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Experimental Design Guidelines for Asphalt Research Contractors Revised 1991

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The Role of the A-001 Contractor

Introduction

The University of Texas at Austin, under the Strategic Highway Research Program (SHRP) contract A-001, is responsible for the overall experimental design, coordination and control of the experimental materials involved with the asphalt research program and has been designated as the Technical Assistance Contractor (TAC). SHRP requires extensive technical support in the design and coordination of the components of the research studies and management of the study materials. SHRP also requires the development of usable products from the research, including information necessary for the writing of specifications and the development of technology transfer materials. The intent of SHRP is to accomplish these services through the Technical Assistance Contractor (A-001).

The different contracts in the SHRP asphalt research program are strongly interrelated, with each contractor employing the research results of others. In order that the work may proceed on schedule and the results of the individual researchers converge on the essential goals of the total program, the TAC will aid SHRP in ensuring timely progress and effective products. Hence, continuing dialogue between the TAC and all asphalt research contractors regarding experimental design is essential. The TAC has been charged with ensuring the quality and proper scope of the asphalt research. For this reason, the final experimental designs and research plans for each contract are to be developed in coordination with the TAC and SHRP. In this manner, SHRP may be assured that no significant gaps exist in the program and that the work will proceed in an efficient and timely manner.

For the majority of the asphalt research contracts, considerable effort will be required for the development of experimental designs and testing of analytical procedures or techniques. This schedule will be closely monitored by the TAC and SHRP, using a project management software system. The scheduling of work by the individual contractors may undergo periodic adjustments as required by the progress of the work and the work of other SHRP contractors. Such adjustments will be developed cooperatively by the individual asphalt research contractors, the TAC, and SHRP.

Objectives of the Asphalt Research Program and Role of Experimental Design

The primary end products of the SHRP asphalt research program are performanced based paving asphalt specifications and, to a lessor extent, accelerated performance tests.

Two objectives that stand out in several of the work statements for the Asphalt Research Projects are as follows:

i. the development and evaluation of new (and innovative) methods for measuring fundamental chemical and compositional properties and laboratory tests for measuring the physical performance of asphalts and asphalt mixtures; and,

ii. the development and evaluation of new (and innovative) methods for relating laboratory performance (and ultimately field performance) of asphalts and asphalt mixtures to measured chemical and compositional properties of the asphalts.

If these objectives are to be satisfied it is imperative that statistically sound experimental designs be developed and followed (Montgomery, 1984). Only in this manner can the reliability of new tests and procedures be properly evaluated and the necessary relationships between the chemical and physical properties of asphalts and asphalt mixtures be reliably related to field performance (ASTM, 1986).

Need for Experimental Design

The SHRP asphalt program offers a unique opportunity to coordinate basic research on the properties and performance of asphalt cement and its role in the performance of asphalt-aggregate mixtures. For this program to be successful, a mechanism must exist which permits contractors doing work under individual contracts to merge, correlate, and draw statistically valid inferences from the data collected from the various studies. To this end, a reference library of asphalt and aggregate materials has been established in Austin, Texas.

Since 32 asphalt cements, 11 aggregate systems, and a number of asphalt modifiers will ultimately be selected for the reference library, it is obvious that all asphalts, modified asphalts, and asphalt-aggregate combinations cannot be tested and evaluated as a part of subsequent asphalt research contracts. Thus, it is essential that experimental design requirements and guidelines be developed to permit the information to be effectively and meaningfully analyzed and correlated among, and within, the asphalt programs.

All asphalt experiments conducted by contractors under the supervision of the TAC must be accomplished using basic statistical procedures and sound experimental designs. In developing the experimental designs, emphasis should be placed on maintaining flexibility to accommodate innovative research, provided that it does not compromise the statistical integrity of the program. Each contractor will be responsible for preparing an experimental design and testing protocol for conducting the experiments which satisfy the project goals of the individual contractor. The TAC will review those designs to assure overall consistency and to provide feedback to the individual project statisticians and researchers. This document will set forth the general guidelines for the design and analysis of the asphalt research program experiments.

Linkage among Experiments

The SHRP asphalt research program consists of a sequence of related interdependent experimental projects. Clearly the success of the entire effort depends upon the success of each of these projects and upon the interchange of ideas and results among projects. It is the responsibility of the TAC to facilitate the interchange of ideas and results among the experimenters in the different projects. Experimental designs and results will be made available to the experimenters from the other projects as early as possible so that all may benefit from this rapid exchange of information.

2

Selection of Materials for the Materials Reference Library

The reference library will contain 32 asphalts and 11 aggregates. Eight asphalts and four aggregates have been designated as core materials and each contractor must test these materials as a minimum. However, for certain evaluation tasks, it will be necessary to include all of the materials in the reference library. It is important that the common core of reference library materials be established for the different Asphalt Program contractors so that their experimental results can be combined and integrated for evaluation purposes. Therefore, the common core of materials was selected to represent those currently or potentially available materials in the United States and Canada. The selected materials also exhibit a sufficiently wide range of properties to serve the needs of the researchers in the asphalt-related contracts.

Selection and Initial Characterization of Asphalts for the Materials Reference Library

The selection of an initial set of 8 asphalts was completed in the Spring of 1988. The preliminary selection of an additional 14 (for a total of 22) asphalts was made in July, 1988. The final 8 asphalts were selected in August 1989. These were selected to provide a wide range of properties and performance. It is essential that the asphalts provide, not only a wide range of values of chemical and compositional properties, but also a certain balance of these values in order to allow the experimenter to evaluate the separate effects of these properties upon the laboratory performance of the materials.

Eight asphalts were chosen to be the core asphalts for the entire SHRP Asphalt Research Program. These eight asphalts must be included in all SHRP experiments, unless elimination or substitution is approved by the TAC, in which asphalt properties are under consideration. Therefore, it was essential that the criteria used for selecting the eight asphalts provide a well-balanced set of asphalts. This was assured by attempting to choose asphalts with performance histories ranging from good to poor and by using aging index (AI) and penetration-viscosity number (PVN), vanadium-nickel ratio, sulfur content, and nitrogen content as a basis for selection. These properties were felt to be closely related to field performance and represent a diverse set of asphalts. The goal of selecting a perfectly balanced set of asphalts cannot be attained because asphalt cements with high PVN and AI values are not marketed and certain combinations of vanadium-nickel ratio, sulfur content, and nitrogens contents do not exist naturally.

As the 32 asphalts in the MRL are sampled, the measured chemical, compositional, and physical properties obtained by the Asphalt Institute and Matrecon will be entered into the Asphalt Program Data Base (Chapter 3). These initial values will be supplemented by data from the various Asphalt Program contractors. The measured values in the data base will represent the mean value and will be accompanied by an estimate of the variation (error) in their measurement (ASTM, 1986). By establishing these initial measured properties, researchers will be able to select additional asphalts (beyond the eight core asphalts) for their future experiments.

The Selection and Initial Characterization of Aggregates for the Materials Reference Library (MRL)

The selection of eleven aggregates for the MRL has been completed and these aggregates are available. A set of four of these have been designated as the core aggregates to be included in all future experiments in which the properties of the aggregates are under consideration. The aggregates were chosen primarily on the basis of their porosity and resistance to moisture damage. Past field performance and composition (calcareous versus siliceous) were also considered so that the eleven aggregates would provide materials with performance histories ranging from good to poor.

Measured property values of these eleven aggregates as obtained from the A-003A and A003B contractors, the Asphalt Institute, and Southwestern Laboratories, will be included in the Asphalt Program Data Base (Chapter 3). As with the asphalt cements, the reported values will contain the estimated mean and an estimate of the variation (error) in their measurement.

3

The Asphalt Program Data Base

The Technical Assistance Contractor (A-001) is responsible for developing and maintaining the Asphalt Program Data Base. This data base, which consists of Summary Data Base and a Archival Data Base, is being developed by the TAC personnel at the University of Texas at Austin. NOMAD II, a commercial data base management program, was chosen as the basis for the data base for this application.

Archival Data Base

The Archival Data Base contains individual data elements, including replicate measurements or measurements from replicate specimens, from each of the experiments conducted by the contractors. The archival data base for an experiment consists of the data elements obtained from the various experiments. This data base, which will be of use primarily to other researchers, will consist of a series of files, one for each experiment, and will be the means for transmitting data to the TAC. As such, it may be thought of as one of the project deliverables. Each data file will have a documentation file (ASCII text) which contains descriptive information for the associated experiment (see Exhibit 3 in Chapter 5 for an illustrative example).

The Archival Data Base will greatly facilitate the timely interchange of data among the different asphalt projects and researchers and will be the source for the data entered into the Summary Data Base. The transmission of these files may take place between researchers, or between the TAC and the researchers, in any convenient manner such as hard copy, electronic mail, or the exchange of a floppy disk.

Summary Data Base

The Summary Data Base will contain a summary of the data elements from the Archival Data Base and will be of value to materials engineers, managers, and administrators. The summary measures of asphalt, aggregate, and mixture properties in the Summary Data Base must all have an appropriate measure of uncertainty. Unless otherwise indicated, in the Data Base a 95% confidence interval will be constructed for the true parameter value. This can be presented as X E where X is the mean and E is the half width of the 95% confidence interval (ASTM, 1986). The all too common practice of arbitrarily assigning an arbitrarily selected value to E is not acceptable. The measures of uncertainty should be provided as part of the Archival Data Base files but they will be reviewed by TAC personnel before they are entered into the Summary Data Base.

Access to Asphalt Program Data Base

Access to the information in the Asphalt Program Data Base will be governed by the Technical Assistance Contractor. Procedures to assure control and publication priority by the experimenters who generate the data will be provided. However, it will be essential to maintain access to basic parameters and characteristics of the asphalts in the data base to assist the other experimenters in the design of subsequent experiments and projects.

4

Application of Experimental Design Concepts to the Research Coordinated by the A-001 Contractor

Need for Statistically-based Experimental Plans

Most researchers develop some sort of an experimental design before they start an experiment. This may be in the form of a formally developed written experiment protocol, or it may be in the form of a loosely thought out objective and loosely defined series of test measurements that are not committed to paper. Whereas the latter may be acceptable for exploratory or developmental experiments, a more formally stated objective and statistically sound experimental plan are required for the evaluation stages of contracts in the SHRP Asphalt Research Program.

It is important to distinguish between the developmental and evaluation stages. The developmental stage includes exploratory or probing experiments and experiments associated with the development of equipment and test procedures. The research activities in the developmental stage are not to be inhibited by unnecessarily rigid guidelines or requirements. Even in the developmental stages, however, the use of experimental designs and statistical methods is encouraged in order to improve productivity and enhance acceptance by others who are knowledgeable in the field. It is difficult to imagine an experiment of any kind which would not benefit from well developed experimental designs and statistical methods.

The evaluation stage includes experiments designed to validate relationships between test variables, to calibrate and validate test procedures and equipment, and to establish specification variables or criteria. In general, data entered into the Asphalt Program Data Base should be generated from experiments that are conducted with the aid of a formally developed experimental design.

Application of Experimental Designs

Statistically-based experimental plans are required in the evaluation stages of all of the SHRP Asphalt Research Program contracts co-ordinated by the TAC for several reasons

i. A written experimental design is a means of transmitting the experimenter's intent to other researchers who will be interacting with the experimenter. This will avoid unnecessary duplication of experimental effort and assist other researchers in developing their experimental designs.

ii. A written experimental design is a means of communicating the intent of the experimenter to the project statistician and to the designated A-001 statistician.

iii. The experimental design provides the basis and documentation for organizing the data for entry into the Archival Data Base.

The experimental plans are best developed jointly by the individual researchers and statisticians in the respective projects. A thorough review of the experimental designs by the project and TAC statisticians will ensure efficiency in the experimental work and will ensure that statistically sound conclusions can be drawn from the experiment when it is completed. With proper experimental design, clear distinction can be made between experimental error and true effects of the variables in the experiment.

Aspects of Good Experimental Design

The following aspects of good statistical experimental design should be an integral part of the research work plans of each contract.
These aspects include as a minimum:

1. independent replication of the procedures and processes used to prepare the samples for measurement in order to provide an accurate estimate of experimental error variance;

2. use of randomization where appropriate to avoid systematic biases caused by the order of measurement;

3. use of blocking and control variables where possible to explain and eliminate extraneous sources of variation (such as technician, day of measurement, batch of reagent, etc.) known to the experimenter;

4. wherever possible, blindness which provides coded labels for identifying specimens being measured so that technicians do not know the true identity of asphalt and replicate samples;

These design issues are summarized in Exhibit 1 and should be considered by each researcher during the process of developing an experiment design.

EXHIBIT 1: Issues to addressed in formulating the Statistical Experimental Design

REPLICATION

Independent replication of the research results requires that the complete set of procedures and processes used to obtain the samples and prepare the test specimens for measurement, be repeated along with the measurements.

RANDOMIZATION

The order of preparation and measurement of the test specimens should be randomized in each replicate set of tests.

BLOCKING

Extraneous sources of variation should be identified, (e.g. technician, day, and batch), and the study organized so that asphalts are crossed with these extraneous factors.

BLINDNESS

Whenever possible, coded labels will be used on asphalts to prevent the technician from identifying (1) the asphalt samples and test specimens and (2) detecting which samples and test specimens are replicates.

5

Guidelines for Development of an Experimental Plan

The guidelines in this document were developed based on the assumption that the research team for each contract will have a project statistician, as required by the contract, who will assist the researchers in generating the appropriate experimental designs. These designs will be reviewed and critiqued as necessary by the statisticians. It is expected that a wide range of statistical methods such as analysis of variance, linear and non-linear regression, and response surface methods will be used by the different research contractors. Each individual researcher will be given the opportunity to select the methods that are best suited to the individual research tasks, subject to review and approval by the TAC. Therefore, early n the development of their work plan, each contractor must submit a plan to the TAC indicating the experimental designs and analytical methods that will be used in the respective contract.

Example of an Experimental Design

A fictitious example of an experimental plan is presented in Exhibit 2. Although this is a relatively simple experiment, as shown in Exhibit 3, it illustrates the essential components of the plan and the information that should be submitted to the TAC for each experiment that is to be included in the Archival Data Base.

The hypothetical experiment described in Exhibit 2 consists of a 23 full factorial design and a control mixture without fibers, with 3 replications resulting in 27 test specimens. The three controlled variables are fiber length, diameter, and content. The response variables are resilient modulus and indirect tensile strength and strain at failure. Adoption of a factorial design with replicates will allow the researcher to make definitive statements about the effect of each of the main variables (fiber content, length, and diameter), interactions among the controlled variables, and experimental error. If possible, the data file, an example of which is given in Exhibit 4, should be submitted with the experimental plan when it is first written. At this point the data file, even though it is empty, serves as an announcement of the data to follow. This will be of use to other researchers in the planning of their work and will be of value to the statisticians in their review of the plan. The researchers should not consider the experimental plan to be final and not subject to change. It may, and should, be changed when warranted, but only after careful discussion with the statisticians.

Reference should be made to Exhibit 2 when developing an experimental plan and the general format given in Exhibit 2 should be adopted by each researcher. It should be noted that the data file contains three types of data as illustrated in Exhibit 4. The first might be called identification data to the extent that it identifies the individual specimen being tested. Included is such information as the specimen number, the asphalt and aggregate code, and the date of the testing. The second type of data specifies the control variables, ie., those that are fixed at different levels by the researcher during the course of the experiment. Test temperature, rate of loading, replicate number, and stress level for the resilient modulus test are examples of this type of data. The third type of data are the response variables,ie.,the response of the test specimen to the imposed test. The strength of the specimen and the resilient modulus are examples of this type of data

Relationship between Experimental Plan and Archival Data Base

Clearly there is a close relationship between the experimental plans and the Archival Data Base. The data files for each experiment may be generated by the experimenter by any means and with any software that is convenient for the experimenter. Computer based files should be transmitted to the TAC on floppy disc in ASCII format as flat files. Flat files are nothing more than a two dimensional array of numbers that may be generated by a spreadsheet or data base program in a personal computer, mainframe statistical analysis program, or even by paper and pencil with an accountant's pad. Although experimenters are encouraged to submit the data files in electronic format, if they do not have the means to do so, the TAC will accept paper copies.

The data file is not a replica of a laboratory notebook. Only those data elements that would be of interest to other researchers should be in the data file. The data file is more likely to contain reduced data rather than raw, unreduced test measurements. The data file should contain only information that other researchers may need when combining data sets and evaluating the combined data. Results for individual specimens or test runs should be reported as opposed to average values.

EXHIBIT 2: Example of Experimental Plan.

File Number: A0100001.TXT

Performing Agency: University of Texas

Project: A-001 Technical Assistance Contractor

Task 0.0: Use of Metal Fibers in Hot-Mix Asphalt

Subtask 0.0.0: Evaluation of Titanium Fibers

Experiment Name: Sample Experiment

Date First Entered into Database: June 31, 1989

Date Last Revision Entered into Database: Jan 23, 1990

Task Manager: J. Fiber
Researchers: S. Bitumen
Experiment Statistician: H. Variance

1. Objective

Task: The object of this task is to determine the effect of the addition of metal fibers on the physical properties of hot-mix asphalt concrete.

Subtask: The objective of this subtask is to study the effect of titanium fibers on the physical properties of hot-mix asphalt concrete.

Experiment: The objective of this experiment is to establish whether or not titanium fibers sufficiently enhance the properties of hot-mix asphalt to warrant further study. This experiment will also be used to determine the experimental error variance for the response variables. (Note: One cannot assume that the experimental error variance will be the same for the control (no fibers) and modified mixes.) If the results from this experiment are positive, a second experiment will be designed to optimize the mixture design variables and the length-diameter of the fibers.

Keywords: diametral resilient modulus, diametral tensile strength, diametral failure strain, metal fibers

2. Scope

In this experiment, a single mix (aggregate and gradation) and asphalt cement will be used to quantify the effect of adding titanium fibers to hot- mix asphalt concrete. Diametral resilient modulus and diametral tensile strength will be measured at one temperature to characterize the physical properties of the mixes. Two different fiber lengths and diameters will be studied at two levels of fiber addition.

EXHIBIT 2: Example of Experimental Plan. (continued)

3. Experimental Protocol

Specimen Preparation The specimens will be prepared in the customary manner (Asphalt Institute MS-1) except that the fibers will be added by hand to the hot mix immediately after mixing. The fibers will be heated to the mix temperature in a separate oven. Compaction of the 2 1/2-in by 4-in diameter specimens will be accomplished with a Fox Model 41-A gyratory compactor and 7% target in air void content.

Specimens will be tested after curing in air for 3 days at 77 F, +/-5 F. The resilient modulus and tension testing will be conducted at 77 F, +/-1 F with a Model 61 STM electrodynamic testing machine. Testing conditions are as follows:

- resilient modulus: Haversine load, 0.1 sec load time, 0.9 sec dwell between load applications

- indirect tension: Conducted immediately after resilient modulus testing, 10 mm/m crosshead speed

4. Experiment Design

Controlled Factors The following controlled variables and levels will be included in the experiment design:

- fiber length, 2 levels (0.5 cm, 1.0 cm)

- fiber diameter, 2 levels (0.005 mm, 0.01 mm)

- fiber content, 3 levels (0%, 0.05%, 0.1% by weight of total mix)

Response Variables Response variables measured for each specimen will include the following:

- resilient modulus at 77 F, 0.1 s haversine load, 0.9 s dwell time, calculated as peak to peak stress divided by the recoverable strain

- indirect tensile strength and strain at failure (at maximum tensile stress)

5. Statistical Design

A full factorial design with three replicates will be used for this experiment. For each replicate all specimens will be prepared on a single day according to the experiment plan. The run order of preparation of the specimens will be randomized, independently for each day (replicate). After the hree day curing period, the specimens will be measured in a random order.

EXHIBIT 2: Example of an Experimental Plain (continued)

Note: After preparation the specimens will be labeled with a randomly assigned code number. This will provide the random order for the measurements, and blinds the technician to the identity of the specimens.

Analysis of variance will be used to identify the magnitude of the effects of the controlled variables on the response variables and to determine the experimental error variance for the control and modified mixes. In addition regression models will be used as appropriate to quantify the effects of the controlled variables on the responses.

6. Anticipated Results

The results of this experiment will be used to determine the effect of adding titanium fibers on the resilient modulus and strength properties of a hot-mix asphalt concrete mixture. The results of this experiment will be used to determine if titanium fibers merit further study as a reinforcement for hot-mix asphalt.

7. Conclusions

To be added when data analysis is completed.

8. Data Description

001.D01 Asphalt number
002.D01 Aggregate number
003.D01 Laboratory sample identification number
004.D01 Replicate specimen number
005.D01 Length of fiber, cm
006.D01 Diameter of fiber, mm
007.D01 Percent of fiber added to mix based on weight of total mix
008.D01 Date when mix was made
009.D01 Mixing temperature, F
010.D01 Bulk specific gravity, ASTM D 2726
011.D01 Maximum theoretical specific gravity, Rice Method, ASTM D 2041
012.D01 Percent air voids in compacted mixture, [100]*[(11)-(10)]/(10)
013.D01 Peak to peak stress in Resilient Modulus Test, psi, MARK IV Schmidt apparatus, ASTM D 3497
014.D01 Recoverable strain - measured 0.1 second after release of load
015.D01 Resilient modulus, [13]/[14], ksi
***The above data are contained in data file with extension .D01
016.D02 Tensile strength, tensile stress at peak load, psi, SHRP APTM 00
017.D02 Failure strain defined as the diametral strain at peak load, in/in, using SHRP ATPM 00
***The above data are contained in data file with extension. D02

EXHIBIT 3: Schematic of Experimental Design

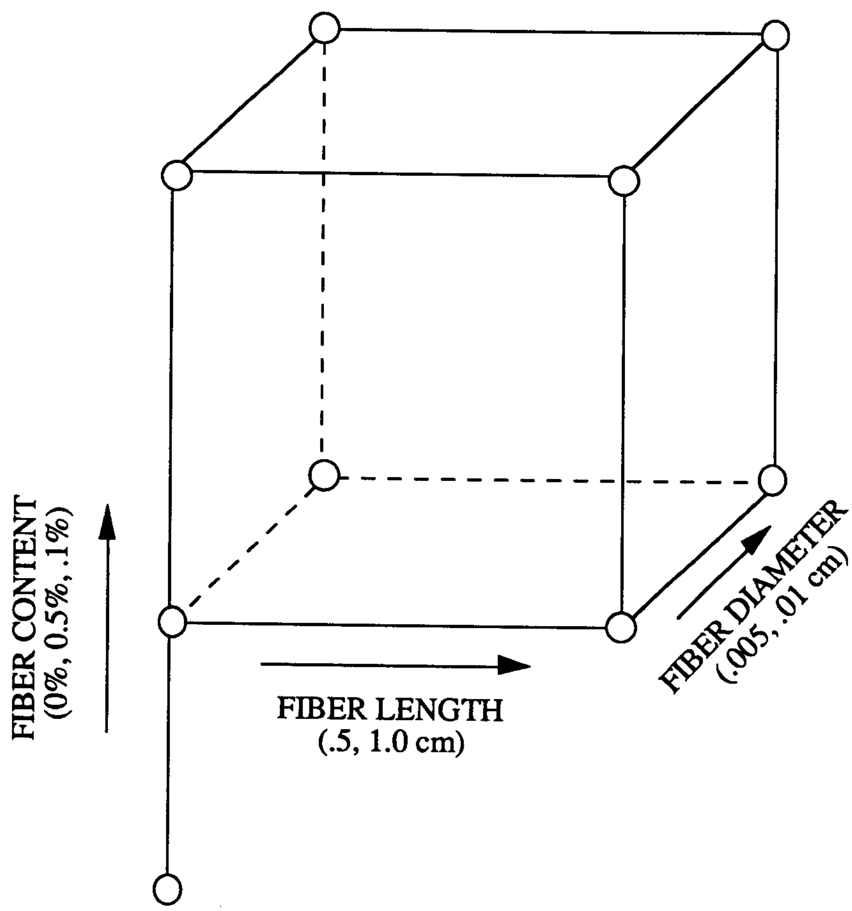

EXHIBIT 4: Example of an Experimental Data File.

A01SAMPL.D01

12/15/89
01/15/90
R.Stone
Closed

A01SAMPL.D02

12/15/89
01/15/90
R.Stone
Closed

Line	1 Asph ID No.	2 Agg ID No.	3 Specimen ID No.	4 * Repl No.	5 Fib Len (cm)	6 Fib Diam (mm)	7 Fib Level (%)	8 Date Specimen Mixed	9 Mix Temp (F)	10 Bulk Sp Gr	11 Theo Sp Gr	12 Mix Voids (%)	13 Diam Stress (psi)	14 Diam Strain (in/in)	15 Diam Mr (ksi)	Line	16 Ten Stren (psi)	17 Fail Strain (%)
7	AAA1	RA	12000891	1	.00	.000	.00	12/20/89	325	2.382	2.457	3.1	104	.000231	450	7	367	1.45
8	AAA1	MM	12000892	2	.00	.000	.00	12/20/89	325	2.379	2.439	2.5	99	.000248	399	8	357	1.39
9	AAA1	MM	12000893	3	.00	.000	.00	12/20/89	325	2.374	2.458	3.5	101	.000271	373	9	345	1.68
10	AAA1	MM	12AAA891	1	.50	.005	.05	12/20/89	*	*	*	*	*	*	*	10	*	*
11	AAA1	MM	12AAA892	2	.50	.005	.05	12/20/89	*	*	*	*	*	*	*	11	*	*
12	AAA1	MM	12AAA893	3	.50	.005	.05	12/20/89	*	*	*	*	*	*	*	12	*	*
13	AAA1	MM	12AAB891	1	.50	.005	.10	12/21/89	*	*	*	*	*	*	*	13	*	*
14	AAA1	MM	12AAB892	2	.50	.005	.10	12/21/89	*	*	*	*	*	*	*	14	*	*
15	AAA1	MM	12AAB893	3	.50	.005	.10	12/21/89	*	*	*	*	*	*	*	15	*	*
16	AAA1	MM	12ABA891	1	.50	.010	.05	12/21/89	*	*	*	*	*	*	*	16	*	*
17	AAA1	MM	12ABA892	2	.50	.010	.05	12/21/89	*	*	*	*	*	*	*	17	*	*
18	AAA1	MM	12ABA893	3	.50	.010	.05	12/21/89	*	*	*	*	*	*	*	18	*	*
19	AAA1	MM	12ABB891	1	.50	.010	.10	12/19/89	*	*	*	*	*	*	*	19	*	*
20	AAA1	MM	12ABB892	2	.50	.010	.10	12/19/89	*	*	*	*	*	*	*	20	*	*
21	AAA1	MM	12ABB893	3	.50	.010	.10	12/19/89	*	*	*	*	*	*	*	21	*	*
22	AAA1	MM	12BAA891	1	1.00	.005	.05	12/19/89	*	*	*	*	*	*	*	22	*	*
23	AAA1	MM	12BAA892	2	1.00	.005	.05	12/19/89	*	*	*	*	*	*	*	23	*	*
24	AAA1	MM	12BAA893	3	1.00	.005	.05	12/19/89	*	*	*	*	*	*	*	24	*	*
25	AAA1	MM	12BAB891	1	1.00	.005	.10	12/18/89	*	*	*	*	*	*	*	25	*	*
26	AAA1	MM	12BAB892	2	1.00	.005	.10	12/18/89	*	*	*	*	*	*	*	26	*	*
27	AAA1	MM	12BAB893	3	1.00	.005	.10	12/18/89	*	*	*	*	*	*	*	27	*	*
28	AAA1	MM	12BBA891	1	1.00	.010	.05	12/18/89	*	*	*	*	*	*	*	28	*	*
29	AAA1	MM	12BBA892	2	1.00	.010	.05	12/18/89	*	*	*	*	*	*	*	29	*	*
30	AAA1	MM	12BBA893	3	1.00	.010	.05	12/18/89	*	*	*	*	*	*	*	30	*	*
31	AAA1	MM	12BBB891	1	1.00	.010	.10	12/20/89	*	*	*	*	*	*	*	31	*	*
32	AAA1	MM	12BBB892	2	1.00	.010	.10	12/20/89	*	*	*	*	*	*	*	32	*	*
33	AAA1	MM	12BBB893	3	1.00	.010	.10	12/20/89	*	*	*	*	*	*	*	33	*	*

Appendix A.

Examples of Issues to be Addressed in the Development of Experimental Designs

In the sections that follow, several examples are given that illustrate the application of statistical principles as applied to an experimental design. These examples are not complete experimental plans but illustrate some of the issues that are likely to be addressed during the development of an experimental plan for the Asphalt Research Program.

1. Example of an Experiment for Developing a Physical Property Test

This example presents a simple experimental design that will be performed as part of the A-002A project. The purpose of this experiment is to develop a procedure for measuring certain physical properties of asphalt and, depending on the suitability of the procedure, it will then be applied to all 32 of the asphalts in the Materials Research Library (MRL).

In this experiment, the response of a loaded beam is used to measure the stiffness of asphalt cement. The experiment will be carried out at two temperatures using two different asphalt cements. The linearity of the stiffness will be established by testing at three load levels.

The objectives of this experiment are as follows:

i) to evaluate the effects of certain factors (e.g. length/depth ratio, etc.) on the performance of the measuring process; and

ii) to evaluate the variance of the experimental error.

When the experiment has been completed, these objectives should be achieved and a decision will be made at that time as to whether or not the process is ready for application to the entire set of 30 asphalts in the Materials Research Library, or if further experimentation is needed.

This experiment will consist of two replications of a complete 23 x 3 factorial experiment. A minimum of two replicates is desirable to provide a valid estimate of experimental error variance. The measured response will be the displacement of the beam as a function of time after loading.

The factors and their levels are as follows:

i. asphalts - two chosen to represent two different rheological types;

ii. aspect ratio (length/depth ratio) of the test beam - two levels chosen;

iii. temperature - two levels chosen (one at the low end and one at the high end of the temperature range); and

iv. load level - three levels chosen, (a low, medium, and high).

One replication of this experiment requires the preparation of 24 samples. These 24 samples will be prepared and tested in a random order before the second replication of 24 samples is prepared. In this way, the replications are true replications and will provide a valid estimate of the experimental error variance. This evaluation of the "repeatability" of the measuring process should be done early in the experimental program, as discussed in Chapter 5.

The data from this experiment will consist of the responses, (i.e. the displacement by time) and the fixed levels of the control variables. For a convenient statistical analysis it will be necessary to first reduce the response portion of the data to one or two calculated fundamental properties related to stiffness or rheological properties. These are the response variables that will be used in a regression analysis from which the following questions are among those that may be answered

i. Is there a strong effect due to the aspect ratio?

ii. Is there a strong effect due to the load level? Is this effect a linear effect?

iii. Does the effect of the aspect ratio depend upon the asphalt used? This is the interaction effect of asphalt and aspect ratio.

iv. Does the effect of the load level depend upon the aspect ratio? This is the interaction effect of load level and aspect ratio.

v. Does the variance of the experimental error depend upon the temperature, rheological type or aspect ratio?

While this experiment may be regarded as exploratory, it is one for which a common statistical design (2 replications (blocks) of a completely randomized 23 x 3 factorial experiment) will be most cost effective. It is generally true that factorial experiments provide more information for a fixed cost than any other method of experimentation. Factorial designs are in contrast to the "one factor at a time" method where all other variables are held constant and one variable is systematically varied. The systematic variation is usually repeated for all of the variables. This "one factor at a time" experimental technique does not generally provide cost-effective results, and, if there are interactions, they are likely to be missed. In fact, these interactions are of extreme importance and often explain the differences observed by two different researchers.

2. Experimental Design Example for Model Development

This example is given for the purpose of illustrating the need for a complete set of measurements on all 32 of the asphalts whenever the relationships between the physical properties are being modeled in terms of the chemical and compositional properties (Box and Draper, 1987). Artificial data are generated for this example but the language is appropriate for the entire A-002A project, and the ideas and results would be appropriate for several of the projects in the Asphalt Research Program.

One of the primary objectives of the Asphalt Research Program is to develop statistical models that will relate the measured physical properties of the asphalts to their measured chemical and compositional properties. This second example demonstrates the importance of providing a complete data base. When the researchers begin to build and evaluate models relating the physical properties of the asphalts and mixtures to the chemical and compositional properties of the asphalts, it is necessary to have all the important variables measured for all 32 asphalts.

As measurements of the physical, chemical, and compositional properties of the asphalts being studied are added to the Asphalt Program Data Base, the process of building models which relate these properties will begin. This is one of the most important, perhaps the most important, product to be produced in the program. Data from the Archival Data Base will be of the most value in model building. The question regarding the sufficient amount of data is answered in terms of how many asphalts have a complete set of measurements, and how many chemical, compositional, and physical properties were measured. In this section one evaluates the importance of obtaining the complete set of measurements on as many asphalts as possible.

For convenient to let Y be a measured physical property of interest and let X_1, X_2 , ..., X_5 be five chemical and compositional measurements whose effects upon Y are being studied. Suppose the true relationship is given by

$$Y = .3X_1 + .25X_2 + .2X_3 + .15X_4 + .1X_5 + \text{error}$$

Certainly it is not likely that the true relationship will be this simple, but this will illustrate the need for more than the measurements from eight asphalts. Now suppose the measured chemical and compositional properties of the eight asphalts are as given in Exhibit 5. With these values for the X's, values for the Y's were generated from the assumed simple model using a variance of the error term of .03. The resulting fitted equation was

$$Y = -.55 + .62X_1 + .59X_2 + .36X_3 + .30X_4 + .25X_5$$

If this fitted equation is used to predict the true model values, the mean square error is .07.

Now suppose the measurements were done on 32 asphalts. An artificial set of X's was generated for the additional 24 required for this illustration. The resulting fitted equation was

$$Y = -.13 + .35X_1 + .33X_2 + .17X_3 + .18X_4 + .05X_5$$

This fitted equation is much more like the assumed true model. If it is used to predict the true model values, the mean square error is .054.

Now it is clear that the additional asphalts have improved the fitted equation by an appreciable amount. In addition, it must be noted that the important question of constructing and evaluating the forms of the model is not considered. With only eight data points it would hardly be possible to evaluate the form of the model.

In conclusion, it seems clear that the success of the model building and parameter estimation process will be totally dependent upon the quality and quantity of the measured properties of the asphalts and every effort should be made to complete this set of measurements for all 32 of the asphalts in the Asphalt MRL.

3. Experimental Design to Illustrate Blocking

Blocking is a technique useful for improving the precision of an experiment by removing a known source of variation. A block is a part of the experiment in which the experimental material and/or measurements are more homogeneous than the rest. Examples of blocks are the runs conducted during a single day, the runs made using the same batch of raw material or reagents, and runs prepared and made by the same operator. These factors (day, batch, and operator) are typically not of particular interest to the experimenter, although they may have a real effect on the measured results. The measurements may be higher or lower for all runs within a block, and the benefits of blocking are accomplished by removing this variation from the experimental error variance.

An example from a task of A-002A will be used to illustrate how blocking can produce considerable benefits with little additional effort. The objective of this experiment is to test the hypotheses that the tensile strength properties of asphalt cement are uniquely related to the test temperature and rate of loading. An additional objective is to develop a statistically sound protocol, to be used in future experiments for determining the fracture properties of aged and unaged asphalt.

The measurement process requires the preparation of dogbone shaped specimens of asphalt prepared under a detailed protocol. Each sample will then be measured in an Ingstron machine under preset temperature (5 levels) and deformation rate (5 levels) conditions. Since the direct tensile strength measurements are known to be quite variable, several replicates of the experiment are planned. In addition, blocking these measurements into sets of runs will also improve the precision of the measurements.

For this experiment the temperature levels will be -30 F, -20 F, 0 F, 20 F, and 40 F. The deformation rates will be .1 in/min, 1 in/min, 2 in/min, 5 in/min, and 10 in/min. All combinations of temperature and strain rate will be used so that one replication of this factorial experiment will require the preparation and measurement of 25 specimens. For each of the aged and unaged specimens there will be 3 complete replications for a total of 150 specimens.

The experiment will be carried out by the preparation of 25 specimens on each of days 1, 2, and 3 and then on day 4 (as called for by the protocol) the complete set of specimens prepared on day 1 will be tested. Likewise, for the specimens prepared on day 2 and 3, they will be tested on days 5 and 6 so that there is a three day delay for each set of prepared specimens. At the beginning of each test day all calibrations will be checked and adjusted as needed in order to maintain the independence required for true replications of the experiment.

Blocking this experiment corresponds to running all temperature and deformation rate measurements in one day on samples prepared under constant conditions in one day. Day to day variation in the measurements and the preparation can thus be eliminated from the experimental error variance resulting in more precise information about the asphalt properties. In addition, no additional effort was required in terms of the number of specimens tested, except to insure that the replications were independently prepared and measured on different days. Although not illustrated here, when the number of treatment combinations and asphalt types exceeds the number of runs which can be done by a single operator in a single day, then an incomplete block design may be desirable (Box, Hunter, and Hunter, 1978).

Exhibit 5. Values of the Measured Chemical and Compositional Properties (X_1 - X_5) for the 8 Core Asphalts

Asphalt Number	X_1	X_2	X_3	X_4	X_5
1	0	1	.2	.8	.4
2	.1	.05	.5	.55	.5
3	0	.85	.5	.9	.65
4	.1	.9	.25	.75	.3
5	.9	0	.4	.8	.1
6	1	.3	.3	.2	0
7	.4	.6	.75	.5	.9
8	.7	.4	.55	.3	.75

References

1. G. E. P. Box, W. G. Hunter, and J. S. Hunter. Statistics for Experiments An Introduction to Design, Data Analysis, and Model Building. Wiley, 1978.
2. G. E. P. Box, and N. R. Draper. Empirical Model-Building and Response Surfaces. Wiley, 1987.
3. W. G. Cochran and G. M. Cox. Experimental Designs. Wiley, 1957.
4. D. C. Montgomery. Design and Analysis of Experiments. 2nd ed. Wiley, 1984.
5. ASTM E-177-86 "Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods." 1986 Annual Book of ASTM Standards, Construction. ASTM, Philadelphia, Pennsylvania.