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INTEGRATION OF CHEMICAL-MATERIAL TEST METHODOLOGY WITH A DATABASE SYSTEM

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RESEARCH AND TECHNOLOGY DIRECTORATE

April 1997

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AUTHORS Wendel J. Shuely

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PREFACE

The work described in this report was authorized under Project No. 10162622A553, CB Defense/General Investigation. This work was started in October 1993 and completed in June 1995.

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INTEGRATION OF CHEMICAL-MATERIAL TEST METHODOLOGY WITH A DATABASE SYSTEM

1. INTRODUCTION

The interaction of a chemical with a material influences the material selection process for a significant fraction of industrial and military products. Concurrent engineering practice and the Army streamlined acquisition process emphasize the consideration of factors such as chemical resistance early in the product development cycle. To support and assist in the early selection of chemically resistant materials, a mainframe dialup computer database had been developed and several revision cycles dealt with data management and technology issues. Recently, numerous problems contributed to user requests for improvements in the material science and test methodology aspects of the system. An example of a serious difficulty was the inability to employ the searchcompare-sort-rank capabilities of a relational database system, which was due to insufficient documentation of the test method parameters held constant and/or the material compositions tested. The system could only be employed in a single transaction mode in which a single or listed chemical-material test result was retrieved and displayed.

Experience as a user, coupled with an evaluation of the material ranking capabilities, led to the formulation of a number of objectives for enhancing the chemicalmaterial compatibility system. Test methods sponsored by standards organizations (such as ASTM and ISO) required further standardization of a specific subset of test parameters (i.e., time, temperature) for generation of data sets that could be held constant for a database query. In addition, these test methods needed further modification to improve safety and to minimize chemi al test volumes. The documentation of the material composition tested often required traceability, even to the lot number and conditioning procedures. After interim system evaluation and testing, it was necessary to integrate the entire data flow process from the test site to storage within the database system.

The material selection and ranking requirements imposed by the mainframe database system were used to generate the material descriptors and test reports employed at laboratory level electronic notebooks. These electronic notebooks became, essentially, laboratory databases. The ASTM E49 Committee was concurrently developing material identification and test report formats, therefore, these formats were adopted whenever possible and developed into laboratory test databases. An investigation of the accuracy and cost factors for data transfer from measurement to database storage led to the addition of the goal of direct test-to-database file transfer via modem, diskette, or other electronic/magnetic mechanism.

Over 50 different standard tests had been employed in the chemical-material compatibility database to obtain chemical effect results. It was judged necessary to minimize the initial number of tests to a core subset and to configure these tests into a cascading flowchart scheme.

An objective was formulated to structure the chemical resistance tests in a scheme designed to screen out nonresistant materials with initial tests and/or theoretical predictions. An investigation of the selection accuracy led to the structuring of the initial

screening scheme as a "deselection" strategy to eliminate nonresistant materials. Implementing this scheme lead to a decreasing number of materials and test results being transferred to the chemical-material compatibility database as a material selection process proceeded (as apposed to a full matrix of materials x tests). Regardless, it was decided to retain independent branches in the scheme to distinguish between effects of chemical on degradation of material properties and the effects of materials on mass transport (e.g., desorption, contamination transfer, etc.) of the chemical in the environment. Further independent scheme branches were required for separate material performance categories (i.e., mechanical, optical, thermal, and electrical).

The description follows a top down structure where the paperless link between the mainframe database and the test site database is reviewed. The modular connection between the mainframe database files and the ASTM E49 "test report" formats is then outlined. The structure is described for converting the ASTM E49 formats to laboratory database for material and test results. A final results section sketches the selection and cascading chemical resistance test methods, with the objective of developing a comprehensive test scheme.

The general results were applied to most materials, including metals and ceramics; however, the specific test methodology and material selection process discussed applies to polymeric materials. The term "integration" within the context of this study refers to the coordination of the entire chemically resistant material selection process, from specification of test methods to be used to the structure of the database query, which is eventually employed to search and rank materials. The term "strategy" is employed herein to denote the fixed overall approach selected for materials testing, reporting, and ranking and the variable system of test flow charts for screening and deselection of candidate materials. The term "direct" transfer is employed here to represent any electronic (modem, network, etc.) or magnetic (diskette, tape, etc.) mechanism of file transfer that avoids transcription errors and delays.

Preliminary steps to improve the chemical-material compatibility database have been documented.¹⁻⁴ Several industrial programs have been initiated to apply database capability to chemical-material compatibility testing, mainly supporting metal corrosion.^{5,6} A test scheme for chemical-composite compatibility has been added to Military Standardization Handbook for Polymer Matrix Composites⁷ and a database has been designed for MIL-HDBK-17.⁸ A chemical-polymer compatibility test program and database are being implemented by industrial laboratories.⁹ Experimental and predictive screening methods have been developed specifically for a chemical-polymer compatibility test program supporting a chemical-material database.¹⁰ Sorption and desorption diffusion test methods have been standardized to provide comparative data for a chemical-materials compatibility database.¹¹

2. METHODOLOGY

2.1 <u>Chemical Resistance Categories</u>.

At one extreme, some materials database users would prefer that the system provide them with a decision on exactly what material they should use for their chemical compatibility application. The database developer, however, normally could only be responsible for the efficient retrieval of data, because the data provided would rarely account for the application-specific mechanical, thermal, optical, or electrical stresses that might have negative synergistic effects on performance.

An example of a midground or an intermediate level of categorization used in traditional hardcopy tabulations and computer databases is the arbitrary partitioning of chemical-material property test data into "Good," "Moderate," and "Poor" type categories. Often, up to 10 qualitative levels are employed, usually as alphanumeric codes. However, this type of feature has been suppressed from the current configuration to avoid misrepresenting the degree of chemical resistance of a material; therefore, the system is strictly a numerical database.

2.2 <u>Criteria for Test Methodology Development</u>.

Regardless of the test method to be employed, a set of criteria for the development and documentation have been devised (Table 1). These criteria are used in research planning and in monitoring the status of each test method. The first criterion is to establish and document the rationale for selecting the method. The second criterion is the development of unique safety equipment procedures or method modifications required for hazardous liquids. The third criterion is the standardization of the procedures. The fourth criterion requires that standard specimens be developed to facilitate the transfer of the method to other test laboratories. The fifth criterion requires the data capture format for direct transfer from test to database. The sixth criterion requires the systematic measurement of the method precision and bias based on an experimental design; round-robin testing might also be included here. Finally, the seventh criterion calls for a plan for transferring the method to other laboratories. Note that the fifth criterion deals with the materials database directly, but each criterion influences the content of the files transferred to the database.

3. RESULTS

3.1 Integration of Mainframe and Laboratory Computer Databases.

3.1.1 <u>Direct Transfer from Test-to-Database</u>.

The first integration requirement involves the paperless transfer on magnetic media (diskettes) from the test instrument to the database. This strategy was initially applied to chemical permeation test data for protective materials within our laboratories.^{1,2} A contrast is shown in Figure 1 between the direct paperless data flow path versus the traditional text flow into a database. The "traditional" path for material data capture involves several steps. These steps are listed as follows:

- initial generation of material property data
- generation of a hardcopy test report

• incorporation of material and chemical description and test results in a draft publication, usually scattered throughout various report sections

- formal referee, editorial, and printing process
- later initiation of a program to identify, select, and queue the reports

• expert review of the publications and extraction of material identification and characterization and test properties spread throughout narrative, tables, figures, and appendixes

• keyed entry and proofreading of the input

Experience has suggested that most of the expense is involved in the knowledgeable review needed to extract data from publications onto data capture forms. Most of the time delay is in the review, editing, and printing step.

The data path being implemented for commercial materials testing contracts is displayed (Figure 1, left side). The investigator or commercial tester is provided a diskette containing blank material and chemical descriptor forms and test report forms in dBase IV or PC (personal computer) database equivalent files. The material test property is measured and the material descriptors, test parameters, and data are transferred to the ASTM E49 forms on the diskette. This transfer can be executed by keyed entry, file export from the test instrument controller, or a combination of both. The investigators, with respect to materials science, provided data review evaluations. The independent, internal review occurs at this point. Data review, with respect to data integrity management, is provided at the PC Buffer-Server by database staff. The programmed data check (the computerized ASTM E49 test report format for sign, magnitude, range, and other editing parameters) has been found to have already minimized most gross errors. The data sets are then incorporated into the mainframe database by file transfer. Note that the investigator still submits the technical publication through the traditional channel in parallel for independent, external review.

3.1.2 Modular Descriptor Formats.

The structure of the mainframe chemical-material compatibility database consists of three supporting modules that document the following:

- chemical identification
- material identification
- test method literature reference

These combine to add documentation to the fourth module, the test property data set that contains the actual test parameters and results. A single computerized data capture format for all chemical-material-test data sets is not feasible because of the unique characteristics of each test. Conversely, a unique, customized data capture form for each chemical-material-test can have highly redundant sections. This intermediate, modular set of data capture formats that parallel the database structure appears optimum. This modular strategy is depicted in Figure 2. Note that the test method literature reference and test report are represented under "Test." The material documentation is provided by the recently adopted ASTM E1308-92 Standard Guide for Identification of Polymers (Excluding Thermoplastic Elastomers) in Computerized Material Property Databases. The ASTM E1308 has been coded into a PC dBase IV menu-driven set of screens, described below. The form previously employed is titled "Chemical Protective and Resistant Material Descriptor Form." These two formats are being merged; they have several identical and unique fields that have been consolidated. The difficulties are in the related but not identical fields.

The chemical or liquid descriptor contains several chemical identification fields, purity, source, and lot numbers. A few additional parameters have been added from the various standard tests on chemical resistance (i.e., ASTM D471/D543/D896 and ISO 175/1817). Liquid state and chemical physical properties are not recorded, because these are linked to other database through the CA Registry Number.

The "Test Report" format uses the general guidelines from ASTM E49, specific fields common to the most widely used chemical resistance tests (Figure 2), and several generic fields for exposure conditions (e.g., time and temperature).

As currently structured, each format is an independent PC database, linked by project reference code. The modules are integrated at a PC buffer-server before uploading to the mainframe database.

3.2 Laboratory and Instrumentation Computer Databases Using ASTM E49 Formats.

3.2.1 ASTM E1308 Format as a Database.

The materials module is described in more detail here. An evaluation of the ASTM E49 E1308 format reveals the following features. The format is essentially a database. Two types of descriptors are those in iterative and single-entry, noniterative sets. The iterative descriptors consist of a material component/composition or classification property that require multiple loops through several fields. These iterative sets of fields have been separated into individual databases linked to the main database that is comprised of single entry fields (Figure 3).

The ASTM E1308 format has been coded into a PC database. The ASTM E1308 database is in menu format with the over 60 fields incorporated into about 15 unique screens. Those screens requiring multiple sets of entries can be cycled through as many times as needed. The tables included in ASTM E1308 have been converted into windowed pick lists for error free selection of entries. This database version of ASTM E1308 has been documented in a draft users/programmers manual.¹² Figures 4-7 show an example of a set of screens in the database.

A typical screen for several single-field entries contained in the main database file is shown in Figure 4. An example of a non-iterative memo field entry is displayed in Figure 5. Iterative fields are structured as linked database files as shown in Figure 3 and an example screen is displayed in Figure 6 for one of several classification properties derived from a tensile test. An overlay window to prompt and control the iteration process is shown in Figure 7. Also note that the example deals with an elastomer in a reasonably successful attempt to adapt and expand the scope of ASTM E1308 (until a dedicated elastomer identification format is completed by ASTM E49).

3.2.2 Test Reports in ASTM E49 Formats.

Several ASTM tests are being adopted or modified to provide test data directly into the chemical-material compatibility database. Among these are ASTM D3132, ASTM D471, and ASTM D543. The ASTM D3132(90), Standard Test Method for Solubility Range of Resins and Polymers, is mainly used to characterize solvents for coating polymers. This method has been adapted for application in an "inverse" mode and is being employed to screen for insoluble and, therefore, potentially chemically-resistant polymers. An "electronic notebook" ASTM D3132 test report and a Summary Test Report have been developed as a PC database. Examples are shown in Figures 8 and 9.

The database report forms displayed in Figures 8 and 9 result from coupling the ASTM E49 database format proposals for a generic test report with our decade long evolution of a hardcopy notebook log for ASTM D3132 experiments. In Figure 8, fields 3-6 are derived from the ASTM E49 proposed format, and fields 7-10 are derived from ASTM E1308, with fields 9, 10, and 12 providing the database link to the full documentation of the material in the ASTM E1308 database (Figures 4-7). The next 10 "liquid" related fields are linked to liquid property databases through the "CAS #." The remaining fields in Figure 8 employ the computational capability within a database to perform the various concentration calculations. The second page of the report form contains the observations noted on polymer solubility according to prompted observation codes, which have been documented.¹⁰ The subset of fields that are abstracted into the "Summary Form" in Figure 9 constitute the file that is transferred to the mainframe chemical-material compatibility database.

3.2.3 <u>Selection and Sequencing of Chemical Resistance Tests Matrix of Material Level</u> Versus Effect.

The liquid-material interaction can be considered from two inverse viewpoints:

- effects of <u>materials on liquids</u>
- effects of liquids on materials

In one case, the effect of the material on the liquid mass transport is of interest. The mass transport processes influence the quantity and rate of vapor transported into the air and the subsequent inhalation hazard. The liquid transport on contact with skin determines the percutaneous hazard.

The inverse effect is the influence of the <u>liquid</u> on the <u>material</u> performance through measurement of the material property effects. This process involves the effect of chemicals on mechanical, thermal, optical, and electrical properties.

These effects can be considered at the material and component levels. Other levels could also be defined, such as piece-part, subsystem, systems, and so on. However, all the levels from component through system can be potentially modeled as assemblies of individual materials. Therefore, the chemical-material interaction is the most basic starting point for chemical-effect test methodology.

3.2.4 <u>Material Test Program Strategy for Sequencing of Test Methods and Direct</u> <u>Transfer of Test Reports to a Database</u>.

Experience has shown that it is difficult to definitively select chemically resistant materials with screening tests or predictive methods. The objective of the initial sequence of predictive methods and screening tests is to eliminate or "deselect" the largest subset of materials that would not be chemically resistant.

A general scheme for this material test strategy can be depicted as a flow chart in which one enters with a set of candidate materials for a hypothetical system (Figure 10). Certain materials in a system might be unique and not readily replaceable with alternate, more chemically resistant materials; therefore, these would not be submitted to a screening test (e.g., polycarbonate aircraft canopies). This is shown by test flow skipping over "screening" in the figure flow chart.

The candidate materials would be submitted to the simplest feasible screening and ranking tests. An evaluation of available tests led to adoption of a polymer-in-liquid solubility test, ASTM D3132. The test consists of placing about 10% uncrosslinked polymeric resin (pellets, powder, gum) into a few milliliters of liquid and observing solubility, insolubility, or hazy/borderline solution appearance. Soluble or partially soluble materials would "fail", and these would be documented in the chemical-material compatibility database.

Completely insoluble and unswollen polymers are candidate resistant and protective materials. These would "pass" onto the two following categories of further tests (Figure 10):

- effects of materials on liquids
- · effects of liquids on materials

A generic test method has been developed that allows measurement of these three properties in one experiment (i.e., equilibrium solubility, fraction extracted, and diffusion coefficient), where equilibrium solubility equals weight gain plus fraction extracted. Specific sections of the generic method have been adapted to specific material physical states, (elastomer or thermoplastic) or application categories (adhesives). The method can be implemented with either a thermobalance or recording balance test configuration (Figure 11). Note that conventional procedures in ASTM D471/D543 use a nonrecording balance. The equilibrium solubility and diffusion coefficient test can be employed as a screening test, although this is believed to be considerably less efficient than use of the ASTM D3132 solubility test. The flow diagram for this strategy is (Figure 11, last element) similar except the solubility (as weight gain) is initially determined on the desorption microbalance. If the <u>S</u>olubility is less than a criterion value (e.g., 10%), then the experiment is completed and equilibrium solubility, fraction extracted, and diffusion coefficient are obtained. If the weight change exceeds the criterion, the test is terminated and only the weight gain value is recorded in the material database.

One can return to the test strategy element (Figure 10) for effects of liquids on materials and expand this branch into a complete strategy for testing liquid effects on material properties (Figure 12). The expanded flow chart shows four general, independent material property categories which are mechanical, electrical, thermal, and optical (following PLASPEC material database categories). A few examples of each test category are shown and these represent the current priority for evaluating material properties and developing tests (within a specific DoD program).

A generic scheme for chemical effects on material properties has been devised (based on the general approach in ASTM D896) to overcome the inconsistencies and overlaps among methods that specify chemical exposure, temperature-humidity conditioning, and property measurement. To solve these problems, each step shown in Figure 13 has been constructed as a distinct module within ASTM E1308 or the individual method test report (e.g., ASTM D471 or ASTM D543). These modules have been written in generic format across several mechanical test types as represented in Figure 13. In this scheme (Figure 13), the specimen is prepared in the standard manner as for any test method. The specimen is then immersed in the chemical according to ASTM D471/D543/ D896. Weight and dimensional changes are determined for any mechanical specimen used. The required temperature/humidity conditioning is then followed before the corresponding property is measured (tensile, flexure, compression, shear, etc.). For each module or test method in the above scheme, a computerized test report has been drafted or a section within ASTM E1308 has been specified for the direct transfer to the chemicalmaterial database.

4. SUMMARY

A scheme of related test methods has been designed and integrated into a chemical-material compatibility database. A polymer solubility test successfully screens out or deselects most nonresistant polymeric materials. The candidate resistant materials are submitted to further testing, either for the effect of chemicals on materials or the effect of the material on chemical transport in, around, and through the polymer. A computerized test report has been developed for each of the chemical-material test methods, and each test report is linked to an ASTM E1308 file to document the material test specimen. These test report files can then be merged and transmitted to the mainframe materials database to provide a paperless transfer mechanism.

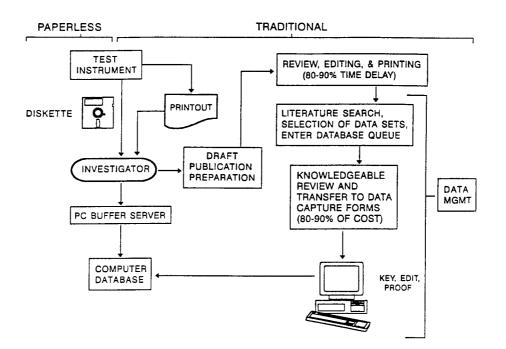


Figure 1. Transfer of Experimental Data Sets from Instrumentation to a Computer Database by the Paperless and Traditional Processes

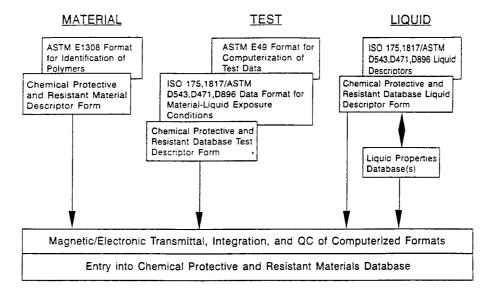


Figure 2. Computerized Format Modules for Documentation of Liquid Effects on Material Properties

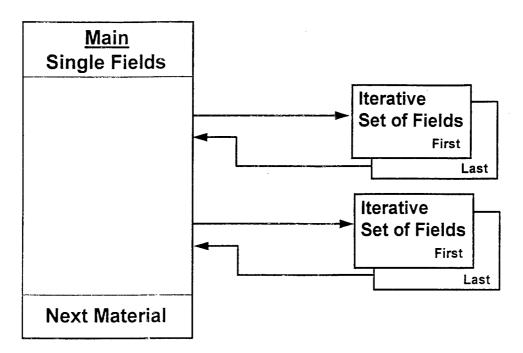


Figure 3. Personal Computer Database Structure Developed for ASTM E1308 Standard Guide for Identification of Polymers (Excludes Thermoset Elastomers) in Computerized Material Property Databases

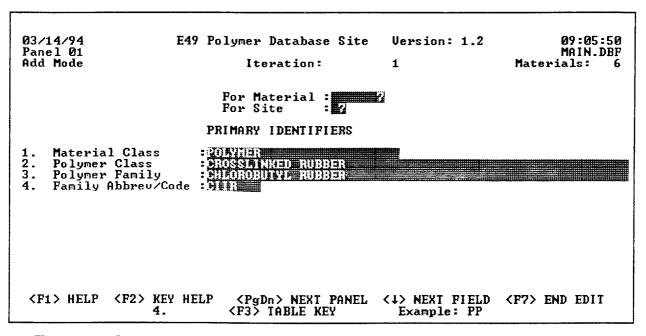


Figure 4. ASTM E1308 Polymer Identification Database: Primary Identifiers

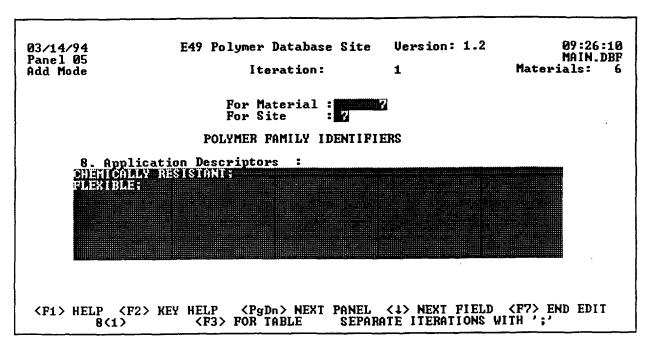


Figure 5. ASTM E1308 Polymer Identification Database: Application Descriptors Memo Field Example

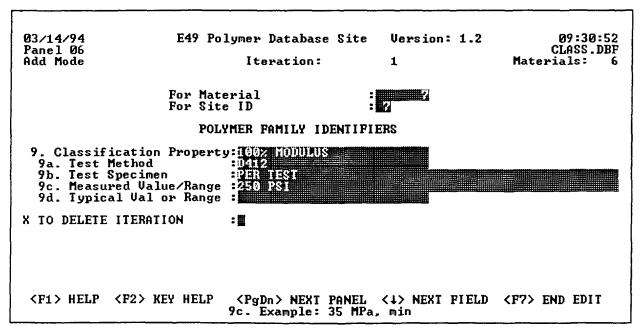


Figure 6. ASTM E1308 Polymer Identification Database: Classification Property Iteration Example

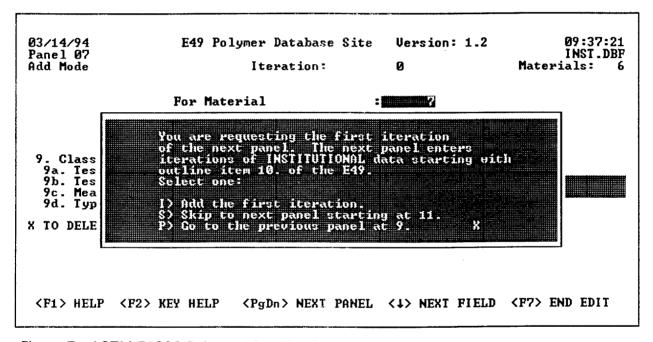
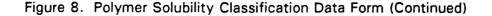


Figure 7. ASTM E1308 Polymer Identification Database: Iteration Branch Control Example

93-001 Log Number : Date 07/16/93 : Operator : Type of Test Polymer-Liquid Solubility : ASTM Test Method D3132 : Date of Standard 1990 : Pub Source of Data Polymer polyisobutylene : Structural Descrip. : linear, uncrosslinked, gumstock, co-chloroisobutyleneASTM E1308 #4 Family Polymer ASTM Code PIB : Abbrev. Code Polymer Lot # Smithers 001 ... ASTM E1308 #18a : Traceability Lot# Polymer Manufacturer : ASTM E1308 Link : 3-chloropropyl thiolacetate (CPTA) Liquid 1 : Liquid 1 CAS # : 13012-54-9 Liquid 1 Lot # Liquid 1 Manufacturer: Liquid 1 Purity Wt% : 90.00% Liquid 2 Liquid 2 CAS # : Ligid 2 Lot # Liquid 2 Manufacturer: Liquid 2 Purity Wt% : 0.00 ? 0.0000 grams Bottle Weight ? 0.0500 grams Bottle, Polymer = 0.0500 grams Polymer Weight Bottle, Polymer, Liquid 1 ? 1.0500 grams = 1.0000 grams Liquid 1 Weight Bottle, Polymer, Liquid 1 & liquid 2 ? 0.0000 grams = 0.0000 grams Liquid 2 Weight Polymer, Liquid 1 & Liquid 2 = 1.0500 grams ? 1.1590 g/mL Density of Liquid 1 Density of Liquid 2 ? 0.0000 g/mL Volume of Liquid 1 0.8628 mL = Volume of Liquid 2 = 0.0000 mL Conc., g/dL, Grams per Deciliter = 5.795 Conc., Weight Ratio = 5.000 Conc., Weight Percent = 4.762%

Figure 8. Polymer Solubility Classification Data Form

```
Observations Codes:
  1 Phase = 1; 2 Phases = 2; Viscous = V; Unchanged Viscosity =
U; Clear = C
  Hazy, low = H1; Hazy, high = Hh; Trace ppt = T
Polymer Sorption Levels: All = a; High = h; Medium = m; Low = 1;
  Unchanged = u
Combined Codes:
  1 Phase Solution (Choose 1)
    1VC (e.g. 1 phase, viscous, clear); 1VCT; 1VH1; 1VHh; 1VUC;
1UHl; 1UHh
  2 Phases (Choose 2 each)
    Liquid: 2L = VC; 2L = VH1; 2L = VHh; 2L = UC; 2L = UH1; 2L =
UHh
    Polymer: /P = A; /P = H; /P = M; /P = L; /P = U
Summary:
        Solubility Code
                          :
                               Ι
        Solution Time, Days: 6
OBSERVATIONS:
Number
             :
                1
             : 07/16/93
Date
Solution Time:
                0
                2L=UC/P=U
Code
             :
                Placed in shaker in hood
Remarks
             :
Number
             :
                2
Date
                07/22/93
             :
Solution Time:
                6
Code
             :
                2L=UC/P=U
Remarks
                Butyl gum floated; totally unchanged appearance
             :
Number
             :
                3
Date
             1
                    - /
                  Solution Time:
                0
Code
             .
Remarks
             :
Number
             :
                4
Date
             :
                  1
                    - /
Solution Time:
                0
Code
             :
                0
Remarks
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Number
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Date
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                  1 1
Solution Time:
                0
Code
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                0
Remarks
             :
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: polyisobutylene
: 3-chloropropyl thiolacetate (CPTA) Polymer Liquid 1 Liquid 2 : Solubility Classification: Ι Observation Code 2L=UC/P=U : Solution Time, Days : 0 : 5.795 Conc., g/dL Conc., Weight Ratio : 5.000 Conc., Weight Percent : 4.762% ASTM E1308 Link : Log Number 93-001 : Polymer-Liquid Solubility Type of Test : : ASTM Test Method D3132 Date of Standard 1990 : Pub Source of Data : Polymer ASTL Code : PIB Polymer Lot # : Smithers 001 Liquid 1 CAS # : 13012-54-9 Liquid 1 Lot # : Liquid 1 Purity Wt% : 90.00% Liquid 1 Density : 1.1590 g/mL Liquid 2 CAS # : Liquid 2 Lot # : Liquid 2 Purity Wt% : 0.00% Liquid 2 Density : 0.0000 g/mL

Figure 9. Polymer Solubility Classification Summary Form

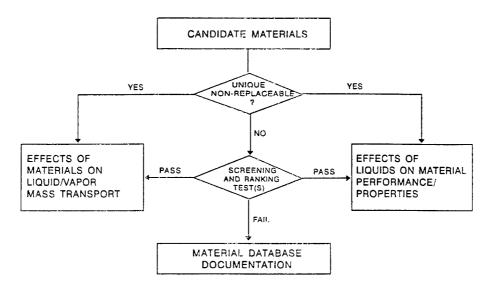


Figure 10. Material Testing Flow Chart for Initial Screening Tests to Deselect Nonresistant Materials

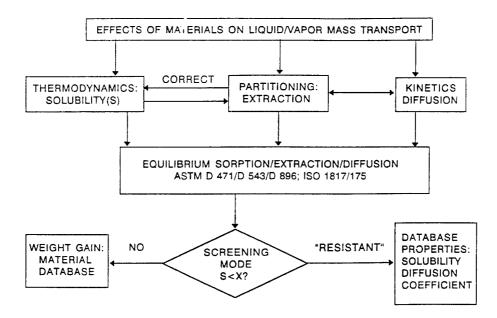


Figure 11. Material Testing Flow Chart Employing Equilibrium Sorption in a Screening Mode and as a Fundamental Property Test

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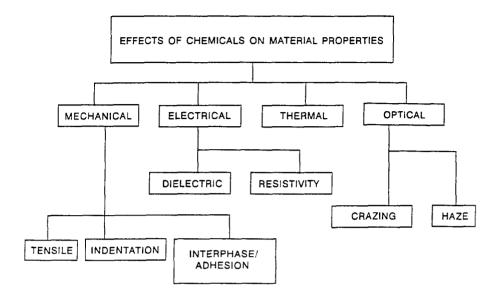


Figure 12. Material Testing Scheme for Effects of Chemicals on Material Properties

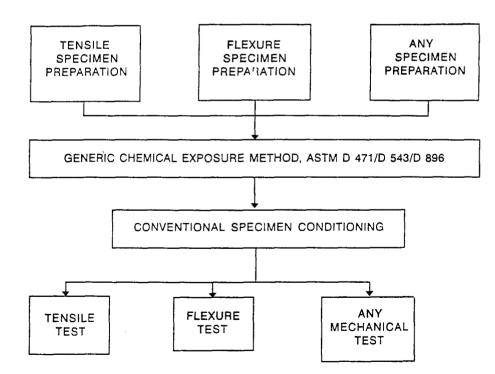


Figure 13. Scheme for Use of a Generic Chemical Exposure Method with Any Test

Table 1. Criteria for Test Methodology Development

Criterion:

- 1. Rationale Documentation
- 2. Special Safety Procedures/Equipment
- 3. Procedures Documented in Standardized Format
- 4. Standard Control Specimens Developed and Stockpiled
- 5. Computerized Test Report in ASTM E 49 Format for Direct, Paperless Transfer to Database
- 6. Method Validation Studies
- 7. Transition Plan for Routine Test Lab and Database Use

Table 2. Material and Component Investigations for Liquid Effects

Level Effect	Materials	Components
Effects of Materials on Liquid/ Vapor Mass Transport		
Effects of Liquids on Material Performance/Properties		

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