

AD-A219 314

DTIC FILE COPY AD

AD-E401 993

Contractor Report ARAED-CR-88017

TEST PLAN DEVELOPMENT FOR PLASTIC AMMUNITION CONTAINERS
VOLUME II - TEST PLAN

J.E. Brzuskiwicz
DSET Laboratories, Inc.
Box 1850 Black Canyon Stage I
Phoenix, AZ 85027

Carlton Morrison
ARDEC
Project Engineer

January 1990

DTIC
ELECTE
FEB 26 1990
S B D



US ARMY
ARMAMENT RESEARCH,
DEVELOPMENT AND
ENGINEERING CENTER

U.S. ARMY ARMAMENT RESEARCH, DEVELOPMENT AND
ENGINEERING CENTER

Armament Engineering Directorate

Picatinny Arsenal, New Jersey

Approved for public release; distribution unlimited.

90 02 23 123

The views, opinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

Destroy this report when no longer needed by any method that will prevent disclosure of contents or reconstruction of the document. Do not return to the originator.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE				
4. PERFORMING ORGANIZATION REPORT NUMBER(S) DSET Report No. R2970-23 (Final Report)			5. MONITORING ORGANIZATION REPORT NUMBER(S) Contractor Report ARAED - CR-88017	
6a. NAME OF PERFORMING ORGANIZATION DSET LABORATORIES, INC.	6b. OFFICE SYMBOL (If applicable)		7a. NAME OF MONITORING ORGANIZATION ARDEC, AED	
6c. ADDRESS (City, State, and ZIP Code) Box 1850 Black Canyon Stage I Phoenix, AZ 85027			7b. ADDRESS (City, State, and ZIP Code) Packaging Division (SMCAR-AEP) Picatinny Arsenal, NJ 07806-5000	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION ARDEC, IMD STINFO BR	8b. OFFICE SYMBOL (If applicable) SMCAR-IMI-I		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAA-21-86-C-0275	
8c. ADDRESS (City, State, and ZIP Code) Picatinny Arsenal, New Jersey 07806-5000			10. SOURCE OF FUNDING NUMBERS	
			PROGRAM ELEMENT NO.	TASK NO.
			PROJECT NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) TEST PLAN DEVELOPMENT FOR PLASTIC AMMUNITION CONTAINERS, VOLUME II - TEST PLAN				
12. PERSONAL AUTHOR(S) J.E. Brzuskiwicz, DSET LABORATORIES, INC. and Carlton Morrison, ARDEC Project Engineer				
13a. TYPE OF REPORT Final	13b. TIME COVERED FROM 08-19-86 to 11-30-88	14. DATE OF REPORT (Year, Month, Day) 90-01-23	15. PAGE COUNT 78	
16. SUPPLEMENTARY NOTATION				
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	Ammunition containers, packaging, plastics, logistics, lifetime prediction models, accelerated testing, Arrhenius relationships test development	
19. ABSTRACT (Continue on reverse if necessary and identify by block number)				
The replacement of wood and metal by plastic materials in ammunition packaging container applications offers the potential for substantial weight and cost reduction. Although plastic packaging has been common in the commercial sector for a considerable number of years, no long-term performance data are available. This is perhaps due to the short design life of commercial packaging. The use of plastic packaging, and polymeric materials in general, for military purposes requires consideration of environmental factors that are not encountered by commercial packaging. Military requirements dictate that ammunition items be offered protection for up to thirty years in a variety of transit and storage conditions. The evaluation and ultimately the qualification of plastic packaging materials and plastic ammunition container designs must be based on test procedures that will allow the accurate prediction of performance. (continued)				
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT ☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL I. Hazendari			22b. TELEPHONE (Include Area Code) (201) 724-3316	22c. OFFICE SYMBOL SMCAR-IMI-I

Paragraph 19. ABSTRACT

Two plastic ammunition containers and their materials were tested using a combination of environmental exposure tests in order to provide the basis for a viable test plan which can be used for a variety of container items, materials, and designs. The test plan developed reflects the testing philosophy required to determine the long-term acceptability of plastic materials and plastic container design. The test data obtained also provides a basis for a continued test effort required to produce accurate lifetime prediction and moisture vapor transmission rate models for the two container items tested.

It was demonstrated that the typical accelerated environmental test procedures, generally followed to evaluate the materials used in military items and to determine the acceptability of items, in fact accelerate effects only to the extent that material degradation and time dependent material's properties allow. The material and full-scale item test data used to prepare the lifetime prediction models developed over the nine month period in which the tests were conducted therefore, do not provide the reliability required to make judgements concerning the acceptability of the two container items tested or for the use of plastic materials in general.

FOREWARD

This is Report No. R2970-23 (Final Report) of DSET Project No. R2970 entitled, "Test Plan for Plastic Containers." Report No. R2970-23 is entitled, "Test Plan Development for Plastic Ammunition Containers" and covers the work conducted to develop accelerated test procedures for predicting the effective lifetime of plastic ammunition packaging containers. The report is presented in two separate volumes. Volume I contains the Final Technical Report and includes the analysis of environmental test data, the characterization of ammunition container logistic chains and container item lifetime prediction analyses. Volume II contains the Final Test Plan prepared on the basis of actual tests conducted on the 155mm Propelling Charge Container and the M2A1 Small Ammo Container.

The project work effort was conducted from August 1986 through September 1988 at DSET Laboratories, Inc. under the technical direction of the Packaging Division of the U.S. Army Armament Research, Development and Engineering Center. Mr. Jasper C. Griggs and Mr. D.E. Jones served as technical consultants to the project during the Phase I effort and assisted DSET in the logistics and literature studies, respectively. The Texas Research Institute, Inc. conducted thermal analysis measurements on container materials during the Phase II effort.

The project was funded under Contract No. DAAA-21-86-C-0275. Volume II of this report fulfills CDRL Item No. A009. The Volume I Final Technical Report fulfills CDRL Item No. A008.

Data are recorded in DSET Logbook No.'s 171, 174 and 200.

Accession For	
NTIS GRA&I	☒
DTIC TAB	☐
Unannounced	☐
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

Copy
Inspected

(THIS PAGE IS LEFT BLANK)

CONTENTS

1.0	<u>INTRODUCTION AND SCOPE.</u>	1
2.0	<u>RELATED DOCUMENTS AND TEST STANDARDS.</u>	2
2.1	ASTM Standards	2
2.2	Industry	2
2.3	Government Standards	3
2.4	Order of Precedence.	3
3.0	<u>PLASTIC AMMUNITION CONTAINER TESTING CONCEPTS</u>	3
3.1	Testing Philosophies	3
3.2	Test Plan Organization	5
4.0	<u>GENERAL TEST PLAN</u>	13
4.1	Definition of Logistics Chain Environment and Container Acceptability Limits	13
4.1.1	Definition of Environmental Parameters.	13
4.1.2	Definition of Container Acceptability Limits.	13
4.1.2.1	Container Materials.	13
4.1.2.2	Container Functional Property Limits	14
4.1.3	Documentation	14
4.2	Container Material Property Tests.	17
4.2.1	General	17
4.2.2	Sample Material Tests and Evaluations	17
4.2.2.1	Optical Properties	17
4.2.2.2	Physical Properties.	19
4.2.2.3	Thermal Properties	19
4.3	Container Material Screening Tests	21
4.3.1	Outdoor Environmental Tests	21
4.3.2	Accelerated Environmental Tests	22
4.3.2.1	Accelerated Natural Outdoor Exposure	22
4.3.2.2	Mil-Std-810D Tailored Simulated Solar Radiation Tests.	22
4.3.2.3	Xenon Arc Lamp Environmental Cabinet Tests	23
4.3.3	Container Material Compatibility Testing.	24
4.3.4	Preliminary Performance Prediction Models	24

CONTENTS (cont)

	<u>Page</u>
4.4 Container Material Qualification Testing	25
4.4.1 General	25
4.4.2 Materials Qualification Test Program Definition . . .	26
4.4.3 Activation Energy Determination Equipment Requirements	28
4.4.4 Activation Energy Determinations for Thermal Degradation of Materials Properties	31
4.4.5 Activation Energy Determinations for Ultraviolet Degradation of Materials Properties	33
4.4.6 Elevated Temperature and Humidity Testing	35
4.4.7 Accelerated Ultraviolet Exposure Testing.	36
4.4.8 Real-Time Outdoor and Indoor Storage Testing.	37
4.4.8.1 Outdoor Real-Time Testing.	37
4.4.8.2 Real-Time Indoor Exposure Testing.	37
4.4.9 Cyclic Environment Testing.	37
4.4.10 Material Data Analysis and Modelling.	38
4.4.10.1 Kinetic Expressions of Ageing Processes . .	40
4.5 Material Production Lot Testing.	41
4.6 Full-Scale Container Item Testing.	42
4.6.1 General	42
4.6.2 Test Equipment.	43
4.6.2.1 Environmental Chamber/Solar Simulator. . . .	43
4.6.2.2 Solar Radiation Instrumentation.	43
4.6.2.3 Temperature/Humidity Measurement Instrumentation.	43
4.6.2.4 Temperature Measurement and Control.	44
4.6.2.5 Pressure Measurement Instrumentation	46
4.6.2.6 Data Recording and Handling Equipment. . . .	46
4.6.3 Full-Scale Item Test Set-Up	46
4.6.4 Data Requirements and Analysis.	48
4.6.5 Design/Development Tests.	58
4.6.6 Production-Lot Tests.	60
5.0 <u>TEST PLAN STATUS AND BACKGROUND</u>	60
5.1 Background	60
5.2 Materials Tests.	61
5.2.1 Materials Acceptability Limits	61
5.2.1.1 Activation Energies	62
DISTRIBUTION LIST.	65

TABLES

	<u>Page</u>
1. Example logistics chain information	14
2. Example checklist describing plastic ammunition container material property acceptability limits.	15
3. Example checklist describing plastic ammunition container functional property acceptability limits.	16
4. Summary of typical material tests	18
5. Temperature and vapor pressure environment data	29
6. General specifications for chilled mirror hygrometers	44
7. Example of container test item characteristics and material data needed for full-scale item testing.	47
8. Example of container full-scale item test data.	50
9. Example of container moisture gain data	51
10. Example of container graph regression information	52
11. Environmental chamber intercalibration data	54

(THIS PAGE IS LEFT BLANK)

FIGURES

	<u>Page</u>
1. Test plan concept summary	6
2. Material selection.	7
3. Material qualification testing.	8
4. Material production-lot testing	9
5. Container development testing	10
6. Container qualification testing	11
7. Container production-lot testing.	12
8. Activation energy determination test equipment concept.	30
9. Materials property degradation curves	33
10. Example of container instrumentation configuration.	45
11. Example of test article set-up and solar simulator target area	49
12. Regression data	57
13. Test plan for 155mm propelling charge container	63

(THIS PAGE IS LEFT BLANK)

TEST PLAN DEVELOPMENT FOR PLASTIC AMMUNITION CONTAINERS - VOLUME II

1.0 INTRODUCTION AND SCOPE

Polymeric materials are seeing increased use in packaging applications for ammunition. The materials and containers used must offer the ammunition items protection for up to 30 years in a variety of storage environments. The environments that must be considered by the packaging design in regard to the ammunition life cycle must include indoor, outdoor, transportation, and maintenance. The purpose of this test plan is to outline test procedures with the objective of predicting the longterm performance of plastic ammunition packaging containers after exposure to storage environments. The test plan is organized into several levels, each of which attempt to successively screen unacceptable materials and designs. The approach to meeting the objective first addresses materials testing for purposes of identifying suitable materials for the application. Second, qualification and pre-production testing of materials and container items to show compatibility of materials and functionality of the container design, and finally the testing required during the production life of the container item are also included.

The application of the test plan is limited to the climatological environment of the container life cycle and does not address the physical environment. However, the testing philosophy and procedures described could be used to prepare test articles to study the effects of the physical environment on features of the container related to handling, transportation, and maintenance. The test plan also does not set limits for acceptability of container materials or design parameters. However, the testing described could be used to establish these limits for plastic packaging if the limits are known or can be determined for packaging designed with other materials. Likewise the test plan does not offer guidance on the selection, formulation, compounding or processing of specific plastic, elastomers, adhesives, or sealants. These parameters however, are critical to the performance of the container and should be included as variables in the test plan when it is implemented. Lastly, the test plan does not address the statistics or sampling of ammunition packaging for the testing required to provide a given degree of confidence or to predict container or ammunition failure rates. The test procedures in the plan could be used to obtain the data needed for this information.

The test plan is based on a preliminary study of the logistics and the testing of the plastic containers reported in Volume I of this report. The actual tests conducted were based on the premise that the ammunition containers were to have a 30-year lifetime in any part of the world with a two-year outdoor, uncontrolled storage. The logistics of the items studied suggested that at the present time less than a 20-year lifetime may be typical. Production rate, war reserve rotation, testing, training, and war are parameters that could change the lifetime requirement for packaging containers. These features of the logistics chain must be considered during the testing of plastic containers since they will have a direct influence on the requirements for the accuracy of the performance prediction models that

will be developed from the test data and can affect the acceptable limits of performance with respect to time.

Packaging items composed of polymeric materials that have a 30-year lifetime requirement necessitate the use of accelerated test procedures to provide engineering data for design purposes and to form the basis for cost and risk analysis. Accelerated testing also has the objective of providing information on the longterm performance of materials and items.

2.0 RELATED DOCUMENTS AND TEST STANDARDS

2.1 ASTM Standards

- D256 Izod Impact Test for Electrical Insulating Materials and Plastics
- D638 Tensile Properties of Plastics
- D790 Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
- D1435 Outdoor Weathering of Plastics
- D1898 Sampling of Plastics
- D2244 Method for Calculation of Color Differences From Instrumentally Measured Color Coordinates
- D3045 Heat Ageing of Plastics Without Load
- D3418 Transition Temperatures of Polymers by Thermal Analysis
- D4065 Determining and Reporting Dynamic Mechanical Properties of Plastics
- D4092 Dynamic Mechanical Measurements on Plastics
- D4230 Measuring Humidity with Cooled-Surface Condensation (Dew Point) Hygrometer
- E793 Heats of Fusion and Crystallization by Differential Scanning Calorimetry
- E903 Solar Absorptance, Reflectance, and Transmittance of Materials Using Integrating Spheres
- G26 Operating Light and Water Exposure Apparatus (Xenon-Arc Type)
- G90 Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight

2.2 Industry

ASHRAE 1985 Fundamentals. American Society of Heating, Refrigerating, and Air-Conditioning Engineers, Inc. Chapter 6. 1985.

2.3 Government Standards

AR-70-38 Research, Development, Test, and Evaluation of Material for Extreme Climatic Conditions.

MIL-STD-210B Climatic Extremes for Military Equipment

MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

MIL-STD-810D Environmental Test Methods and Engineering Guidelines

2.4 Order of Precedence

In the event of a conflict between the text of this test plan and the referenced standards, this document shall take precedence.

3.0 PLASTIC AMMUNITION CONTAINER TESTING CONCEPTS

3.1 Testing Philosophies

The use of polymeric materials for ammunition packaging necessitates philosophical discussion for testing approaches, techniques and data analysis. This discussion relates to the general inherent properties of polymers in regards to processing, environmental degradation, and especially accelerated environmental tests. Environmental effects on materials in this sense was used to form the basis of the testing approach described in this test plan. Polymeric materials as compared to other engineering materials are not isomorphic. Thus, considerable testing tailored for the characteristics of each specific material is required to determine the distribution of performance with respect to the environment occurring over a thirty-year use period as well as the distribution to be expected over the lifetime of the production program.

The performance properties of polymeric materials are temperature dependent. This general feature requires that testing be conducted over a range of temperatures in order to predict the performance of container materials and suggests that the container material performance can be described by an Arrhenius relationship. As noted in Volume I, lifetime prediction errors increase with test temperatures above actual use temperatures. Thus, in order to develop prediction models with a high degree of accuracy it will be necessary to conduct tests using temperatures close to actual use temperatures. The choice of appropriate test temperatures however, will also depend on the thermal stability of the material. The approach to developing performance prediction models for polymeric materials will require the determination of activation energies to describe the temperature dependency of the material's properties over time.

The outdoor environment is more aggressive than indoor storage environments. The two-year outdoor exposure period requirement for plastic ammunition containers could be used for materials screening test purposes since this period is short as compared to the expected life cycle. Unacceptable performance after a two-year real-time or two-year equivalent accelerated outdoor test would indicate that the material or container item is not suitable for use over a 30-year life cycle where an outdoor exposure would more likely occur late in the container life cycle. Those materials and container items which exhibit acceptable performance after a two-year outdoor or a two-year equivalent outdoor exposure early in the life cycle would be subjected to further testing. This subsequent testing would have the objective of determining the effects of longterm indoor storage on the ability of the container material to provide adequate performance during a two-year outdoor exposure late in the life cycle.

Accelerated testing requires consideration of the characteristics of the materials and container items in order to establish conditions that will accelerate materials degradation and degradation effects in a manner than can be modelled to a real-time basis. Materials degradation and functional container properties such as moisture vapor transmission, are related to diffusion processes. Materials in thick cross-section or containers with large wall thickness would be expected to degrade at a different rate than if the same material was used in a thin cross-section. Surface degradation can also affect the rate of bulk degradation and accelerated test results in this case would be misleading. Accelerated test methods must therefore be designed to evaluate surface and bulk effects as they relate to realtime performance.

Sample configuration, the measurement techniques used to evaluate performance, the rate of acceleration and the severity of test conditions must be taken into account in order to select appropriate test conditions. The thermal history of container materials is important to field performance and to the interpretation of accelerated test results. Samples or container items with different thermal histories could react to the solar environment at significantly different rates and by entirely different mechanisms.

The degradation of materials due to outdoor exposure and simulated solar radiation must be evaluated in the context of the true solar spectrum of solar ultraviolet radiation. It is obvious that ultraviolet irradiation with energy at wavelengths significantly below that of the sun's terrestrial cut-off produces effects in some materials that are different from those occurring during natural exposure to sunlight. Most materials have strong absorption coefficients for ultraviolet in the wavelength region below 300nm. In fact a number of the more important polymeric materials transmit ultraviolet from natural sunlight, but begin absorbing only below 298-305nm. The irradiation of such materials with light from artificial sources that typically have intensities at 290nm from one million to ten million times greater than they would receive in natural sunlight may overwhelm the photochemistry of the system and produce unrealistic results.

Moisture gain by the container over long periods of time must also be tested for. The mechanism of moisture gain, also a diffusion-controlled process, is material specific and temperature dependent. The absorption and desorption of moisture by the container do not necessarily occur at the same rate. Thus, the accelerated test conditions used to evaluate different container items may require different test cycles in order to accelerate effects. A container that has a low moisture vapor transmission rate would typically be tested using constant, high vapor pressure conditions in order to cause the container to gain moisture as rapidly as possible. Daily temperature and water vapor pressure swings would tend to have little effect on moisture content even over long periods of time. On the other hand, a cyclic test would be used for containers with relatively high moisture vapor transmission rates since daily temperature and moisture vapor pressure changes could cause changes in the moisture content of the container and thus subject its contents to the deleterious effects of moisture absorption and desorption.

3.2 Test Plan Organization

The features of the test plan for plastic ammunition containers are summarized in Figures 1 through 7. Figure 1 exhibits the overall test plan concept. Figures 2 through 7 summarize the details of the plan. The test plan is divided into materials, component and full-scale item testing. Each level of testing includes accelerated and real-time test procedures. The accelerated testing described in the following paragraphs is used to provide screening information and to make preliminary performance predictions during the early phases of a container program. These data, combined with real-time test data in later stages of the program, will be used to refine the performance prediction models developed for the item during the program and build confidence in them.

The test plan concept in Figure 1 requires definition of the logistics chain environment, container functional requirements, and the desired life cycle in order to determine the container material and container design requirements. The combination of these requirements and definitions set the container test parameters by imposing limits on test environments, appropriate property measurements and test procedures. These requirements also set requirements for container materials and container design features.

Materials tests, as outlined in Figure 2, include screening, characterization and compatibility tests. These tests are to be conducted to show compliance with requirements and to provide data for performance prediction. Appropriate property measurements are to be conducted during accelerated and real-time environmental tests. As preliminary tests, the data obtained will be used to form the basis for qualifying materials for use in pre-production and production items. The qualification test process outlined in Figure 3, requires that an appropriate sampling plan be developed to show that the materials to be used for container items will statistically perform as required. The qualification tests to be conducted will also include an in-depth study of processing parameters which could affect long-term performance of the material. The resulting test data will be used to

PLASTIC AMMUNITION CONTAINER

TEST PLAN CONCEPT

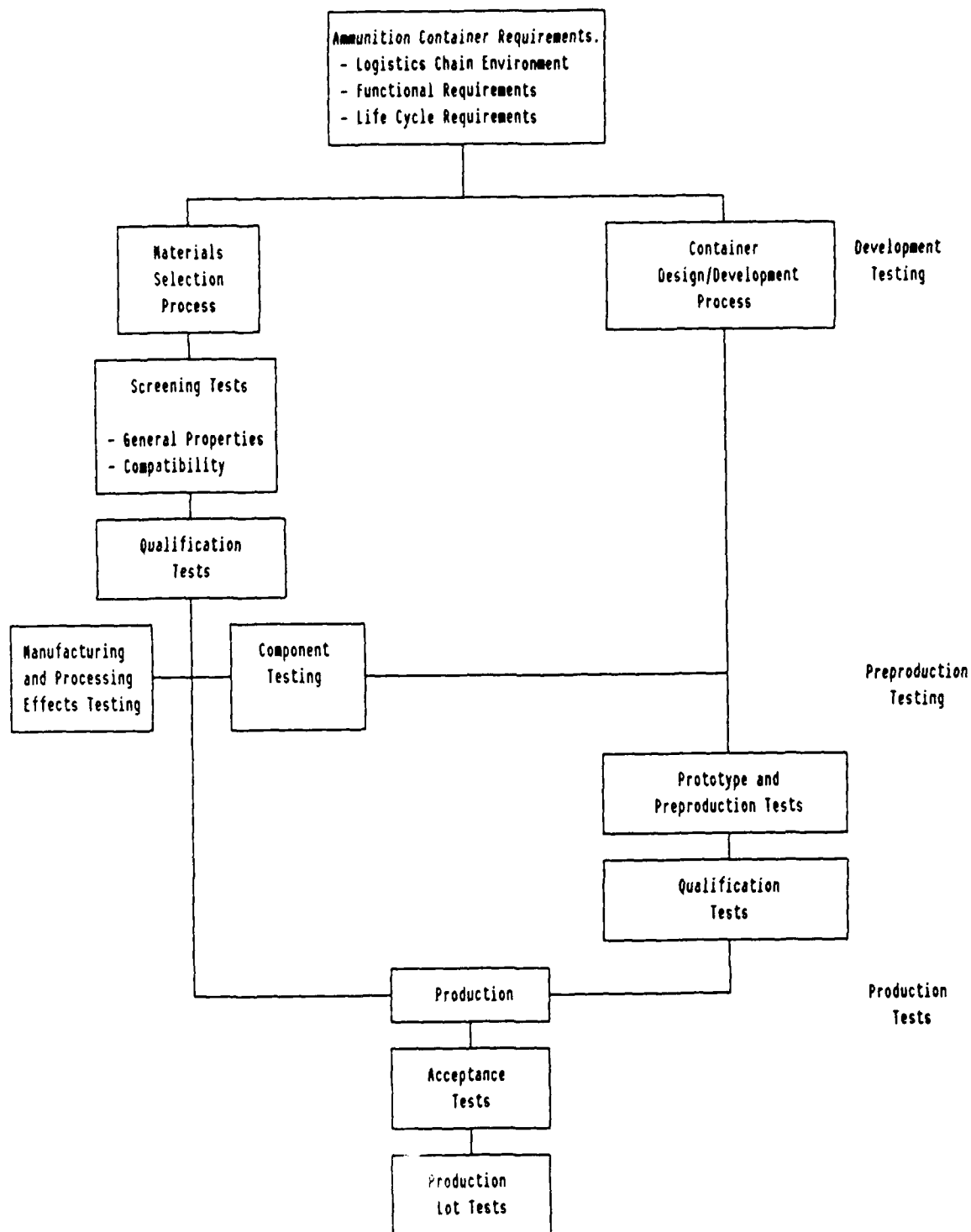


Figure 1. Test plan concept summary

PLASTIC AMMUNITION CONTAINER MATERIAL SELECTION PLAN

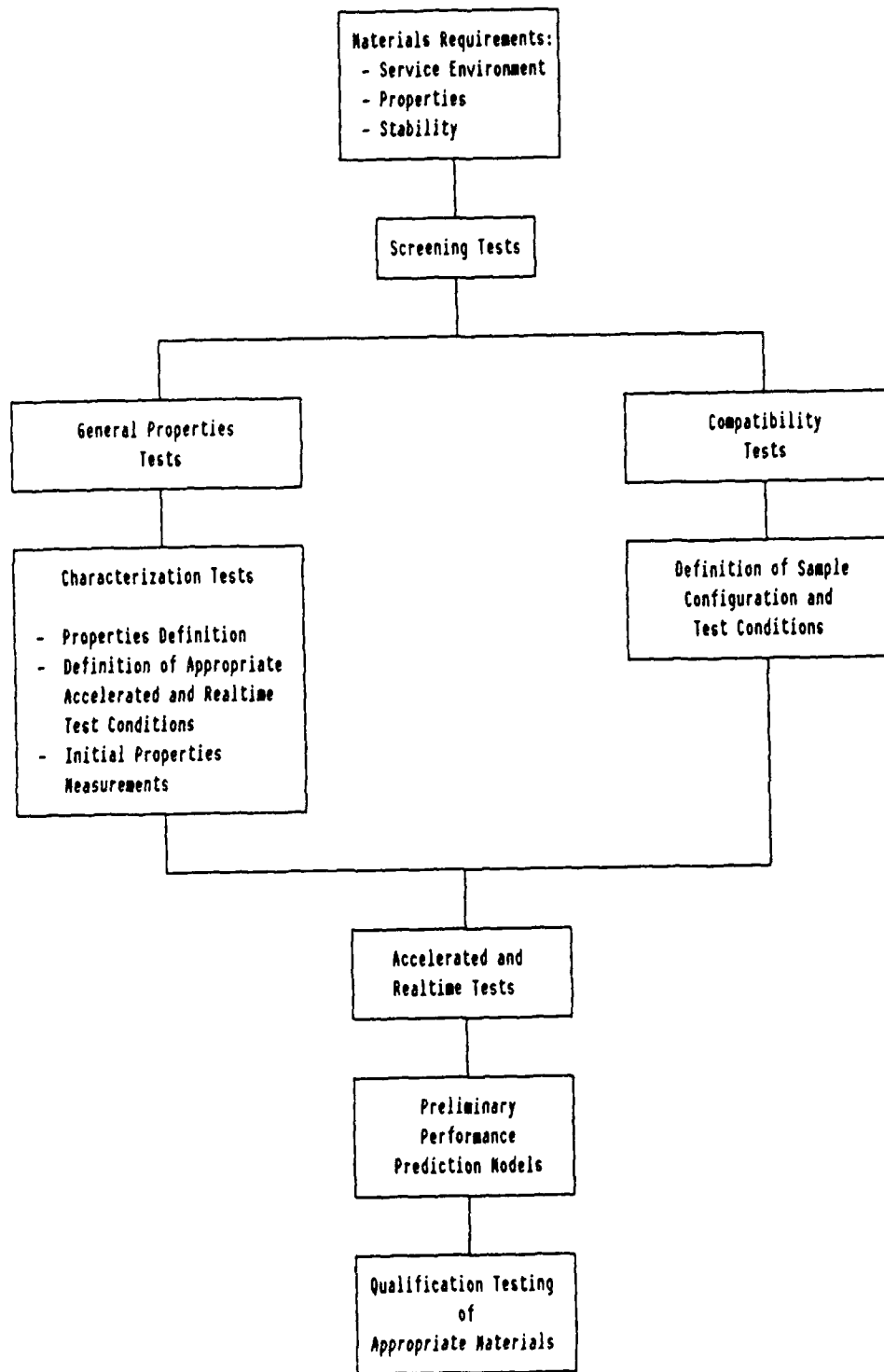


Figure 2. Material selection

PLASTIC AMMUNITION CONTAINER MATERIAL QUALIFICATION TESTING

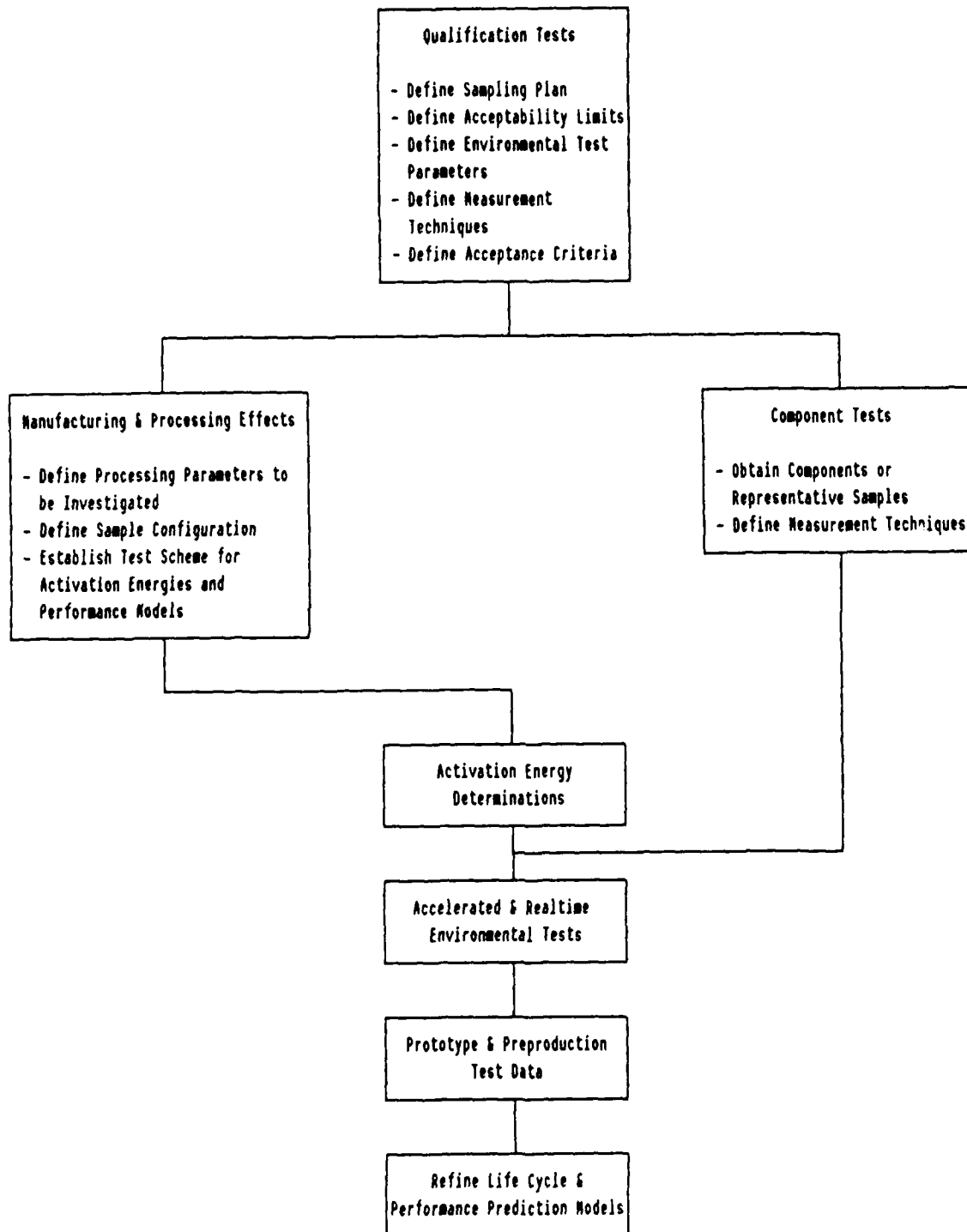


Figure 3. Material qualification testing

PLASTIC AMMUNITION CONTAINER MATERIAL PRODUCTION-LOT TESTING

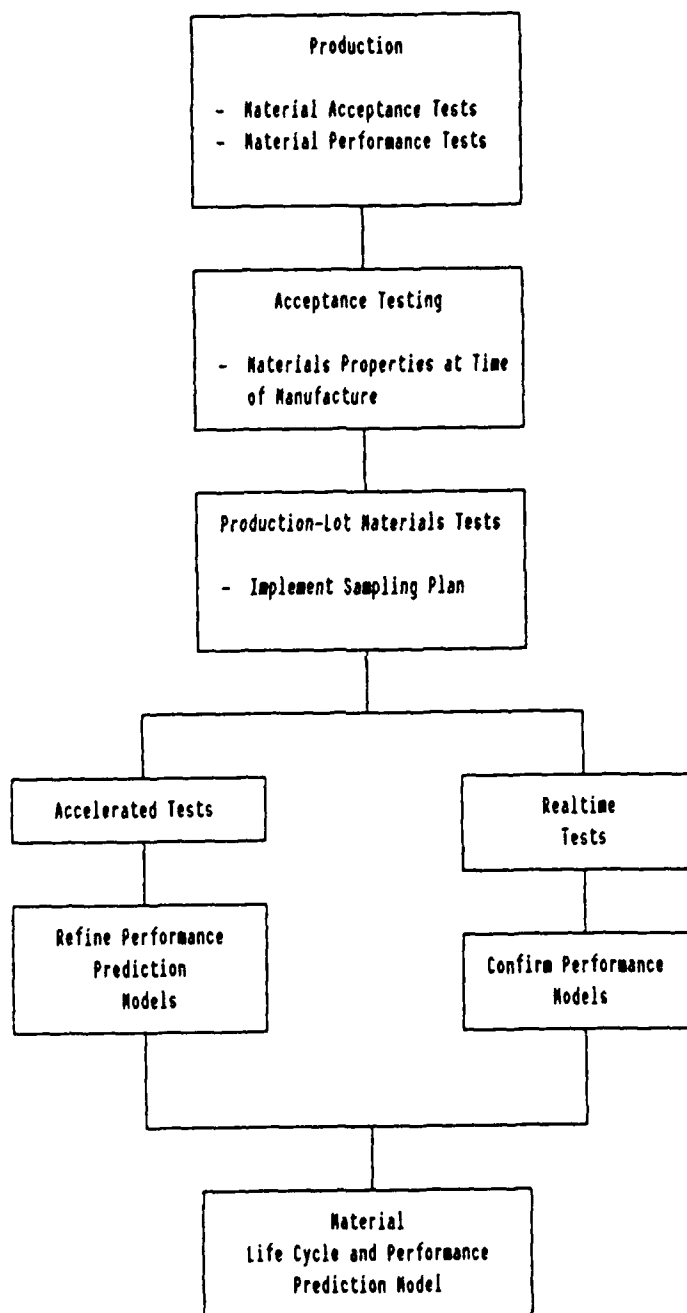


Figure 4. Material production-lot testing

PLASTIC AMMUNITION CONTAINER DESIGN/DEVELOPMENT TEST PLAN

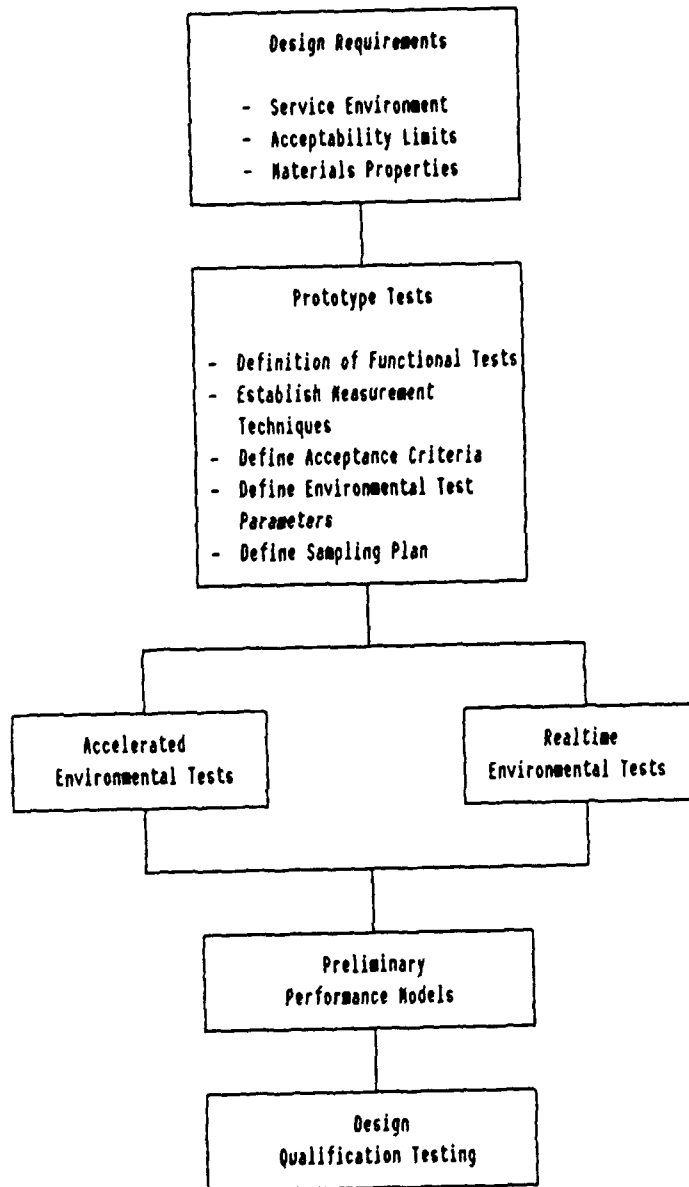


Figure 5. Container development testing

PLASTIC AMMUNITION

CONTAINER QUALIFICATION TESTING

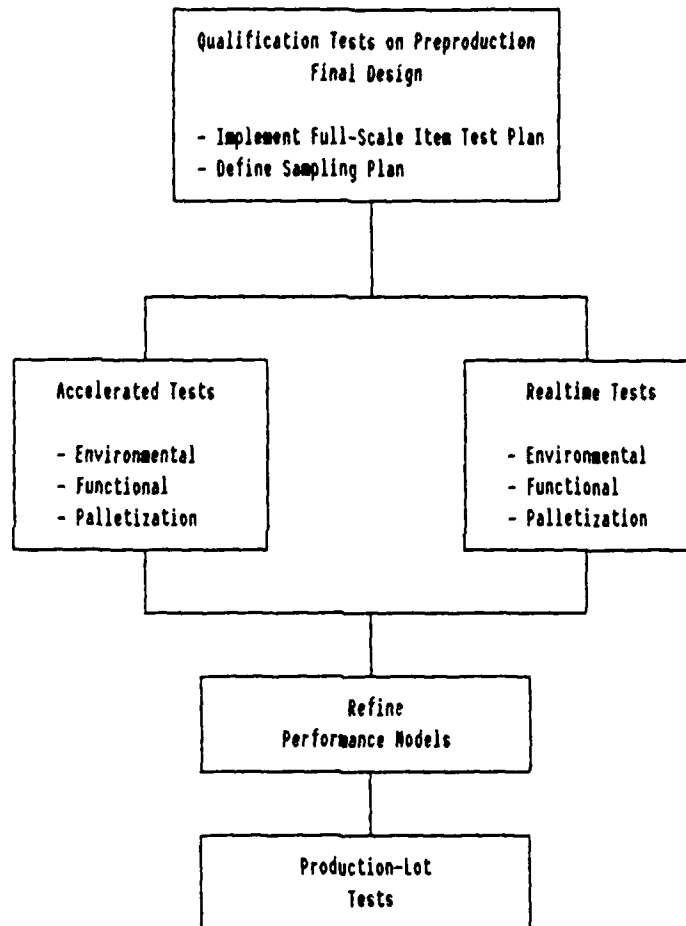


Figure 6. Container qualification testing

PLASTIC AMMUNITION CONTAINER

PRODUCTION-LOT TESTING

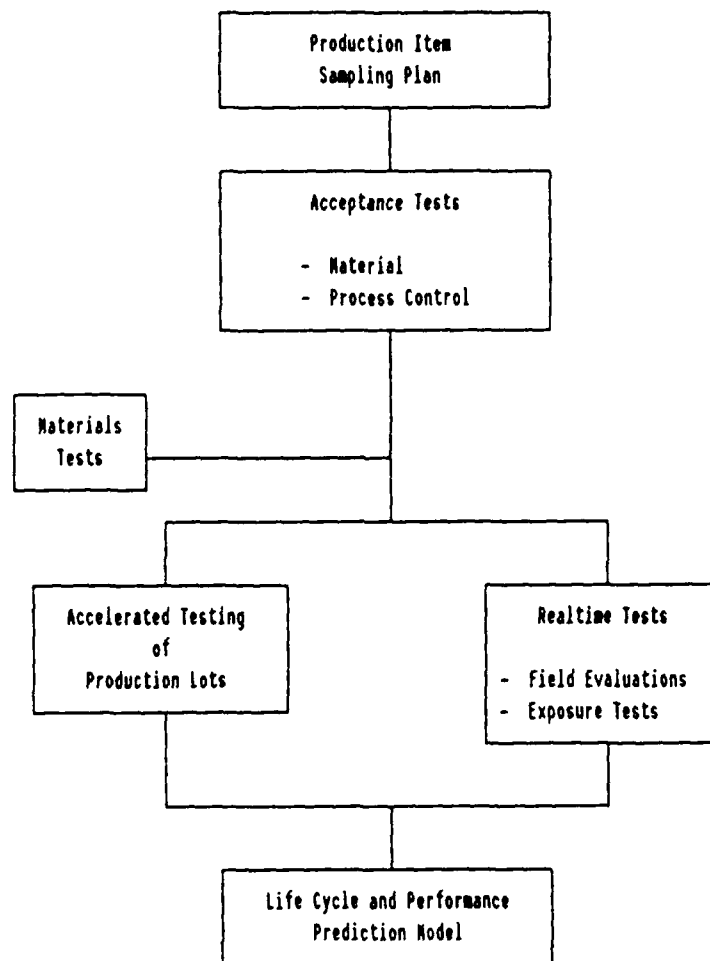


Figure 7. Container production-lot testing

refine performance prediction models. The production-lot testing scheme shown in Figure 4 has the objective of building confidence in and testing the accuracy of the performance prediction models developed for the container materials during the development and qualification test efforts.

The full-scale item testing shown in Figures 5 through 7 can run concurrent with the materials testing. Full-scale item testing, as for the materials testing, includes development, qualification and production-lot tests. Full-scale item tests involve container properties measurements, real-time and accelerated environmental tests. Full-scale item functional test data combined with container material data can then be used for overall life cycle predictions.

4.0 GENERAL TEST PLAN

4.1 Definition of Logistics Chain Environment and Container Acceptability Limits

4.1.1 Definition of Environmental Parameters

The logistics chain environment must be characterized in order to establish environmental test conditions, set materials properties requirements and identify container lifetime requirements. Temperature, humidity in terms of vapor pressure, and time at condition information at the environmental extremes should be considered as a minimum. Actual environmental data for the logistics chain are preferred, however, in the absence of actual data Mil-Std-210B and AR-7038 may be used as guidelines.

The solar radiation environment of the logistics chain should also be characterized. Total solar radiation below 3000nm maybe used to characterize the solar environment of the logistics chain. Total ultraviolet radiation below 385nm should be used for intercomparison and normalization of test data obtained from both accelerated and real-time tests. 308 MJ/m² ultraviolet below 385nm is a typical yearly average for the tropical Florida environment while 333 MJ/m² ultraviolet below 385nm is the typical yearly ultraviolet radiation for the Arizona desert environment. 308 MJ/m² ultraviolet is typically used in the commercial sector to normalize accelerated test conditions to real-time test conditions.

4.1.2 Definition of Container Acceptability Limits

4.1.2.1 Container Materials

Acceptability limits for critical container materials properties must be set in order to determine end points for testing, identify specific measurement techniques and to prepare lifetime

prediction models. The limits of acceptability for materials are typically described in terms of engineering properties such as tensile strength, impact strength, modulus, creep, etc. Other properties may be used depending on the availability of appropriate test specimens, test objectives and material properties of interest.

4.1.2.2 Container Functional Property Limits

Acceptability limits must also be set for the container item. Moisture vapor transmission, sealability and impact resistance are functional properties of major concern. Other factors which could influence acceptability limits include palletization loads and container component failure.

4.1.3 Documentation

Tables 1 through 3 exhibit sample documentation which summarizes logistics chain, container and container material information of the type required before a suitable test scheme can be designed. Other formats are acceptable. Other pertinent information should also be recorded.

Table 1. Example logistics chain information

Container item description:	155mm propelling charge container
Typical load plant environment:	13°C mean temperature, 20 dynes/cm ² mean moisture vapor pressure
Estimated time at temperature extremes during permanent storage:	183,960 hrs. at 25°C 78,840 hrs. at -2°C
Estimated time at moisture vapor pressure extremes during permanent storage:	131,400 hrs. at 11 dynes/cm ² 131,400 hrs. at 37 dynes/cm ²
Solar radiation environment at permanent storage site:	10.4 MJ/m ² total daily average
Comments:	Item could spend more than one year at load plant before shipment

Table 2. Example checklist describing plastic ammunition container material property acceptability limits

Material description:	Pigmented high density polyethylene, Phillips HXM 50100
Material function and processing information:	Blow molded ammunition container bodies 0.25 in. (0.64mm) minimum thickness
Nominal properties:	See vendor technical data sheet
Material sensitivities and comments:	1) Susceptible to ultraviolet degradation 2) Increases in percent crystallinity affect physical properties
Required lifetime:	Greater than 30 years indoor storage; two-year outdoor storage

<u>Critical properties</u>	<u>Test method</u>	<u>Acceptability limit</u>
Tensile strength	ASTM D638 Type IV, 2 in./min. (50mm/min.)	3600 psi (26 MPa) after 20 years
Modulus	ASTM D790	175,000 psi (1206 MPa) after 20 years
Spectral reflectance	ASTM E903	15% max. at 600nm after 616 MJ/m ² ultraviolet radiation
Percent crystallinity	ASTM E793	TBD
Impact resistance	ASTM D256	TBD

Table 3. Example checklist describing plastic ammunition container functional property acceptability limits

Container item description:	155mm propelling charge container	
Functional description of item:	Riveted handle, screw on cap with O-ring, and vent port, fiberboard reinforcing tube inside container body.	
Required lifetime:	Greater than 30 years indoor storage; two-year outdoor storage.	
<u>Critical properties</u>	<u>Test method</u>	<u>Acceptability limit</u>
Moisture gain	Not specified	Less than 0.00Xg after 30 years
Cap seal break pressure	Not specified	No change from original after 30 years
Handle integrity	Not specified	No change from original after 30 years
Seal integrity	Not specified	No change from original after 30 years as shown by moisture gain data and internal pressure decay data.
Drop impact	See drawing	No change from original after 30 years

4.2 Container Material Property Tests

4.2.1 General

Container material property tests are to be used to evaluate container materials during environmental exposure tests. The environmental exposure tests to be conducted are categorized into screening, qualification, and production phase testing. Screening tests have the objective of identifying candidate materials which are likely to exhibit stable physical properties over a two-year outdoor storage period early in the material life cycle. Promising materials would be subjected to additional testing in order to be qualified for use in specific packaging container applications. Production testing has the objectives of identifying lot-to-lot variability and to refine performance prediction models.

Screening tests, which should cover assembly, subassembly, and component materials, will also provide information on the compatibility of materials in specific packaging applications. Real-time and accelerated exposure test procedures are to be used to characterize materials failure modes, such as cracking, fading, embrittlement or loss of impact strength, and to establish appropriate accelerated test parameters for subsequent qualification and production lot testing. Real-time exposure testing must be included in each phase of testing to serve as a point of reference for evaluating the results of accelerated testing and determine confidence levels for performance prediction models.

Typical material test procedures and properties for two types of container materials are listed in Table 4. A similar set of measurements should be selected for each material on the basis of the container material performance properties to be investigated.

4.2.2 Sample Material Tests and Evaluations

4.2.2.1 Optical Properties

Sample materials tests and evaluation procedures are to be selected on the basis of material properties critical to container functional requirements. The measurements selected must provide a quantitative measure of change in properties resulting from environmental exposure in order to determine a rate of change with respect to environmental conditions.

Table 4. Summary of typical material tests

<u>Material</u>	<u>Properties measured</u>	<u>Measurement technique</u>
Pigmented high density polyethylene	Spectral reflectance	ASTM E903
	CIE color	ASTM D2244
	Tensile strength	ASTM D638
	Thermal decomposition	TGA
	Heat of fusion, heat flow and degree of crystallinity	DSC
	Coefficient of thermal expansion	TMA
Glass reinforced polyester composite	Spectral reflectance	ASTM E903
	CIE color	ASTM D2244
	Thermal decomposition and composition changes	TGA
	Storage and loss modulus, and damping	DMA
	Coefficient of thermal expansion	TMA

Optical properties, such as reflectance, transmittance, or color may be used to evaluate visual changes or determine the extent of surface changes. Changes in optical properties often precede changes in physical or bulk properties, especially after outdoor exposure. Specific test methods such as ASTM E903 and D2244 are used for these purposes. Other test methods having sufficient sensitivity to detect changes in specific materials are also appropriate. The noted test methods describe requirements for sample preparation, instruments, instrument operation, and reporting.

4.2.2.2 Physical Properties

Physical property measurements are also to be used to provide a quantitative measure of change in properties resulting from environmental exposure and are to be selected on the basis of critical container properties. Physical property measurement results, unlike optical property measurements which describe surface changes only, depend on both surface and bulk properties. Therefore, measurement speeds and conditions must be selected with great care in order that measurement data represents the performance of the material when used in container items.

A wide range of standardized physical property test methods exist which are appropriate for container materials at every level of assembly. A few of these tests are ASTM D256, D638, and D790. Tests of these types are often used successfully for environmental screening tests and to provide engineering information. The use of these measurement techniques for performance prediction models must be confirmed empirically by relating the specific test method and results directly to a performance property of the container. This is required since test specimens are rarely fabricated using actual container items and as a result of processing differences may not truly represent the material performance in a container.

4.2.2.3 Thermal Properties

The performance of polymeric materials in outdoor and indoor storage environments depends greatly on the thermal properties of the material. The use of thermal analysis techniques therefore offer practical and accurate methods for determining the useful lifetime of many plastic materials. The data obtained from thermal analysis measurements are also well-suited to modeling because the data are closely related to the chemical and thermal processes that occur as a result of specific material property degradation mechanisms. These mechanisms, once related to the polymer structure dependent properties, can be used to predict changes in basic engineering properties.

Whereas the successful use of physical property tests depends on sample geometry and test parameters as related to the functional properties of the container items; the successful use of thermal analysis depends on the ability to manufacture reproducible test specimens and quantitatively relate test data to physical phenomenon. The ability to

manufacture reproducible test specimens is primarily a materials-related problem area and may prevent the use of thermal analysis techniques. Quantitative relationships between physical phenomenon or properties and thermal analysis test data must be developed empirically. Therefore, an ideal measurement program would involve both physical property tests for engineering data and thermal analysis tests for basic materials property data.

A number of standardized thermal analysis test methods may be found in the ASTM. These include, but are not limited to ASTM D3418, D4065, D4092, and E793. These methods may require modification to accommodate specific materials. Scientific literature should also be referred to for appropriate techniques for specific materials.

Several thermal analysis techniques are particularly well-suited for the thermoplastic, elastomeric, and reinforced composite materials used for packaging containers. These are Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analysis (DMA), Thermogravimetric Analysis (TGA), and Thermomechanical Analysis (TMA).

DSC is used to follow thermodynamic changes in container materials by measuring the heat flow into and out of test samples. Physical transitions such as T_g , melting temperature and crystallization and; chemical transitions such as decomposition are used to study both physical and chemical processes. The degree of crystallinity, as related to the heat of fusion has a fundamental effect on material physical properties such as modulus, permeability, density and melting point. The decomposition temperature obtained from DSC relates to the oxidizable groups present in the material. Lower onset of decomposition temperatures indicate that fewer oxidizable groups are present in the material or the material has been preoxidized. The onset of decomposition temperature is not a quantitative value in respect to specific physical properties in the sense that it only represents the temperature at which the material decomposes after passing through the melt phase. The stability of the polymer, and thus the stability of properties which are dependent on the oxidation state of the polymer, is related to the decomposition temperature. Therefore, as a temperature value it can be used to indicate the oxidative stability of the polymer. The heat flow value associated with the onset of decomposition temperature is the area under the decomposition curve. It represents the energy required to decompose the sample and is dependent on the molecular weight, mass, and surface area of the sample in addition to the thermal properties of the material.

A second example of the use of thermal analysis to predict lifetime is the use of TGA to study decomposition kinetics. This technique assumes first order kinetics and uses extrapolation to estimate the long lifetimes encountered at normal use temperature. Further, the technique assumes that the limit of acceptability, or lifetime, of the material is dependent on the thermal stability of a polymer or the presence of a stabilizer, or a combination of the two. The thermal decomposition of materials is related to the presence of stabilizers and various additives in addition to the stability of the polymer. TGA is useful in studying various stages of thermal degradation and the compositional changes that occur during

the ageing process. However, the TGA data must be associated with a functional property in order to predict useful lifetimes. TGA can also provide structural property data to the extent that properties are dependent on the chemical makeup of the material.

TMA is used to determine dimensional changes in materials as a function of temperature. This measurement technique provides information on the thermal expansion, T_g and the softening point. DMA is closely related to TMA. DMA provides modulus and damping data. Storage and loss moduli and the loss tangent can be used to evaluate the integrity of the adhesion between the polymer and fiber reinforcement in the case of composite materials. These properties also relate to crystallinity. Thus, DMA can be used to study other properties such as creep, flexural modulus, and shear modulus.

4.3 Container Material Screening Tests

4.3.1 Outdoor Environmental Tests

As noted in paragraph 4.2.1, the objective of materials screening tests is to identify materials which retain critical properties after a two-year outdoor storage test. Test specimens are to be manufactured from a single material batch or lot to minimize lot-to-lot differences and processing effects on test results. Test specimens are to be prepared in sizes, shapes, and sufficient numbers needed to meet the requirements of the specific measurement procedures selected to evaluate properties during the course of a two-year environmental exposure test.

Properties measurements are to be made prior to the exposure test and at predetermined intervals covering a minimum of a two-year period. Exposure test sites should be selected to represent the extremes of the logistics chain environment. As a minimum, the material should be tested in a desert environment and in a tropical environment.

The intervals at which properties measurements are to be made should be based on 3, 6, 12, 18, and 24 month equivalents of total ultraviolet radiation below 385nm. If historical total ultraviolet radiation data is not available for the exposure test site used, measurement intervals are to be based on a one-year equivalent of 308 MJ/m² total ultraviolet below 385nm. Thus, measurements are to be made approximately after 77, 154, 308, 462, and 616 MJ/m² of ultraviolet radiation have been deposited on sample surfaces.

Procedures for sample mounting, data collection, and reporting, applicable to plastic materials, are described in ASTM D1435. Exposure testing may be stopped prior to 24 months if measurement data suggests a failure.

4.3.2 Accelerated Environmental Tests

4.3.2.1 Accelerated Natural Outdoor Exposure

Accelerated natural outdoor testing is limited to the desert environment. Test procedures, sample mounting and reporting requirements are covered in ASTM G90. Test specimens are to be manufactured from a single material batch or lot in sufficient numbers to conduct a two-year equivalent test. Test specimens are to be prepared in sizes and shapes needed to conduct the specific measurements required to evaluate material properties.

Properties measurements are to be made prior to the exposure test and at predetermined intervals covering a minimum of a two-year equivalent ultraviolet radiation exposure. The intervals at which properties measurements are to be made should be based on 3, 6, 12, 18 and 24 month equivalents of total ultraviolet radiation below 385nm. A one-year equivalent ultraviolet exposure is to be based on 308 MJ/m² total ultraviolet below 385nm.

Monitoring sample temperature to establish temperature extremes during the test is also recommended. At least one representative test specimen should be used for this purpose. Summertime test start dates are also recommended to maximize solar radiation and temperature during the initial portion of the test. Exposure testing may be stopped prior to the equivalent of 24 months of total ultraviolet exposure if measurement data suggests a failure.

4.3.2.2 Mil-Std-810D Tailored Simulated Solar Radiation Tests

Method 505.2 - Procedure II of Mil-Std-810D is to be used as a guideline for general procedures and equipment requirements for materials screening tests. Procedure II describes a steady state test, which when combined with elevated temperature and humidity conditions may be used to evaluate the effects of extreme temperature and humidity in a solar radiation environment.

As described in Method 505.2, the solar simulator to be used produces approximately a one-sun intensity, which when operated continuously can provide over twice the total energy of the natural sun. However, the screening tests are to be scheduled on the basis of total ultraviolet below 385nm, rather than on the basis of total radiation as described in the standard. 308 MJ/m² is to be used as a one-year equivalent. As noted for the environmental tests described in the preceding paragraphs, properties measurements are to be made prior to the test and after 77, 154, 308, 462, and 616 MJ/m² of total ultraviolet irradiation.

The temperature to be used during the test should be based on the maximum recommended service temperature for the material. The test chamber temperature should be adjusted to obtain a sample temperature

just below the maximum service temperature when irradiated. The test temperature will depend on the absorptivity of the material. This procedure requires that at least one representative test specimen be instrumented with a thermocouple and be located within the solar simulator target area. Humidity should be set to just below the condensation point and maintained at that level throughout the test.

Reporting requirements are described in Mil-Std-810D. Procedures for sample mounting and data collection will depend on the design of the specific test chamber to be used. Samples should be normal to the simulated solar radiation and located within a target area with a defined intensity and uniformity. Exposure testing may be stopped prior to the equivalent of 24 months of total ultraviolet exposure if measurement data suggest a failure.

4.3.2.3 Xenon Arc Lamp Environmental Cabinet Tests

ASTM G26 Method A describes a standard practice for operating xenon-arc lamp exposure apparatuses and is to be used as a guideline for general procedures and equipment requirements. The method involves continuous ultraviolet radiation exposure with an intermittent water spray cycle. Test equipment capable of temperature and humidity control during the ultraviolet radiation exposure is available and is preferred for screening tests.

The xenon arc lamp should be fitted with filters to remove ultraviolet below 295nm in order to simulate the spectral distribution of natural ultraviolet. The designs of many of the commercially available apparatuses do not allow continuous direct measurement of sample temperature during the test. However, an approximation of the actual sample temperature during the test can be obtained by holding a representative test specimen, instrumented with a thermocouple, stationary while being irradiated. As discussed in paragraph 4.3.2.2, sample temperatures close to the maximum service temperature occurring in the logistics chain during outdoor exposure are recommended. In the event the exposure test temperature cannot be established in this manner, the guidelines for using black panel temperature are to be followed. Humidity during the test is to be maintained continuously at a level just below the condensation point.

The intensity of ultraviolet from xenon arc lamps varies between lamps and as the lamps age during the course of a test. However, most commercially available test equipment will provide 308 MJ/m² of total ultraviolet below 385nm in approximately 1,000 hours of continuous exposure. As for the other exposure tests described in previous paragraphs, 308 MJ/m² of total ultraviolet is to be used as a one-year equivalent. The measurement intervals to be used for a two-year equivalent test are 77, 154, 308, 462, and 616 MJ/m². Equipment manufacturer or test service recommendations for the time period required to obtain the noted irradiation levels are to be used if ultraviolet irradiance cannot be characterized for the lamps used for the test.

Reporting requirements and sample mounting procedures are described in ASTM G26. Exposure testing may be stopped prior to the equivalent of 24 months of total ultraviolet exposure if measurement data suggest a failure.

4.3.3 Container Material Compatibility Testing

Ammunition containers are often composed of a variety of plastics, metals, elastomers, and cellulosic materials in a wide range of thicknesses. These materials could also contain a variety of processing aids, have lubricants, paints, or sealants applied to their surfaces during the assembly of the container. The reaction of one component of the container with another could adversely affect the performance of the container assembly. Preliminary compatibility tests, as part of the two-year outdoor screening tests, recommended for exterior container materials, are therefore also recommended.

The details of test specimen design must be based on a consensus between design and test engineers since materials compatibility tests generally entail nonstandard specimen geometries and often times subjective evaluations of performance. Wherever possible, standardized measurement techniques, such as described in paragraph 4.2, should be used. The environmental test procedures described in paragraph 4.3 should be conducted on appropriate test specimens.

Examples of material compatibility tests might include the following container design situations:

- Rivets or other fasteners in plastic
- Gaskets or seals exposed to the environment at seams
- Identification inks, coatings, or paints used on container surfaces
- Residual mold release agents on container surfaces
- Decontamination chemical effects
- Cleaning agent effects
- Outer wrap or strapping effects on container materials
- Dissimilar plastics

4.3.4 Preliminary Performance Prediction Models

The results of the screening and materials characterization tests can be used to predict materials performance in outdoor environments to the extent that the environmental tests recommended were based on a two-year or two-year equivalent ultraviolet basis. The use of solar radiation environmental testing should present a worst case environment for materials early in their life cycle. However, it must be recognized by the design and test engineers that the performance of the material in actual container items may also depend on the thermal degradation of the material, the effects of which may not result from the screening tests described in the preceding paragraphs.

Screening test data should therefore be used for engineering information and to provide guidelines for the qualification testing of candidate materials which exhibited acceptable performance during the screening tests. The screening test data can be added to the qualification test data since, as will be discussed in following paragraphs, the qualification testing will consider the longterm environmental performance of the materials in similar environments in addition to the other environmental aspects of the logistics chain. Preliminary performance prediction models can be prepared from screening test data that includes appropriate time and time at temperature information using the modeling concepts discussed in paragraph 4.4.10.

4.4 Container Material Qualification Testing

4.4.1 General

Candidate materials which exhibit promise for meeting performance requirements after a two-year outdoor storage early in the material life cycle are to be subjected to further testing and characterization in order to qualify for use in container items. Qualification testing should ideally be conducted prior to prototype production. However, the advantages of concurrent prototype item development with material qualification should also be considered. If certain materials already have a substantial performance or test history, the risks in conducting concurrent item development and material qualification are obviously reduced. The qualification test program to be used has therefore been organized to allow flexibility in how the program is to be implemented by addressing basic materials testing and component testing.

The materials qualification tests have the objectives of determining the effects of manufacturing variables, material lot-to-lot and batch-to-batch differences and the development of longterm performance prediction models. The confidence in the results of the qualification tests and resulting performance prediction models will depend on the extensiveness of the testing conducted. The use of statistical sampling plans and statistical experimental designs is highly recommended. A typical sampling plan may be found in Mil-Std-414 or ASTM D1898. The general literature is also recommended for information on statistical experimental design.

The development of material performance prediction models will be based on quantifying temperature dependent thermal and ultraviolet induced properties degradation. Test data will be evaluated in terms of Arrhenius relationships with the objective of determining an apparent activation energy which can be used to predict performance over the temperature extremes of the logistics chain environments in an early stage of a production program and to evaluate the effects of material formulation and processing changes in later stages of production. The following considerations are instrumental to the development of a performance model.

- Until substantial experience is obtained with a particular material in a particular container design, the performance prediction models must be continually updated and tested by field performance data.
- The efficacy of typical and newly developed accelerated test methods must be proven for specific materials used in particular container designs.
- The models developed must be based on materials properties test data that can quantify pertinent degradation mechanisms. This may require that a number of materials properties be studied in order to identify the properties most critical to performance.
- Real-time testing must be a part of the test program in order to test the models developed and to provide test items which can be used to refine the models with respect to predicting performance when container items are exposed to outdoor storage environments late in their life cycle.

4.4.2 Materials Qualification Test Program Definition

The components and assembly design of a particular container are to be evaluated to determine which materials properties are best suited to identifying and characterizing environmental performance. It is assumed that screening test data will be available during this analysis to support and justify the sample plan, test specimen design and environmental test parameters to be used. The manufacturing process intended for the container item is to be reviewed to identify processing conditions which could affect the performance of the material in the container design. This review is to be used to define the process variables that must be evaluated as part of the qualification test program. Acceptability limits for the properties selected are to be established. An environmental test matrix, including real-time testing, accelerated testing and the tests required to determine activation energy, is to be defined.

The qualification testing described in the following paragraphs may be used at the various stages of the production program. Testing of preproduction and production lot materials is advisable. Real-time test results should be used for reference and referee purposes to confirm accelerated test results and the prediction models developed from activation energy determinations. Activation energy determinations should be considered

as the first scheduling priority since the test schedule involved with this testing is likely to provide data in a shorter time period than the other tests. When possible, real-time testing should be initiated concurrent or prior to the activation energy determinations since, being based on real-time, these tests will take longer to accomplish than other tests.

A conceptual approach to determining apparent activation energies needed for performance prediction models is described in paragraphs 4.4.4 and 4.4.5. The approach is presented as a concept because the performance prediction models needed for plastics used in ammunition packaging requires a high degree of certainty that is not usually carried out in a practical sense by the general scientific community and because of the diversity of materials and container designs. Thus, standardized procedures for performance prediction modeling specifically applicable to ammunition packaging applications are not available. However, specific test methods, found in the ASTM standards and general literature, can be used to obtain materials property data for performance prediction modeling.

The use of activation energy, or rather using Arrhenius relationships, to predict longterm performance may not be suited for all materials or all container designs. The activation energy determinations outlined in paragraphs 4.4.4 and 4.4.5 have the objective of identifying a materials constant that relates material properties to temperature and time and since accelerated testing techniques are used, this approach to performance prediction model development will at a minimum enhance the understanding of plastic materials used in ammunition packaging if not become the accelerated test method of choice. The concept involves making materials properties measurements after several intervals of environmental exposure at four different temperatures, separated by at least 10°C, covering a range close to the logistics chain temperature range. The use of test temperatures much higher than actual service temperatures may detrimentally affect the accuracy of the resulting performance prediction models by causing unnatural degradation mechanisms. An isothermal method is recommended, rather than an isoconversional method where materials are tested to a predetermined endpoint, because it allows flexibility for a variety of test sample designs, measurement techniques and materials. Both approaches to determining activation energy should be evaluated however, because the isoconversional approach entails much shorter time periods to obtain data.

A realistic humidity level, described as moisture vapor pressure, representative of the logistics chain temperature range will be selected and held constant during the tests in order to reduce the influence of moisture as a variable on the test results. Activation energy determinations have been organized into two categories. The first is based on thermal degradation of properties for indoor storage environments and the second is based on ultraviolet exposure for outdoor environments. In order to predict container performance in outdoor environments near the end of the life cycle, it will be necessary to conduct additional studies on test specimens which have undergone elevated temperature and humidity exposure for a significant period of time.

The accelerated testing and real-time testing have also been categorized into indoor and outdoor environments. As noted above, real-time testing is to be included in the test program to serve as a reference and as a referee to accelerated testing. The real-time and accelerated tests follow accepted commercial and military practices and therefore the results from this testing may be compared to an already existing body of test data for some materials.

4.4.3 Activation Energy Determination Equipment Requirements

The equipment to be used for activation energy determinations can be a complete facility or a combination of components that allow materials to be subjected to continuous constant condition environmental tests at elevated temperature with various relative humidity levels. A concept drawing of the equipment is shown in Figure 8. Ideally, materials should be tested using the same ultraviolet radiation for tests at all four temperatures. In the event that a complete system of equipment is not available, a separate test at each temperature may be used. Activation energy determinations are to be made with and without simulated solar radiation. Simulated solar radiation can be provided by any combination of sources which will produce an acceptable spectral energy distribution and at least a one-sun equivalent intensity in the ultraviolet region below 400nm. These sources could include xenon, fluorescent, and metal halide lamps. Until test data is obtained that suggest otherwise, control and accurate recording of test conditions and sample temperatures are to be considered more important than the actual test conditions used.

Table 5 describes the temperature and humidity relationship for the temperature range that is recommended for the activation energy determinations. These parameters dictate the environmental chamber equipment requirements for both the indoor and outdoor environmental testing. The vapor pressure requirement at the high and low ends of the temperature scale in Table 5 may not be controllable using typical equipment. The available test equipment shall therefore be set to control humidity to the required level and the chamber humidity environment shall be intercalibrated with temperature to determine the moisture vapor pressure occurring at the set-point temperature and humidity. Typical or average moisture vapor pressure values obtained from the intercalibration shall be used for purposes of reporting and data analysis. The temperatures and humidities listed in Table 5 are typical of temperate, hot-humid and hot-dry climates and thus represent a range of conditions that could occur in the logistics chain environment. The higher temperatures noted can occur in the hot-humid and hot-dry climates when pigmented plastics are exposed to solar radiation on hot days.

Table 5. Temperature and vapor pressure environment data

<u>Air Temperature (°C)</u>	<u>Dew Point (°C)</u>	<u>Relative Humidity (%)</u>	<u>Vapor Pressure (dynes/cm²)</u>
30	28	87	37
35	28	68	37
40	28	51	37
45	28	40	37
50	28	31	37
55	28	24	37
60	28	19	37
65	28	15	37
70	28	12	37

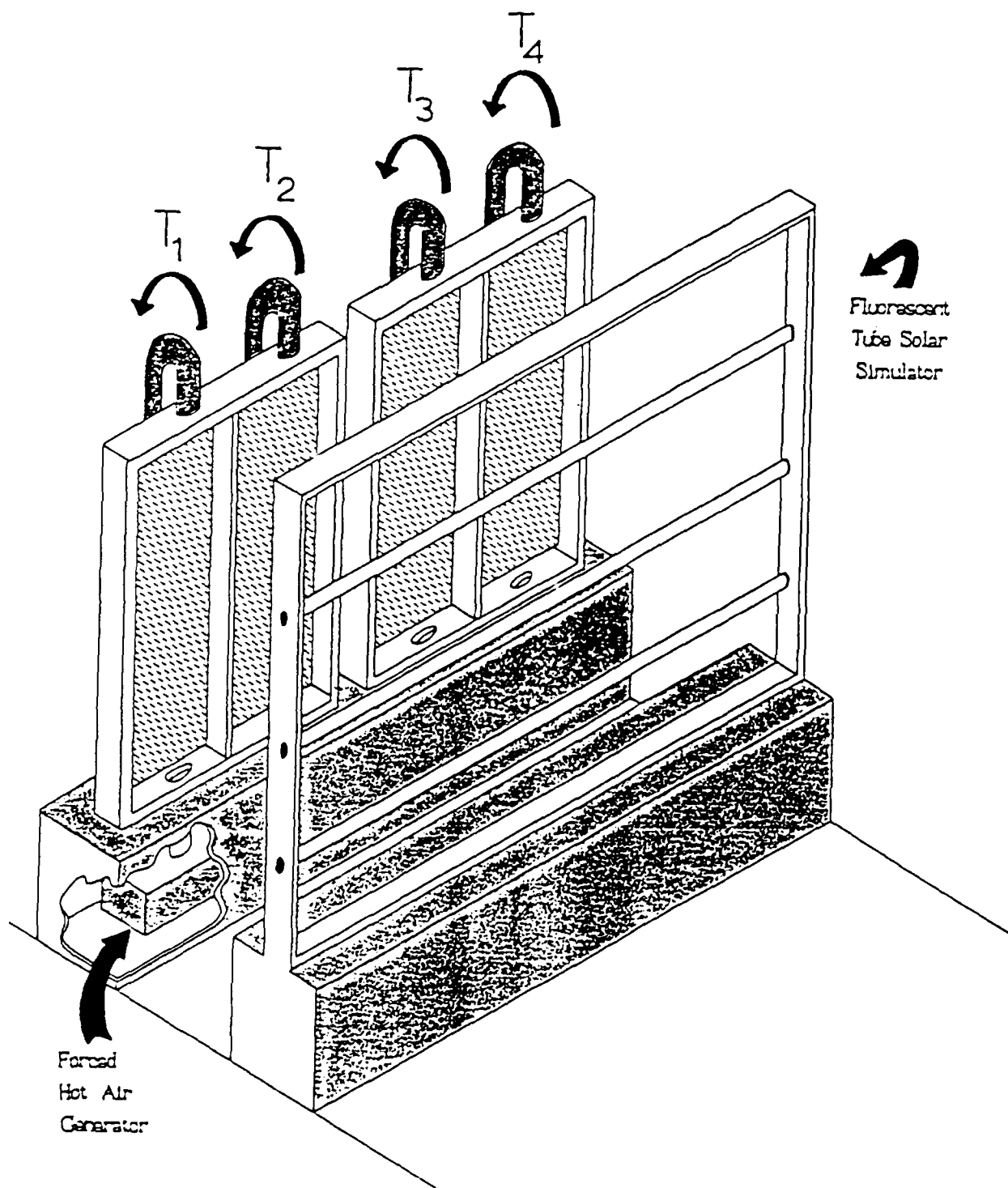


Figure 8. Activation energy determination test equipment concept

The solar radiation source to be used for activation energy determinations for the solar radiation environment shall have a spectral energy distribution as described in Mil-Std 810D. The intensity of the ultraviolet radiation from the source may be greater than prescribed by Mil-Std 810, however. Filters are to be used to eliminate radiation below 295nm. The spectral energy and intensity distributions shall be mapped at the target area periodically. The uniformity of radiation at the target area should be a minimum of $\pm 10\%$. Total ultraviolet radiation below 385nm shall be measured and recorded continuously during tests with simulated solar radiation.

The test chamber equipment must also provide for continuous monitoring and recording of temperatures for at least one test specimen of each material being tested in the same test. Temperature data may be taken as an hourly average or at hourly intervals since constant conditions will be used during the test. Mil-Std 810D shall be referred to for equipment operation, maintenance, and reporting requirements that are appropriate for the tests being conducted.

4.4.4 Activation Energy Determinations for Thermal Degradation of Materials Properties

General scientific literature and the ASTM provide several thermal analysis techniques for directly determining activation energy values. These techniques are useful from the standpoint that they provide lifetime data in short periods of time. These techniques usually involve testing the materials at temperatures much higher than actual service temperatures which contribute uncertainty to the resulting lifetime models. They are also limited to thermal degradation without consideration of the moisture environment. The use of these direct methods therefore requires some tradeoff between the accuracy of the lifetime prediction and the time required for the testing. Thermal analysis, as a measurement technique used in conjunction with the other measurement techniques noted in paragraph 4.2 however, can be used to obtain property degradation data which can be used to develop more realistic models following the exposure test scheme suggested below.

An isothermal exposure method for determining activation energy is recommended. The recommendation is made on the basis that the method will allow accurate determination of activation energies and not be limited to any single material property or property measurement technique. The versatility of this method is allowed by the exposure test equipment concept described in paragraph 4.4.3 which can be scaled for any type of test specimen size and shape. The environmental exposures required can be carried out using any combination of ovens, temperature/humidity cabinets or environmental chambers since a solar radiation source is not needed. Thus, any combination of material property measurements can be used to characterize materials' performance.

As outlined in paragraph 4.4.2, appropriate properties for the material to be tested are to be selected and test specimen geometries are to be defined. Test sample materials are to be obtained in quantities sufficient to conduct the necessary exposure testing. Test specimens are to be obtained from at least two different lots of materials to determine lot-to-lot variations. Specimens representative of different processing and manufacturing parameters should also be included in the test program.

Four specific test temperatures are to be selected for exposure testing on the basis of the logistics chain environment and the maximum recommended service temperature for the material to be tested. The temperature and humidity conditions listed in Table 5 can be used as a guideline. Other factors that should be considered relate to the thermal properties of the material. These factors include, but are not limited to, decomposition temperature, crystallization temperature, glass transition temperature, and melting temperature. The four temperatures should be separated by a minimum of 10°C. The objective of this selection process is to identify the highest temperatures that the materials can be tested at without changing degradation kinetics. Higher temperatures will allow shorter exposure test times.

Material screening test data is to be reviewed with respect to the time at temperature performance of the material in order to establish the exposure test schedule and to set appropriate exposure intervals at which properties are to be measured. A minimum of six exposure intervals should be planned for materials properties measurements including pre-exposure and initial characterization measurements. The exposure schedule and measurement intervals are to be set so that properties data is obtained after the end of the induction phase of the properties degradation curve and primarily in the rate controlling region of the properties degradation curve. Setting the properties measurement intervals on this basis assumes that the material will fail or reach the limit of acceptability in the rate controlling region of the degradation curve. A material which fails in the induction period may not allow accurate performance prediction models to be developed. A conceptual materials property degradation curve is shown in Figure 9, where property measurement data, P, are plotted against time or dose.

Additional test specimens, when available, should be subjected to the environmental exposures in order to prepare test specimens with a significant degree of known test history for subsequent testing with simulated solar radiation, the study of longterm ageing effects or other appropriate tests and evaluations. Test specimens generally are not to be returned to the exposure test after measurements have been made. Test specimens should be visually evaluated before measurements and again after the measurement when the specimens have been stored for some period of time at typical laboratory or room conditions. Property measurement data are to be recorded with the environmental exposure test history, test conditions, and other pertinent information.

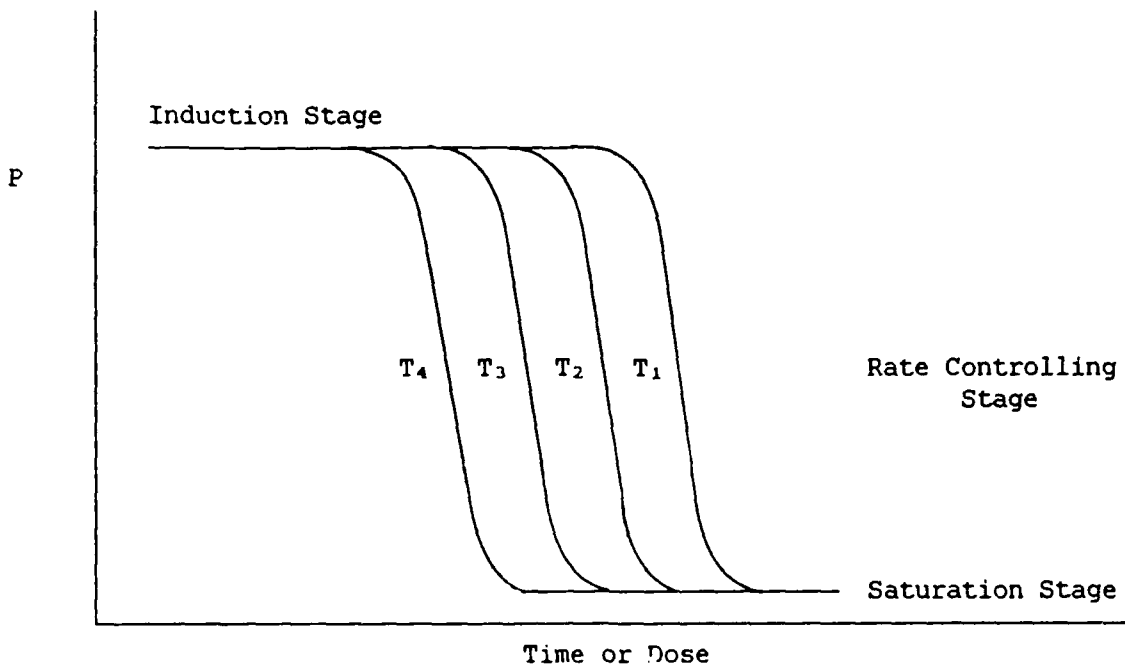


Figure 9. Materials property degradation curves

4.4.5 Activation Energy Determinations for Ultraviolet Degradation of Materials Properties

Activation energies for ultraviolet degradation of materials are also to be determined using isothermal exposure testing at four different temperatures with simulated solar radiation. As noted in paragraph 4.4.3, the source of the radiation must be well characterized with respect to wavelength distribution and intensity. Appropriate properties for the material to be tested are to be selected and test specimen geometries are to be defined. Test sample materials are to be obtained in quantities sufficient to conduct the necessary exposure testing.

Test specimens are to be representative of lot-to-lot variations and different processing and manufacturing parameters. Newly manufactured test specimens are to be tested to obtain data for models which predict outdoor performance early in the life cycle. Test specimens which have undergone prior real-time exposure with or without solar radiation are to be tested to obtain data for models which predict outdoor performance late in the material life cycle. The prior history of the test specimens is to be documented in order to relate the results of the testing to the respective portion of the life cycle.

The four specific test temperatures to be used are to be selected for exposure testing on the basis of the logistics chain environment and the maximum recommended service temperature for the material to be tested. The temperature and humidity conditions listed in Table 5 can be used as a guideline. Other factors that should be considered relate to the thermal properties of the material. These factors include, but are not limited to, decomposition temperature, crystallization temperature, glass transition temperature, and melting temperature. The four temperatures should be separated by a minimum of 10°C. The objective of this selection process is to identify the highest temperatures that the materials can be tested at without changing degradation kinetics. Higher temperatures will allow shorter exposure test times. Exposure tests with solar radiation will usually require relatively short time periods to accomplish.

Test specimen temperatures while under a solar load will typically be greater than the environmental chamber setpoint temperature. The actual sample temperature during the test will depend on the absorption characteristics, the mass of the specimen and the thermal conductivity of the material. The environmental chamber temperatures used for this testing may therefore be lower than the chamber temperatures used for the thermal degradation activation energy determinations. Preliminary testing may be required to establish the appropriate chamber setpoint conditions with a solar load.

Material screening test data is to be reviewed, with respect to the time at temperature and resistance to ultraviolet radiation in order to establish the exposure test schedule and to set appropriate exposure intervals at which properties are to be measured. A minimum of six exposure intervals, on the basis of 308 MJ/m² of total ultraviolet below 385nm being an equivalent year of ultraviolet exposure, should be planned for materials properties measurements. These intervals include pre-exposure and initial characterization measurements. The exposure schedule and measurement intervals are to be set so that properties data are obtained after the end of the induction phase of the properties degradation curve and primarily in the rate controlling region of the properties degradation curve. Setting the properties measurement intervals on this basis assumes that the material will fail or reach the limit of acceptability in the rate controlling region of the degradation curve. A material which fails in the induction period may not allow accurate performance prediction models to be developed.

Additional test specimens, when available, should be subjected to the environmental exposures in order to prepare test specimens with a significant degree of known test history for subsequent study of longterm ageing effects or other appropriate tests and evaluations. Test specimens generally are not to be returned to the exposure test after measurements have been made. Test specimens should be visually evaluated before measurements and again after the measurement when the specimens have been stored for some period of time at typical laboratory or room conditions. Property measurement data are to be recorded with the environmental exposure test history, test conditions, and other pertinent information.

4.4.6 Elevated Temperature and Humidity Testing

Constant condition elevated temperature and humidity exposure tests are to be conducted to evaluate the performance of container materials in these environments and to prepare test specimens for solar radiation exposure testing and activation energy determinations which represent a late stage in the container material life cycle. Although the testing is to be conducted on a time at temperature or real-time basis, the tests will achieve a degree of acceleration by nature of the continuous elevated temperatures used. Conditions of elevated temperature with ambient humidity and elevated temperature with high humidity are to be used. Elevated temperature with ambient humidity testing can be conducted using mechanically assisted convection ovens, forced air ovens, or an environmental chamber. Elevated temperature with high humidity testing is to be conducted in an environmental chamber capable of maintaining constant conditions for long periods of time.

Polymeric materials generally contain a variety of stabilizers which allow the material to maintain desirable properties for long periods of time in typical service environments. Very often the action of these stabilizers depends on the thermal environment by reacting with the polymer, degradation products of the polymer or a chemical aspect of the environment such as moisture. The rate at which the stabilizers are consumed or decomposed at the surface and in the bulk of the material will greatly influence the stability of the properties of the polymer and thus the performance of the material in longterm applications. Elevated temperatures can also cause changes in the structure of the polymer which affect performance properties. Just a few of these changes include crystallization, molecular weight distribution, and crosslink density. Thermal analysis measurement techniques such as TGA or DSC can be used to characterize these features of the polymeric material and determine appropriate test temperatures by identifying critical temperatures where temperature dependent degradation mechanisms may be changed by the test temperature.

A test temperature is to be selected that will minimize the possibility of changing degradation mechanisms. The humidity level to be used during the elevated temperature/humidity tests should be selected similarly and defined in terms of moisture vapor pressure. The moisture vapor pressure used for this testing should be based on the maximum vapor pressure of the logistics chain environment and will very likely be representative of the hot-humid environment. The minimum temperature to be considered however, is the maximum temperature of the logistics chain environment that the container item will experience during its life cycle. Predetermination of a specific test and measurement schedule for this testing may not be possible because it is likely that more than one material will be tested during a single test in order to operate exposure test equipment at capacity and because of the wide variety of materials and materials stabilization schemes possible for container items. When possible, data obtained from screening tests and activation energy determinations should be used to

determine property measurement intervals, at least for the initial stages of exposure. The objective of the time intervals selected for properties measurements should be the development of a properties degradation curve, such as the concept shown in Figure 9.

Test specimens are to be representative of lot-to-lot variations and different processing and manufacturing parameters. Test specimens are to be obtained and tested in a quantity sufficient to conduct the exposure test long enough to reach the end of the induction phase of the material's degradation with enough sample material remaining at this point in the test to continue the test through the rate controlling phase, conduct activation energy determinations, and to conduct accelerated tests with solar radiation. Industry and military standard test methods such as ASTM D3045 and Mil-Std 810D are to be used as guidelines for equipment operation and maintenance, data, and reporting.

4.4.7 Accelerated Ultraviolet Exposure Testing

The objective of accelerated ultraviolet exposure testing for materials qualification purposes is the characterization of the outdoor performance of container materials both early and late in the materials' life cycle. Newly manufactured test specimens representative of at least two different lots of material are to be tested in order to characterize an early portion of the life cycle. Test specimens obtained after longterm exposure to the elevated temperature and elevated temperature/humidity testing described in paragraph 4.4.8 are to be used to characterize the performance of the material later in its life cycle. Test specimens subjected to other real-time exposure testing without solar or simulated solar radiation may also be included in this testing.

The accelerated ultraviolet exposure testing is to be based on a typical one-year equivalent of total ultraviolet for a minimum total of two equivalent years. As for other solar radiation testing discussed in this test plan, 308 MJ/m² total ultraviolet below 385nm is to be used as the basis for a one-year equivalency. The accelerated test procedures outlined in paragraph 4.2 and used for the screening tests are recommended. The same test and measurement schedule used for screening tests is also recommended. However, it is advisable that these tests be carried out until the materials reach a failure point in order to test the accuracy of the lifetime prediction models developed during the course of the test program. The specific industry and military standard test procedures followed are to be used as guidelines for equipment operation, data, and reporting.

4.4.8 Real-Time Outdoor and Indoor Storage Testing

4.4.8.1 Outdoor Real-time Testing

Real-time outdoor exposure testing used for qualification test purposes shall be conducted as described in paragraph 4.3.1 for up to two years. Desert and tropic environments are to be used at a minimum. Testing in temperate and arctic environments can also be conducted to provide additional data. Test samples representative of at least two production lots or batches are to be tested in sufficient quantities to conduct subsequent indoor exposure tests. The temperature of the sample materials should be measured and recorded continuously if possible. Where facilities do not allow continuous temperature monitoring, material temperature is to be measured on bright sunny days at various times during the year in order to approximate the maximum temperature during solar exposure under a variety of ambient temperature environments. Test specimens are to be removed periodically during the test to conduct property measurements in order to prepare a property degradation curve as shown in Figure 9. Measurement data and exposure test data are to be recorded as described in paragraph 4.3.1.

4.4.8.2 Real-time Indoor Exposure Testing

Real-time indoor exposure testing used for qualification test purposes are to be conducted using bunker or warehouse conditions in desert and tropic environments. Testing in temperate and arctic environments can also be conducted to provide additional data. Test samples representative of at least two production lots or batches are to be tested in sufficient quantities to conduct the test for as long as practical. Additionally, test samples which have been subjected to prior outdoor exposure are also to be included in the indoor exposure tests. Accelerated environmental test data should be used when available to set the duration of the real-time test and the intervals after which materials should be conducted which a frequency that will allow a property degradation curve, like that shown in Figure 9 to be prepared.

4.4.9 Cyclic Environment Testing

Naturally occurring temperature, humidity and solar radiation cycles can cause significant materials degradation, irrespective of the time at temperature effects resulting from the real-time and accelerated test procedures described in preceding paragraphs. This degradation is usually evidenced by surface cracking or craze and results from the mechanical stresses caused by the cycling of temperature or the absorption and desorption of moisture. A cyclic environmental test using environmental chambers is not recommended as an accelerated test for the outdoor environment since this equipment generally cannot be controlled to cause condition changes as quickly as naturally occurring events. Conversely the indoor storage

result in other modes of materials failure such as creep, fatigue or compression set. Thus, for the indoor environment constant condition exposure testing very probably would produce an acceleration of effects since the effects of typical cyclic environmental chamber tests would tend to be time at condition dependent. The effects of environmental cycles on material performance would also tend to be more significant late in the material's life cycle after stabilizing additives have been consumed or volatilized. The relation of moisture absorption and desorption to a real time base is even more complex since absorption and desorption are material specific and depend on temperature and humidity.

The use of cyclic environmental testing for qualification test purposes should be limited to evaluating materials properties at environmental extremes rather than as an accelerated test. The low temperature brittleness of a material as related to a transition temperature, or expansion and contraction features as related to temperature and humidity are two examples where cyclic testing could provide valuable data for qualification. The design of specific test procedures for such testing is however dependent on the inherent features of the material being tested.

4.4.10 Material Data Analysis and Modelling

The analysis of test data has two objectives. First is the determination of the appropriateness of a particular material for ammunition packaging applications on the basis of initial properties and environmental stability as shown by the results of screening tests. Second is the development of a performance database, comprised of both accelerated exposure test results and real-time test results, from which material property data can be used to prepare and test models which will predict the longterm performance of the material.

The approach recommended to mathematically correlate durability characteristics obtained from accelerated tests to real time tests is to attempt to kinetically relate a material's exposure-induced properties to the level of stress (e.g., exposure) taking into account structure-property relationships. This requires information on degradation rate constants as a function of structure and temperature-related changes in those rate constants. The development of complete expressions that predict performance apriori of the actual exposure tests is essentially impossible. This is due primarily to a lack of information on well-defined structure-property and stress-property relationships for the same properties. However, the degradation rates of certain important properties of plastics may be described by S-shaped curves in which the property, or retention of property, is plotted as the dependent variable as a function of the environmental stress. Plotted as change in property, the relationship takes the form where the independent variable acts as the driving force which is a combination of solar radiation, temperature, oxygen and moisture in this treatment of the ageing process. The successful application of this modelling approach depends on the availability of property data in the rate controlling region of the curves in Figure 9 and the identification of the limit of acceptability.

Examination of the concepts of induction, rate-controlled, and saturation processes is a prerequisite to a thorough understanding of the ageing process. The term induction as represented in Figure 9; is a convenient descriptor for the magnitude of the independent variable necessary to initiate the rate-controlled property change. More fundamentally, induction describes both the reaction rates specific to initiation and the quantity of stress required for the production of a sufficient number of defects, or "observable events," that are necessary in order to be measurable by whatever diagnostic tools are employed to assess the dependent variable. The rate-controlling step in Figure 9 is that portion of the reaction, or ageing process, that is represented by a single linear rate equation. More fundamentally, it is that time period of stress application after initiation where the concentration of reactive sites available for reaction obeys a well defined depletion rate. When this does not occur, either the rate controlling step cannot be mathematically represented as a linear relationship or the saturation phase has been reached. Obviously, saturation is a descriptor that is employed to describe the material condition in which most, if not all, reactive sites, or potential events, have been depleted during the application of stress.

The linear portion of a plot of change in property as a function of exposure can be described by writing a slope intercept equation, $y = mx + b$, where $\Delta P = mE + b$, m is the slope and b is the intercept. Solving for E , we obtain

$$E = 1/m \{ \Delta P - b \} \quad (1)$$

where $-b/m = i$, the intercept on the abscissa that is the induction measured in terms of the independent variable as exposure. Equation 1 can be written in terms of loss of property as a function of exposure, giving

$$\Delta P = m\{E - i\} \quad (2)$$

permitting prediction of durability of that property in the rate controlling step in terms of the exposure. It is important to note that few materials plot as a normal linear relationship defined by equation 2 and that generally a log-normal and sometimes a log-log plot is required to achieve a linear relationship. This is particularly true for systems with exponential or logarithmic decay in the availability of reactive sites, or where the stress such as ultraviolet radiation, exhibits such a decay as it penetrates into the material's matrix. In this case equation 2 becomes

$$\Delta P = m\{\log E - \log i\}, \text{ or } \log \Delta P = m\{E - i\} \quad (3)$$

Correlation of accelerated and real-time exposures should seldom, if ever, be performed on exposed, or stressed, materials in only the induction or the saturation phase since these expressions only explain linear portions of the ageing process. These regions are used in combination to define the entire exposure process; however, acceptance decisions should never be made when either the real-time or the accelerated exposure level is representative of one of these regions -- especially in the induction phase.

4.4.10.1 Kinetic Expressions of Ageing Processes

The instantaneous rates at which the concentration of reactive molecular sites change with time (both the creation of new and the depletion of existing species) in photochemical, oxidative, or photo-oxidative reactions may be represented by the derivative dC/dt -- which states a reaction velocity. In zero order reactions, we simply set the derivative equal to the proportionality factor "k", the value of which depends on the temperature as well as the order. In first, second and higher order reactions which may be also opposing, or back reactions, or secondary reactions of different order, these expressions become complicated by the mathematical description of the instantaneous concentrations of unreacted species, new species, etc. Nonetheless, it is instructive to examine the simple first order reaction $[mM + nN = \text{products}]$ where by definition the rate depends only on the concentration of, for example, M.

$$dM/dt = -kM \quad (4)$$

which, on integrating, becomes

$$\ln M = -kM + b \quad (5)$$

which is similar to equation 3.

The influence of temperature on these relationships is shown by a consideration of the Van't Hoff and Arrhenius equation. Arrhenius developed the relationship

$$d\ln(k)/dT = E/RT \quad (6)$$

from the Van't Hoff equation for the temperature coefficient of the equilibrium constant, where k is the specific rate constant, E is the material specific activation energy for the reaction and R and T are the gas constant and the absolute temperature, respectively. Integration of equation 6 yields the expression

$$\ln(k) = - E/RT + \ln(A) \quad (7)$$

where A is a constant of integration known as the Arrhenius factor. Equation 7 is useful for plotting exposure data to develop activation energies. If we consider that the reaction rate "k" can be described by the relationship dP/dt as the change of property with time and if we consider the expression dt to be represented by an incremental exposure then we can, as an example, expose a plastic for a specific ultraviolet exposure (in kJ/m^2) at several different temperatures and employ equation 7 to determine its activation energy. This can be done by plotting dP/dE_{uv} versus $1/T$ and computing the activation energy from the relationship $\tan \alpha = E/R$ from

$$dP/dE_{uv} = E/R (1/T) - \ln(A) \quad (8)$$

Placing equation 7 in exponential form, as presented in equation 8, a thermal reaction coefficient S for exposure of this plastic at temperature T can be developed as shown in equation 9.

$$S = dP/dE_{uv} = A e\{-E/RT\} \quad (9)$$

A thermal velocity coefficient V can be computed from the ratio of two thermal reaction coefficients S and S' normalized to an arbitrarily selected reference temperature T' such that

$$V = S/S' = e\{-E/RT\}/e\{-E/RT'\} \quad (10)$$

and since this velocity coefficient applies to the independent variable, or ultraviolet exposure in this example, we may now thermally adjust the measured ultraviolet exposure data used to expose a plastic whose activation energy we either know or have determined. Using this thermally adjusted ultraviolet irradiance data, we may now plot the change in property (or log of the change in property) versus the thermally adjusted ultraviolet exposure E_{tuv} (or log of exposure) to give for example,

$$\Delta P = m (\log E_{tuv} - \log i), \text{ or} \quad (11)$$

$$\log \Delta P = m (E_{tuv} - i) \quad (12)$$

These equations may be considered as representing a "unified exposure theory" that will permit the normalization of both ultraviolet irradiance data and exposure temperature in exposure tests. This, in turn, means that differences in exposure results can then be interpreted in terms of differences in environmental constituents such as humidity/moisture/dew formation and localized environmental constituents (ozone, smog, acid rain, etc.). Although this approach emphasized ultraviolet effects for outdoor exposure, the analysis technique is applicable to the thermal effects that occur as a result of indoor exposure.

4.5 Material Production Lot Testing

Qualified materials used in production runs of ammunition containers are to be tested as a matter of quality assurance. The use of statistical sampling plans as found in MIL-STD 414 and ASTM D1898 are recommended to select materials and determine the number of individual tests required to characterize a production lot.

A full test matrix, including accelerated and real-time testing as described for the qualification tests, should be conducted on the first several production lots. The data to be obtained from this testing can be used to refine performance prediction models by combining it with the materials screening and qualification test data.

4.6 Full-Scale Container Item Testing

4.6.1 General

Full-scale container item testing is categorized into development, qualification and production-lot testing. The objectives of the development testing are to be characterize the functional characteristics of the container and to demonstrate the accelerated testing procedures that will predict the long-term reliability of the container. The objective of the qualification and production-lot tests to be conducted is to demonstrate the ability of the container to meet performance requirements prior to production and to establish predictive performance models based on production item performance.

Plastic ammunition packaging containers will have moisture absorption and desorption characteristics that will vary from design to design depending on the materials comprising the container body and seals. The absorption and desorption of moisture from the container do not necessarily occur at the same rate either due to the inherent properties of container materials or because of time at temperature related phenomenon which do not allow moisture vapor pressure inside the container to exceed the level outside the container. Two general types of performance in regard to moisture vapor transmission are anticipated for plastic ammunition container packaging at this time. The first case is a well sealed container composed of materials with low moisture vapor permeability constants. After long periods of time this type of container will exhibit either a net gain of moisture or a net loss of moisture depending on the amount of water sealed into the container at the load plant and the vapor pressure differential occurring at the permanent storage area. The second case is a container that is less well sealed either due to the design of the seal or due to high moisture permeability of the container materials. This type of container will allow moisture to be transmitted into or out of the container in a time frame relating to daily or time of year temperature and dew point swings. The characterization of the moisture vapor transmission features of each type of container item will require different test procedures to be conducted in order to predict failure of the ammunition items resulting from longterm storage and to identify the accelerated test conditions that will allow the prediction of longterm results.

The daytime solar radiation outdoor storage environment requires that the moisture vapor transmission of the containers be characterized while under solar load. During daytime periods absorption of the solar radiation causes surface temperatures to rise above ambient temperature which would cause moisture in the container walls to flow in a direction depending on the moisture vapor pressure differential between the outside and the inside of the container.

4.6.2 Test Equipment

4.6.2.1 Environmental Chamber/Solar Simulator

Environmental exposure test chamber equipment as described in Mil-Std 810D may be used for full-scale item tests. The size of the chamber and the size of the solar simulator required will depend on the size of the test item or items to be tested. The environmental chamber is to be capable of producing various conditions over a broad temperature range, specifically the temperature range occurring in the logistics chain. Relative humidity control capability covering a range between 20% and 95% is required in order to produce a variety of moisture vapor pressures. Ideally the test chamber would be instrumented so that temperature, humidity and solar radiation data can be recorded at hourly intervals throughout the test.

The solar simulator array can be comprised of any combination of light sources which, as described in Mil-Std 810D Method 505.2 - Procedure II, can produce a one-sun intensity and provide twice the total energy of the natural sun when operated continuously. The spectral distribution of the simulated solar radiation is to be measured over the target plane that the test items are to be located. The target plane is to be mapped for uniformity. Uniformity of the irradiance shall be $\pm 10\%$ over the entire target area. The radiation intensity in the target area shall be monitored and recorded continuously during the test.

4.6.2.2 Solar Radiation Instrumentation

A spectroradiometer capable of measuring the complete solar spectrum between 290nm and 2600nm wavelength shall be used to determine the spectral energy distribution at the target plane. The location of the light sources and the power input of the light sources are to be adjusted in order to obtain a uniform one-sun intensity over the target area. PSP type radiometers or the equivalent may be used to map the energy distribution of the target plane after the spectral energy distribution is characterized using the spectral radiometer. The irradiance during the testing can be monitored using a Licor LI-200 sensor or the equivalent. The measurements noted shall be conducted behind the window or filter that will be used over the environmental test chamber during the test.

4.6.2.3 Temperature/Humidity Measurement Instrumentation

The requirement of detecting, measuring and recording small changes over a broad range of moisture vapor pressure which can occur inside the container test items makes the use of a chilled mirror type hygrometer almost prerequisite. The instrument selected for use shall also provide for the measurement of temperature inside the container item. ASTM D4230 describes the instrument and its operating procedure. The

humidity transducer may be located inside or outside of the container item depending on the container size, location of the test item in the chamber and the temperature range used during the test. The specific set-up used shall be determined experimentally. A set-up with the humidity sensor located inside the container is shown in Figure 10. A chilled mirror hygrometer, such as the EG&G Model 911 Dew-All, may require modifications to allow specific data recording devices to be used and special calibration to allow long sensor to instrument readout distances to be used. The instrument manufacturer should be consulted prior to specifying and purchasing humidity measurement instrumentation. A general specification for the chilled mirror hygrometer is shown in Table 6.

Table 6. General specifications for chilled mirror hygrometers

Dew Point Range.....	-40°C to + 60°C
RH Range.....	5% to 100%
Dew Point Accuracy (Nominal).....	± 0.3°C
RH Accuracy (Nominal).....	± 0.5%
Dew Point Depression.....	45°C
Air Flow Rate.....	0.25 to 2.5 liter/min.
Operating Temperature for the Transducer.....	-50°C to + 70°C

4.6.2.4 Temperature Measurement and Control

Type T thermocouples are suitable to monitor the environmental chamber temperature and the test article temperature during testing. The thermocouple used to monitor the chamber temperature should be shielded from solar radiation during the test. A thermocouple imbedded in a small sample of the test item material may be used to measure and monitor the test article under a solar load during the test. This sample should be in full view of the solar radiation. Wet and dry bulb temperature sensors integrated in the control circuit of the environmental chamber used are suitable for chamber control and monitoring chamber conditions. Detail requirements for test item and test chamber temperature measurements and recording may be found in Mil-Std 810D. Temperature data should be recorded at the same frequency and time intervals used for humidity and solar radiation measurements.

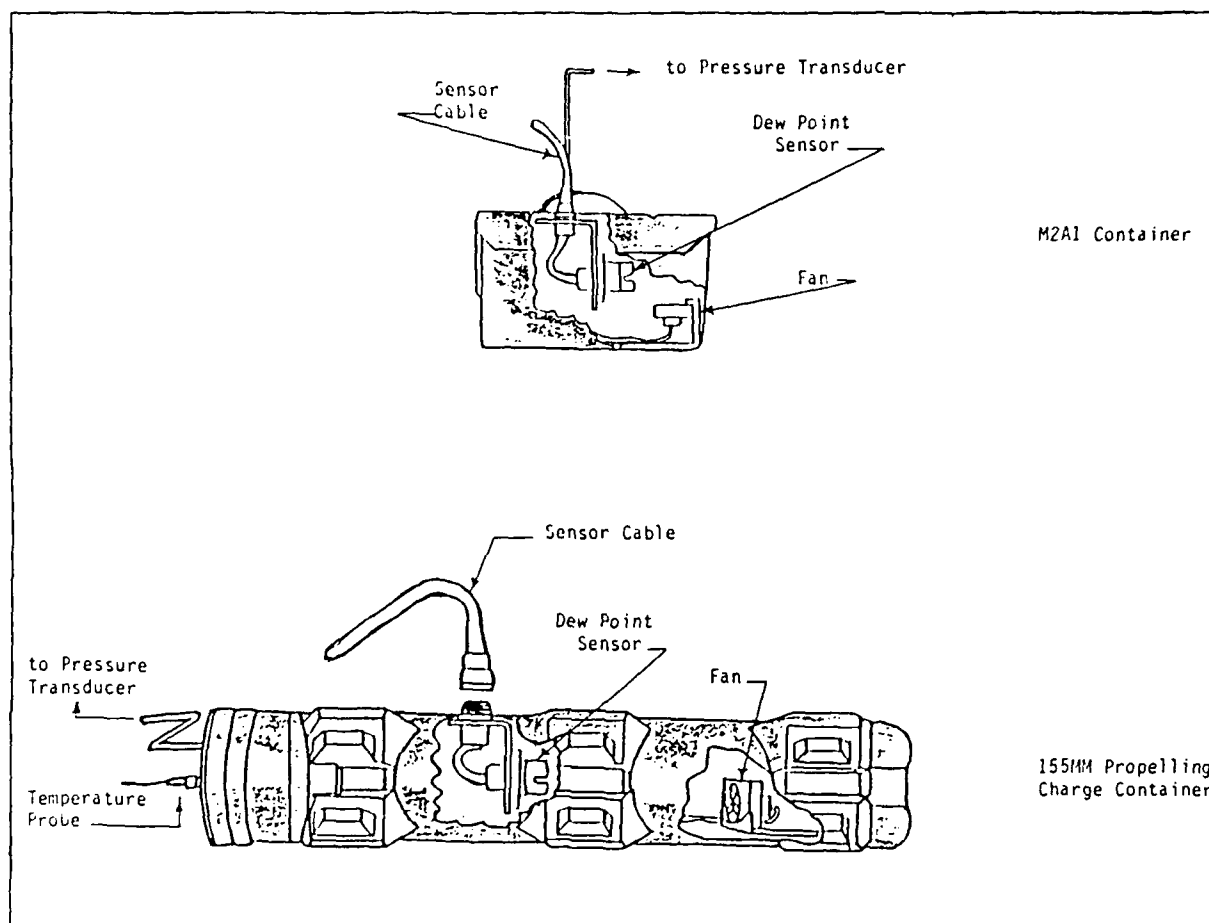


Figure 10. Example of container instrumentation configuration

4.6.2.5 Pressure Measurement Instrumentation

A stainless steel diaphragm pressure transducer with an accuracy of 1% of full scale and an appropriate readout or an equivalent pressure measuring device is to be used to monitor pressure inside the test item during the test. Depending on the test temperature to be used, it may be necessary to locate the transducer outside the environmental chamber. The transducer should be attached to the test item using stainless steel tubing. The test item and transducer shall be checked for major leaks by pressurizing the test item and measuring the pressure drop over a 24 hour period. The magnitude of the pressure drop of a well sealed assembly will depend on the seal design, the moisture content of the container, temperature and ambient temperature changes. Final proof of the integrity of the assembly seal shall be the ability of the container instrumented with the pressure and humidity transducers to remain sealed during several high and low temperature cycles with a high humidity condition. The integrity of the seal will be recognized by a pressure change to commensurate with the temperature change and volume of the container without a major change in dew point inside the container.

An Omega Engineering Model PX 120-15GV transducer and an Omegaraometer DP2000S process monitor or equivalent may be used to measure and monitor pressure during the test. The transducer and the readout used shall be calibrated to a National Bureau of Standards traceable standard in accordance with Mil-Std 45662.

4.6.2.6 Data Recording and Handling Equipment

Multichannel data-logging equipment is necessary to take data during the test. This equipment must be compatible with the temperature, humidity, solar radiation, pressure and test chamber transducers and readouts. An integrated data recording and handling system is recommended since several thousand data points may be taken during a typical test and an integrated system will make data analysis less time consuming. Separate data-logging and data analysis equipment can be used if necessary.

Data analysis can be accomplished using a micro-computer and software suitable to plot graphs, tabulate data, calculate moisture gain, calculate moisture vapor pressure changes and rates of moisture vapor pressure change.

4.6.3 Full-Scale Item Test Set-Up

The environmental chamber controller shall be intercalibrated with the container temperature and humidity measurement instrumentation. This is required since the actual conditions inside the container may be different than indicated by the chamber controller setpoint due to the condition and ageing of the wet bulb thermometer. Also, the wet and dry bulb thermometers may be at the limit of their capacity during some of the test

conditions required. The test chamber moisture vapor pressure will also tend to cycle with the addition of moisture to the chamber during the test. The moisture vapor pressure environment inside the test chamber must be accurately known in order to calculate the container moisture vapor pressure differential and thus the moisture vapor transmission rate occurring during the test.

The container test item or items shall be prepared for testing by first removing all fiberboard material that may be contained inside. Container handles or attachments that do not influence the sealing of the container may be removed as required to allow sensors to be attached to the test item. Pertinent information on the container test item is to be obtained from literature or by test. An example of the information needed for full-scale container test items is shown in Table 7. The container volume is needed to calculate moisture gain. The percent moisture by weight of the container material, even if only a handbook value for the container item material, is useful in checking initial calculations and determining test conditions and times.

The test items are to be prepared by drilling or cutting holes for instrument feed-throughs as required. Existing ports or vents should be used wherever possible. An example of instrumentation with test items was shown in Figure 10. As noted in paragraph 4.5.2.5, the test item and instrumentation assembly shall be leak tested before testing.

The test item or items with any additional sample material shall be loaded into the test chamber. Fixtures are to be used as required to locate the test items in the target plane of the solar simulator. An example of the configuration used for testing containers in a vertical plane is shown in Figure 11. Other configurations can also be used.

Table 7. Example of container test item characteristics and material data needed for full-scale item testing

Container Weight.	4.11 kg.
Container Volume (23°C)	23.6 liter
Container Volume (60°C)	23.7 liter
Coefficient of Thermal Expansion of Container Material	1.85 x 10 ⁻⁴ cm/cm/°C
Temperature Dependent Pressure rise calculated for Container Volume at 60°C	0.12 atm
Maximum Water Absorption by Weight of a Typical High Density Polyethylene.	0.01%
Maximum Possible Weight of Water in 4.111 kg of High Density Polyethylene.	0.4111g

The container test item should be sealed in an environment having a relative humidity that is close to the midpoint of the range of sensitivity of the humidity measuring instrumentation. For chilled mirror hygrometers a relative humidity of approximately 50% is suitable. This humidity level would allow the moisture inside the container to be measured over a wide range of test temperatures without exceeding the maximum measurable dew point depression.

Several trial runs will be necessary to checkout instrumentation and chamber equipment for proper operation. These trials should include constant condition testing for at least 48 hours at several temperature and humidity conditions and several high and low temperature cycles to ensure that the test item and instrument assembly are well sealed. The chilled mirror hygrometer may also require adjustment at each temperature extreme.

4.6.4 Data Requirements and Analysis

An example of the raw test data to be taken during the test is shown in Table 8. The moisture vapor pressure differential dependence of moisture gain requires that long periods of time at constant test chamber conditions will be required to cause measurable changes in dew point temperature inside well sealed containers. Thus, temperature measurements to within only one degree Celsius are required. Tables 9 and 10 exhibit the calculated data needed to determine moisture vapor transmission.

There are several mechanisms by which moisture can gain entry into plastic ammunition container packaging and effect damage to the contents of the container. First, moisture can be brought into the container by the contents of the container, by the inside components of the container and by the container material itself. This is a result of moisture being absorbed during manufacturing and loading. Moist air could also be entrapped when the container is sealed at the load plant. This moisture can be desorbed and reabsorbed under certain conditions of temperature and vapor pressure. The rates of moisture vapor absorption and desorption are not necessarily equal but both are temperature dependent. This is illustrated in equation 13 where the moisture content, E , of a material desorbing moisture is shown as a function of time, t , the moisture content at time zero, E_0 , a temperature dependent coefficient, q , and a material constant K , which depends on temperature and water vapor partial pressure.

$$E = \frac{E_0}{(1+Kt)^q} \% \quad (13)$$

Although the application of this equation to describe the moisture content of the container items is complex, since K and q must be determined for each temperature used, the relationship suggests a fundamental rule of thumb. That is, drying time, or desorption of moisture, occurs in short period of time if the water vapor partial pressure at the surface of

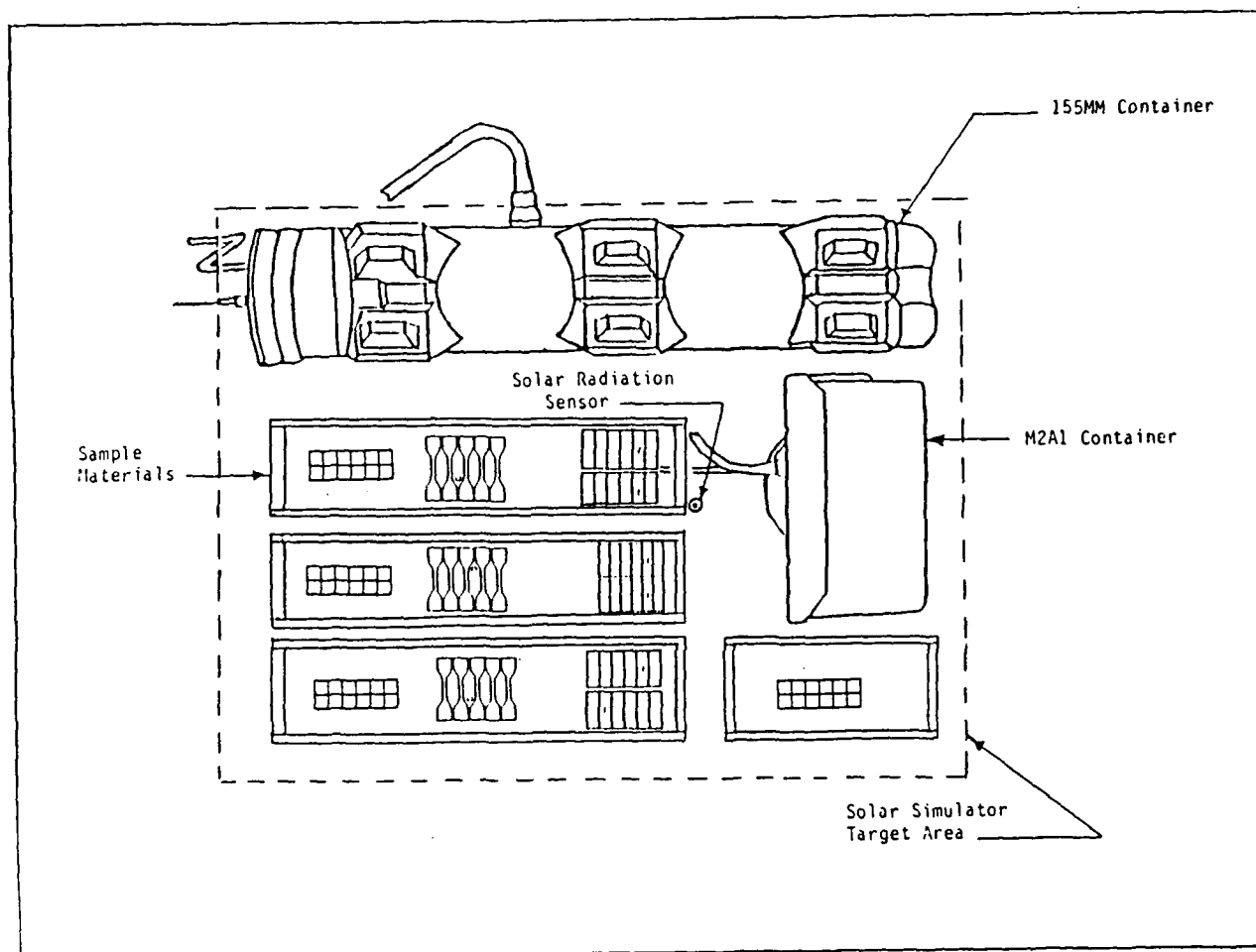


Figure 11. Example of test article set-up and solar simulator target area

Table 8. Example of Container Full-Scale Item Test Data

TEST SEQUENCE	CYCLE TIME (HR.)	DATE-TIME	CHANNEL 2	CHANNEL 3	CHANNEL 4	CHANNEL 5	CHANNEL 6	CHANNEL 10	CHANNEL 12
			DEW POINT (MV)	%RH (MV)	TEMPERATURE (MV)	ALARM	CONTAINER PRESSURE (MV)	SURFACE TEMPERATURE (Deg. C)	CHAMBER TEMPERATURE (Deg. C)
	41.75		2303.30	414.70	2977.90		142.30	59.40	59.20
	42.00		2296.30	410.60	2978.60		143.00	59.20	59.20
	42.25		2304.20	418.10	2976.10		142.90	59.40	59.30
	42.50	14:00	2310.30	418.90	2977.00		143.50	59.40	59.20
	42.75		2315.70	422.50	2977.50		143.20	59.50	59.10
	43.00		2307.40	418.10	2975.70		143.10	59.40	59.20
	43.25		2315.20	422.10	2975.10		143.00	59.50	59.30
	43.50	15:00	2320.00	429.90	2973.90		143.80	59.40	59.10
	43.75		2324.20	429.90	2974.00		143.60	59.50	59.30
	44.00		2315.60	426.00	2974.00		143.90	59.40	59.30
	44.25		2322.50	429.90	2975.00		143.70	59.40	59.30
	44.50	16:00	2327.70	434.70	2974.20		143.80	59.40	59.40
	44.75		2331.40	434.50	2974.90		144.10	59.50	59.10
	45.00		2322.00	430.70	2975.50		143.90	59.40	59.30
	45.25		2329.10	434.90	2976.40		143.50	59.40	59.10
	45.50	17:00	2333.80	438.80	2976.60		142.20	59.50	59.20
	45.75		2337.50	438.40	2974.90		142.30	59.50	59.20
	46.00		2327.50	434.10	2976.00		142.70	59.20	59.10
	46.25		2334.30	438.60	2975.50		142.60	59.30	59.20
	46.50	18:00	2338.90	442.50	2975.40		142.70	59.30	59.00
	46.75		2342.20	443.40	2975.50		143.40	59.30	59.10
	47.00		2331.90	438.90	2975.40		141.80	59.40	59.30
	47.25		2339.10	442.50	2974.30		141.80	59.30	59.20
	47.50	19:00	2343.40	446.20	2974.60		140.90	59.20	59.20
	47.75		2347.10	447.00	2975.70		140.60	59.20	59.10
	48.00		2336.20	438.40	2975.70		140.60	59.40	59.30
	48.25		2343.50	446.40	2975.80		139.60	59.20	59.20
	48.50	20:00	2347.50	446.90	2977.00		139.10	59.30	59.10
	48.75		2350.70	450.80	2978.30		139.00	59.30	59.30
	49.00		2340.30	438.40	2978.60		139.20	59.30	59.20
	49.25		2347.40	446.20	2978.70		138.70	59.30	59.00
	49.50	21:00	2351.30	450.30	2978.30		138.90	59.10	59.10
	49.75		2354.40	450.80	2978.40		138.50	59.30	59.30
	50.00		2343.30	443.20	2978.10		138.60	59.20	59.00
	50.25		2350.50	450.30	2979.10		138.60	59.20	59.00
	50.50	22:00	2354.70	450.30	2979.70		138.60	59.00	59.00
	50.75		2357.80	450.80	2979.40		138.90	59.20	59.10
	51.00		2346.70	447.00	2980.00		138.80	59.10	58.90
	51.25		2354.20	450.20	2980.00		138.30	59.20	58.90
	51.50	23:00	2358.00	450.30	2980.80		138.20	59.20	59.20
	51.75		2361.10	450.30	2980.60		138.30	59.30	59.10
	52.00		4000.20	3982.00	2978.80	3806.50	138.40	59.20	59.00
	52.25		3979.30	3983.10	2978.30	3819.60	137.90	59.20	58.90
	52.50	041638 00:00	3973.60	3982.60	2978.40	3826.90	137.60	59.20	58.90
	52.75		3972.00	3983.60	2978.50	3831.50	138.30	59.20	59.10
	53.00		3975.90	3982.90	2978.80	3834.60	137.60	59.30	59.00
	53.25		3972.00	3983.70	2979.60	3835.60	137.30	59.20	59.00

Table 9. Example of Container Moisture Gain Data

TEST SEQUENCE	TIME	ELAPSED TIME (HRS)	CONTAINER		CHAMBER VAPOR PRESSURE (DYN/CM ²)	CHAMBER	
			MOISTURE (G)	MOISTURE GAIN RATE (G/HR.)		VAPOR PRESSURE (DYN/CM ²)	CONTAINER PRESSURE DIFFERENCE (DYN/CM ²)
TRIAL #6	155.5	0	0.4204		77.9	28.1	49.8
SOLAR RADIATION	164	8.5	0.4864	0.0077647		29.9	48
48C/80%RH	176.25	20.75	0.5174	0.0046746		31.8	46.1
	186.25	30.75	0.5468	0.0041105		33.6	44.3
	199.25	43.75	0.5812	0.0036754		35.7	42.2
	225.25	69.75	0.6215	0.0028831		37.8	40.1
	250.25	94.75	0.6602	0.0025308		40.1	37.8
	280.25	124.75	0.7032	0.0022669		42.5	35.4
	318.5	163	0.7465	0.0020006		45	32.9
	358.5	203	0.7923	0.0018320		47.6	30.3
	407.5	252	0.8407	0.0016678		50.3	27.6
SEPARATE RUN	617.5	0	0.7875		77.9	47.6	30.3
	700.5	83	0.8864	0.0011915		53.2	24.7
	751.5	134	0.94	0.0011380		56.3	21.6
	775.5	158	0.94	0.0009651		56.3	21.6
TRIAL #4	144.25	0	0.3114			19.4	58.5
SOLAR RADIATION	148.75	4.5	0.3542	0.0095111		22	55.9
48C/80%RH	153.25	9	0.3781	0.0074111		23.4	54.5
	156.75	12.5	0.3786	0.005376		24.9	53
	160.75	16.5	0.4294	0.0071515		26.5	51.4
	174	29.75	0.4558	0.0048537		28.1	49.8
	178.75	34.5	0.4849	0.0050289		29.9	48
	183.25	39	0.5158	0.0052410		31.6	46.3
TRIAL #6	39.75	0	0.2866		15	18.2	-3.2
DARK	41	1.25	0.3058	0.01536		19.4	-4.4
60C/20%RH	43.5	3.75	0.3262	0.01056		20.6	-5.6
	51.75	12	0.3477	0.0050916		22	-7
SEPARATE RUN	129.5	0	0.2867			17	-2
	130.5	1	0.3058	0.0191		19.3	-4.3
	133.25	3.75	0.3262	0.0105333		20.6	-5.6
	137.75	8.25	0.3477	0.0073939		22	-7
	141.25	11.75	0.3705	0.0071319		23.4	-8.4
	146	16.5	0.3948	0.0065515		24.9	-9.9
	150.5	21	0.4204	0.0063666		26.4	-11.4
TRIAL #6	853.75	0	0.9286		165	56.3	108.7
DARK	856.75	3	0.9786	0.0166666		59.5	105.5
60C/90%RH	869.75	16	1.0373	0.0067937		62.8	102.2
	884.25	30.5	1.0993	0.0055967		66.3	98.7
	903.25	49.5	1.1648	0.0047717		69.9	95.1
	922.25	68.5	1.2339	0.0044569		73.8	91.2
	946.25	92.5	1.307	0.0040908		77.9	87.1
	966.25	112.5	1.3841	0.0040488		82	83

Table 10. Example of Container Graph Regression Information

SOLAR CYCLE 1ST GROUP REGRESSION

Regression Output:

Constant -8.40338
Std Err of Y Est 0.148746
R Squared 0.915799
No. of Observations 10
Degrees of Freedom 8

X Coefficient(s) 0.067272

Std Err of Coef. 0.007211

SOLAR CYCLE LAST GROUP REGRESSION

Regression Output:

Constant -8.064651
Std Err of Y Est 0.1691406
R Squared 0.6309609
No. of Observations 7
Degrees of Freedom 5

X Coefficient(s) 0.0581066

Std Err of Coef. 0.0198735

60C/20XRH CYCLE 1ST GROUP REGRESSION

Regression Output:

Constant -2.24738
Std Err of Y Est 0.110052
R Squared 0.980792
No. of Observations 3
Degrees of Freedom 1

X Coefficient(s) 0.427333

Std Err of Coef. 0.059801

60C/20XRH CYCLE 2ND GROUP REGRESSION

Regression Output:

Constant -3.671755
Std Err of Y Est 0.2380916
R Squared 0.7477093
No. of Observations 6
Degrees of Freedom 4

X Coefficient(s) 0.1376770

Std Err of Coef. 0.0399867

COMBINED SOLAR CYCLE DATA REGRESSION

Regression Output:

Constant -8.19398
Std Err of Y Est 0.142323
R Squared 0.959663
No. of Observations 20
Degrees of Freedom 18

X Coefficient(s) 0.061211

Std Err of Coef. 0.002957

COMBINED 60C/20XRH CYCLE DATA REGRESSION

Regression Output:

Constant -3.733787
Std Err of Y Est 0.2969115
R Squared 0.5992185
No. of Observations 9
Degrees of Freedom 7

X Coefficient(s) 0.1391354

Std Err of Coef. 0.0430080

60C/90XRH CYCLE REGRESSION

Regression Output:

Constant -9.96592
Std Err of Y Est 0.307885
R Squared 0.683806
No. of Observations 7
Degrees of Freedom 5

X Coefficient(s) 0.050879

Std Err of Coef. 0.015472

the material is relatively high. This is related to the fact that the heat conduction between the source of heat and the material is greater at higher pressures and that the resistance to diffusion in a moist surface layer is smaller than in a dry one. The relationship of time, temperature and vapor pressure on the desorption of moisture from the contents of ammunition containers and the container material is important to both real life performance and the selection of accelerated test conditions since the movement of moisture from these sources can cause degradation of the contents and the container material by both physical and chemical mechanisms.

Moisture can also enter the container through pinholes and channels through the container material. These pinholes and channels could occur around pigment particles, reinforcing fillers or at a seal. The entry of moisture through pinholes is dependent on whether the air outside the container is mostly air or mostly vapor and on the size of the pinhole in relation to the mean free path of the water molecule, which is approximately 7×10^{-5} cm. Therefore, the transmission of moisture into the container by these mechanisms depends greatly on the vapor pressure differential occurring between the outside and inside of the container. These entry mechanisms then, allow categorization of moisture gain into low pressure and high pressure situations. In simplified terms, moisture gain by these mechanisms can be expressed by equation 14,

$$Q = k * m * P \quad (14)$$

where Q is the rate of flow, k is a permeability constant, m is a constant dependent on the area and thickness of the material and P is the vapor pressure differential. m varies for pinhole and channel size and length and is also dependent on the vapor pressure regime.

The natural humidity environment for temperate and tropical climates is generalized for typical daily moisture vapor pressure in a temperate climate cycle from approximately 15 dynes/cm² to approximately 20 dynes/cm² over a temperature range between 18°C and 25°C. The peak moisture vapor pressure occurs in the summertime and can be as high as 27 dynes/cm². Typical daily moisture vapor pressure in a tropical climate cycles between approximately 29 dynes/cm² to approximately 37 dynes/cm² over a temperature range between 26°C and 32°C. The peak moisture vapor pressure can be as high as 43 dynes/cm². The vapor pressures, shown in Table 11, used for several elevated temperature/humidity tests cover a range between 15 dynes/cm² and 165 dynes/cm² over a temperature range between 20°C and 60°C.

Desert environments therefore do not present vapor pressure environments greater than the worst case tropical environments although analysis of typical dew point and air temperature data for some desert regions would show overlap during certain times of the year. However, important consideration must be given to the effects of the high temperatures resulting from the absorption of solar radiation during outdoor storage on the diffusion of the moisture that might be contained in the surface of the container material. This heating could drive moisture into the container

over long periods which would never be predicted by environmental chamber tests with short cycle times. On the same basis, arctic environments could also cause high vapor pressure environments during daytime, outdoor exposure.

Table 11. Environmental chamber intercalibration data

Chamber Set-Point Conditions	Average Instrument Readings	Vapor Pressure (dynes/cm ²)
60°C/90%RH	87.6%RH 56.1°C Dew Point	165.0
60°C/20%RH	8.5%RH 13.1°C Dew Point	15.0
48°C/80%RH	76.9%RH 41.2°C Dew Point	77.9

The categorization of moisture gain into high and low vapor pressure regimes can be used to determine the severity of test conditions relative to real environmental conditions. The extent to which test conditions can be used to increase moisture vapor transmission rate and thus accelerate effects of moisture damage however, may be entirely dependent on a given material's inherent moisture permeation coefficients. Therefore, the use of an acceleration or a test severity factor based entirely on the ratio of test conditions to real environmental conditions to predict results must be judicious. As an example, if normal vapor pressure is about 43 dynes/cm² for the worst tropical conditions, while the test conditions had vapor pressures in the range of 340 dynes/cm². The ratio of the vapor pressures would suggest an acceleration factor of 7.9. However, more appropriate is the determination of test severity on the basis of a ratio of transmitted moisture at different vapor pressure conditions. In this example if 0.6mg of transmitted moisture was measured at constant conditions of 40°C with a vapor pressure of 65 dynes/cm², while 0.012mg were measured at constant conditions of 25°C with a vapor pressure of approximately 30 dynes/cm², the acceleration factor based on transmitted moisture is 0.6/0.012 or 50, while an acceleration factor based on the test condition is 65/30 or 2.2.

The application of this model to the case of an ammunition container must account for the changes in vapor pressure differential which occur as the container gains moisture. Thus, as the container gains moisture the moisture vapor transmission rate would tend to decrease. That is as the vapor pressure differential between the environment outside the container and the inside of the container approaches zero the moisture vapor transmission rate also approaches zero. Since the containers could be deployed to hot and dry and cold and dry environments, the containers could also lose moisture over long periods of time.

Moisture vapor transmission rate constants and permeation constants are temperature dependent material properties. These constants generally increase logarithmically with increasing temperature. This is to be expected since water vapor pressure also increases with temperature in a logarithmic manner. As suggested by equation 14, when material thickness is incorporated into the constant m , moisture vapor transmission rate is found to be inversely dependent on thickness. Full-scale item tests and test data analysis should be normalized for the effects of thickness and temperature by using chamber/container vapor pressure differential and the use of container volume to first determine moisture gain and their moisture gain rate.

Moisture gain is to be expressed in terms of grams per hour as a function of vapor pressure differential for the test conditions used. This is based on the concept of the rate of change in vapor pressure differential as function of time. This latter relationship is shown by equation 15.

$$\ln(P/P_0) = -rt \quad (15)$$

In equation 15,

P = water vapor pressure differential
 P_0 = P at time zero
 t = time
 r = a rate constant derived from a material water vapor transmission rate constant.

Equation 15 is analogous to the equation for exponential decay shown in equation 16 and can be applied to both the vapor pressure differential between the test chamber and the container, moisture gain and to the relationship between the vapor pressure differential and the rate of moisture gain.

$$X = X_0 e^{-rt} \quad (16)$$

If equation 15 is to be applied to the rate of moisture permeation into the container at any time, t , then X as the moisture vapor transmission rate at time t in equation 17, is proportional to the product of the moisture vapor transmission rate in the container, X_0 , at time t_0 and the vapor pressure differential, P , between the chamber and the container. In order to express the rate of moisture permeation as a function of vapor pressure differential, both the quantity of water and vapor pressure differential must be considered as a function of time.

$$X = X_0 e^{-mP} \quad (17)$$

The differential equation describing the function of moisture gain and vapor pressure differential and its solution are given in equations 18 and 19, where C is a nonzero constant.

$$D_t(X) = mX \cdot D_t(P) \quad (18)$$

$$X = Ce^{-mP} \quad (19)$$

In equation 17 it is assumed that P is zero when the chamber vapor pressure equals the vapor pressure in the container and when $X = X_0 = C$, equation 20 results.

$$X_0 = C e^{-m \cdot 0} \quad (20)$$

In practice the weight of moisture in the container as a function of time is calculated using the humidity ratio and dry air volume from psychrometric tables as determined from the container dew point and temperature. The vapor pressure of the container as a function of time is determined likewise, while the vapor pressure of the test chamber is determined from chamber calibration experiments. Finally, the vapor pressure differential between the test chamber and the container is to be calculated as a function of time from the vapor pressure data.

The amount of water occurring in the container during the course of environmental testing can be calculated using equation 21,

$$\frac{W_s}{V_a} V = W_{H_0} \quad (21)$$

where W_s and V_a are the humidity ratio and dry air volume and W_{H_0} is the weight of water contained in the container air volume, V . W_s is obtained at the measured dew point. V_a is obtained for the temperature measured inside the container during the test. Both values are for temperatures to the nearest degree.

The total water content inside the container at a given time during the test is to be associated with an elapsed time and test chamber vapor pressure. Elapsed time is determined by subtracting the given time in hours from time zero. Time zero can be determined from data, where the chamber and container conditions are stabilized. Water gain rate, in terms of grams per hour is then calculated by subtracting the moisture content of the container at a given time from the water content at time zero and dividing by the elapsed time. Container moisture vapor pressure are determined with the dew point temperature and the inside container temperature data and psychrometric tables. Vapor pressure differential as a function of time is calculated by subtracting the test chamber vapor pressure from the container vapor pressure at a given time during the test.

Moisture gain rate data can then plotted with their linear regressions as a function of vapor pressure differential. The regression line values are obtained using the natural logarithm of the moisture gain rates. The slope of the regression is taken as the moisture vapor transmission rate constant over the vapor pressure range during the test. The rate constant is expressed in terms of grams of moisture per hour per dyne/cm². Examples of calculated data are shown for a container item shown in Tables 9 and 10 and Figure 12.

155MM CONTAINER FULL-SCALE TEST

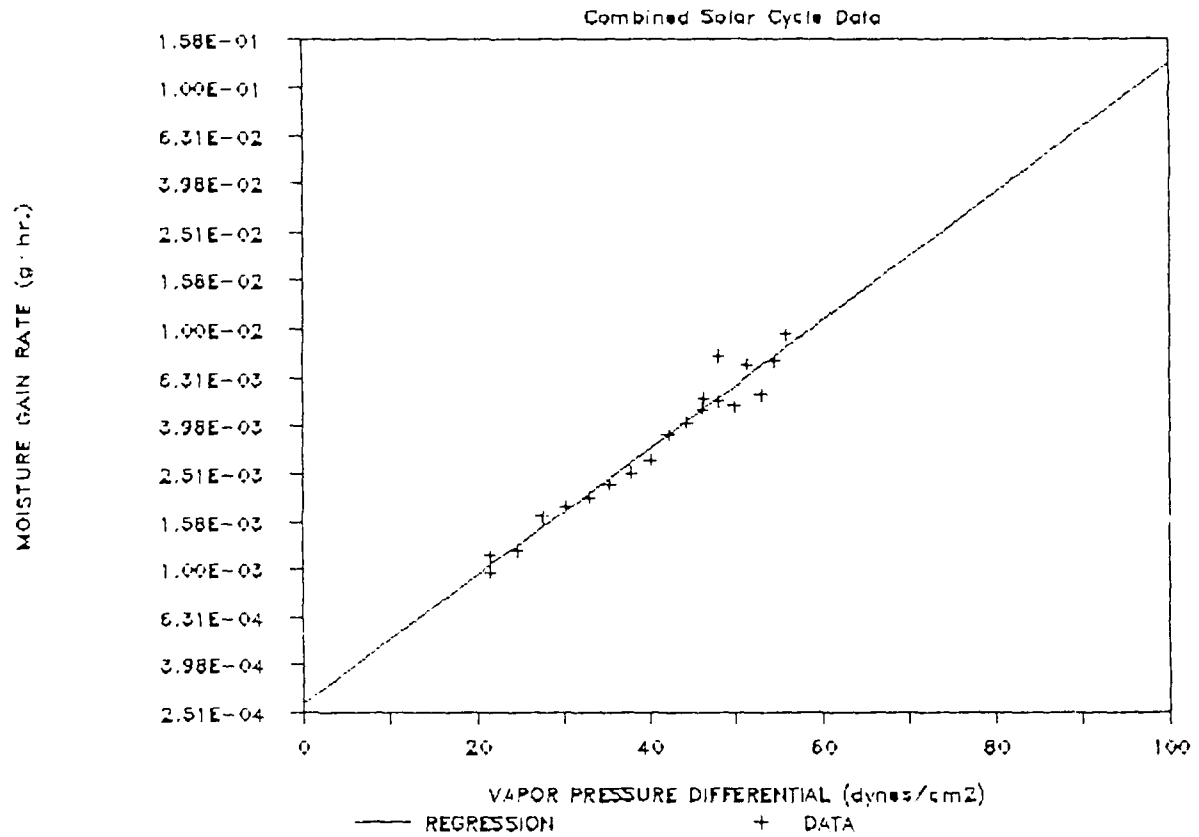


Figure 12. Regression data

4.6.5 Design/Development Tests

The objective of design and development testing of full-scale items is to first characterize the time frame in which the container item gains and loses moisture. This information is to be used to establish test conditions and test cycles which will allow the performance of the container design to be predicted. Well sealed containers made from materials with low moisture permeability will generally have either a net gain or loss of moisture depending on the moisture vapor pressure differential occurring between load plant and the permanent storage area. Containers of this type are best tested using constant conditions in order to establish typical moisture vapor transmission rate coefficients that can be used to predict the time required to reach a critical moisture level inside the container at the permanent storage site. Less well sealed containers shown to absorb and desorb moisture during typical daily or time of year temperature cycles, are to be tested using various temperature and humidity cycles in order to predict the amount of moisture absorbed or desorbed by the container in a typical temperature cycle. The net gain or loss of moisture by the container resulting from these cycles would then be used to predict the time required to reach a critical moisture level at the permanent storage area. The appropriateness of the container design as packaging for a particular ammunition item can be determined from this characterization.

A second objective of design and development testing is to provide quantifiable data for comparing different designs and containers fabricated using different materials since prototype and pre-production container items are most likely to be available for design and development testing. Lastly design and development testing is to be used to establish the performance requirements and qualification test conditions for production items.

As noted in paragraph 4.6.3, the test items are to be sealed in an environment that will allow dew point to be measured over the widest possible temperature range. As an example, if the container is sealed at 20°C/50% RH, the test chamber should be operated at those same conditions in order to determine the amount of moisture in the container and to equilibrate the container at these conditions. After the container and chamber have been stabilized as shown by a constant dew point temperature inside the container, the container should be subjected to a high moisture vapor pressure by increasing the chamber temperature and humidity conditions. A temperature of 60°C at approximately 80%RH provides the worst case moisture vapor pressure conditions that can be reproducibly controlled by typical test chambers. This test condition causes a moisture vapor pressure greater than the typical extreme of the logistics chain environment but allows the container item to be characterized in a relatively short period of time.

The container item should be subjected to the elevated temperature and humidity conditions until a sufficient number of data points are taken to allow a moisture gain rate to be established. The time required to obtain a sufficient number of data points is dependent on the moisture vapor transmission rate of the container and can be recognized by the dew

point temperature inside the container approaching the dew point of the test chamber. This could take as long as several thousand hours for well sealed containers. The test chamber should then be set to the starting conditions or to an even lower moisture vapor pressure represented in the logistics chain environment. These conditions should be held constant until a sufficient number of data points are obtained to allow a moisture loss rate to be established.

The time period required for the moisture vapor pressure differential between the container and the test chamber to reach zero, either by absorption or desorption, should be compared to daily and time of year temperature and relative humidity changes in order to determine whether a cyclic test or constant condition test should be used to further characterize the container. Containers that show a change in moisture vapor pressure differential over a short period of time within the range represented between the load plant environment and the transportation and permanent storage environments of the logistics chains should be tested using a cyclic test. Containers that show little change in moisture vapor pressure differential with respect to time are better testing using constant conditions.

Paragraph 4.6.4 describes the concept of using moisture gain to determine an acceleration factor for testing and the use of moisture vapor pressure differential to predict moisture gain or loss with respect to time. The consideration of how a container item gains or loses moisture can be used to establish specific test conditions for characterizing performance. The maximum test temperature that can be used will be limited by the maximum service temperature recommended for the container materials. This temperature will also place limitations on the maximum moisture vapor pressure that can be used. A thorough characterization study must also include tests using temperatures and moisture vapor pressures typical of the logistics chain.

The effects of sunlight during outdoor storage can be determined using simulated solar radiation cycles. The test chamber conditions should be set to maintain the test item temperature while under a solar load at the temperatures used for testing without solar radiation. The test chamber moisture vapor pressure and the resulting moisture vapor differential occurring between the chamber and the container can then be used to determine the effects of daytime exposure during outdoor storage. The container could gain moisture for some period of time even though the vapor pressure differential between the chamber and the container might suggest otherwise, due to the moisture contained in the container material and the container temperature. The daytime and nighttime effects on moisture gain by the container can thus be determined.

Each container design change and material change that may occur during the container item development should be evaluated. In order to develop confidence in the performance data obtained from the testing, enough testing to show reproducibility must be accomplished.

4.6.6 Production-Lot Tests

Production container items are to be subjected to qualification test procedures on a lot-to-lot basis using a suitable sampling plan. As test data is accumulated, especially moisture vapor transmission data, it is to be compared to field performance data. Acceptability limits and performance prediction models can then be adjusted.

Depending on the measurement techniques used to evaluate the materials comprising the container item during the material qualification process, actual full-scale items may be used to prepare test specimens for the material property tests called for in paragraph 4.5. Where possible, container items which have been used in the field should be used as test specimens as well.

5.0 TEST PLAN STATUS AND BACKGROUND

5.1 Background

The approach taken to develop accelerated test procedures for the 155mm Propelling Charge container and the M2A1 container pursued the application of Arrhenius relationships to materials degradation and correlating the moisture vapor pressure differential with full-scale container moisture gain rate. Materials property and moisture gain data were analyzed with regards to the ammunition item logistics chain environment in order to prepare lifetime prediction models. Accelerated test conditions were chosen at the high end of the logistics chain temperature and humidity extremes. Tests were conducted primarily with constant condition tests in order to maximize the time at condition during the course of the testing. Testing with simulated solar radiation was conducted using several flux rates including natural sunlight. Materials degradation test data were then extrapolated to lower temperatures using a range of activation energies to prepare the models.

Accelerated testing of plastic materials is usually conducted at temperatures well above actual service temperatures and with high intensity ultraviolet radiation. However, differences between the service temperature and the accelerated test temperatures, require that the activation energy value used for performance prediction be accurate. As an example, a small difference in temperatures between real and accelerated conditions would require that the activation energy be determined to within 300 calories per mole in order to determine a rate of degradation with a 10% error. On this basis, a general rule that should be applied to the design of accelerated tests is to conduct the tests at close to real temperature conditions. The tradeoff of speed or degree of acceleration for total test time should be decided on first by determining the accuracy of results required and second, by the inherent properties of the materials to be tested. The comparison of

the performance of the high density polyethylene (HDPE) used for the 155mm Propelling Charge container to the performance of the glass reinforced polyester used for the M2A1 container serves as a case in point for the latter consideration.

The HDPE was found to exhibit a measurable degree of degradation at the surface and in the bulk as a result of exposure to the same conditions that the degradation of the glass reinforced polyester could only be determined visually or by colorimetric methods. The M2A1 container could therefore be tested using more extreme conditions or for longer periods of time to produce a given degree of degradation. Conversely, the moisture gain characteristics of the M2A1 container suggest that this feature of the item be tested using vapor pressure differentials representative of the logistics chain over a long period of time. This approach would tend to accelerate the effects of absorption and desorption on the container contents. The 155mm Propelling Charge container on the other hand was found to have a net increase in moisture over the range of vapor pressure differential representative of the logistics chain and thus would be best tested using high vapor pressure differentials to accelerate results.

As noted in Volume I, activation energy values for the HDPE were taken from literature and also chosen arbitrarily to test the performance prediction models developed. In order to meet the project objective for a 30-year lifetime prediction model with a 10% certainty, the activation energies for photodegradation and thermal degradation of the HDPE must be determined and applied to the model. Thermal history resulting from manufacturing processes or from storage of the container item could also affect the longterm performance of the container material. This aspect of determining the ability of the container material to meet longterm requirements also needs further investigation.

The test plan for the container items tested is summarized in Figure 13. The plan is divided into materials and container item testing which at its end should produce lifetime prediction models with the desired degree of confidence. As noted in the diagram real-time testing is recommended for both materials testing and for container item testing. This feature of the plan is necessary to test the prediction models. The details of the plan are as follows:

5.2. Materials Tests

5.2.1. Material Acceptability Limits

A definition of a material failure is required. Ideally the definition should be quantitative in terms of a critical property. Appropriate properties include impact resistance, tensile strength, elongation, and creep resistance. In the case of the 155mm Propelling Charge container, the thermal properties of the HDPE were used to evaluate the change in material properties. However, there was no reference point between

the thermal properties measured and the engineering properties which are typically used to define a failure point. Therefore, full-scale items which have been subjected to exposure tests must be tested to determine the acceptability of the material in providing adequate protection to the contents. These full-scale tests could be drop tests, palletization load tests, moisture vapor transmission tests or any other tests which would allow the thermal properties measurements to be related to a physical limit of acceptability. Future accelerated and real-time testing could then be conducted to the failure point.

5.2.1.1 Activation Energies

In order to improve the confidence of the lifetime prediction models described in Volume I, the activation energy of the HDPE used for the container must be determined for photodegradation processes, to represent outdoor storage effects, and for thermal degradation process, which represent indoor storage effects. The activation energies should be determined using at least four different ambient temperatures between 30°C and 70°C. The exposure test equipment described in paragraph 4.4.3 and the measurement techniques used during the Phase II effort can be used to determine both activation energies.

An investigation of the effects of thermal history and humidity could be conducted concurrent with this effort. This information will identify the effects of manufacturing and fabrication processes on the durability of the material as well as indicating the variance likely to be typical for the material.

HDPE 155MM PROPELLING CHARGE

AND

POLYESTER M2A1 CONTAINERS

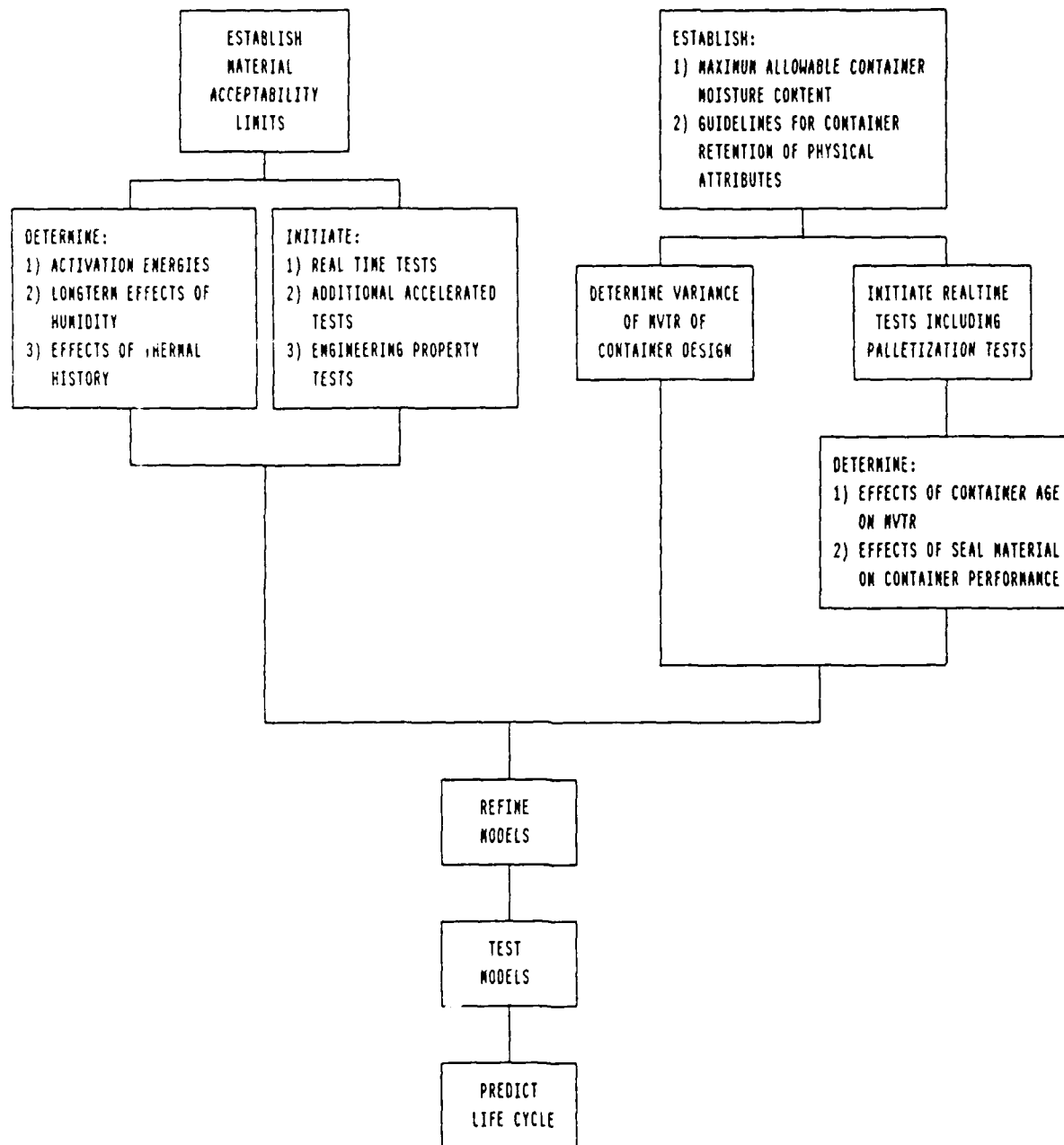


Figure 13. Test plan for 155mm Propelling Charge Container

(THIS PAGE IS LEFT BLANK)

DISTRIBUTION

Commander
Armament Research, Development and Engineering Center
U.S. Army Armament, Munitions and Chemical Command
ATTN: SMCAR-AEP(D)
Carl Morrison
Packaging Division
Building 455
Picatinny Arsenal, NJ 07806-5000

Commander
Armament Research, Development and Engineering Center
U.S. Army Armament, Munitions, and Chemical Command
ATTN: SMCAR-IMI-I (Bldg 59)
SMCAR-AMSMC-QAR-R(D)
SMCAR-AMSMC-GCL(D)
Picatinny Arsenal, NJ 07806-5000

Administrator
Defense Technical Information Center
ATTN: Accessions Division (12)
Cameron Station
Alexandria, VA 22304-6145

Director
U.S. Army Materiel Systems Analysis Activity
ATTN: AMXSY-MP
Aberdeen Proving Ground, MD 21005-5066

Commander
Chemical Research, Development and Engineering Center
U.S. Army Armament, Munitions and Chemical Command
ATTN: SMCCR-MSI
Aberdeen Proving Ground, MD 21010-5423

Commander
Chemical Research, Development and Engineering Center
U.S. Army Armament, Munitions and Chemical Command
ATTN: SMCCR-RSP-A
Aberdeen Proving Ground, MD 21010-5423

Director
Ballistic Research Laboratory
ATTN: AMXBR-OD-ST
Aberdeen Proving Ground, MD 21005-5066

DISTRIBUTION (cont)

Chief

Benet Weapons Laboratory, CCAC
Armament Research, Development and Engineering Center
U.S. Army Armament, Munitions and Chemical Command
ATTN: SMCAR-CCB-TL
Watervliet, NY 12189-5000

Commander

U.S. Army Armament, Munitions and Chemical Command
ATTN: SMCAR-ESP-L
Rock Island, IL 61299-6000

Director

U.S. Army TRADOC Systems Analysis Activity
ATTN: ATAA-SL
White Sands Missile Range, NM 88002

Mr. J.E. Brzuskiwicz

R/NBD Division (2)
DSET LABORATORIES, INC.
Box 1850 Black Canyon Stage I
Phoenix, AZ 85027