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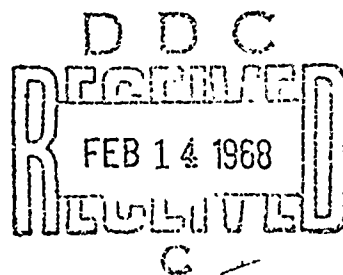
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PROPELLANT SPECIFICATIONS PREPARATION AND USE

BY
FORREST S. FORBES

TECHNICAL REPORT AFRPL-TR-67-256

OCTOBER 1967



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AIR FORCE ROCKET PROPULSION LABORATORY
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

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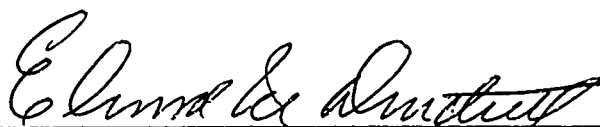
FOREWORD

This report has been prepared to describe some of the "behind the scenes" activity of rocket propellant specification preparation, and to emphasize the proper role of the specification in the control of propellant quality. Although specification preparation is somewhat routine, and represents only a small portion of the Air Force Rocket Propulsion Laboratory's overall propellant effort, it is one of the major end products of the Laboratory's extensive research and development program.

Much of the difficulty that arises in the field due to poor propellant quality can be traced to the improper use of the propellant specifications. It is hoped that this report will contribute to improved communication on this subject among the laboratory scientists, logistics personnel, quality control inspectors, and the ultimate consumer.

A condensed version of this report was presented by the author at the Sixty-First National Meeting of the American Institute of Chemical Engineers on 21 February 1967 at Houston, Texas. The approval for public release is included within this report.

This report has been reviewed and approved.



ELWOOD M. DOUTHETT

Colonel, USAF

Commander, Air Force Rocket Propulsion Laboratory

ABSTRACT

The specification is the key document that establishes the technical requirements for propellant procurements. These requirements are dictated by the needs of the various rocket applications, with due consideration given to the manufacturing ability of the suppliers. The specification also contains quality assurance provisions that detail the sampling techniques and analytical procedures to be used to insure that the propellant conforms to the specified requirements.

Propellant specifications are prepared by the cognizant technical laboratory, and authenticated annually for the procuring activity. Specifications are continually revised and updated as required by the rocket community. Examples are presented showing the rationale for the establishment of specific requirements and analytical methods for several current propellants.

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SECTION I

INTRODUCTION

The Air Force currently budgets more than \$2,000,000 annually for liquid propellant development. The Army, Navy and NASA also spend appreciable funds to improve the properties or characteristics of propellants. The end result of these efforts is a document called a specification or, when prepared by one of the three services, a "Mil Spec".

The specification is intended primarily for use in procurement. It clearly and accurately describes the essential technical requirements for the propellant, and includes the procedures by which it will be determined that these requirements have been met.

The details of specification preparation, coordination and format are prescribed by Defense Standardization Manual, M 200B, "Standardization Policies, Procedures and Instructions".* The considerations for the establishment of the technical requirements and test methods are many and varied. Essentially, each requirement for each propellant is an individual determination. The general criteria and the rationale for the selection of specific requirements and analytical methods for several selected propellants are discussed in later sections of this report.

Throughout the entire logistics cycle of propellant manufacture, transportation, storage, servicing operation, and ultimate use, propellant quality can be affected. The military specification is designed to control the propellant at the point of government acceptance, usually

* Issued by Office of the Assistant Secretary of Defense Installations and Logistics.

at the place of manufacture or in the shipping container as it leaves the vendor's plant. Specifications are not designed for control of propellant quality during the various logistic handling and transfer operations, but in practice, are often so used. However, the specification limits of certain propellants are defined so as to allow for some deterioration in propellant quality during a normal cycle between procurement and ultimate use.

All propellant specifications now in use by the Department of Defense are of the military series, although some Federal specifications have been used in the past. The reason for separate "Mil Specs" is that chemicals that have multiple usage, such as ammonia (which is also used for refrigeration) or oxygen (used for aircraft breathing systems), must meet entirely different requirements for different applications. Little, if any, simplification results from placing such varied requirements and quality assurance provisions into one document. In fact, confusion usually results. The coordination, revision, and authentication procedures become burdensome when several offices representing different technical specialties are involved in furnishing engineering support to the procuring activity.

The current military specifications for liquid propellants are listed in Table I.

Although this report is limited to a discussion of liquid propellant specifications, the general philosophy and description of procedures and format apply essentially to all commodity specifications prepared by the Department of Defense.

TABLE I LIQUID PROPELLANT SPECIFICATIONS

TABLE I LIQUID PROPELLANT SPECIFICATIONS

<u>Spec. No.</u>	<u>Date</u>	<u>Propellant</u>	<u>Status</u>
1. MIL-P-7254E(1)	8 Sep 59 (1) 17 Aug 61	Nitric Acid	Rev F planned 3rd Qtr FY68
2. MIL-N-8722A(USAF)	6 Feb 59	n-Propyl Nitrate	To be cancelled upon approval of Army-Navy
3. MIL-P-8845 (ASG)	20 Apr 59	Ethylene Oxide	Rev planned 4th Qtr FY68
4. MIL-P-16005D	18 Mar 65	Hydrogen Peroxide	Rev E due 1st Qtr FY68 (will include 98%)
5. MIL-P-23686A(1)	15 Apr 64 (1) 15 Mar 65	Mixed Amine Fuel, MAF-3	
6. MIL-P-23741A(1)	15 Apr 64 (1) 15 Mar 65	Mixed Amine Fuel, MAF 1	
7. MIL-P-25508D	16 Mar 62	Oxygen	Rev E planned 1st Qtr FY69
8. MIL-P-25576C	10 Feb 67	Kerosine	(Former Name: RP-1)
9. MIL-P-25604C	20 May 63	uns-Dimethylhydrazine	Rev D due 4th Qtr FY 68
10. MIL-P-26536B	13 Mar 64	Hydrazine	Rev C due 3rd Qtr FY 69
11. MIL-P-26539B	15 Oct 65	Nitrogen Tetroxide	Rev C planned 4th Qtr FY69
12. MIL-P-26694B	30 Jun 67	uns-Dimethylhydrazine-JP-4 (40% UDMH 60% JP-4)	
13. MIL-P-27201A	1 Sep 64	Hydrogen	Rev B planned 1st Qtr FY69
14. MIL-P-27401B	19 Sep 62	Pressurizing Agent, Nitrogen	Rev C planned 1st Qtr FY69
15. MIL-P-27402A	24 Feb 67	Hydrazine, uns-Dimethylhydrazine (50% N ₂ H ₄ - 50% UDMH)	
16. MIL-P-27403(USAF)	11 Mar 63	Pentaborane	Inactive - To be cancelled
17. MIL-P-27404	3 Apr 62	Monomethylhydrazine	Revision due 3rd Qtr FY 69
18. MIL-P-27405(USAF)	Not issued	Fluorine	Spec. due 2nd Qtr FY68
19. MIL-P-27406(USAF)	9 May 66	Ammonia	
20. MIL-P-27407(1)(USAF)	4 Feb 64 (1) 8 Jan 65	Pressurizing Agent, Helium	
21. MIL-P-27408(1)(USAF)	31 Jul 64 (1) 5 May 67	Nitrogen Tetroxide- Nitric Oxide (90% N ₂ O ₄ - 10% NO)	Rev. A due 3rd Qtr FY68
22. MIL-P-27411(USAF)	Not issued	Chlorine Trifluoride	Spec due 2nd Qtr FY68

TABLE I LIQUID PROPELLANT SPECIFICATIONS (CONT'D)

<u>Spec. No.</u>	<u>Date</u>	<u>Propellant</u>	<u>Status</u>
23. MIL-P-27412(USAF)	12 Jan 65	Aluminum-Hydrazine	Rev A planned 3rd Qtr FY 68
24. MIL-P-45700A	10 Mar 61	Aniline-Furfuryl Alcohol	
25. MIL-P-45702A	25 Nov 60	Furfuryl Alcohol	Rev A planned 3rd Qtr FY 68
26. MIL-P-45734(ORD)	24 Mar 61	uns-Dimethylhydrazine-Jet Fuel (17% UDMH - 83% JP-4)	To be cancelled. Incorporated in Rev B of MIL-P-26694A(USAF)
27. MIL-P-27413(USAF)		Chlorine Pentafluoride	Spec due 4th Qtr FY 68
28. MIL-P-27414(USAF)		Mixed Hydrazine Fuels	New Spec forecast 2nd Qtr FY68 - Possibly 2 Specs.
29. MIL-O-81399(AS)	13 Sep 66	Chlorine Trifluoride	To be replaced by MIL-P-27411

SECTION II

THE ANATOMY OF A SPECIFICATION

Specifications are identified by number and title, e. g. , MIL-P-27201A, Propellant, Hydrogen. All military specification numbers begin with "MIL", followed by the first letter of the specification title, which for liquid rocket propellant specifications is the letter "P". The number assignment is prescribed by a system that precludes duplication of numbers for any military specifications or standards whether prepared by the Army, Navy, Air Force, or the Defense Supply Agency. A suffix letter is used as a revision indicator. A parenthetical numerical indicator is used to identify an amendment. An amendment is a short-form revision and, when approved, becomes a part of the basic specification as an attachment. When more than one amendment is required on a specification, the latest will contain all previous amendment data.

All liquid rocket propellant specification titles begin with the word "Propellant". It is standard practice to follow the basic noun in the title with the proper chemical name of the material insofar as possible. The title often becomes a little unwieldy as is the case of MIL-P-27402(USAF): Propellant, Hydrazine-unsDimethlhydrazine (50%/N₂H₄-50% UDMH), but it is not ambiguous.

A specification that has not been fully coordinated among the Army, Navy and Air Force is a "limited coordinated specification". It is identified by the symbol designation of the preparing agency. MIL-P-16005D, for example, is a fully coordinated specification, while MIL-P-27405 (USAF) has received coordination only within the Air Force.

A specification consists of six sections, entitled and numbered as follows:

1. SCOPE
2. APPLICABLE DOCUMENTS

3. REQUIREMENTS
4. QUALITY ASSURANCE PROVISIONS
5. PREPARATION FOR DELIVERY
6. NOTES

The information contained in these sections is described below.

1. SCOPE

Section 1 is a brief statement sufficiently complete and comprehensive to describe generally the item covered by the specification in terms that may be easily interpreted by manufacturers, users, suppliers and others familiar with the terminology and trade practices. A listing of the different types or grades is presented as applicable. The term "type" implies chemical or physical differences in like items which are for different but specific, equally important uses and is designated by a Roman numeral. The oxygen specification, MIL-P-25508D, for example, covers two types: Type I, Gaseous; Type II, Liquid. The term "grade" is used to imply differences in quality and is designated by capital letters, thus, "Grade A", "Grade B", etc. This term has seldom been used in propellant specifications.

2. APPLICABLE DOCUMENTS

Only those documents identified and cited in Sections 3, 4, and 5 of the specification are listed in Section 2. References are confined to documents currently available at the time of issuance of the specification. Documents often referenced are Military Standards for marking for shipment and storage, Federal Test Methods or ASTM Standards, specifications for shipping containers and samplers, and ICC regulations. Nongovernment documents may be referenced when such documents are widely recognized by industry and are accepted by the using governmental agencies; care must be taken, however, to assure the availability of copies and prior approval of the copyright owner.

3. REQUIREMENTS

Section 3 is the heart of the specification. It must state only the actual minimum needs of the government and describe the required propellant in a manner which encourages maximum competition, and eliminates, insofar as is possible, any restrictive features which might limit acceptable offers to products of one or of a relatively few suppliers. The essential requirements and description applying to the chemical and physical characteristics are stated in this section. The minimum standards of quality and workmanship which the commodity must meet to be acceptable are also stated as definitively as practicable. The requirements should also be so worded as to provide a definite basis for rejection in those cases where the quality and workmanship are such that the item is unsuitable for the purpose intended. (The processes for establishing the technical requirements are discussed under Section IV of this report.)

4. QUALITY ASSURANCE PROVISIONS

This section includes all of the examinations and tests to be performed in order to determine that the propellant offered for acceptance conforms to the requirements in Section 3 of the specification.

The sampling plan is described, identifying the lot, the number and size of each sample, and the mode of obtaining a sample.

Detailed descriptions of the tests, equipment, reagents, analytical methods, and criteria for determining requirement conformance are presented in this section. Standard test methods are usually included only by reference.

5. PREPARATION FOR DELIVERY

The applicable requirements for packaging, marking for shipping, and hazardous-substance labeling are covered in Section 5 of the

specification. The containers (drum, cylinder, tank car, etc.) and materials thereof which are suitable for the specified propellant are also identified.

6. NOTES

This last section of the specification contains information of a general or explanatory nature only. It may contain miscellaneous information relative to the intended use of the propellant, detailed ordering data to be incorporated into invitations for bids, definitions, NASA coordination, etc. The information contained in Section 6 is, contractually, nonmandatory upon the contractor.

7. AMENDMENTS

Amendments are separately issued documents carrying the same headings and titles, and the symbols and numbers of the specification with which they are associated.

Amendments are issued when a significant change, addition, or correction to the specification has been made, but a complete revision of the specification is not warranted. Amendments are prepared and coordinated in the same manner as specifications.

SECTION III

THE MECHANICS OF SPECIFICATION PREPARATION

When a new propellant is made available to the rocket community, the first specification, usually an informal one, is often based on the product as produced by the chemical manufacturer. The rocket industry requirements are certainly considered, but may not be well defined at this point in time. A limiting factor in establishing firm requirements is often a lack of good analytical procedures. Although considerable help is received from industry in this area, it is preferable that the test procedures be finalized in the laboratories of the preparing service. This enables them to be in a better position to give technical support to government quality-control inspectors, and to better serve as a referee should conflicts arise. When reasonable requirements and adequate analytical procedures have been identified, we have the makings of a Mil Spec, and a draft is prepared. It is circulated to all interested organizations for comments. When all comments have been reviewed and differences resolved, the specification is formally coordinated and issued. As new requirements are identified, or better analytical procedures are developed, the cycle starts over and a revised specification or an amendment is issued. Figure 1 depicts the specification preparation cycle.

Propellant specifications are dynamic by intent. The preparing activity welcomes suggestions that will improve and simplify the procurement requirements of the propellant. All specifications, including the limited coordinated documents, are reviewed by the propellant manufacturer, engine manufacturer, missile system contractor, the three Services, and NASA. Other governmental agencies are consulted as required.

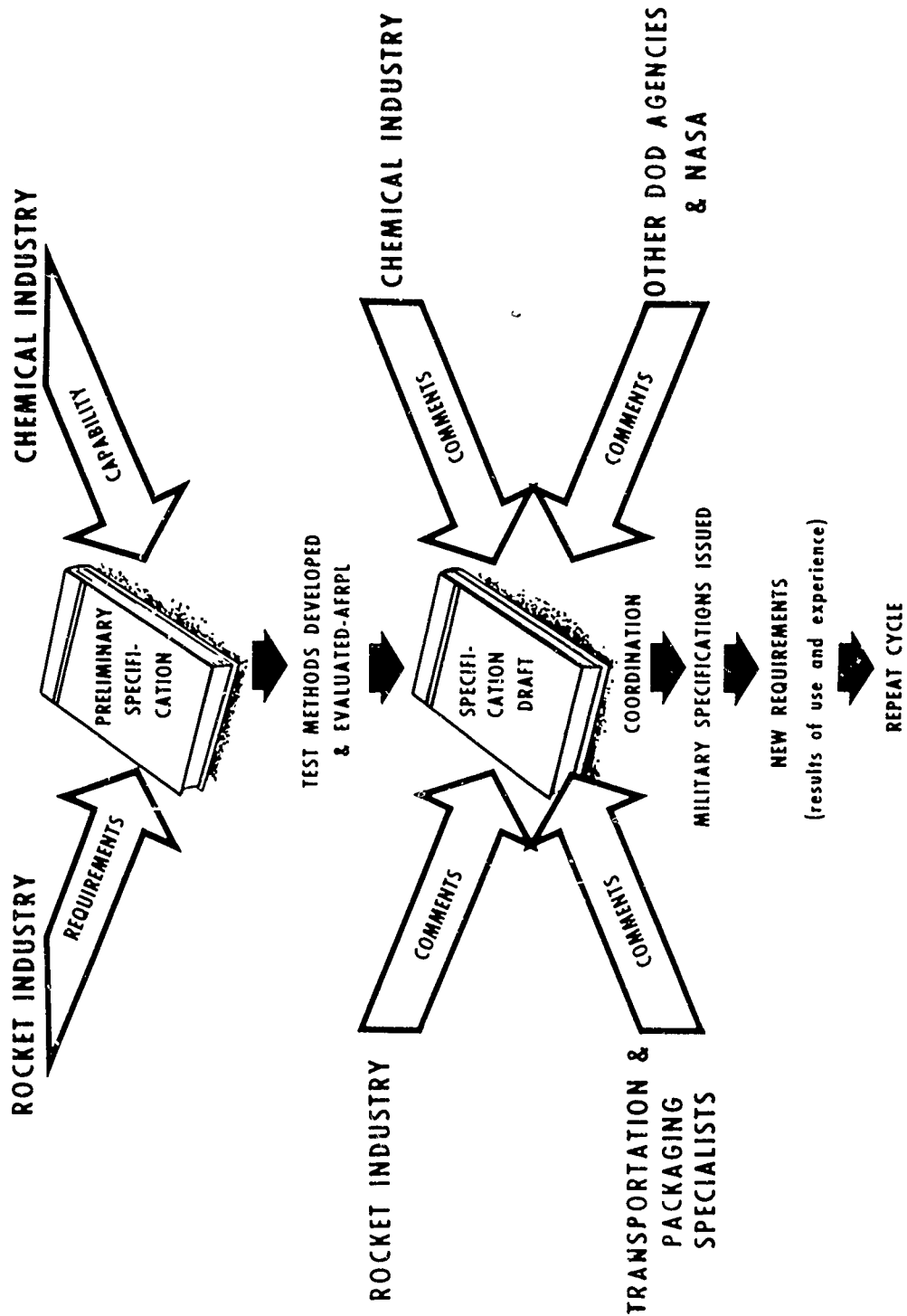


Figure 1. Specification Preparation Cycle.

SECTION IV

ESTABLISHING SPECIFICATION REQUIREMENTS

The establishment of detailed requirements for Section 3 of the specification is an important and difficult task, carrying considerable responsibility. The propellant quality established must meet the needs of the specific system, e. g. , the Titan II Bullpup B, Atlas, Centaur, Agena, Saturn, etc. Several criteria are used to specify propellant quality. Performance is usually paramount, thus nonenergetic materials or diluents should be kept to a minimum. Impurities that may present a hazardous condition (e. g. , hydrocarbons in liquid oxygen) must be controlled. Impurities that cause decomposition or corrosion cannot be tolerated especially for "storable" propellants such as nitrogen tetroxide for use in the Titan II. However, the requirements cannot be too stringent if adequate quantities of propellant at a reasonable cost are to be made available.

When a new propellant is made available to the rocket community, the first specification (usually an informal one) is based on the product as it comes from the manufacturer. Inert, or nonenergetic impurities are generally limited to 3 and sometimes 5 wt. percent. If the propellant is cryogenic, condensable materials may be controlled to 0.5 wt. percent or lower.

As experience is gained during rocket engine development, refinements in propellant quality are made. An impurity that affects the ignition or combustion process may be found or suspected. Sometimes additives may be tried to promote improved performance. Controlled storage tests may indicate poor stability, resulting in corrosion, gas evolution, change in chemical composition or other undesirable effects. The propellant manufacturer also strives to improve his products, and as his production and facilities increase, higher purity often results.

Thus, from the considerations of storability, performance, safety, and manufacturing capability, a formal specification emerges and is published as a limited coordinated military specification. Other factors often enter into the considerations. For example, there must be analytical procedures capable of measuring the propellant assay and controlled impurities or constituents with the desired precision. It is not reasonable to set a limit for some impurities of 0.1 wt. percent, when the analytical method will detect only 0.5 wt. percent. Although a value is occasionally established on the grounds that it appears "reasonable" or is "believed" to be significant, this is usually limited to physical properties which are relatively simple to measure. Some propellants are subject to inherent decomposition, degradation, or contamination during the time they are in transit between the manufacturer and user. When this can occur, the specification must be made more stringent than required by the user to allow for such changes.

Changes to specifications can originate from many sources:

- a. The preparing activity may find new requirements as a result of continued surveillance, additional experience, or long-term-storage studies.
- b. A manufacturer may change his process, resulting in new levels of purity, or introduction of different impurities.
- c. New applications resulting in additional or more stringent requirements may develop.
- d. Advances in analytical chemistry can result in quality-control improvement.

At times it becomes very difficult to define the chemical composition of a propellant sufficiently to assure proper quality control. For these instances a performance test can be specified, and those manufacturers' products that pass the test can be placed on a Qualified Products List (QPL). This, of course, requires the establishment of a test procedure and fabrication of equipment that will simulate actual

operational usage. Unfortunately, it is extremely difficult to achieve satisfactory simulation in a scaled laboratory reactor, and correlation with actual operation must be established. The use of actual operational systems hardware will assure accurate evaluation, but it is very costly, and the use of propellant in different systems compounds the expense. A manufacturer whose product is included in a QPL must assure that he will not change his process or formulation procedures. If a change occurs, the product must be reexamined. The QPL procedure has been used for many years for such materials as aircraft lubricants, but has never been used for rocket propellants although it was strongly considered for hydrogen peroxide.

Current military propellant specification test requirements are summarized in Table II. The following discussion describes how some of these requirements evolved for several selected propellants.

1. FUMING NITRIC ACID

In the early 1950's, white fuming nitric acid (WFNA) was the commonly used type. It had two major deficiencies: it was very corrosive, resulting in contamination; and it was unstable, resulting in composition change and gas evolution on storage. Through research studies it was noted that the addition of 2 to 3 wt. percent water and 12 to 13 wt. percent NO_2 resulted in a very stable material (1,2). The addition of hydrogen fluoride to the nitric acid was found to reduce corrosion of both aluminum and stainless steel containers by a factor of 10 or greater (3). The optimum amount of HF providing the greatest corrosion reduction was 0.5 wt. percent. HF forms a passive fluoride film on the container surfaces (4). Each time the propellant is transferred to a different container, some HF would be consumed. To allow for this loss, the specification requirement was set at 0.6 to 0.8 wt. percent. No method has been found to prepassivate the containers. It appears that an equilibrium is established between the fluoride on the wall and

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS

<u>Propellant & MIL-SPEC No.</u>	<u>Requirements</u>	<u>Control Test</u>
1. Nitric Acid IIIA MIL-P-7254E	Total Solids 0.1% by wt NO ₂ 13-15% by wt HNO ₃ 81.6-84.8% H ₂ O 1.5-2.5% by wt HF 0.6-0.8% by wt	Residue Ignition Ceric Titration NaOH Titration By difference Zr/Al Calorimetric
2. N-Propyl Nitrate MIL-P-8722A	Min Assay wt 95.0% Water max 0.03% by wt Acidity max 0.01% by wt Specific Gravity 1.064±0.002 Index of Refraction 1.398±0.001	Gas Chromatography Karl Fischer Alkali Titration FTM 791 Refractometer
3. Ethylene Oxide MIL-P-8845	Acidity 0.005% by wt Water 0.03% by wt Total Iron 0.1 mg/l Aldehydes 0.3% by wt Chloride 0.2% by wt	Alkali Titration Karl Fischer Colorimeter Iodine Titration Chlorides AgNO ₃ Colorimeter
4. Hydrogen Peroxide MIL-P-16005D	Assay 90.0-91.0% by wt Aluminum max 0.50 mg/l Chloride max 1.0 mg/l Ammonia max 3.0 mg/l Nitrate 3.0-5.0 mg/l Phosphate max 0.20 mg/l Sulfate max 3.0 mg/l Tin 1.0-4.0 mg/l Carbon 200.0 mg/l Evaporation Residue max 20 mg/l Stability max 5.0% loss for 24 hrs at 100°C±1°C Particulate max 1.0 mg/l	Ceric Sulfate Titration Spectrophotometer (color) Spectrophotometer (turbidimetric) Spectrophotometer (color) Spectrophotometer (color) Spectrophotometer (turbidimetric) Spectrophotometer (color) Hydrochloric Titration (combustion) Evaporation Steam Bath Filtration (millipore filter)

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS (CONT'D)

5. Mixed Amine Fuel MAF-3 MIL-P-23686A	Assay UDMH 20.0%±1.0 by wt Assay DETA 80.0%±1.0 by wt Water max 1.0% by wt Particulate max 10.0 mg/l Density 0.912-0.920 at 25°C	KIO ₃ Titration HCl-ethylene glycol-isopropanol Titration Vapor phase Chromatography Filtration (millipore) FTM 791, method 402.2
6. Mixed Amine Fuel MAF-1 MIL-P-23741	Assay UDMH 39.0±1.5 Assay DETA 49.7±0.7 Assay ACN 10.0±1.0 Water max 1.0% by wt Particulate max 0.2 wt% Density 0.869±0.002	KIO ₃ Titration HCl-ethylene glycol-isopropanol Titration Infrared Spectrometry Vapor Phase Chromatography Filtration (millipore) FTM 791, method 402.2
7. Oxygen Type II (Liquid)	Assay min 99.5% by vol when gasified Hydrocarbons max 25 ppm by wt Carbon 66.7 ppm by vol methane Acetylene max 0.5 ppm by wt, 0.62 ppm by vol Moisture max 26.3 ppm by vol when gasified Particulate max 1.0 mg/l	Orsat Hydrocarbon Analyzer, Flame Ionization Ilosvay Accelerated Gravimetric Filtration-millipore
8. Kerosine MIL-P-25576C	Distillation 10% 365°F to 410°F End Point 535°F max Residue max 1.5% by vol Distillation loss max 1.5 by vol Gravity °API min 42.0 Gravity °API max 45.0 Existent gum max 7 mg/100 ml Potential gum max 14 mg/100 ml Sulfur max 0.05% by wt	Method 1001 Method 401 Method 401 Method 3302 Method 3354 Method 5201

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS (CONT'D)

8. (Continued)	Mercaptan-Sulfur max 0.005% by wt Freezing Point max -40°F BTU Heat of Combustion 18,500 157/min Viscosity max 16.5 centistokes at -30°F Aromatics max 5.0% by vol Olefins max 1.0% by vol Smoke Pt., mm, min 25.0 Copper Corrosion max 1 ASTM Water Reaction Flash Pt. min 110°F Aniline Point Particulate max 1.5 mg/l	Method 5204 Method 1411 Method 2502 Method 305 FTM 791 Method 3703 Method 3701 Method 2107 Method 5325 Method 3251 Method 1102 Method 3601 Filtration
9. Unsymmetrical Dimethylhydrazine (UDMH) MIL-P25604C	Assay min 98.0% by wt Transmittancy 90% min Density 0.783-0.786 Melting Point -70°F max Distillation 10% 143°F min Distillation 90% 105°F max Water max 0.3% by wt Particulate max 10 mg/l	KIO₃ Titration Potentiometer Calorimeter FTM 791, Method 402.2 Capillary Tube
10. Hydrazine MIL-P26536B	Assay min 97.5% by wt Water max 2.5% by wt Particulate max 10 mg/l Density 1.002 min at 25°C Density 1.006 max at 25°C	Gas Chromatography Filtration
11. Nitrogen Tetroxide MIL-P-26539B	Assay min 99.5% by wt Moisture Content max 0.10% by wt Chloride as Nitrosyl Chloride max 0.08% by wt Particulate max 10 mg/l Qualitative-homo Color	KIO₃ Titration Potentiometer Gas Chromatography Filtration (millipore) FTM 791, Method 402.2 Total Acidity Water Equivalent (Phase Separation) Spectrophotometer (turbidimetric) Filtration Visual

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS (CONT'D)

12.	UDMH - JP-4 40% 60% (JP-X Type I) MIL-P-26694B	Assay UDMH min 39.0% by wt Assay UDMH max 41.0% by wt Assay JP-4 min 59.0% by wt Distillation 10% 155.0°F max 50% 300.0 70% 370.0 90% 470.0 Density 0.788-0.746 at 25°C No Phase Separation at -65°F max Particulate max 10 mg/l	} KIO ₃ Titration Potentiometer
13.	Hydrogen	Assay min 99.995% by vol Para H ₂ min 95.0% Oxygen max 1 ppm Inert Gas max 48.0 ppm Carbon Gases max 1.0 ppm	FTM 791 Method 402.2 Filtration (millipore) Gas Chromatography In-stream Analyzers Gas Chromatography Gas Chromatography Gas Chromatography
14.	Nitrogen, Press. Agent, Type I MIL-P-2740/B	Assay min 99.9% by vol Hydrocarbons 25 ppm max carbon 58.3 ppm max methane Oxygen max 0.5% by vol Moisture max 26.3 ppm by vol Particulate max 1.0 mg/l	Difference of 100%-impurities Hydrocarbon Analyzer Oxygen Analytical Apparatus (by color) Accelerated Gravimetric Filtration (millipore)
15.	N ₂ H ₄ - UDMH 50% 50% MIL-P-27402A	Assay N ₂ H ₄ 51% by wt Assay UDMH 47% min by wt Water max 1.8% Particulate max 10 mg/l	Acetylation Acetylation Gas Chromatography Filtration
16.	Pentaborane MIL-P-27403 (USAF)	Assay min 99.0% by wt Dissolved Solids max 1.1% by wt Thermal Stability max 1.5% by wt	Freezing Point Method Vacuum Evaporation Apparatus Vacuum Evaporation Apparatus
17.	Monomethylhydrazine (MMH) MIL-P-27404	Assay min 98.0% by wt Density 0.872±0.004 Melting Point max -64.5°F Transmittancy min 90% Particulate max 10 mg/l	KIO ₃ Titration Potentiometer FTM 791 Method 402.2 Capillary tube Calorimeter Filtration (millipore)

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS (CONT'D)

18.	Fluorine (Tentative) MIL-P-27405 (USAF)	Assay min 99.0% CF ₄ + CO ₂ max 0.2 O ₂ + H ₂ max 0.80	By difference Infrared Spectrophotometry Gas Chromatography
19.	Ammonia MIL-P-27406 (USAF)	Assay min 99.5% by wt Moisture max 0.5% by wt Oil max 5.0 ppm	By difference Karl Fischer Evaporation
20.	Helium, Press. Agent MIL-P-27407 (USAF)	Assay min 99.995% by wt Hydrocarbons max 1.0 ppm by vol Oxygen max 1.0 ppm by vol Other Gaseous max 39.0 ppm by vol Moisture max 9.0 ppm by vol	By difference Hydrocarbon Analyzer Oxygen Trace Analyzer Gas Chromatography Accelerated Gravimetric Method and Others
21.	N ₂ O ₄ - NO 90% 10% MIL-P-27408 (USAF) (1)	Assay NO 10.0-11.0% by wt Assay N ₂ O ₄ min 98.8% by wt Water Equivalent max 0.10% by wt Nitrosyl Chloride max 0.080% by wt	Reaction Bomb $\bar{c}$ O ₂ = Weight Increase Acid/Base Titration Water Equivalent Apparatus Spectrophotometer
22.	Chlorine Trifluoride (Tentative) MIL-P-27411 (USAF)	Assay min 99.0% by wt HF max 0.3% Nonvolatile max 0.002% ClO ₂ + ClO ₂ F max 0.2%	By Gas Chromatography Near IR Evaporation Gas Chromatography
23.	Aluminum Hydrazine Gel (Alumazine) MIL-P-27412 (USAF)	Aluminum, 43.0 + 0.5% by wt Hydrazine, 56.0% by wt min Modified polyacrylic acid, 0.30 - 0.35% by wt Water, 0.6 by wt max Density, 1.366 - 1.380 gm/cc Limiting flow characteristics Yield stress, 1400 - 2000 dynes cm ²	CuSO ₄ Titration Gas Chromatography Precipitation with HCl Gas Chromatography Oil Pycnometer Brookfield viscometer Rising sphere rheometer

TABLE II
SUMMARY SPECIFICATION REQUIREMENTS (CONT'D)

24.	Aniline-Furfuryl MIL-P-45700A	Assay Furfuryl Alcohol 46.3-46.7% by wt Assay Hydrazine 6.8-7.2% by wt Water max 1.5% Other max 0.7% Aniline Remainder Specific Gravity 1.070-1.085 at 60° F Melting Point max -42.8°C	MIL-P-45702A MIL-P-26536B MIL-A-10450 Method C of ASTM D 891-51
25.	Furfuryl Alcohol MIL-P-45702A	Furfuryl max 0.7% Cloud Point max 22°C Refractive Index 1.486 ± 0.003 at 20°C Specific Gravity 1.135 ± 0.006 at 20°C	Iodine Titration Refractometer Hydrometer
26.	UDMH - JP-4 17% 83% (JP-X Type II) MIL-P-26694B (Formerly MIL-P-45734 ORD)	Assay UDMH 17 + 0.3% by wt Assay JP-4 83 + 0.3% by wt Distillation 10% 245°F max 50% 340 90% 465.8 Density 0.792 - .749 at 25°C Particulate max 150 mg/l	KIO ₃ Titration Potentiometer KIO ₃ Titration Potentiometer FTM 791, Method 402.2 Filtration (millipore) Gas Chromatography Near IR
27.	Chlorine Pentafluoride (Tentative) MIL-P-27413 (USAF)	Assay 99.5% by wt min HF 0.2% by wt max	Gas Chromatography Near IR
28.	Mixed Amine Fuels	Not Defined	
29.	Chlorine Trifluoride	Assay min 99.0% HF 0.4% max ClO ₂ F + ClO ₂ 0.2% max ClF ₂ , F ₂ , Cl ₂ 0.4% max	Gas Chromatography Near IR Gas Chromatography

the fluoride in the nitric acid, which is disrupted upon transfer and must be reestablished.

The resulting nitric acid mixture was called inhibited red fuming nitric acid (IRFNA) and was designated Type IIIA in the specification (the "A" signifying the HF additive). It was recommended for all new applications. Some existing applications continued to use WFNA, which was designated Type I. Types II ($6\frac{1}{2}\%$ NO_2) and IV (22% NO_2) were used briefly for special systems but were dropped as soon as the stability of the Type IIIA was firmly recognized by the industry.

A problem arose with the use of IRFNA in the Agena engine. Solids were found plugging the gas generator screens. The Agena project personnel requested that the permissible solids content of the IRFNA be reduced from 0.10 to 0.04 wt. percent. This low value is extremely difficult to maintain because of the corrosive nature of IRFNA, thus it is not a practical operational requirement. Special container selection, cleaning and surveillance is required, and its storage life is short.

For these reasons it was decided not to change the Type IIIA specification, but to add a new type, IIIB, for those applications requiring extra low solids content and which could tolerate the special handling procedures required.

2. HYDROGEN PEROXIDE

Hydrogen peroxide (H_2O_2) is a monopropellant that can be catalytically decomposed, producing water (steam) and oxygen. Impurities that promote decomposition must be avoided if good storage life is to be obtained; however, stabilizers which are also catalyst poisons cannot be tolerated if catalyst packs are not to fail prematurely.

Hydrogen peroxide stability is controlled by limiting the active oxygen loss, not by limits on the impurities. The maximum active

oxygen loss permitted is 5% measured over a 24-hour period at 100°C, and provides reasonable stability. The stability of product from current production is well within this limit.

Impurities that affect catalyst pack performance were identified under an extensive Navy Program (5), and these are listed in the specification. Chloride ion was found to cause corrosion of the container, thus it is controlled. Further corrosion inhibition is afforded by the addition of nitrates. Phosphate is a severe catalyst poison. Since it cannot be entirely eliminated, tin (as stannous chloride) is added to offset its effects, hence the minimum limit on Sn. The effect of carbon has been the subject of considerable speculation. Some carbonaceous materials are known to be more harmful than others, but it is extremely difficult to identify them in the H_2O_2 . Catalyst pack failures in the X-15 research aircraft precipitated an intensified effort to determine the adequacy of the carbon limit of 200 ppm. Hydrogen peroxide is currently made by three different processes (electrolytic, propane oxidation, and the modified anthraquinone), each leaving a carbonaceous residue of a different composition and amount. Extensive evaluation and analysis failed to indicate any performance difference among the products of these three processes (6). Consideration was given to the establishment of a Qualified Products List. A performance test using a small laboratory reactor was evaluated, but it would not correlate with X-15 experience, and was subsequently dropped. The X-15 problems were later traced to faulty catalyst packs, which points up the necessity of identifying the actual failure mode before accusing poor propellant quality.

3. LIQUID OXYGEN

The prime requirement for oxygen purity is based on safety. Hydrocarbons in oxygen produce a very hazardous condition (7). Impurities that have low solubilities are especially undesirable since they can act as ignition sources. Acetylene is limited to 0.5 ppm

maximum for this reason. During transfer and storage considerable oxygen boils off (8). This causes the impurities to concentrate and to precipitate if their solubilities are reached.

4. AMMONIA

The purity of ammonia produced in the United States is consistently high. The Air Force has found it necessary to control only the oil and moisture content. Excessive oil in ammonia will deposit in engine passages and on the ignitors and can cause malfunctions. Water can cause corrosion.

The Federal Specification 0-A445a, covering refrigeration grade ammonia, was previously used. It controlled pyridine, naphthalene, and hydrogen sulfide, which were found not to affect rocket engine performance in the quantities normally present. Oil was not controlled. A military specification was therefore prepared that was more responsive to the rocket industry, and did not contain any unnecessary requirements.

5. CHLORINE PENTAFLUORIDE

The specification for this material is in preparation. The purity of the material first produced in a pilot plant was better than 97 percent. This purity is very high for initial pilot-plant production of a new compound. With continued process experience, the purity of ClF_5 has increased. The requirements of the present interim specification call for 99.0 wt. percent assay; however, all of the material from current production is considerably better. A value of 99.5 wt. percent for the assay is under consideration for the Mil Spec. Thus, for chlorine pentafluoride, the specification values will be based on the manufacturer's present quality.

Of the known impurities, HF is the most undesirable because it is an inert from the performance standpoint. It will be controlled at a

value of between 0.1 to 0.5 wt. percent. The other impurities, Cl_2 , ClF_3 , and ClO_2 , that would be present in small amounts (less than 0.5 wt. percent for all) are of no concern at this time.

SECTION V

SELECTING ANALYTICAL PROCEDURES

The requirements delineated in a specification have little meaning unless they can be measured with the necessary accuracy and preciseness. The development and evaluation of suitable test methods is usually the most difficult and time-consuming part of specification preparation. The Quality Assurance Provision section of the specification which includes these methods represents the bulk of the specification. The specification must present the procedures in sufficient detail so that they can be performed by any reasonably skilled analytical chemist in an average control laboratory. Analytical methods that involve considerable specialized experience or are unduly subject to personnel error are to be avoided. Methods that require highly specialized or costly equipment are not desirable since the government must, either directly under cost reimbursable contracts, or through higher propellant price, pay for such items. Government quality-control laboratories must also, of course, acquire whatever equipment is specified.

Initial test methods are usually developed by the organization that first synthesized or formulated the propellant. As samples and evaluation quantities are made available to the rocket community, additional personnel become involved with analysis. Since propellant quality can be highly variable in the early stages of its development, many analyses of the propellant are made, primarily to determine quality for correlation with performance data (rather than for quality control). In these early stages, most of the analyses are performed by research personnel of the propellant manufacturer, rocket engine contractor, or the cognizant government laboratory; and these highly skilled people often develop refined or superior procedures.

The specification-preparing activity collects all available methods and evaluates them for simplicity and accuracy. The methods may be modified, or new ones developed if necessary. Most of the evaluation is performed by government personnel in the preparing agency's laboratories for two major reasons:

- a. Conflicts that arise between the propellant manufacturers and the rocket engine developer can be resolved more equitably.
- b. The preparing activity is the organization that receives the technical questions on the procedures, and chemists who have personal experience in performing the various procedures can give better technical support.

The preparing activity works closely with industry to obtain quality assurance provisions that are satisfactory. Many difficulties and differences arise which are settled by concentrated efforts. Everyone realizes that such provisions must be adopted, and if not satisfactory, they will be a continual irritation to all concerned. Personnel often visit other laboratories to resolve differences by working with others who are perhaps more experienced, or who have developed better techniques. "Round Robin" samples are another means of checking the results of different laboratories (but, of course, one must assure that the propellant doesn't change during such a laboratory interchange). The resulting procedures are written to provide stepwise instructions for the analyses, spelling out volumes and weights of materials to be used as well as phenomena to be observed, such as color changes. All required reagents and equipment are identified. If calibration charts or calculation formulas are needed, they are also included.

The test methods used in current specifications are summarized in Table II. The following discussion describes some of the analytical methods used, and the rationale for their selection.

1. NITROGEN TETROXIDE (N_2O_4)

N_2O_4 Assay - The standard acid-base technique is used. An ampoule of N_2O_4 is broken in a solution containing excess sodium hydroxide, and back-titrated with nitric acid to the phenolphthalein end point. This is a straightforward procedure that any chemist can use and obtain the required accuracy.

Moisture Content - This is determined by evaporation of the N_2O_4 to a cloudy end point, holding the temperature of the sample at 28° to 32°F . The moisture content is calculated by the following formula:

$$\begin{array}{l} \text{moisture content} \\ \text{(percent by weight)} \end{array} = \frac{\text{volume at end point}}{\text{initial volume}} \times 1.6$$

This gives a relative, not absolute value for the amount of water present. Other methods considered use expensive instruments (e. g., NMR spectrography), but the increased precision does not warrant the higher cost.

Nitrosyl Chloride (NOCl) - The chloride ions in the N_2O_4 are reacted to silver chloride. By using a filter photometer and a calibration curve, the chlorine ion content is determined. However, since the chloride is present in the N_2O_4 as nitrosyl chloride, the NOCl content is determined by calculation.

2. THE HYDRAZINES: HYDRAZINE (N_2H_4), MONOMETHYLHYDRAZINE (MMH) AND U-DIMETHYLHYDRAZINE (UDMH)

N_2H_4 , MMH or UDMH Assay - This is determined by titration with potassium iodate (KIO_3), a direct and well-accepted method. Gas chromatography (G. C.) is also a good technique for this parameter. Since the specification is being revised to incorporate the use of G. C. for the water-content measurement, it may be desirable to use it for the assay as well.

Water Content - Gas chromatography is the technique now used; it provides for a direct determination (9). This method is not yet incorporated into the specifications, but it has been provided to the procuring activity, and it is used for all current procurements. The old method incorporated in the hydrazine and MMH specification yielded water content by difference. This is very inaccurate, especially since all soluble impurities would be included. Water content in UDMH is determined by distillation. This method also gives a direct determination which is superior to that obtained by difference. The distillation method was already developed for UDMH at the time this specification was written. Subsequent studies indicated the superiority of the gas chromatographic technique, which will be used in the revised specifications of these three fuels, and any mixtures thereof.

3. NITRIC ACID

HNO₃ Assay - Standard titration using sodium hydroxide. Simple and accurate.

NO₂ Content - Standard ceric sulfate titration.

Water - Obtained by difference. This is a grossly inaccurate method and is subject to considerable technician error, but a better method is unknown.

Hydrogen-fluoride - Colorimetric method using zirconium-alizarin reagent to complex the fluoride ion. A standard fluoride solution is used to prepare a calibration curve. This is an easy method. Other methods are available and are referenced in the specification, but offer no advantage.

4. HYDROGEN PEROXIDE

H₂O₂ Assay - Standard titration with ceric sulfate to a ferroin end point. This is a straightforward method not requiring expensive instrumentation.

Ions - Al, Cl, NH₄, NO₃, PO₄, SO₄, are all determined by spectrophotographic absorbance. This is the simplest, most accurate method for identifying trace quantities of these ions, although it is very time-consuming for occasional analysis. Tin is determined by polarography.

Carbon Contents - Performed by combusting the sample in a furnace to convert all carbon materials to CO₂, which is determined by titration. An easy test, but does not identify individual carbonaceous species.

5. HYDROGEN

H₂ Assay - Determined by difference. Very accurate method because O₂, N₂, and other gases can be determined with great accuracy. Other impurities cannot be present because of the nature of the product and manufacturing process.

Gas Impurities - O₂, N₂, CH₄, CO₂, CO, He, and Ar are determined by gas chromatography, an accurate and easy method.

6. FLUORINE

F₂ Assay - This is obtained by difference. Impurities (HF, CF₄, N₂, O₂, CO₂ and He) can be determined very accurately, permitting the F₂ assay to be determined by difference.

Impurities - HF and CF₄ are determined by near-infrared and infrared spectroscopy, respectively. These methods are not ideal, but are the most practical for the present state-of-the-art from the

standpoint of accuracy, ease of operation, and economy. A gas chromatographic method would be more desirable; however, there are no chromatographic columns available yet which will provide adequate separation of all volatile impurities. Nitrogen, oxygen and helium are determined by gas chromatography after they have been removed from the fluorine. As part of this procedure a direct determination of F_2 is made to establish sample size so that the percent of N_2 , O_2 , HF, CF_4 , and He can be found.

7. CHLORINE PENTAFLUORIDE

The specification for ClF_5 is in preparation, and various analytical procedures are being evaluated. Gas chromatography appears to be the most suitable method for determining the impurities: HF, ClF_3 , ClO_2 , Cl_2 , and ClF_5 assay will then be obtained by difference (10).

8. AMMONIA

NH_3 Assay - Determined by difference. This is the most simple and accurate method, since the oil and H_2O determinations are known with considerable accuracy.

Oil Content - Determined by extraction from the residue of an evaporated sample and weighed directly. It is best to have the direct weight of the oil, since there may be several different oils present.

Water - Obtained by the Karl Fisher method. Titration with Fisher reagent is a direct and simple method of determining water content, since the Fisher reagent is specific for H_2O .

The purity of ammonia is consistently high, eliminating the need for other analyses.

SECTION VI

PROPELLANT PROCUREMENT

The Air Force has been designated by the Department of Defense as single manager for the rocket propellant commodity class, Federal Supply Classification No. 9135. This means that the Air Force procures all propellants that are covered by a limited coordinated or coordinated military specification for all of the Services and NASA. San Antonio Air Material Area (SAAMA), San Antonio, Texas is the procuring activity that accumulates the user quantity requirements and then contracts with industry for the manufacture and delivery of the required propellants. The Air Force Rocket Propulsion Laboratory (AFRPL), Edwards, California furnishes the engineering support to SAAMA by preparing the Air Force and fully coordinated specifications, and by resolving technical questions that arise. Annually, AFRPL authenticates an AFLC/AFSC Form 1-8 (Figure 2) which provides SAAMA with the necessary technical information for the propellant procurement. If a satisfactory specification exists, it is identified in Block IV of the form and Block III is annotated with an "A". Should there be deficiencies in the specification, or if no specification exists, this fact is noted in Block II; the appropriate letter is entered in Block III; and a Supplemental Data Sheet (SDS), AFLC/AFSC Form 5 (Figure 3) with the necessary information is attached. Supplemental Data Sheets are prepared to furnish the procurement activity with the latest available information quickly. Formal distribution for other purposes is not normally provided. The information contained in an SDS will eventually be incorporated into the applicable specification.

If a propellant is not covered by a military specification, SAAMA will procure the propellant only if requested to do so, and the requiring activity will then furnish the engineering support. A specification becomes "coordinated" when the three Services concur on the document,

and its use is then mandatory. A limited coordination specification is identified by the symbol designation of the preparing activity, and its use is mandatory for only that activity (optional for others).

The specification, or other technical information forwarded with the AFLC/AFSC Form 1-8, becomes the basis for the propellant procurement, and is incorporated into the invitation to bid and the resulting contract.

I. AFIC/AFSC ENGINEERING ACTIVITY RTD/AFRPL (RPPS)		ADVANCE PROCUREMENT DATA SUPPORT WORKSHEET		XIII. INITIATING ACTIVITY MAAHA (MAOR)		XIV. FY RGR FY 67	
II. REMARKS		XV. PROCUREMENT INFORMATION AFSC ☒ ANA/ATD ☐ MTP/Procurement Service/Theme ☐ CASL ☒ Item for use in FY 66 ☐ MAP ☐ Series ☐ XVI. FEDERAL STOCK NUMBER OR AERNO (if not assigned, give Council NO.) 9135-209-7943		XXII. DATES PC-TESTED C/N2000-9135-3		Date F/C 30 Apr 66	
Supplemental Data Sheet AFIC/AFSC Form 5 attached		DESCRIPTION: PROPELLANT, NITRIC ACID SPEC: MIL-P-7254 USE: Rocket Engine Oxidizer					
This form replaces or was replaced by AFIC Form 444 dated _____ (see item)							
Stock No. _____							
III. APPROVED OR DISAPPROVED A. Approved B. Approved. See Remarks. C. Approved provided recommended changes to above description are accomplished. D. Disapproved. Item series disapproved (See Remarks). E. Disapproved. See Remarks.							
IV. APPLICABLE PROCUREMENT DOCUMENT 1. Specification No. MIL-P-7254 2. Drawing No. _____ 3. See Remarks.							
V. SPECIFICATION OR DRAWING OPTIONS 1. See specification or drawing for options to specify on ID's or RFP's. 2. See Remarks.							
VI. QUANTITY OF PREPRODUCTION TEST SAMPLES (FIRST ARTICLE) FOR TEST 1. Quantity (See Notes 1, 11, 13, & 14 in Block XVII) 2. See Remarks.							
VII. ESTIMATED PREPRODUCTION TEST SAMPLE (FIRST ARTICLE) TESTING TIME 1. Days _____ 2. See Remarks.							
VIII. PREPRODUCTION TEST SAMPLE (FIRST ARTICLE) INSPECTION OPTION 1. At plant or approved testing laboratory sponsored by Government and AFIC/AFSC. 2. At plant or approved testing laboratory sponsored by Government and AFIC/AFSC. 3. At plant or approved testing laboratory sponsored by Government and AFIC/AFSC. 4. At AFIC/AFSC. See Remarks.							
IX. QUALIFICATION REQUIREMENT 1. Specification of drawing does not require qualification testing. 2. See Remarks.							
X. GOVERNMENT LOANED EQUIPMENT 1. GLE required as listed in spec, obtain from AFIC/AFSC Engineering Activity. 2. GLE required as listed in spec, obtain from AFIC/AFSC Engineering Activity. 3. GLE required as listed in spec, obtain from AFIC/AFSC Engineering Activity. 4. See Remarks.							
XI. GOVERNMENT FURNISHED PROPERTY 1. See Remarks.							
XII. COPIES OF CONTRACTS AND AMENDMENTS THEREOF RESULTING FROM THIS CLEARANCE 1. One copy of technical specification and amendments thereto will be forwarded by the contracting officer to AFIC/AFSC Engineering Activity. 2. See Remarks.							
XIII. AUTHENTICATION (Name, organizational code and telephone number) 1. AFIC/AFSC AUTHENTICATION (Name, organizational code and telephone number) 2. AFIC/AFSC AUTHENTICATION (Name, organizational code and telephone number)							
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XXX. AUTHENTICATION (Name, organizational code and telephone number) 1. AFIC/AFSC AUTHENTICATION (Name, organizational code and telephone number) 2. AFIC/AFSC AUTHENTICATION (Name, organizational code and telephone number)							

Figure 2. Advance Procurement Data Support Worksheet.

ADVANCE PROCUREMENT SUPPLEMENTAL DATA SHEET			FY67
ORGNL CODE	FEDERAL STOCK NO. OR SERNO	SOS DATE	
RTD/AFRPL (RPPS)	9135-209-7943	15 June 1965	
	CLASS CODE	SERIAL NUMBER	SUPERSEDES SOS DATED
	9135	N/A	15 July 1964
NOMENCLATURE AND TYPE NUMBER			
Propellant, Nitric Acid			
APPLICABLE SPECIFICATION AND/OR DRAWING			
MIL-P-7254, Revision E, Amendment 1			
ADDITIONAL SUPPLEMENTARY DATA			
Basic specification, page 2, paragraph 3.5: Revise to read "3.5 Qualitative. The propellant shall be a single phase liquid when examined visually by transmitted light." Page 9, add 6.3 Definitions. and 6.3.1 Single phase liquid. A single phase liquid is devoid of any visible foreign liquid but may contain solid material as permitted within this specification.			

AFLC/AFSC FORM 5
JUL 62

REPLACES AFSC FORM 44B, JUL 61, WHICH IS OBSOLETE.

AF-WP-Q-APR 64 7650

Figure 3. Advance Procurement Supplemental Data Sheet.

SECTION VII

THE SPECIFICATION IN ACTION

The government quality-control inspectors use the specification to assure compliance with the contract, usually at the point of manufacture. Samples are taken as prescribed and analyzed in government laboratories. Frequently, the government inspectors will accept the vendor's analyses when the vendor has demonstrated satisfactory quality-control procedures. In these instances, the vendors provide a certificate of analysis and permit the government inspector to spot check their procedures. Should the vendor not elect to use the analytical methods detailed in the specification, a waiver is requested subject to approval of the cognizant engineering activity.

The acquisition of samples and their shipment to a distant laboratory is not without problems. For the propellants that are relatively nonreactive and are liquid under normal temperatures, common sample bottles may be used. Cryogenic propellants such as liquid hydrogen require special samplers. Samples of cryogenic propellants are obtained in the liquid state. The special cryogenic sampler (11) allows the operator to vaporize the total sample without loss, thus maintaining the original composition and purity of the sample, and yet providing safe conditions for carrying or shipping the sampler to a quality-control laboratory.

The specification is a procurement document, and should not be used for field quality control or surveillance (Figure 4). After the propellant is accepted at the vendor's plant, it can be exposed to many sources of contamination beyond the control of the vendor (12). Some propellants exhibit slow chemical changes (e. g. , fuming nitric acid). Cryogenic propellants are subject to boil-off, thus concentrating the impurities. The specification requirements are selected to allow for

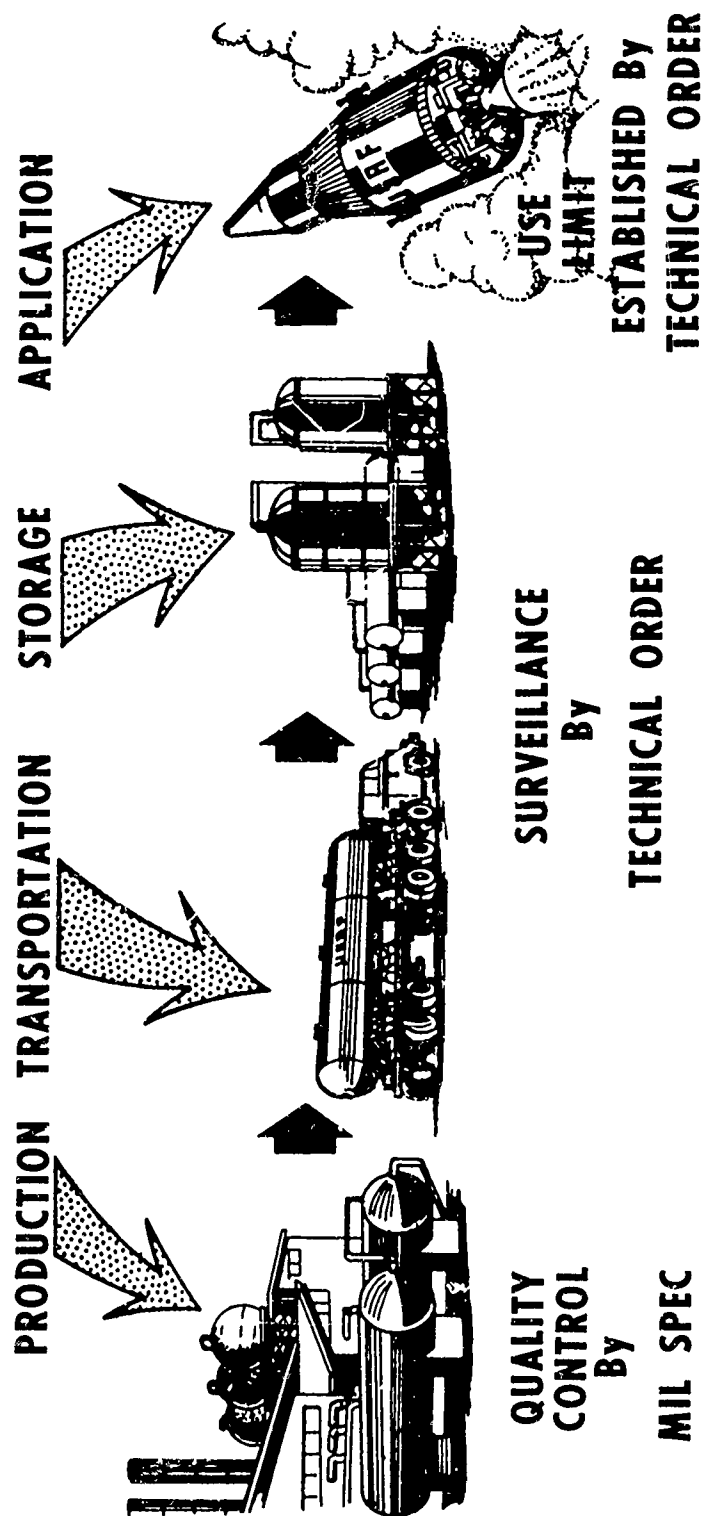


Figure 4. Propellant Logistics.

some propellant change, thus assuring that the delivered propellant is usable. Otherwise, the propellant that just meets the procurement requirements at acceptance may not do so at the point of delivery.

The quality of the propellant required at the destination will depend on the application. Each user should specify a "use limit" that is meaningful for his specific engine, weapon system, etc. This "use limit" should represent the minimum quality that the propellant must have and still perform satisfactorily in the specific system. For missiles that are stored in a ready condition, a "loading limit" may be established that identifies the propellant quality that must be met at the time the missile is serviced. The values of the "loading limit" are a function of the storage period, and are selected so that the "use limit" will not be reached by the expected degradation during this period. Thus, a propellant may be permitted to degrade from the military specification quality to the "loading limit", with further degradation to the "use limit". The "loading" and "use limits" germane to a given system or application are included in the applicable operating manuals or technical orders.

Although the procurement specification requirements cannot properly be used for field use, the quality assurance provisions can. The included analytical methods and sampling procedures are very valuable for all surveillance activities, since these techniques are usually selected for their simplicity and readily available equipment.

Engine model specifications should not always stipulate that the rocket engine use propellants conforming to the requirements of the procurement specification. If the propellant is subject to degradation during shipping and storage, "use" or "loading limits" should be established during the engine development program. This will preclude the problem of trying to supply specification-grade propellants in the field, when the propellant will begin to deviate from the specification as soon as it leaves the manufacturing plant.

SECTION VIII

SUMMARY

The military specification is an important document that defines the required quality of rocket propellants procured by the government. It is used by quality control inspectors as the criteria for the acceptance of propellants from suppliers.

The two most important parts of a specification are the Requirements and Quality Assurance Provisions sections. The Requirements section contains the specific properties, composition and impurity limits that the propellant must meet. The requirements are established to assure that the propellant will accomplish the purposes intended. Data from R&D programs, field use, and engine manufacturers are used in the preparation and revision of specifications. The application requirements must be balanced by the ability of the chemical industry to produce large quantities of the propellant at an acceptable price. The factors of safety, storability and analytical difficulty are as important as performance in setting specification values.

The Quality Assurance Provisions section consists of the test methods or analytical procedures used to determine conformity of the propellant with the specified requirements. The methods selected must provide the necessary accuracy, and should be simple and easy to perform. The use of specialized and expensive instruments is to be avoided when possible. Most analytical methods are currently based on standard titration techniques, though the use of gas chromatographic procedures is increasing (13). The most difficult and time-consuming function of specification preparation is the development of suitable analytical methods.

Military specifications, being procurement documents, are not adequate for field quality control. Most propellants are subject to decomposition or contamination, causing deviation from the specification requirements, during shipping or storage. "Loading limits" and "use limits" should be established by the using organization to assure control of propellant quality between procurement and ultimate consumption. The specification analytical procedures are satisfactory for field use and are widely used.

The specification is a dynamic instrument, the product of extensive cooperation and coordination among the rocket and chemical industries, and government agencies. Specifications are continually reviewed, and are revised frequently as propellant technology advances.

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DOCUMENT CONTROL DATA - R&D		
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1. ORIGINATING ACTIVITY (Corporate author) Air Force Rocket Propulsion Laboratory Edwards, California 93523		2a. REPORT SECURITY CLASSIFICATION Unclassified
		2b. GROUP
3. REPORT TITLE Propellant Specifications - Preparation and Use		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Special Report		
5. AUTHOR(S) (Last name, first name, initial) Forbes, Forrest S.		
6. REPORT DATE October 1967	7a. TOTAL NO. OF PAGES 53	7b. NO. OF REFS 13
8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S) AFRPL-TR-67-256	
b. PROJECT NO. 3148		
c.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
d.		
10. AVAILABILITY/LIMITATION NOTICES Distribution of the document is unlimited.		
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Air Force Rocket Propulsion Laboratory
13. ABSTRACT The specification is the key document that establishes the technical requirements for propellant procurements. These requirements are dictated by the needs of the various rocket applications, with due consideration given to the manufacturing ability of the suppliers. The specification also contains quality assurance provisions that detail the sampling techniques and analytical procedures to be used to insure that the propellant conforms to the specified requirements. Propellant specifications are prepared by the cognizant technical laboratory, and authenticated annually for the procuring activity. Specifications are continually revised and updated as required by the rocket community. Examples are presented showing the rationale for the establishment of specific requirements and analytical methods for several current propellants.		

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