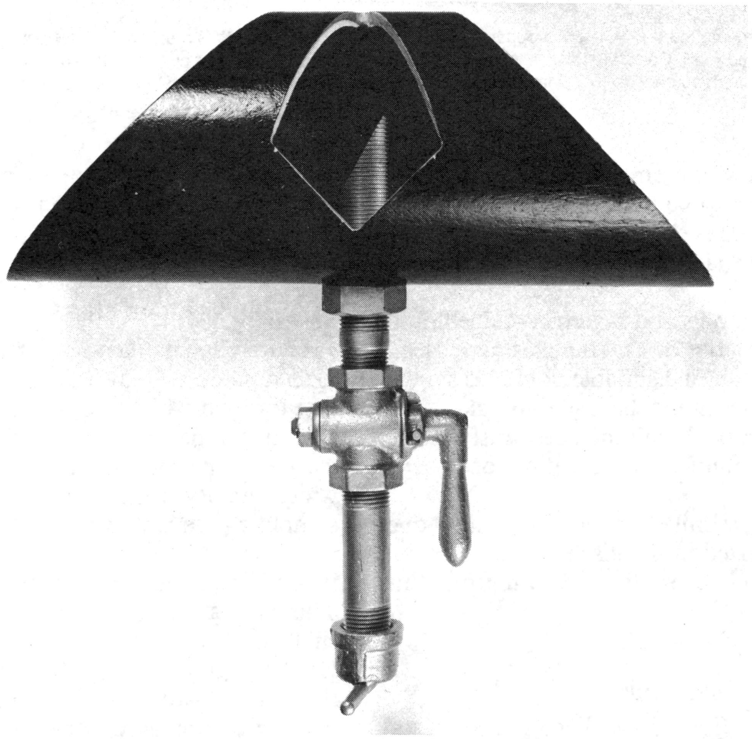


FIG. 5 In-Line Asphalt Sampling Device

Samples shall be taken from at least 3 in. (76 mm) below the surface and at least 3 in. from the side of the container. A clean hatchet may be used if the material is hard enough to shatter and a broad, stiff putty knife if the material is soft. When more than one package in a lot is sampled, each individual sample shall be not less than ¼ lb (0.1 kg) in weight. When the lot of material is obviously from a single run or batch of the producer, all samples from the lot shall be melted and thoroughly mixed, and an average 1-gal (1-L) sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and the batches can be clearly differentiated, a composite 1-gal sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches, each sample shall be examined separately.

14. Sampling Crushed or Powdered Materials

14.1 *Bulk Storage*—Solid bituminous materials in crushed ASTM fragments or powder shall be sampled in accordance with Method D 346. The gross sample shall be not less than 50 lb (25 kg) from which shall be selected the 2 to 3-lb (1 to 1.5-kg) sample for test.

14.2 *Drums, Barrels, Bags and Cartons*—Where the crushed or powdered material is in barrels, drums, or bags, a number of packages shall be selected at random as described in Section 13. A sample at least 1½ lb (0.7 kg) in weight shall be selected from near the center of each container to yield a gross sample weighing not less than 50 lb (22.7 kg) from which the 2 to 3-lb (1 to 1.5-kg) sample for test shall be selected as prescribed in Practice D 346.

15. Sampling at Point of Shipment Delivery

15.1 Sampling of bituminous materials shall be done as soon as practicable after the bituminous material has arrived at plant site, storage site, or job destination or at the time of unloading.

15.2 The required number of samples shall be taken from each delivery of bituminous material. In the case of small delivery units such as distributors, the samples shall be taken to represent a maximum of 10 000 gal (40 L).

15.3 Sampling shall be done by one of the three following methods:

15.3.1 In accordance with Section 8.

15.3.2 By bleeding through a sample valve or tap in the transfer line during the unloading of the approximate middle third of the load, or

15.3.3 By means of a sampling device inserted to a level of approximately the middle third of the load or tank.

15.4 Tests for material acceptability shall be made on one of the samples taken. The other samples are to be retained for confirmation in the event the first sample tested should fail to comply with requirements.

16. Precautions

16.1 Because of the numerous types and grades of bituminous materials that are alternately shipped and stored in the same or similar containers, the opportunity for contaminating these containers with residues, precipitates, or cleaning solvents is ever present. Numerous opportunities also exist for obtaining samples which are not strictly representative of the material or are contaminated after removal. Therefore it is incumbent upon the producer, transporter, user, and sampler to exercise continuous precaution in the sampling and handling of these materials.

17. Keywords

17.1 bituminous materials, crushed materials, liquid asphalts; powdered materials; sampling; semisolid materials; uncrushed solid materials

SUMMARY OF CHANGES

This section identifies the location of selected changes to this practice that have been incorporated since the last issue. For the convenience of the user, Committee D-4 has highlighted those changes that may impact the use of this practice. This section may also include descriptions of the changes or reasons for the changes, or both.

(1) Note 1 was added.
(2) Section 17 was added.

slots, thence, into the riffle pans, one half of the sample being collected in each pan. Under no circumstances shovel the sample into the riffle, or dribble into the riffle from a small-mouth container. Do not allow the crushed coke to build up in or above the riffle slots. If it does not flow freely through the slots, shake, or vibrate the riffle to facilitate even flow. The opening of the riffle slot should be 2.5 to 3 times the size of the largest particle.

10.2.3 After crushing the gross sample of not less than 14 kg (30 lb) (see Table 2) for 4.75-mm (No. 4) sieve size, using suitable pulverizing equipment, further reduce this subsample to 2.36-mm (No. 8) sieve size and divide by riffling to not less than the quantity specified in Table 3 for 2.36-mm (No. 8) sieve size.

10.2.4 With suitable pulverizing equipment, preferably a roll crusher or ball mill, reduce the 2.36-mm (No. 8) sieve size subsample to a 250-µm (No. 60) sieve size.

10.2.5 As an alternative to the procedure given in 10.2.4, the 2.36-mm (No. 8) sieve size subsample may be reduced to pass 95 % through a 850-µm (No. 20) sieve. Divide this subsample by riffling with the small riffle to not less than the quantity specified in Table 3, and then reduce to 250-µm (No. 60) sieve size as described in 10.2.4.

10.2.6 Thoroughly mix the laboratory analysis sample, weighing not less than 50 g, preferably, by mechanical means, before extracting portions for analysis.

10.3 *Procedure B, Hand Division:*

10.3.1 The method of dividing by hand the quantity of coke in a gross sample shall be carried out as described even though the initial size of coke and impurities may be less than indicated in Table 2.

10.3.2 Do the progressive crushing and division in the weight of the sample to the quantities indicated in Table 2 by the following method, which is described and illustrated in the accompanying Fig. 2, until the maximum size of the coke is 13 mm (½ in.), and the quantity is 27 kg (60 lb). Crush the gross sample to a maximum size of pieces of 50 mm (1 in.).

10.3.2.1 Repeat the alternate-shovel method of dividing the gross sample until the sample is divided to approximately 57 kg (125 lb) and take care to observe before each division in quantity that the sample has been crushed to the fineness specified in Table 2. Shovel the crushed coke into a conical pile (Fig. 2 (2)) by depositing each shovelful of coke on top of the preceding one, and then form into a long pile in the following manner: Take a shovelful of coke from the conical pile and spread it out in a straight pile (Fig. 2 (3A)) having a width equal to the width of the shovel and a length of 1.5 to 3 m (5 to 10 ft). Spread the next shovelful directly over the top of the first shovelful, but in the opposite direction, and so on back and forth, piling and, occasionally flattening the pile, until all the coke has been formed into one long pile, proceeding as follows: Beginning on one side of the pile, at either end, and shoveling from the bottom of the pile, take one shovelful (Fig. 2 (4), shovelful No. 1) and set it aside; advancing along the side of the pile a distance equal to the width of the shovel, take a second shovelful (shovelful No. 2) and discard it; again advancing in the same direction one shovel width, take a third shovelful (shovelful No. 3) and add it to the first. Take the fourth (shovelful No. 4) in a like manner and discard, retain the fifth (shovelful No. 5), and so on, advancing always in the same direction around the pile so that its size will be gradually divided in a uniform manner. When the pile is removed, about half of the original quantity of coke should be contained in the new pile formed by the alternate shovelsful that have been retained (Fig. 2 (5A) shows the retained half and (5B), the rejected half).

TABLE 3 Preparation of Sample for Chemical Analysis

Crush to Pass at Least 95 % Through Sieve	Divide to Minimum Weight, g
4.75 mm (No. 4)	2000
2.36 mm (No. 8)	500
850 µm (No. 20)	250
250 µm (No. 60) (100 % through)	50

10.3.3 After the gross sample has been divided by crushing and by the alternate-shovel method to approximately a 27-kg (60-lb) sample of 13-mm (½-in.) coke, proceed using the methods in 10.2.3.

10.4 The report of analysis should include a brief description of the method of taking the sample, by such characteristic expressions as *belt sample, top-of-car*, etc. Information should be given as to how the gross sample is crushed and divided in the description that accompanies the sample sent to the laboratory.

Designation: D 346 – 90

An American National Standard

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St. Philadelphia, Pa 19103
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

Standard Practice for
Collection and Preparation of Coke Samples for Laboratory Analysis[1]

This standard is issued under the fixed designation D 346; 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.

INTRODUCTION

Coke, especially run of oven coke or foundry coke, or both, is a difficult material to sample. It is imperative that every sample be collected and handled carefully and conscientiously and in strict accordance with the standard procedure described herein.

Gross samples of not less than the quantities designated in this practice must be taken, whether the coke to be sampled consists of a few tons or several hundred tons.

1. Scope

1.1 This practice covers procedures for the collection and reduction of samples of coke to be used for physical tests, chemical analyses, and the determination of total moisture.

1.2 The values given in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 167 Test Method for Specific Gravity and Porosity of Lump Coke[2]
D 293 Method of Sieve Analysis of Coke[2]
D 2013 Method of Preparing Coal Samples for Analysis[2]
D 2234 Test Methods for Collection of a Gross Sample of Coal[2]
D 3038 Method of Drop Shatter Test for Coke[2]
D 3302 Test Method for Total Moisture in Coal[2]
D 3402 Method of Tumbler Test for Coke[2]

3. Significance and Use

3.1 This practice may be used to provide a representative sample of the coke from which it is collected. Because of the variability of coke and the wide variety of sampling equipment, caution must be exercised in all stages of sampling, from system specifications and equipment procurement to equipment acceptance testing and actually taking the final sample.

3.2 This practice also provides an analysis sample of coke from the gross or divided sample. The analysis samples can be used to determine the value of the coke represented, its ability to meet specifications, as well as for other purposes.

4. Place of Sampling

4.1 Sample coke while it is being loaded into or unloaded from railroad cars, ships, barges, or trucks, or when discharged from supply bins, grab buckets, belt conveyers, or other coke conveying equipment. Sample close to the point of interest.

5. Collection of Gross Sample

5.1 Collect increments regularly, systematically, and with such frequency, so that the entire quantity of coke sampled will be represented proportionately in the gross sample, and a gross sample of the required amount will be collected. The standard gross sample shall equal or exceed the quantities given in Table 1.

5.1.1 The quantity of sample to be taken will depend on the size of the coke being sampled and the amount of information to be obtained from the sample.

5.1.1.1 The size of the sieve analysis sample is governed by the homogeneity of the coke being sampled. The quantities given in Table 1 represent the minimum quantity to be collected. The sieve analysis sample will supply sufficient coke for any subsequent physical tests and laboratory analysis. Total moisture of the coke should not be determined on the sieve analysis sample.

5.1.1.2 For the drop shatter test, a minimum of 75 kg (165 lb) of coke retained on 50-mm (2-in.) square mesh sieve if furnace coke is being processed, or 140 kg (300 lb) of coke retained on 75-mm (3-in.) square mesh sieve if foundry coke is being processed, is required. The quantities given in Table 1 represent the minimum amounts required of sized coke.

5.1.1.3 The tumbler test requires a minimum of 35 kg (75 lb) of coke that will pass a 75-mm (3-in.) square mesh sieve and can be retained on a 50-mm (2-in.) square mesh sieve, or that which will pass a 64-mm (2½-in.) square mesh sieve and can be retained on a 38-mm (1½-in.) square mesh sieve. The quantities given in Table 1 represent the minimum amount of the required sized coke.

5.1.1.4 The apparent specific gravity test required a min-

---

[1] This practice is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.23 on Sampling.

Current edition approved Sept. 28, 1990. Published January 1991. Originally published as D 346 – 32 T. Last previous edition D 346 – 78.

[2] *Annual Book of ASTM Standards*, Vol 05.05.

TABLE 1 Minimum Gross Sample Weights of Coke, kg (lb)

Usage	Foundry Coke		Furnace Coke		Coke Breeze[A]
	Run of Oven	Sized	Run of Oven	Sized	
Sieve analysis (size consist)	364 (800)	227 (500)	227 (500)	182 (400)	45 (100)
Drop shatter test	182 (400)	159 (350)	136 (300)	91 (200)	
Tumbler test	68 (150)	45 (100)	57 (125)	57 (125)	
Apparent specific gravity	46 (100)	46 (100)	46 (100)	46 (100)	23 (50)
Coke moisture	46 (100)	46 (100)	46 (100)	46 (100)	23 (50)
Chemical analysis	113 (250)	113 (250)	57 (125)	57 (125)	34 (75)

[A] Coke passing a ¾-in (19-mm) square hole sieve, or smaller top size

imum of 23 kg (50 lb) of representative coke pieces. The quantities given in Table 1 represent the minimum amounts of the required coke pieces.

5.1.1.5 The determination of total moisture of the coke requires approximately 45 kg (100 lb) of coke. The quantities given in Table 1 represent the minimum amount to be collected.

5.1.1.6 The quantities given for samples for chemical analysis represent the minimum amounts to be collected.

NOTE 1—Samples collected from the surface of coke in piles, bins, cars, ships, or barges are, in general, unreliable because of size segregation and should not be used for determining conformance to specifications unless the purchaser and the seller so agree If necessary to collect a sample of coke from the surface of a loaded railroad car, take nine equal increments about 305 mm (1 ft) below the surface Locate the nine points as shown in Fig 1 The diameter of the hole must be at least 3 times that of the largest piece in the shipment

6. Condition of Increment Collection

6.1 Four conditions of increment collection are recognized:

6.1.1 *Condition A (Stopped-Belt Cut),* in which a loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the coke stream. The distance between the parallel faces shall not be less than three times the length of the largest piece.

6.1.2 *Condition B (Full-Stream Cut),* in which a full cross section is removed from a moving stream of coke.

6.1.3 *Condition C (Part-Stream Cut),* in which a portion, not a full cross section, is removed from a moving stream of coke.

6.1.4 *Condition D (Stationary Sampling),* in which a portion of coke is collected from a pile, a railroad car, a barge, or a shiphold.

NOTE 2—See Section 6 of Test Methods D 2234

7. Size of Increments

7.1 To collect increments, use a shovel or specially designed tool or mechanical means for taking equal increments. When increments are collected from the surface of a loaded rail car or truck shipment, the gross sample shall consist of nine increments of approximately equal quantity. When increments are taken from piles, conveyor belts, barges, etc., the gross sample shall consist of not less than 25, nor more than 50 increments, of approximately equal quantity.

NOTE 3—When large-size coke, such as foundry coke, is being sampled, it is likely that quantities larger than those given in Table 1 will be collected. However, the minimum number of increments collected should not be decreased unless such is agreed upon between the purchaser and the seller

NOTE 4—If the sample for chemical analysis is obtained from the sieve analysis sample, a sample not smaller than that shown in Table 1 should be reconstituted from the size fractions obtained when the sieve analysis sample was processed

8. Quantities Represented

8.1 Take a gross sample for each 454 Mg (500 tons) or fraction thereof, or in case of larger tonnages, for such quantities as may be agreed upon. Sample separately each lot of coke arising from a different source or known to be of different quality or size.

9. Processing of the Test Sample

9.1 Process samples collected for sieve analysis of coke in accordance with the procedure given in Method D 293.

9.2 Process samples collected for the drop shatter test in accordance with the procedure given in Method D 3038.

9.3 Process samples collected for the tumbler test in accordance with the procedure given in Method D 3402.

9.4 Process samples collected for the determination of apparent specific gravity and porosity in accordance with the procedure given in Test Method D 167.

9.5 Process samples taken for the determination of total moisture in accordance with the procedure given in Test Method D 3302 and Method D 2013.

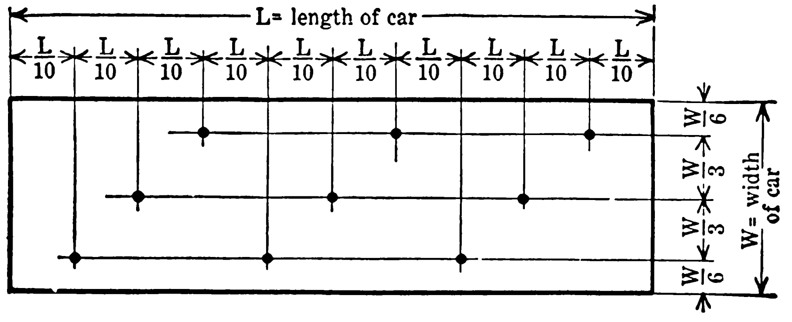


FIG. 1 Location of Sampling Points from Exposed Surface of Car

9.6 Prepare samples taken for chemical analysis as directed in Section 10.

10. Preparation of Coke Sample for Laboratory Analysis

10.1 Crush, mix, and divide the entire gross sample in quantity to convenient size for transmission to the chemical laboratory. Crush the sample, preferably by means of jaw or roll crushers, or on a chilled iron or hard steel plate by impact of a tamper, hard bar, or sledge, avoiding all rubbing actions as otherwise the ash yield can be materially increased by the addition of iron from the sampling apparatus. Do the crushing under such conditions as to prevent loss of coke or accidental mixture of foreign matter.

10.2 *Procedure A, Manual Riffling*·

10.2.1 Determine the number of passes required in the riffling operation from the total volume of the gross sample and the minimum permissible weight in accordance with Table 2.

TABLE 2 Weights of Coke Samples with Corresponding Crushing Sizes

Weight of Sample to Be Divided		Largest Size of Coke and Impurities Allowable in Sample Before Division	
kg	lb	mm	in
113 or over	250 or over	25	1
57	125	19	¾
27	60	13	½
14	30	5	4 mesh

10.2.2 Divide the crushed gross sample by using a large riffle. Riffles properly used will reduce sample variability but cannot eliminate it. Pass the crushed coke through the riffle from a feed scoop, feed bucket, or riffle pan having a lip or opening the full length of the riffle. When using any of the above containers to feed the riffle, spread the crushed coke evenly in the container, raise the container, and hold it with its front edge resting on top of the feed chute; then slowly tilt it so that the coke flows in a uniform stream through the hopper straight down over the center of the riffle into all the

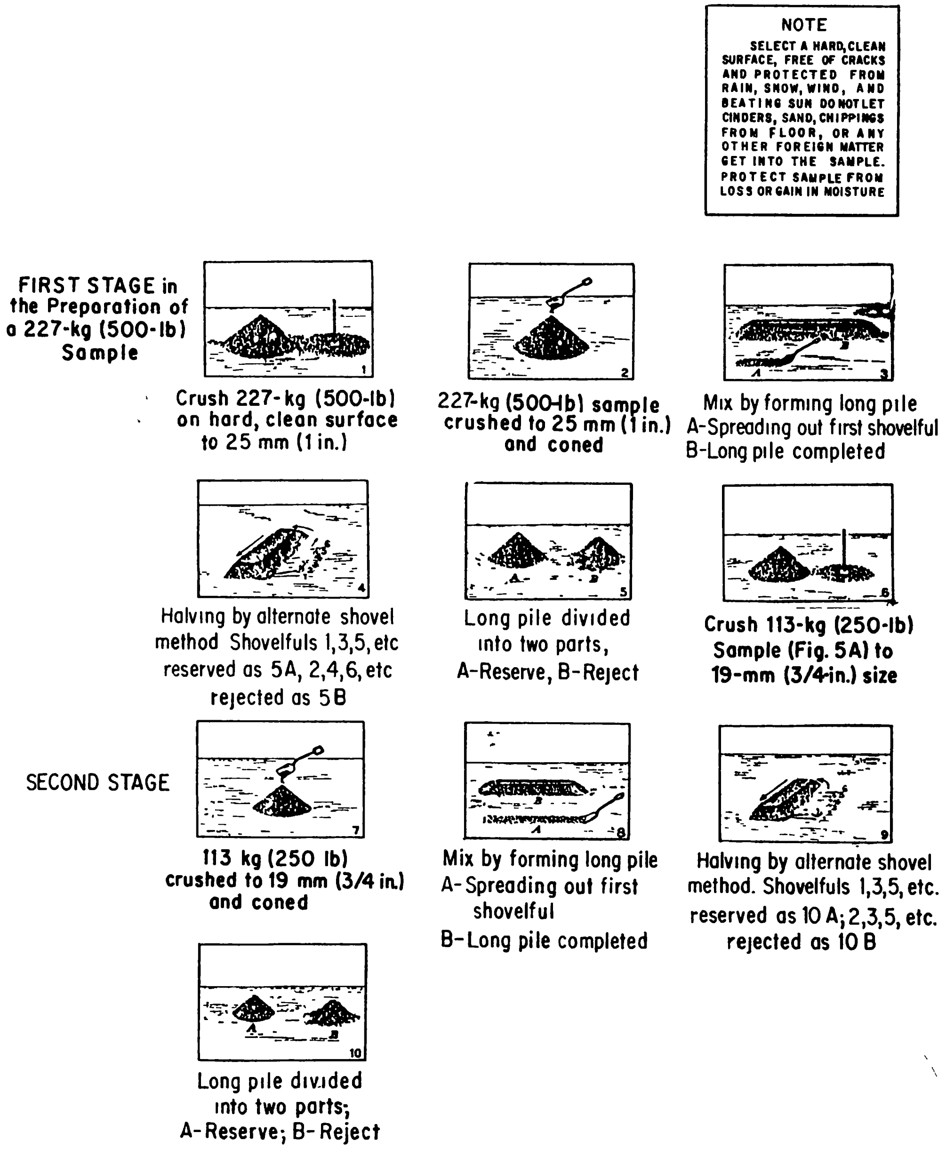


FIG. 2 Standard Hand Method of Crushing and Reducing the Gross Sample Quantity of Coke