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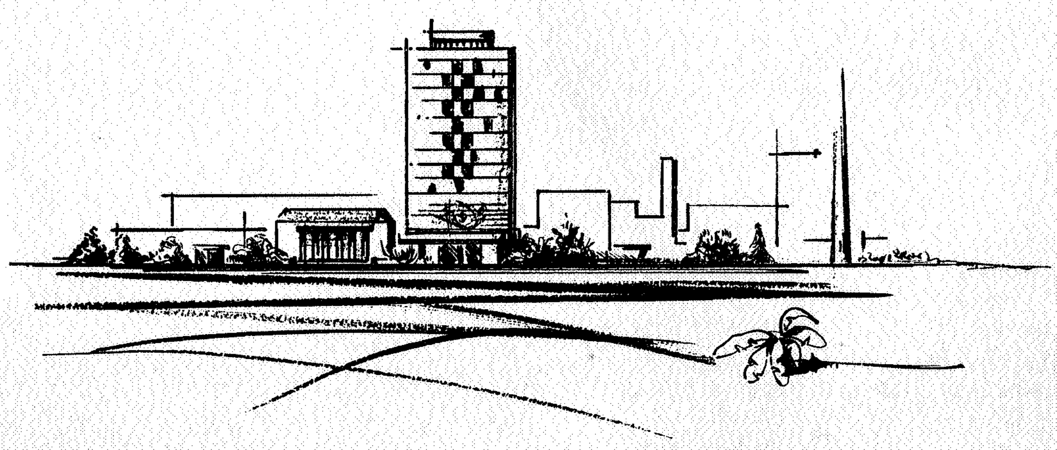

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July 26, 1967

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama 35812

Attention R-ME-MMC

Gentlemen:

Final Report on Contract NAS8-20317
Packaging and Preservation of Space-Vehicle Hardware

This report summarizes work carried out on the subject contract during the period June 9, 1966, through June 8, 1967. Twenty copies including one reproducible copy are enclosed.

Very truly yours,

C. W. Cooper
Principal Investigator

CWC:jb

FINAL REPORT
JUN 9-1966 - JUN 8-1967

on

PACKAGING AND PRESERVATION OF SPACE-VEHICLE HARDWARE

to

GEORGE C. MARSHALL SPACE FLIGHT CENTER

July 9, 1967

by

C. W. Cooper, J. Mason Pilcher,
J. A. Gieseke, A. R. Bunk, and W. E. Clark

NASA Contract No. NAS8-20317

Period Covered: June 9, 1966, through June 8, 1967

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ABSTRACT

This study of the packaging and preservation of space-vehicle hardware is divided into three phases--criteria control and standards, material development and application, and development of new procedures using the new materials cleaned under the new standards.

The first segment of the effort which included contacts with major suppliers and users of clean packaging materials as well as an analysis of relevant specifications and standards resulted in recommendations for the preparation of any future standards. One critical shortcoming of all currently existing standards is the omission of any means for qualifying a material on the basis of its more important single property--slough resistance.

Other portions of the study are concerned with evaluation of new materials and recommendations for preferred packaging systems. Materials were found which are superior in abrasion resistance to the presently preferred Nylon 6. A polycarbonate film was the best of these. Composites employing this material as well as various combinations of polyester film, polyvinyl fluoride, a polyvinylidene chloride copolymer, and fluorohalo polymers are suggested.

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INTRODUCTION

The objectives of this program have been to (1) develop standards and testing methods for evaluating the cleanliness level of hardware and packaging materials, (2) select more suitable materials for packaging applications, and (3) develop new procedures using the new materials cleaned under the new standards. The program has been divided into these three specific phases which are spelled out in the Work Scope and the report follows this outline. Phase I deals with Criteria Control and Standards, Phase II New Materials (an experimental phase), and Phase III Test and Evaluation. The first two of these have been detailed in Monthly Reports Nos. 7 and 11, respectively. This report reviews the findings of the initial two phases and includes also a summary of Phase III activity.

It is important to note that the slough tendency of packaging materials in contact with aerospace hardware is the principal property of concern in clean packaging materials. Commercial films--or composites--now exist which can contribute the desired degree of LOX compatibility, gas and moisture vapor barrier properties, and sealability.

SUMMARY AND CONCLUSIONS

The program was divided into three phases. Phase I has been concerned with criteria control and standards; Phase II, material development and application; and Phase III, test and evaluation.

In the first phase, technical representatives of the major suppliers of clean room products and of several users of these materials were contacted for comments and opinions relative to clean packaging. These comments are listed under categories of materials, performance criteria, specifications, and general. In addition, relevant specifications and standards in the broad area of cleaning, cleanliness measurement, handling, and packaging space hardware were obtained, cataloged, and evaluated. This, together with discussions with users and a review of pertinent literature, confirmed the need for a standardized and effective means for the measurement of the most important single characteristic of a clean packaging material--its resistance to sloughing.

The specifications and standards considered sufficiently important for review and analysis were divided into two classes--those dealing with film and those concerned primarily with hardware cleanliness or cleanliness measurement. Based on this analysis plus field comments and opinions of Battelle staff members, a compilation of a set of eight points to be considered in any future preparation of specifications was made.

Phase I activity also included a study of current methods of evaluating cleanliness levels. This was helpful in subsequent effort in determining performance characteristics of new materials.

The Phase II effort was concerned with four basic properties of packaging materials--resistance to sloughing, LOX compatibility, good moisture and gas barrier characteristics, and adequate sealability. Currently existing films or other packaging materials or composites are capable of contributing all but the first of these characteristics. Since the development of wholly new materials was beyond the scope of this project, activity centered around the selection of potentially useful materials or systems not now being employed for this application.

Nylon 6 is the material presently preferred for slough resistance. In the examination of other candidate materials, it became necessary to develop and evaluate techniques for measuring this characteristic since no prescribed method now exists for this. Three such methods were evaluated--mechanical abrasion (an adaptation of the Taber Abraser), packaged part tumbling, and a reciprocating stylus technique. The first of these appears to be the most reproducible.

In the course of measuring slough characteristics of various polymeric films, it became apparent that two or three of these are more abrasion-resistant than nylon. The best of these was a polycarbonate film.

In the third phase of the program, recommendations are made relative to means for prescribing standards for slough resistance. In addition, suggestions are made regarding preferred materials and composites, and techniques for reducing the tendency of a packaging material to abrade during use. Methods for measuring cleanliness level of packaging materials and packaged parts are outlined also.

PHASE I. CRITERIA CONTROL AND STANDARDS

Field Contacts

To provide input of practical value to the program, several contacts were made in the field with producers and users of clean room products and one meeting was arranged with a major technical society for establishing standards--ASTM. The producers of clean room products visited and key personnel with whom discussions were held are listed below. These currently are the four major suppliers to the clean packaging industry.

Clean Room Products, Inc.
Farmingdale, New York 11735

Mr. Leon Hertzson, President

Specialty Converting, Inc.
1930 Hoyt Avenue
El Monte, California 91733

Mr. Charles Forbes, President

Scientific Enterprises, Inc.
468 Polygon Market
Broomfield, Colorado 80020

Mr. R. E. Bolasny, President

Richmond Corporation
27427 Pacific Avenue
Highland, California 92346

Mr. Ralph Richmond, President
Mr. Dan C. Anderson, Vice President

The users visited together with personnel from whom opinions were obtained were:

Grumman Aircraft Engineering Corporation
Bethpage, New York 11714

Mr. C. T. Williamsen
Staff Engineer, Quality Control

General Electric Company
King of Prussia
Philadelphia, Pennsylvania 19101

Mr. Robert Waite

Pertinent Observations from the Field

The important comments and observations obtained in discussing problems of clean packaging with suppliers and users of these materials are summarized below. For convenience, these items have been roughly categorized as relating to materials, performance criteria, specifications, and general.

Materials. Polychlorotrifluoroethylene films are not appropriate for clean packaging because of slough tendencies and susceptibility to hydrocarbon contamination in solvent cleaning.

Abrasion resistance of Nylon 6 is sufficiently good to reduce the importance of LOX compatibility in certain situations. Humidity should be controlled in producing packaging materials to minimize development of a static charge.

It is apparent that both producers and users of clean room materials rely on available products without sufficient consideration of new possibilities. This is perhaps justified since large producers of film generally do not conduct research on nonsloughing materials or on cleaning techniques largely because (for them) the market is small.

One supplier believes packagers should use a definite (and specified) material for certain types of parts. Most major users depend upon standard films available from clean packaging suppliers. Users are aware of the hazards of aluminum foil as a packaging material. Since the organization contacted is involved in various space projects, its packaging needs vary and the requirements are usually specified by the customer.

Tapes for sealing are accepted by many but at least one supplier does not believe in them because of potential contamination with oxidizable materials.

Performance Criteria. There is need for improved means for checking contamination levels, especially surface contamination. Need exists for a means to determine the best films to use in various applications.

Methods of static control include operation at higher humidities, α-ionization, static bars (air ionizer), and surface treatment such as coatings or conductive additives.

Hot wire cut-off for films is not recommended--it generates particles along the edge.

Heat-sealing should be controlled by time, not temperature, because of differing rate of conductivity of sealed sections and variations in web thickness.

At least one supplier is interested in static-free films, particularly a polyethylene, which is reported to bleed off a charge if grounded.

No supplier or user presently conducts any routine measurement of slough characteristics of packaging materials. There is need for a more practical and definite method for counting particles per unit area.

Test methods should be kept as simple as possible with respect to both equipment and procedures.

Cleanliness levels should be the same for all parts going into the same system.

One supplier is very much interested in control of static charge during film handling as a means for reducing the quantity of particulate contamination. The same supplier believes many standards for cleanliness (and other characteristics) have been set up arbitrarily and would like greater consideration to be given to "what materials to use, levels to specify, and the total system".

Specifications. A primary goal in any overhaul of specifications, according to one supplier, would be a precise definition of permissible particle count and micron levels for packaging materials.

The same supplier believes MSFC-SPEC-164 to be the one specification used by most contractors although MBO-295005 is in conflict with it.

The KSC-C-123D specification was indicated as essential to Huntsville. This specification is the same as MSFC-10M01671.

There is need for a standardized material specification.

The state of the art does not provide for compliance with the specification for gas bearing systems (no particles larger than 20 microns).

MSFC-10419906--"Cleanliness of Components for Use in Gyro Air Supply Systems"--is not applicable to film. This specification requires that there be no particle larger than 20 microns which is not achievable.

One supplier suggested a specification containing (1) material controls--specified film, and (b) cleanliness levels for certain uses--five levels (one solvent to test all levels). Particle count and size per unit area to be stated.

A major converter mentioned MIL-STD-22191 as one which presumably places packaging materials in classes. It mentions Scotchpak (Mylar-polyethylene laminate) but excludes all others.

The new specification--MIL-STD-1246A--prepared by Dennis Conley of General Electric is of interest to most suppliers and users contacted.

In the collective opinion of staff members of a major supplier, the more important specifications include MSFC-SPEC-164 (use levels for NVR analysis), KSC-C-123D, MSFC-SPEC-106A (LOX compatibility), MBO-295-005 (Cleanliness Regulations for Clean Packaging, at various levels, North American), ARP-598 and -743 (hydraulic fluid), MSFC-PROC-195 (designed around a component), and MSFC-SPEC-456 (LOX compatibility).

Another supplier believes the more important specifications to be MBO-295-005, MSFC-SPEC-164, KSC-C-123D, MSFC-10M01671, QA-002, MIL-F-22191, L-P-378A, and Y-939. The same supplier does not like the fact that cleaning specifications are being used as packaging specifications. He recognizes a need for better education of producers and users of packaging materials.

One supplier believes specifications should cover the practical aspects of material control. That is, there should be simple foolproof tests to qualify all materials.

Specification should spell out physical characteristics of film--not require that certain commercial types be used.

One major user summed up the problems in cleanliness specifications as:

(a) Too much unrealistic reliance on previously applied cleanliness specifications which are often extended to situations where they are not necessarily appropriate.

(b) Lack of information on the degree of cleanliness needed for a part to function properly.

(c) A need for designers and engineers of space hardware to be knowledgeable in the problems of contamination, methods of cleaning, and cleanliness levels.

(d) Definition of the time a part must meet the required cleanliness level--before packaging, after packaging, on receipt, at time of use, etc.

General. It would be advisable to have a "standard" part on which evaluation of cleaning techniques, cleanliness measurement, and packaging and packing materials and methods could be carried out.

Films should be coded for ready identification. Human errors account for many mistakes and problems.

Other important factors for consideration include tighter tolerances, biocleanliness, better definition of film types, environmental sensitivity of films, contamination tolerances.

There is need for innovation--entirely new ways of packaging.

One other rather important contact was made. This was with the American Society for Testing and Materials (ASTM), probably the most important technical society in this country engaged in setting standards. After discussing the activities of various committees in the area of clean packaging, the Assistant Technical Secretary of ASTM--Mr. Samuel F. Etris--concluded that the committee members and other interested people had not (up to that time) been able to decide on a unified organizational approach to the problem. The result is that several committees have standards related to the general problem but no specific studies have been made. It was suggested that Battelle use existing ASTM standards directly where applicable and as guidelines in formulating any new standard procedures.

Mr. Etris felt that Committee F-2 on Flexible Barrier Materials would be of help in supplying new information on testing methods for the various films that might be used in the clean packaging field. (Etris was instrumental in setting up this committee.) He indicated that they are presently working on test methods for seam strength, grease penetration, and evaporation of solvent from films. Some of the work on the properties of film has been or is being transferred to Committee D-20 on Plastics.

The problem of evaluating abrasion or sloughing of films was discussed. Mr. Etris stated that there is presently no test on sloughing and that the abrasion tests are designed for plastic sheet rather than film. Mr. Whittier of ASTM felt that a more gentle reciprocating rubbing of film (similar to that which was being planned at Battelle and which became the reciprocating stylus method) might be more appropriate than the standard abrasion test (ASTM-D-1175-64T).

The use of a standard part was felt to be a reasonable approach to determining certain packaging problems. However, it was felt that a manipulation similar to that used in the mechanical testing of packing materials (ASTM D-782-60T) would be preferable to shipping the packaged part. This procedure rolls the packages in a hexagonal tumbler.

Mr. Etris indicated that the following portions of ASTM books might be a help in devising a sloughing test:

Surface Contamination (Part 8)
Committee F-1: Materials for Electronic Devices and Microelectronics

Atmospheric Analysis (Part 23)
Committee D-22: Methods of Atmospheric Sampling and Analysis

Current Specifications and Standards

In the earlier portions of the study, relevant specifications and standards in the broad area of cleaning, cleanliness measurement, handling, and packaging of space hardware were obtained, cataloged, and evaluated. The examination of these documents, discussions with users, and a review of pertinent literature confirmed the need for a standardized and effective means for the measurement of the most important single characteristic of a clean packaging material--its tendency to abrade or slough, thus generating an objectionable particulate contaminant.

Some 70 documents were obtained. Using the specifications outlined in the NASA contract, i.e., MSFC-SPEC-164, -166A, -246, and Drawing 10419906, all of the standards referenced were ordered along with others believed to be pertinent to the problem. Additional specifications were selected based on information in various papers written on the subject of clean packaging or referred to Battelle staff members through contacts in the field. Many of the specifications received bear only indirectly on the problem since they dealt with such subjects as cleaning solvents, cleaners, gases, shipping containers, etc. Specifications believed to be of greatest help are those which can be listed under one or more of the following headings: Packaging of Space Hardware, Cleaning of Space Hardware, Testing of Hardware for Cleanliness, and Films for Packaging. There are approximately 20 specifications covering these areas. Only one of these mentions that the cleanliness level required for the packaging film must equal that of the hardware being packaged. This is the proposed Military Specification MIL-STD-1246A. All others, at best, state that the film must be clean without defining the degree of cleanliness required.

Appendix A lists the 70-some specifications under three main headings (Federal, Military, and NASA), as well as under various categories such as Packaging of Space Hardware, etc.

Current Methods of Evaluating Cleanliness Level

A portion of the work during this program was directed toward the analysis of standards and testing methods for evaluating the cleanliness level of hardware and packaging materials. Along with these analyses, a review was made of currently used methods for measuring cleanliness levels. This effort has been made because the value of any specification is dependent on the methods used in making the cleanliness measurements.

Direct Visual Examination

One method of evaluating the cleanliness level of hardware and packaging materials is to visually examine the surface of interest. This method is most applicable to the detection of relatively gross quantities of contaminants such as shop dirt, corrosion products, oils, and greases. Clean packaging material and relatively flat precision parts may be examined with the aid of a light microscope. If particles can be readily distinguished from the surface, the particle size distribution may be determined. A standard method for measuring and counting particulate contamination on surfaces is outlined in ASTM Standard Method F-24-65. Ultraviolet light may be used as an aid in detecting contaminants which naturally fluoresce such as some mineral oils, greases, and lints. Practically all standards and specifications regarding hardware and packaging cleanliness refer to a direct visual examination as the first step in determining cleanliness.

Wipe Test

The wipe test consists of lightly wiping the surface of interest with a clean cloth or filter paper and visually examining the wiping medium for contaminants. The wiping medium may be white or colored to aid in contaminant detection and identification. The method is particularly useful in gaining a first estimate of the cleanliness levels of chambers and passageways of intricate parts which cannot be visually inspected directly.

Water Break

There are several so-called "water break" methods in which nonwettable, or hydrophobic, contaminants are detected by observing the behavior of water on the surface of interest. On a surface free of contamination, a water film will spread evenly and as the water runs off, there is essentially a zero angle of contact between the film and the surface. In the presence of hydrophobic material, a water film will tend to "break" and not run off a surface evenly. Also, the contact angle is steeper and proportional to the amount of hydrophobic contamination present. Relatively small components and areas of packaging materials may be inspected using this method by merely dipping them in clean, pure water and observing the water break and the receding contact angle as the water runs off. Hof[(1)*], in a review of cleanliness determination procedures, indicates that slight water breaks are made more apparent by examining the surface of interest under kerosene.

The atomizer test is a method in which a fine water mist is sprayed on the surface of interest and the spreading behavior of the water is observed. For a clean surface, the impacting water droplets will spread out evenly. On a surface containing hydrophobic contaminants, there is less wetting and discrete water droplets remain intact. White[(2)] reports that the appearance of sprayed surfaces may be compared to photographs of sprayed surfaces with known contamination levels and a "grade number" assigned to the surface in question to describe its cleanliness level. The usefulness of this test is limited by the subjectivity of assigning a grade number to a surface and the fact that the behavior of the spray droplets is somewhat dependent on the nature of the hydrophobic contaminant.

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* References are on page 72.

To make the water break method a quantitative measure of cleanliness, White suggests that the contact angle between water droplets and the surface in question be accurately measured with a reflection goniometer. The contact angle is a fundamental surface property which appears to have a wide range of values for different surface contaminants and levels of contamination. This method is still in the process of development and may be extremely useful when the relationship between the contact angle and the type and degree of contamination is more firmly established.

Fluid Extraction Inspection

Fluid extraction methods involve the rinsing or purging of surfaces of interest and the subsequent examination of the flushing medium for extracted contamination. Both liquid solvents and compressed gases are used. Particulate matter may be filtered from the flushing medium and the particle concentration and size distribution may be determined using a standard counting method such as ASTM Standard Method F-24-65. Also, the total filterable solids may be weighed following a procedure outlined in ASTM Standard D-2387, Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis.

The amount of soluble contamination extracted from a surface is determined in a number of ways. One method is to allow the filtered flushing solvent to evaporate and to obtain the weight of the remaining substance called "nonvolatile residue". The cleanliness meter, described by Marsh[(3)], is an instrument capable of determining the concentration of nonvolatile residues in concentrations as low as from one to ten parts per million. The instrument operates as follows: The filtered flushing solvent containing soluble residue is passed through a nebulizer where the solvent is dispersed as a fine aerosol. Clean air is added to dry the aerosol stream and the nonvolatile residue concentration is determined with a light scattering photometer.

A third method of detecting the presence of soluble contaminants is with spectrographic procedures. A sample is obtained by washing the surface of interest with a solvent or by repeatedly depositing and picking up a drop of solvent on the surface. An infrared spectrophotometer is then used to analyze the sample for contaminants of interest. Although this method is most often used to detect the presence of condensable hydrocarbon residues on components of liquid oxygen systems, many contaminants may be detected and identified using spectrographic procedures.

The cleanliness level as determined by fluid extraction procedures is an accurate representation of the cleanliness level of a surface in question only if all significant contamination has been extracted for analysis. The utmost care should be taken during the processes of flushing, purging, and immersion to insure that all contamination is removed. Also, the cleanliness level of the flushing medium should be accurately known and taken into consideration when analyzing cleanliness measurements.

Evaporation Rate

A relatively new technique for the quantitative detection of surface contamination has been reported by Anderson(4). Surface contaminants are detected by measuring the rate of evaporation of an added radioactive volatile test solution with a Geiger-Mueller radiation detector tube. Evaporation rate is a function of surface properties such as roughness and porosity and the amount and type of contamination present. Under similar conditions, the rate of evaporation from a clean surface is always greater than from a contaminated surface. By comparing the evaporation rate of the test solvent from a surface in question to the rate associated with a known level of contamination on the same surface, a quantitative measure of the cleanliness level may be obtained. Recent specifications for the Meseran, a practical instrument using the evaporation rate principle, indicate that the method is sensitive to less than a single layer of residue molecules. The process appears to be most useful for repetitive examinations of similar surfaces when the nature of the contamination is known.

Miscellaneous Methods

An acidity and alkalinity test is often performed on surfaces of interest with pH-indicating paper and a small drop of clean distilled water. An abnormal pH reading may indicate the presence of a contaminant.

Hof[(1)] refers to a ring test in which a drop of water in a surface tension tester ring is repeatedly lowered to touch a surface of interest. The number of contacts with the surface which results in the total transfer of the drop to the surface is called the B number and is considered a measure of the surface wettability. The B number is dependent on the amount and type of contamination present and is, in general, smaller for lesser amounts of residue.

Review and Analysis of Major Documents

As previously noted, from the standpoint of particulate contamination, slough or abrasion resistance is probably the most important single characteristic of protective packaging films used in contact with space-vehicle hardware. Even though this is undoubtedly true, no existing specification either presents a method for measuring sloughing characteristics or establishes acceptable limits of in-service contamination by sloughing.

Those specifications and standards considered to be sufficiently important for review and analysis have been divided into two broad classes--those dealing with film and those concerned primarily with hardware cleanliness or cleanliness measurement. These are listed below:

Class I - Film

L-P-387A
MIL-B-131B
MIL-B-22205A
MIL-L-10547B
MIL-F-22191A
MSFC-SPEC-456
MSFC-SPEC-C-12A

Class II - Hardware Cleanliness

MIL-STD-1246A (Proposed)
MSFC-10M01671
MSFC-DWG-10419906B
MSFC-SPEC-164
MSFC-PROC-166C
MSFC-PROC-195
KSC-C-123D
MIL-SPEC-M9950
MSC-SPEC-5A
MSC-SPEC-7

In certain instances, the organization and language commonly used in the standards and specifications make them somewhat difficult to interpret and the stated objectives sometimes restrict applicability. Many of the existing standards were prepared several years ago and nearly half were not directed toward space applications. One example of this is the suggested use of aluminum foil, a readily abradable material, in MSFC-DWG-10419906 and also in MIL-M-9950. However, most are well conceived and provide useful outlines for establishing cleanliness levels for parts. In many cases, justification for the degree of cleanliness specified would strengthen the technical relevance of the chosen levels. Also, some correspondence between cleanliness levels stated in the various specifications appears to be desirable. At present, it is possible for a part to be specified to a cleanliness level required by one standard, such as MIL-STD-1246A, for which there is no counterpart in other standards. All parts comprising an assembly should be cleaned to the same level using the same standard in order for the assembly to meet a congruent cleanliness level.

The levels of cleanliness required for various packaging materials and components are dependent on the application of the components. Various levels are listed which are intended to insure the desired reliability for the component or assembly when the specified cleanliness level is attained. The major listings of cleanliness levels for surfaces are found in MSFC-PROC-166A, MSFC-DWG-10419906B, MSFC-PROC-195, MSFC-10M01671, KSC-C-123D, MSC-SPEC-C-7, MSC-SPEC-C-11, and MIL-STD-1246A (Proposed). With the exception of MIL-STD-1246A (Proposed), these specifications are directed toward particular systems. Cleanliness levels for packaging materials have often been ambiguously specified as "clean" except in the more recent cases where it is specified that the package must be as clean as the part which is to be packaged.

Any or all cleanliness requirements can be criticized because of the general lack of information relating part, assembly, or system reliability to cleanliness level. However, experience and logic undoubtedly provide a basis for most choices of required cleanliness levels. It seems unlikely that many slightly different levels are really necessary. A list of well ordered levels such as those proposed in MIL-STD-1246A could serve adequately as a reference for all requirements. This is particularly true in view of the uncertainties relating reliability and cleanliness. To be sure, it appears logical that the cleaner the system, the more reliable it will be. However, it cannot be generally stated, for instance, that a single 1-micron particle is more or less desirable than a large number of 0.1-micron particles. Small differences, then, between cleanliness levels probably reflect little or no difference in reliability, or at least the effect is unknown. For these reasons, it is suggested that one uniform series of well ordered levels should be employed. There is no reason to establish a completely new series of cleanliness levels for each new piece of hardware. Instead, it is necessary only to specify to which of a standard series of cleanliness levels each part should conform.

It is also believed that the proper method of presenting cleanliness levels is through the use of a continuous particle size distribution. This will allow more latitude in particle counting techniques by permitting use of various reticles as well as micrometer eyepieces. This approach has been used in the proposed MIL-STD-1246A along with a breakdown by size increment.

Some further comments on the proposed MIL-STD-1246A seem to be justified because of inconsistencies between the continuous particle size distributions and the tabular representations. The tabular listings appear to be inconsistent with respect to the continuous distributions from which they were derived. The continuous distributions are presented as a plot of particle size against the number of particles having a size greater than the indicated size (i.e., cumulative number of particles). The proper procedure to find the number in any size increment is to subtract the number of particles greater than the larger size from the number of particles greater than the smaller size. Apparently, the procedure used to obtain the tabular levels in MIL-STD-1246A was to read the number of particles corresponding to the midpoint of the range under consideration.

A simple example can be used to indicate the error of using the midpoint to obtain the number of particles in a size range. Figure 1 shows the continuous distribution for level 50 as taken from Chart 1 of MIL-STD-1246A. The following illustrates the differences between procedures.

Particle Size, microns	Point on Graph	Number of Particles Greater than Indicated Size
5	A	170
10	B	60
15	C	26
25	D	7

Particles greater than 5 microns	170
Minus particles greater than 15 microns	-26
Particles in size range 5-15 microns	144
Particles at midpoint (b)	60
Particles greater than 5 microns	170
Minus particles greater than 25 microns	-7
Particles in size range 5-25 microns	163
Particles at midpoint (C)	26

It is obvious that the midpoint procedure as used to obtain Table 1 in MIL-STD-1246A is not correct because this method indicates fewer particles in the 5-25-micron range than in the 5-15-micron range. It is suggested that Table 1 in MIL-STD-1246A be revised to be consistent with Chart 1 which gives the continuous distributions.

The following conclusions can be drawn which relate to measurement and levels of cleanliness.

(1) Many and different cleanliness levels exist in specifications and standards. These present a confusing and inconsistent picture of what is required to assure adequate reliability.

(2) Of the many methods available for measuring surface cleanliness, the more qualitative methods are suitable for measurement of contaminants which are on the surface while the more quantitative methods are for determining what has been removed from the surface. The amount of contaminant removed from the surface does not necessarily indicate how much contaminant remains on the surface.

(3) A more generally applicable and in many ways superior method of representing particulate cleanliness levels is by means of a continuous particle size distribution rather than a series of discrete size groups.

Pertinent observations on the specifications and standards in question follow:

(1) Low-density polyethylene, because it sloughs readily, is a poor choice of material for use in direct contact with critical components of space hardware, yet it is suggested in MSFC-SPEC-164.

(2) No existing specification takes cognizance of the presence of processing aids and antiblocking or slip agents in polyolefin films. Some of these materials are volatile, particularly under vacuum, and provide a source of contamination.

(3) It is characteristic of several specifications that polyethylenes are mentioned without defining the type.

(4) Specification MSFC-PROC-166C and MSFC-10M01671 indicate use of Saran film in contact with space hardware. While under certain conditions this might be a slight improvement over low-density polyethylene, the same specification includes aluminum foil as an overwrap, increasing the chance of contamination in shipping or in opening the packages.

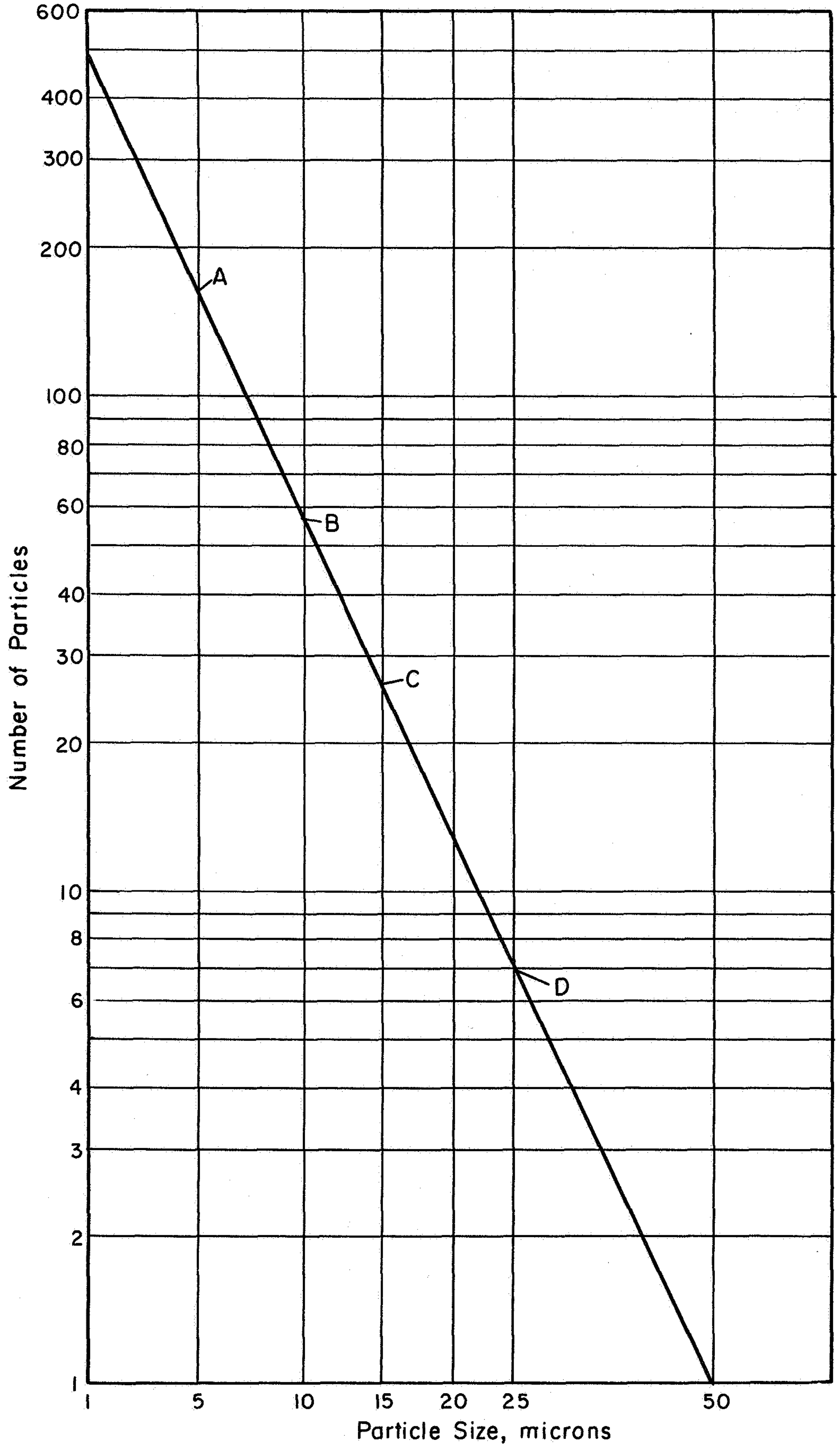


FIGURE 1. PARTICLE-SIZE DISTRIBUTION FOR LEVEL 50 FROM MIL-STD-1246A

(5) Specification KSC-C-123D represents an improvement in certain respects over earlier documents, yet it mentions commercial films by name without noting the required performance. Also, it mentions polyethylene with no indication of type.

(6) MIL-STD-1246A is probably the best of the specifications reviewed in terms of required physical properties and cleanliness levels of packaging materials, but its references are in terms of parts, requiring, in turn, reference to parts specifications.

The specifications for packaging materials themselves are less than adequate for use by the aerospace industry. Any attempt to improve the presently used standards should have simplification as a primary goal. A real need exists for a NASA standard for clean packaging materials. It is recognized that polymeric films offer by far the strongest approach to solving problems of clean packaging where contact with a clean part is essential. Therefore, as a first and most important step, it is proposed that a specification be developed which will apply only to materials of this class. Such a document should presuppose that new and improved materials will appear (the polycarbonates represent one very interesting class as will be shown in a subsequent section of the report) and make possible their acceptance on the basis of performance characteristics alone.

Considerations for Future Specifications

It is important that attention be given to properties of film or film composites--both available and potentially achievable. The more important properties are listed below. Importance of these, of course, varies with the end use. LOX compatibility should be an overriding factor where this property is deemed essential. It could be shown as a go, no-go delineation of materials.

Film Properties

LOX compatibility
Abrasion or slough resistance
Cleanliness, cleanability
Moisture-vapor transmission rate (MVTR)
Gas barrier characteristics
Waterproofness
Heat sealability
Purity
Tensile properties
Burst strength
Folding endurance
Bondability
Transparency
Blocking resistance
Grease resistance
Chemical resistance
Temperature resistance (low and high)

The points believed to be important in any future consideration of specifications are listed below:

(1) Properties listed should be at specified levels defining upper and lower limits of each level.

(2) Cleanliness levels of packaging materials should be defined in such a way as to be compatible with levels specified for critical parts.

(3) The specification preferably should not identify the material as a single type or trade name (except as an illustration), but rather should permit inclusion of any product meeting the required standards.

(4) Tests to qualify films should be identified to the degree possible by existing ASTM standards.

(5) In the event of need for a user or converter to carry out a final qualifying test, such tests should not only be simple and foolproof but should also be described clearly.

(6) To the degree possible, the specification should stand on its own. Frequent reference to previous specifications, some of which may have fallen into disuse, will weaken the document and increase the chance that certain requirements might be ignored.

(7) Sealing tapes should be covered by a recommendation for a tape of specific class.

PHASE II. MATERIAL DEVELOPMENT AND APPLICATION

Phase II of this program was concerned with four basic properties of packaging materials--resistance to sloughing, LOX compatibility, good moisture and gas barrier characteristics, and adequate sealability. It has been pointed out in earlier reports that presently existing films or other packaging materials--or indeed composites of these materials--are capable of contributing all but the first of these characteristics. Until now, Nylon 6 has been considered by many to be the best choice as a slough-resistant material. However, because of limited available information on both slough resistance of packaging materials and slough-measuring techniques, this conclusion might not be completely valid. Thus, it has been necessary also to work toward improved quantitative methods for measuring abrasion or sloughing tendencies and to investigate the performance of films on which no data exist. This section summarizes Phase II activity which includes a general appraisal of candidate packaging materials and experimental measurements of the generation of particulate matter by specific films.

The development of wholly new packaging materials--particularly the preparation of new polymeric entities combining all four of the desirable characteristics for clean packaging is obviously beyond the scope of the project. In fact, the industry for years has been working toward obtaining film-forming polymers combining more ideal barrier and sealing properties while maintaining other desirable attributes such as high strength, good machinability, transparency, resistance to large changes in temperature, low cost, etc. To date, the best answers can be obtained only through the use of composites. However, Battelle background in products of this class plus information available from both the field and the literature have been helpful in reaching some of the important conclusions.

Sloughing

The cleanliness level of space-vehicle hardware is maintained through the use of clean packaging materials and procedures. It is essential that a clean package not only protect a critical part from the exterior environment but that the package itself does not contaminate the part. To meet the latter requirement, clean packaging materials should have at least the same degree of cleanliness as the part to be packaged and should not slough significantly during handling, shipping, and storage of the packaged part.

Evaluation of the sloughing tendency of films was investigated using three methods: the Taber Abraser method, the tumble box method, and the reciprocating stylus method. The objective was to provide definite information on the sloughing tendencies of films for the purpose of evaluating relative performance as clean packaging materials, as well as to determine the effectiveness of the procedures. It was necessary to develop and evaluate these techniques since up to this point no prescribed method existed for determining this particular property of a packaging material.

Sloughing can be specifically defined as the breaking away of small pieces from the surface of the packaging material. In this manner, the package becomes a source of contamination. Sloughing seems to be caused by two separate mechanisms. Movement of the packaged part within the package can cause sloughing through abrasive action as the part rubs against the package. Also, movement of the package itself can cause sloughing through flexing of the packaging material. A suitable procedure for measuring sloughing tendencies of films should provide a measure of total sloughing resulting from both flexing and abrasion unless it can be determined that one or the other mechanism predominates.

A general problem concerning measurement techniques is determination of the proper quantity to be measured. Since the particles sloughed are of primary concern, it seems logical to use particle count and size distribution as the measured quantity. However, the weight loss during abrasion has often been used as an indication of sloughing because of its simplicity.

Aside from measurements of abrasion resistance, there have been only a few experimental studies of sloughing tendencies and these have been applied to only a limited number of films. It is not now possible, therefore, to compare methods or films on a quantitative basis.

However, several of the methods described below have been useful in providing a comparison of sloughing among films and metal foils and have provided a basis for investigations of alternate procedures.

The most widely used measure of resistance to material loss by films is the Taber Abraser method. Federal Standard No. 101a[5] refers to a widely used abrasion test described in ASTM Standard Test Method D-1175-64T[6]. This method covers the measurement of abrasion resistance using a double-head, rotary platform tester such as the Taber Abraser. Abrasion resistance is measured by clamping the material in question onto a flat circular platform which turns at 70 rpm under two grinding wheels. The degree to which each material abrades is determined by measuring weight loss of the material under the specified test conditions.

A second type of sloughing test is the flexed sheet method. Marshall Space Flight Center Memorandum No. R-ME-MMC-96-94 refers to a study in which 1-foot-square samples of clean packaging materials were first cleaned and then carefully flexed, roughed, and smoothed[7]. The materials were then washed with 100 ml of solvent which was filtered through a membrane filter. The particles collected were counted in the size intervals 10 to 25, 25 to 50, 50 to 100, and those greater than 100 microns.

A third type of sloughing test can be called the handled-package method. NASA Test Report No. TR-92-D, entitled "Plastic Film Packaging Materials", refers to a slough testing procedure in which 6 x 8-inch clean packages containing standard weights were flexed and twisted according to a standard procedure to simulate handling and shipping conditions[8]. A bag made of each material tested and containing no part was left open on one end to serve as a control condition for each test. Membrane filter samples were obtained for test bags and control bags by washing out each with a clean solvent. Particles were counted in the size intervals 15 to 30, 30 to 45, 45 to 65, 65 to 100, and those greater than 100 microns. The difference in particle count between the test bag and the control bag for each material was considered to be the amount sloughed during flexing.

A final type of sloughing test has employed the Gelbo Tester, a device capable of simultaneously crumpling and twisting sleeves of clean packaging materials. Standard parts might also be placed in the sleeves and the tester operated to determine the degree to which various materials slough when subjected to this treatment. The system appears to be applicable to the measurement of sloughing, but no data are available at this time on its use.

Results for the three methods mentioned above are very limited. Abrasion resistance, as determined with the Taber Abraser, is often available in manufacturers' listings of film specifications, however, the number of revolutions, the type of abrasive wheel used, and weights added are not standardized and direct comparisons between the films are not always possible. The other methods have been applied to only a few films and these films were all different for each method so no comparison between methods is possible. The available results for each film are probably inconclusive because of the limited amount of testing and, further, the results for the handled package method do not seem to be dependable.

Experimental Procedures

Mechanical Abrasion. This method involved the use of a Taber Abraser, which is a rotary platform, double-head abraser commonly used to test the abrasion resistance or durability of textile fabrics. A detailed description of the apparatus and procedure are given in ASTM Standard D-1175-64T[6]. Abrasion resistance is measured with the Taber Abraser by allowing the test material, which is clamped onto a flat circular platform, to revolve under two circular rubber-base or vitrified-base abrasive wheels. The degree of abrasive action is controlled by varying the type of wheels used and the pressure applied to the wheels. The degree to which each clean packaging material sloughs was determined by measuring the material's weight loss after a specific number of revolutions under an appropriate abrasive wheel and pressure combination. In preparation, a special collar or clamp was constructed to hold the films on the circular platform. The inside diameter of the collar was designed to provide a pressed fit over the circular abrasion platform with the test film in place. This pulled the film tight and held it securely.

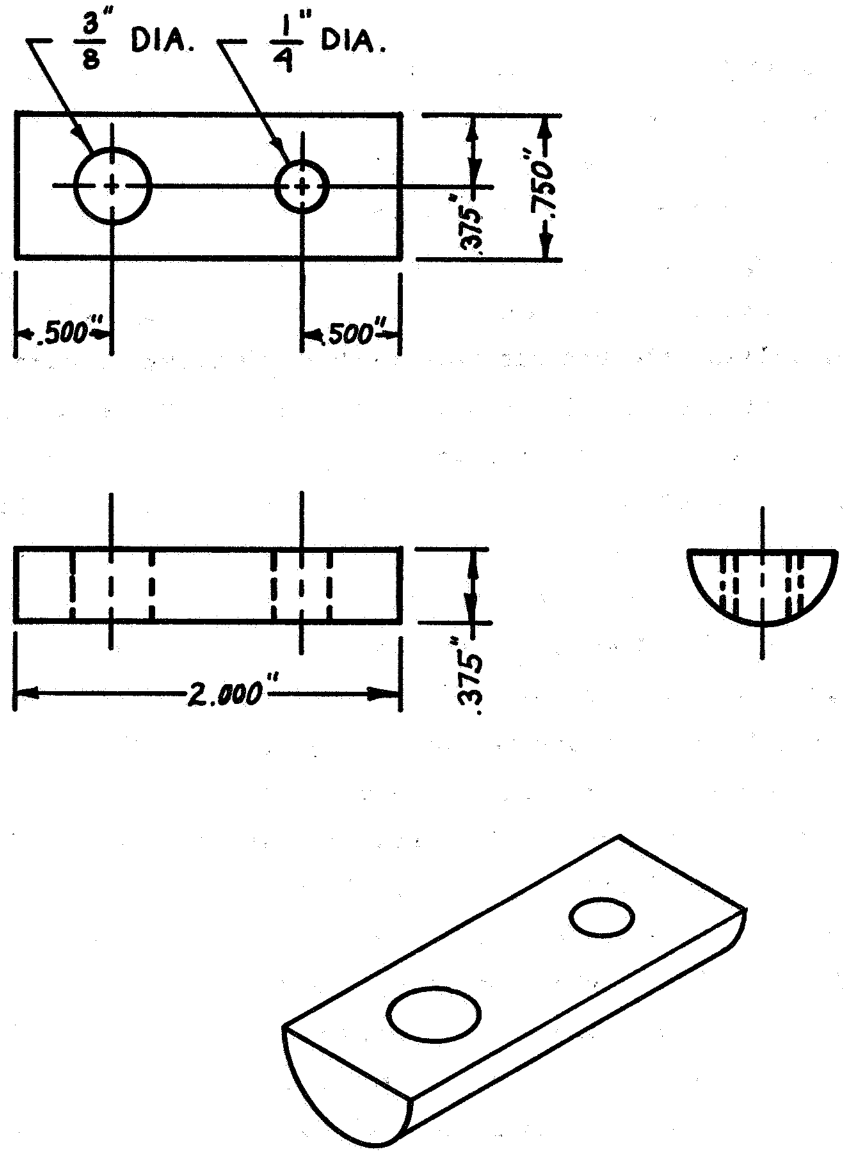
One unforeseen problem arose in the experiments. This was the fact that the accumulation of static charges on the films during handling and abrasion seriously affected weighing. For this reason, the static charge was removed from the film by rinsing it in a solvent such as ethanol or trichlorotrifluoroethane. This procedure also helped to remove particles of the abrading wheel prior to weighing.

The specific experimental procedure was to cut from the test film a circular section slightly larger in diameter than the rotating platform of the abraser. The film specimen was weighed and clamped to the circular platform. The platform was then rotated at 70 rpm in contact with two CS-17 wheels, each of which was loaded with a weight of 1000 grams. The weight loss was determined for various numbers of revolutions by removing, cleaning, and weighing the film. The film was then repositioned on the abraser platform and abrasion was continued for another predetermined number of revolutions. This was continued until the film was torn or a hole developed in it.

Packaged Part Tumbling. This second experimental method is designed to subject the clean packaging material to the type of manipulation which might occur during actual shipping and handling. Clean packages, made by heat sealing the various packaging materials and containing a "standard part", were subjected to a tumbling action in a revolving hexagonal drum. After a specific number of revolutions, the particles were washed from the inside of the package and counted. Although this test is most suitable for those materials which may be heat-sealed, nonheat-sealable materials could have been evaluated by using taped joints.

A standard stainless steel part, shown in Figure 2, was designed to have a variety of edge and surface configurations. The part is a 2-inch-long, 3/4-inch-diameter cylinder which is cut in half and has two holes, 1/4 and 3/8 inch in diameter, drilled in it.

The hexagonal drum used to tumble the packaged parts is a scaled-down version of a drum referred to in the ASTM Tentative Method



SPECIFICATIONS:

MATERIAL - STAINLESS STEEL TYPE 304

FINISH - 32 TO 64 RMS ON ALL SURFACES

TOLERANCE - ALL DIMENSIONS ± .005"

EDGES - BREAK ALL EDGES 45° x 1/32"

WEIGHT - 50.0 ± 0.5 GRAMS

FIGURE 2. CLEAN PACKAGING STANDARD PART

of Testing Shipping Containers in Revolving Hexagonal Drums, ASTM Designation D-782-60T[9]. Figure 3 is a photograph of the tumble box along with the driving mechanism. It is seen that the box has a hexagonal cross section and is lined with 1/4-inch foam rubber. The box is 24 inches long, 9 inches on each of the six sides, and has a 22-inch-diameter Plexiglas cover on each end. The covers provide the circular shape for rolling the box and have 6-3/4-inch-diameter openings in each end. An additional 1/4-inch-thick strip of foam rubber about 1 inch wide was centered on each flat side to promote tumbling. In preliminary experiments, it was found that a speed of 12 rpm resulted in satisfactory tumbling action and package size of 3 x 3 inches seemed to be best suited to the size of the standard parts and the tumbling motion.

A Model B Coulter counter was calibrated and tested for use in determining the number of particles sloughed from materials evaluated by means of the tumble box and reciprocating stylus methods. The Coulter counter is capable of electronically counting and sizing particles from about 0.5 to 800 microns in diameter. In this device, particles are suspended in an electrically conductive liquid. The counter works in the following way: An aperture with an electrode on either side is immersed in a beaker of electrolyte containing the particle suspension. The electrolyte is drawn through the aperture at a rate of a few milliliters per minute. The resistance between the immersed electrodes is momentarily changed with the passage of each particle through the aperture and a voltage pulse is produced of a magnitude proportional to the particle volume. Each pulse which occurs is then electronically amplified, scaled, and counted.

The Coulter counter was used in the experimental program for several reasons. First, particle number vs size distribution may be determined for a sample in less than 15 minutes as compared with 2 to 3 hours required to obtain similar results with a light microscope. Another reason for utilizing the automatic counter is the fact that the experimental data are not as subjective and dependent on operator performance as results obtained using microscopy. It was found that by using the Coulter counter, more measurements of slough could be obtained and the results were more accurate and reproducible.

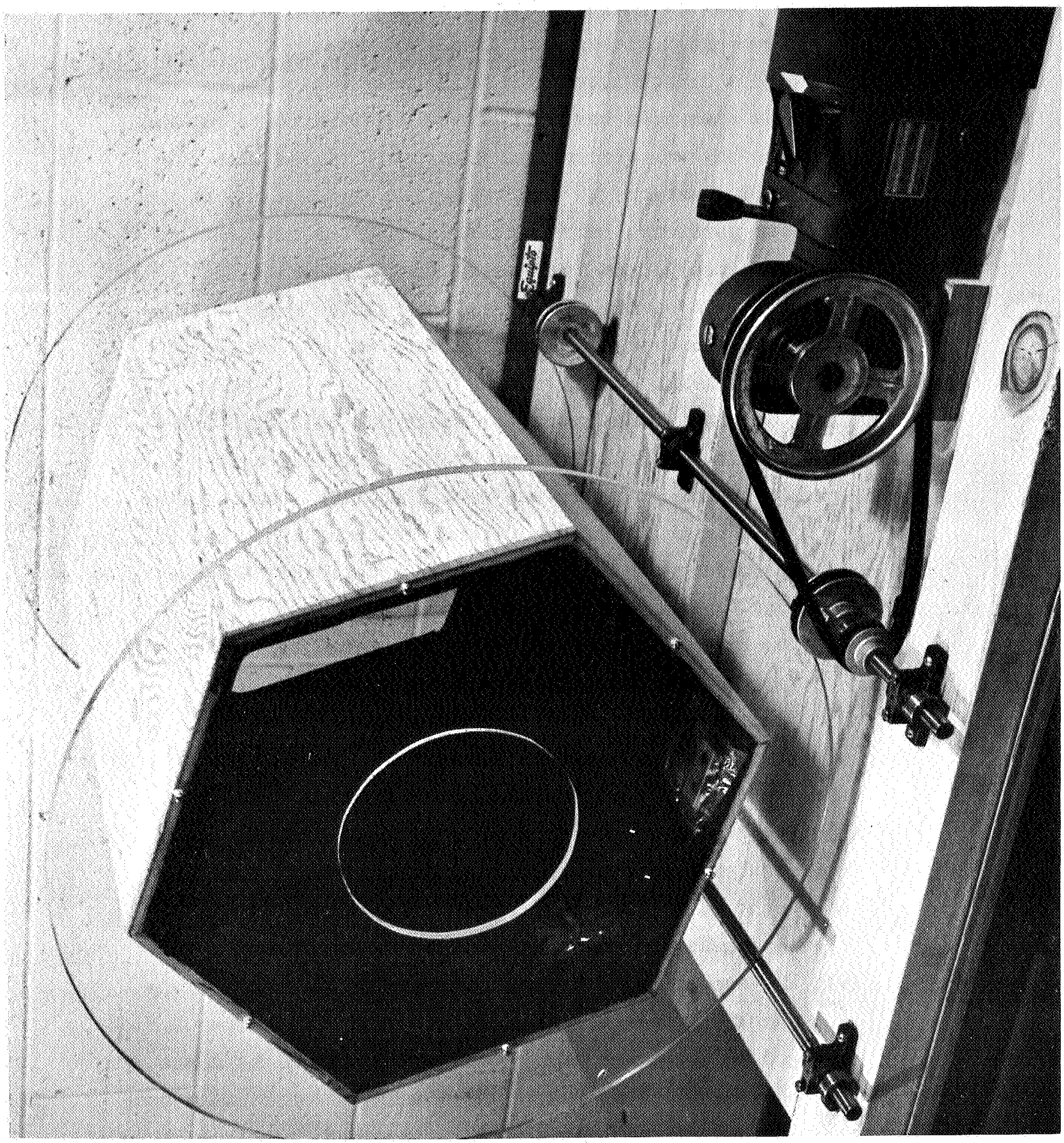


FIGURE 3. PHOTOGRAPH OF TUMBLE BOX

The experimental procedure used to obtain data with the tumble box method was as follows:

(1) A standard part as shown in Figure 2 was cleaned and thoroughly rinsed with distilled water.

(2) A 3 x 3-inch-square bag with three sealed sides and one open side was made of the material to be tested.

(3) The bag was cleaned and thoroughly rinsed with distilled water.

(4) The part was placed in the bag and the combination repeatedly rinsed with a filtered 2 percent NaCl water solution having less than 30 particles greater than 5 microns and containing a trace of wetting agent (Triton-X).

(5) The final 300 ml of rinse water was collected and the number-size distribution of the particles contained in the solution determined with a Coulter counter. The size distribution was determined three times and an average count calculated which constitutes the so-called "background" for each run.

(6) The bag containing the part was sealed, placed in the tumble box, and tumbled for 30 revolutions.

(7) The bag and part were removed from the tumble box, the bag slit open, and the interior rinsed with 300 ml of the clean rinse agent described in Step 4.

(8) The number-size distribution of the particles contained in the rinse water was determined with the Coulter counter. Again, three size distributions of the sample were measured and an average count calculated.

(9) The number and size of the particles sloughed during tumbling were determined by subtracting the Step 8 background count for each size increment from the count obtained in Step 9.

All cleaning, rinsing, and packaging were carried out in a laminar flow clean bench. Background counts were at a level such that the number of particles greater than 5 microns appearing in successive rinses was similar within the limits of counting accuracy.

<u>Reciprocating Stylus</u>. This testing method subjected a strip of clean packaging material, held firmly against a stainless steel surface, to a reciprocating scraping action by a sharp-edged stylus. The test was conducted in the funnel of a microanalysis filter holder. The funnel contained a stainless steel platform holding the clean packaging material to be tested. As the stylus reciprocated across the material, a flow of clean air carried the sloughed particles to the filter. After a specific number of cycles, the apparatus was washed with clean solvent. The number and size of the sloughed particles were then determined for each material.

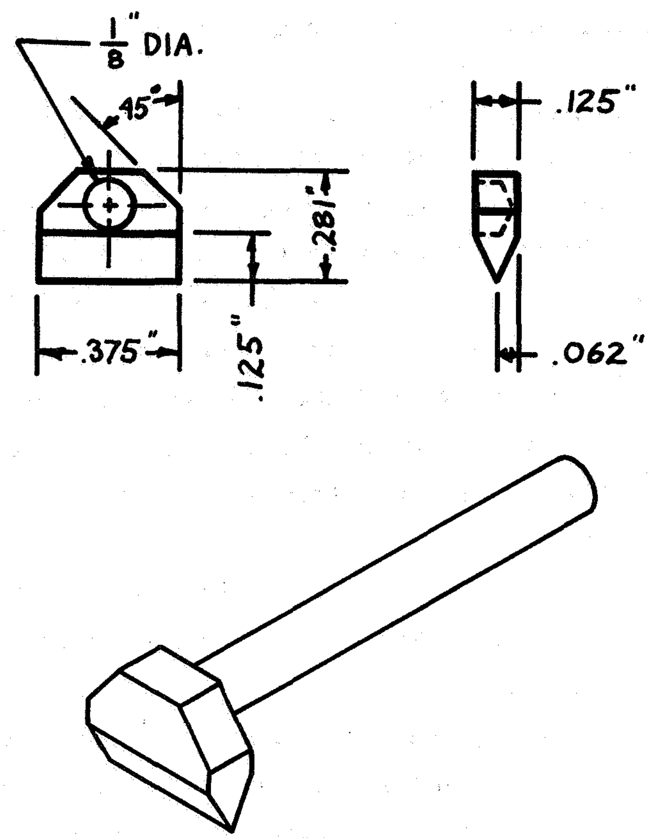
The dimensions and specifications of the stylus which were used are shown in detail in Figure 4. The reciprocating motion was supplied by a mechanism similar to that described in the ASTM Proposed Method of Test for the Erasing Quality of Paper[10]. The experimental apparatus is shown in Figure 5 and the stylus, abrasion surface, and filter holder are shown in Figure 6. The filter holder had an inside diameter of 11/16 inch and length of 3 inches. The film support surface was made of stainless steel, had a length of 2-3/4 inches and a width of 5/8 inch. The stylus shaft was weighted at a point 2-7/8 inches from the stylus, the total shaft length was 5-3/4 inches, and the weight on the stylus edge was 55.5 grams. The stylus reciprocated 23.5 cycles per minute and in all tests 100 cycles were used. The solvent rinse was a 1 percent aqueous solution of sodium chloride.

The experimental procedure for using the reciprocating stylus method was as follows:

(1) The stylus was sharpened and dressed on an Arkansas stone.

(2) The filter holder, film support surface, and stylus were cleaned.

(3) The filter holder was assembled using a membrane filter having a pore size of 0.45 micron.



SPECIFICATIONS:

MATERIAL - STAINLESS STEEL TYPE 304

TOLERANCE - ALL DIMENSIONS ± .005"

STYLUS EDGE - SHARPENED TO HAVE AN ANGLE OF 90 DEGREES

FIGURE 4. CLEAN PACKAGING SLOUGHING STYLUS

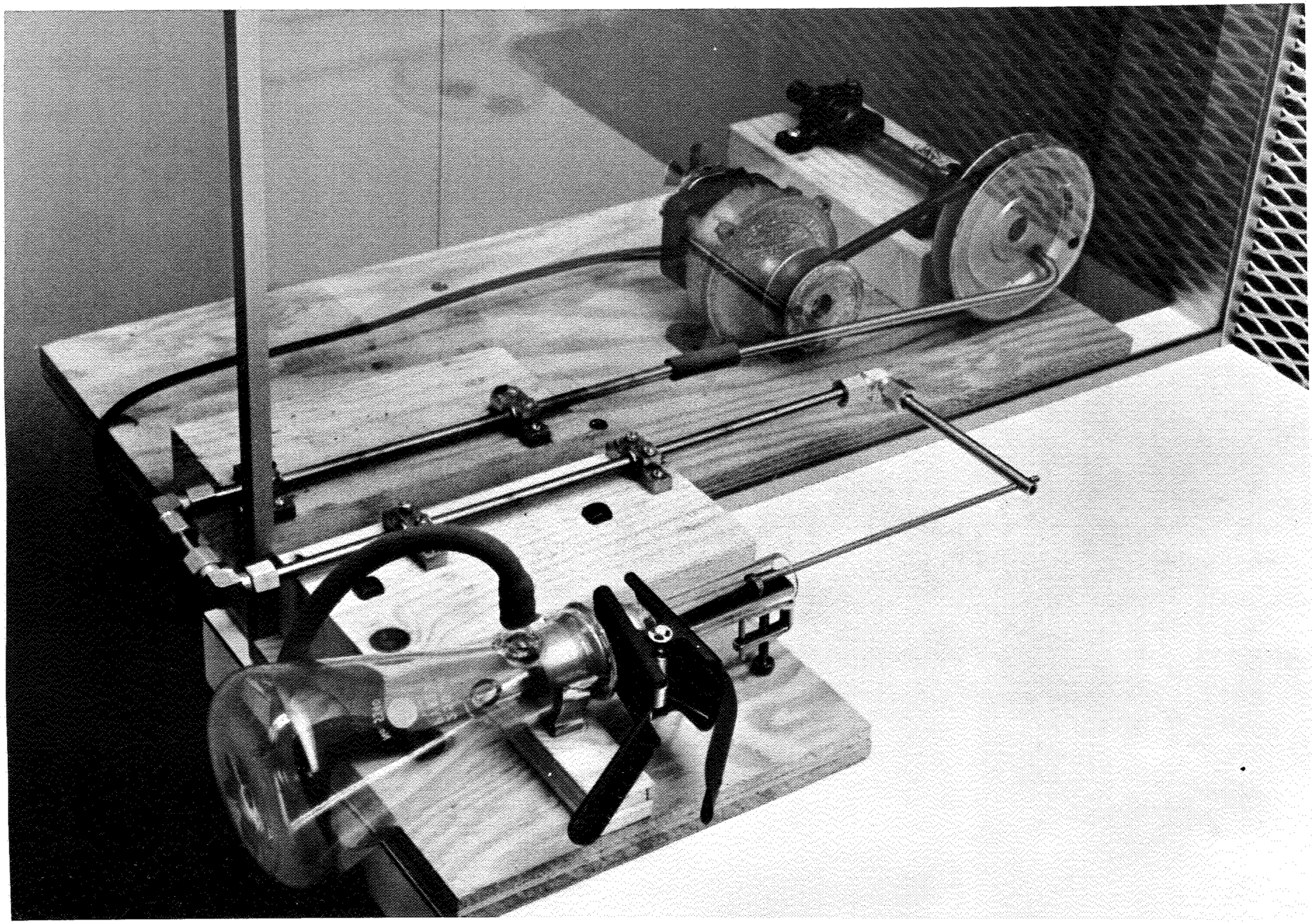


FIGURE 5. EXPERIMENTAL EQUIPMENT FOR RECIPROCATING STYLUS METHOD

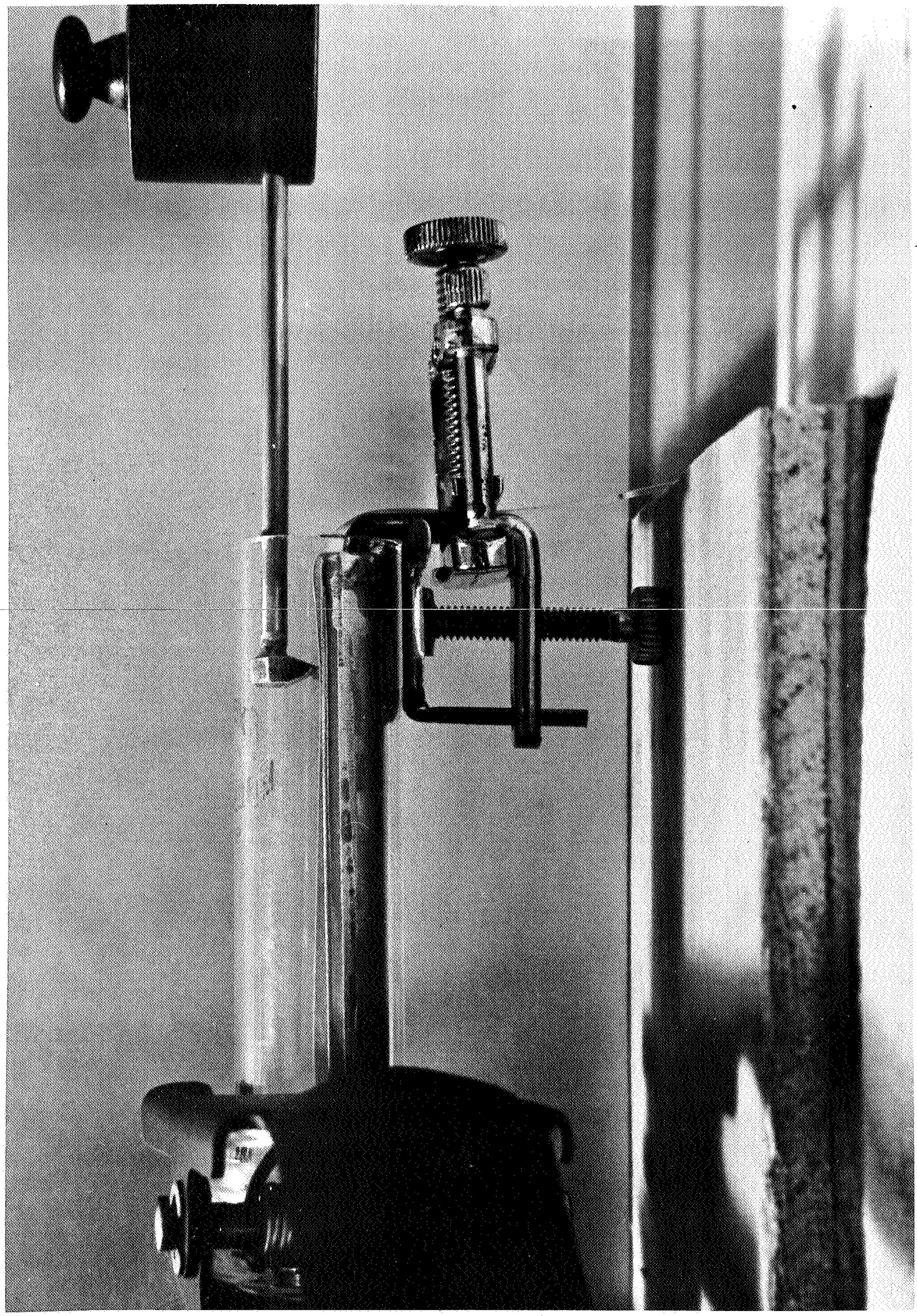


FIGURE 6. PHOTOGRAPH OF STYLUS AND ABRASION SURFACE IN FILTER HOLDER

(4) A strip of film was cut, placed on the film support surface, and this combination was then fixed in the filter holder.

(5) The interior of the filter holder, the film, and the film support surface were repeatedly washed until a consistent particle count was obtained.

(6) A vacuum pump was turned on to draw air through the filter.

(7) The stylus was drawn back and forth across the film for 100 cycles.

(8) The interior of the filter holder with film, filter, film support surface, and stylus were rinsed clean with 300 ml of rinse solution.

(9) The particle size and count in the rinse solution were determined using a Coulter counter.

Comparative Evaluation of Procedures

The experimental results are based on the sloughing tendencies of four films as determined by the three methods described previously. The four films used in this study were low-density polyethylene, Nylon 6, Aclar 22C, and Lexan. These specific films were chosen somewhat arbitrarily but the primary reason was that they gave significantly different results with the Taber Abraser method.

Taber Abraser. The weight loss in milligrams for each of the four films, as determined using the Taber Abraser method, is shown in Figure 7. It is readily apparent that while some scatter in the data exists, this method clearly distinguishes the four films. The data appear to be reproducible to at least ±50 percent and in most cases the scatter is well within these limits. From most to least abrasion resistance, the films can be listed in order as Lexan, Nylon 6, Aclar 22C, and low-density polyethylene. It should be noted that this listing represents comparative abrasion resistance and not necessarily slough resistance.

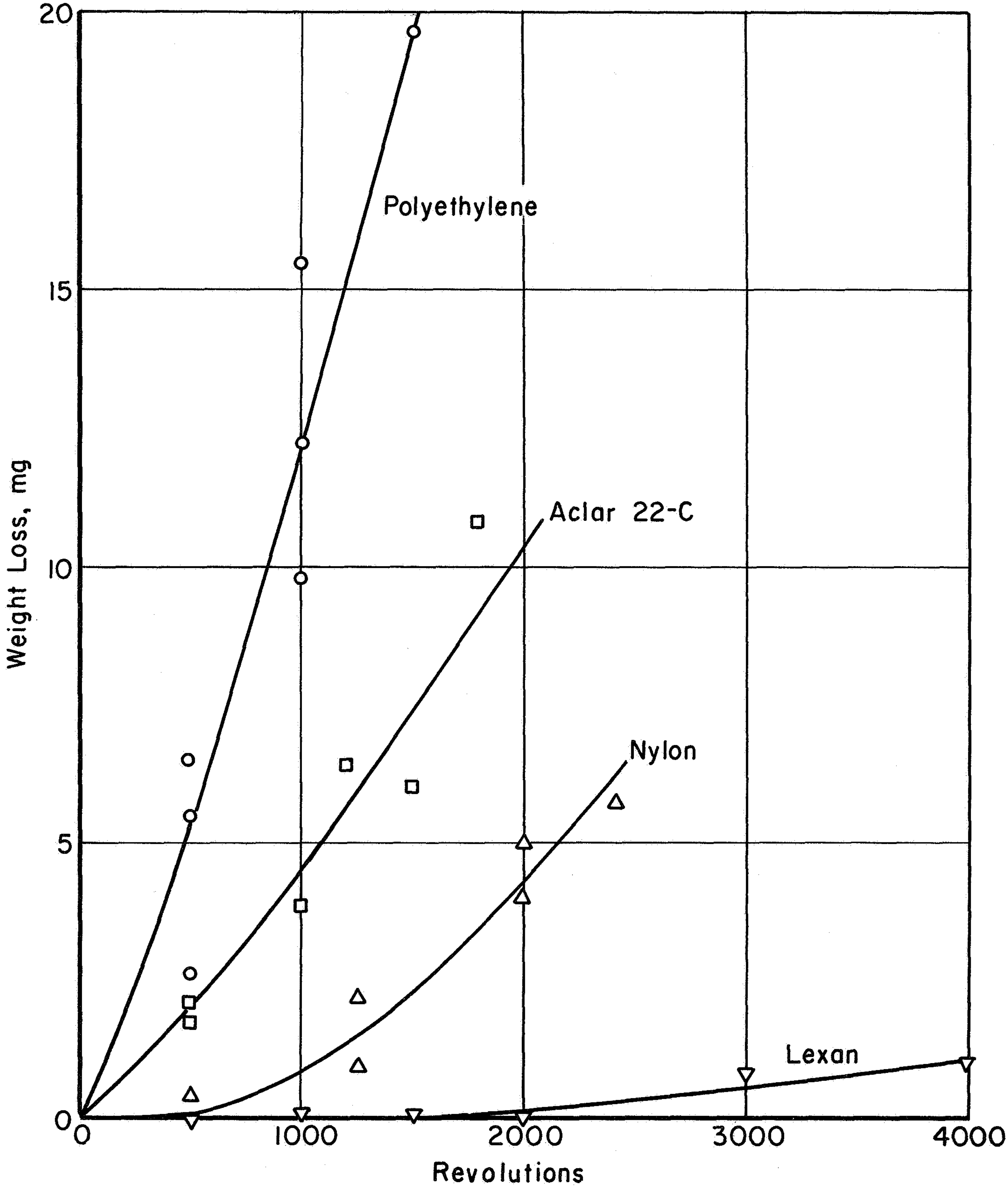


FIGURE 7. WEIGHT LOSS FROM FOUR FILMS DURING ABRASION

Tumble Box. Experimental measurements of slough resistance were performed with the tumble box method in triplicate for the four films except in the case of polyethylene where seven replications were made. The results for polyethylene are presented in Figure 8. Considerable scatter is evident in the data which fall in a range from about one-fifth to five times the values from a curve representing the best fit to the data. A comparison of the best fit curves for all four films is presented as Figure 9. There is apparently little significant difference among the films, except for Aclar 22C which definitely seems to be different. The scatter in the data for the individual films tends to mask any difference in sloughing tendency which might otherwise appear.

The random errors associated with the automatic particle counting and its sampling system are small. The relative error in such measurements, estimated from triplicate counts at each particle size on each wash solution, is ±50 percent or less. Therefore, the scatter can be attributed largely to the tumbling and washing procedures. It is believed that the largest source of scatter is the degree of tightness with which the part is held in the package. It was found that a loosely packaged part would form a hole in the package after a very few revolutions of the tumble box.

Reciprocating Stylus. The reciprocating stylus method was used to determine sloughing by abrasion for three of the four films evaluated with the other two methods. The results for polyethylene are presented in Figure 10. The scatter of the data appears less than that for the tumble box. However, the entire group of data indicate an expected range of data from about one-half to two times the mean values. The comparison of results for the three films indicated in Figure 11 shows that results for the films are similar. In view of the data scatter, no meaningful distinction between films is possible.

Comparison of Methods. A comparison and evaluation of the three methods for determining the slough resistance of clean packaging materials must necessarily be made on the basis of whether the measurement being made is meaningful as well as whether the technique and

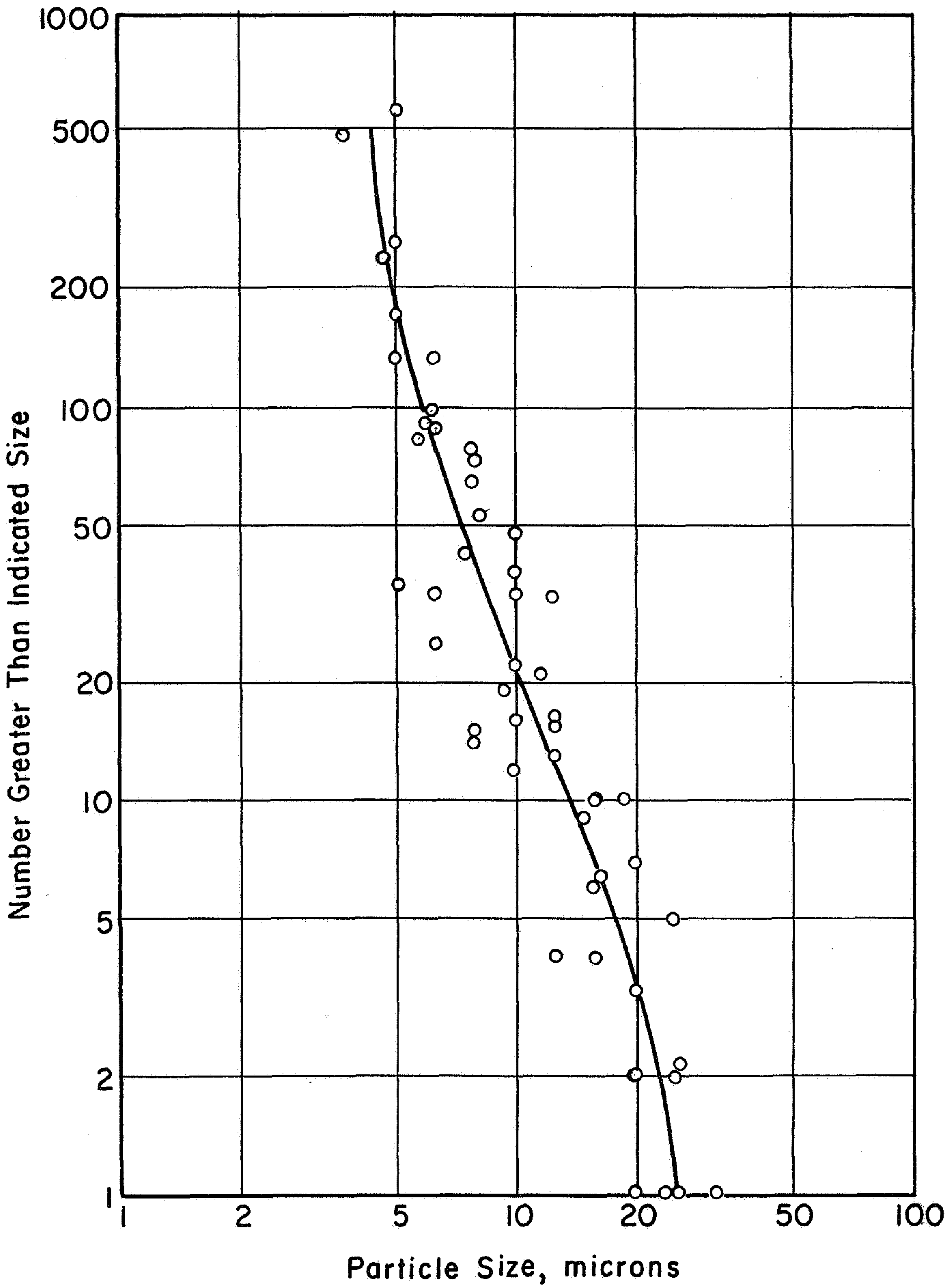


FIGURE 8. PARTICLES FORMED DURING TUMBLING OF POLYETHYLENE PACKAGED PART

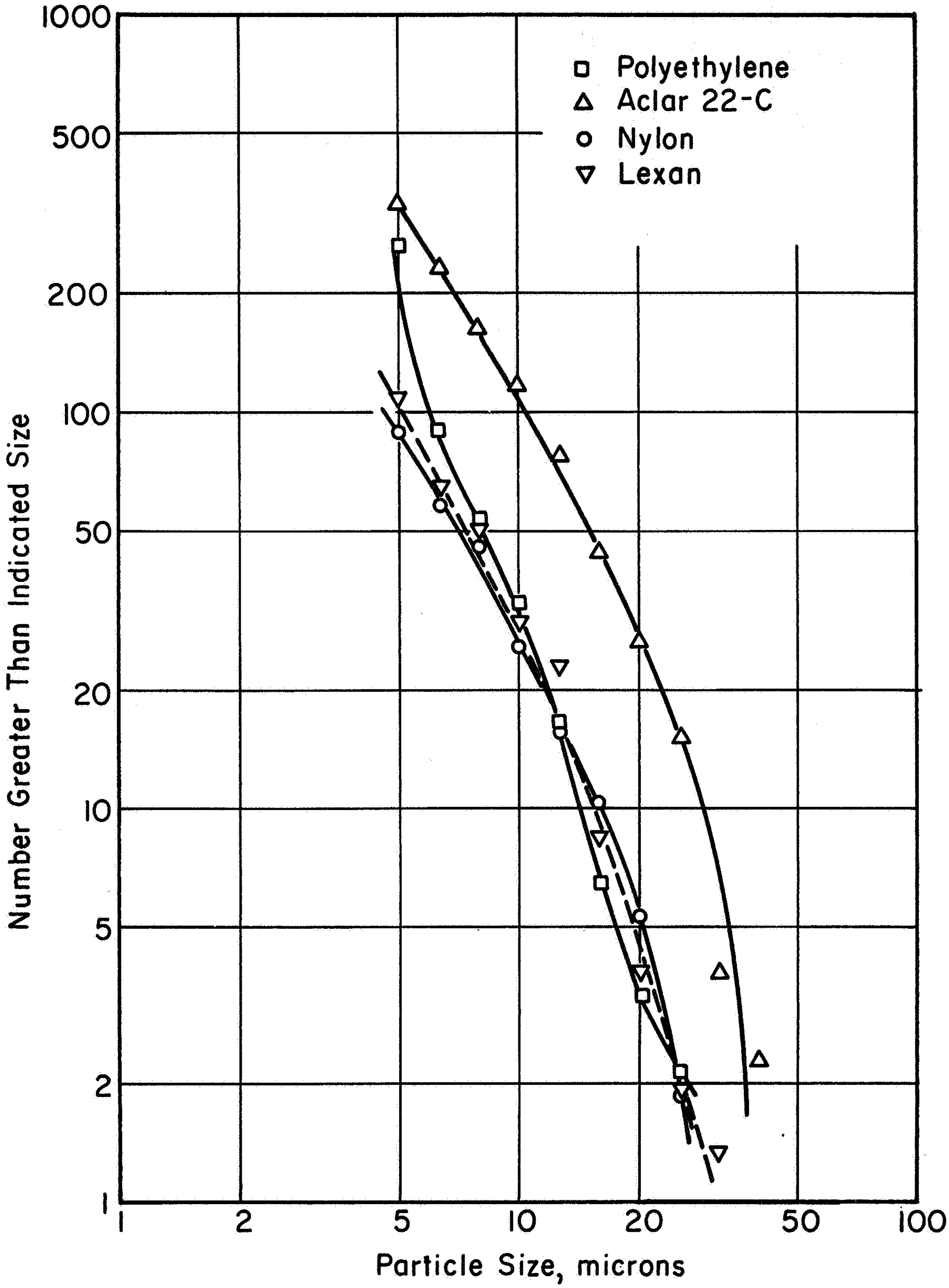


FIGURE 9. PARTICLES FORMED DURING TUMBLING OF PACKAGED PARTS

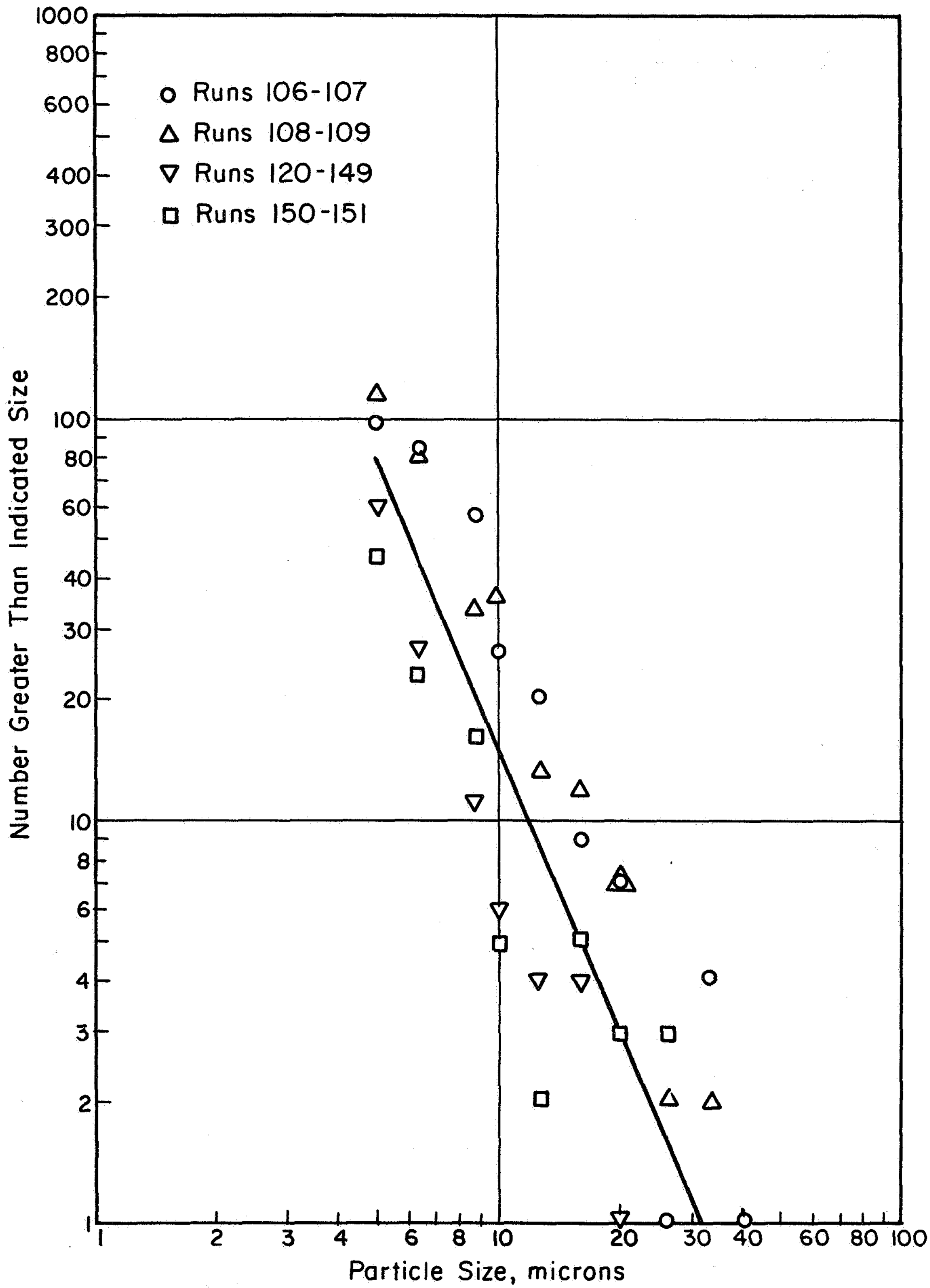


FIGURE 10. PARTICLES ABRADED FROM POLYETHYLENE WITH RECIPROCATING STYLUS

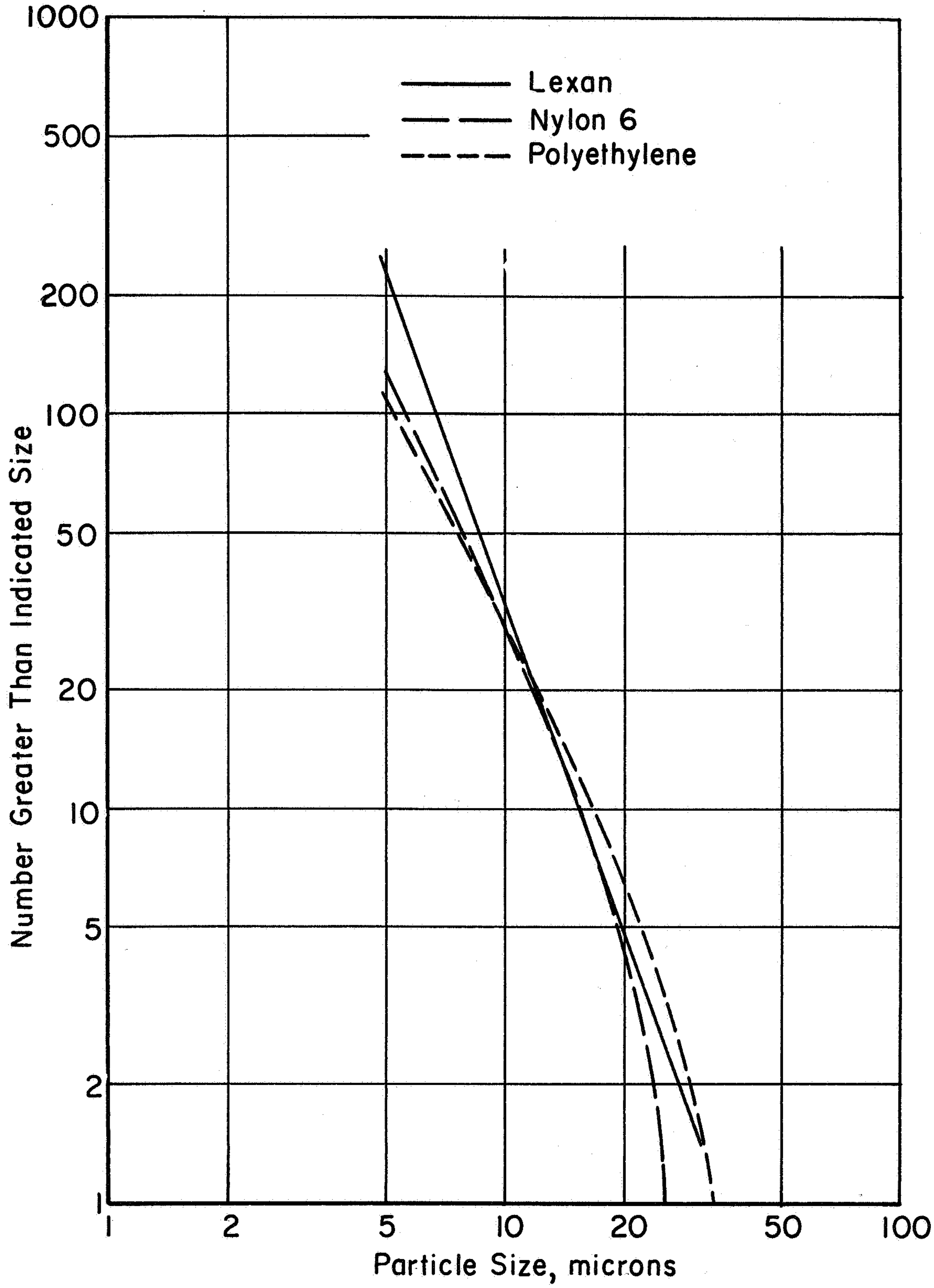


FIGURE 11. PARTICLES ABRADED FROM POLYETHYLENE, NYLON 6, AND LEXAN WITH RECIPROCATING STYLUS

measurements are satisfactory. The Taber Abraser method is based on a rather severe abrasion which affects the interior of the film as well as its surface. It is expected that sloughing is generally a surface phenomenon in a practical situation and, therefore, the suitability of a severe abrasive action to determine sloughing tendency is not known. Also, the measured quantity is weight loss rather than a more pertinent measurement of particles sloughed. However, there should be a direct relationship between weight loss and the number of sloughed particles. The Taber Abraser method does have certain advantages in that it is simple to perform, provides reproducible results, can be standardized easily, distinguishes between films, and places films with presently known performance characteristics in their expected order.

The tumble box and reciprocating stylus methods have the attractive feature of using particle count and size as the measured quantities. However, neither method provides much distinction between different films because of the scatter inherent in the data. The tumble box is the only method which incorporates an indication of both abrasion and flexing mechanisms of particle formation and the standardized tumbling of the package provides the closest simulation of a practical situation. In addition, use of the tumble box method could be easily standardized through tumble box and standard part designs and those persons interested in slough measurements are probably well equipped and capable of making the necessary particle counts.

Method Improvements. Since these studies of methods for determining sloughing tendencies of clean packaging materials were necessarily exploratory in nature, there are some improvements which now appear to be desirable. The tumble box method would probably be more suitable if a lighter standard part were used and if the tumbling were continued for considerably more revolutions. The sharp corners of the standard, 50-gram part tended to form holes in some of the packages if they were tumbled much more than 30 revolutions. Numerous improvements in the reciprocating stylus method could be made to facilitate performance of the experiments, with the major change being an enlargement of the equipment.

The tumble box method provided interesting information concerning some other aspects of clean packaging. Perhaps the most salient point is the strong dependence of sloughing and maintenance of package integrity on the tightness of the package. As more care was taken to expel as much air from the bag as possible, prior to the final sealing operation, it was found that there was less part movement, less sloughing, and less chance that the integrity of the package would be violated. It is expected that the large amount of scatter in the tumble box results are attributable to differences in air expulsion before sealing. It appears that this may be as important as the choice of clean packaging material in reducing the amount of sloughing.

Based on the experimental studies reported above, the following conclusions can be made:

(1) The Taber Abraser has not been shown conclusively to be a measure of sloughing. However, abrasion is an indication of slough tendency. Thus, the Taber instrument is a useful tool in classifying materials.

(2) The tumble box and reciprocating stylus methods in their current state of development cannot distinguish satisfactorily between variations in sloughing tendencies for different films.

(3) The tumble box method seems to be most representative of sloughing in practical situations and, with some suitable revisions, this method could probably be improved significantly.

(4) The tightness of the package is probably as important as the packaging film in determining the extent to which sloughing occurs within the package during tumbling.

Results

The measurements of abrasion resistance for 25 films evaluated with the Taber Abraser method are presented in Figures 7, 12, 13, and 14. These films are identified as to type and source in Table B-1 in Appendix B.

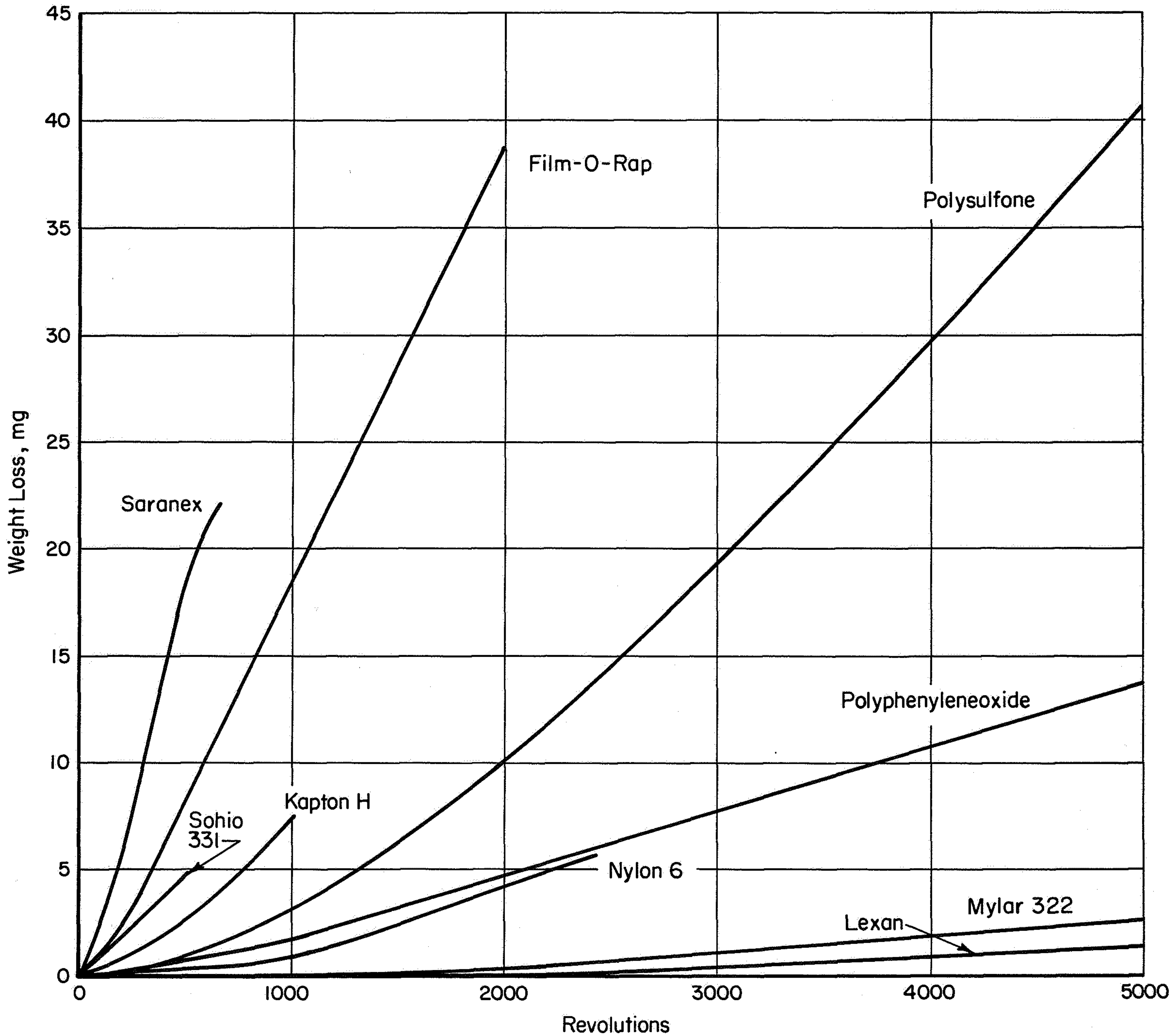


FIGURE 12. AROMATIC RING POLYMERS AND OTHERS - ABRASION WEIGHT LOSS

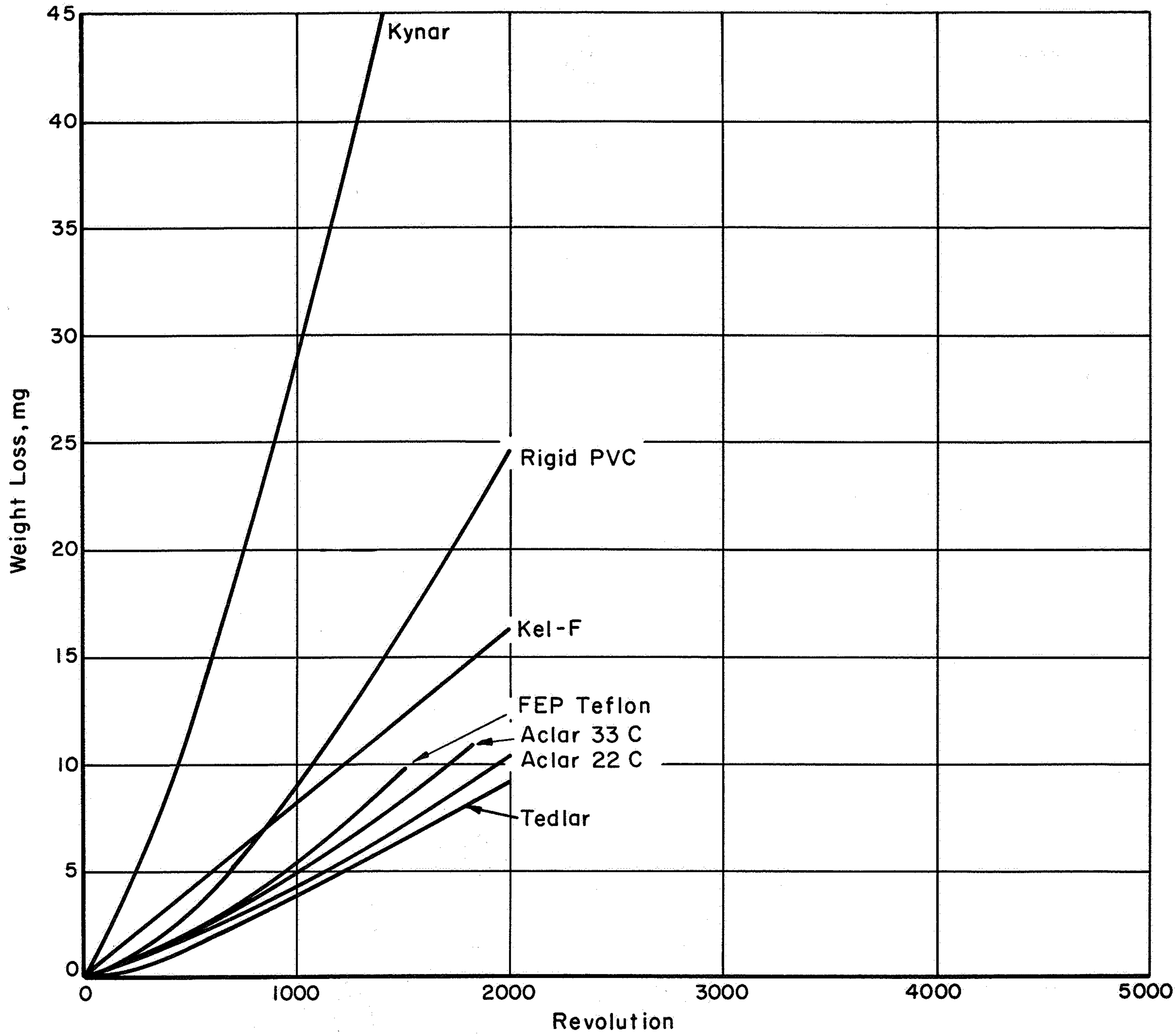


FIGURE 13. OLEFIN AND COPOLYMER FILMS - ABRASION WEIGHT LOSS

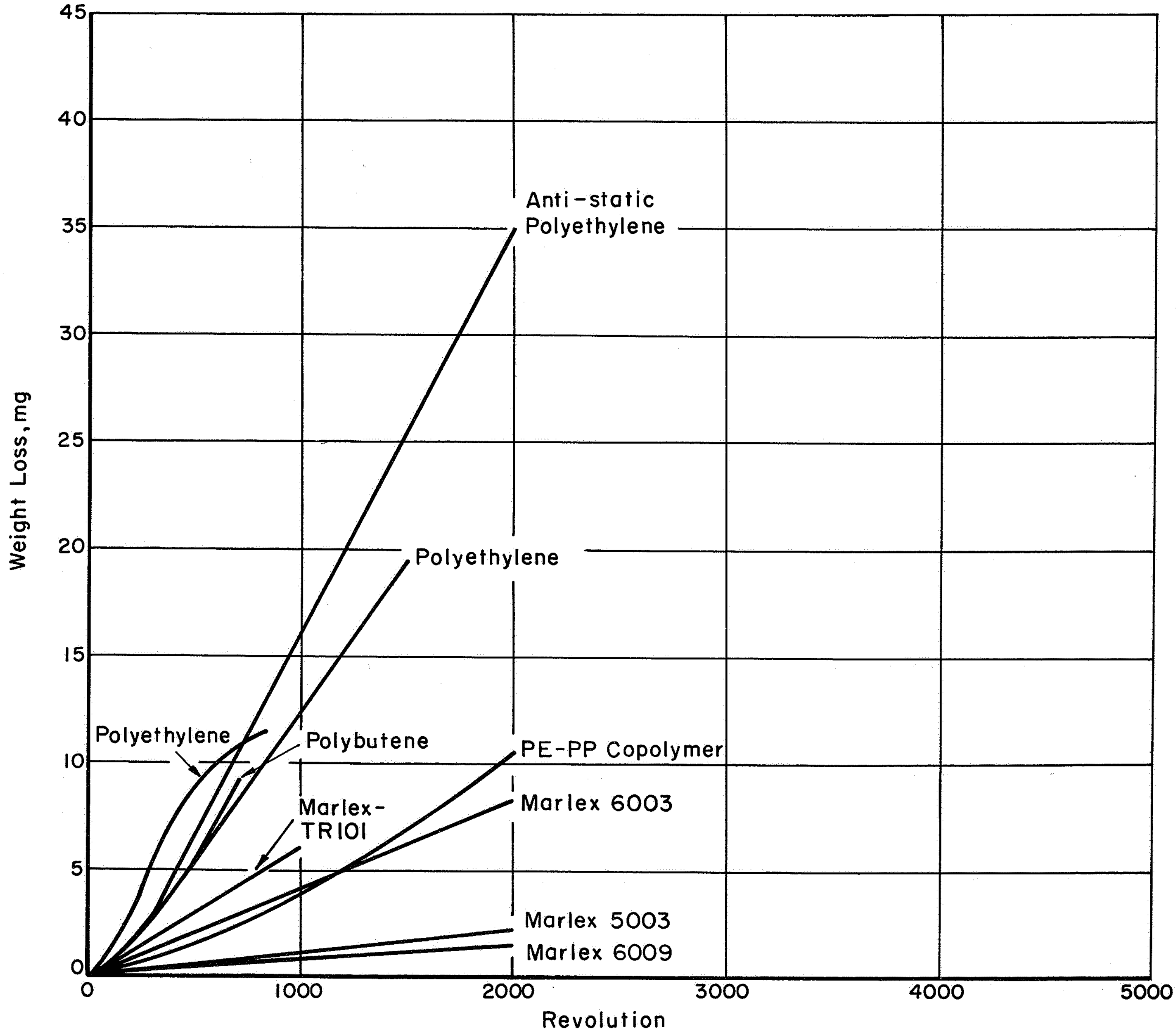


FIGURE 14. HALOGEN-CONTAINING POLYMERS - ABRASION WEIGHT LOSS

Figure 7 shows the results, with datum points, for three of the more commonly used materials plus Lexan which was found to be most abrasion-resistant of the films tested. All of the films are presented without datum points in Figures 12, 13, and 14. Detailed data for films evaluated on the Taber Abraser are presented in Tables B-2 through B-9 in Appendix B.

Abrasion measurements were carried out for as many revolutions of the Taber Abraser as was possible and this was usually determined to be the point at which the film developed a hole or was torn. The results indicate, as would be expected, that the maximum possible number of revolutions is dependent on film thickness as well as abrasion resistance. For comparative purposes, the weight loss for each film at both 1000 and 2000 revolutions was used as a basis for comparison. The attached Table 1 shows the comparative results for all of the films tested. It should be noted that some of the values are extrapolations. It can be seen that there are wide variations in weight loss among the films, indicating large differences in abrasion resistance.

The measurements of slough resistance were made using the tumble box method and the results are presented for the individual films in Figures 8, 15, 16, and 17. The results are presented as the number of particles sloughed as a function of particle size for 30 revolutions of the tumble box. A comparison of the four films is shown in Figure 9.

The reciprocating stylus method for abrading films is a less severe one than is the Taber Abraser technique. Figures 10, 18, and 19 show the results obtained for three films in terms of the number of particles sloughed as a function of size per 100 cycles of the stylus. A comparison of the three films is given in Figure 11.

The results for the tumble box and reciprocating stylus methods are not conclusive. As was indicated in earlier sections concerned with evaluation of these methods, the scatter in the data is such that differences among the films are indistinguishable. In contrast, the Taber Abraser results show significant differences among films.

The validity of an abrasion measurement to completely characterize a sloughing tendency is not known. However, abrasion of a

TABLE 1. ABRASION RESISTANCE OF SELECTED FILMS AS DETERMINED WITH THE TABER ABRASER[a]

Film Identification	Weight Loss, mg	
	At End of 1000 Rev.	At End of 2000 Rev.
Lexan (polycarbonate)	0	0.1
Mylar 322 (polyester	0	0.5
Marlex 5003 (high-density polyethylene)	0.9	2.3
Marlex 6009 (high-density polyethylene)	1.0	1.5
Nylon 6 (polyamide)	1.0	4.0
Polyphenylene Oxide	1.7	4.3
Polysulfone	3.5	9.5
Marlex 6003 (high-density polyethylene)	4.0	8.1
Tedlar (polyvinyl fluoride)	4.0	9.2
Dow PZ2003.00 (polyethylene-polypropylene copolymer)	4.0	11
Aclar 22C (polyfluorohalocarbon)	4.5	10.4[b]
Aclar 33C (polyfluorohalocarbon)	5.1	12
FEP Teflon (fluorinated ethylene-propylene copolymer)	5.4	14[b]
Marlex TR101 (high-density polyethylene)	6.1	12[b]
Kapton H (polyimide)	7.5	15[b]
Kel-F (polychlorotrifluoroethylene)	8	16
Genotherm U.S. 200 (rigid polyvinyl chloride)	9	24
Sohio 331 (polyacrylonitrile-based film)	9.6[b]	19[b]
Polypropylene	12[b]	15[b]
Polyethylene (low density)	12	27[b]
Polybutene	14[b]	30[b]
Antistatic Polyethylene	16	35[b]
Film-O-Rap (polyethylene-Mylar-polyethylene-Aclar laminate, polyethylene side)	18	39[b]
Kynar (polyvinylidene fluoride)	29	64[b]
Dow PZ5527.05, Saranex side (vinylidene chloride copolymer)	35[b]	72[b]

(a) No. CS-17 wheels, 1000 g/wheel.

(b) Estimated by extrapolation of data.

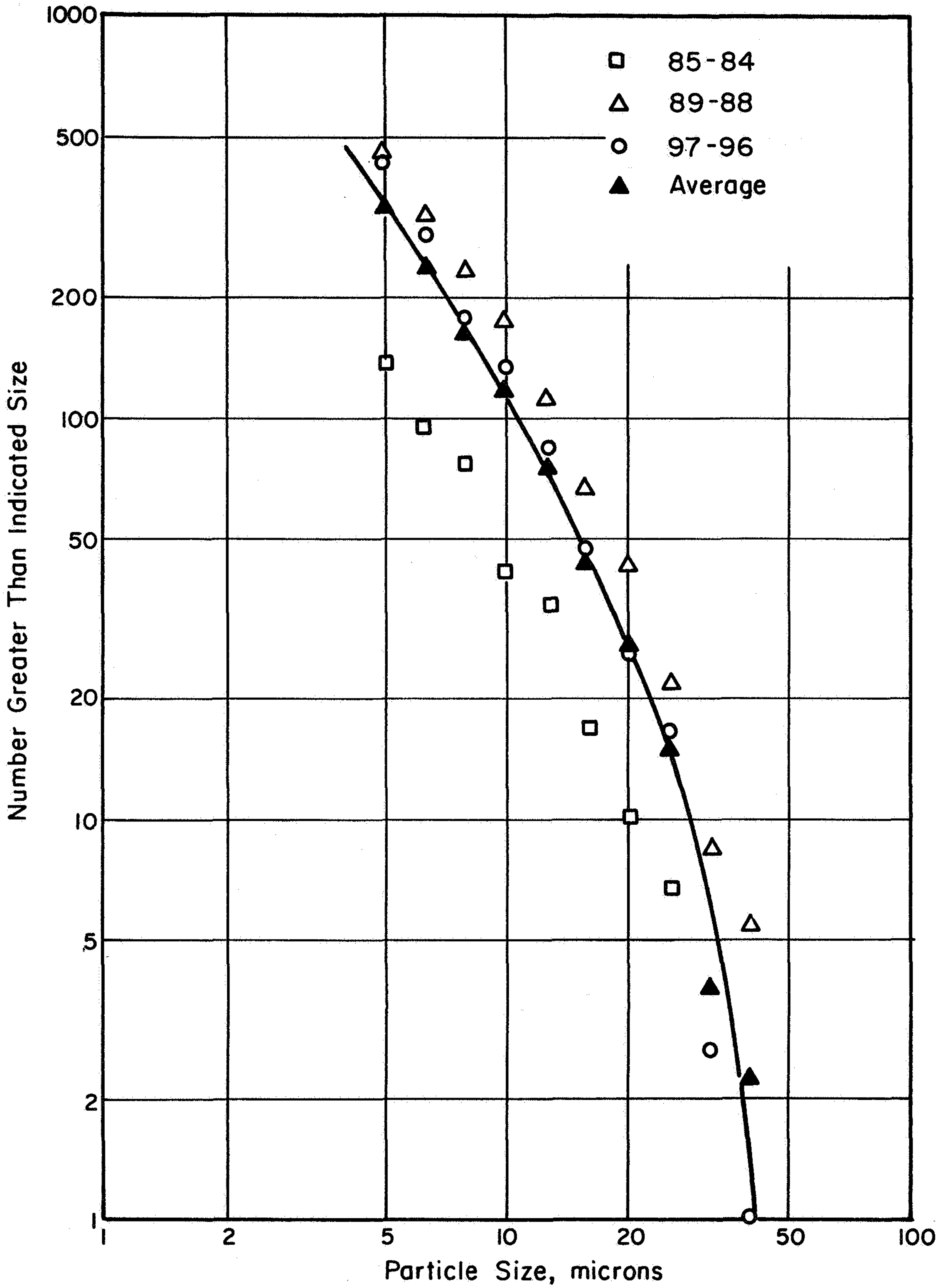


FIGURE 15. PARTICLES FORMED DURING TUMBLING OF ACLAR 22C PACKAGED PART

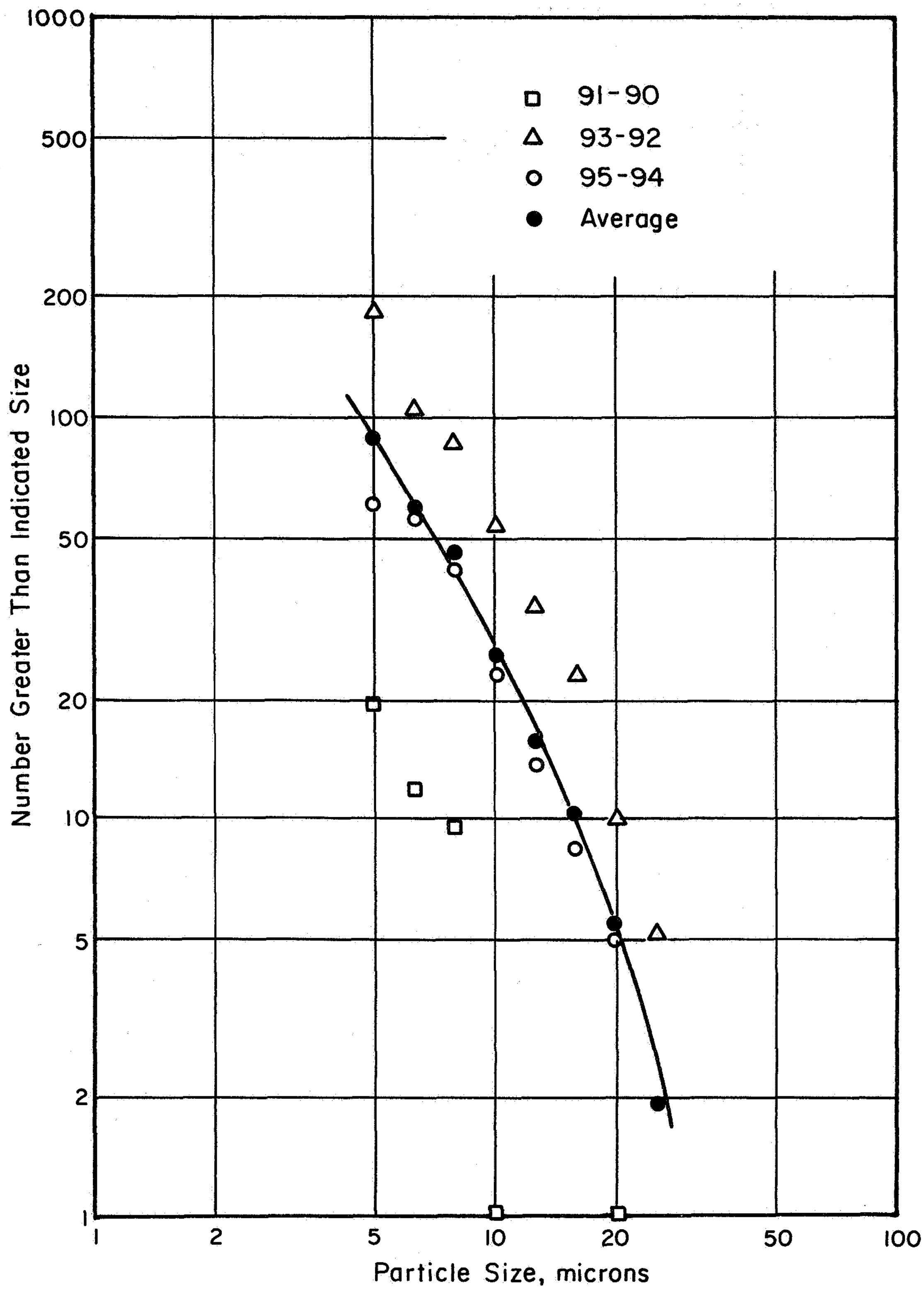


FIGURE 16. PARTICLES FORMED DURING TUMBLING
OF NYLON 6 PACKAGED PART

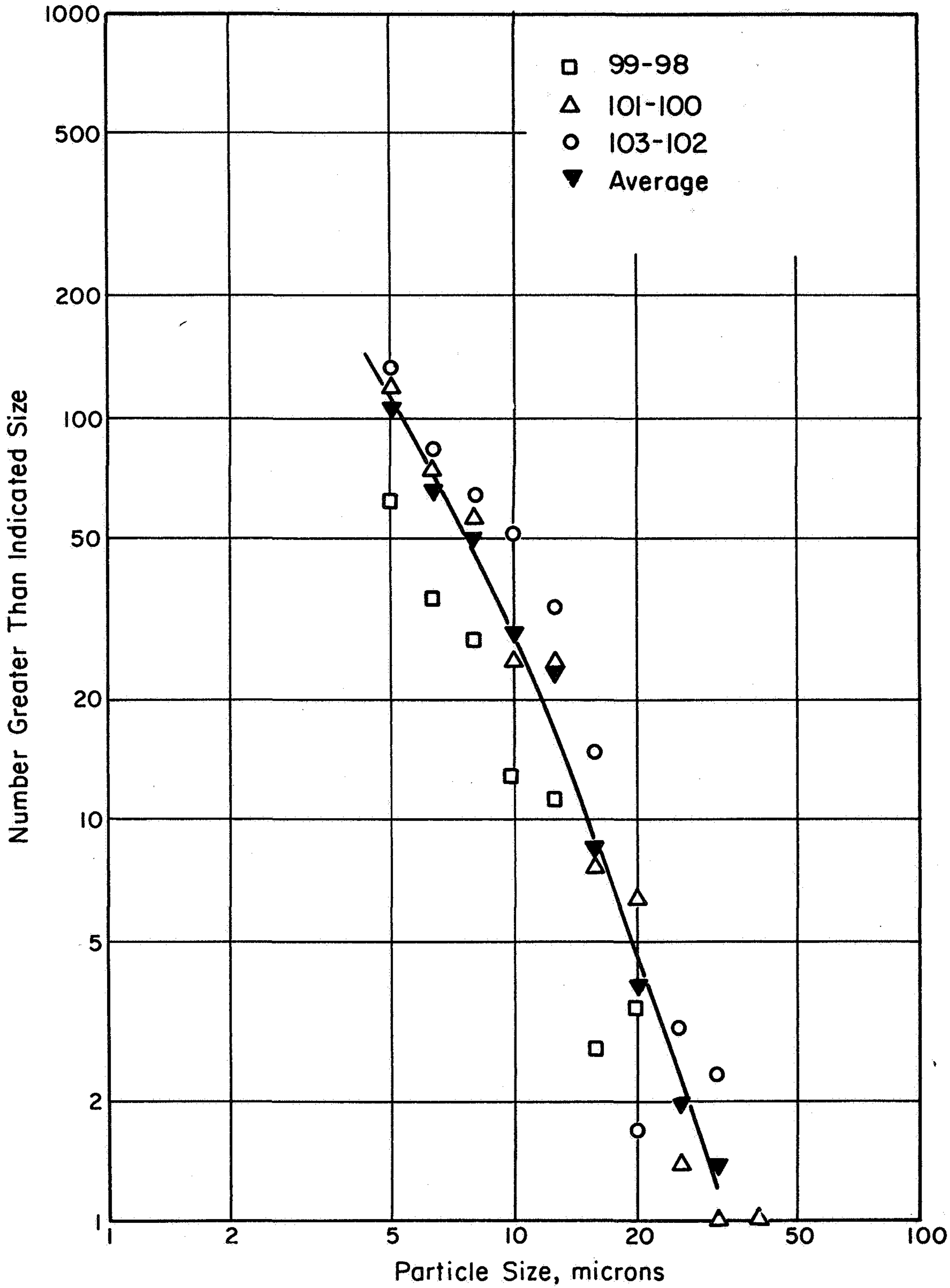


FIGURE 17. PARTICLES FORMED DURING TUMBLING OF LEXAN PACKAGED PART

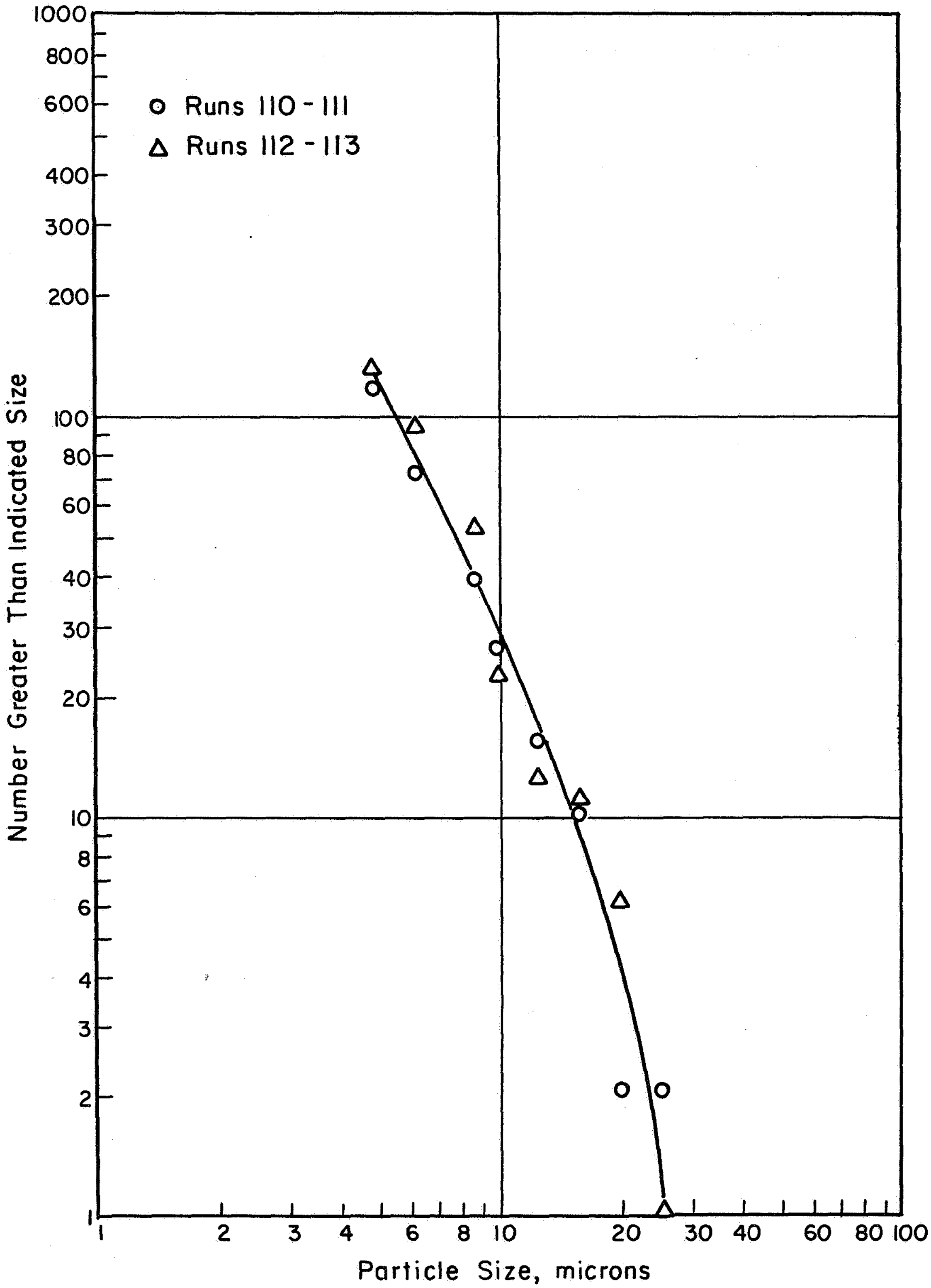


FIGURE 18. PARTICLES ABRADED FROM NYLON 6
WITH RECIPROCATING STYLUS

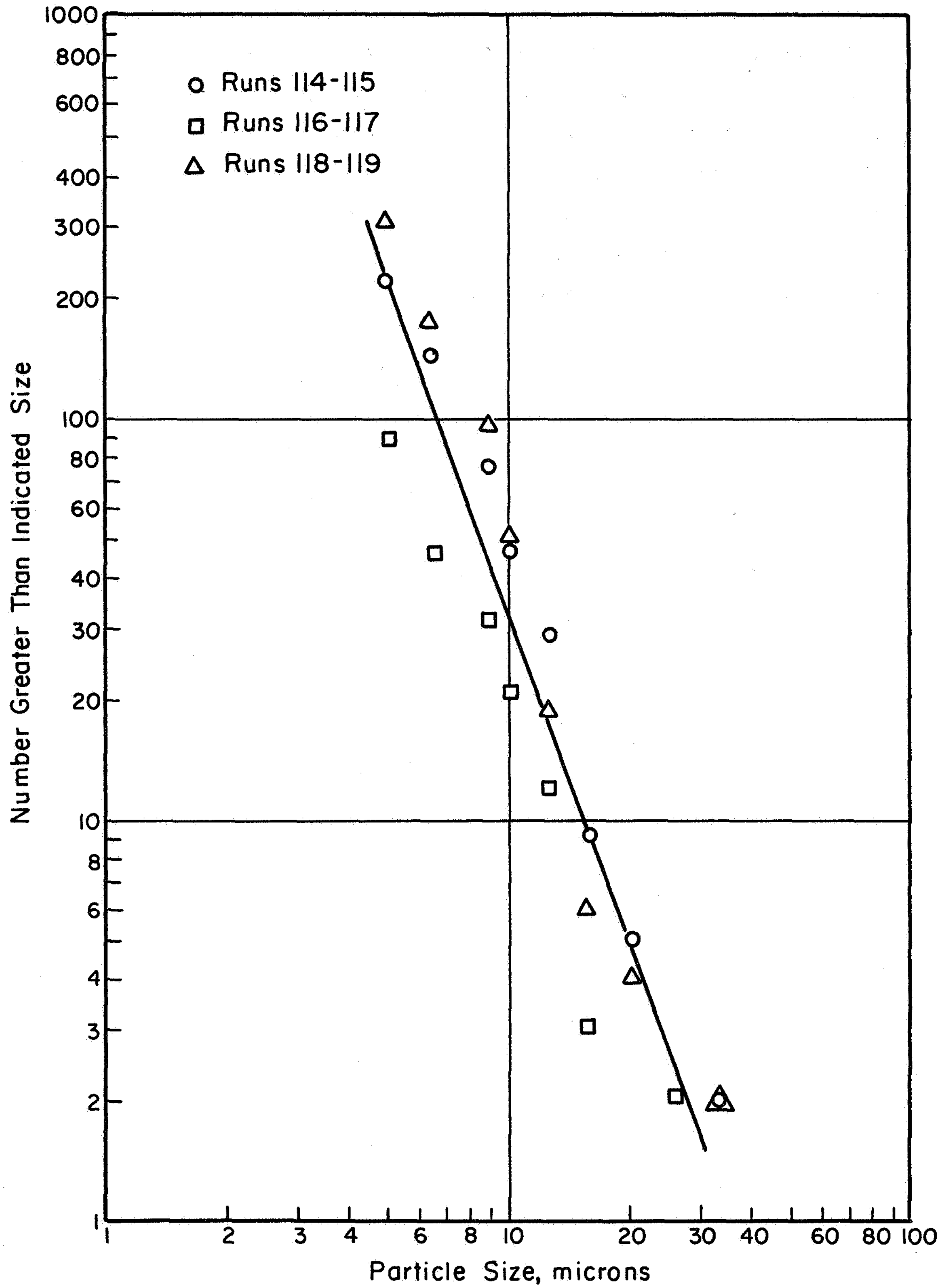


FIGURE 19. PARTICLES ABRADED FROM LEXAN WITH RECIPROCATING STYLUS

film can be an important contributing factor. Therefore, it is suggested that the Taber Abraser Method for measuring abrasion characteristics be used until a procedure such as the tumble box method which provides an action more representative of that causing sloughing can be perfected. The Taber Abraser technique appears to be an obvious choice at this time. Thus, the information provided in Table 1 can be used as a tentative guide in selecting clean packaging materials.

LOX Compatibility

The number of LOX-compatible materials is extremely limited and almost all of these are either fluoro- or chlorofluoroethylene polymers in which virtually all of the hydrogen has been replaced by one of these halogens. For example, the LOX-compatible films listed in NASA Report TMX-985, August, 1964, are shown below:

Producer	Producer's Designation	Polymer Type	Film Thickness, mils
3M	Kel-F, 8105	Chlorotrifluoroethylene	5
3M	Kel-F, KX202	Ditto	2
3M	Kel-F, KX8110	"	10
3M	Kel-F, 8210	"	10
3M	Kel-F, 8205	"	5
Du Pont	Teflon	Tetrafluoroethylene	2
Ditto	Ditto	Ditto	6
"	Teflon FEP, 554	Fluorinated ethylene propylene	1
"	Ditto	Ditto	5
"	"	"	10

The materials previously mentioned must be prepared for LOX service by cleaning and processing by applicable MSFC Standards. The films listed below must be similarly approved but, in addition, it is generally required that each manufacturer's lot must be individually tested and found acceptable.

Producer	Producer's Designation	Polymer Type	Film Thickness, mils
Allied Chemical	Aclar Type 22A	Fluorochloropolymer	5
Ditto	Ditto	Ditto	2
"	Aclar Type 191	"	2
Du Pont	H Film[(a)]	Polyimide	2
Ditto	ML Film	Ditto	2

(a) Now identified as Kapton.

Data needed for approval of Pennsalt's Kynar (polyvinylidene fluoride) are not complete as yet. Therefore, the material has not been approved for use in LOX environments. According to NASA Report TMX-53052, Kynar has less sensitivity to LOX than most films with the exception of Kel-F, Aclar, and Teflon. Tedlar (Du Pont polyvinyl fluoride) has been found to be unacceptable. This is not surprising since only one of four hydrogens of the monomeric unit has been replaced by fluorine. It is interesting to note that the one nonhalo carbon of the group--the polyimide--contains a relatively small amount of hydrogen based on its molecular size. However, most of these hydrogens are aromatic in nature and are thus less reactive than aliphatic hydrogens.

Moisture and Gas Barrier Characteristics

Of the four films most widely used in the clean packaging field--Aclar 33C, low-density polyethylene, Saran (a polyvinylidene chloride copolymer), and Nylon 6--all but the last have relatively good water vapor barrier characteristics. Two of these materials--Aclar and Saran--are also effective barriers for gases, particularly oxygen. In fact, from the standpoint of gas barrier characteristics, these are among the best of all films. Polymers other than these with good to relatively good gas barrier properties include polyvinyl fluoride (Tedlar), polyester (Mylar), polyvinylidene fluoride (Kynar), phenoxy, and polyacrylonitrile.

With respect to water barrier characteristics, Aclar and Saran again represent the better two of the four commonly used materials. Of the other films examined, those having good water barrier characteristics include polychlorotrifluoroethylene (Kel-F), high-density polyethylene, polybutene, polyacrylonitrile, a fluorinated ethylene-propylene copolymer (FEP Teflon), and polyvinylidene fluoride (Kynar). Table 2 lists the properties of these and other films examined during the program. Obviously, both gas and moisture permeability characteristics of any otherwise desirable material can be upgraded by lamination or coating with a polymer with good barrier properties such as polyvinylidene chloride.

Sealability

Virtually all thermoplastic films are sealable to at least some degree. Exceptions to this, of course, are the highly oriented crystalline films such as Mylar which distort and lose strength on heating to fusion. Those films which are not readily sealable, as in the case of barrier properties, can be improved either by lamination with a sealable material such as polyethylene, by application of a coating such as one based on polyvinylidene chloride, or by the use of tapes or zipper closures.

Of the 15 or so types of films evaluated for possible clean packaging of space hardware, none has any appreciable advantage over the present films in sealability. There is a fairly wide range of fusion temperatures of the materials considered, starting at about 200 and going to about 700 F. The polyimide film is at the extreme upper end of the scale, but this is not truly a heat-sealing type. Table 2 also lists heat sealing ranges of the films examined.

Abrasion resistance or the tendency of packaging materials to slough remains as the factor of prime importance in the solution of clean packaging problems. Of the three methods investigated for measuring slough characteristics of polymeric films--Taber Abraser, tumble box, and reciprocating stylus--the first of these has produced the most consistent results. Polycarbonate is the most attractive of the films examined from the standpoint of abrasion loss.

TABLE 2. FILM PROPERTIES

Film Type	Examples	Heat Sealing Range, F	Specific Gravity	Tensile Strength, psi	Grease and Oil Resistance	Water Resistance	Resistance to Heat, F	Water Vapor Permeability(a)	Permeability to Gases(b)	References(c)
Fluoro-Halocarbon	Kel-F FEP-Teflon Aclar 33C	350-700	2.2	2,000-10,000	E	E	350-400	0.03-0.4	For FEP-Teflon: H_2=2,340 O_2=350 CO_2=325 N_2=133 For Alcar 33C: O_2=7 CO_2=16	DuPont trade literature, Bulletin T-1B Allied trade literature, Bulletin ADT2-365 Modern Plastics Encyclopedia 1967
Polyamide	Nylon-6	400-475	1.129	9,000-18,000	E	E-P	200-375	5.4-20	O_2=2.6 H_2=90-250 CO_2=9.7-45 N_2=0.9-6	Modern Plastics Encyclopedia 1967
Polyethylene (low density)	Marlex 1712	250-400	0.910-0.925	1,250-2,500	P	E	180-200	1.3	CO_2=2700 O_2=500 N_2=180	Modern Plastics Encyclopedia 1967
Polyethylene (high density)	Marlex 6003	275-400	0.941-0.965	2,400-6,100	G-E	E	250	0.3	CO_2=580 O_2=185 N_2=42	Modern Plastics Encyclopedia 1967
Polyvinyl fluoride	Tedlar	400-425	1.38	7,000-18,000	E	E	220-250	3.24	CO_2=15 H_2=58 N_2=0.25 O_2=3	Modern Plastics Encylopedia 1967
Vinylidene Chloride – Vinyl chloride copolymer	Saran	240-315	1.57-1.68	8,000-20,000	E-G	E	150-200	0.25-0.76	CO_2=12 (50% R.H.) O_2=2.4 (50% R.H.)	Modern Plastics Encyclopedia 1967
Polycarbonate	Lexan	400-430	1.20	8,400-8,800	G	G	270	11.0	O_2=900-950 CO_2=1500-1700 N_2=160-200	Modern Plastics Encyclopedia 1967
Polyimide	Kapton H	non-sealing	1.42	25,000	E	E	750	5.4	CO_2=45 H_2=250 N_2=6 O_2=25	Modern Plastics Encyclopedia 1967
Polyester (PE-terephthalate)	Mylar 322	325-450	1.23-1.395	25,000-30,000	E	E	300	1.7-1.8	CO_2=16 O_2=3.5-6 H_2=100 N_2=0.7-1.0	Modern Plastics Encyclopedia 1967
Polyvinylidene fluoride	Kynar	575-700	1.75-1.78	5,500-7,400	E	E	300	0.06-0.1(b)	O_2=4.0 x 10^{-3} N_2=9.2 x 10^{-4} CO_2=1.5 x 10^{-3}	Pennsalt Trade Literature Modern Plastics Encyclopedia 1967
Phenoxy	–	CA 200 250	1 182-1.324	9,000-9,500	E	E	550	3.5	O_2=5-8 CO_2=15-30	Modern Plastics Encyclopedia 1966
Polybutene	–	CA 275	0.912	2,800-3,300	G	G	190	0.6	O_2=2500 CO_2=5880	Grace Trade Literature
Polypropylene oxide	–	CA 380-400	1.06	11,600	–	–	345-375	–	–	General Electric Trade Literature
Polysulfone	–	CA 350	1.24	10,200	G	G	345	–	–	Literature obtained from Mobil Chemical Co.
Polyacrylonitrile	Sohio 331	–	1.17	20,000	E	G	482 50 psi	0.077	O_2=0.034 He=62.5 H_2=17.2 NH_3=0.054	Trade Literature from Sohio (SF331-12464)
Biaxially oriented polyethylene	Clysar 300 EH30	230-400	0.923	2,300-2,500	–	G-E	–	–	–	Plastics World, October, 1966
Polyvinyl chloride	Reynolon 5155	305-370	1.30	12,500	E	G	90	4.0	O_2=75 N_2=20 CO_2=500	Trade Literature from Reynolds Metals Co. Bulletin No. 407-1-4(10M465)
Rubber Hydrochloride	Snug-Pak	225-250	1.11	3,500-5,000	E	G	–	1.75	CO_2=288-13,500 O_2=38-2,250	Modern Plastics Encyclopedia, 1967 Snug Pack Literature from Tee-Pak

NOTES: (a) g/24 hr/100 sq. in./mil at 25 C. (b) cc/100 sq. in./mil./24 hr/atmos./at 25 C. (c) Values corrected to be consistent.

LOX-compatible materials are still limited to presently known fluorine-containing polymers and no really new films with both good gas and water vapor barrier characteristics have been developed. The polyimides may provide a degree of LOX compatibility coupled with relatively high impermeability to gases. However, the moisture barrier property of these films is not exceptionally good. Virtually all of the thermoplastic materials are sealable. Improvements in this property as well as barrier characteristics may be imparted to slough-resistant or LOX-compatible films by lamination or coating.

PHASE III. TEST AND EVALUATION

New Standards

Recommendations designed as guides in the preparation of any new specifications or standards were outlined in the Phase I Section of this report. In addition, comments which are believed to be in order relative to sloughing of packaging materials are presented here.

There exists a need for a standard procedure with which the sloughing tendencies of clean packaging materials may be measured. Such a standard procedure or method should be based on combined abrasive and flexing actions upon the films. This seems to be best represented by the previously described tumble box method. However, until this method is improved, it is suggested that the ASTM standard method for measuring abrasion resistance (ASTM Standard Test Method D-1175-64T) be used to indicate slough resistance.

Improvement of the tumble box technique seems to be possible through several modifications of the equipment and procedure. First, a lighter standard part should be used. This could be readily accomplished by drilling several more holes through the part in addition to the two already there (see Figure 2, Phase II) or by reducing the size of the part. In any case, the weight should probably be of the order of 20 grams rather than its present weight of 50 grams. Another improvement in the technique would be to tumble the parts for longer periods of time. This should tend to reduce the differences between slough data for packages caused by the random tumbling action. Also, continued tumbling will cause more particles to be sloughed and counted, giving a higher statistical credibility to each datum on particle count. Perhaps the most important improvement would be to standardize the means for expelling air from the package prior to sealing. Air expulsion or tightness of the package was found to be a major factor in determining the extent of sloughing. A template placed over the package and part during sealing might be used to standardize this factor and reduce scatter in the sloughing data. These changes should lead toward developing the tumble box method into a more suitable means for evaluating sloughing tendencies of clean packaging materials.

Materials

Reduction of the tendency of a package to generate particulate contamination seems to be the most obvious single goal of major importance to pursue in improving the packaging of space hardware. As has been pointed out, packaging materials with other desirable attributes presently exist but good abrasion or slough resistance cannot be achieved by conventional means such as coating or lamination. This is a characteristic which must be inherent. Nylon 6, from the standpoint of abrasion resistance, is the best of the materials presently used. Yet nylon, which is also a relatively good gas barrier, is somewhat water-sensitive and is quite permeable to water vapor. Of the films presently used in various space packaging applications, the fluorochloropolymers such as the Aclars and certain copolymers of vinylidene chloride (Saran) offer much better and really quite adequate barrier properties.

The current study has shown that films made from polycarbonate (Lexan) are most attractive from the standpoint of abrasion loss. These heat-sealable films in comparable thicknesses are several times better than nylon. Mylar, although not readily sealable without disorientation and weakening, is also considerably more abrasion-resistant than nylon. Like nylon, however, it is only a fair gas barrier and offers relatively poor protection from water vapor. Of the existing films, certain high-density polyethylenes offer probably the best obtainable combination of properties. They are roughly equivalent to nylon in abrasion resistance as measured by the Taber Abraser technique. In addition, they offer much better water vapor protection coupled with ready heat sealability. Gas barrier properties--in some instances less important--are relatively poor. The best of the films from the standpoint of water and gas barrier properties are the vinylidene chloride copolymer, Saran; the polyfluorochloropolymers such as Aclar 33C, Kel-F, and FEP Teflon; and polyvinylidene fluoride, Kynar.

Table 3 sums up the more important characteristics of the previously mentioned films. In addition to the properties shown, all of these polymers offer excellent resistance to oils and greases. If the generation of particulate matter must be kept to a minimum, one of the more abrasion-resistant films can be used as an inner wrap. This, together with an overwrap of one of the barrier films, would provide both cleanliness and protection.

Laminates and Coatings

Previous sections of the report have mentioned the use of composites of materials as a means of obtaining a desired combination of properties. Although overwraps may perform similarly, lamination of two or more materials or the application of a coating (which is really a solution or hot-melt-applied laminate) will often provide a means of obtaining a single sheet which will perform well as a unit. Frequently, laminates are also coated to achieve a particular characteristic such as heat sealability or impermeability to moisture. Slough resistance is the one property believed at this time to be difficult if not impossible to obtain through coating methods since most solution-coated materials--particularly the conventional thermoplastic ones--are relatively soft. However, polycarbonate conceivably might be applied as a thin coating to a barrier film or even to a foil as a means of developing an improved degree of abrasion resistance.

Materials such as the Sarans, of course, are also frequently applied to more permeable substrates to provide protection. These coatings, which are also readily heat-sealable, slough readily and should not be used adjacent to a clean part. One excellent possibility as combined materials might be an extrusion coating of one of the preferred high-density polyethylenes on a base sheet of polycarbonate. This would provide a material with both moisture protection and good resistance to sloughing. The latter would be possible no matter which side was adjacent to the clean part. Gas barrier characteristics could be added by coating or overwrapping with a Saran material. Other possible laminates with

TABLE 3. FILM PROPERTIES

Film	Abrasion Loss mg. at end of 2000 rev.[a]	Water Vapor Permeability[b]	Permeability to Gases[c]			
			CO_2	H_2	N_2	O_2
Polyethylene (low density)	27	1.3	2,700	---	180	500
Polyethylene (high density) Marlex 5003 or 6009	1.5-2.3	0.3	580	---	42	185
Aclar 33c	12	0.03-0.055	16-40	220-330	2.5	7-15
Saran	72	0.3-0.8	12	---	---	2.4
Nylon-6	4	5.4-20	9.7-45	90-250	0.9-6	2.6
Lexan	0.1	11.0	1500-1700	1600	160-200	900-950
Mylar 322	0.5	1.7-1.8	16	100	0.7-1.0	3.5-6.0
Kel-F	16	0.03-0.55	16-40	220-330	2.5	7-15
FEP Teflon	14	0.4	325	2,340	133	350
Kynar	64	0.06-0.1	1.5×10^{-3}	---	9.2×10^{-4}	4.0×10^{-3}

(a) Using Taber Abraser under conditions described in this report.
(b) g/24 hr/100 sq.in./mil at 25 C.
(c) cc/24 hr/100 sq.in./mil/atmos. at 25 C.

obvious advantages are combinations of Mylar and high-density polyethylene, Lexan with Kynar, Lexan with Kel-F, and Lexan with Saran. With Lexan or Mylar adjacent to the hardware, both low slough characteristics and good barrier properties could be achieved. The converter could readily identify one or both sides, thus minimizing the possibility of using the structure wrong side out.

Of the several commercial and experimental laminate films examined, two are of potential interest. One from Dow, and identified as 4-mil PX 2003.00, is a structure composed of polyethylene-polypropylene copolymer/Saran/polyethylene-polypropylene copolymer. The other is Film-O-Rap 7750, a 0.75-mil Aclar/0.75-mil polyethylene/0.5-mil Mylar/2.5-mil polyethylene. The first of these exhibits better than average abrasion resistance and good barrier properties while the latter may be of interest as a LOX-compatible wrap. Experimental confirmation of the latter, of course, would be necessary. The laminate is well marked so that identification of the Aclar side should present no problem.

Reduced Abrasion

Shrink packaging was investigated as a possible means for reducing abrasion of a sealed bag or wrapping material. This technique takes advantage of a built-in shrinkage factor in the film. This is usually achieved by biaxially orienting a particular type of sheet at an elevated temperature and then cooling it in a restrained condition. On reheating, the plastic "memory" of the sheet acts to return it to a size approaching its preoriented dimensions. It was believed that a close fitting wrap might help to minimize any abrasive action caused by motion of the package in handling or shipping. If a shrink package could also be made from an abrasion-resistant material, it was thought also that a dual advantage might be obtained.

Three types of shrinkable films were evaluated in a method which involved a comparison of the degree of particulate matter generated when the films were used in a shrink versus a nonshrink package. The films investigated included a 1.5-mil plasticized polyvinyl chloride, a 1-mil oriented low-density polyethylene, and a 0.6-mil rubber hydrochloride. The packages were sealed bags containing the standard part described in the section of Phase II on packaged part tumbling and shown in Figure 2 in the same section. Two sets of bags were evaluated. One set was sealed and tumbled without preshrinking and a second set was sealed, shrunk with the aid of heat, and then tumbled.

In the procedure used, film was cut 2-1/2 by 3 inches and sealed so that three sides were closed. The bag was rinsed several times with Genesolv D to remove the major dirt. After rinsing off the machined part, it was placed in the bag. The bag and part were then rinsed three times with Genesolv D. A background count was obtained using 20 ml of Genesolv D to rinse out bag with part. This was filtered and the background count obtained by using a microscope and counting half of the filter. The particle counts were then doubled to obtain the final number-size distributions. The same procedure was used to obtain particle counts for the tumbled and the shrunk and tumbled bags. After obtaining the background count, the bag was sealed and tumbled 30 revolutions at a rate of 6 revolutions per minute. After tumbling, the bag was cut open and the standard part removed. The bag and part were rinsed with 30 ml of Genesolv D, which was then filtered and counted. To shrink the package, a heating gun was used allowing just enough heat to shrink the film snugly to the part. The tumbling action was the same as described above.

The results of these evaluations are shown in Table 4. With the films investigated by this technique, more particles were generated in all cases when the package had been shrunk and then tumbled than when tumbled without preshrinking. Apparently, the shrinking process or abrasion caused by the shrinking action is a significant factor in particle generation. The effect of heat may also enter into the cause for contamination. This would be particularly true in the case of the plasticized polyvinyl chloride where heat would tend to volatilize the plasticizer. The performance of this wrap was, in fact, poorest of the three. Lower molecular weight, more volatile components may also be present in the other two films and these might be affected by heat in the same manner as the plasticizer was.

TABLE 4. PARTICULATE EFFECT ON SHRINK VERSUS NON-SHRINK PACKAGING

Particle Size, Microns	Polyvinylchloride (1.5 mils)						Oriented Polyethylene (1 mil)						Rubber Hydrochloride (0.6 mil)					
	Tumbled			Shrunk & Tumbled			Tumbled			Shrunk & Tumbled			Tumbled			Shrunk & Tumbled		
	B	T	Diff.	B	S&T	Diff.	B	T	Diff.	B	S&T	Diff.	B	T	Diff.	B	S&T	Diff.
4.69- 9.38	92	186	94	76	526	450	106	132	26	266	330	64	6	28	22	98	126	28
9.38- 18.8	138	386	248	204	580	376	176	172	0	332	698	366	144	90	0	198	246	48
18.8 - 37.7	246	806	560	236	6054	5818	350	422	72	522	768	246	206	230	24	564	362	0
37.7 - 75.0	26	62	36	50	246	196	36	74	38	28	132	104	18	36	18	48	100	52
75.0 -150.0	14	14	0	6	66	60	6	20	14	6	60	54	2	22	20	10	36	26
150.0	6	4	0	4	34	30	4	50	46	0	34	34	2	8	6	4	10	6

Note: B = background count.
T = count after tumbling.
S&T = count after shrinking and tumbling.

Another approach to reducing abrasion in a package has been described by Gordon Walker(11). Walker mentions the use of a rigid plastic container to enclose filter elements. The package is designed so that the filter is suspended and the filtering medium does not touch any part of the enclosure. Thus, the part is protected from both contamination and from damage by handling. If a container of this type were made of an abrasion-resistant material such as polycarbonate, particle generation conceivably could be further reduced.

Reduced Corrosion

The corrosion-resistant packaging of hardware, if particulate matter were not a factor, is outlined in several methods of waterproof packaging in MIL-P-116E. Methods 1C-3, 1A-8, and 1C-6 are such examples. If particulate matter must be kept low, then a package system as described earlier must be used. Other methods of packaging as outlined in MIL-P-116E are Method 1A-2, in which a dip coat compound is used to make the package waterproof, and another type which uses both a desiccant and a humidity indicator and is outlined in Method 11b which describes the use of two barrier materials. If a floating barrier type package is needed, Method 1A-16 can be used and if a rigid container is needed, Method 11d can be used. The latter has a barrier wrap along with a desiccant and humidity indicator.

Cleanliness Level Measurement

The most generally accepted method of determining the number and size of particles rinsed from critical surfaces is to filter the rinse solution and to count and size the collected particles with a light microscope. Although this method is simple to understand and allows a direct visual examination of the particles of interest, it has several shortcomings. Specifically, with the microscope considerable time is required to count a filter sample and the counts are subjective in nature and highly dependent on operator performance.

Particles in rinse solutions may also be counted and sized with automatic instruments. Although procedures utilizing automatic particle counters are not as widely accepted or as standardized as microscopic procedures, automatic counters, in general, give results which are relatively more accurate and reproducible than microscope counts and which may be obtained in considerably less time. There are currently a number of automatic particle counters commercially available for which results have been compared with those obtained with a microscope on identical samples. A few automatic counters are discussed below with emphasis on how they operate, over what range of sizes they are applicable, and how results obtained using them compare with results using a microscope.

The HIAC automatic particle counter is a device by which particles are counted and sized as they pass through a beam of light. Specifically, the instrument flow system is such that fluid borne particles pass one by one through a critically sized light beam which is directed toward a photomultiplier tube. As each particle passes through the light beam, it is counted and sized according to the degree to which the light intensity is decreased. The particle size reported, then, is the diameter of a spherical particle having an equivalent projected area. Particles may be counted in either 4 or 5 increments over a number of different size ranges. Each size range over which the instrument may be used is controlled by interchangeable sensing elements called "microcells". The microcell used also determines the sample flow rate and the maximum particle concentration allowable to avoid the chance of more than one particle passing through the sensing zone at a time. The size range over which available microcells are applicable, the flow rate used, and the maximum particle concentration per ml of fluid are shown in Table 5. The HIAC counter is applicable for both batch samples and continuous in-line monitoring.

TABLE 5. OPERATING CHARACTERISTICS OF HIAC COUNTER FOR VARIOUS MICROCELLS

Size Range, microns	Sample Flow Rate, cc/min	Maximum Particle Concentration, per cc fluid
5-150	5	3400
10-300	300	850
20-500	800	300
35-1000	3000	75
85-2500	15000	12

An evaluation of the HIAC counter has been conducted by Romine and Gayle(12). Particle counts were obtained with the automatic counter and with a microscope for the size ranges 10 to 25, 25 to 50, 50 to 100, and greater than 100 microns. The longest dimension was reported as the particle size when the sample was counted with a microscope. In general, the results obtained using the two methods correlated closely. Some discrepancy from an exact correlation was expected since the HIAC sizes particles according to their projected area rather than their longest dimension. Consequently, long particles or fibers were recorded as being smaller when counted by the HIAC counter than when observed with the microscope. The automatic counter could be adjusted to compensate for this difference if the shape of the particles of interest was known and remained constant.

The Coulter counter is an automatic counting and sizing instrument which was originally developed to count blood cells. It has, however, been adapted to a wide range of industrial uses and is capable of counting and sizing particles from about 0.5 to 800 microns in diameter. The counter operates as follows: An aperture with an electrode on either side is immersed in a beaker of electrolyte containing the particles of interest. The electrolyte is drawn through the aperture at a controlled rate which depends on the aperture size. The resistance between the immersed electrodes is momentarily changed with the passage of each particle and a voltage pulse is produced of a magnitude proportional to the particle volume. Each pulse which occurs is electronically amplfied, scaled, and counted. The particle size indicated by the counter is then the diameter of a spherical particle having a volume equivalent to the envelope of the particle which passes through the aperture. The size range over which the instrument may be used is controlled by the aperture diameter. Apertures are available from 30 to 2000 microns in diameter. It is recommended that the aperture used should be at least twice the diameter of the largest particle to be sized. The smallest particle which may be accurately sized with each aperture is from 1.5 to 2 percent of the aperture diameter.

The Coulter counter has been compared with microscope measurements of the thickness of wool fibers[13]. The wool fibers sized were first cut to uniform lengths of 98 ±10 microns so the fiber thickness could be calculated from the volume-related Coulter counter output. The fiber diameter distribution of equivalent samples measured with a microscope and with the Coulter counter were found to be in excellent agreement and the reproducibility of the Coulter counter was found to be equivalent to or greater than the standard microscope method.

Irani and Callis[14] have compared particle size distributions obtained with several methods including a Coulter counter. They show that for smaller sized particles the Coulter counter does not give results consistent with those obtained by other methods. They indicate that the range of most accuracy for the Coulter counter is probably between 5 and 100 microns. Some problems of coincidence occur when high concentrations of particles are counted. In almost all cases involving clean parts, packages, and fluids as used in the aerospace industries, coincidence corrections should be small. An analysis of various methods for correcting for coincidence has been performed by Princen and Kwolek[15].

<u>The Royco liquid-borne particle counter</u> is capable of automatically counting and sizing particles greater than 5 microns in diameter. The instrument draws the liquid sample of interest through a viewing cell where it is illuminated by a lamp and lens system. A photomultiplier tube is located at a 90-degree angle from the illumination axis to receive light scattered from the particles. The particles

pass through the viewing field in a fine stream and are counted individually, the amount of scattered light being proportional to the square of the particle diameter. Classification is determined by an electronic pulse height analyzer, and particle concentration totals can be displayed in as many size ranges as desired with the appropriate data display module. The instrument operates at a sample flow rate of from 100 to 500 cc/min, and is capable of sizing particles suspended in a wide range of liquid mediums by changing the sensor cell. Although no comparisons of results obtained with the Royco counter and with a microscope on identical samples are available from the open literature, it is understood that comparative measurements of this type are currently being made.

REFERENCES

(1) Hof, G. F., Present Practices in the Verification of Cleanliness, Sandia Laboratory Report, No. SCTM 147-63(25), Albuquerque, New Mexico, September, 1963.

(2) White, M. L., The Detection of Organic Contaminants, presented at AACC Fourth Annual Technical Meeting, Miami Beach, Florida, May 24-28, 1965.

(3) Marsh, R. C., Cleanliness Meter and Its Application to Solvent Cleaning, presented at AACC Fifth Annual Technical Meeting, Houston, Texas, March 29-April 1, 1966.

(4) Anderson, J. L., Quantitative Detection of Surface Contaminants, Contamination Control, June, 1963.

(5) "Preservation, Packaging, and Packing Materials: Test Procedures", Federal Test Method Standard No. 101a (January 15, 1962).

(6) "Tentative Methods of Test for Abrasion Resistance of Textile Fabrics", ASTM Designation: D1175-64T, 1966 Book of ASTM Standards, Part 24, ASTM, Philadelphia, Pennsylvania (1966), p 225.

(7) "Laboratory Analysis of Intimate Wraps", MSFC Memorandum No. R-ME-MMC-96-94 (July 16, 1964).

(8) "Plastic Film Packaging Materials", NASA Test Report No. TR-92-D (October 9, 1964).

(9) "Tentative Method of Testing Shipping Containers in Revolving Hexagonal Drum", ASTM Designation: D782-60T, 1966 Book of ASTM Standards, Part 15, ASTM, Philadelphia, Pennsylvania (1966), p 223.

(10) "Proposed Method of Test for Erasing Quality of Paper", 1965 Book of ASTM Standards, Part 15, ASTM, Philadelphia, Pennsylvania (1965), p 822.

(11) Walker, Gordon, Contamination Control in the Saturn S-IVB Stage Hydraulic System, presented at the American Association for Contamination Control Annual Technical Meeting, Washington, D.C., May 16-18, 1967. Douglas paper No. 4203.

(12) Romine, J. O., and Gayle, J. B., "Evaluating the HIAC PC-101 Automatic Particle Counter", The Journal of the American Association for Contamination Control, January, 1964.

(13) O'Connell, R. A., and Martsch, R. J., "Progress in the Application of an Electronic Counter for Determining Wool Fineness Distribution", Proceedings of Western Section of the American Society of Animal Production, University of Wyoming, 1962.

(14) Irani, R. R., and Callis, C. F., Particle Size: Measurement, Interpretation, and Application, John Wiley and Sons, Inc., New York (1963), pp 135 and 148.

(15) Princen, L. H., and Kwolek, W. F., "Coincidence Corrections for Particle Size Determinations with the Coulter Counter", Review Sci. Instr., 36, 646 (1965).

APPENDIX A

APPENDIX A

Federal Specifications

Federal Standard No. 595 - Federal Standard Colors

Federal Standard No. 751a - Federal Standard Stitches, Seams, and Stitchings

Federal Test Method Standard No. 101a - Preservation, Packaging, and Packing Materials:Test Procedures
and Change Notice 1, 2, 3, 4, and 5

Federal Standard No. 209 - Clean Room and Work Station Requirements, Controlled Environment

L-P-378a - Plastic Film (Polyethylene Thin Gage)

O-A-51d - Acetone, Tech.

O-A-86 - Hydrochloric Acid, Tech.

O-A-88 - Nitric Acid, Tech.

O-C-105c - Calcium Chloride, Tech.

O-E-751b - Petroleum Ether, Tech.

O-E-760b - Ethyl Alcohol

O-H-765a - Hydrochloric Acid, Tech.

O-H-795 - Hydrofluoric Acid, Tech.

O-N-350 - Nitric Acid, Tech.

O-O-670 - Orthophosphoric Acid, Tech.

O-P-313 - Phosphoric Acid, Tech.

O-S-595a - Sodium Dichromate, Tech.

O-S-642b - Sodium Phosphate, Tribasic, Tech.

O-T-634b - Trichloroethylene, Tech.

P-D-220a - Detergent, General Purpose (P-C-431)

BB-N-411b - Nitrogen, Tech.

NN-P-00515b - Plywood, Container Grade

QQ-C-576b - Copper Flat Products, etc.

UU-T-81g - Tags, Shipping and Stock

PPP-B-601, PPP-B-621, PPP-B-636, PPP-B-640C - Boxes, etc.

CCC-T-191 - Textiles, Test Methods

PPP-D-723 - Drums, Fiber

PPP-F-320C - Fiberboard, etc.

PPP-T-60 - Tape, Pressure-Sensitive Adhesive, etc.

TT-S-735 - Standard Test Fluids, Hydrocarbon (MIL-S-3136)

TT-I-735a - Isopropyl Alcohol (MIL-I-10428A)

Military Specifications

MIL-STD-105D - Sampling Procedures and Tables, etc.

MIL-STD-129D - Marking for Shipment and Storage

MIL-STD-1246A - Proposed Degree of Cleanliness, Cleaning, Controlled Environment and Protection Requirements

MIL-A-148D - Aluminum Foil

MIL-B-131D - Barrier Material, Water Vaporproof, Flexible, Heat-Sealable

MIL-B-22205A - Bags, Transparent, Flexible, Heat Sealable, for Packaging Applications

MIL-C-632B - Cleaner, Vacuum (Industrial Type)

MIL-C-18718A - Cleaning Compound, Solvent

MIL-C-21100A - Cleaner, Vacuum, Industrial, External Filter Type

MIL-D-16791E - Detergents, General Purpose

MIL-F-22191A - Films, Transparent, Flexible, Heat Sealable, for Packaging Application

MIL-L-10547B - Liners, Case, and Sheet, Overwrap; Water-Vaporproof or Waterproof, Flexible

MIL-M-9950 (USAF) - Missile Components; LOX, Liquid N_2, Gaseous O_2, Gaseous N_2, Instr. Air, He and Fuel Handling Systems, Cleaning and Packaging for Delivery

MIL-P-27401B - Propellant Pressurizing Agent, N_2

MIL-S-4461C - Sealing Machines, Heat, Bench, and Portable

MIL-T-27602A (USAF) - Trichloroethylene, Oxygen Propellant Compatible

MIL-A-10428 - Isopropyl Alcohol, Grade A (see TT-I-735a)

MIL-S-3136 - Standard Test Fluids, Hydrocarbons (see TT-S-735)

MIL-P-116E - Methods of Preservation

MIL-STD-794(WP) - Parts and Equipment, Producers for Packaging and Packing of

NASA Specifications

MSFC-STD-105A - Age Control of Synthetic Rubber

MSFC-STD-246 - Design and Operational Criteria of Controlled Environment Areas

MSFC-PROC-166C - Hydraulic System Detail Parts, Components, Assemblies, and Hydraulic Fluids for Space Vehicles, Cleaning, Testing, and Handling

MSFC-PROC-195 - Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of Gas Bearing, Gas Supply, and Slosh Measuring System

MSFC-PROC-245 - Carbon Tetrachloride Scrubber Method for Analysis of Condensable Hydrocarbon Contamination in Compressed Gases

MSFC-PROC-404 - Gases, Drying and Preservation, Cleanliness Level and Inspection Methods

MSFC-SPEC-164 - Cleanliness of Components for Use in O_2, Fuel, and Pneumatic Systems

MSFC-SPEC-217 - Trichloroethylene, Tech.

MSFC-SPEC-233A - Nitrogen, Instrument Grade

MSFC-SPEC-234 - Nitrogen - Space Vehicle Grade

MSFC-SPEC-237A - Solvent, Precision Cleaning Agent

KSC-C-123D - Cleanliness Levels, Cleaning, Protection, and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystems, and Systems for Fluid Use in Support Equipment

MSFC-10419906 - Cleanliness Levels, Cleaning, and Inspection Procedures for Component Parts of Gas Bearing and Slosh Measuring Systems

MSFC-10M01671 - Cleanliness Levels, Cleaning, Protection and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystems, and Systems for Pneumatic Use in Support Equipment

MSFC-SPEC-456 - LOX Compatible Film

NASA-TM-X-53052 - Compatibility of Materials with LOX

NPC-200-2 - Quality Program Provisions for Space System Contracts

NPC-200-3 - Inspection System Provisions for Suppliers of Space Materials, Parts, Components, and Services

Specifications on Cleaning Solvents

O-A-51d - Acetone, Tech.

O-E-751b - Petroleum Ether, Tech.

O-E-760b - Ethyl Alcohol

O-T-634b - Trichloroethylene, Tech.

MIL-T-27602A (USAF) - Trichloroethylene, Oxygen Propellant Compatible

MIL-A-10428 - Isopropyl Alcohol, Grade A (see TT-I-735a)

MSFC-SPEC-217 - Trichloroethylene, Tech.

MSFC-SPEC-237A - Solvent, Precision Cleaning Agent

Specifications on Gases

BB-N-411b - Nitrogen, Tech.

MIL-P-27401B - Propellant Pressurizing Agent, Nitrogen

MSFC-PROC-404 - Gases, Drying and Preservation, Cleanliness Level and Inspection Methods

MSFC-SPEC-233A - Nitrogen, Instrument Grade

MSFC-SPEC-234 - Nitrogen, Space Vehicle Grade

MSFC-PROC-245 - Carbon Tetrachloride Scrubber Method for Analysis of Condensable Hydrocarbon Contamination in Compressed Gases

Specifications on Cleaning Materials Other Than Solvents

O-A-86 - Hydrochloric Acid, Tech.

O-A-88 - Nitric Acid, Tech.

O-H-765a - Hydrochloric Acid, Tech.

O-H-795 - Hydrofluoric Acid, Tech.

O-N-350 - Nitric Acid, Tech.

O-O-670 - Orthophosphoric Acid, Tech.

O-P-313 - Phosphoric Acid, Tech.

O-S-595a - Sodium Dichromate, Tech.

O-S-642b - Sodium Phosphate, Tribasic, Tech.

P-D-220a - Detergent, General Purpose (P-C-431)

BB-N-411b - Nitrogen, Tech.

MIL-D-16791E - Detergents, General Purpose

MIL-C-18718A - Cleaning Compound, Solvent

Specifications on Shipping Containers

NN-P-00515b - Plywood, Container Grade

PPP-B-601 - Boxes

PPP-B-621 - Boxes

PPP-B-636 - Boxes

PPP-B-640c - Boxes

CCC-T-191 - Textiles, Test Method

PPP-D-723 - Drum, Fiber

PPP-F-320c - Fiberboard, etc.

UU-T-81g - Tags, Shipping and Stock

Miscellaneous Specifications

QQ-C-576b - Copper Flat Products (Bar, Sheet, Plate, and Strip), etc.

MIL-S-4461C - Sealing Machines, Heat, Bench and Portable

NPC-200-2 - Quality Program Provisions for Space System Contracts

NPC-200-3 - Inspection System Provisions for Suppliers of Space Materials, Parts, Components, and Services

Specifications on Standards

Federal Standard No. 595 - Federal Standard Colors

Federal Standard No. 751a - Federal Standard Stitches, Seams, and Stitchings

Federal Test Method Standard No. 101a - Preservation, Packaging, and and Change Notice 1, 2, 3, 4, and 5 Packing Materials:Test Procedures

Federal Standard No. 209 - Clean Room and Work Station Requirements, Controlled Environment

TT-S-735 - Standard Test Fluids; Hydrocarbon (MIL-S-3136)

MIL-STD-105D - Sampling Procedures and Tables, etc.

MIL-STD-129D - Marking for Shipment and Storage

MIL-STD-1246A - Proposed Degree of Cleanliness, Cleaning, Controlled Environment and Protection Requirements

MIL-STD-794(WP) - Parts and Equipment, Procedures for Packaging and Packaging of

MSFC-STD-105A - Age Control of Synthetic Rubber

MSFC-STD-246 - Design and Operational Criteria of Controlled Environment Areas

Specifications on Cleaning of Packaging Films, etc.

MIL-STD-1246A - Proposed	Degree of Cleanliness, Cleaning, Controlled Environment, and Protection Requirements

Specifications on Packaging of Space Hardware

Federal Test Method Standard No. 101a and Change Notice 1, 2, 3, 4, and 5 - Preservation, Packaging, and Packing Materials:Test Procedures

Federal Standard No. 209 - Clean Room and Work Station Requirements, Controlled Environment

O-C-105c - Calcium Chloride, Tech.

PPP-T-60 - Tape, Pressure Sensitive Adhesive, etc.

UU-T-81g - Tags, Shipping and Stock

MIL-STD-129D - Marking for Shipment and Storage

MIL-STD-1246A - Proposed Degree of Cleanliness, Cleaning, Controlled Environment, and Protection Requirements

MIL-M-9950 (USAF) - Missile Components, Gaseous Nitrogen, Gaseous Oxygen, Liquid O_2, Liquid N_2, Instr. Air, Helium and Fuel Handling Systems, Cleaning and Packaging for Delivery

MIL-P-116E - Methods of Preservation

MIL-STD-794(WP) - Procedures for Packaging and Packing of Parts and Equipment

MSFC-STD-246 - Design and Operational Criteria of Controlled Environment Areas

MSFC-PROC-166C - Hydraulic System Detail Parts, Components, Assemblies, and Hydraulic Fluids for Space Vehicles, Cleaning, Testing, and Handling

MSFC-SPEC-164 - Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems

KSC-C-123D - Cleanliness Levels, Cleaning, Protection, and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystem, and Systems for Fluid Use in Support Equipment

MSFC-10419906 - Cleanliness Levels, Cleaning and Inspection Procedures for Component Parts of Gas Bearing and Slosh Measuring Systems

MSFC-10M01671 - See KSC-C-123D (Newer specification on same subject matter)

Specifications on Cleaning of Space Hardware

Federal Standard No. 209 - Clean Room and Work Station Requirements, Controlled Environment

MSFC-STD-246 - Design and Operational Criteria of Controlled Environment Areas

MSFC-PROC-166C - Hydraulic System Detail Parts, Components, Assemblies, and Hydraulic Fluids for Space Vehicles, Cleaning, Testing, and Handling

MSFC-PROC-195 - Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of Gas Bearing, Gas Supply, and Slosh Measuring Systems

MSFC-SPEC-164 - Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems

KSC-C-123D - Cleanliness Levels, Cleaning, Protection, and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystem, and Systems for Fluid (Pneumatic) Use in Support Equipment

MSFC-10419906 - Same as MSFC-PROC-195.

MSFC-10M01671 - Same as KSC-C-123D

NPC-200-3 - Inspection System Provisions for Suppliers of Space Materials, Parts, Components, and Services

MIL-STD-1246A - Proposed Degree of Cleanliness, Cleaning, Controlled Environment, and Protection Requirements

SPECIFICATIONS ON TESTING OF HARDWARE FOR CLEANLINESS

MIL-STD-1246A - Proposed Degree of Cleanliness, Cleaning, Controlled Environment, and Protection Requirements

MIL-M-9950 (USAF) - Missile Components; Liquid Oxygen, Liquid Nitrogen, Gaseous Oxygen, Gaseous Nitrogen, Instrument Air, Helium and Fuel Handling Systems, Cleaning, and Packaging for Delivery

MSFC-PROC-166C - Hydraulic System Detail Parts, Components, Assemblies, and Hydraulic Fluids for Space Vehicles, Cleaning, Testing, and Handling

MSFC-PROC-195 - Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of Gas Bearing, Gas Supply, and Slosh Measuring Systems

MSFC-SPEC-164 - Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems

KSC-C-123D - Cleanliness Levels, Cleaning, Protection and Inspection Procedures for Parts, Field Parts, Assemblies, Subsystems, and Systems for Fluid (Pneumatic) Use in Support Equipment

MSFC-10419906 - See MSFC-PROC-195

MSFC-10M01671 - See KSC-C-123D

Specifications on Films and Their Properties

Federal Test Method Standard No. 101a - Preservation, Packaging, and
and Change Notice 1, 2, 3, 4, and 5 Packing Materials:Test Procedures

L-P-378a - Plastic Film (Polyethylene Thin Gage)

MIL-A-148D - Aluminum Foil

MIL-B-131D - Barrier Material, Water Vaporproof, Flexible, Heat-Sealable

MIL-B-22205A - Bags, Transparent, Flexible, Heat-Sealable, for Packaging Applications

MIL-F-22191A - Films, Transparent, Flexible, Heat-Sealable, for Packaging Applications

MIL-L-10547B - Liners, Case, and Sheet, Overwrap; Water-Vaporproof or Waterproof, Flexible

MSFC-SPEC-456 - LOX Compatible Film

NASA-TM-X-53052 - Compatibility of Materials with LOX

NASA-TM-X-985 - Compatibility of Materials with LOX

APPENDIX B

TABLE B-1. FILMS

Film	Film Type	Source
Aclar 22 C	Fluorohalocarbon	Allied Chemical Co.
Aclar 33 C	Fluorohalocarbon	Allied Chemical Co.
Dow PZ2003.00	PE-PP copolymer/saran/PE-PP copolymer	Dow Chemical Co.
Dow PZ5527.05	Oriented Polypropylene/saran	Dow Chemical Co.
Film-O-Rap	Aclar/PE/Mylar/PE	Rap Industries
Genotherm U.S. 2000	Rigid Polyvinyl chloride	American Hoechst Corp.
Kapton H	Polyimide	E.I. du Pont deNmours & Co., Inc.
Kel-F	Polychlorotrifluoroethylene	Erie Enameling Co.
Kynar	Polyvinylidene fluoride	Mono-Sol Division, Baldwin Montrose Chemical Co., Inc.
Lexan	Polycarbonate	General Electric Co.
Marlex 5003	High-density polyethylene	Mehl Mfg. Co.
Marlex 6003	High-density polyethylene	Mehl Mfg. Co.
Marlex 6009	High-density polyethylene	Mehl Mfg. Co.
Marlex TR101	High-density polyethylene	Mehl Mfg. Co.
Mylar 322	Polyester	E.I. du Pont De Nemours & Co., Inc
Mylar (amorphous)	Polyester	E.I. du Pont de Nemours & Co., Inc
Nylon-6	Polyamide	E.I. du Pont de Nemours & Co., Inc
Polybutene	------	Mobil Chemical Co.
Polyethylene (low density)	------	Clean Room Products, Inc.
RC-AS-1200 Antistatic Polyethylene	------	Richmond Corp.
Polyphenylene oxide	------	Mobil Chemical Co.
Polypropylene (biaxially oriented)	------	Kordite, Division of Mobil Chemical Co.
Polysulfone	------	Mobil Chemical Co.
Sohio 331	Polyacrylonitrile	Standard Oil Company of Ohio
Tedlar	Polyvinyl fluoride	E.I. du Pont de Nemours & Co., Inc
FEP-Teflon	Fluorinated ethylene-propylene coated polytetrafluoroethylene	E.I. du Pont de Nemours & Co., Inc

TABLE B-2. WEIGHT LOSS OF ABRADED FILMS

Run Number	1	5	11	2	8	13	15	16
Material	Low-Density Polyethylene 6 mils			Nylon-6 2 mils		Sohio 331 1 mil		
Revolutions[a]	Weight Loss In Milligrams							
0	0	0	0	0	0	0	0	0
100							1.9	0.3
200							2.9	
300						3.1	4.1	
500	6.5	5.5	2.6		0.4			
1000	15.5	12.2	9.8					
1250				0.9	2.2			
1500	26.0	19.7						
2000				4.0	5.0			
2417				5.7				
Cleaning Solvent	EtOH[b]	EtOH	TCTFE[c]	EtOH	TCTFE	TCTFE	TCTFE	TCTFE

Notes: (a) Taber Abraser, CS-17 wheels, 1000 grams per wheel.
(b) EtOH is Ethanol.
(c) TCTFE is trichlorotrifluoroethane.

TABLE B-3. WEIGHT LOSS OF ABRADED FILMS

Run Number	17	18	19	20	28	27	30	33
Material	Sohio 331 1 mil	Tedlar 3 mils		Lexan 3 mils			Aclar 33C 2 mils	
Revolutions[(a)]	Weight Loss In Milligrams							
0	0	0	0	0	0	0	0	0
100	0.3							
200	1.3							
250		0.5						
300	2.2							
400	3.2							
500	4.0	1.6	1.3	0.1		2.1		3.4
1000		4.2	3.8	0		4.6	3.3	
1500		6.8	6.9	0			5.4	
2000		9.0	9.0	0		10.1		
3000				0.8				
4000				1.0				
6000				1.9	0.8			
8000				2.8				
10000				3.8	1.9			
Cleaning Solvent	TCTFE[(b)]	TCTFE	TCTFE	TCTFE	TCTFE	EtOH[(c)]	EtOH	EtOH

Notes: (a) Taber Abraser, CS-17 wheels, 1000 grams per wheel.
(b) EtOH is Ethanol.
(c) TCTFE is trichlorotrifluorothane.

TABLE B-4. WEIGHT LOSS OF ABRADED FILMS

Run Number	34	31	32	35	36	39	40	41
Material	Aclar 33C 2 mils	FEP Teflon 5 mils		Aclar 22C 5 mils		RC-AS-1200 Antistatic Polyethylene, 6 mils		
Revolutions[(a)]	Weight Loss In Milligrams							
0	0	0	0	0	0	0	0	0
500	3.6	2.1	2.6	1.8	2.1	7.2	6.5	6.3
1000	7.8	4.9	6.0	3.9		17.8		14.9
1200					6.4			
1500	11.4	7.7	9.6	6.0		27.2	24.0	25.2
1800					10.8			
Cleaning Solvent	EtOH[(b)]	EtOH	EtOH	EtOH	EtOH	EtOH	EtOH	EtOH

Notes: (a) Taber Abraser, CS-17 wheels, 100 g/wheel.
(b) EtOH is Ethanol.

TABLE B-5. WEIGHT LOSS OF ABRADED FILMS

Run Number	42	43	45	46	48	49	50
Material	Kapton H 2 mils		Film-O-Rap 7750 (PE-Side) 4-1/2 mils			Mylar 322 10 mils	
Revolutions[a]	Weight Loss In Milligrams						
0	0	0	0	0	0	0	0
250			2.4				
500	0.6	3.2	8.5	7.5	0		
750			14.5				
1000		7.4	19.8	17.2	0		
1500				27.1			
1875			36.6				
2000					1.6	0.5	
3000					2.8		
4000					3.5	1.0	
5000					5.0		0.9
6500						4.7	2.5
8000							3.5
8500						6.8	
10000						7.9	4.7
Cleaning Solvent	EtOH[b]	EtOH	EtOH	EtOH	EtOH	EtOH	EtOH

Notes: (a) Taber Abraser, CS-17 wheels, 1000 g/wheel.
(b) EtOH is Ethanol.

TABLE B-6. WEIGHT LOSS OF ABRADED FILMS

Run Number	54	55	58	59
Material	Polyphenylene oxide 3 mils		Polysulfone 3 mils	
Revolutions[(a)]	Weight Loss In Milligrams			
0	0	0	0	0
1500		3.2	8.4	
1520				4.2
2200	4.5			
3000		7.4		
3050				13.7
3400			28.1	
4300	11.0			
4500				30.3
4850			43.5	
5000		11.9		
5219	13.2			
Cleaning Solvent	EtOH[(b)]	EtOH	EtOH	EtOH

Notes: (a) Taber Abraser, CS-17 wheels, 1000 g/wheel.
(b) EtOH is Ethanol.

TABLE B-7. WEIGHT LOSS OF ABRADED FILM

Run Number	121	122	123	124	125	126	127
Material	Kynar 5 mils Cordoflex KC-30		Dow PZ 5527.05, 3-1/2 mils Saranex side		Polybutene 3 mils		
Revolutions	Weight Loss In Milligrams						
0	0	0	0	0	0	0	0
200			5.7	6.6			
250					2.4	1.4	1.3
400			14.7	14.7			
500	10.6	8.0			7.8	5.7	6.1
600			20.3	22.1			
750					13.0	9.4	7.7
1000	26.1	22.6					
2000	51.1	49.9					

Note: Ethanol used as the cleaning solvent.

TABLE B-8. WEIGHT LOSS OF ABRADED FILM

Run Number	128	129	130	131	132	133	164	165
Material	Dow PZ 2003.00, 4 mils PE/PP Copolymer Side		Polypropylene, 2 mils		Marlex 6003 4-1/2 mils HDPE		Amorphous Mylar, 5 mils	
Revolutions	Weight Loss In Milligrams							
0	0	0	0	0	0	0	0	0
250			3.6	2.0				
500	2.0	1.2	6.9	7.2	2.7	1.3	0.8	1.2
750			9.8	11.4				
1000	5.0	3.5			5.1	3.2	2.7	4.1
2000	10.8	10.2			8.7	7.3	9.0	10.6

Note: Ethanol used as the cleaning solvent.

TABLE B-9. WEIGHT LOSS OF ABRADED FILM

Run Number	134	135	136	137	138	139	140	141
Material	Genotherm U.S. 2000 Rigid PVC, 6 mils			Kel-F, 5 mils			Marlex 5003, HDPE 6 mils	
Revolutions	Weight Loss In Milligrams							
0	0	0	0	0	0	0	0	0
500	6.9	2.9	0	3.5	4.7	5.0	0.7	0.3
1000	18.7	8.9	6.2	6.9	8.4	8.3	1.3	0.8
2000	36.3	21.0	22.1	16.0	17.6	15.7	3.0	2.0

Note: Ethanol used as the cleaning solvent.

TABLE B-10. WEIGHT LOSS BY ABRADED FILM

Run Number	142	143	144	145	146	147	148
Material	Marlex 5003 HDPE 6 mils	Marlex 6009 HDPE 5 mils			Marlex HDPE 4 mils		
Revolutions	Weight Loss In Milligrams						
0	0	0	0	0	0	0	0
250					1.9	1.9	1.1
500	0.3	0.4	0.8	0.1	3.8	3.1	2.7
1000	0.8	0.7	1.3	0.9	6.2	5.9	5.5
2000	2.2	1.4	1.5	1.6			

Note: Ethanol used as the cleaning solvent.