4 May 1966

3668

TECP 700-700
Materiel Test Procedure 5-2-585
White Sands Missile Range

U. S. ARMY TEST AND EVALUATION COMMAND
COMMON ENGINEERING TEST PROCEDURE

CHEMICAL AND METALLURGICAL TESTS

1. OBJECTIVE

The object of this MTP is to present the theory and the practical methods currently used to conduct typical chemical and metallurgical tests on specific test specimens.

2. BACKGROUND

Much literature concerning chemical and metallurgical analysis and testing is available. The references in Section 4 were chosen to reflect those documents which currently are considered most informative in specialized areas. These documents are referenced throughout the test of this MTP and should be consulted for a full understanding of chemical and metallurgical analysis and testing.

A number of the tests presented in this MTP were developed as a result of specific requirements brought about by the advances in missiles in recent years. Other tests are derived from older standard methods of performing chemical analysis.

3. REQUIRED EQUIPMENT

Table I lists all equipment required for these tests.

4. REFERENCES

A. Beckman GC-2 Gas Chromatograph Instruction Manual
B. Beckman Application Data Bulletin GC-86-MI
C. Mason, D. M. and Vango, S. P., Simplified Method for Testing the Acceptability of Fuming Nitric Acid for Storage and Use as an Oxidizer for Rocket Fuels, California Institute of Technology, Jet Propulsion Laboratory, Pasadena, California, Report No. 20-205.
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L. Wiberley, S. E., Spraque, J. W., and Campbell, J. E., Analytical Chemistry, Volume 29, Page 210, 1957
M. Standards for Protection Against Radiation, Title 10, Atomic Energy Commission, Chapter 1, Part 20
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Q. Metal Handbook, American Society for Metal, Cleveland, Ohio, 1948
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5. SCOPE

5.1 SUMMARY

The chemical and metallurgical tests, described in paragraphs 6.2.1 through 6.2.22 of this MTP, are summarized as follows:

a. Determination of Resin in Corporal Fuels -- Paragraph 6.2.1 is a test to determine resin formed in furfuryl alcohol or in other mixtures containing furfuryl alcohol in which the resin has a higher boiling point than other components of the mixture.

b. Determination of Sn (Tin) in H_2O_2(Hydrogen Peroxide) -- Paragraph 6.2.2 is a test to analyze the presence of tin in hydrogen peroxide using the polarographic method of chemical analysis.

c. Analysis of Aniline Using the $HClO_4$ (Perchloric Acid) Method -- Paragraph 6.2.3 is a test used for the analysis of pure aniline and various mixtures of aniline in furfuryl alcohol and water. The perchloric acid method of analysis is relatively simple and easily conducted.

d. Analysis of H_2O_2 (Hydrogen Peroxide) by the Permanganate Method -- Paragraph 6.2.4 is a test to analyze aqueous solutions of hydrogen peroxide over a wide range of peroxide concentrations.

e. Analysis of Aniline by Gas Chromatography -- Paragraph 6.2.5 is a gas chromtographic analysis of aniline that may be used in any mixture in which none of the other components give a chromotographic peak at the same time as aniline, under the conditions of analysis. The accuracy of this method is approximately 1 percent of the total weight percent of the aniline present.

f. Analysis of UDMH Using the KIO_3 (Potassium Iodate) Method -- Paragraph 6.2.6 is a test used to analyze the UDMH present in JP fuels.

g. Analysis of UDMH in JP-17 Fuel Using the Water Extraction Method -- Paragraph 6.2.7 is a test used to analyze the UDMH present in JP-17

Table I. Tabular Listing of Instrumentation, Equipment, and Supplies by Test Where Used (Sheet 1 of 4)

INSTRUMENTATION, EQUIPMENT and SUPPLIES	CONDUCT OF TEST PARAGRAPH WHERE USED																				
	6.2.1	6.2.2	6.2.3	6.2.4	6.2.5	6.2.6	6.2.7	6.2.8	6.2.9	6.2.10	6.2.11	6.2.12	6.2.13	6.2.14	6.2.15	6.2.16	6.2.17	6.2.18	6.2.19	6.2.20	6.2.21	6.2.22	6.2.23
APRON, Acid Resistant															X								
ARM SLEEVES, Acid Resistant															X								
BALANCE, Analytical			X	X					X	X	X	X				X							
BALANCE, Pan				X	X																		
BALANCE, Micro (± 0.002mg)																						X	
BALLOON, Large																				X	X		
BEAKER, 5ml			X																				
BEAKER, 20ml										X													
BEAKER, 50ml						X																	
BEAKER, 250ml			X			X																	
BEAKER, Large Polyethylene										X													
BOATS, Weighing, Small																						X	
BOTTLE, 1 liter						X																	
BOTTLE, 2 liter			X			X																	
BOTTLE, 9 liter				X						X	X												
BOTTLE, Aspirator, 1 liter																					X	X	
BOTTLE, Eyedropper, 1 oz.				X																			
BOTTLE, Wash			X	X																			
BULB, Rubber															X								
BURETTE, 50ml	X	X	X																				
BURETTE, 100ml						X																	
BURNER, Meeker															X								
CELL, IR Absorption (inlet & outlet stopcocks)																				X	X		

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Table I. Tabular Listing of Instrumentation, Equipment, and Supplies by Test Where Used (Sheet 2 of 4)

INSTRUMENTATION, EQUIPMENT and SUPPLIES	CONDUCT OF TEST PARAGRAPH WHERE USED																				
	6.2.1	6.2.2	6.2.3	6.2.4	6.2.5	6.2.6	6.2.7	6.2.8	6.2.9	6.2.10	6.2.11	6.2.12	6.2.13	6.2.14	6.2.15	6.2.16	6.2.17	6.2.18	6.2.19	6.2.20	6.2.21	6.2.22	6.2.23
CELL, Sealed Sample (NaCl windows-0.100mm spacers)																X							X
CELL, Teflon (CaF_2 windows-2 in. path length)																X							X
CELL, Variable Path Liquid (0.100mm path length)																							
CHROMATOGRAPH, Gas					X			X											X	X			
CONDENSER, Water Cooled																X							
CONNECTIONS, Metal-T Tubing																				X	X		
CRUICIBLES, 25 ml Platinum															X								
CRUICIBLES, 25ml Porcelain	X																						
DARKROOM FACILITIES																							X
DESSICATOR										X					X					X			
DIE, KBr Pellet																						X	
ELECTRODE, Fisher		X																					
EXTRACTOR, Soxhlet																X						X	
EYEDROPPER			X																				
FACE SHIELD										X					X								
FILM VIEWER, High Intensity																							X
FLASK, Boiling (24/40 ground glass joints)																X						X	
FLASK, Erlenmeyer											X												
FLASK, 25ml Volumetric																X							
FLASK, 50ml Volumetric		X										X				X							
FLASK, 100ml Volumetric						X																	
FLASK, 250ml Volumetric			X									X											

Table I. Tabular Listing of Instrumentation, Equipment, and Supplies by Test Where Used (Sheet 3 of 4)

INSTRUMENTATION, EQUIPMENT and SUPPLIES	CONDUCT OF TEST PARAGRAPH WHERE USED																					
	6.2.1	6.2.2	6.2.3	6.2.4	6.2.5	6.2.6	6.2.7	6.2.8	6.2.9	6.2.10	6.2.11	6.2.12	6.2.13	6.2.14	6.2.15	6.2.16	6.2.17	6.2.18	6.2.19	6.2.20	6.2.21	6.2.22	6.2.23
FLASK, 500ml Volumetric		X																					
FLASK, 250cc Volumetric												X											
FLASK, 1 liter Volumetric												X											
FLASK, 2 liter Volumetric												X											
FUNNEL, 250ml Separatory							X			X													
GLASS ROD										X		X											
GLOVES, Acid Resistant										X					X								
GRADUATE CYLINDER, 100ml		X	X	X		X	X																
GRADUATE CYLINDER, 1 liter						X																	
HACKSAW																						X	
HOLDER, KBr Pellet																						X	
HOSE PIECES, Assorted Vacuum																				X	X		
HOSING, Rubber																X						X	
HOT PLATE		X													X								
INFRARED LAMP	X																						
MANTLE, Heating (with variac)																X						X	
MOTOR, ph (with glass stirrer)						X																	
OVEN, Laboratory 150 degree			X	X																			
OVEN, Vacuum										X	X												
PIPETTES, 3ml						X																	
PIPETTES, 10ml	X	X														X							
PRESS, 20 Ton																						X	
PUMP, Vacuum (1mm Hg Vacuum)																				X	X	X	

Table I. Tabular Listing of Instrumentation, Equipment, and Supplies by Test Where Used (Sheet 4 of 4)

INSTRUMENTATION, EQUIPMENT and SUPPLIES	CONDUCT OF TEST PARAGRAPH WHERE USED																					
	6.2.1	6.2.2	6.2.3	6.2.4	6.2.5	6.2.6	6.2.7	6.2.8	6.2.9	6.2.10	6.2.11	6.2.12	6.2.13	6.2.14	6.2.15	6.2.16	6.2.17	6.2.18	6.2.19	6.2.20	6.2.21	6.2.22	6.2.23
PYCNOMETER, 50ml									X														
REFRACTOMETER, Immersion									X														
REFRIGERATOR												X											
RING STAND and RING							X																
SCREEN, Lead																							X
SPACERS, 5mm Stainless Steel																							X
SPATULA, Flat Blade																					X		
SPECTROGRAPH, NSL Emission																	X						
SPECTROPHOTOMETER, Beckman												X											
SPECTROPHOTOMETER, Perkins-Elmer (NaCl optics)																X				X	X	X	X
STOPCOCKS, 3-way, Glass																				X	X		
SYRINGE										X						X							
SYRINGE, Hypodermic, 2ml						X																	
THERMOMETER									X														
THIMBLES, Soxhlet Extractor																X						X	
TONGS	X																						
TONGS, Platinum Tipped															X								
TUBE, Polyethylene Sampling										X													
TUBING, Assorted Glass (ball & socket joints)																				X	X		
TWEEZERS																						X	
"WIG-L-BUG" and ACCESSORIES																						X	
X-RAY MACHINE																							X

fuel, which is a mixture of 17 parts of UDMH and 83 parts of JP-4 by weight.

h. Analysis of Redstone Alcohol Using Gas Chromatography -- Paragraph 6.2.8 in an analysis for the presence of methanol, ethanol, or water if a component is present in amounts from 0.05 to 100 percent in a mixture with one or both of the other components.

i. Analysis of Redstone Alcohol Using the Refractive Index Method -- Paragraph 6.2.9 is a method used for the analysis of concentrated Redstone alcohol and may be used to analyze a mixture of ethyl alcohol, methyl alcohol, and water in any ratio.

j. Determination of NO_2 (Nitrogen Dioxide) in RFNA -- Paragraph 6.2.10 is a test for analyzing the presence of nitrogen dioxide, nitric acid hydrofluoric acid, and water. This method is limited to an acid medium. The presence of oxidizable metal ions in the solution will interfere with the accuracy of this method. The amount of nitrogen dioxide present does not restrict the use of this method.

k. Determination of the Total Acidity in RFNA -- Paragraph 6.2.11 is a test to determine the total acidity of aqueous solutions.

l. Determination of HF (Hydrogen Fluoride) in RFNA -- Paragraph 6.2.12 is a test to determine the amount of hydrogen fluoride present in RFNA. The presence of hydrogen fluoride in RFNA materially lowers the corrosion rate when RFNA is in contact with aluminum and stainless steel alloys. The inhibiting action of the fluoride ion is greatest around 0.6 weight percent hydrogen fluoride. This method is limited to solutions that contain no chloride or sulfate ions. The presence of iron and aluminum in amounts exceeding 500 micrograms per 50-ml portion will also interfere with this method.

m. Determination of HNO_3 (Nitric Acid) in RFNA -- Paragraph 6.2.13 is a test to determine, by difference, the amount of nitric acid present in RFNA.

n. Determination of Water in RFNA -- Paragraph 6.2.14 is a test to determine the percent of water present in RFNA.

o. Determination of Total Solids in RFNA -- Paragraph 6.2.15 is a test to determine the amount of total solids in RFNA of any concentration.

p. Quantitative Determination of Trinitrotoluene (TNT), Nitrocellulose (NC), and Cyclotrimethylene Trinitramine (RDX) in Explosives Using Extraction Techniques and Infrared (IR) Spectroscopy -- Paragraph 6.2.16 is a test to determine the concentration, percent by weight, of TNT, NC, and RDX when in a mixture with each other.

q. Quantitative Analysis of the Alloying Elements in Steels Using the NSL Emission Spectrograph -- Paragraph 6.2.17 is a test to analyze the presence of boron, chromium, managanese, molybdenum, nickel, silicon, and vanadium in steels.

r. Quantitative Analysis by Gas Chromatography Employing the "Marker" Method -- Paragraph 6.2.18 describes the "marker" method for the quantitative determination of the presence of a particular component in a mixture. The "marker" method of analysis can be used with a mixture containing any number of components, so long as the peak of the "marker" does not overlap the peak of any of the components. The accuracy obtainable for a particular component is within 1 percent of the true amount.

s. An IR Spectroscopic Procedure for Determining the Percentage of SF_6 (Sulfur Hexafluoride) in Gas Samples -- Paragraph 6.2.19 is a procedure used to determine the concentration, percentage by volume, of sulfur hexafluoride in gas samples. The determination is made at 660.0 mm Hg and 23° C over the concentration range from approximately 50 to 100 percent with a standard error of 1.6 percent.

t. An IR Spectroscopy Procedure for Determining the CO (Carbon Monoxide) and CO_2 (Carbon Dioxide) Content of Air Samples (Missile Exhaust) -- Paragraph 6.2.20 is a test to determine the carbon monoxide and carbon dioxide content of gas samples from their IR absorption band.

u. An IR Spectrophotometric Method for the Analysis of Dioctyl Adipate (DOA) in a Mixture with Ammonium Perchlorate, Aluminum, and Polyvinyl Chloride Using a KBr (Potassium Bromide) Pellet Technique -- Paragraph 6.2.21 is a test providing a means for the quantitative analysis of dioctyl adipate in missile propellants.

v. An IR Spectroscopic Method for Determining the Water Content of Various Organic Solvents -- Paragraph 6.2.22 is a test to determine the water content of the following organic solvents:

Solvent	Range (ppm water)
Benzene	16-700
Carbon Tetrachloride	5-90
Toluene	5-400

5.2 LIMITATIONS

Due to the variety of ideas concerning chemical and metallurgical analysis and testing and the great number of such tests that could be conducted, a detailed coverage of the entire field is beyond the scope of this MTP. The more common types of analyses and the recommended methods for obtaining these analyses in the laboratory are discussed. The tests presented are representative of the general field. Frequent reference is made throughout this pamphlet to applicable MTP's, specifications, and other publications for the specific details in accomplished difficult analyses utilizing complex test methods. These references should be consulted to obtain a full understanding of the tests.

6. PROCEDURES

6.1 PREPARATION FOR TEST

Personnel conducting the tests or making calculations should be thoroughly familiar with the instrumentation, equipment, and supplies required. The following general procedures should be used throughout the various tests described in paragraphs 6.2.1 through 6.2.22.

a. Follow standard laboratory, military, and manufacturer's safety practices and testing techniques to avoid injury to personnel or contamination of the test specimens.

b. Consult all pertinent paragraph, table, and figure references, prior to commencing a particular test.

c. Ensure that applicable instructions and specifications are available.

d. Prepare a test log book or folder for recording all pertinent test results.

6.2 TEST PROCEDURE

6.2.1 Determination of Resin in Corporal Fuels

NOTE: The evaporation of a fuel sample by using an IR lamp is a method that may be used to determine resin formed in furfuryl alcohol or mixtures containing furfuryl alcohol. This method is based on the consideration that the boiling temperature of the resin is higher in comparison to the other components.

a. The fuel sample shall be analyzed in duplicate.

b. The crucibles shall be dried for approximately 45 minutes under an IR lamp and then the crucibles shall be dried in a dessicator.

c. A constant weight for each crucible shall be determined and recorded.

d. 10 ml of each fuel sample shall be pipetted into separate crucibles.

NOTE: The density of the sample at 60°F will be found and recorded.

e. An IR lamp shall be positioned 5 inches above the crucibles and heated until the samples no longer flow when tilted at a 45° angle.

f. As each sample is dried, it shall be placed in a dessicator to cool.

g. The crucibles shall be removed from the dessicator, an IR lamp shall be positioned 6 inches above the crucibles, and the crucibles shall be heated for 45 minutes.

h. The crucibles shall be cooled in a dessicator and each sample shall be weighed and the weight recorded.

6.2.2 Determination of Sn (Tin) in H_2O_2 (Hydrogen Peroxide)

NOTE: The polarographic method is well suited for analysis of tin in hydrogen peroxide because of the sensitivity of the method. The Fisher electropode, a polarograph employing a dropping mercury electrode polarographic cell, is used in this analysis. By using the polarographic method, a concentration of 0.1 mg of tin per liter of H_2O_2 can be detected. Accuracy of small concentrations is no better than 10 percent.

a. The following reagents shall be obtained before conducting this test and shall be used to detect the presence of tin in a hydrogen peroxide sample:

1) NH_4Cl, (Ammonium Chloride, Reagent grade)
2) HCl, (Hydrochloric Acid, ACS grade)
3) NaOH, (Sodium Hydroxide, ACS grade)
4) Sn (Tin, CP grade)
5) Gelatin (Freshly prepared 0.25 percent aqueous solution)
6) Hg, (Mercury, equivalent to triple distilled)
7) N_2, (Nitrogen, Oxygen free)

b. Preparation of standard polargram

1) 0.5000g of tin shall be accurately weighed and completely dissolved in 250 ml of concentrated hydrochloric acid.
2) The solution shall be transferred to a 500-ml volumetric flask and diluted to the mark with distilled water.

3) One ml of this solution (one mg of tin) shall be pipetted into a 50-ml volumetric flask containing 10.7 g of ammonium chloride and approximately 30 ml of distilled water.
4) One ml of 0.25 percent gelatin solution shall be added, the solution shall be brought to room temperature and diluted to the 50 ml mark with distilled water.
5) 20 ml of this prepared solution shall be pipetted into a polarographic cell and the solution shall be flushed in the cell for 15 minutes with oxygen free nitrogen.
6) The solution shall be polargraphed between -0.25 and -0.75 volts at 2X sensitivity and the diffusion current shall be determined and recorded.

c. Determination of the tin content of the unknown sample.

1) 150 ml of the hydrogen peroxide sample shall be measured in a graduated cylinder and poured into a 500-ml widemouthed erlenmeyer flask.
2) 150 ml of distilled water and 5 ml of 2N NaOH shall be added to the flask.
3) The contents of the flask shall be warmed gently on a hot plate until the peroxide decomposition is vigorous.
4) The flask shall be removed and placed under a hood until the decomposition ceases.
5) The flask shall be placed on the hot plate and the solution shall be brought to boiling while carefully reducing the volume to approximately 10 ml.
6) 7.5 ml of 35 percent hydrochloric acid shall be poured from a burette into a 50-ml volumetric flask.
7) The 10 ml of decomposed peroxide shall be poured from the erlenmeyer flask into the 50-ml volumetric flask.
8) The erlenmeyer flask shall be washed into the volumetric flask with three 10-ml portions of ammonium chloride solution (total of 10.7 g ammonium chloride).
9) One ml of 0.25 percent gelatin solution shall be added to the volumetric flask and the flask shall be brought up to volume with distilled water.
10) 20 ml of the solution shall be accurately transferred into the polarographic cell and the cell shall be flushed with oxygen free nitrogen for 15 minutes.
11) The solution shall be polarographed between -0.25 and -0175 volts at 2X sensitivity and the diffusion current shall be determined and recorded.

6.2.3 . Analysis of Aniline Using the $HClO_4$ (Perchloric Acid) Method

NOTE: The perchloric acid method of analyzing aniline in furfuryl alcohol, water, or any mixture that does not contain a component, such as hydrazine, that exhibits basic properties in a non-aqueous solution, depends upon the neutralization of aniline (a weak base). This titration exhibits a sharp change in slope in its titration curve and for this reason an indicator may be used to indicate the end point

a. The following reagents shall be procured prior to conducting this test and shall be used during this test to analyze aniline in a nonaqueous solution:

1) Na_2CO_3, Sodium Carbonate (ACS grade)
2) $HClO_4$, Perchloric Acid, 60 percent by volume (ACS grade, 40 ml per liter of acetic acid)
3) $HC_2H_3O_2$, Acetic Acid (ACS grade, one liter per standard $HClO_4$ solution; 100 ml per standardization or titration; 250 ml per sample)
4) Methyl Violet, Crystal (0.1 g per 10 ml of indicator solution.

b. Preparation and Standardization of Reagents

1) The methyl violet indicator solution shall be prepared by weighing approximately 0.1 g of crystalline methyl violet, transferring it to a dropper bottle, and dissolving in 10 ml of glacial acetic acid.

NOTE: The prepared methyl violet solution is stable and may be kept for future analyses.

2) The perchloric acid solution shall be prepared by measuring approximately 12 ml of perchloric acid, 60 percent by volume, into a 100-ml graduate, adding the perchloric acid to one liter of glacial acetic acid and mixing thoroughly in a 2-liter bottle.
3) The perchloric acid solution standardization.

(a) 0.1 g of sodium carbonate, which has been previously dehydrated in an oven for one hour at 105° C, shall be weighed and added to a 250-ml beaker. The weight of sodium carbonate shall be recorded.
(b) The sodium carbonate shall be dissolved in approximately 75 ml of glacial acetic acid and by means of an eye-dropper one drop of the methyl violet indicator solution shall be added.
(c) The burette shall be rinsed at least 3 times with perchloric acid solution and then the burette shall be filled with the perchloric acid solution.
(d) The solution shall be added to the beaker at the rate of 5 to 10 ml per minute while constantly stirring.
(e) The solution shall be titrated until the color of the solution being titrated changes from dark blue to blue green.
(f) The volume of perchloric acid solution required for titration shall be recorded.
(g) The normality of the perchloric acid solution shall be determined before proceeding.

c. Determination of unknown sample

1) Approximately 10 ml of the fuel sample to be analyzed shall

be transferred to a 1-oz eyedropper bottle and the bottle shall be weighed to the nearest tenth of a milligram and the weight recorded.

2) 40 ml of glacial acetic acid shall be poured into a 250-ml volumetric flask.
3) 1.5 g of the fuel shall be introduced into the volumetric flask using an eyedropper.
4) The eyedropper bottle shall be weighed to determine the weight of the fuel sample and the weight shall be recorded.
5) Volumetric flask shall be filled to the mark with glacial acetic acid and thoroughly mixed.
6) 50 ml of glacial acetic acid shall be transferred into a 250-ml beaker and a 25-ml aliquot of the fuel sample shall be pipetted into the beaker.
7) One drop of the indicator solution shall be added to the beaker.
8) The burette shall be rinsed at least three times with the perchloric acid solution and then filled with the perchloric acid solution.
9) The solution shall be added to the beaker at the rate of 5 to 10 ml per minute while constantly stirring.
10) Titration shall be continued until an end point is reached. The volume of perchloric acid solution required for titration shall be recorded.

NOTE: The end point is reached when the color of the solution changes from dark blue to blue green. If the end point is passed, the color of the solution changes to green, then yellow, and the titration must be rejected.

11) Duplicate titrations shall be conducted on the sample being analyzed and these must check within 0.1 percent.

6.2.4 Analysis of H_2O_2 (Hydrogen Peroxide) by the Permanganate Method

NOTE: The permanganate method may be used for analyzing the presence of hydrogen peroxide in aqueous solutions over a wide range of peroxide concentrations. This method depends upon the reaction between acidified hydrogen peroxide and a standardized potassium permanganate solution, $5H_2O_2 + 3H_2SO_4 + 2KMnO_4 \rightarrow 2MnSO_4 + K_2SO_4 + 8H_2O + 5O_2$.

a. Reagents -- The following reagents are used during this test to detect the presence of hydrogen peroxide in an aqueous solution and shall be procured prior to conducting this test.

1) $KMnO_4$, Potassium Permanganate Solution (0.5N).
2) H_2SO_4, Sulfuric Acid Solution (20 percent by volume).
3) $Na_2C_2O_4$, Sodium Oxalate (Primary Standard)
4) Distilled Water

b. All glassware intended to contain hydrogen peroxide solutions, shall first be cleaned as follows:

1) The glassware shall be washed in a solution of a synthetic detergent.
2) The glassware shall be immersed in a 10 percent sodium hydroxide solution for one hour at room temperature.
3) The glassware shall be rinsed with tap water.
4) The glassware shall be immersed in sulfuric acid, 35 percent by volume, for one hour at room temperature.
5) The glassware shall be rinsed in distilled water and dried in an oven at 105° C.

c. Preparation of potassium permanganate solution.

1) 16 g of reagent grade potassium permanganate crystals shall be dissolved per liter of distilled water.
2) The solution shall be allowed to stand at room temperature (60° F to 90° F) for at least one week.
3) The aged solution shall be filtered through glass wool into a 9-liter bottle; the first 25 ml filtrate shall be discarded.

d. Standardization of potassium permanganate solution.

1) The potassium permanganate solution shall be standardized before use and weekly thereafter.
2) A small beaker shall be weighed and the weight shall be recorded.
3) 1 g + 0.1 mg of sodium oxadate, which has been dyhydrated in a vacuum oven for one hour at 110° C, shall be added to the beaker.
4) The beaker shall again be weighed and the weight recorded.
5) The sodium oxadate assay shall be recorded.
6) This beaker and its contents shall be placed in a 500-ml erlenmeyer flask to which 200 ml of 20 percent sulfuric acid and 40 ml of distilled water have been added.
7) The solution shall be heated to a temperature between 80° and 90° to dissolve the sodium oxadate and these temperature limits shall be maintained during the course of the titration.
8) A burette shall be rinsed at least four times with the potassium permanganate solution,then the burette shall be filled with the solution.
9) The solution shall be added to the flask at the rate of 10 to 20 ml per minute while constantly stirring. Titration shall be continued until a persistent faint pink coloration appears.
10) The volume of titrant shall be recorded.

NOTE: If the end point is passed, the titration must be rejected. The first titration may be used to estimate the amounts of permanganate solution required for the other samples. The equation for this reaction is:

$$2KMnO_4 + 5Na_2C_2O_4 + 8H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 5Na_2SO_4 + 10CO_2 + 8H_2O.$$

e. Determination of unknown sample.

1) A clean dry weighing bottle (10-ml beaker) shall be weighed to the nearest 0.1 mg.
2) 3 to 4 drops of hydrogen peroxide (approximately 0.4 g of 75.7 percent H_2O_2) shall be introduced into the bottle by means of a clean eyedropper.
3) The bottle with its contents shall be accurately weighed to determine the sample weight.
4) The weighing bottle containing the hydrogen peroxide shall be placed into a clean 500-ml widemouthed erlenmeyer flask to which 70 ml of 20 percent by volume sulfuric acid has previously been added.
5) The burette shall be rinsed at least four times with the 0.5N potassium permanganate solution and then filled with the solution.
6) The solution shall be added to the flask at the rate of 10 to 30 ml per minute while constantly stirring.
7) Continue titration until the end point is reached. The amount of potassium permanganate solution required for titration shall be recorded.

NOTE: The end point is reached when a persistent pink coloration appears. If during the titration the solution in the flask turns brown, the sample should be rejected and the analysis repeated. The brown coloration will indicate a deficiency of acid or too rapid addition of the potassium permanganate solution to the contents of the flask.

8) Duplicate titrations of the sample being analyzed shall be conducted and these shall check within 0.07 percent.

6.2.5 Analysis for Aniline by Gas Chromatography

NOTE: The accuracy of this method is approximately 1 percent of the total weight percent of aniline present in the mixture being analyzed. Consult reference A of this MTP for further understanding of the use of the gas chromatograph.

a. The following conditions shall be set on the chromatograph, preferrably the night before the analysis is to be made:

1) Pressure to 20 psig on the instrument and 30 psig on the tank gauge.
2) Temperature to 190° C setting and the recorder power shall be turned on.

b. When the temperature equilibrium has been reached, the power switch shall be turned to "+" and the current shall be adjusted to 255 ma.

c. A few minutes shall be allowed for the instrument to stabilize so that there is no baseline drift.

d. A 0.005-ml sample of reagent grade aniline shall be prepared.

e. The ambient temperature in the area where the sample is being prepared shall be recorded.

f. The recorder chart drive shall be turned to the on position, the

recorder pen shall be zeroed with attenuation at "1", and the aniline sample shall be injected.

g. The time shall be recorded and the attenuation shall be turned to "20".

h. Ten minutes later, another aniline sample shall be injected and still ten minutes later, another sample.

i. The three aniline peaks shall then be recorded in succession.

j. The recorder chart drive shall be turned to the off position.

k. The first peak shall be discarded and the unit area counts under each of the other two peaks shall be determined in the manner shown in Figure 1.

l. From the two unit area counts, the average for the two peaks shall be determined and the number of counts per gram of aniline shall be determined assuming the reagent grade aniline to be 99.9 percent pure.

NOTE: The weight of a 0.005-ml sample of aniline from 20° to 25° C is 5.09×10^{-3} grams

m. The sampler glass needle assemble shall be cleaned before proceeding further.

n. A 0.005-ml sample of the aniline mixture to be analyzed shall be prepared.

o. The ambient temperature in the area where the sample is prepared is recorded and the sample density at this temperature shall be determined and recorded.

p. The recorder chart drive shall be turned to the on position, the recorder pen shall be zeroed with attenuation at "1", and the sample injected.

q. After the dissolved air peak has been recorded, approximately 30 seconds, the time shall be recorded and the attenuation shall be set at "20" if the percent of aniline is suspected to be over 60 percent, "10" if the percent of aniline is expected to be 30 to 60 percent, and "5", "2", or "1" if only small amounts of aniline are expected to be present.

NOTE: A trial run may be necessary to determine the best attenuation setting to be used.

r. The sample peak shall be recorded between 24 and 31 minutes after the air peak has been recorded.

s. Other aniline samples shall be run in the same manner, cleaning the sampler needle glass assembly between each sample and after all samples have been analyzed.

t. The average unit area counts for the test sample shall be determined and recorded.

u. The instrument power switch shall be turned on, the recorder power shall be turned off, instrument pressure shall be turned to 3 to 5 psig and the temperature setting shall be turned to 100° or lower.

6.2.6 Analysis of UDMH Using the KIO_3 (Potassium Iodate) Method

NOTE: This method is based upon the oxidation of UDMH. The products of oxidation of UDMH under the conditions of this titration are not definitely known. However, the following equation can be written which is in accord with the stoichiometry observed:

$$10\ (CH_3)_2NNH_2 + 4KIO_3 + 4HCl \longrightarrow 5\ (CH_3)_2\ NN - NN\ (CH_3)_2 + 4\ KCl + 2I_2 + 12H_2O$$

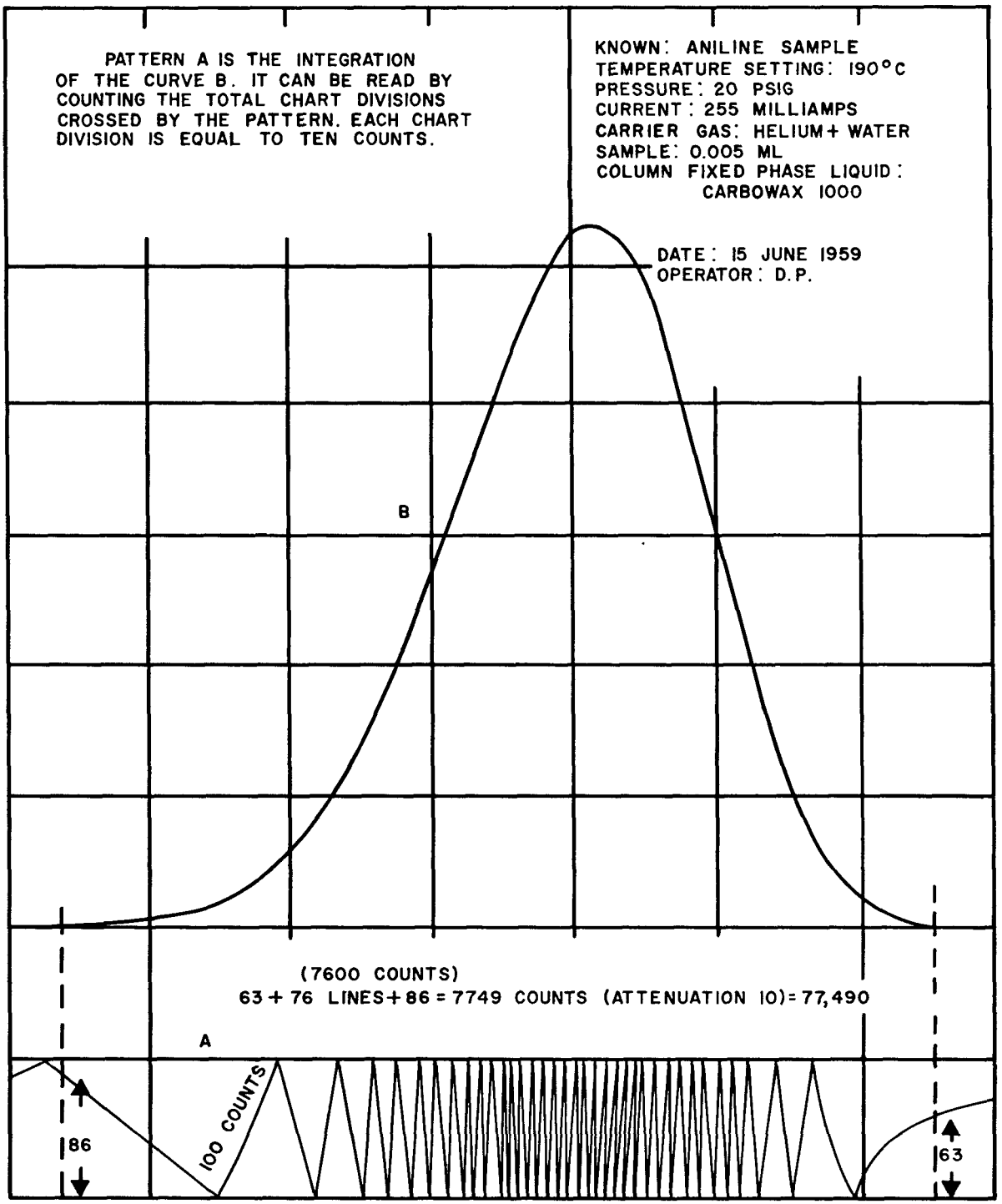

FIGURE 1. DETERMINING THE UNIT AREA COUNTS FOR SAMPLE PEAKS

After the UDMH has been oxidized, additional potassium iodate oxidizes the I_2 (iodine) formed during the oxidation of the UDMH:

$$2I_2 + KIO_3 + 6HCl \rightarrow KCl + 5ICl + 3H_2O$$

Therefore, one mole of potassium iodate oxidizes two moles of UDMH under the conditions of the titration.

a. The following reagents are used during this test to analyze UDMH and shall be procured before initiation of testing.

1) KIO_3, Potassium Iodate (ACS grade - 21.4 g dissolved in water and made up to 1 liter).
2) HCl, Hydrochloric Acid (ACS grade - add 750 ml water and make up to 1 liter, 12N).
3) HCl, Hydrochloric Acid (ACS grade - add 42 ml and water to make up to 500 ml, 1N).
4) $HC_2H_3O_2$, Acetic Acid (ACS grade - 25 ml per run)

NOTE: Before using the potassium iodate, it shall be dried in an oven at 105° C for one hour. In addition to the reagents listed, dry ice and alcohol are required, during this test, for use as a cold bath.

b. Preparation and Standardization of Reagents.

1) 0.1M Potassium Iodate Solution

(a) The standard 0.1M potassium iodate solution shall be prepared by weighing 21.4 g of the previously dried potassium iodate in a 50-ml beaker.
(b) The weighed iodate shall be transferred to a one-liter volumetric flask.
(c) The flask shall be filled with water to the one-liter mark, and the potassium iodate dissolved. From the weight of the potassium iodate and its assay, the molarity of the solution shall be calculated.

$$M = \frac{g\ KIO_3}{Mol.\ wt.\ KIO_3} \times \text{assay/volume in liters}$$

(d) The molarity shall be recorded.

2) 9 N Hydrochloric acid solution

(a) The 9N hydrochloric acid solution shall be prepared by measuring 750 ml of concentrated hydrochloric acid into a large graduate cylinder.
(b) The acid shall be slowly added to one liter of distilled water, mixed well, and stored in a 2-liter bottle.

3) 1N Hydrochloric Acid Solution

(a) The 1N hydrochloric acid solution shall be prepared by measuring 42 ml of concentrated HCl into a 100-ml graduate cylinder.
(b) The acid shall be slowly added to 500 ml of distilled water, mixed well, and stored in a one-liter bottle.

c. Analysis of Concentrated UDMH

1) The ph meter and auxiliary equipment for making the potentiometric titration shall be assembled.

WARNING

UDMH has a flash point of 0° C. No smoking or open flames are permitted in the vicinity when this material is being handled. UDMH is also toxic and caustic. Do not inhale fumes or permit the liquid to contact the skin or eyes.

2) 50ml of 1N hydrochloric acid shall be pipetted into a 100-ml volumetric flask, a stopper shall be inserted and the sample shall be weighed to the nearest tenth of a mg.
3) The flask and its contents shall be cooled in a dry ice and alcohol bath for 30 seconds.
4) Approximately 3 ml of UDMH shall be pipetted into the flask while the flask is swirled. The pipette tip shall not be permitted to touch the acid.
5) The flask shall be stoppered and shaken thoroughly.
6) Distilled water shall be added to the 100-ml mark to dilute the solution and the contents shall be thoroughly mixed.
7) Approximately 75 ml of 12N of hydrochloric acid shall be poured into a 250-ml beaker and 20 ml of the sample solution shall be pipetted into the beaker.
8) The contents of the beaker shall be chilled to -5° C in a cold bath.
9) The beaker shall be placed in position for titration with the platinum and calomel electrodes immersed in the sample solution.
10) The sample shall be titrated with standard 0.1M potassium iodate solution to an endpoint voltage of 0.70 ± 0.04 mv.
11) The temperature of the solution shall be maintained between -5 and +5° C throughout the titration.

NOTE: The color of the sample solution is successively amber, brown, and light yellow at the endpoint. Make the titration rapidly until the yellow color starts to appear then make the voltage measurement to determine the exact endpoint.

12) The volume of potassium iodate solution required to reach the endpoint shall be recorded.

d. Analysis of mixtures of UDMH in JP-4 fuels

1) The ph meter and auxiliary equipment shall be assembled.

2) 25 ml of glacial acetic acid shall be cooled in a 250-ml beaker to near freezing point.
3) About 1.9 ml of the UDMH mixture shall be drawn into a clean and dry syringe.
4) After removing the syringe from the mixture, a small amount of air shall be drawn into the syringe to minimize evaporation losses.
5) The syringe needle shall be wiped off and the syringe weighed on an analytical balance.
6) About 1.6 ml of the UDMH mixture shall be ejected into the cooled glacial acetic acid.
7) The needle shall be withdrawn from the solution, a small amount of air drawn into the syringe, the needle dried, and the syringe reweighed.

NOTE: The difference in the two weights of the syringe is considered the weight of the sample.

8) The weight of the sample shall be recorded.
9) 100 ml of 9N HCl that has been cooled to between 0 to 5° C shall be added to the beaker.
10) The beaker shall be placed in position for titration with the platinum and calomel electrodes immersed.
11) The sample shall be titrated with the standard 0.1M potassium iodate solution until an endpoint is obtained.

NOTE: An endpoint is obtained generally when one drop of this solution will give a potential drop from 0.35 to 0.25 volts. Throughout the entire titration, the temperature of the solution shall be maintained between 0 and 5° C. The color of the solution changes from brown to yellow before the potentiometric endpoint is reached. Any JP-4 which is floating on the surface of the solution during the titration will be brown within a few drops of the end point.

12) The volume of potassium iodate solution required to reach the endpoint shall be recorded.

6.2.7 Analysis of UDMH in JP-17 Fuel Using the Water Extraction Method

NOTE: This method is based upon the solubility of JP-4 and UDMH in water.

WARNING

UDMH/JP-4 mixtures are flammable and should not be handled in the vicinity of flames, sparks, or strong oxidants. UDMH is toxic and caustic, thus inhalation of fumes and contact of the liquid with the skin and eyes must be avoided.

a. 100 ml, ±0.1 ml, of the JP-17 sample shall be measured into a graduate cylinder.
b. The density of JP-17 shall be recorded.

c. 100 ml fuel sample shall be transferred to a separatory funnel and approximately 100 ml of water shall be added to the sample.

d. The density and assay of the UDMH used in preparing the JP-17 shall be recorded.

e. The funnel shall be stoppered, inverted, and shaken vigorously for approximately 30 seconds.

f. The stopcock shall be opened to release any pressure, thus venting the separatory funnel.

g. The funnel shall be allowed to stand for 15 minutes to ensure complete separation.

h. The bottom layer, containing UDMH and water, shall be drained and discarded.

i. 10 ml of water shall again be added to the sample, the sample shall be shaken vigorously, any pressure shall be released and time shall be allowed for complete separation. The top layer shall be transferred to a 100-ml burette and water shall be added to bring the total liquid volume to 100 ml. 10 minutes shall be allowed for complete separation of the water and JP-4 fuel and the volume of JP-4 fuel shall be recorded.

6.2.8 Analysis of Redstone Alcohol Using Gas Chromatography

NOTE: The accuracy of the Redstone alcohol analysis by gas chromatography is:

Table II. Redstone Alcohol Analysis

Component	Absolute Deviation from True Value	Standard Deviation
MeOH, Methanol	± 0.1	± 0.06
EtOH, Ethanol	± 0.2	± 0.15
H_2O, Water	± 0.2	± 0.15

Consult References A and B of this MTP for further understanding of the use and application of the gas chromatograph.

NOTE: To perform this test, a pressure vessel is required in addition to the equipment specified. A thick, 9-liter, glass container with a narrow neck and a specially made cover may be used. The cover should be a stainless steel fitting that fits over the lip of the lip of the glass container and holds a two-hole rubber stopper in place against a pressure of approximately 50 psig. During the actual test, the container is used with a pressure of 30 psig. As a safety precaution, place the glass container in a polyethylene container. Into the 9-liter container, place 2 or 3 liters of water and a glass tube through one hold of the rubber stopper until it is well below the level of the water. Connect rubber tubing from a carrier gas cylinder to the glass tube. Place a 3/4-in. length of glass tubing in the remaining hole in the rubber stopper and connect this tube to the carrier gas line leading into the chromato-

graph instrument.

a. The following conditions shall be set on the chromatograph:

1) Pressure -- 20 psig on the instrument and 30 psig on the tank gauge.
2) Temperature -- 100°C setting (88° C actual)
3) Current -- 300 ma.
4) Recorder Power -- ON position (the night before use, if possible).

b. When equilibrium has been established and there is no zero baseline drift of the recorder pin, a 0.005-ml liquid sample of the alcohol to be analyzed shall be prepared.

c. One droplet shall be forced out of the sampler to make the volume slightly less than 0.005 ml.

d. The chart drive switch shall be set to the off position, the attenuation to "1", the pen on zero, and the liquid sample injected.

e. The needle shall be inserted far enough so that the retaining assembly of the sample touches the inlet.

NOTE: If the needle does not go in easily, remove it and try again. Forcing the needle against the metal shelf inside of the instrument will bend the needle.

f. The plunger shall be depressed quickly, and the sampler removed 10 seconds later.

g. Approximately 45 seconds after sample injection, an air peak will appear, the instrument timer shall then be set for 60 minutes.

h. When more than eight but less than 10 minutes have elapsed, the pen shall be zeroed, the attenuation set to "20", and the recorder chart drive switch positioned to the on position.

i. Methanol and ethanol peaks shall be recorded successively during an 8-minute period.

j. After the pen is back to the baseline, the chart drive shall be turned off.

k. Thirty minutes from the beginning time, the attenuation shall be set to "1", the pen zeroed, and the recorded chart drive switch positioned to the on position.

l. A water peak shall then be recorded.

m. The tail of the water peak shall be allowed to run until the pen has completely leveled or begins to rise slightly.

n. The chart drive shall be turned off, and the chart paper removed and recorded.

o. After all samples are analyzed, the instrument power shall be turned to the on position, the instrument pressure gauge set to 3 to 5 psig, and the chart drive recorder turned off.

p. The sampler glass needle assembly shall then be cleaned.

6.2.9 Analysis of Redstone Alcohol Using the Refractive Index Method

NOTE: This analysis is based upon the relationship between the specific gravity of the alcohol mixture at 15.6/15.6° C and its refractive index (immersion) scale reading at 20° C.

a. The pycnometer shall be assembled and weighed on an analytical balance. The weight shall be recorded.

NOTE: Care must be taken to zero the balance and ensure that the pycnometer is free from dust or lint during all weighings.

b. The pycnometer shall be filled completely with water, ensuring that air bubbles are not trapped in the bottle, then the water shall be cooled to below 15.6° C.

c. The thermometer shall be inserted with a turning motion to avoid bubbles.

NOTE: The thermometer should fit firmly in place but shall not be forced into position.

d. The outside of the pycnometer shall be wiped dry, the temperature shall be allowed to rise to 15.6° C, the side arm shall be dried and a cap shall be placed on the side arm.

e. The pycnometer shall be weighed, and the weight recorded.

f. The thermometer and the side arm cap shall be removed and dried.

g. The bottle shall be rinsed at least three times with the alcohol mixture ensuring that the alcohol flows through the side arm.

h. The bottle shall be filled and cooled below 15.6°C.

i. The thermometer shall be inserted, the temperature shall be allowed to rise to 15.6° C, and the side arm dried as the alcohol expands.

j. A cap shall be placed on the side arm and the pycnometer shall be weighed. The weight shall be recorded.

k. The refractive index of the alcohol shall be determined at 20° C.

6.2.10 Determination of NO_2 (Nitrogen Dioxide) in RFNA

NOTE: An attempt to prepare nitrogen dioxide results in an equilibrium mixture of both nitrogen dioxide and N_2O_4 (nitrogen tetroxide). This method of analysis is based upon the following reaction using $Ce(H\ SO_4)_4$ (0.1N ceric acid sulfate) in one molar H_2SO_4 (surfuric acid) as the oxidizing agent:

$$2H_2O + N_2O_4 + 2Ce^{++++} \longrightarrow 2Ce^{+++} + 2NO_3^- + 4H^+$$

The nitrogen dioxide in samples of RFNA is first reacted with an excess of ceric acid sulfate and then back-titrated with $Fe\ SO_4\ 7H_2O$ (ferrous sulfate) to determine the amount of excess or unreacted ceric acid sulfate. The back-titration end point color is red-orange using ferroin as the indicator.

a. The following reagents are used during this test to analyze nitrogen dioxide in RFNA and shall be procured before beginning the test.

1) $Ce(H\ SO_4)_4$ 0.1N Ceric Acid Sulfate
2) $FeSO_4\ 7H_2O$. 1.1N Ferrous Sulfate
3) 0.025M Ferroin (Indicator)
4) $Os\ O_4$ (Catalyst) 0.01M Osmium Tetroxide

b. Preparation and Standardization of Reagents

1) The ceric acid sulfate solution (0.1N) preparation

(a) 500 g of ceric acid sulfate shall be weighed on a pan balance.
(b) A funnel shall be placed in the neck of a 9-liter bottle and the 500 g washed into the bottle using distilled water.
(c) Distilled water shall be added until the 9-liter bottle is approximately half full. 300 ml of sulfuric acid shall be slowly added while the bottle is continually agitated.
(d) The bottle shall be filled with distilled water to the 9-liter mark and the solution thoroughly mixed.
(e) When cool, this solution shall be filtered into another 9-liter bottle.

2) Ceric acid sulfate (0.1N) standardization

(a) The primary standard arsenious oxide (As_2O_2) shall be heated for at least one hour in a vacuum over at 110° C.
(b) The arsenious oxide shall be cooled in a dessicator until ambient temperature is reached.
(c) Three samples (approximately 0.20 g) of the dry arsenious oxide shall be weighed on an analytical balance and the weight recorded.
(d) Each sample shall be placed into a 500-ml erlenmeyer flash and 100 ml of distilled water and four pellets of NaOH (sodium hydroxide) shall be added.
(e) Each sample shall be heated until the arsenious oxide is completely dissolved.
(f) 35 ml of 5N sulfuric acid shall be added and then cooled to room temperature.
(g) Five drops of 0.01M osmium tetroxide catalyst and two drops of ferroin shall be added.
(h) The ceric solution shall be shaken, the burette rinsed with the ceric solution, and the arsenious oxide solution titrated to the disappearance of the red-orange color.
(i) Record volume of titrant.
(j) Normality of Ce ($H SO_4$) shall be calculated before proceeding.

3) 0.1N ferrous sulfate solution preparation.

(a) 280 g of ferrous sulfate heptahydrate ($FeSO_4$ $7H_2O$) shall be weighed on a pan balance.
(b) A funnel shall be placed in the neck of a 9-liter bottle and the ferrous sulfate shall be washed into the bottle using distilled water.
(c) Distilled water shall be added until the 9-liter bottle is approximately half full.
(d) 100 ml of concentrated sulfuric acid shall be slowly added while continually agitating the bottle.

(e) The bottle shall be filled with distilled water to the 9-liter mark and the solution thoroughly mixed.

4) During this test ferroin indicator (1-10 orthophenanthroline ferrous sulfate (0.025M)) shall be used.
5) The osmium tetroxide preparation.

(a) 2-ml of concentrated sulfuric acid shall be added to a one-liter volumetric flask that is approximately half full of distilled water.

WARNING

Osmium tetroxide is extremely toxic. Inhalation of its fumes could be fatal. The entire preparation of the osmium tetroxide reagent should be performed under a well ventilated laboratory hood.

(b) The contents of the flask shall be diluted to the one-liter mark using distilled water and the solution shall be thoroughly mixed.
(c) 100 ml of the solution shall be removed and poured into another one-liter volumetric flask.
(d) This solution shall again be diluted to the one-liter mark and thoroughly mixed.
(e) The 100 ml of this second solution shall be poured into a graduate cylinder and the graduate placed in a large polyethylene beaker.
(f) 1/4 g of osmium tetroxide (contained in a sealed glass vial) shall be added to the graduate containing the sulfuric acid.
(g) Using a glass rod, the osmium tetroxide shall be pushed to the bottom of the graduate, crushed completely, and dissolved completely.
(h) This solution shall be poured into a reagent bottle.
(i) All equipment and hands shall be washed thoroughly in soap and warm water.

c. Analysis of Nitrogen Dioxide in RFNA.

1) This procedure shall be run in duplicate for each acid sample.
2) The ampules shall be weighed on an analytical balance and the weight recorded, and then placed into 20-ml beakers.
3) The top of the sample shall be covered with a finger and the beaker filled with powdered dry ice.
4) The beakers shall be placed under a ventilating hood using a face shield and gloves.
5) A polyethyene sampling tube shall be inserted into a syringe and the tube rinsed twice with the acid sample.
6) Using the syringe and sampling tube an acid sample of 1.2 g shall be injected into each ampule.
7) Each ampule shall be sealed with an oxy-torch, being careful not to leave cracks or openings in the ampules.

8) The ampules shall be removed from the dry ice and rinsed in warm water until unfrozen.

NOTE: The ampules shall be carefully checked for leaks. If an ampule is leaking, it shall be placed below the surface of a saturated sodium bicarbonate solution and the ampule broken.

9) The sealed ampules shall be dried thoroughly, weighed on an analytical balance, and the weight recorded.
10) The ferrous sulfate and ceric sulfate bottles shall be thoroughly shaken, and the burettes connected to these bottles shall be drained 2 or 3 times.
11) 50.00 ml of the ceric solution shall be measured into a ground glass, 500-ml, stoppered bottle.
12) An ampule shall be carefully placed into the bottle.
13) The bottle shall be stoppered tightly and the bottle shaken until the ampule is completely crushed.
14) The bottle shall be shaken intermittently for 15 minutes to allow the reaction to complete.
15) Two drops of ferroin shall be added and the excess ceric solution shall be back-titrated with the ferrous solution to the red-orange endpoint.
16) The amount of titrant shall be recorded.
17) The ferrous-ceric conversion factor shall be determined by titrating 35 ml of the ceric solution with the ferrous solution using ferroin as an indicator.
18) The amount of titrant shall be recorded.

6.2.11 Determination of the Total Acidity in RFNA

NOTE: The process of neutralization is used for determining the quantity of acid present. In neutralizing RFNA with NaOH (sodium hydroxide), the amount of acid may be ascertained by determining the volume of sodium hydroxide of known concentration that is required to neutralize a known weight of RFNA. The total acidity of the RFNA is expressed in HNO_3 (nitric acid). The NO_2 (nitrogen dioxide) is oxidized with H_2O_2 (hydrogen peroxide) and forms part of the total acidity. The equation representing the reaction of hydrogen peroxide with nitrogen dioxide is:

$$H_2O_2 + 2NO \longrightarrow 2HNO_3$$

a. The following reagents are used during this test to determine the total acidity in RFNA and shall be procured before beginning this test.

1) NaOH, Sodium Hydroxide, (0.5N, Phenolphthalein Indicator).
2) Potassium Hydrogen Phthalate.

b. Preparation and Standardization of Reagents.

1) Preparation of 0.5N sodium hydroxide solution.

(a) The 0.5N sodium hydroxide solution shall be prepared by measuring into a glass graduate cylinder 250 ml of liquid sodium hydroxide (C.P. Grade, 50/50 by weight, carbonate free).
(b) The hydroxide shall be poured into a 9-liter bottle and the bottle shall be filled with distilled water to the 9-liter mark.
(c) The solution shall then be thoroughly mixed.

2) Standardization of 0.5N sodium hydroxide solution.

(a) The sodium hydroxide solution shall be standardized by heating approximately 10 g of potassium hydrogen phthalate, using a vacuum oven, at 110° C for one hour.
(b) The phthalate shall be cooled in a dessicator to room temperature.
(c) Three samples, 3 to 4 grams each, of the dried phthalate shall be weighed on an analyitcal balance and each sample placed into a 250-ml erlemeyer flask.
(d) 100 ml of distilled water shall be added to each sample and heated gently until the phthalate is dissolved.
(e) The phthalate shall be cooled to room temperature.
(f) Four drops of phenolphthalein indicator shall be added and the solution titrated with the sodium hydroxide solution until the solution changes from colorless to a faint pink.
(g) The normality shall then be calculated

$$\text{Normality NaOH} = \frac{\text{g phthalate x assay of phthalate}}{\text{ml of NaOH x 0.20422 (milliequivalent weight of potassium hydrogen phthalate)}}$$

(h) The three samples must agree within one part per thousand.
(i) The normality of NaOH shall be recorded.

3) Preparation of phenolphtalein indicator.

(a) The phenolphthalein indicator shall be prepared by weighing on an analytical balance approximately one g of phenolphthalein.
(b) The phenolphthalein shall be dissolved in 70 ml of ethyl alcohol, 30 ml of distilled water shall be added and the solution shall be thoroughly mixed.

c. Analysis for Total Acidity in RFNA.

1) This procedure shall be run in duplicate for each acid sample.
2) Weighed samples shall be obtained in the same manner as described in paragraph 6.2.10c. The weight shall be recorded.
3) The ampule containing the weighed sample of acid shall be placed into a glass stoppered bottle containing 5 ml of hydrogen peroxide (30 percent) and approximately 70 ml of

distilled water.

4) The bottle shall be shaken until the ampule is completely crushed.
5) 15 minutes shall be allowed for the hydrogen peroxide to react with the nitrogen dioxide and the bottle shall be shaken intermittently during the 15 minutes.
6) The stopper shall be removed from the sample bottle and any solution clinging to the cap shall be washed into the bottle.
7) Four drops of phenolphthalein indicator shall be added.
8) The solution shall be titrated with NaOH until a faint pink color appears.
9) The amount of titratant required shall be recorded.
10) A "blank" correction shall be determined by titrating, with the sodium hydroxide, a solution composed of 70 ml distilled water, five ml hydrogen peroxide, and four drops of the indicator.
11) Record the amount of titrant required.
12) Steps 10 and 11 shall be repeated until 3 blank determinations have been made.

6.2.12 Determination of HF (Hydrogen Fluoride) in RFNA

NOTE: This method is based on Beer's law that the complexing of F (fluoride) with Zr (zirconium) causes a bleaching effect on reddish zirconium-alizarine dye and can be utilized in an analysis for the fluoride ion. To determine the fluoride content in RFNA, four solutions and a spectrophotometer (similar to the Beckman DU Spectrophotometer) are necessary. Reference D of this MTP gives instructions for operating a Beckman DU Spectrophotometer. One solution contains a known concentration of fluoride in zirconium-alizarine and is used to correct variables in the spectrophotometer that affect its functioning the same from day to day, such as fluctuations in electrical circuits and photo-tubes. The second solution contains an excess of fluoride ion in zirconium-alizarine and is used to provide a means for adjusting the spectrophotometer to 100 percent relative transmittance. A third solution contains only zirconium-alizarine (blank) and a fourth solution contains the unknown fluoride. The third and fourth solutions are used to determine the percent of fluoride in the unknown through the use of a prepared calibration curve.

a. Preparation of Reagents

1) Zirconium-alizarine Preparation

(a) 0.40 g of zirconium oxychloride hydrate and 0.80 g of alizarine sodium sulfonate (alizarine red S) shall be weighed on an analytical balance.
(b) Each of the above shall be dissolved in separate 250-cc volumetric flasks containing approximately 200 cc of distilled water.
(c) Each flask shall be filled with distilled water to the 250-cc mark and mixed thoroughly.

(d) 10 ml of this solution shall be pipetted into another one-liter volumetric flask and the flask filled to the one-liter mark with distilled water.

b. Test Procedure

1) Preparation of Calibration Curve

(a) Weigh 2.06 g ± 0.1 mg of sodium fluoride on an analytical balance and record the weight.
(b) Transfer the sodium fluoride to a one-liter volumetric flask and diluted to the one-liter mark with distilled water.
(c) 10, 8, 6, 5, 4, and 2 ml of this solution shall be pipetted into separate 250-ml volumetric flasks and each filled to the 250-ml mark with distilled water.
(d) 2 ml from each of these 250-ml flasks shall be pipetted into a corresponding number of 50-ml volumetric flasks.
(e) A sufficient amount of color reagent shall be prepared by mixing the zirconium-alizarine reagent with distilled water in the ratio of 1:1.
(f) The bleach solution shall be prepared by pipetting 5 ml of the stock bleach solution into a 50-ml volumetric flask.
(g) 5 ml of the prepared color agent shall be added to each of the 50-ml volumetric flasks.
(h) A blank solution shall be prepared by pipetting 5 ml of the prepared color reagent into a 50-ml volumetric flask and this flask shall be labeled "blank".
(i) Each volumetric flask shall be filled to the 50-ml mark with distilled water, shaken thoroughly, and allowed to stand for 90 minutes.
(j) During this 90-minute period, the spectrophotometer shall be turned on and the following adjustments made:

(1) The cell-shifting control set to the out position
(2) The shutter closed
(3) The selector switch set to one
(4) The wavelength set to 530 millimicrons
(5) The slit width set to 0.02 mm.
(6) The sensitivity set to midrange
(7) The blue photocell placed in position

(k) Four, matched, 1-cm path length, liquid absorption cells, shall be cleaned, filled with distilled water, and their absorbance determined and recorded.

NOTE: These absorption values shall be the cell corrections. If the absorbance of the cells is greater than ±0.01, the cells shall be recleaned and their absorbance determined again.

(l) When 15 minutes of the 90-minute waiting period remain, the bleach solution shall be placed in cell 1, the

blank solution in cell 2, and the first two standards in cells 3 and 4.

(m) After the spectrophotometer has warmed up, the dark current knob shall be turned until the galvanometer reads zero.

(n) The bleach solution (cell 1) shall be placed into position with cell-shift control, the absorbance set on zero, the shutter opened, and the sensitivity knob turned until the galvanometer reads zero.

NOTE: Do not touch the dark current knob once the dark current has been zeroed.

(o) The blank solution (cell 2) shall be placed in position.

(p) The absorbance-transmission knob shall be turned until the galvanometer reads zero and the absorbance reading shall be recorded.

(q) The first standard (cell 3) shall be placed in position and its absorbance determined.

(r) The second standard (cell 4) shall be placed in position and its absorbance determined.

(s) The measured absorbance shall be corrected by adding or subtracting the cell corrections previously determined.

(t) The difference in corrected absorbance between the blank solution and the standards against the known fluoride concentration for each 50-ml volumetric flask shall be plotted.

NOTE: Figure 2 illustrates a typical calibration curve.

2) Determination of Fluoride Content

(a) Two pellets (approximately 0.2 g) of potassium hydroxide shall be placed in a weighing bottle for each sample.

(b) Each bottle shall be filled half full with distilled water and stirred with a glass rod.

(c) Each bottle shall be capped, cooled to room temperature, weighed and the weight recorded.

(d) Approximately one g acid sample (0.7 ml) shall be added using a polyethylene pipette.

NOTE: The tip of the pipette should be about one-half inch above the liquid level. While adding the acid, the bottle lid should be held over the bottle to condense any water vapor which may be coming off.

(e) The bottle shall be capped and allowed to come to room temperature.

(f) The bottle shall be re-weighed to obtain the sample weight and the weight recorded.

(g) The sample shall be transferred from the weighing bottle into a 250-ml beaker and then the weighing bottle shall be carefully rinsed into the beaker.

(h) Each sample shall be buffered to ph 7 with a 5 percent

solution of potassium hydroxide.

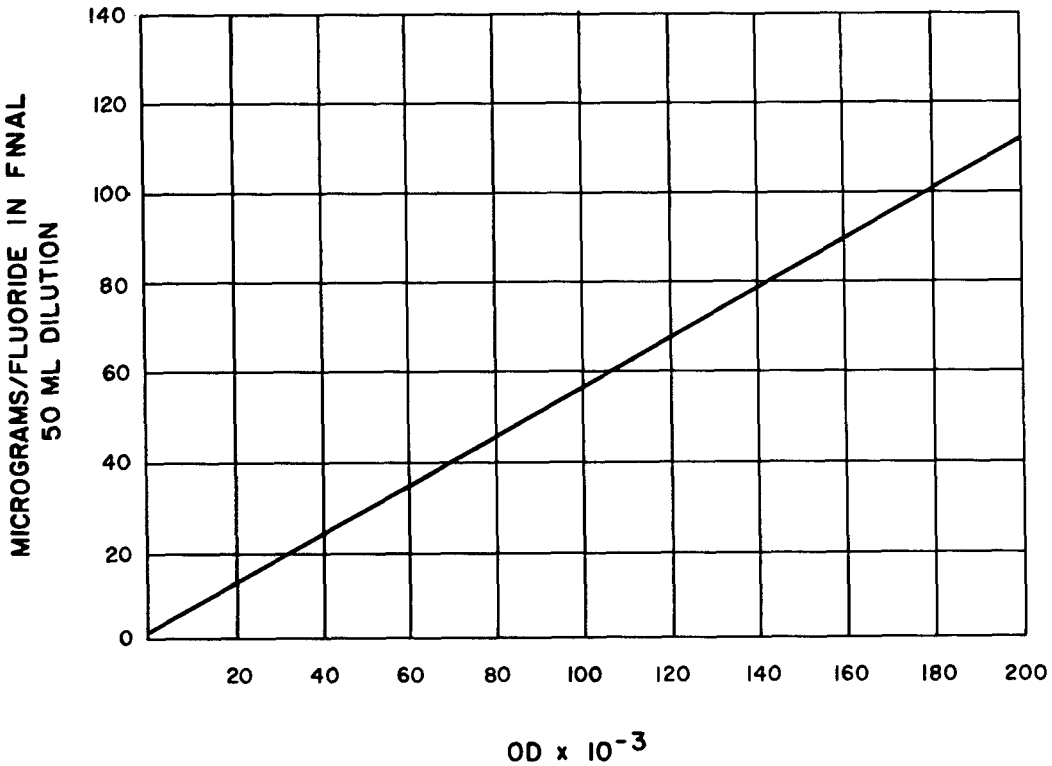

FIGURE 2 TYPICAL CALIBRATION CURVE

(i) Each buffered sample shall be transferred to a 250-ml volumetric flask and each beaker carefully rinsed.

(j) Each flask shall be filled with distilled water to the 250-ml mark and each flask shaken thoroughly.

(k) 2 ml shall be pipetted from each 250-ml volumetric flask into separate 50-ml volumetric flasks.

(l) 2 ml of the standard fluoride stock solution shall be pipetted into a 50-ml volumetric flask and labeled "standard"

(m) 5 ml of the bleach stock solution shall be pipetted into a 50-ml volumetric flask and labeled "bleach".

(n) The following adjustments shall be made on the spectrophotometer after at least a 10-minute warm up period:

(1) The cell-shifting control set to the out position
(2) The shutter closed
(3) The selector switch set to one
(4) The wavelength set to 530 millimicrons
(5) The slit width set to 0.02 mm.
(6) The phototube selector turned to the blue tube.

(o) When 15 minutes of the 90-minute waiting period remain, the four, matched, 1-cm, liquid absorption cells shall

be filled as discussed in paragraph 6.2.12b, 1.

(p) The absorbance of the standards in cells 3 and 4 shall be determined.

NOTE: Duplicate determinations on the same cell shall agree within 0.002 absorbance units.

(q) The standards in cells 3 and 4 shall be replaced with the first two samples.

(r) The absorbance of the samples shall be determined.

NOTE: Duplicate determinations on the same cell should agree within 0.002 absorbance units.

(s) The difference in absorbance between the blank solution and the other solutions (samples and standards) shall be determined.

(t) The micrograms of fluoride present in each sample and standard run shall be determined from the calibration curve previously prepared.

6.2.13 Determination of HNO_3 (nitric Acid) in RFNA

NOTE: The percent of nitric acid in RFNA is determined by difference. Knowing the percent of HF (hydrogen Fluoride), NO_2 (nitrogen dioxide), and the total acidity in RFNA, the amount of HNO_3 (nitric acid) can be determined.

a. The percent nitrogen dioxide shall be determined as in paragraph 6.2.10.

b. The percent total acidity shall be determined as in paragraph 6.2.11.

c. The percent hydrogen fluoride shall be determined as in paragraph 6.2.12.

6.2.14 Determination of Water (H_2O) in RFNA

NOTE: The percent of water in RFNA is determined by difference. Knowing the percent of HNO_3 (nitric acid), NO_2 (nitrogen dioxide), and HF (hydrogen fluoride), the amount of water can be determined by difference.

a. The percent nitrogen dioxide shall be determined as in paragraph 6.2.10.

b. The percent hydrogen fluoride shall be determined as in paragraph 6.2.12.

c. The percent nitric acid shall be determined as in paragraph 6.2.13.

6.2.15 Determination of Total Solids in RFNA

NOTE: This method is based upon the fact that the more volatile components of this acid mixture are evaporated at a lower temperature than is required to vaporize the solid particles

present.

a. Each sample shall be run in duplicate.

b. While holding the platinum crucibles with platinum tipped tongs, the crucibles shall be heated to constant weight using a meeker burner.

CAUTION

Care must be exercised in heating the platinum crucibles so as not to hold them in the reducing part of the flame. Use platinum tipped tongs to handle the crucibles at all times.

c. The crucibles shall be cooled momentarily and then placed into a dessicator for additional cooling.

d. The crucibles shall be weighed on an analytical balance to the nearest tenth of a milligram and the weight recorded.

e. 10 ml of the sample shall be transferred to each crucible.

f. The crucibles containing the acid shall be placed on the top of an aluminum foil covered hot plate under a hood.

g. The acid shall be allowed to evaporate until dry, while ensuring that the acid does not spatter or boil too vigorously.

h. The crucibles with their dried contents shall be heated to a constant weight using a meeker burner.

i. The crucibles shall be cooled momentarily and placed in a dessicator for additional cooling.

j. When the crucibles are cooled to room temperature, they shall be weighed on an analytical balance to the nearest tenth of a milligram and the weight recorded.

6.2.16 Quantitative Determination of TNT, NC and RDX in Explosives Using Extraction Techniques and IR Spectroscopy

NOTE: This procedure is based on the difference of solubilities of components in organic solvents and on IR absorption. The procedure is further based on the following facts:
TNT is soluble in chloroform
NC is soluble in ethanol
RDX is soluble in acetone
TNT has an IR absorption band near 10.6 microns
NC has an IR absorption band near 11.9 microns
RDX has an IR absorption band near 9.0 microns

a. Extraction

1) The unknown sample shall be reduced to a fine powder.
2) A clean soxhlet extractor thimble shall be weighed and recorded and then approximately 1 g of the powdered sample shall be placed in the thimble and the thimble weighed again.
3) The weight of the sample shall be determined.
4) A soxhlet extractor and condenser shall be assembled on a ring stand.
5) To extract TNT from the sample, approximately 250 ml of chloroform shall be placed in a clean 500-ml boiling flask

with 24/40 ground glass joints and several boiling stones shall be added.

6) The boiling flask shall be assembled on the soxhlet extractor and cooling water turned into the condenser.
7) A heating mantle shall be placed around the boiling flask and the variac adjusted until several drops per second are dropping from the condenser onto the sample.
8) The extraction should continue for at least four hours or overnight.
9) The thimble shall be removed from the extractor and dried until a constant weight is obtained.
10) The weight shall be recorded.
11) The boiling flask shall be removed from the soxhlet extractor and attached to a straight water cooled condenser.
12) The chloroform shall be distilled, the distillate collected until 10 to 15 ml of chloroform and extract remain in the boiling flask.
13) The distillate shall be labled as "recovered" chloroform.
14) A tared 50 ml volumetric flask having a ground glass stopper shall be weighed and the weight shall be recorded.
15) The contents of the boiling flask shall be carefully poured into a tared 50-ml volumetric flask having a ground glass stopper.
16) The boiling flask shall be rinsed several times with 5-ml aliquots of the recovered chloroform, then the volume of the sample and chloroform shall be brought to the 50-ml mark on the volumetric stem by adding recovered chloroform.
17) The 50-ml flask shall be weighed, the weight recorded and the contents mixed by tipping the flask upside down several times.
18) This procedure shall be repeated for making an NC extraction using ethanol in place of chloroform.
19) This procedure shall be repeated for making an NC extraction using acetone.

NOTE: If the explosive sample is composed only of TNT, NC, and RDX, there should not be any sample remaining after the third extraction. However, other explosive ingredients may be present in the extracted TNT, NC, and RDX mixtures. For this reason, the amounts of TNT, NC, and RDX shall be determined using an IR spectrophotometer.

b. Recording IR Absorption Data

1) General Set Up

(a) The double-beam IR spectrophotometer (similar to Perkins-Elmer Model 21) shall be placed in operating condition.
(b) A 0.1-mm spacer shall be placed between the sealed NaCl windows and the path length shall be accurately determined.
(c) The spectrophotometer control devices shall be set to the values listed in Table III for the various determinations.

2) To record the IR absorption data for NC:

(a) The scanning system shall be set to 10 microns on the wavelength counter.
(b) The IR sealed cell shall be filled with ethanol and the cell placed in the sample beam holder.
(c) The variable path cell shall be filled with ethanol and the micrometer indication adjusted to 100 microns.
(d) The variable path-length cell shall be placed in the compensation beam holder and the cell adjusted until the ethanol is compensated.

Table III. Control Device Settings for the Double-Beam IR Spectrophotometer (Perkins-Elmer Model 21)

CONTROL DEVICE	SETTINGS		
	NC	TNT	RDX
Test Signal	Off	Off	Off
Gain Control	5-6 (normal)	5-6 (normal)	5-6 (normal)
Response Control	1	1	1
Auto Suppression	Off	Off	Off
Pen Switch	Off	Off	Off
Filter Control	"in"	"in"	"out"
Manual-Program	Manual	Manual	Manual
Slit Width (microns)	250	150	125
2X-1X Switch	1X	1X	1X
Pen Speed	11	11	11
Expansion Control	1X	1X	1X
Amplifier Balance Control No Pen Drift with Both Beam Shutters Closed			

(e) The ethanol in the sample cell shall be replaced with the sample NC extraction.
(f) A 10-to 13-micron wavelength scan at a slit width of 25 microns shall be recorded.
(g) Baselines for all the recorded scans shall be constructed by drawing the best straight line through the minimum absorption points for each scan and the absorption value for each scan shall be recorded.
(h) The true path length shall be recorded.

3) To record the IR absorption data for TNT:

(a) The scanning system shall be set to 9.5 microns on the wavelength counter.
(b) Steps (b) through (e) of paragraph 6.3.16.b.2 shall be repeated using chloroform in place of ethanol.
(c) A 9.5- to 11.5-micron wavelength scan at a slit width of 150 microns shall be recorded.
(d) Steps (g) and (h) of paragraph 6.2.16.b.2, shall be repeated.

4) To record the IR absorption data for RDX:

(a) The scanning system shall be set to 9.0 microns on the wavelength counter.
(b) Steps (b) through (e) of paragraph 6.2.16.b.2 shall be repeated using aceton in place of ethanol.
(c) A 9- to 12-micron wavelength scan at a slit width of 125 microns shall be recorded.
(d) Steps (g) and (h) of paragraph 6.2.16.b.2 shall be repeated.

NOTE: Consult Reference E of this MTP for further information pertaining to the details of TNT, NC, and RDX extraction and recording IR absorption data.

6.2.17 Quantitative Analysis of the Alloying Elements in Steels Using the NSL Emission Spectrograph

NOTE: The analysis of alloying elements in steels may be determined over the concentration range indicated in Table IV.

Table IV. Concentration Range for Analysis of Alloying Elements in Steels

ELEMENT	CONCENTRATION RANGE (percent)
Boron (B)	0.002 - 0.01
Chromium (Cr)	0.3 - 1.1
	2.5 - 25.0 (Stainless Steel)
Manganese (Mn)	0.3 - 1.5
Molybdenum (Mo)	0.2 - 1.0
Nickel (Ni)	0.1 - 1.0; 0.5 - 5.0
Silicon (Si)	0.1 - 0.3
	0.3 - 1.2
Vanadium (V)	0.005 - 0.15

The emulsion calibration equation for Eastman S A I plates is:

$$\text{Log } I = 2.15147585 - 3.88563838 \times 10^{-2} (Tr) + 1.29888039 \times 10^{-3} (Tr)^2 - 2.70118813 \times 10^{-5} (Tr)^3 + 2.78016109 \times 10^{-7} (Tr)^4 - 1.13007283 \times 10^{-9} (Tr)^5$$

This equation was evaluated from a plate made while rotating the log step sector in front of the slit and is to be used for the evaluation of log I for lines from 2200A to 3300A. All samples analyzed during this test must have a flat surface. Polish the surface of each sample on a belt sander to ensure that it is clean and flat.

a. Procedures

1) The excitation sequence shown in Table V shall be employed for the analysis of up to six unknowns.

Table V. Excitation Sequence for Analysis of up to Six Unknowns

Spectra No.	Sample
1	Carbon
2	Standard
3	Unknown
4	Unknown
5	Unknown
6	Standard
7	Unknown
8	Unknown
9	Unknown
10	Standard

2) The following conditions shall be set on the grating control panel of the emission spectrograph instrument:

(a) Separation set to 0
(b) Light set for grating #1 to 100 percent and #2 to 0.
(c) Separation of spectra set to 6 mm.
(d) Movement of plate set to down and automatic.
(e) Wavelength range for grating #1 set to the value listed in Table VI for the element being determined.

3) The spectrograph slit controls shall be set as follows:

(a) Slit width set to 20 microns.
(b) Filter #2 (7%/100%/33% transmission) placed into position.
(c) Slit height set to 6.5 mm.

4) Spectrograph excitation unit shall be set to the following conditions:

   (a) Capacitance and inductance switches set to "short".
   (b) Selector switch set to the type of excitation listed in Table VI for the element being determined.
   (c) Spark power rotated to its counterclockwise limit.
   (d) Arc power rotated to position #10.
   (e) The prespark and total timers set to the values listed in Table VI for the element being determined.

5) A carbon electrode shall be placed in each holder and the air flow regulator turned on so that a reading of three pounds is obtained.
6) The spectrograph shall be started and the spark power turned clockwise until the number of discharges established for the method listed in Table VI is obtained on the oscilloscope.
7) The spectrograph shall be stopped.
8) The plate position shall be set to 6 on the plate position scale.
9) The plate holder back cover shall be pulled up on until it has reached the upper limit of travel as indicated by a metallic "click" sound.
10) The unit shall be started and allowed to run the programmed cycle.
11) The appropriate National Bureau of Standards (NBS) spectrographic steel standard, listed in Table VI, shall be placed in the lower electrode clamp and a 3/16-inch O.D. electrode shall be placed point down in the upper clamp.
12) The unit shall be started and, during the prespark time, the electrodes shall be adjusted to the red lines on the burner housing.

NOTE: This adjusts the electrode gas to 3 mm. Do not change the adjustment during the exposure.

13) Steps 11 and 12 shall be repeated using the unknown sample in place of the standard. No more than three unknowns shall be run in series.
14) Steps 11 and 12 shall be repeated using the standard.
15) Steps 11 through 14 shall be repeated to analyze up to nine unknowns.
16) Upon completion of excitation the master switch (excitation unit) shall be turned to off, the lamp on the burner housing turned to off, and the air turned to off.
17) The plate holder back cover shall be pushed down on until it is in a closed position.
18) The plate holder shall be removed and taken to the darkroom.
19) The plate shall be developed and recorded as data.

6.2.18 Quantitative Analysis by Gas Chromatography Employing the "Marker" Method

NOTE: Gas chromatography makes possible both a qualitative and

Table VI. Instrument Parameters

Element	NBS Std	Wavelength Range, A	Excitation Selector Switch	Prespark Timer	Total Timer	Discharges per Cycle
B	464	2100-3100	Uni-arc	10	15	2
Cr (Low)	421	2300-3300	Spark	10	21	10
Cr (High)	442	2000-3000	Spark	10	15	10
Mn	412	2300-3300	Spark	10	21	10
Mo	446	2300-3300	Spark	10	18	10
Ni (Low & High)	412	2400-3400	Spark	10	30	10
Si (Low)	417	2300-3300	Spark	10	24	10
Si (High)	404A	2300-3300	Spark	10	21	10
V	444	2300-3300	Spark	10	21	10

quantitative analysis of volatile mixtures. In the internal normalization method of quantitative analysis, either the exact volume or weight of a sample must be known, or the areas of the peaks of all the components in the mixture must be known. If the mixture contains water, complications arise due to the "tailing off" of the water. The area under the water peak is difficult to accurately determine when it "tails off", especially if area determinations are made with a disc integrator. The "marker" method eliminates complications that arise from the "tailing off" of water and eliminates the need to know the weight or volume of the sample. The method consists of adding to the sample an accurately known amount of a pure substance not present in the sample. The pure substance must not give a chromatographic peak at the same time as a component of the sample. It is best that the pure substance have a peak that is bracketed by the peaks of the components of the sample, if possible. For accuracy, the height of the marker peak should not differ much from that of the component of interest. If the sample contains only two components, x and y, chromatograms of the marker and x are obtained from mixtures of x and the marker in several accurately known proportions. The proportions chosen should be close to the proportion that is to be used in the actual sample. The ratio of the areas of the peaks for the marker and for x are plotted against the ratio of the amounts actually present. This generly results in a straight line. In the actual analysis, a known amount of marker is added to the sample and a chromatogram obtained. The ratio of the area under the marker peak to the area under the x peak is determined and the amount of x can then readily be determined from the plot of knowns. The same method is repeated for the determination of the amount of y. Operating conditions of the gas chromatograph must remain the same during the entire analysis. Areas of peaks may be determined by means of a planimeter or by an integrating unit attached to a strip-chart recorder which automatically measures the areas.

6.2.18.1 Analysis of Redstone Alcohol by the "Marker" Method

The accuracy of the "marker" in the analysis of Redstone alcohol is as follows:

	Absolute Deviation from True Value
MeOH, Methyl alcohol	± 0.2
EtOH, Ethyl alcohol	± 0.7
H_2O, Water	± 0.9

Note: $CHCl_3$ (chloroform) may be used as the marker during this analysis.

a. The following conditions shall be set on the gas chromatograph:

1) Pressure to 20 psig on the instrument and 30 psig on the tank gauge.
2) Temperature to 100° C.

3) Current to 300 ma.
4) Power to on for at least four hours prior to the test.

b. Known solutions of ethyl alcohol, methyl alcohol and water shall be prepared as to bracket the required concentrations.

c. One gram of each solution shall be accurately weighed to four decimal places, the weight recorded, and the weight percent of each constituent recorded.

d. To each of the above weighed solutions one gram of chloroform, which has been weighed and whose weight shall be recorded, shall be added.

e. Each solution shall be thoroughly mixed by shaking for about ten seconds.

f. 0.005 ml of one of the chloroform, alcohol, water mixtures shall be injected into the sample inlet of the gas chromatograph.

NOTE: When the chloroform peak forms on the chart paper, the run can be considered complete, as there is no need to obtain the water peak. Figure 3 illustrates methyl and ethyl alcohol, chloroform, and water peaks.

g. Step f shall be repeated for each of the standards and the chart paper shall be noted to identify which sample was run.

h. One gram of Redstone alcohol shall be accurately weighed and the weight recorded.

i. One gram of chloroform (ACS grade) shall be accurately weighed, the weight recorded and then added to the weighed Redstone alcohol.

j. With the gas chromatograph instrument settings the same as previously discussed, 0.005 ml of the mixture shall be inserted into the sample inlet.

NOTE: When the chloroform peak forms on the chart paper, the run can be considered complete, as there is no need to obtain the water peak.

6.2.19 An IR Spectroscopic Procedure for Determining the Percentage of SF_6 (Sulfur Hexafluoride) in Gas Samples

NOTE: This procedure is based upon the absorption band of sulfur hexafluoride at approximately 7.2 microns.
NOTE: The transfer of a gas sample to the IR gas absorption cell is discussed in detail in paragraph 6.2.20.

a. The double-beam IR spectrophotometer (similar to Perkins-Elmer Model 21) shall be placed in operating condition.

b. The spectrophotometer control devices shall be set to the values listed in Table VII.

c. Chart paper shall be placed on the recorder drum.

d. The zero and the 100 percent transmittance readings of the pen shall be adjusted to the zero of the chart paper.

e. The scanning system (wavelength counter) shall be set to 6.5 microns wavelength.

f. The compensation shutter and the sample-beam shutter shall be opened and the 6.5-micron through 8.0-micron wavelengths shall be recorded.

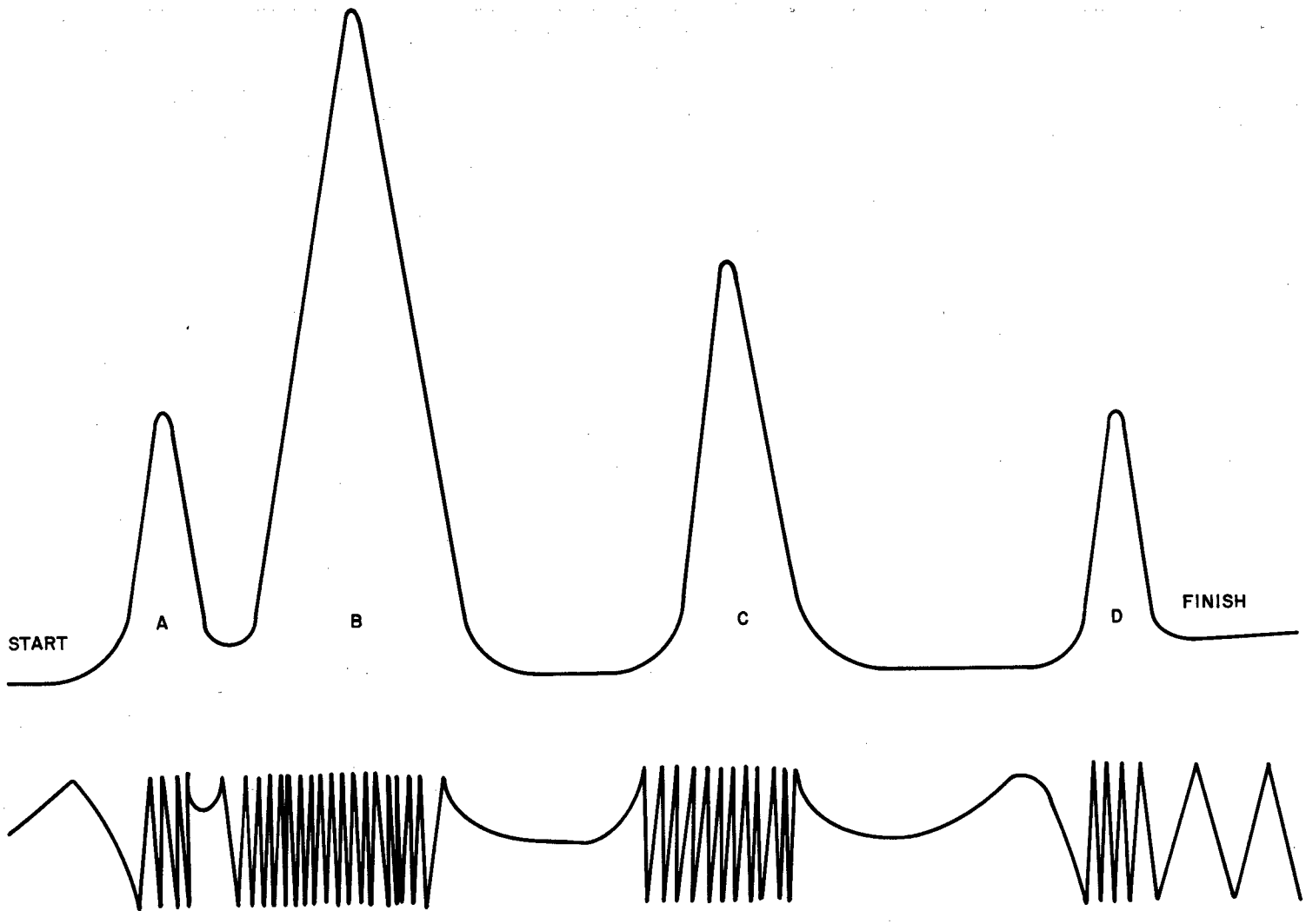

FIGURE 3. COMPARISON OF METHYL ALCOHOL, ETHYL ALCOHOL, CHLOROFORM, AND WATER PEAKS

g. The sample bottle shall be removed from its connection and replaced with a fresh sample bottle as described in paragraph 6.2.20b.

h. A sample of pure (99 percent) sulfur hexafluoride shall be transferred into the cell and the 6.5-micron through 8.0-micron wavelengths recorded.

Table VII. Control Device Settings for the Double-Beam IR Spectrophotometer (Perkins-Elmer Model 21) for Analyses of SF_6

CONTROL DEVICE	SETTING
Test Signal	Off
Gain Control	5-6 (normal)
Response Control	1
Auto Suppression	Off
Pen Switch	Off
Filter Control	"Auto" or "Out"
Manual - Program	Manual
Resolution Control	33 (65-micron reading on the slit counter)
2X - 1X Switch	1X
Pen Speed	11.5
Expansion Control	1X
Amplifier Balance Control	No Pen Drift with Both Shutters Closed

6.2.20 An IR Spectroscopy Procedure for Determining the CO (Carbon Monoxide) and CO_2 (Carbon Dioxide Content of Air Samples (Missile Exhaust))

NOTE: This procedure may be used to determine the concentration (percent by volume) of carbon monoxide and carbon dioxide in air at 650 mm Hg and 20° C over a concentration range of approximately 0.003 to 100 percent and 0.01 to 60 percent, respectively for each gas. The equations relating concentration to absorbance may not be transferred and must be established independently for each gas. The standard error estimate (SEE) for a carbon monoxide determination is 0.003 percent and the standard deviation for carbon dioxide determination is 0.008 percent. This procedure can be adapted for determining concentrations of other gas mixtures in various

path length IR absorption cells. This procedure is based upon the fact that carbon monoxide has an absorption band at a wavelength near 4.65 microns and that carbon dioxide has an absorption band at a wavelength near 4.30 microns.

a. Cell Filling

1) To transfer a gas sample to the IR gas absorption cell, an apparatus similar to that illustrated in Figure 4 shall be assembled.
2) A small amount of Apiczon N or L vacuum grease shall be applied to all vacuum hose connections and stopcocks.
3) A balloon neck shall be placed around a rubber stopper for the aspirator bottle and the balloon placed in the bottle.
4) The stopper shall be pushed tightly down into the aspirator bottle neck.
5) Stopcocks A, C, D, and E shall be checked to ensure that they are closed, the vacuum pump shall be started, and stopcock D shall be slowly opened.
6) If there is a gurgling noise in the pump after a minute of pumping, there may be an air leak in the hoses, connections, or in the gas cell. The stopcocks and the gas cell window seals shall then be checked.
7) If the pump gurgling continues, the hosing shall be checked for leaks.
8) Valve B from the gas cell shall be closed, opened stopcock C, and any leaks as indicated by gurgling in the vacuum pump shall be noted.
9) A leak may be determined by pumping down a section of the system, such as the gas cell, then closing off that section from the system a few minutes, opening the section into the vacuum, and listening for air being pumped out.
10) The three-way stopcock B shall be positioned so that the balloon and the tubing between stopcock B and Valve A on the sample bottle are evacuated.
11) Leaks shall be checked for as previously discussed.
12) Stopcock D shall be closed and the closed valve A momentarily opened.
13) Stopcock B shall be set closed to the gas cell and opened between the balloon and the sample bottle.
14) Valve A on the sample bottle shall be opened and stopcock E shall be slowly opened to begin evacuation of the aspirator bottle (the balloon should begin expanding) drawing in the gas from the sample bottle.
15) Stopcock E shall be closed to stop the evacuation of the aspirator bottle when the balloon has expanded to the capacity of the aspirator bottle.
16) Valve A shall be closed and the stopcock B shall be set open to the gas cell and balloon and closed to Valve A.
17) Stopcock C shall be opened and stopcock E set so that the aspirator bottle is open to the atmosphere but closed to the vacuum pump.
18) The balloon should collapse, as gas is forced into the gas cell.

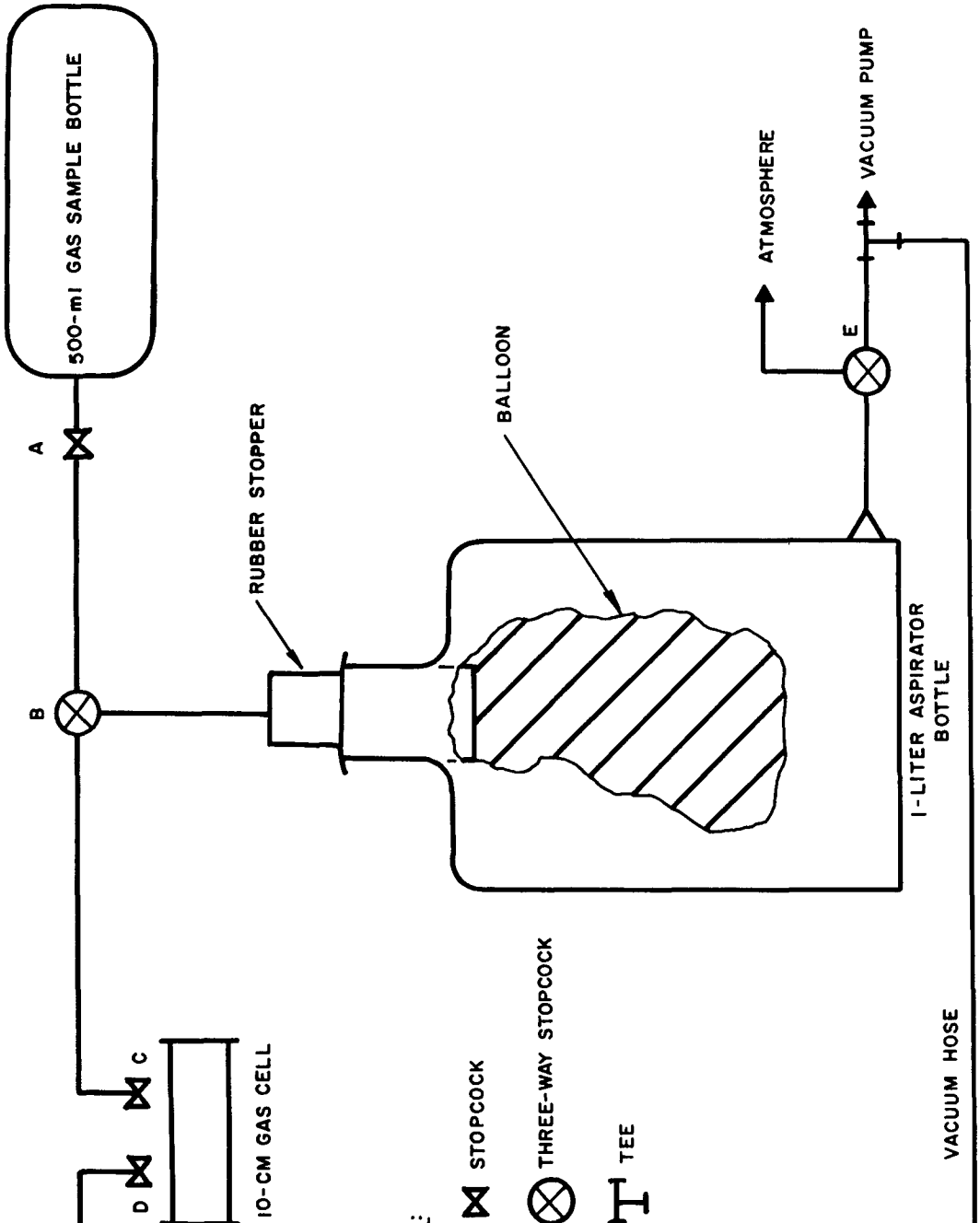

FIGURE 4. APPARATUS USED FOR TRANSFERRING GAS SAMPLE TO AN IR GAS ABSORPTION CELL.

19) Stopcock C shall be closed after the balloon has collapsed and this procedure repeated if the gas sample bottle contains less than 50 cc of gas or if the gas was originally under a partial vacuum.

NOTE: It is important that the gas cell be filled with gas sample to a pressure near ambient pressure (650-660 mm Hg.) If there are not 500 cc of gas available to fill the 10-cm path gas cell, use a 5-cm path cell which will require about 200 cc of gas. The carbon monoxide and dioxide absorption values observed in the 5-mm cell (1/2-path length) will need to be doubled to find the percent using the equations described in paragraph 6.4.21.

20) The room temperature and barometric pressures at which this test is conducted shall be recorded.

b. Recording the Carbon Monoxide and Carbon Dioxide IR Absorption Bands.

1) The double-beam IR spectrophotometer (similar to Perkins-Elmer Model 21) shall be placed in operating condition.
2) The spectrophotometer control devices shall be set to the values listed in Table VIII.

Table VIII. Control Device Settings for the Double-Beam IR Spectrophotometer (Perkins-Elmer Model 21) to Obtain Quantitative Data for CO and CO_2 Gas

CONTROL DEVICE	SETTING
Test Signal	Off
Gain Control	5-6 (normal)
Response Control	1
Auto Suppression	Off
Pen Switch	Off
Filter Control	"Auto" or "Out"
Manual - Program	Manual
Resolution Control	51 (100-micron reading on Slit Counter)
2X - 1X Switch	1X
Pen Speed	11
Expansion Control	1X
Amplifier Balance Control	No Pen Drift With Both Shutters Closed

3) Chart paper shall be placed on the recorder drum. The zero and the 100 percent transmittance readings of the pen shall be adjusted to the zero of the chart paper.
4) The scanning system (wavelength counter) shall be set to 4.0 microns wavelength.
5) The compensation shutter and the sample beam shutter shall be opened.
6) The expansion control shall be switched to 10X or 20X.
7) The pen shall be brought on scale by first turning the 100 percent adjust control to the limit clockwise and then turning the pen position control clockwise until the pen is reading approximately at the 50 percent transmittance reading of the chart.
8) The 4.0-micron through 5.0-micron wavelengths shall be recorded.
9) The expansion control shall be switched to 1X.
10) The sample bottle shall be removed from its connection and replaced with a fresh sample bottle.
11) The gas transfer system shall be disconnected at the ball joint connection and the vacuum hose disconnected from stopcock D.
12) Stopcocks D and C shall be opened and a small purge of nitrogen or other nonabsorbing gas containing no detectable carbon monoxide or dioxide passed through stopcock D and out through stopcock C of the gas cell.
13) This procedure shall be repeated to record the absorbance of the blank.

6.2.21 An IR Spectrophotometric Method for the Analysis of DOA (Dioctyl Adipate) in a Mixture with Ammonium Perchlorate, Aluminum, and Polyvinyl Chloride Using a KBr (Potassium Bromide) Pellet Technique

NOTE: This test is conducted to determine the percent by weight concentration of DOA. The procedure also may be adopted for determining component concentration of other organic and inorganic constituents of propellants. The following absorption law relationships are followed in the potassium bromide pellet analysis technique:

(a) The necessity for determining the sample thickness is eliminated by incorporating an internal standard Na N_3 (sodium azide) in the potassium bromide.
(b) The absorbance of a material to be analyzed at a wavelength L_1 is given by A_{L1} bc', and the absorbance of the internal standard at wavelength L_2 by $A_{L2} = a_{L2}$ bc".
(c) Since both absorbances are measured on the same potassium bromide pellet, dividing the equation gives:

$$\frac{A_{L1}}{A_{L2}} = \frac{a_{L1}\ bc'}{a_{L2}\ bc''}$$

The b's cancel and since a_{L1} and a_{L2} are constants at the wavelength at which the measurements are made and c", the concentration of the internal standards, is constant,

these constants can be combined to form a single constant represented to K:

$$\frac{A_{L1}}{A_{L2}} = Kc'$$

(d) A plot of $\frac{A_{L1}}{A_{L2}}$ even with regular type versus c' (concentration of the unknown) will give a straight line in accordance with "Beer's" law.

This procedure is based on the fact that DOA has a strong absorption band near 4.75 microns.

6.2.21.1 Test Preparation

a. The following reagents are used during this test to determine concentrations of DOA and shall be procured before testing:

1) MEK Methyl Ethyl Keton, (Reagent grade)
2) KBr Potassium Bromide, (ACS grade)
3) NH_4 ClO_3 Ammonium Perchlorate, (ACS grade)

b. Preparation of Synthetic Propellant

1) Sufficient DOA, ammonium perchlorate, and potassium bromide shall be weighed on a microbalance to the nearest 0.002 mg. to make a one g sample whose DOA and ammonium perchlorate composition approximates the composition of the propellant to be analyzed.

NOTE: The resulting mixture is referred to as synthetic propellant.

2) The synthetic propellant shall be moistened with methyl ethyl ketone, transferred to a mortar, and the synthetic propellant ground and mixed until uniform.
3) This propellant shall be placed into an evacuatable desiccator to remove all traces of methyl ethyl ketone.

c. Preparation of Potassium Bromide/Sodium Azide Mixture.

1) Approximately 20 g of this mixture containing about 0.13 percent sodium azide shall be prepared by weighing sufficient azide and adding it to powdered potassium bromide.
2) This mixture shall be thoroughly mixed by grinding until uniform.
3) The uniformity shall be checked by preparing three pellets from the mixture.
4) The absorbance of the sodium azide peak at 4.75 microns shall be determined.
5) The pellets shall be weighed on an analytical balance and the absorbance of the azide peak shall be divided by the weight of the pellet.

NOTE: This ratio must be nearly equal for the three pellets or

a new mixture shall be prepared.

d. Preparation of a synthetic Propellant Mixture

1) One g of a synthetic propellant mixture shall be prepared by weighting approximately 0.005 g of the synthetic propellant on a micro balance and adding 0.995 g of the potassium bromide/sodium azide mixture

NOTE: All weighings should be made to the nearest microgram.

2) All weights shall be recorded.
3) The synthetic propellant mixture shall be transferred to a "wig-l-bug" vial and ground on the "wig-l-bug" for about two minutes.
4) A pellet shall be made from this mixture.

e. Preparation of a Propellant Mixture.

1) A one-g mixture of the missile propellant to be analyzed shall be prepared by weighing approximately 0.005 g of powdered (ground) propellant on a micro balance and adding 0.995 g of potassium promide/sodium azide mixture.
2) All weights shall be recorded.
3) The propellant mixture shall be transferred to a "wig-l-bug" vial and ground on the "wig-l-bug" for about two minutes.
4) A pellet shall be made from this mixture.

6.2.21.2 Recording IR Absorption Data for DOA

Perform the following:

a. The double-beam IR spectrophotometer (similar to Perkins-Elmer Model 21) shall be placed in operating condition.

b. The spectrophotometer control devices shall be set to the values listed in Table IX

c. Chart paper shall be placed on the recorded drum and the zero and 100 percent transmittance readings of the pen adjusted to the zero of the chart paper.

d. The scanning system shall be set to 4.0 microns on the wavelength counter.

e. The pellet made from the synthetic propellant mixture, paragraph 6.3.21b shall be placed into the KBr pellet holder and the holder inserted into the IR sample beam.

f. The compensation beam shutter shall be adjusted so that the pen records at 85 percent transmittance.

g. The resolution control shall be adjusted so that a slit of 75 microns is obtained on the slit counter.

h. The spectra shall be recorded from 4.0- to 5.5-micron wavelengths.

i. The NaN peak should not occur below 20 percent transmittance and if it does, the pellet shall be remade using less material.

j. At the 5.5-micron wavelength, the slit width shall be adjusted to 100 microns and the DOA peak recorded from 5.5 to 6.1 microns.

Table IX. Control Device Settings for the Double-Beam IR Spectrophotometer (Perkins-Elmer Model 21) for Analysis of DOA

Control Device	Setting
Test Signal	Off
Gain Control	5-6 (Normal)
Response Control	1
Auto Suppression	Off
Pen Switch	Off
Filter Control	"Auto"
Manual-Program	Manual
Resolution Control	NaN -39 (75-micron reading on slit counter) Dioctyl adipate peak -51 (100-micron reading on slit control)
2X-1X Switch	1X
Pen Speed	11.5
Expansion Control	1X
Amplifier Balance Control	No pen drift with both beam shutters closed.

k. The pellet holder shall be removed from the sample beam and the pellet rotated 45° from its original position with a pair of tweezers.

l. The holder shall be reinserted into the sample beam and the recording procedure repeated.

m. The aforementioned procedure shall be repeated until the pellet has been rotated 180° or four times.

n. This procedure shall be repeated using the pellet made from the propellant mixture prepared in paragraph 6.2.21.le.

6.2.22 An IR Spectroscopic Method for Determining the Water Content of Various Organic Solvents

NOTE: This procedure describes a method for determining the water content of benzene, carbon tetrachloride, and toluene. The method is general and can be adopted to other solvents. The procedure is based on the fact that water has a strong absorption peak at 2.75 microns.

a. Determining Water in Benzene.

1) The double-beam IR spectrophotometer (similar to Perkins-Elmer Model 21) shall be placed in operating condition.

2) The spectrophotometer control devices shall be set to the values listed in Table X.
3) The chart paper shall be placed on the recorder drum and the zero and 100 percent transmittance readings of the pen adjusted to the zero of the chart paper.
4) Two fixed-path length cells shall be filled with dried benzene and one cell placed in the compensation beam and the other in the sample beam.
5) The scanning system shall be set to 2.4 microns wavelength and the pen adjusted to read 50 percent on the chart paper with the expansion control set at 10X.
6) Several scans shall be recorded from 2.4 to 3.0 microns wavelength.
7) The dried benzene in the sample cell shall be replaced with the sample to be analyzed.
8) The zero setting shall be rechecked and the pen reading adjusted at 2.4 microns to 85 to 90 percent transmittance on the chart paper.
9) The 2.4- to 3.0-micron wavelength shall be scanned with the expansion control positioned at 1X.
10) The scan shall be repeated at greater expansions if the peak at 2.75 microns can be retained on the chart paper.

Table X. Control Device Settings for the Double Beam-IR Spectrophotometer (Perkins-Elmer Model 21) for Analysis of Water in Benzene.

Control Device	Setting
Test Signal	Off
Gain Control	5-6 (Normal)
Response Control	1
Auto Suppression	Off
Pen Switch	Off
Filter Control	Auto
Manual-Program	Manual
Resolution Control	126 (250-micron slit opening)
2X - 1X Switch	1X
Pen Speed	11
Expansion Control	1X
Amplifier Balance Control	No pen drift with both shutters closed.

b. Determining Water in Carbon Tetrachloride.

1) The control device settings for the spectrophotometer shall be the same as shown in Table X with the exception that the resolution control shall be at 30 (56-micron slit opening).
2) The chart paper shall be placed on the recorded drum and the zero and 100 percent transmittance readings of the pen adjusted to the zero of the chart paper.
3) A teflon 2-inch path length cell shall be filled with dry carbon tetrachloride and the cell placed in the sample beam.
4) The scanning system shall be set to 2.4 microns wavelength. and the pen adjusted to read 85 to 90 percent transmittance with the expansion contro positioned at 1X.
5) Several scans from 2.4 to 3.0 microns wavelength shall be recorded.
6) The dry carbon tetrachloride in the sample cell shall be replaced with the sample to be analyzed.
7) The zero setting shall be rechecked and the pen reading adjusted at 2.4 microns to 85 to 90 percent transmittance on the chart.
8) The 2.4- to 3.0-micron wavelength shall be scanned with the expansion control set at 1X.

c. Determining Water in Toluene.

1) The control device settings for the spectrophotometer shall be the same as shown in Table X with the exception that the resolution control shall be set at 85 (175-micron slit opening) and the pen speed shall be set at 11.5.
2) The chart paper shall be placed on the recorder drum and the zero and 100 percent transmittance readings of the chart paper.
3) Two fixed-path length cells (5-mm spaces) shall be filled with dried toluene and one of the cells placed in the compensation beam and the other in the sample beam.
4) The scanning system shall be set to 2.4 microns wavelength and the pen adjusted to read 85 percent transmittance on the chart paper with the expansion control set at 1X.
5) Several scans shall be recorded from the 2.4- to 3.0-micron wavelength.
6) The dried toluene in the sample cell shall be replaced with the sample to be analyzed.
7) The zero setting shall be rechecked and the pen reading at 2.4 microns adjusted to 85 percent transmittance on the chart with the expansion control set at 1X.
8) The 2.4- to 3.0-micron wavelength shall be scanned with the expansion control set at 1X.
9) The scan shall be repeated at greater expansions if the peak at 2.75 microns wavelength can be retained on the chart paper.

6.3 TEST DATA

6.3.1 Preparation for Test

None

6.3.2 Determination of Resin in Corporal Fuels

The following values shall be recorded:

a. Weight of empty crucible in grams.
b. Weight of resin plus crucible in grams.
c. Volume of sample in milliliters.
d. Density of sample in grams/milliliters.

6.3.3 Determination of Sn (Tin) in H_2O_2 (Hydrogen Peroxide)

The following values shall be recorded:

a. Diffusion current of unknown sample.
b. Diffusion current of known standard.

6.3.4 Analysis of Aniline Using the $HClO_4$ (Perchloric Acid) Method

a. Perchloric acid solution standardization

The following values shall be recorded:

1. Weight of $NA_2\ CO_3$ (Sodium Carbonate) in grams.
2. Assay of $NA_2\ CO_3$ (Sodium Carbonate).
3. Volume of $HClO_4$ (perchloric acid) required for titration in milliliters

b. Determination of unknown sample

The following values shall be recorded:

1. Weight in grams of eyedropper with fuel.
2. Weight in grams of eyedropper after fuel sample is removed.
3. Volume of $HClO_4$ (perchloric acid) required for titration in milliliters.
4. Volume of $HClO_4$ (perchloric acid) required for duplicate titration in millimeters.

NOTE: Steps 3 and 4 must agree to within 0.1 percent.

6.3.5 Analysis of H_2O_2 (Hydrogen Peroxide) by the Permanganate Method

a. Standardization of potassium permanganate solution

The following values shall be recorded:

1) Weight of beaker with $NA_2C_2O_4$ (sodium oxadate) in grams
2) Weight of empty beaker in grams
3) $NA_2C_2O_4$ (sodium oxadate) assay
4) Volume of $KMnO_4$ (potassium permanganate) required for titration in milliliters.

b. Determination of unknown sample

The following values shall be recorded:

1) The weight of empty weighing bottle in grams.
2) The weight of weighing bottle with H_2O_2 (hydrogen peroxide) in grams.
3) The volume of $KMnO_4$ (potassium permanganate) required for titration in milliliters.
4) The volume of $KMnO_4$ (potassium permanganate) required for duplicate titration in millimeters.

NOTE: Steps 3 and 4 must agree within 0.07 percent.

6.3.6 Analysis for Aniline by Gas Chromatography

Record the following values:

a. The ambient temperature in the area where the standard is being prepared in degrees C.
b. The average unit area counts for the reagent grade aniline.
c. The ambient temperature in the area where the test sample is being prepared in degrees C.
d. The density of the test sample in grams/milliliter.
e. The average unit area counts for the test sample.

6.3.7 Analysis of UDMH using the KIO_3 (Potassium Iodate) Method

The following values shall be recorded.

a. The molarity of the potassium iodate solution.
b. The weight of the sample shall be recorded in grams.
c. The volume of KIO_3 required for titration shall be recorded in milliliters.

6.3.8 Analysis of UDMH in JP-17 Fuel Using the Water Extraction Method

The following values shall be recorded:

a. The density of JP-17 in grams/milliliter.
b. The density of the UDMH used in preparing the JP-17 in grams/milliliter.
c. The assay of the UDMH used in preparing the JP-17 in percent.
d. The volume of JP-4 shall be recorded in milliliters.

6.3.9 Analysis of Redstone Alcohol Using Gas Chromatography

The chart recordings shall be recorded.

6.3.10 Analysis of Redstone Alcohol Using the Refractive Index Method

The following values shall be recorded:

a. The weight of the empty pycnometer in grams.
b. The weight of the pycnometer and water at 15.6°C in grams.

c. The weight of the pycnometer and alcohol mixture at 15.6° in grams.

d. The Refractive index scale for the alcohol mixture at 20°C.

6.3.11 Determination of NO_2 (Nitrogen Dioxide) in RFNA

a. Standardization of Ce $(H\ SO_4)_4$ (ceric Acid Sulfate)

1) Record the weight of the three samples of the primary standard arsenious oxide in grams.
2) Record the assay of the arsenious oxide.
3) Record the volume of Ceric acid sulfate required for each of the titrations of the three samples in mls.

b. Analysis of Nitrogen Dioxide in RFNA.

Record the following values:

1) Weight of empty ampule in grams.
2) Weight of ampule and sample in grams.
3) Volume of ferrous solution used in titration of the ceric acid sulfate and sample in ml.
4) Volume of ferrous solution used in titration of 35ml ceric acid sulfate in ml.

6.3.12 Determination of Total Acidity in RFNA

a. Record the normality of NaOH.
b. Record the sample weight in grams.
c. Record the volume of NaOH required for titration in ml.
d. Record the volume of NaOH required in the three blank determinations in mls.
e. Repeat steps b and c for duplicate runs.

6.3.13 Determination of HF (Hydrogen Fluoride) in RFNA

Record data in accord with sample data sheet shown in fig 5.

6.3.14 Determination of HNO_3 (Nitric Acid) in RFNA

a. Percent nitrogen dioxide

1) Standardization of Ce $(H\ SO_4)_4$ (Ceric Acid Sulfate)

6.3.14 Determination of HNO_3 (Nitric Acid) in RFNA

a. Percent nitrogen dioxide

1) Standardization of Ce $(H\ SO_4)_4$ (Ceric Acid Sulfate)

(a) Record the weight of the three samples of the primary standard arsenious oxide in grams.
(b) Record the assay of the arsenious oxide.
(c) Record the volume of Ceric acid sulfate required for

HF Analysis			Date 13 Oct 1961	Analyst BA
Sample Number	736	737		
Final Weight	43.3326	43.5650		
Initial Weight.	42.3209	42.4948		
Sample Weight	1.0117	1.0702		
Blank	.207 .206	.207 .206		
Sample	.136 .136	.138 .138		
Delta OD	.071 .070	.069 .068		
Delta OD Over.	.071	.069		
Microgram F Ion	41.5	40.4		
% HF Uncorrected	.540	.500		
Standard Correction	-.072	-.036		
% HF Corrected	0.478	0.464		

Cell Correction		
Cell Number	3	4
Blank	.207 .207	.207 .207
Std. HF	.131 .131	.136 .136
Delta OD	.076 .076	.071 .071
Microgram	44.5	41.8
Std. HF, Known	.514	.514
Std. HF, Observed	.586	.550
Standard Correction	-.072	-.036

Figure 5. Sample Data Sheet

each of the titrations of the three samples in grams.

2) Analysis of Nitrogen Dioxide in RFNA.

Record the following values:

(a) Weight of empty ampule in grams.
(b) Weight of ampule and sample in grams.
(c) Volume of ferrous solution used in titration of the ceric acid sulfate and sample in ml.
(d) Volume of ferrous solution used in titration of 35ml ceric acid sulfate in ml.

b. Percent total acidity

1) Record the normality of NaOH.
2) Record the sample weight in grams.
3) Record the volume of NaOH required for titration in ml.
4) Record the volume of NaOH required in the three blank determinations in milliliters.
5) Repeat steps 2 and 3 for duplicate runs.

c. Percent hydrogen fluoride

Record data in accord with sample data sheet shown in fig. 5.

6.3.15 Determination of Water (H_2O) in RFNA

a. Percent nitrogen dioxide

1) Standardization of Ce $(H\ SO_4)_4$ (Ceric Acid Sulfate)

(a) Record the weight of the three samples of the primary standard arsenious oxide in grams.
(b) Record the assay of the arsenious oxide.
(c) Record the volume of Ceric acid sulfate required for each of the titrations of the three samples in milliliters.

2) Analysis of Nitrogen Dioxide in RFNA.

Record the following values:

(a) Weight of empty ampule in grams.
(b) Weight of ampule and sample in grams.
(c) Volume of ferrous solution used in titration of the ceric acid sulfate and sample in ml.
(d) Volume of ferrous solution used in titration of 35ml ceric acid sulfate in ml.

b. Percent total acidity

1) Record the normality of NaOH.
2) Record the sample weight in grams.

3) Record the volume of NaOH required for titration in ml.
4) Record the volume of NaOH required in the three blank determinations.
5) Repeat steps 2 and 3 for duplicate runs.

c. Percent hydrogen fluoride

Record data in accord with sample data sheet shown in fig 5.

6.3.16 Determination of Total Solids in RFNA

Record the following data:

a. The weight of the empty crucible in grams.
b. The weight of the crucible with sample residue in grams.
c. Steps a and b shall be repeated for the second sample.

6.3.17 Quantitative Determination of TNC, NC, and RDX in Explosives Using Extraction Techniques and IR Spectroscopy

Record the following data:

a. Weight of empty thimble in grams.
b. Weight of thimble with sample in grams.
c. Weight of thimble in grams after TNT extraction.
d. Weight of empty 50 ml flask for TNT extraction in grams.
e. Weight of 50 ml flask with TNT extraction in grams.
f. Weight of thimble in grams after NC extraction.
g. Weight of empty 50 ml flask for NC extraction in grams.
h. Weight of 50 ml flask with NC extraction in grams.
i. Weight of thimble in grams after RDX extraction.
j. Weight of empty 50 ml flask in grams.
k. Weight of 50 ml flask with RDX extraction in grams.
l. The absorption value for NC-ethanol mixture.
m. The true path length for the NC determination in mm.
n. The absorption value for the TNT chloroform mixture.
o. The true path length for the TNT determination in mm.
p. The absorption value for the RDX acetone mixture.
q. The true path length for the RDX determination in mm.

6.3.18 Quantitative Analysis of the Alloying Elements in Steel Using the NSL Emission Spectrograph

The developed film shall be retained.

6.3.19 Analysis of Redstone Alcohol by the "Marker" Method

a. Record the percent weight of water, ethyl alcohol and methyl alcohol in each of the standard solutions.

b. Record the weight of each of the 1 gram samples of the known solutions to four decimal places and in units of grams.

c. Record the weight of chloroform, in grams to four decimal places, added to each of the above standards.

d. Record the weight of the Redstone alcohol in grams to four decimal places.

e. Record the weight of the chloroform added to the Redstone alcohol in grams to four decimal places.

f. All chart recordings shall be retained.

6.3.20 An IR Spectroscopic Procedure for Determining the Percentage of SF_6 (Sulfur Hexafluoride) in Gas Samples

All chart recordings shall be retained.

6.3.21 An IR Spectroscopy Procedure for Determining the CO (Carbon Monoxide) and CO_2 (Carbon Dioxide) Content of Air Samples (Missile Exhaust)

a. All chart recordings shall be retained.

b. Record the temperature in degrees F and the pressure in mm of Hg of the testing area.

6.3.22 An IR Spectrophotometric Method for the Analysis of DOA in a Mixture with Ammonium Perchlorate, Aluminum, and Polyvinyl Chloride Using KBr (Potassium Bromide) Pellet Technique

a. Synthetic Propellant

1) Record weight of tare in grams.
2) Record weight of tare and DOA in grams.
3) Record weight of tare in grams.
4) Record weight of tare and NH_4 Cl O_4.
5) Record weight of tare in grams.
6) Record weight of tare and KBr in grams.

b. Synthetic Propellant Mixture

1) Record weight of tare in grams.
2) Record weight of tare and synthetic propellant in grams.
3) Record weight of tare, synthetic propellant, and potassium bromide/sodium azide mixture in grams.

c. Propellant Mixture

1) Record weight of tare in grams.
2) Record weight of tare and propellant in grams.
3) Record weight of tare, propellant and potassium bromide/sodium azide mixture in grams.
4) Record all chart recordings.

6.3.23 An IR Spectroscopic Method for Determining the Water Content of Various Organic Solvents

All chart recordings shall be retained.

6.4 DATA REDUCTION AND PRESENTATION

6.4.1 Preparation for Test

None

6.4.2 Determination of Resin in Corporal Fuels

The resin content of the fuel samples is calculated in weight percent in the following manner:

$$\frac{\text{Weight of Resin}}{\text{10ml x Density of Sample at 60° F}} \times 100 = \text{Weight \% Resin}$$

An example of this calculation is as follows:

Weight of resin plus crucible = 19.5941g
Weight of empty crucible = 19.5249g
Sample volume = 10ml
Density at 60° F of sample = 1.080 g/ml

$$\frac{(19.5941 - 19.5249)}{10 \times 1.080} \times 100 = 0.64 \text{ Weight \% Resin}$$

6.4.3 Determination of SN(Tin) in H_2O_2 (Hydrogen Peroxide)

The amount of tin in the hydrogen peroxide is determined by the following equation:

$$\text{Tin (mg/liter)} = \frac{20\ (\text{Diffusion Current of Unknown})}{3\ (\text{Diffusion Current of Known})}$$

6.4.4 Analysis of Aniline Using the $HClO_4$ (Perchloric Acid) Method

a. The normality of the perchloric acid solution is found using the following formula:

$$\text{Normality} = \frac{\text{g of } Na_2CO_3 \times Na_2CO_3 \text{ assay}}{\text{ml of } HClO_4 \text{ used} \times 0.052995 \text{ (milliequivalent weight of } Na_2CO_3)}$$

b. Total weight of fuel is found by taking the difference in weight of the eyedropper with fuel and the weight of the eyedropper without fuel. The weight of the fuel sample in aliquot is found by multiplying the above weight by 25 milliliters and dividing by 250 milliliters.

c. The weight percent aniline is found by multiplying the ml of perchloric acid times the K factor and dividing by the weight of the sample. The K factor is found by multiplying the normality of the perchloric acid by 0.09312 which is the milliequivalent weight of aniline. To convert the quotient to percent, multiply by 100. An example of this calculation is as follows:

Normality of perchloric acid solution = 0.1010 N
Weight of fuel sample in aliquot $= \frac{5.3524 \text{ g} \times 25 \text{ ml}}{250 \text{ ml}}$
$= 0.53524$ g
Perchloric acid solution used = 28.36 ml
$K = N\ HClO_4 \times 0.09312 = 0.1010 \times 0.09312 = 0.0094505$

$$\text{Weight \% Aniline} = \frac{28.36 \text{ ml} \times 0.009405}{0.53524 \text{ g}} \times 100 = 49.83\%$$

6.4.5 Analysis of H_2O_2 (Hydrogen Peroxide) by the Permanganate Method

a. The normality of the potassium permanganate solution shall be determined by the following formula:

$$\text{Normality} = \frac{\text{g of } Na_2C_2O_4 \times Na_2C_2O_4 \text{ assay}}{\text{ml of } KMnO_4 \times 0.067000 \text{ (milliequivalent weight of } NA_2C_2O_4)}$$

b. The weight percent of hydrogen peroxide is found by multiplying the ml of potassium permanganate times the K factor and dividing by the weight of the sample. The K factor is found by multiplying the normality of potassium permanganate by 0.017010 which is the milliequivalent weight of hydrogen peroxide. An example of this calculation follows:

Normality of $KMnO_4$ = 0.5145 N
Weight of sample = 0.4489 g
$KMnO_4$ used = 38.80 ml

$K = N \text{ of } KMnO_4 \times 0.017010 = 0.5145 \times 0.017010 = 0.008752$

$$\text{Weight \% } H_2O_2 = \frac{38.80 \text{ ml} \times 0.008752}{0.4489 \text{ g}} \times 100 = 75.65\%$$

6.4.6 Analysis for Aniline by Gas Chromatography

To calculate the percent weight of aniline in a mixture, determine the unit area counts for the aniline peak of the sample in the same manner as with the pure aniline peak. Knowing the counts per gram of aniline, the counts for the sample peak, the volume of the sample, and the density of the sample, the percent weight of aniline in the sample can be calculated. An example of this calculation follows:

Counts/gram of aniline = 100,000 counts/5.09×10^{-3} g
$= 19.646 \times 10^{-6}$ counts/gram
Sample volume = 0.005 ml
Sample Density = 1.083 g/ml
Sample weight = (1.083 g/ml (0.005 ml))
$= 5.415 \times 10^{-3}$ g
Counts for aniline peak of sample = 48,000 counts

$$\text{Weight of aniline in sample} = \frac{48 \times 10^3 \text{ counts}}{19.646 \times 10^6 \text{ counts/gram}} = 2.443 \times 10^{-3} \text{ g}$$

$$\text{Percent weight of aniline in sample} = \frac{2.443 \times 10^{-3} \text{ g} (100)}{5.415 \times 10^{-3} \text{ g}} = 45.1\%$$

6.4.7 Analysis of UDMH using the KIO_3 (Potassium Iodate) Method

The weight percent of UDMH is determined as follows:

$$\text{Weight \% UDMH} = \frac{\text{Molarity of } KIO_3 \times \frac{\text{ml of } KIO_3}{1{,}000} \times 2 \times \text{mol. wt. UDMH} \times 100}{\text{Sample Weight}}$$

$$\text{Let K} = \frac{\text{Molarity of } KIO_3 \times 2 \times \text{mol. wt. of UDMH} \times 100}{1{,}000}$$

Then:

$$\text{Weight \% UDMH} = \frac{\text{ml of } KIO_3 \times K}{\text{Sample Weight}}$$

An example of this calculation follows:

Molarity of KIO_3 solution = 0.1002M
Sample weight = 0.4691 g
ml of KIO_3 used = 38.13 ml

$$K = 0.1002 \times 2 \times \frac{60.10 \times 100}{1{,}000} = 1.204$$

$$\text{Weight \% UDMH} = \frac{38.13 \times 1.204}{0.4691} = 97.86\%$$

6.4.8 Analysis of UDMH in JP-17 Fuel Using the Water Extraction Method

To calculate the weight percent of UDMH, the following constants must be employed:

1) Density and assay of UDMH used in preparing the JP-17.
2) Density of JP-17.
3) Loss of JP-4 caused by water extraction experimentally determined as 0.50 ml.

The weight percent of UDMH is given by the formula:

Weight % UDMH = vol. JP-17 - vol. JP-4 + loss JP-4 by water extraction
= 100.00 - 82.86 + 0.50 = 17.64 ml
Assay UDMH = 98.45 % by weight
Density UDMH = 0.7850 g/ml
Density JP-17 = 0.6513 g/ml
Volume JP-17 used = 100.0 ml

$$\text{Weight \% UDMH} = \frac{17.64 \text{ ml} \times 0.7850 \text{ g/ml} \times 0.9845 \times 100}{100.0 \text{ ml} \times 0.6513 \text{ g/ml}} = 20.93\%$$

6.4.9 Analysis of Redstone Alcohol Using Gas Chromatography

Fasten the recorder chart on a flat surface. Draw a baseline of the methanol and ethanol peaks in the following manner: Place a straight edge across the bottom of the peaks so that it is touching the straight line segment of the recorder pen trace on the right side of the methanol peak and touching the straight line segment of the recorder pen trace on the left side of the ethanol peak. The straight edge will not touch the dip between the peaks and will be nearly parallel to the horizontal lines on the recorder chart paper. Draw a line along the straight edge. Figure 6 illustrates the methanol and ethanol peaks and the properly drawn baseline. Divide the two peaks as shown at the lowest point between them. Using a planimeter, integrate the area under each peak until results are obtained which agree with 0.02 square inches of each other. The baseline of the water peak is drawn in the following manner:

Place a straight edge along the bottom of the peak so that it is touching the straight line segment of the recorder pen trace on the right side of the water

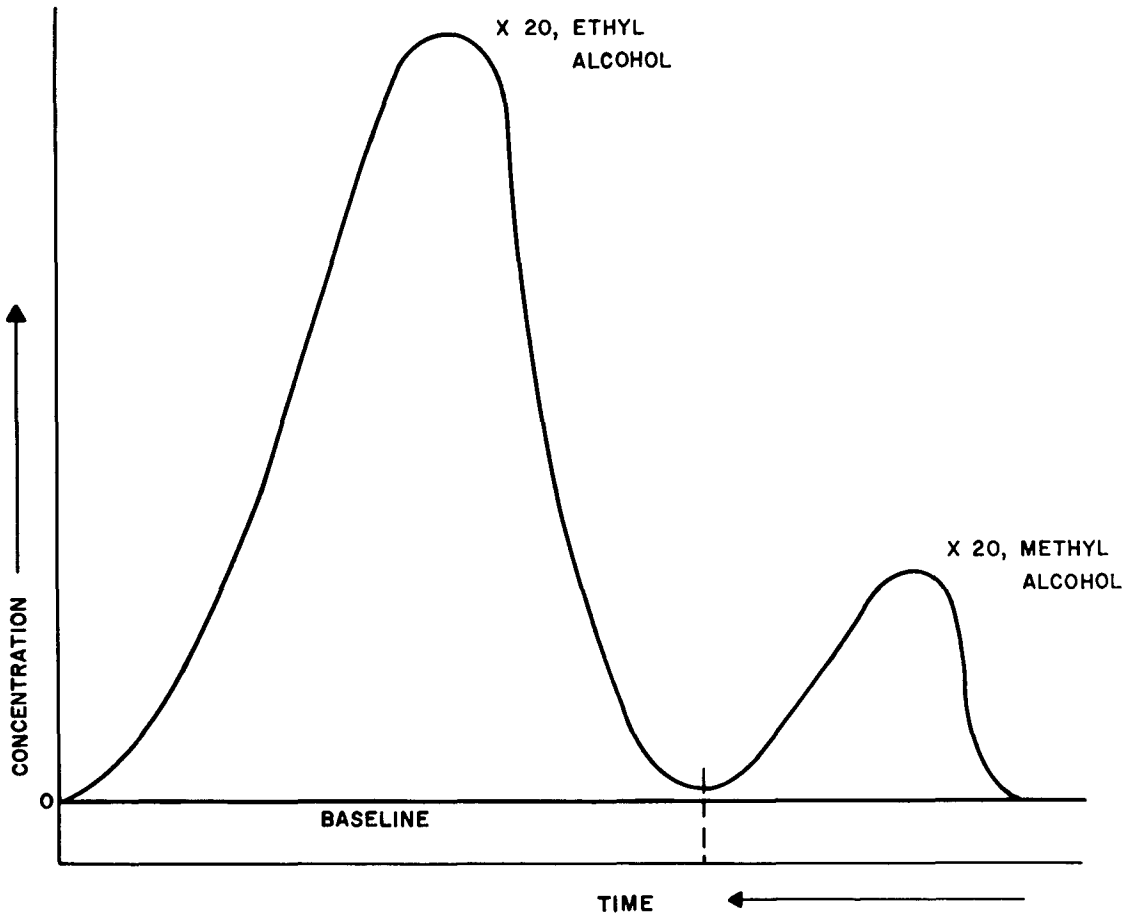

FIGURE 6. METHANOL AND ETHANOL PEAKS AND THEIR BASELINE

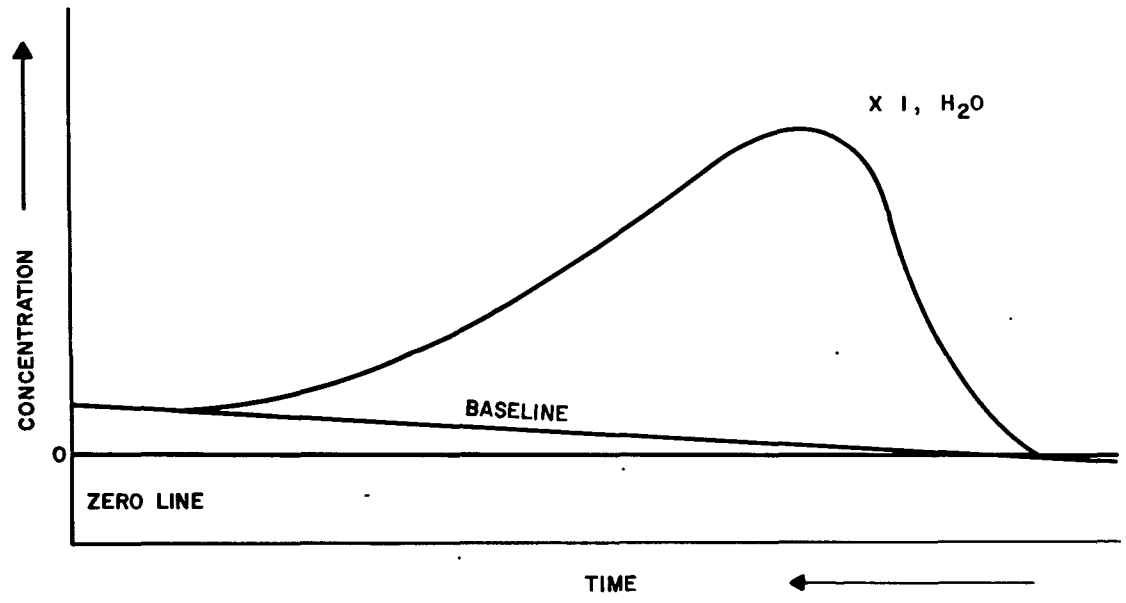

FIGURE 7. WATER PEAK AND ITS BASELINE

peak and touching the lowest point of the recorder pen trace on the left side of the water peak. Figure 7 illustrates the water peak and its baseline. Draw a line along the straight edge. Integrate the area until results are obtained which agree within 0.2 square inches of each other. Multiply the average area for each of the three peaks by the attenuation of each peak. Calculate the weight percent of MeOh, EtOH, and H_2O from the following:

$$\text{C.T.A.} = \frac{Am}{Km} + \frac{Ae}{Ke} + \frac{Aw}{Kw}$$

$$\text{Weight \% MeOH} = \frac{Am/Km\ (100)}{\text{C.T.A}}\ Km = 1.0\%$$

$$\text{Weight \% EtOH} = \frac{Ac/Ke\ \ (100)}{\text{C.T.A.}}\ Ke = 0.895\%$$

$$\text{Weight \% } H_2O = \frac{Aw/Kw\ (100)}{\text{C.T.A.}}\ Kw = 0.94\%$$

Am = (area under MeOH peak) (attenuation used)
Ae = (area under EtOH peak) (attenuation used)
Aw = (area under H_2O peak) (attenuation used)
Km = sensitivity factor for MeOH taken arbitrarily as one
Ke = sensitivity factor for EtOH
Kw = sensitivity factor for H_2O
C.T.A. = Calculated total area

6.4.10 Analysis of Redstone Alcohol Using the Refractive Index Method

The specific gravity at 15.6/15.6° C is given by the formula:

$$\frac{\text{(Weight of pycnometer + alcohol) - Weight of pycnometer}}{\text{(Weight of pycnometer + water) - Weight of pycnometer}}$$

The details for calculating the weight percent of ethyl alcohol, methyl alcohol, and water in a mixture of these components are discussed in Reference T of this MTP, "Handbook of Chemistry and Physics," and are represented by the following example:

Specific gravity of alcohol mixture at 15.6/15.6° C = 0.97917
Refractive index scale at 20° C = 30.00
Density ethyl alcohol = 13.80 %
Density methyl alcohol = 12.83 %
Refractive index scale (13.80%) ethyl alcohol = 38.34
Refractive index scale (12.83%) methyl alcohol = 22.48

The percent by weight of the alcohol which is methyl alcohol is equal to:

$$\frac{38.34\ (\text{Scale of EtOH} - 30.00)\ (\text{Scale of sample})}{38.34 - 22.48\ (\text{Scale of MeOH})} \times 100 = 52.58\%\ \text{by wt. EtOH}$$

Assume that all of the alcohol in the sample is ethyl alcohol and the percent by weight of the mixed alcohol in the sample is equal to:

12.83 (% MeOH based on gravity) x 0.5258 + 13.80 (% EtOH base on gravity) x (1-0.5258) = 13.29% by wt. mixed alcohol

The weight percent of ethyl and methyl alcohol in the sample is equal to:

13.29 (% mixed alcohol) x 0.5258 (% mixed alcohol that is MeOH) = 6.99% MeOH
13.29 (% mixed alcohol) x (1-0.5258) (% mixed alcohol that is EtOH) = 6.30% EtOH

The weight percent of water in the alcohol mixture is equal to:

$$100.00 - 6.99\ (\%\ MeOH) - 6.30\ (\%\ EtOH) = 86.71\ \%\ H_2O$$

6.4.11 Determination of NO_2 (Nitrogen Dioxide) in RFNA

a. Determine the normality of the $Ce(H\ SO_4)_4$ using the following formula for each of the three standard samples

$$\text{Normality Ce } (H\ SO_4) = \frac{20.22 \times (\text{wt. } As_2O_3) \times \text{assay } As_2O_3}{(\text{ml Ce } (H\ SO_4)_4}$$

The three samples must agree to within two parts per thousand.

b. The ferrous - ceric conversion factor shall be determined by dividing the volume (35 ml) of ceric acid sulfate by the volume of ferrous solution required to titrate that volume (35 ml) of ceric acid sulfate. For example:

$$\frac{35 \text{ ml } Ce(H\ SO_4)_4}{36.42 \text{ ml Fe } SO_4\ 7\ H_2O} = 0.9610 \text{ (Conversion factor)}$$

c. Determination of net volume of ceric acid sulfate solution used in reaction with test sample.

Multiply the ml of ferrous solution used in back-titrating the samples by the conversion factor and subtract the product from the ml of ceric solution used to obtain the net ml of ceric solution used in the reaction. For example:

Ml of ferrous solution used in back-titrating the sample = 10.00 ml
10.00 ml x 0.9610 = 9.610 ml ceric solution
Net ml of ceric solution used = 50.00 ml - 9.61 ml = 40.39

d. Determination of NO_2 weight percentage

Calculate the total amount of oxides as nitrogen dioxide using the following equation:

$$\text{Wt. \% } NO_2 = \frac{\text{Net ml } Ce(H\ SO_4)_4 \times \text{N } Ce(H\ SO_4)_4 \times \text{milliequivalent wt. } NO_2 \times 100}{\text{Sample weight}}$$

Net ml $Ce(H\ SO_4)_4$ = 40.39 ml
Normality of $Ce(H\ SO_4)_4$ = 0.1000
Milliequivalent weight NO_2 = 0.04601 g
Sample weight = 1.2907 g

$$\text{Wt. \% } NO_2 = \frac{40.39 \times 0.1000 \times 0.04601}{1.2907} \times 100 = 14.40\ \%$$

6.4.12 Determination of Total Acidity in RFNA

a. Average the three blank determinations and subtract this average from the total volume of NaOH used in titration of test sample.

b. The percent total acidity is determined as follows:

$$\%\ \text{Total Acidity (as } HNO_3) = \frac{\text{ml of NaOH x N of NaOH x 0.063016 (milliequivalent wt. of } HNO_3) \times 100}{\text{Sample weight}}$$

6.4.13 Determination of HF (Hydrogen Fluoride) in RFNA

Calculate the number of micrograms of fluoride in the 50-ml volumetric standard fluoride stock solution. The following equation is used to calculate the percent HF in each sample and standard run, assuming a 1-g sample weight:

$$\% \text{ HF} = \frac{(\text{micrograms of fluoride in 50-ml volumetric})\ (0.01316)}{\text{Sample weight}}$$

Compare the known percent HF for the standard with that determined experimentally. Apply this correction algebraically to the appropriate sample run. Reference C of this MTP gives further specific information pertaining to the testing of RFNA for acceptability.

6.4.14 Determination of HNO_3 (Nitric Acid) in RFNA

a. Percent nitrogen dioxide

1) Determine the normality of the $Ce(H\ SO_4)_4$ using the following formula for each of the three standard samples.

$$\text{Normality Ce } (H\ SO_4)_4 = \frac{20.22 \times (\text{wt. } As_2O_3) \times \text{assay } As_2O_3}{(\text{ml Ce } (H\ SO_4)_4}$$

The three samples must agree to within two parts per thousand.

2) The ferrous - ceric conversion factor shall be determined by dividing the volume (35 ml) of ceric acid sulfate by the volume of ferrous solution required to titrate that volume (35 ml) of ceric sulfate.
For example:

$$\frac{35 \text{ ml } Ce(H\ SO_4)_4}{36.42 \text{ ml Fe } SO_4\ 7\ H_2O} = 0.9610 \text{ (Conversion factor)}$$

3) Determination of net volume of ceric acid sulfate solution used in reaction with test sample.

Multiply the ml of ferrous solution used in back-titrating the samples by the conversion factor and subtract the product from the ml of ceric solution used to obtain the net ml of ceric solution used in the reaction.

For example:

Ml of ferrous solution used in back-titration the sample = 10.00 ml
10.00 ml x 0.9610 = 9.610 ml ceric solution
Net ml of ceric solution used = 50.00 ml - 9.61 ml = 40.39

4) Determination of NO_2 weight percentage
Calculate the total amount of oxides as nitrogen dioxide using the following equation:

$$\text{Wt. } \% NO_2 = \frac{\text{Net ml } Ce(H\ SO_4)_4 \times \text{N } Ce(H\ SO_4)_4 \times \text{milliequivalent wt. } NO_2 \times 100}{\text{Sample weight}}$$

Net ml $Ce(H\ SO_4)_4$ = 40.39 ml
Normality of $Ce(H\ SO_4)_4$ = 0.1000
Milliequivalent weight NO_2 = 0.04601 g
Sample weight = 1.2907 g

$$\text{Wt. \% } NO_2 = \frac{40.39 \times 0.1000 \times 0.04601}{1.2907} \times 100 = 14.40\ \%$$

b. Percent total acidity

1) Average the three blank determinations and subtract this average from the total volume of NaOH used in titration of test sample.
2) The percent total acidity is determined as follows:

$$\text{\% Total Acidity (as } HNO_3) = \frac{\text{ml of NaOH} \times \text{N of NaOH} \times 0.06316 \text{ (milliequivalent wt. of } HNO_3) \times 100}{\text{Sample weight}}$$

c. Percent hydrogen fluoride

Calculate the number of micrograms of fluoride in the 50-ml volumetric standard fluoride stock solution. The following equation is used to calculate the percent HF in each sample and standard run, assuming a 1-g sample weight:

$$\text{\% HF} = \frac{\text{(micrograms of fluoride in 50-ml volumetric) (0.01316)}}{\text{Sample weight}}$$

Compare the known percent HF for the standard with that determined experimentally. Apply this correction algebraically to the appropriate sample run. Reference C of this MTP gives further specific information pertaining to the testing of RFNA for acceptability.

d. The weight percent of nitric acid in RFNA is determined as follows:

$$\text{Wt \% } HNO_3 = \text{\% total Acidity} - (3.15 \times \%HF + 1.37 \times \%NO_2)$$

6.4.15 Determination of Water (H_2O) in RFNA

a. Percent nitrogen dioxide

1) Determine the normality of the $Ce(H\ SO_4)_4$ using the following formula for each of the three standard samples

$$\text{Normality Ce } (H\ SO_4)_4 = \frac{20.22 \times (\text{wt. } As_2O_3) \times \text{assay } As_2O_3}{(\text{ml Ce } (H\ SO_4)_4}$$

The three samples must agree to within two parts per thousand.
2) The ferrous - ceric conversion factor shall be determined by dividing the volume (35 ml) of ceric acid sulfate by the volume of ferrous solution required to titrate that volume (35 ml) of ceric acid sulfate.
For example:

$$\frac{35 \text{ ml } Ce(H\ SO_4)_4}{36.42 \text{ ml Fe } SO_4\ 7\ H_2O} = 0.9610 \text{ (Conversion factor)}$$

3) Determination of net volume of ceric acid sulfate solution used in reaction with test sample.

Multiply the ml of ferrous solution used in back-titrating the samples by the conversion factor and subtract the product from the ml of ceric solution used to obtain the net ml of ceric solution used in the reaction.

For example:

Ml of ferrous solution used in back-titrating the sample = 10.00 ml
10.00 ml x 0.9610 = 9.610 ml ceric solution
Net ml of ceric solution used = 50.00 ml - 9.61 ml = 40.39

4) Determination of NO_2 weight percentage

Calculate the total amount of oxides as nitrogen dioxide using the following equation:

$$\text{Wt. \% } NO_2 = \frac{\text{Net ml } Ce(H\,SO_4)_4 \times N\ Ce(H\,SO_4) \times \text{milliequivalent wt. } NO_2 \times 100}{\text{Sample weight}}$$

Net ml $Ce(H\,SO_4)_4$ = 40.39 ml
Normality of $Ce(H\,SO_4)_4$ = 0.1000
Milliequivalent weight NO_2 = 0.04601 g
Sample weight = 1.2907 g

$$\text{Wt. \% NO} = \frac{40.39 \times 0.1000 \times 0.04601}{1.2907} \times 100 = 14.40\ \%$$

b. Percent total acidity

1) Average the three blank determinations and subtract this average from the total volume of NaOH used in titration of test sample.
2) The percent total acidity is determined as follows:

$$\text{\% Total Acidity (as } HNO_3) = \frac{\text{ml of NaOH} \times \text{N of NaOH} \times 0.063016\ (\text{milliequivalent wt. of } HNO_3) \times 100}{\text{Sample weight}}$$

c. Percent hydrogen fluoride

Calculate the number of micrograms of fluoride in the 50-ml volumetric standard fluoride stock solution. The following equation is ustr to calculate the percent HF in each sample and standard run, assuming a 1-g sample weight:

$$\text{\% HF} = \frac{(\text{micrograms of fluoride in 50-ml volumetric})\ (0.01316)}{\text{Sample weight}}$$

Compare the known percent HF for the standard with that determined experimentally. Apply this correction algebraically to the appropriate sample run. Reference C of this MTP gives further specific information pertaining to the testing of RFNA for acceptability.

d. The weight percent of nitric acid in RFNA is determined as follows:

$$\text{Wt \% } HNO_3 = \text{\% total Acidity } -(3.15 \times \%HF + 1.37 \times \%NO_2)$$

e. The weight percent of water in RFNA is determined as follows:

$$\text{Wt \% } H_2O = 100 -(\% NO_2 + \% HF + \% HNO_3)$$

6.4.16 Determination of Total Solids in RFNA

a. Determine the weight of the residue by subtracting the weight of the empty crucible from the weight of the crucible with the residue.

b. Average the weight of the residue for the two samples.

c. Determine weight percent of total solids in RFNA shall be determined as follows:

$$\% \text{ Total Solids} = \frac{\text{Average Weight of Residue}}{10 \text{ ml} \times (\text{density of } HNO_3)} \times 100\%$$

6.4.17 Quantitative Determination of TNC, NC, and RDX in Explosives Using Extraction Techniques and IR Spectroscopy

a. NC determination

1) The absorbance for any difference in path length from 0.100 mm shall be corrected using the following formula:

$$A \text{ (corrected)} = \frac{A \text{ (uncorrected)} \times b \text{ (true path length)}}{b_2 \text{ (0.100-mm desired path length)}}$$

2) The concentration of the NC in the NC/ethanol mixture shall be determined using the following formula:

$$\text{Concentrated percent NC by weight} = \frac{A}{0.202}$$

3) The weight of NC present in the NC/ethanol mixture shall be determined by multiplying the weight of the mixture by the percent NC in the mixture.

NOTE: The weight of the mixture shall be determined by subtracting the weight of the empty 50 ml flask from the weight of the 50 ml flask with the mixture.

4) If NC was the only substance extracted by the ethanol, then the weight of the NC determined in step 3 should equal the total weight extracted by the ethanol.

NOTE: The total weight extracted by the ethanol shall be determined by subtracting the weight of the thimble after the ethanol extraction from the weight of the thimble before the ethanol extraction.

5) To determine the percent NC of the original sample, the weight of NC calculated in step 3 shall be divided by the

total sample of weight and multiplied by 100.

NOTE: The total sample weight shall be determined by subtracting the empty thimble weight from the weight of the thimble with the sample.

b. TNT

1) The absorbance for any difference in path length from 0.100 mm shall be corrected using the following formula:

$$A\ (\text{corrected}) = \frac{A\ (\text{uncorrected}) \times b\ (\text{true path length})}{b_2\ (0.100\text{-mm desired path length})}$$

2) The concentration of the TNT in the TNT/chloroform mixture shall be recorded using the following formula:

$$\text{Concentrated percent TNT by weight} = \frac{A}{0.241}$$

3) The weight of the TNT present in the TNT/chloroform mixture shall be determined by multiplying the weight of the mixture by the percent TNT in the mixture.

NOTE: The weight of the mixture shall be determined by subtracting the weight of the empty 50 ml flask from the weight of the 50 ml flask with the mixture.

4) If TNT is the only substance extracted by the chloroform then the weight of the TNT determined in step 3 should equal the total weight extracted by the ethanol.

NOTE: The total weight extracted by the chloroform shall be determined by subtracting the weight of the thimble after chloroform extraction from the weight of the thimble before the chloroform extraction.

5) To determine the percent TNT of the original sample, the weight of TNT calculated in step 3 shall be divided by the total sample weight and multiplied by 100.

c. RDX

1) The absorbance for any difference in path length from 0.100 mm shall be corrected using the following formula:

$$A(\text{corrected}) = \frac{A\ (\text{uncorrected}) \times b\ (\text{true path length})}{b_2\ (0.100\ \text{mm desired path length})}$$

2) The concentration of the RDX in the RDX/acetone mixture shall be determined by the following formula:

$$\text{Concentrated percent RDX by weight} = \frac{A}{0.439}$$

3) The weight of RDX present in the RDX/acetone mixture shall be determined by multiplying the weight of the mixture by

the percent RDX in the mixture

NOTE: The weight of the mixture shall be determined by subtracting the weight of the empty 50 ml flask from the weight of the 50 ml flask with the mixture.

4) If RDX was the only substance extracted by the acetone then the weight of the RDX determined in step 3 should equal the total weight extracted by the acetone.

NOTE: The total weight extracted by the ethanol shall be determined by subtracting the weight of the thimble after the acetone extraction from the weight of the thimble before the acetone extraction.

5) To determine the percent RDX of the original sample, the weight of RDX calculated in step 3 shall be divided by the total sample weight.

6.4.18 Quantitative Analysis of the Alloying Elements in Steels Using the NSL Emission Spectrograph

a. The transmittance readings (Tr) of the analytical lines listed in Table VII shall be determined for the element being determined.

b. The average transmittance reading shall be determined for each line.

c. A log I value shall be evaluated for each line using the emulsion calibration equation:

$$\text{Log I} = 2.15147585 - 3.88563838 \times 10^{-2} (\text{Tr}) + 1.29888039 \times 10^{-3} (\text{Tr})^2 - 2.70118813 \times 10^{-5} (\text{Tr})^3 + 2.78016109 \times 10^{-7} (\text{Tr})^4 - 1.13007283 \times 10^{-9} (\text{Tr})^5$$

d. Algebraically subtract the log I value for each Fe line from the log I value of the corresponding element line for each sample and standard.

NOTE: This will give a log I ratio, $\log \frac{(\text{I element line})}{(\text{I Fe line})}$, for each sample and standard.

e. Average the log I value calculated for the standard run before and after the samples in the excitation sequence.

f. Algebraically subtract the average log I ratio from the log I ratio previously determined, listed in Table XI from the appropriate element calibration equation.

g. The difference is applied as a correction to the unknown samples by adding or subtracting the correction algebraically, depending upon whether the difference is positive or negative.

h. Evaluate the concentration of the known using the appropriate calibration equation listed in Table XII.

NOTE: A sample calculation is shown in Table XIII.

i. The log I ratio of the external standard on this, Table XIII is larger than the log I ratio used in the calibration equation.

Table XI. Analytical Lines

Element	Element Line	Internal Standard Line	Log I ratio (NBS std)
B	2496.78	2496.99	+0.0252015
Cr (Low)	2822.37	2813.61	-0.5218975
Cr (High)	2324.89	2351.01	+0.09589388
Mn	2886.68	2906.12	+0.0664151
Mo	2775.40	2770.51	+0.02426097
Ni (Low)	3012.00	3011.48	-0.14732378
Ni (High)	3012.00	3011.48	-0.15198915
Si (Low)	2881.58	2874.17	-0.33817684
Si (High)	2516.12	2509.12	-0.49032368
V	3110.71	3116.59	+0.37794851

Table XII. Calibration Equations

Element	Cone Range (percent)	Calibration Equation
B	0.002-0.01	Log (Cone x 10^3) = 4.3961 x 10^{-2} + 2.8435 (corrected log I ratio)
Cr (Low)	0.3 - 1.1	Log (Cone x 10) = 1.2833 + 1.1593 (corrected log I ratio)
Cr (High)	2.5 - 25.0 (stainless steel)	Log (Cone) = 1.09471 + 1.2002 (corrected log I ratio)
Mn	0.3 - 1.5	Log (Cone x 10^{-2}) = 0.85028 + 1.2804 (corrected log I ratio)
Mo	0.2 - 1.0	Log (Cone x 10) = 0.71370 + 0.91161 (corrected log I ratio)
Ni (Low)	0.1 - 1.0	Log (Cone x 10) = 1.1232 + 2.2637 (corrected log I ratio)
Ni (High)	0.5 - 5.0	Log (Cone x 10) = 1.0325 + 1.7502 (corrected log I ratio)
Si (Low)	0.1 - 0.3	Log (Cone x 10) = 7.6663 + 1.4929 (corrected log I ratio)
Si (High)	0.3 - 1.2	Log (Cone x 10) = 1.2833 + 1.3690 (corrected log I ratio)
V	0.005-0.15	Log (Cone x 10^2) = 5.4388 + 1.1379 (corrected log I ratio)

Table XIII. Sample Calculations

Spectra No.	Sample	Average TR READING Mn Line	Calculated Log I (Mn)
1	Standard NBS #412A	46.275	1.62919807
2	Unknown	59.25	1.51490269
3	Unknown	66.225	1.44803376
4	Unknown	55.575	1.57812297
5	Standard NBS #412A	43.650	1.65195696
Spectra No.	**Sample**	**TR READING; Fe Line**	**Calculated Log I (Fe)**
1	Standard NBS #412A	56.925	1.53963499
2	Unknown	54.825	1.52791291
3	Unknown	52.8	1.57260806
4	Unknown	58.4	1.522684466
5	Standard NBS #412A	56.525	1.53934994

Spectra No.	Log I Ratio (log I (MV) - Log I (Fe))	Corrected Log I ratio*
1	+0.08956308	---
2	-0.01301022	-0.03945127
3	-0.12457430	-0.15101535
4	+0.05543831	+0.02899726
5	+0.11260702	---

*Ave log I Ratio of external standard = 0.10108505

Log I ratio of external standard from a typical calibration equation: $\frac{(0.074644)}{(+0.02644105)}$

j. The difference is then subtracted algebraically from the unknown log I ratios to correct for the increase.

NOTE: Table XIV illustrates this correction

k. Consult Reference F of this MTP for further information pertaining to the details of the emission spectrographic method of analyzing alloying elements in steels.

Table XIV. Corrected Calculations

Spectra	Calculated Concentration	Rounded Calculated Concentration
1	---	---
2	0.79977	0.80
3	0.65692	0.66
4	0.88741	0.89
5	---	---

6.4.19 Analysis of Redstone Alcohol by the "Marker" Method

a. Standard chromatographs shall be made from the runs on the standards, by plotting the ratio of the counts under the alcohol peaks and chloroform peak against the ratio of the actual percent of the alcohol and chloroform.

NOTE: See Figure 8 for the plot of the count ratio of methyl alcohol and chloroform against their weight percent ratio. See Figure 9 for the plot of the count ratio of ethyl alcohol and chloroform against their weight percent ratio.

b. From the chart of the Redstone alcohol determine the ratio of the counts under each of the alcohol peaks and the chloroform peak.

c. From the standard chromatograms, prepared in step 1, and from the actual amount of chloroform present, the amount of alcohol present shall be determined.

For example: If that ratio of the counts under the methyl alcohol peak and the chloroform peak is 0.400, Figure 8 indicates that this is equivalent to a weight percent ratio of 0.200. Assume that the actual weight percentage of chloroform present in the mixture is 59.00. The amount of methyl alcohol can be determined as follows:

$$0.200 = \frac{\%\ \text{MeOH (by volume)}}{59.00}$$

$$\%\ \text{MeOH} = 11.8\%$$

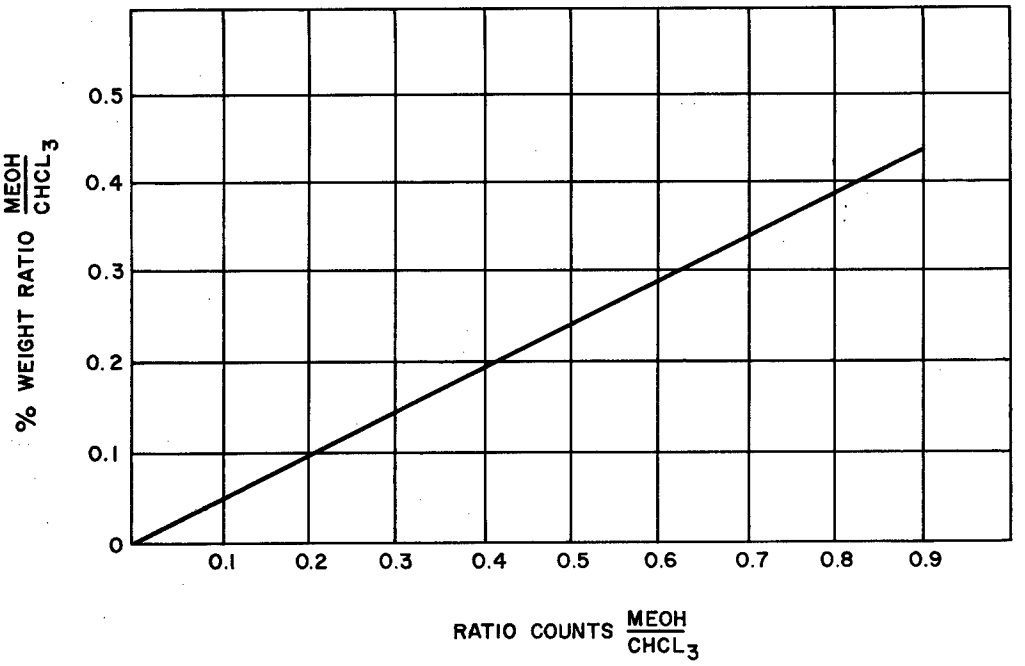

FIGURE 8. PLOT OF THE COUNT RATIO OF METHYL ALCOHOL AND CHLOROFORM

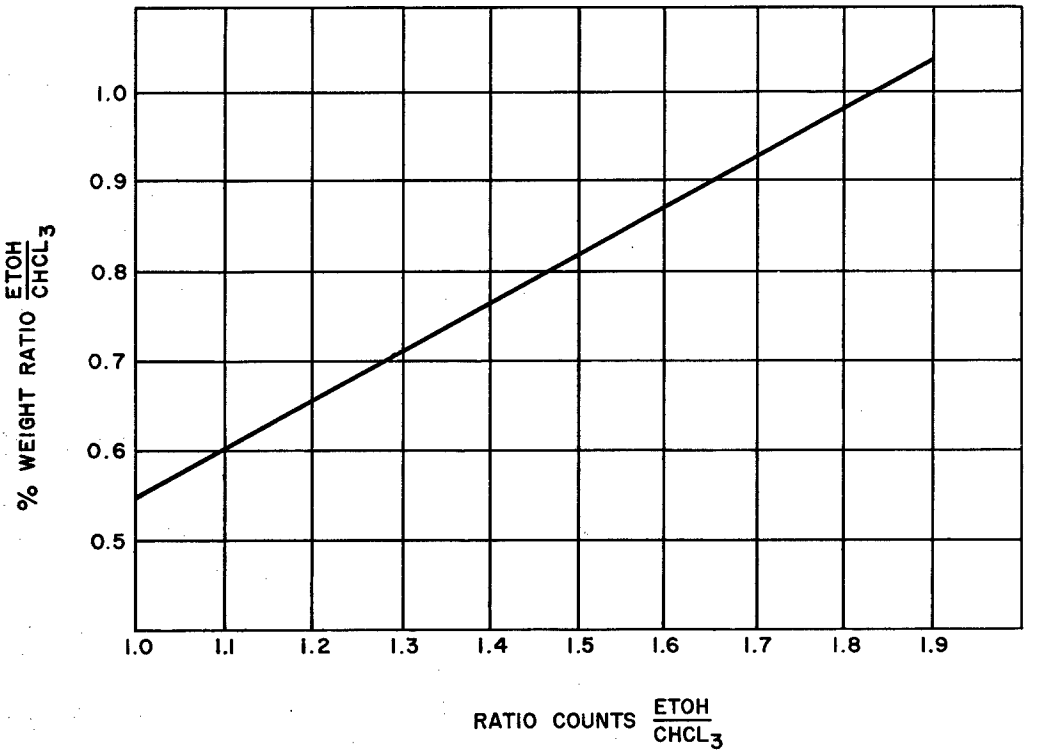

FIGURE 9. PLOT OF THE COUNT RATIO OF ETHYL ALCOHOL AND CHLOROFORM

After the methyl and ethyl alcohol percentage has been determined, the water percentage is obtained by difference. An example:

MeOH	= 11.8%
EtOH	= 82.3%
Total alcohol	= 94.1%
H_2O	= 5.9%

6.4.20 An IR Spectroscopic Procedure for Determining the Percentage of SF_6 (Sulfur Hexafluoride) in Gas Samples

a. Draw a baseline for each sample and standard.

b. Record the I_B (percent transmittance of the baseline) near the 7.2-micron band for each sample.

c. Record the I_S (percent transmittance of the absorption peak) near the 7.2-micron band for each sample and standard.

d. Calculate and record the I_B/I_S ratio for each sample and standard.

e. Record the absorption (log I_B/I_S) ratio for each sample and standard.

f. The absorption values for each sample are corrected by reference to the pure (99 percent) sulfur hexafluoride absorption in the following manner.

$$\% SF_6 \text{ (by volume)} = 2.3 \times 10^{-3} + 575.7 \text{ (A) (Equation relating absorption to concentration)}$$

Note

The equation relating absorption to concentration will vary from instrument to instrument.

$$\frac{99.00 - 2.3 \times 10^{-3}}{575.7} = A = 0.17196$$

g. To obtain the corrected absorption for each sample, multiply the absorption value for each sample b the ratio:

$$\frac{\text{A observed (standard)}}{\text{A calculated (standard)}}$$

h. Use the following equation to calculate the concentration of each sample from the corrected absorption values:

$$\text{Cone (\% volume) } SF_6 = 2.3 \times 10^{-3} + 575.7 \text{ (corrected absorption)}$$

NOTE: The corrected absorption equation will vary from instrument to instrument.

Standard Error = 1.58% at 658 mm Hg and 23° C.

i. Consult Reference 4G of this MTP for further details pertaining to recording the sulfur hexafluoride absorption band and calculating the results of the analysis.

6.4.21 An IR Spectroscopy Procedure for Determining the CO (Carbon Monoxide) and CO_2 (Carbox Dioxide) Content of Air Samples (Missile Exhaust)

a. Draw a baseline for the absorption bands of each sample as illustrated in Figure 10.

b. Record the I_B (percent transmittance of the baseline) near the 4.30-micron and 4.65-micron wavelengths for each sample.

c. Record the I_S (percent transmittance of the absorption peak) near the 4.30-micron and 4.65-micron wavelengths for each sample.

d. Calculate and record the I_B/I_S ratios for each absorption band.

e. Divide each sample I_B/I_S values by the blank I_B/I_S values for each wavelength and record the result as the net I_B/I_S value for each sample.

f. Record the absorption (log I_B/I_S net) for the absorption bands of each sample.

NOTE: Table XV is an example for recording these data.

g. Use the following equations to calculate the concentration from the absorption values and record the results as percent by volume:

CO_2 (4.30 microns): CO_2 (% volume) = $2.9 \times 10^{-3} + 3.760A$, SEE = 7.8×10^{-3} % at 650 mm Hg and 20° C

CO (4.65 microns): CO (% volume) = $5.0 \times 10^{-4} + 6.232A + 3.237 \times 10^{2}A^{2}$, SEE = 2.8×10^{-3} % at 650 mm Hg and 20° C

h. Determine the true percent carbon monoxide (corrected) from Figure 11 which relates the observed (calculated) percent carbon monoxide to the true (corrected percent).

Table XV. Recording CO and CO_2 Absorption Data

WAVELENGTH	RUN	I_B	I_S	I_B/I_S	I_B/I_S (NET)	LOG I_B/I_S (NET)
4.30	Blank	95.34	97.35	0.9793		
4.30	Sample	93.40	91.73	1.0182	1.0397 *	0.01691
4.65	Blank	95.33	95.33	1.0000		
4.65	Sample	93.34	92.62	1.0078	1.0078 **	0.00337

* 1.0182 / 0.9793 = 1.0397

** 1.0078 / 1.0000 = 1.0078

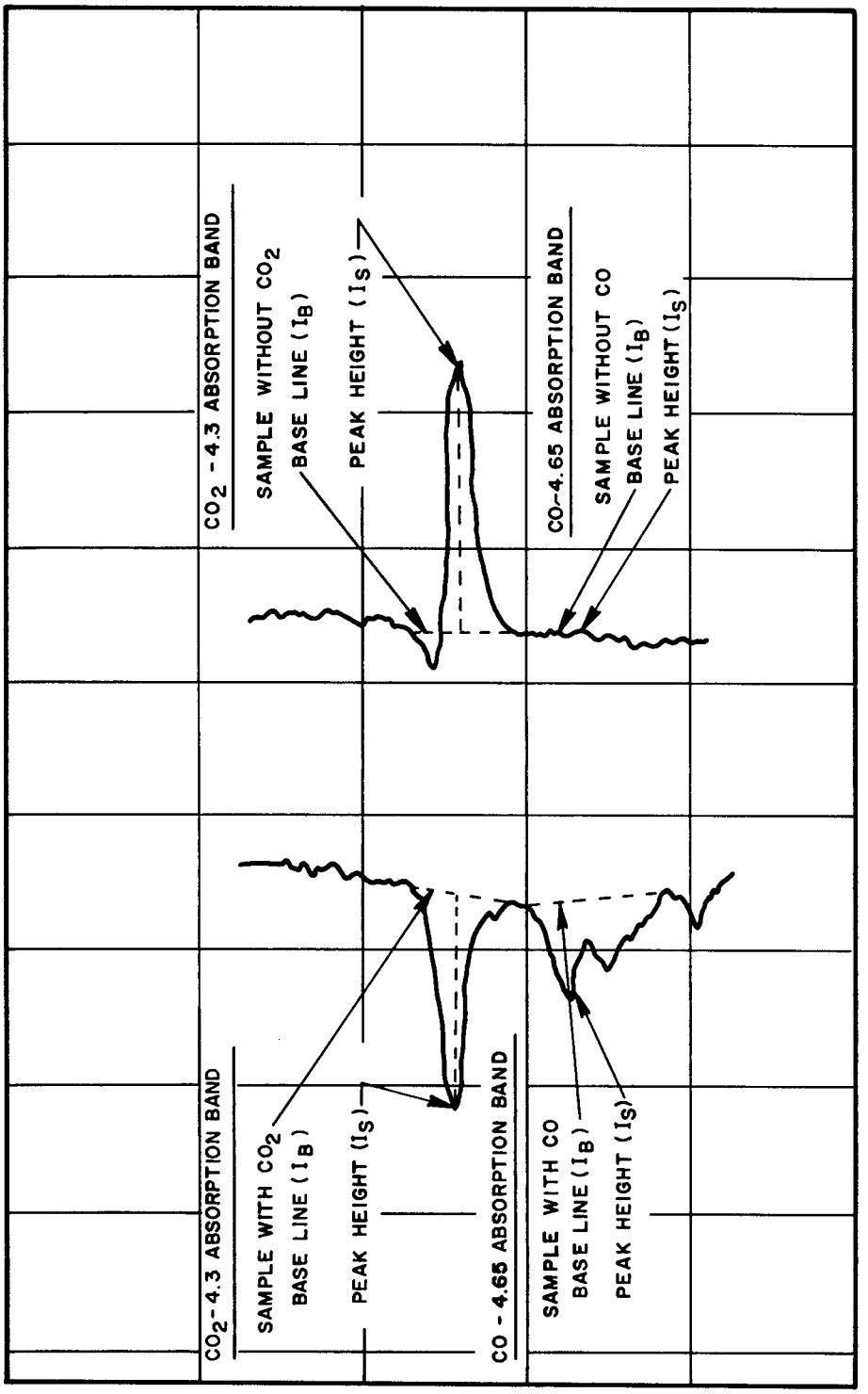

FIGURE 10. 4.0-TO 5.0-MICRON WAVELENGTHS SCAN FOR NITROGEN PURGE (BLANK) THROUGH A 10 cm. GAS CELL AND A GAS CONTAINING CO AND CO_2 (EXPANSION SCALE 10 X)

i. Construct a graph similar to Figure 11 for carbon monoxide and another for carbon dioxide, as necessary, to correct for changing instrument conditions which may affect the absorptivities. Sample calculations of CO and CO_2 concentrations are as follows:

$$\% \text{ CO} = 5.0 \times 10^{-4} + 6.232\ (0.00337) + 3.237 \times 10^{2}\ (0.00337)^{2} = 0.0242$$

(From Figure 11 the % CO true) = 0.034

$$\% \text{ CO} = 2.9 \times 10^{-3} + 3.670\ (0.01691) = 0.059$$

Consult References 4H through 4J of this MTP for further details pertaining to recording and calculating carbon monoxide and carbon dioxide content of air samples.

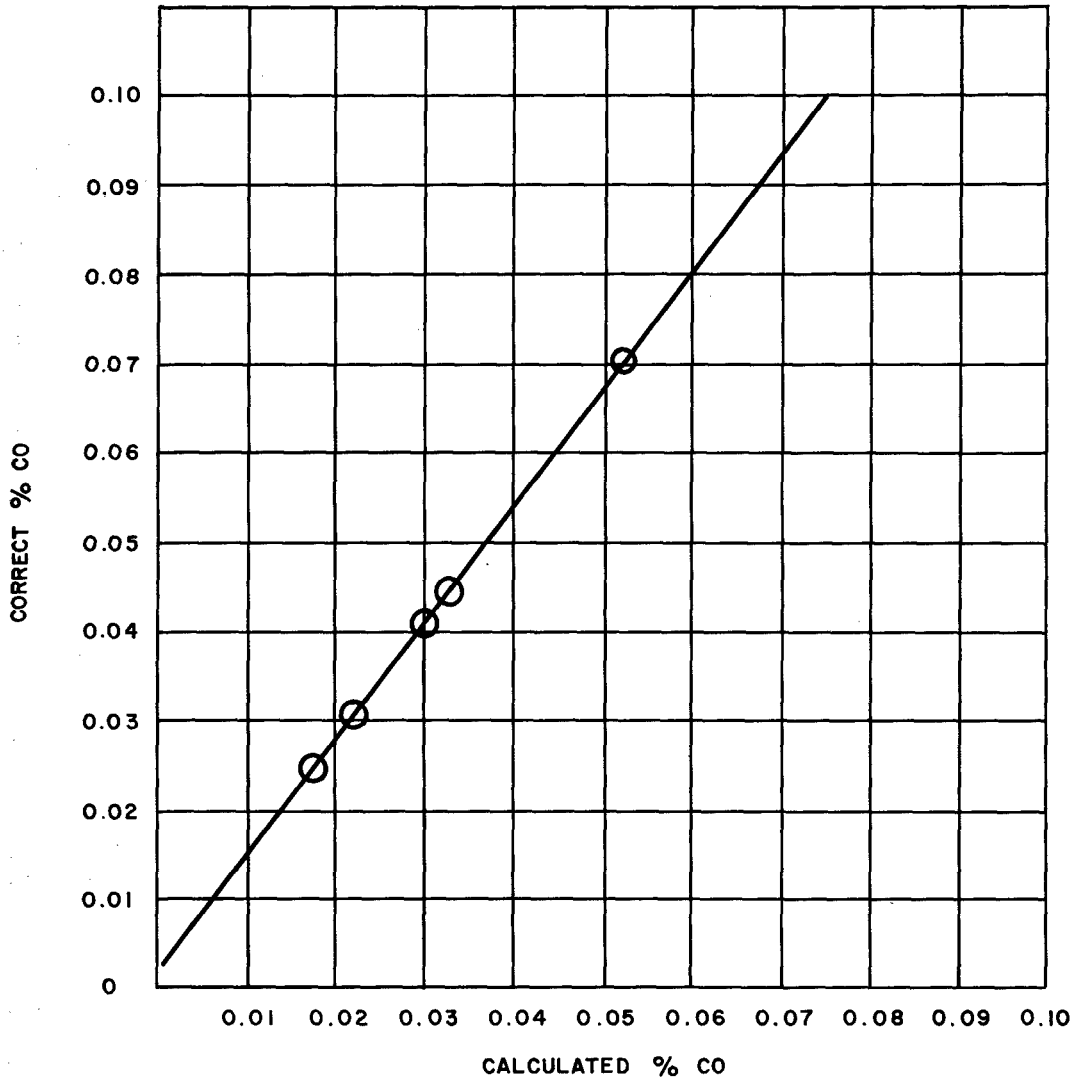

FIGURE II. CORRECTION CURVE FOR PERCENT CO DETERMINED BY IR PERCENT CO CORRECTED VERSUS PERCENT CO CALCULATED FROM CO ABSORPTION EQUATION

6.4.22 An IR Spectrometric Method for the Analysis of DOA in a Mixture with Ammonium Perchlorate, Aluminum, and Polyvinyl Chloride Using KB_r (Potassium Bromide) Pellet Technique

a. Construct base lines for all of the recorded scans by drawing the best straight line through the minimum absorption points for each scan.

b. Record the absorption values log (I base/I sample for the sodium azide peak and the DOA peaks.

c. Divide the A (absorbance) value for the DOA peak by the A value for the sodium azide peak for each scan $\frac{A\ (DOA)}{A\ (Na\ N_3)}$

d. Determine the average value of this absorbance ratio for the synthetic propellant mixture pellet and also for the actual sample propellant.

e. The DOA in the propellant shall be determined using the relationship:

$$\frac{\text{Concentration DOA in Synthetic Propellant}}{\text{Synthetic Propellant Mixture}} = \frac{\text{Concentration DOA in Propellant}}{\text{Average } \frac{A(DOA)}{A\ (Na\ N_3)} \text{ Propellant Mixture}}$$

The weight fraction of DOA in the synthetic propellant is determined by dividing the weight of DOA in the amount of synthetic propellant mixture by the total mixture weight used to prepare the pellets. The following are sample calculations:

(1) Synthetic Propellant

Tare + DOA	10.160035 gms	percent
Tare	10.128926 gms	
DOA	0.031109 gms	9.86
Tare + $NH_4\ ClO_4$	10.336129	
Tare	10.160035	
$NH_4\ ClO_4$	0.176094	55.82
Tare + KB_r	10.444412	
Tare	10.336129	
KB_r	0.108283	34.32 (replaces aluminum and polyvinyl chloride)

(2) Synthetic Propellant Mixture

Tare + "synthetic propellant"	8.207142	
Tare	8.202113	
"synthetic propellant"	0.005029	
Tare + "synthetic propellant" + KB4 Na N_3	9.202181	
Tare	8.202113	
"synthetic propellant mixture"	1.000068	

(3) Weight Percent DOA in Synthetic Propellant Mixture

$$\text{Wt \% DOA} = \frac{0.005029 \text{ gms} \times 0.0986}{1.000068 \text{ gms}} \times 100 = 0.04958$$

(4) Propellant Mixture

Tare + propellant	10.134896
Tare	10.129932
Propellant	0.999976

(5) Absorption Data

Item	A (DOA) /A ($Na N_3$)
"propellant mixture" pellet	0.20780
"synthetic propellant mixture" pellet	0.19772

(6) Weight Percent DOA in Propellant Mixture

$$\frac{0.04958}{0.19772} = \frac{\text{Wt \% of DOA in "propellant mixture"}}{0.20780}$$

Wt % DOA in "propellant mixture" = 0.05211%

(7) Weight Percent DOA in Propellant

$$\text{Wt \% DOA in propellant} = \frac{0.0005211 \times 0.999976}{0.004964} \times 100 = 10.5\%$$

6.4.23 An IR Spectroscopic Method for Determining the Water Content of Various Organic Solvents

a. To determine the amount of water in each sample of benzene and toluene analyzed:

1) Draw base lines from 2.4 to 3.0 microns wavelength for each scan.
2) Record the I_B (percent transmittance of base line) at the 2.75-micron absorption peak on dried benzene or toluene compensated scans.
3) Record I_o at the 2.75 micron negative peak (may not be negative if different windows are used) for the dried benzene or toluene compensated scans.
4) Determine the average I_o/I_b ratio for several scans.
5) Record I_B and I_S values for each sample and determine the average I_B/I_S ratio for each sample.
6) Calculate I_o/I_S ratio for each sample from the product of I_o/I_B and I_B/I_S for each sample.
7) Find log I_o/I_S (absorption values for each sample).
8) Use the following equation to calculate the ppm water in benzene:

$$H\,O\ (ppm) = 4.89 \times 10^3\, A + 5.4875 \times 10^2 A^2;\ \text{Standard Error} = \pm 15.5 \text{ ppm}$$

9) Use the following equation to calculate the ppm water in toluene:

$$H_2O \text{ (ppm)} = A/6.174 \times 10^{-4}$$

b. To determine the amount of water in carbon tetrachloride analyzed:

1) Draw base lines from the 2.4- to 3.0-micron wavelengths for each scan and record the I_B (percent transmittance of base line) at the 2.75-micron water absorption peak.
2) Record the I_S (percent transmittance of absorption peak) at the 2.75-micron peak and determine I_B/I_S ratio for each sample.
3) Use the following equation to calculate the ppm water in carbon tetrachloride:

$$H_2O \text{ (ppm)} = A/8.656 \times 10^{-3}$$

NOTE: Consult Reference K of this MTP for further information concerning the determination and calculation of water in various solvents.

APPENDIX A

General Information for Film Radiography

a. Principle and Basis

A radiograph is a photographic record produced by passing X-rays, gamma rays or other forms of radiation through an object and recording the transmitted radiation on photographic film. The degree of darkening of the film depends on the amount of radiation striking the film. Every material will absorb radiation to a different degree depending on its density. Therefore, varying densities or the absence of material in a specimen will record as contrasting shades of darkening on the film after it is developed.

b. Considerations

1) Equipment Requirements

There are many types of X-ray machines. Some are water cooled, and still others use combined cooling systems. The requirements of each X-ray machine by necessity will depend on its intended application. The two most important factors to consider are the maximum useful kilovoltage and the manner in which the X-ray tube is mounted. The maximum useful kilovoltage determines the maximum thickness of material which can be penetrated. The mounting of the X-ray tube determines the ease and speed with which the tube can be positioned to radiograph a particular item. A permanent installation is one where the X-ray machine is operated in only one room. Such a unit includes a shielded control panel, shielded room, cooling system, power supply, and a hoisting mechanism.

Mobile units consist of an X-ray unit with a control system and cooling system mounted on a van which can transport the entire unit. Radioactive sources for mobile units consist of a series of Cobalt 60 sources which can be transported on a flat bed truck. The weight of the lead safes necessary to store the radioactive sources depends on the strength of the isotope and will require a crane to load or unload the vehicle. Most of the lead safes have small wheels which enable the safe to be moved over short distances. These sources may have collimating cones for beam applications or they may be of the panoramic type.

There are a variety of portable X-ray machines ranging in weight from 50 to 100 pounds and have a self-contained cooling system. Such units can be positioned easily. Radioactive sources for portable X-ray units consist of a series of Iridium 192 radioactive sources which can be stored in lead safes. These units may have collimating cones for beam applications or they may be of the panoramic type. Thulium is another isotope which shows promise in the radiography of light weight metals.

2) Shielding of Film

Any object to be radiographed should be placed on a lead screen to protect the film from scatter radiation which may fog the film. Nearby concrete walls and other structural members can also cause scatter. The proper interpretation of any radiographic film will be enhanced by adequate protection

from scatter radiation. Additional protection from scatter radiation can be gained by placing the film between two lead screens. Usually, the back screen is twice as thick as the front screen.

3) Darkroom Facilities

When the volume of work is intermittent, one room containing the processing tanks, wash tanks, film driers, and film storage cabinets should be sufficient to handle the film processing. If the volume of work is large, it may be advantageous to have separate rooms for the film driers and film storage cabinets. Automatic film processing should be used whenever the film processing time becomes excessive. The only labor involved in the automatic film process is feeding and removing film from the machine. The darkroom should be adjacent or as close to the radiographic unit as possible. The film sustains a minimum of transportation damage by reducing the distance. Hazardous conditions such as explosives may not permit such an arrangement, but whenever possible, such a design will lead to a more efficient operation.

4) Viewing Equipment

The interpretation of radiographic film can best be accomplished in a separate room where the lights can be dimmed without inconvenience. There are a variety of film viewers available, but for most film interpretations, a high intensity viewer with an appropriate mask to accommodate different sizes of film will suffice.

5) A General Radiographic Technique

Radiography may be accomplished on an assembly line basis or it can be usefully employed on a few but highly critical parts. (a) Selecting the Kilovoltage -- Graphs, tables, and slide rules will enable the radiographer to choose suitable kilovoltage for any material and thickness of material to be radiographed. In practice, the lowest possible kilovoltage should always be used in order that maximum subject contrast may be obtained. X-ray machines are more versatile than radioactive sources in this respect because the kilovoltage can be varied within the limits of each machine. Radioactive sources have inherent levels of energy and thus are not as versatile. (b) Time of Exposure -- Radiographic film does not respond to all radiation that strikes it. Since only a small percentage of the radiation is useful in producing an image, it is generally necessary that the exposure time be not less than one minute. In the radiography of light weight metals, such as aluminum and magnesium, it is even more important to use an exposure time of at least one minute. However, when radiography is not intended to reveal the quality of the base material, but rather internal circuitry, shorter exposure times may be used. Excessive exposure times may cause film to be darkened by scatter radiation. The three factors that govern exposure time are milliamperage or source strength, time of exposure, and film to source distance. (c) Film to Source Distance of Radiation -- In any radiographic technique, the greatest possible distance should be used to obtain the maximum detail definition. However, the distance should not be so large that exposure time becomes excessive. The direction of radiation should be perpendicular to the surface of the object except in those cases where more information can be obtained by changing the direction of radiation, and in the fillet type welds. (d) Location and Identification of Film -- Stored film should be located as close as possible to the

object being radiographed. Each film should be marked with images of lead numbers or letters placed adjacent to the object being radiographed. (e) Sensitivity -- Sensitivity is indicative of the clearness of the image on the film. Penetrameters are used as a check of sensitivity. Penetrameters are made of the same material as the object being radiographed and are placed on the side of the object closest to the radiation. (f) Density of X-ray Film-- Density is the darkness or the degree of blackening of the radiographic film. Density does not have any units of measure as such, but is a measure of the amount of light that passes through an exposed film. Radiographic film with a density range between 1.5 and 3.5 can be interpreted with standard viewers. (g) Selection of Film -- Defects may range in size, number, and orientation. Therefore, to locate small defects, a fine grained high contrast film should be used. Other films with higher speed and coarser grain may be used whenever the standards are less critical.

6) Interpretation of X-ray Film

The interpretation of X-ray film is a skill that can be acquired only by experience and a good knowledge of the quality standards required for the end use of each particular item inspected. Because it is nearly impossible for any item to be perfectly sound, it is then necessary for the radiographer to judge the degree of imperfection to determine if standards are met. It is important that reference radiography and specifications are available to aid the radiographer.

7) Safety

In permanent installation, the hazard of exposure to radiation can be solved by shielding. Generally, for low kilovoltage radiography, it is sufficient to line the control panel and the walls with lead sheets. For a 250-kilovolt machine, 1/4-inch thick lead will suffice. For installations involving 1 MEV and higher radiography, it is more practical to erect concrete walls of sufficient thickness to protect operating personnel. Thirty inches of concrete are sufficient for a 2-MEV machine. In the field, protection can best be accomplished by distance. Proper barricades and signs should be erected and warn personnel of dangers in the area. Portable lead shields may be used as necessary. There are a variety of survey meters available for detecting radiation. At least two meters should be in use during any radiographic operation. Survey meters with a range from 0 to 2.5 roentgens should be adequate. Survey meters should always be used in place of Geiger counters since a Geiger counter will not operate in a high radiation field.

8) Materials

Film should be stored under conditions specified by the manufacturer. Most brands of film are standardized. The source of supply and delivery dates are important since expired film should not be used. Chemicals used in the developing process should be replaced or replenished as specified by the manufacturer. Storage of chemicals should also be in accordance with manufacturer's specifications. It is recommended that a single brand of film and chemicals be used in the developing process. The selection of casettes depends upon the specific item and configuration of the item being radiographed. Some casettes are more flexible than others and the method of fastening may vary. Casettes having snap-on fasteners are preferred by most radiographers. Intensifying

lead screens can vary in thickness according to preference, but they are usually 0.005 and 0.010 inches thick in the front and back respectively. Screens should be cleaned with a damp cloth to remove foreign matter that might scratch the film.

9) Workmanship

All radiography should be accomplished with film that is not defective in any manner and the finished X-ray film should be free of marks which may interfere with its interpretation.

10) Identification of Parts

All items radiographed should be identified on the X-ray film with either lead figures or an appropriate marker. Proper identification facilitates matching the film with the radiographed item. Consult References M through P of this MTP for further information concerning film radiography.

APPENDIX B

A General Procedure for Investigation of Service Metal Failures

a. Principle and Basis

The procedures described will serve as a guide for service metal failure investigations. The investigation will not be based on a statistical quantity of samples but will be concerned with the isolated causes which occur. The emphasis is on the approach rather than on specific detailed procedures. Consult References Q through S of this MTP for further information regarding the investigation of failures in service metals.

b. Considerations

1) Design Factors

Stresses induced in a moving part by other parts with which it comes into contact can be a combination of direct stress, bending, and torsional stress. The final design of a component is a compromise of the stresses to which it will be subjected. Every component should be examined for possible areas of stress concentrations such as sharp fillets, key ways, and oil holes. Excessive stress concentrations can initiate minute fractures which could result in the total fracture of a component.

2) Mechanical Considerations

Components, whether wrought or cast, generally experience maximum stresses on the surface. A component should be examined for surface defects such as roughness, scratches, cuts, and tool marks that may be sources of eventual fracture.

3) A General Investigation Technique

It is important that the investigator become thoroughly familiar with the function of the failed component. The investigator should observe similar components in actual operation and whenever possible, should examine components which have been in operation for an equal time. Determine the actual field conditions under which the component failed, such as temperature, contamination (sand, corrosive chemicals, etc.), method of fastening (rivets, welding, bolts), continuous or intermittent operation. Determine how the component was fabricated and what properties are advantageous to such a process. The fabrication methods are numerous, each having advantages and disadvantages as well as economic implications. The quantity of a single component to be fabricated may in itself determine how the component will be manufactured. Also determine the type of surface treatment, if any, on the failed component, such as shot peening, plating, and surface hardneing. Laboratory tests on failed components must follow general guidelines because of the many variables in any failure analysis. The size and number of specimens available may also influence how extensive the investigation may be. A general systematic plan for investigation is to determine the type of fracture, such as tension, shear, fatigue, compression or torsional. Examine microstructure to determine proper heat treatment, carburization or decarburization, microhardness, grain orientation and size, and thermal changes (welding). Examine microstructure to

determine inclusions, flow lines in wrought components, and standard hardness. Conduct mechanical tests to determine tensile strength, elongation, yield point, reduction in areas, and impact values. Analyze specimen for element composition. Conduct radiographic and ultrasonic tests to determine the nature of internal defects, such as cracks, porosity, and inclusions.

4) Specifications

It is essential that manufacturers' specifications be available to assist the investigator in his findings and conclusions.