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Statistical Consulting in the Frame of VAMAS

The Role of the Technical Working Area / Advisory Group

Statistical Techniques for Interlaboratory Studies and Related Projects

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PREFACE

The present Technical Report was written with the intention of fulfilling four needs: (i) for an introduction of research workers in the Technical Working Areas (TWAs) of VAMAS to modern statistical methods and techniques appropriate to improve the effectiveness of experimental research in the scope of the VAMAS co-operative projects in materials science and engineering; (ii) for illustrating the power of such approaches by giving an outline of more recent applications in science and technology; (iii) for developing a feeling on the role and importance of statistical consulting under these circumstances; and (iv) for presenting objectives and a programme to provide and promote statistical consulting within VAMAS by developing the TWA / Advisory Group 'Statistical Techniques for Interlaboratory Studies and Related Projects'.

Statistical consulting in the broadest sense of the word amounts to a co-operation of one or more statisticians with scientists or engineers who need statistical assistance to solve a certain problem. In more sophisticated situations, the treatment of the problem can require additional research or the development of computer programs on the statistical side. On the other hand, there are situations in which statistical consulting reduces to the immediate response of a statistician to a telephone query. Between these two limiting situations, there is a variety of scenarios which lead to different requirements on the statistical side of such a co-operation, for example, situations in which the computer-aided application of existing standards is sufficient.

It is obvious that effective statistical consulting requires specific skills. Apart from the statistical knowledge, a consultant must be able to understand the scientific or engineering side of such a problem and to help formalizing it. Moreover, he must be able to communicate with his clients, locate, adapt or develop procedures in a timely fashion and use computers effectively.

In particular, each interlaboratory experiment demands such an intimate co-operation between statisticians and experimenters with equal responsibility for both. This refers likewise to the main phases of such an experiment: experimental design, design and preparation of samples, organization, instruction and execution of the experiment, and analysis, interpretation and discussion of the results, where in corresponding materials science and engineering experiments the requirement of distributing identical test material can lead to a massive sophistication on the statistical side of such an experiment.

The present report considers the indicated topics more detailed. Apart from the Introduction (Chapter 1), which reviews, in particular, first approaches to a classification of interlaboratory studies, Chapter 2 analyses the objectives and means of statistical consulting within VAMAS. Chapter 3 outlines the preliminary organizational activities and Chapter 4 is essentially a review of more recent interlaboratory studies in science and technology. Moreover, Appendix 1 contains a reprint of the minutes of the initial meeting of the TWA / Advisory Group and Appendix 2 summarizes objectives, activities and results.

The first mentioned author is grateful to Prof. Dr.-Ing. M. Pfender, president of the Federal Institute for Materials Testing, 1954-1972, for drawing his attention to the organizational, statistical and standardizational aspects of interlaboratory studies (DIN 51 848: Testing of mineral oils: testing errors) in 1968. The authors would also like to thank Prof. Dr. Dr. h.c. H. Czichos, president of the Federal Institute for Materials Research and Testing, member of the VAMAS Steering Committee, for initiating the present project and encouraging the preparation of this report. Moreover, the authors are indebted to the VAMAS Steering Committee under the chairmanship of Dr. K. Hossain (National Physical Laboratory, Teddington, UK) for promoting this project at the Tokyo meeting, 1991, and the Berlin meeting, 1992, of this committee. At different stages of the organizational work the authors received also advice and particular support from Mr. Y. Maezawa (Science and Technology Agency, Tokyo, Japan), Mr. G. D. Quinn, chairman of TWA 3: Ceramics (National Institute of Standards and Technology, Gaithersburg, USA) and Mrs. K. Riley (Institute of Standards Research, American Society for Testing and Materials (ASTM), Philadelphia, USA). Also, the authors should like to mention gratefully that the iso bulletin was placed at their disposal regularly by Dr. C. J. Favré (International Organization for Standardization (ISO), Geneva, Switzerland).

The main part of this report was in the focus of the Berlin initial meeting of the TWA / Advisory Group. For helpful discussions and constructive comments, the authors wish to express their gratitude to Dr. C. Amzallag (IRSID SNC, Group USINOR-SACILOR, Firminy, France), Prof. Dr. K. Doerffel, (Technical University 'Carl Schorlemmer', Leuna-Merseburg, FRG), J. Häyrynen, Technical Research Centre of Finland, Espoo, Finland), W. J. Muster (Swiss Federal Laboratories for Materials Testing and Research, Dübendorf, Switzerland), Dr. M. A. Nemeth (Monsanto Co., St. Louis, USA), Prof. Dr. K. Suzuki (University of Electro-Communications, Tokyo, Japan), Th. Svensson (Swedish National Testing and Research Institute, Borås, Sweden), and Dr. L. Wolstenholme (City University, London, UK).

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As data become more complex, so do the methods for examining data sets.

St.D. Brown, R.S. Bear, Jr., and Th.B. Blank

I never lose a chance to get experience with data; I enumerated a district in the Census 1940; I've been out on interviews at least 40 different times on Census work, labor force, social surveys, market research; I've used the telephone; I've collected data on hundreds of physical and chemical trials and on reliability and testing and inspection in plants; to me this experience is extremely important.

W.E. Deming

INTRODUCTION

1-1 INTERLABORATORY STUDIES AND VAMAS STANDARD TEST METHODS. As in numerous fields of science and technology, interlaboratory studies are developing into an important tool of research and testing in connection with the collaborative projects of VAMAS [1 - 5]. With respect to advanced materials, this is especially true for activities in the field of pre-standards measurement research aiming at the improvement of the intercomparability of test results, the development and evaluation of standard test methods, the computerization of materials data [6], in particular the compilation, computerization and networking of corresponding databases [7, 8], and the preparation of internationally acceptable standards and specifications [9, 10]. According to [11], it is, in particular, the need for standardized test methods which has long been identified as an important priority. While for metallic materials test methods are well established, for brittle materials (technical ceramics) [12, 13], composites and other advanced materials there is considerable difficulty at present in achieving consensus on such methods, so that there are often large disagreements on results and also on what is actually being measured by a non-standard method. - For terminological and other details of test methods, see [14].

1-2 EXPERIMENT AND THEORY. Apart from those fields of science and technology which apply directly to materials science and engineering [15], more precisely, to the development, production and processing of advanced materials [16, 17], an effective application of the mentioned experimental and statistical tool in testing and standardization of this kind of materials assumes, furthermore, an intimate interaction of the experimental branches of the scientific and technological fields, relevant in the present circumstances, with their theoretical counterparts and important auxiliary sciences like mathematical modelling and computer simulation [18, 19], so that some kind of balancedness is established between experiment, theory and computer experiment [20].

1-3 ENGINEERING PROPERTIES OF ADVANCED MATERIALS. Since, in particular, engineering properties, i.e., mechanical and thermophysical properties, of advanced materials are, at present, in the focus of research, development and testing, the theoretical contribution of applied mechanics to the knowledge on important classes of advanced materials deserves special consideration. For example, the branch of composite materials mechanics investigates the performance and behaviour of particulate, fiber reinforced, and layered materials, together with corresponding structural elements, from the viewpoints of statics, dynamics,

stability, fracture, and damage processes. An essential part of this branch is micromechanics. It allows a prediction of macro-phenomena from the mechanical processes and properties at the micro-level [21 - 24].

More precisely, such an interaction between experiment, theory and computing in the field of applied mechanics, in particular with respect to microstructural phenomena, continuum level phenomena and behaviour in service, forms the basis of further progress in constitutive modelling of advanced materials, providing the necessary constitutive laws [25] for a computational transfer of properties from the macro-level to the level of mechanical engineering projects and showing again the role of standard test methods for the present class of materials and the importance of interlaboratory research in this area.

1-4 STATISTICAL CONSULTING AND RESEARCH ON ADVANCED MATERIALS. Basic and more general aspects of statistical consulting are considered in [26, 27]. As indicated in the Preface, the ability of a consultant to understand the scientific or engineering side of a problem under consideration is a skill of particular importance.

More precisely, there is no doubt that a statistician, who intends to co-operate with an experimenter in conducting an interlaboratory study from the field of advanced materials, should have, at least, basic knowledge on properties, testing, failure modes and fracture of this kind of materials, observing that [28] mentioned the following working fields for the present phase of VAMAS:

- metals and metal matrix composites;
- polymers and polymer matrix composites;
- ceramics and ceramic matrix composites;
- non-material specific test techniques;
- materials classification and data.

With respect to composites, the study of monographs or handbooks like [29 - 39] is recommendable. Science and technology of this kind of reinforced materials are considered in journals like [40], as well. An outline on metal matrix composites is given in [41]. This kind of materials is treated more detailed, for example, in the monographs and handbooks [42 - 47]. Advanced ceramics are considered, for example, in [48 - 53]. From the viewpoint of abstracts, this field is covered by [54]. For metals like superalloys, it is referred to [55].

The whole field of advanced materials is covered by handbooks according to [56 - 58]. The field of higher temperature superconductivity is in the focus of [59]. Tribological examples from composite materials mechanics and other advanced materials subjects are contained in [60 - 62]. - For more recent papers from English language periodicals, see [63].

1-5 INTERLABORATORY RESEARCH. From the viewpoint of analytical chemistry, the following explication of the notion 'interlaboratory research' was given [64]:

The measurement of the same quantity in different laboratories according to a pre-established programme. - Interlaboratory research is conducted mainly on

- estimation of the true (real) value (analysis of standards);
- estimation of the repeatability and reproducibility of any analytical method;
- analysis of the suitability of a method for the given analytical task.

Obviously, by this explication the notion under consideration is subjected to several unnecessary restrictions, for example, to the consideration of simple univariate, measurable quantities.

Preserving this main restriction and considering realizations of the concept of the generic interlaboratory study in science and technology under aspects of purpose, importance, structure and complexity of the underlying model, a distinction between 'strict sense' and 'wide sense' interlaboratory studies is suggested. Such a distinction allows, at least, some more and wider insight into the field of 'univariate' interlaboratory research. Moreover, it serves for organization and clarification. - Observe, especially, that the conduct and analysis of an interlaboratory study can be highly affected in situations in which the test material representing the quantity under consideration is altered by the test or measurement (destructive testing).

1-5-1 Strict Sense Interlaboratory Studies. According to different purposes, one can discriminate in the first case between five different types of interlaboratory studies [65], where the first and, to a certain extent, the second and the fifth type seem to be of particular importance from the viewpoint of the development of standard test methods and other VAMAS activities:

- Collaborative Studies: determination of performance characteristics of test methods [66 - 70];

- **Comparative Studies:** comparison of the performance of several test methods [71];
- **Proficiency Studies:** determination of the performance of laboratories [72 - 76];
- **Consensus Studies:** determination of an estimate of the 'true value' of a material;
- **Certification Studies:** assignment of a 'true value' to a material with a stated uncertainty [2, 77 - 80].

1-5-2 Wide Sense Interlaboratory Studies. Interlaboratory research and testing, however, produced numerous additional kinds of interlaboratory studies. For example, the conduction of large-scale investigations with interlaboratory character can be observed inside and outside of VAMAS, in the scope of which test methods are applied with respect to which the factor 'laboratory' is negligible, such that test conditions and specimens can be distributed arbitrarily to the participating laboratories within the frame of such a study. In most of these cases regression is an appropriate method of analysis and the term 'distributed study' can be used to characterize the situation.

Examples of particular frequency are studies which aim at a simple interlaboratory comparison of experimental results and corresponding uncertainties [81 - 84], given a certain material, possibly via sample exchange [85], which has to be analysed by one and the same method or by different methods. Obviously, the execution of such a wide sense interlaboratory study can be helpful during the preliminary phase of all types of strict sense interlaboratory studies, as well. In particular, a thorough investigation of measurement errors or other adverse influences can take place in the frame of such a study. Moreover, it can focus the development and comparison of specimens or aspects of the calibration of specimens or test devices [86], providing for traceability.

Apart from materials or test methods, the behaviour of mathematical objects can be focussed in the scope of interlaboratory comparisons, too. This is true, for example, for statistical models and procedures of analysis.

Co-operative interlaboratory studies, as recommended in [87] and applied according to [88] in the field of food safety, are more sophisticated examples of wide sense interlaboratory studies in the frame of which several laboratories analyse a series of specimens for a certain property by any appropriate, however not prescribed method, where the specimens are sub-samples from bulk material. The statistical approach for an analysis of this kind of studies is of nested analysis of variance type and aims at a realistic quantification of random

and systematic errors in the system. The design of such a study has elements from strict sense interlaboratory studies of the first and the third type.

Corresponding wide sense interlaboratory studies are characterised by their individual purpose, structure, organization and analysis [89, 90], which indicates the occurrence of individual statistical questions and problems, showing again the importance of statistics, in particular computational statistics, under the present circumstances. Note, finally, that, up to now, there is no corresponding classification in the variety of such interlaboratory studies.

1-5-3 Remark. An outline of activities to promote and standardize interlaboratory research, together with more detailed informations on the importance of strict sense and wide sense interlaboratory studies in science, technology, and industry will be given in Chapter 4.

1-6 VAMAS INTERLABORATORY STUDIES AND STATISTICS. Referring to interlaboratory studies in the frame of VAMAS and the corresponding state-of-the-art as given in Chapter 4, this means that bridge works are required which, on the one hand, make accessible knowledge from standards and guidelines together with experiences from interlaboratory studies in other fields of science and technology, in particular from industrial studies, and in the scope of which, on the other hand, more advanced and specific applicable knowledge is developed from computational statistics and related computational fields.

1-7 STATISTICAL CONSULTING AND OBJECTIVES OF THE TWA / ADVISORY GROUP. Therefore, the activities of the TWA / Advisory Group aim in a natural manner at four topics which can be regarded as topics of a statistical program accompanying the experimental work of VAMAS in all fields directed to the development, engineering and testing of advanced materials, in other words, a program which promotes the enhancement of the communication between experimental materials science and the relevant computational fields on these kinds of materials in the field of interlaboratory research:

- Support of the work of other TWAs by offering an overall service in the field of interlaboratory studies, including statistical computer simulation, to increase the effectiveness of such studies;
- Optimization of specimens and test devices to be analysed in the frame of interlaboratory studies by application of classical and more recent design methods (Taguchi) or mathematical modelling and computer simulation, observing the requirements of available models and approaches, as well.

- Problem-oriented statistical research in the field of interlaboratory studies to improve the structure and the applicability of existing models and approaches, including the development of corresponding computer programs;
- Consulting in all fields of mathematics and computational statistics, in particular, in connection with applied subjects, including certain fields of chemometrics.

This assumes that sufficient specialized and qualified members of the TWA / Advisory Group can be interested in statistical consulting and, in particular, in the task of a further maturing of the general concept of interlaboratory studies and its VAMAS advanced materials realizations.

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STATISTICAL CONSULTING

In the present Chapter an attempt is made to give some impression of the rich diversity of facilities available for statistical consulting in the frame of VAMAS. Especially, emphasis is given to an illustration of the objectives of the TWA / Advisory Group, as set up in the Introduction. A collection of research proposals is presented to make plausible the importance of problem-oriented statistical research in supporting collaborative engineering projects and to assist in better understanding of the role of research in the scope of statistical consulting of the experimental TWAs of VAMAS.

2-1 SERVICE: FOCUS ON COLLABORATIVE STUDIES. In the scope of strict sense interlaboratory studies, collaborative studies are of special importance. In the present outline, their affairs are focused, without restricting the generality, under the assumption that identical test material (see Section 4-1) can be distributed, to give an example of that part of statistical consulting which amounts to immediate statistical and computational support required for all kinds of interlaboratory studies. Recall that this type of studies aims at the investigation of performance characteristics for methods of test or analysis, for example, at the determination of precision data, provided that there are significant laboratory effects.

2-1-1 Design and Statistical Model. A service which provides for competent guidance is of particular importance in the design phase of collaborative and all other kinds of interlaboratory studies. Apart from preliminary considerations on method, material and participating laboratories, conducting a collaborative study starts, commonly, with considerations on appropriate designs. This is the first point, where statistical knowledge and knowledge of corresponding standards or guidelines is required: As well laboratories as materials, more precisely true values of characteristic properties of materials, to be included in such a study, can be considered as a random sample of a corresponding population. On the other hand, the experimenter can be interested only in a set of concrete laboratories or in a set of concrete materials. In this way, random effects, fixed effects or mixed models occur. Corresponding approaches are mainly based on the models of the random effects one-way layout or the random effects two-way layout. In certain cases, for example, if the materials to be included are test devices consisting of several materials, the model of a three- or n-way layout can be appropriate [1, 2]. A similar assertion is true with respect to nested, or crossed and nested designs [3 - 6]. The types of

these models are relevant in connection with the statistical analysis of the results from the collaborative study to be performed.

Secondly, if a collaborative study is executed in the frame of the development of a test method, materials have to be included which allow an assessment of that test method in relation to its discriminating power [7].

2-1-2 Size of Design and Computer Simulation. A third important point in this pre-experimental phase is the determination of the size of the collaborative study, observing the requirements that the expected performance characteristics, for example precision data estimates and the estimates in connection with the discriminating power, should be sufficient precise and that the final design should be the most cost-efficient one. Here, the results from small pilot experiments, prescribed by certain standards, can serve as input data for statistical computer simulations. Such simulations allow a prognosis of the actual precision of the precision data estimates and the discriminating power values to be obtained from the experimental results, given values of the number of laboratories and the number of materials to be included in the study. In addition, the (small) influence of the number of replicates can be seen.

2-1-3 Analysis and Results. While in this design stage closest co-operation between experimenter and statistician is necessary, in the following analytical stage, the proposed service assumes that the statistician has to provide for suitable techniques, commonly of the analysis-of-variance type, standards, computer programs and computer facilities which allow an analysis of the results of the executed collaborative study. This stage includes the performance of the computations of the required quantities.

Closer cooperation as part of such a service is again necessary in the stage in which the obtained experimental and computational results have to be interpreted. This interpretation considers, usually, stragglers and outliers with respect to the included laboratories and materials and amounts, finally, to an overall assessment of the obtained results and a discussion of their applicability and use, for example, in connection with the intercomparability of test results, the development of a test method, or the preparation of a standard.

2-2 OPTIMIZATION OF SPECIMENS AND TEST EQUIPMENT. The planning of an acceptable efficient interlaboratory study can include in certain cases additional experimental and theoretical, possibly, computer-aided preliminary investigations, which are necessary to provide for test specimens, sometimes additionally for measurement systems or test devices [8, 9], to guarantee an optimal

behaviour in respect of the results expected from the interlaboratory study under consideration, where one has to observe, possibly, the standard character of a test method under development and requirements for a corresponding certification procedure.

2-2-1 Specimens for Friction and Wear. Observe, for example, that the specimen geometry can require such an adjustment. - On the other hand, test specimens and test devices in the case of certain VAMAS interlaboratory studies on friction and wear form highly complicated and sensitive vibrational systems which require measures [10] to guarantee that these systems behave, at least approximately, in the same manner in all participating laboratories, such that additional errors from this source can be neglected.

2-2-2 Nonlinearity and Chaos. Since, moreover, it cannot be excluded that these systems behave nonlinear and since it is well-known that already elementary nonlinear mechanical vibrational systems can behave chaotic, this example, together with the fact that chaotic behaviour was traced already in connection with rolling friction [11], gives an impression from the extent and the depth to which preliminary investigations of this kind can be necessary to obtain sufficient intercomparable test results and acceptable small precision data estimates. Note, in addition, that the phenomenon 'chaos' seems to pollute the results of interlaboratory studies and corresponding precision data in many fields of materials research and testing [12, 13].

2-2-3 Classical and Taguchi Methods. Referring to the methods which are available to approach such problems, one can cite first the classical statistical methods of experimental design [14 - 16], in particular, methods for the treatment of response surface designs [17]. More recent, sometimes more simple and efficient design methods from the field of quality engineering are due to Taguchi [18, 19]. An outline of his concept of parameter design was given in [20]. For more recent details, see [21, 22]. Corresponding software packages for experimental planning and analysis, 'Taguchi Analyst', 'Qualitek-3/4', are now available [23, 24]. In connection with a Taguchi approach for optimizing the performance of an analytical device see [25]. [26] focusses as well classical as Taguchi methods. Summing up, one can no longer exclude that Taguchi's methods are starting a new career in testing and metrology. This should be observed in the present scope, too.

2-2-4 Further Approaches. A treatment of more complicated problems can require much more sophisticated methods, for example identification of dynamical

systems, modal analysis, mathematical modelling and computer simulation [27] or approaches by finite or boundary elements. In this manner, corresponding activities can reach the level of research which is appropriate for journals like [28] and [29].

2-3 RESEARCH PROPOSALS. Observe again that continuing research is an extremely important component of statistical consulting. - Continue now to consider again collaborative studies and corresponding methods of analysis. This type of strict sense interlaboratory study is, on the one hand, of particular importance in respect of applications in the scope of VAMAS. On the other hand, statistical pre-standards research is required to improve corresponding concepts and approaches [30]. As already mentioned, from the viewpoint of VAMAS, comparative studies are of importance, too. In this case, the existing statistical and standardizational basis is much weaker than in the case of collaborative studies. Hence, existing approaches require much more research work to allow an application under the present circumstances.

Moreover, research and development on computer programs is necessary in cases in which the requirements of existing models and procedures are not satisfied or in which no appropriate procedure is available at all. In other words, the investigation, assessment, control or consideration of the effects of departures from assumptions underlying available statistical models [31] and approaches is one of the most important tasks on the statistical side of VAMAS interlaboratory research. This refers to general strict sense and wide sense interlaboratory studies, likewise.

As already indicated, the existing experimental designs and the corresponding statistical approaches are mainly directed towards the case of a univariate measurable quantity, though 'multivariate' interlaboratory problems are occurring, increasingly, in materials research and testing, analytical chemistry and in connection with problems from environmental control and protection. This implies that corresponding research is necessary which generalizes procedures from the univariate situation to the multivariate case.

Examples of research proposals are the following, where priorities should be given according to the needs occurring in connection with the treatment of concrete VAMAS advanced materials interlaboratory studies:

2-3-1 ISO 4259. The standard ISO 4259 (global analysis) contains several shortcomings, in particular, the re-estimation of outlying observations from the remaining values and the use of Student's t in the estimates of the preci-

sion data. Moreover, in comparison with ISO 5725 or ASTM E 691 (level-by-level analysis), concrete applications of this standard are rare for reasons of its more sophisticated structure and a lack of easy-to-use versions of computer programs. This implied that the importance of this standard is disputable in fields other than the motor and oil industries. Remedy is possible as follows:

- Substitution of the re-estimation procedure on the basis of methods of estimating variance components from unbalanced data [32];
- Substitution of Student's t by multipliers of Mandel and La-shof, [33, 34];
- Preparing and testing of a FORTRAN 77 version of an existing SAS computer program for ISO 4259 (and ASTM RR-D2-1007) [35].

In the Federal Institute for Materials Research and Testing, a first attempt for the development of such a computer program according to DIN 51 848 was made in 1981/82 [36, 37]. In particular, the transformation problem could be solved in an acceptable manner in the original version, i.e., without a dummy variable, on the basis of common regression. The project was interrupted because of a change in the computer system. Some of the experiences led to [38].

2-3-2 ISO/DIS 5725. The revised and extended version of the 1986 standard ISO 5725 comprises five parts. Up to part 5, the corresponding documents were issued 1990/91. They cover accuracy (trueness and precision) of measurement methods and results. More precisely, the following subjects are in the focus: (i) general principles and definitions concerning the accuracy of measurement methods and results; (ii) basic approaches for estimating the two extreme measures for the precision of measurement methods (repeatability and reproducibility); (iii) intermediate measures of the precision of measurement methods (weaker versions of the reproducibility conditions); (iv) measures for the trueness of a measurement method; (v) alternatives for the approaches according to (ii) and (iv); (vi) practical applications.

- Developing computer programs for selected details can be helpful to enlarge the range of engineering applications and the significance in the scope of VAMAS.

2-3-3 Recomputations. Since collaborative studies were already executed in the scope of VAMAS, for example, in the field of friction and wear [1], corresponding data are available to serve as a numerical basis for comparative applications of the statistical procedures contained in different documents. -

Corresponding computer programs are available in the case of level-by-level analysis, too [39 - 41].

- Numerical investigations and recomputations of estimates from data of collaborative studies, already executed and analysed, by existing or rearranged computer programs, observing the fact that on-going testing and validation of these computer programs is necessary.

2-3-4 Comparison of different Procedures and Documents. Apart from [42], more profound comparisons are lacking, which focus the effects of the different statistical models, the different treatments of the outlier problem and the different computational procedures used in the relevant standards, guidelines and other, more specific approaches. Statistical computer simulation is an appropriate method. Results could contribute to a rational assessment of corresponding documents and a judgement of their usefulness in the scope of VAMAS.

- Comparative statistical investigations and Monte Carlo simulations on the different statistical approaches to the evaluation of collaborative studies, considering, in particular, different statistical models [43 - 46] and the different treatments of stragglers and outliers [47, 48].

2-3-5 Negative Estimates of Variance Components. In the frame of an application of the random effects or mixed models, the analysis-of-variance method can lead to negative estimates of variance components [49] which cannot be tolerated in the further course of the analysis. One can observe, in particular, that, in the case of random models, the probability to obtain this phenomenon increases according to the number of factors [50]. The state-of-the-art with regard to this situation is poor. There are even standards which do not consider this phenomenon. In other words, further research on this subject is required:

- Further research on methods which guarantee positive estimates of variance components in the frame of appropriate analysis-of-variance approaches is necessary. This should lead to additional computer programs.

2-3-6 Interval Estimates of Precision Data. The output from the usual statistical procedures and computer programs for the analysis of data from collaborative studies is, commonly, poor. Apart from outliers and estimates of the true value, results are point estimates for the precision data. Interval estimates, i.e., confidence intervals, are of importance, too. Moreover, operating

characteristic curves for testing of the usual hypotheses on the variance components of the corresponding analysis-of-variance models together with operating characteristic curves for acceptance criteria based on the precision data are lacking in the common approaches.

- Determination of confidence intervals for the precision data [51 - 53], and the true value. Moreover, generalization and adjustment of the usual set-up of the computation of characteristic curves for acceptance criteria. A corresponding supplementation of the relevant standards and guidelines should be considered in both cases. Computer programs should be developed, in particular, in the first case.

2-3-7 Appropriateness of a Test Method. There exist empirical approaches for an assessment of the appropriateness of estimates of the reproducibility of a test method or method of analysis. One approach is due to Horwitz. Large-scale applications of this approach in which a resulting characteristic (HOR-RAT) played the leading role (data bases on analytical assays) were published, recently, [54, 55]. For an additional approach see [56].

- Investigation and generalization of the Horwitz equation and the Horwitz ratio by the Monte Carlo method taking into account a possible supplementation of the relevant standards and guidelines.

2-3-8 Discriminating Power. The problem of the determination of the discriminating power of a test method and the corresponding evaluation on the basis of two specified materials, treated in [57] in respect of ISO 5725 or ASTM E 691, requires further statistical and computational research.

- Improving existing computational approaches for an assessment of a test method in relation to its discriminating power by consideration of occurring outliers and negative variance components estimates. Adjusting of these approaches to further standards and guidelines.

2-3-9 Severity. In connection with the monitoring of test methods by collaborative studies at regular time intervals, the notion of the severity together with corresponding statistical approaches for its determination from collaborative studies is of importance.

- Investigation of options for a definition of the notion of the severity of a test method. Developing of statistical approaches and computer programs for the computation of point and interval estimates from the results of collaborative studies.

2-3-10 Conformance with Specifications: Supplier and Receiver. Another variety of problems, the existing solution of which can be made more realistic and practicable by computer simulation according to the Monte Carlo method, is formed by problems which refer to specifications and their impact on the relation between supplier and receiver. Though both of the relevant standards are designated for an application in the field of petroleum products, one can assume that research on the basis of the indicated method is of importance in connection with the performance of advanced materials standard test methods, as well.

- Probabilistic modelling, quantification and investigation of sensitivities of approaches proposed by ISO 4259 and ASTM D 3244 on product quality conformance, in particular precision data, specification limits and the position of supplier and receiver.

2-3-11 Changing of Normality Assumption. Standards like ISO 5725 and ISO 4259 assume normality of the data in connection with the identification of outliers and the definition of repeatability and reproducibility. ASTM E 691 makes a corresponding assumption only in connection with a determination of these two quantities. The required analysis of variance does not depend on a normality assumption. Hence, one should investigate the following possibilities:

- Relaxation or substitution of the normality assumption. Investigation of possible generalizations of the indicated approaches to the case of a lognormal distribution or other appropriate distributions of the data (without transformations). Preparing of suitable computer programs.

2-3-12 Bayesian Approach for Collaborative Studies. Collaborative studies can be very expensive. This can, in particular, happen in cases in which the determination of very precise estimates of the precision data of a test method are required, i.e., in cases in which, usually, the execution of a very large interlaboratory study is necessary. The available statistical approaches and computer programs for the analysis use classical statistical notions and techniques. Bayesian counterparts [58] would allow a consideration of informations from previous or similar interlaboratory studies which amounts, usually, to more precise statistical estimates from medium-sized studies and hence to a reduction of the experimental expenses.

- The practicability and the use of a development of Bayesian counterparts to the classical statistical approaches of analysis, in particular, to those contained in relevant standards - ISO 5725, ASTM E 691, ISO 4259, ASTM RR D2-1007 - and guide-

lines [59, 60] should be investigated, where one has to observe ISO/DIS 5725 (see 2-3-2).

Possible solutions should consider the fact that data for repeated studies are, commonly, obtained by the same laboratories of one and the same research group. This situation affects the modelling and analysis of many interlaboratory studies. Note that the relevant problem of random effects in connection with finite populations was already considered in [32]. Possibly, multistage approaches make sense. Results could give rise to the development of computer programs.

2-3-13 Qualitative Data. The data from an interlaboratory study can be qualitative [61 - 63]. This means that the usual ideas on collaborative studies have to be modified, suitably.

- Modification of the quantitative concept of the collaborative study and the statistical approach for an analysis to the case of qualitative data. Development of corresponding computer programs.

2-3-14 Comparative Studies. Referring to the comparison of test methods in the scope of comparative studies [64], there is a lack of standards and guidelines, i.e., ample room for pre-standardization research. Recently, a Taguchi approach was used to plan and to evaluate a comparative study [65, 66]. A computer program based on a specific approach to the analysis of comparative studies is contained in [67].

- Pre-standardization research on comparative studies including the development of further computer programs.

2-3-15 Modelling and Simulation of Interlaboratory Studies having a Specific Design. The need for research and development on interlaboratory studies with design departing from the usual models was already emphasized. From the viewpoint of VAMAS, it should be stressed again that the present proposal is of particular importance in connection with the design and analysis of advanced materials interlaboratory studies. There is no doubt that an extensive spectrum of methods from computational statistics will have application in this field. A regular column on statistical computing and software is contained in [68].

- Development of statistical models, approaches and computer programs in specific cases of interlaboratory studies, which are not covered by existing standards and guidelines, including generalized strict sense and wide sense interlaboratory studies, likewise. Preparing and conducting of computer simulation experiments in the preliminary stages of forthcoming studies.

2-3-16 Systematic Measurement Uncertainties. The relevant standards and guidelines disregard the influence of systematic uncertainties [69, 70, 71]. In general, such uncertainties [72] can be combined with random uncertainties by randomization [73, 74].

- The possibilities of obtaining statistical approaches and corresponding computer programs for a consideration of systematic uncertainties should be investigated. An example of an appropriate design structure is given by [75, 76]. See also ISO/DIS 5725.

2-3-17 Interlaboratory Studies from the Multivariate Point of View. To illustrate the situation, note at first that multivariate interlaboratory studies are conducted, for example, by the motor and oil industries in the field of engine testing. In the scope of such studies, the interaction of fuel and oil with a testing engine is investigated. The multivariate character results in this case from the fact that a single test result in the frame of such an interlaboratory study consists of a set of observations on characteristics like piston cleanliness, weight of deposits, ring sticking, cylinder wear, viscosity increase, oil consumption, and sludge.

As indicated before, similar situations can be observed in several other fields of science and technology. In the scope of VAMAS corresponding situations occurred, for example, in the field of friction and wear. Although, one can consider and treat the output from such an interlaboratory study 'componentwise', a generalization of the univariate models and procedures to the multivariate case is necessary.

- The applicability and use of statistical multivariate methods [77 - 80] (multivariate outliers [81], multivariate analysis of variance (MANOVA) [82, 83], principal component analysis [84], pattern recognition and cluster analysis [85, 86]) for the planning and analysis of multivariate interlaboratory studies, in particular collaborative and proficiency studies, should be further investigated.

2-3-18 Additional Proposals. Furthermore, the following proposals could be of importance:

- The role of robust estimates in the frame of the analysis of interlaboratory studies [87, 88] should be investigated.
- To cover the case of time-dependent observations in the frame of interlaboratory studies, additional research on the analysis of time series collected in an experimental design [89] is neces-

sary.

- The use of more advanced sampling [90] and resampling [91] approaches under the present circumstances should be examined.
- The applicability of concepts and techniques from information theory [92, 93] in the field of interlaboratory studies should be investigated.
- The state-of-the-art in the field of expert systems for interlaboratory studies deserves special monitoring [94, 95].
- An important boundary field of the present activities is research on the limit of detection [96].
- Certain scientific aspects of certification and accreditation should also be considered in the frame of this kind of research.
- The terminological and semantical basis of the mentioned concepts requires further consideration. For similar activities in the scope of VAMAS see [97].

2-4 CONSULTING OF OTHER TWAs. Providing for an immediate access to relevant theoretical and computer-oriented knowledge and corresponding experiences, possibly via a network of hot line services (FAX, computer mail) in the frame of the TWA / Advisory Group is of great importance for several reasons:

- Specialists from these fields can try on request to help the experimenter, immediately, in analysing or solving occurring non-experimental problems.
- Activities of this kind allow to gain a more thorough impression on corresponding needs of other TWAs, such that a feedback on this part of the work of the TWA / Advisory Group is implied.
- Client-consultant interactions can give rise to new and cooperative research projects in the TWA / Advisory Group.
- In this way, one can contribute, at least in the frame of VAMAS, to a bridging of the traditional gap between experiment and theory showing, in particular, that statistics is not an obstacle but a help.
- Consulting of this kind produces an additional impact on the work of VAMAS by increasing the efficiency and effectiveness of its experimental activities.

More precisely, one can assume that consulting activities covering the relevant theoretical fields in the frame of the TWA / Advisory Group include the translation of problems into mathematical or statistical framework, the adap-

tion of theories and methods to problems of interest, the communication of concepts and methods to the client, literature reviews on such problems and concrete support in connection with problem solving.

As subjects in the computational fields, in particular computational mathematics and computational statistics, topics like hardware and operating systems, including intelligent laboratory systems and computer nets, software, including libraries, packages, public-domain, easy-to-use and user-developed programs, and scientific computing, in particular supercomputing, offer themselves.

Examples of fields, where co-operative research from consulting activities should be expected, are fatigue and friction and wear. While with respect to the first subject probabilistic and reliability-oriented models are of interest, a co-operation with respect to the second subject can include synergetical approaches as well as mathematical modelling and computer simulation.

Apart from the statistical techniques for improving the design of experiments and, hence, their effectiveness, from the viewpoint of chemometrics [98, 99], the following topics are considered to be important for the present part of the work of the TWA / Advisory Group: parameter estimation, multivariate statistics, calibration, optimization, signal processing, resolution of peaks, pattern recognition and artificial intelligence. For more advanced subjects, see [100, 101].

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ORGANIZATIONAL ASPECTS

3-1 STRUCTURE AND STAFF. Taking into account the objectives illustrated in the foregoing chapter, the design of the organizational structure of the TWA / Advisory Group represents itself in a natural manner by two working levels. While it is intended to organize, on the one level, small groups for the treatment of more sophisticated problems in connection with the design and analysis of advanced materials interlaboratory studies and for corresponding pre-standardization research, attached to institutes in the frame of the TWA / Advisory Group, the work on the other level shall comprise, in the first place, the announced activities of service, consulting and optimization, provided that this can be done without additional research.

The progress of the research groups and consequences for the working programme can be discussed in the scope of the second level, for example, during annual meetings of the staff of the TWA / Advisory Group. In addition, liaison reports on the progress of interlaboratory studies and their analysis can be given. This part of the work should aim also at an elaboration of rules or recommendations for the conduct, analysis and interpretation of interlaboratory studies and the development of some kind of bye-law for VAMAS interlaboratory studies taking into account certain quality requirements. Moreover, results, manuscripts, papers and proposals or contributions for standardization committees can be circulated for comments and review at any time. An additional task which should belong to this level is the organization and execution of meetings, workshops or scientific conferences. Important subject could be, for example, 'Computers and Computing in Interlaboratory Research and Testing' or 'Computational Materials Science and Engineering'.

It is obvious with respect to the second level that, for the purpose of a continuing communication between the annual meetings and, in particular, the communication with other TWAs, the usual facilities (FAX, computer mail) are sufficient. An effective research work on the first level, however, requires a concentration of the members of a group at one place.

Referring to the staff of the TWA / Advisory Group, attention should be given to the fact that the time of members from the industry is more limited than that of members from research institutes. This means that contributions of members of the first kind can be expected, essentially, in the field of consulting and only to a limited extend in the field of service. In connection with the

forming and reinforcement of research groups, the temporary employment of external researchers will be necessary. Moreover, in certain cases the introduction of positions of guest researchers will have to be taken into consideration.

Observing the proposed research activities, it is the opinion that the organizational structure of a TWA is more appropriate in the present case than the structure of an advisory group.

3-2 SUPPORT FROM VAMAS. Up to now, the chairmen of five TWAs responded positively to a first call for co-operation: TWA 1 found the proposed activity timely and important. Data on previous interlaboratory studies are available on request [1, 2]. TWA 3 found that statistical methods are very important in analyzing interlaboratory studies. A list of projects already performed, a list of publications, and copies of the reports [3, 4] were supplied, together with an extremely valuable report on the experiences and the work of this TWA. TWA 4 provided an extensive list of publications, contributions to conferences, and reports, mentioned, however, that Canada will only passively participate in the present round of VAMAS activities. TWA 5 mentioned its pleasure to help launching the new item and offered all of the data concerning their work. TWA 10 gave a report on their previous interlaboratory studies and supplied copies of the reports [5, 6]. The former chairman of VAMAS, Mr. M. K. Hossain, mentioned that the statistical analysis of data is currently an important concern in his division of NPL and proposed a discussion of a preferred approach towards the end of the running year. - Concerning the first period of VAMAS activities, [7] was placed at the disposal of the authors of this manuscript.

An earlier version of this manuscript was presented to members of the VAMAS Steering Committee by the first mentioned author on June 6 during a meeting in Berlin, in the presence of an observer from ISO. The overall opinion on this project was positive, though the well-known additional amount of persuasive work will be necessary to convince all members of this committee from the fact that statistical support of experimental investigation and corresponding problem-oriented research in statistics are two sides of one and the same medal. - The publication of a shorter version of this manuscript in the VAMAS Bulletin was proposed to intensify communication with other TWAs. The final designation of the group as a TWA or an Advisory Group is still open. Several members of the Steering Committee prefer the latter.

To support the German kernel of the TWA / Advisory Group consisting of Mr.

Fritz, Mr. Gerisch and Mrs. Steinborn, the following scientists were named in the frame of VAMAS: Mr. Amzallag (France), Mr. Cosoli (Italy), Mr. Suzuki (Japan), Mrs. Croarkin (USA), Mr. McCune (USA), and Mrs. Wostenholme (UK).

3-3 SUPPORT FROM OTHER ORGANIZATIONS. Furthermore, NORDTEST named Mr. Svensson (Sweden). The National Engineering Laboratory mentioned that Mr. Dower (UK) may be able to contribute to the programme. The Technical Research Centre of Finland (VTT) named Mr. Häyrynen and Mr. Isomursu who are working on statistical materials characterization and on fuzzy logic in connection with interlaboratory studies, respectively. Mr. Horwitz, AOAC, Food & Drug Administration, gave notice that he will be in a position to co-operate with VAMAS to a reasonable extent. Mr. Feinberg, Statistics Committee of AOAC, is interested in collaborating to any project that can improve the processing of interlaboratory data. Mr. Muster, who is working on advanced materials, was named by EMPA (Switzerland). Mrs. Nemeth, AOAC, is working on collaborative testing in food safety. An additional member from the French oil industry, interested in offshore materials, was announced for autumn of the running year.

3-4 CONTINUING NEGOTIATIONS. There is a certain chance that the staff of the TWA / Advisory Group will obtain additional support from institutes of the following countries: Canada, France, Germany, and USA. - To reinforce the capacity of the group, announcements on the designed activities in Journals like [8] and [9] are taken into consideration.

3-5 PROFESSIONAL STRUCTURE OF THE TWA / ADVISORY GROUP. Looking at the occupational activities of the scientists interested in the work of the TWA / Advisory Group, till now, one can claim some kind of multi-disciplinary character: There are, in particular, mathematicians, pure and applied statisticians, chemometricians, engineers from materials science and technology, and specialists from statistics and standardization committees. Hence, one can assume that the group will be able to develop activities which cover the required wide spectrum of services, research and consulting to a helpful extent.

3-6 FINANCIAL ASPECTS. While the expenses occurring in connection with activities for services, optimization and consulting, including charges for using communication facilities and for travelling, shall be beared by the participating institutes, since VAMAS does not provide financial support, funding by the corresponding national (Japan: [10]) and international organizations (Commission of the European Communities, BCR [11]) will be necessary to allow the employment of external researchers. In the case of guest researchers, scholar-

ships (Germany: [12]) or funds from the budget of participating institutes (Germany: [13]) could be an appropriate solution.

3-7 START OF WORK. It is intended to start the work of the TWA / Advisory Group on the basis of the present version of the programme, after the approval by the mentioned scientists during a workshop in Berlin (see Chapter 5) and after a confirmation of the project by the VAMAS Steering Committee, on the forementioned second level in the field of service and consulting. In this phase, an important point will be the extension of the communication with other TWAs, in particular, with members working in fields in which this TWA / Advisory Group is planning activities. Moreover, forthcoming interlaboratory studies on wear behaviour and, possibly, on fracture toughness will be focussed on this level.

The preliminary work on the first level will be chiefly of an organizational and administrative nature. In particular, the problem of initiating and funding the research groups will be in the focus. Moreover, high priority will be given to an exchange of experiences on the applicability of appropriate software, including the existing international problem-solving systems (IMSL, SAS), and to a completion of existing pools of computer programs for the analysis of interlaboratory data.

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COLLABORATIVE ACTIVITIES IN SCIENCE AND TECHNOLOGY

4-1 IDENTICAL TEST MATERIAL. Observing the fact that the statistical roots of interlaboratory studies, in particular of collaborative studies, can be traced back to the petroleum industry, where basic notions like repeatability and reproducibility were introduced during World War II, one can realize that these notions meet, in the first place, the requirements of liquid substances from which, usually, 'identical test material' can be provided without problems, regardless whether the test material is altered by the test or measurement or whether it remains unchanged. Indeed, classical collaborative studies assume that such test material is investigated by the same method under repeatability conditions - same operator, same apparatus, same laboratory, and short interval of time - and reproducibility conditions - different operators, different apparatus and different laboratories -, where it is sometimes appropriate to modify the reproducibility conditions [1]: different operators, different apparatus, etc., however same laboratory. For more severe changes, see ISO/DIS 5725.

In the case of solid substances, as in the frame of common advanced materials collaborative studies, however, the preparation of identical test material can be a problem, because no specimen behaves like another one. Depending on the property under investigation and the complexity of materials mechanical processes [2], this fact has to be considered either on the experimental side or, if necessary, in the frame of individual statistical models. Nevertheless, there are applications of the classical approach in testing solid materials, for example, in the case of polymeric materials and products [3 - 5]; see also [6].

This supports the above-mentioned thesis according to which, in many situations, the consideration and adjustment of the indicated discrepancies between experimental practicability and available statistical concepts forms a fundamental statistical task in the scope of advanced materials interlaboratory studies, other than simple applications of existing standards or guidelines.

4-2 MORE ON WIDE SENSE INTERLABORATORY STUDIES. An important field of application of interlaboratory comparisons is metrology. Journals like [7] contain many examples from measurement, calibration and testing. A workshop and seminar on uncertainties was conducted recently [8]. - An interlaboratory study from materials testing is considered in [9]. For miscellaneous examples of this kind of wide sense interlaboratory studies, see [10 - 12].

4-3 DIFFERENT OUTPUT FROM INTERLABORATORY STUDIES. Strict sense interlaboratory

studies of one and the same type can differ, in addition, by their structure, design and data. To go into the details, a method can produce quantitative or qualitative data. Moreover, there are wide sense interlaboratory studies the data or results of which are mathematical models, for example, equations of regression lines [13]. In most cases, the data obtained and analysed in the frame of an interlaboratory study are of experimental nature. There are, however, studies on artificial data, for example, in connection with simulation experiments [14] or, as in the scope of certain advanced materials interlaboratory studies [15, 16], on results from finite element analyses.

4-4 WARNING. According to these circumstances, the description of the different kinds of interlaboratory studies, in particular in the strict sense case, require different statistical models. This implies that the planning and co-ordination of such studies has to be performed under different aspects. Furthermore, an application of different statistical techniques for the analysis, the evaluation and the interpretation of the results of such studies is necessary. It follows that, though corresponding expert systems are under development, the chances of obtaining disappointing results from interlaboratory studies are great, in particular, if unexperienced groups of laboratories plunge into such studies without careful planning, competent guidance and with lack of access to sufficient statistical and computational facilities.

4-5 PROMOTION AND HARMONIZATION. To bridge gaps occurring in this manner and to harmonize the design and the execution of strict sense interlaboratory studies, national and international agencies (NBS [17], ASQC [18], ASTM, ISO, BSI, AFNOR, ASA [19]) provided for corresponding methods and standards. In addition, special committees of professional associations and institutes developed and improved guidelines for this kind of studies (AOAC: Interlaboratory Studies Committee [20, 21 - 23], Statistics Committee [24]; ACS; Laboratory of the Government Chemists [25]). Other organizations (IUPAC [26, 27], EUROLAB [28]) support the execution of interlaboratory studies and the preparation of standards and guidelines by regular workshops and symposia. To simplify the communication, standard formats of data and results were established for the most important types of studies [29]. Moreover, specific standard formats and protocols for an effective transmission of data in computer nets were developed [30, 31]. As mentioned above, computer programs for the analysis of the results of such studies are already available (ASTM [32], BSI). The state-of-the-art in the field of interlaboratory studies in the frame of chemometrics is also reviewed at regular intervals [33, 34].

4-6 MISCELLANEOUS INTERLABORATORY STUDIES. Under aspects of quality and trade, political organizations (Commission of the European Communities) and industrial associations installed institutes (BCR [35, 36]) or committees for the planning, guidance, analysis, monitoring and certification mainly in connection with industrial interlaboratory studies, executed in the corresponding sphere of activity (CEC (London): Lubricants and Engine Fuels: Statistical Applications Advisory Committee [37]; DIN, ASTM [38]). Finally, apart from the activities in the scope of VAMAS, many institutions (NIST, NRI, NPL, BAM) organize and analyse interlaboratory studies, essentially on the national or local level, in different fields and for different purposes, including the certification of test methods and the accreditation of laboratories. In these ways, activities on an interlaboratory scale penetrated nearly all spheres of life. Apart from science and technology [39], this refers, in particular, to: environment [40 - 42], agriculture and food [43, 44]; chemistry [45 - 48], clinical analysis [49, 50], medicine, drugs and cosmetics; textiles; pulp and paper; paint, and all other kinds of materials [51 - 56].

4-7 CONCLUSION. Summing up, one must say, that interlaboratory studies can form an approved, very effective and extremely helpful device to promote research work, the development of new test methods, and further practical applications on the borderline between materials, on the one hand, and measurement and testing as well as specifications and standards, on the other hand, provided that the described assumptions and requirements are considered carefully and that the given warning is taken seriously.

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APPENDIX A

MINUTES OF THE FIRST MEETING OF THE TWA / ADVISORY GROUP

5-1 **PRELIMINARY REMARK.** In the following, a reprint of the draft minutes for the first meeting of the TWA / Advisory Group is given. Up to the end of the year 1992, the following members signalized agreement: D. Amzallag (France), T. Fritz (FRG), W. Gerisch (FRG), W. Muster (Switzerland), S. Steinborn (FRG), T. Svensson (Sweden), and M. A. Nemeth (USA). In comparison with the original version, the present version of this document contains several additional telephone numbers, FAX numbers and revised addresses.

In addition, M. A. Nemeth announced possible contributions to chapter 14 of these minutes as follows: Service: Design of experiments; commercial packages on PC, Macintosh, VAX, and IBM mainframe: includes SAS, BBN products, JMP, DataDesk, Statview, Super Anova, Design Expert, Design Ease, Statgraphics, ASTM E691; collaborative studies. - Optimization: Response service methodology. - Research: Multivariate methods. - Consulting: Statistics; experimental design and data analysis.

5-2 **DOCUMENT.**

12/10/92

DRAFTMINUTES of the INITIAL MEETING (WORKSHOP)

of the

Technical Working Area / Advisory Group

'STATISTICAL TECHNIQUES FOR ADVANCED MATERIALS INTERLABORATORY STUDIES'

Held on 27th November 1992

at the

Federal Institute for Materials Research and Testing (BAM), Berlin, FRG

ATTENDANCE

D. Amzallag		France
K. Doerffel		FRG
T. Fritz		FRG
W. Gerisch	(Chairman)	FRG
J. Häyrynen		Finland
W. Muster		Switzerland
K. Suzuki		Japan
S. Steinborn		FRG
T. Svensson		Sweden

1 INTRODUCTION

- 1-1 Purpose: Apart from short presentations of the participants on their institute and work, the meeting aimed, essentially, at a discussion of the background document on the situation in fields in which activities of the Group can be expected. In particular, it focussed concrete preliminary steps which had to be taken before the work of the Group can start.
- 1-2 BAM: The Chairman introduced BAM, its Group of Mathematics and Computer Services, and himself.
- 1-3 VAMAS: An outline on structure and organization, objectives, technical work, achievements and future trends was given. - Reference: M. K. Hos-sain, Current status and future trends, ISPRAM '91, VAMAS Bulletin No. 15, January 1992, 2-5.

2 MEMBERSHIP LIST

The membership list was obtained from the list of persons invited to the meeting by deleting the following names for reasons of working fields different from the designed working area of the Group: Dr. Cosoli, Italy, Mr. Isomursu, Finland and Dr. Ito, Japan.

- 2-1 Remark: The complete membership list is given in an Appendix to these Minutes (see Chapter 12). An additional Appendix contains a list of scientists from non-G7 countries which apply for membership (see Chapter 13).
- 2-2 Remark: It was agreed to use first names in the future.

3 APOLOGIES FOR ABSENCE

Apologies were received from C. Croarkin, R. J. Dower, M. Feinberg, W. Horwitz, M. A. Nemeth, and L. Wolstenholme.

4 MATTERS ARISING

- 4-1 VAMAS Calender: The following meetings were mentioned: TWA 14 'Classification Ceramics': 10-11 December 1992 (BAM) [TWA = Technical Working Area]; VAMAS Steering Committee: 22-23 April 1993 (NIST).
- 4-2 Report: A report on the short presentation of the Chairman to the VAMAS Steering Committee during its Berlin meeting on 4-5 June 1992 was given. The presentation cited the Minutes of the Tokyo meeting of the VAMAS Steering Committee on 19-20 Dezember 1991, recalled the objectives of the Group and gave an outline on the development up to the organization of the present meeting in Berlin. Copies of an earlier version of the background document were distributed and discussed.

5 VAMAS AND INTERLABORATORY STUDIES

Mrs. Steinborn (BAM) resumed the presentation of VAMAS more detailed. She

mentioned that one of the most effective ways of determining quantitatively the performance of test methods applied in different testing institutes is the execution of interlaboratory studies. Moreover, she emphasized the importance of this kind of studies for prestandardization research in the frame of VAMAS. In the last years nearly in all VAMAS TWAs studies of this kind, however, in many cases with different structures and different purposes, were performed. More than 500 laboratories from member and non-member states took part in such investigations. Finally she assumed that the enormous efforts in money, material and hands, necessary to execute efficient interlaboratory studies, may have led, as in many other fields of science and technology, to the decision to set up a VAMAS working group to support the execution of advanced materials interlaboratory studies from the statistical point of view and to improve their efficiency by statistical methods.

6 INSTITUTE AND WORK OF PARTICIPANTS IN THE FIELD OF INTERLABORATORY STUDIES OR RELATED SUBJECTS

- 6-1 C. Amzallag: IRSID, Centre de Recherche d'USINOR SACILOR: Structure and organization of research in the 2nd largest steel corporation of the world. - An outline on research in fatigue, in particular on interlaboratory research in fatigue rupture and statistics as an important tool, was given. A nuclear programme, automotive applications, contributions to VAMAS on low-cycle fatigue testing and activities in the field of standardization (afnor) were mentioned.
- 6-2 K. Doerffel: Technical University Leuna-Merseburg: Analytical and geochemistry; analytical applications of spectroscopical methods; statistical methods in chemistry; time series analysis; surface analysis; international interlaboratory tests in chemistry. - A geometrical pre-test for the possible support of multivariate interlaboratory studies on the quantitative occurrence of similar chemical substances (Fe_2O_3 , Al_2O_3 , SiO_2 , TiO_2) was presented. Graphs for different laboratories were compared. The importance of the pre-test was explained.
- 6-3 T. Fritz (BAM): A system of computer programs for the analysis of collaborative studies on the basis of ISO 5725 was presented which allows an automatic processing of round robin data from their collection in the laboratories to the output of numerical and graphical results. The run time for 35 laboratories and 15 materials was about 6 minutes. The program is used in BAM for the analysis of environmental interlaboratory studies, proficiency investigations and accreditation.
- 6-4 W. Gerisch (BAM): An abstract to a paper on the discrimination of test methods was presented. The computer programs contained in this paper will allow an evaluation and a more simple application of Bye-Law No. 6, Chapter D 'Requirements to be met before Full Approval ('A') is granted to a Test Method' of the Co-ordinating European Council for the Development of Performance Tests for Transportation Fuels, Lubricants and other Fluids, London.
- 6-5 J. Häyrynen: Technical Research Centre of Finland: Structure and organization of VTT; main objective support of industry. - It was reported on the activities of the metals laboratory, in particular, on the investigation of structural materials and the role of environmental conditions, real world and simulated. Moreover, it was mentioned that about 30 in-

terlaboratory programmes were conducted on conventional materials and test methods. The investigation of advanced materials was found to be affected by the present recession in Finland.

- 6-6 W. Muster: Swiss Federal Laboratories for Materials Testing and Research: Structure and organization of EMPA. - A participation in the VAMAS fracture toughness test round robin on ceramics was mentioned. An outline on stochastic investigations (probability of failure, Weibull distribution) on strands and cables was given. Furthermore, proficiency testing on modern NDE equipment was discussed. Finally, the importance of computer tomography for research and testing of advanced materials was demonstrated using the detection of flaws and the distribution of the defect size in the case of ceramics as an example.
- 6-7 K. Suzuki: University of Electro-Communications. - Working area is the field of quality control (member of the Deming Price Committee. - Reference: T. Ryan, Statistical Methods for Quality Improvement, New York: Wiley 1989, Chapter 2, Japan's approach to quality control; added by the Chairman). The following working fields were mentioned and discussed: statistical investigations on lifetime, experimental design for Weibull regression using orthogonal array tables (examples: L_4 , L_8 , L_{16} , L_{32}); analysis of degradation data for reliability assessment, in particular lifetime parameters; evaluations of lifetime from field performance data; analysis of the relations between field performance and laboratory experiment.
- 6-8 T. Svensson: Swedish National Testing and Research Institute: Structure and organization of SP; additional activities on weight and measure. - From the view point of the activities of the Department of Materials and Mechanics, welded structures and ceramics were focussed. A cooperation in the frame of VAMAS was mentioned. An outline on the following working fields was given: models for the stochastic approach to fatigue, time series analysis, signal processing; statistics on interlaboratory studies.
- 6-9 Remark: In connection with the presentations, documents were placed at the disposal of the Chairman. These documents are listed in an Appendix to these Minutes (see Chapter 16).

7 COMMENTS ON THE BACKGROUND DOCUMENT

- 7-1 Table of contents of the background document and overall comments
- 7-1-1 Mr. Doerffel proposed that the document should be supplemented by a Summary and contributed a corresponding draft. This was accepted.
- 7-1-2 Mr. Suzuki emphasized that the effectiveness of the Group should soon be shown on the basis of a collaborative study with actual results.
- 7-2 Classification of interlaboratory studies: An appropriate classification of interlaboratory studies and the use of corresponding terms was left as an unsolved problem. The background document discriminates provisionally between 'strict sense' and 'wide sense' interlaboratory studies.

In the first case the classification of Mr. Horwitz is of importance (see Chapter 9-3-1).

7-3 Distribution of identical test material: The cases of liquid and solid test material were discussed for collaborative studies. In connection with the applicability of existing standards and guidelines in the case of solid specimens, corresponding assumptions were considered as a severe handicap.

7-4 Objectives of the Group

It was agreed on the following items:

7-4-1 Service: Support of the work of other TWAs, including statistical computer simulation, to increase the effectiveness of VAMAS inter-laboratory studies.

7-4-2 Optimization: Application of methods of experimental design (classical and Taguchi) or mathematical modelling and computer simulation to improve specimens or test devices, observing available models and approaches.

7-4-3 Research: Problem-oriented statistical research on interlaboratory studies; improvement of the structure and applicability of existing models and approaches; development of computer programs.

7-4-4 Consulting: All fields of mathematics, computational statistics, certain fields of chemometrics, materials research and testing.

7-5 Research proposals: The list of research proposals was recalled.

7-5-1 Mr. Suzuki drew the attention to the new ISO/DIS 5725, 1992, in particular to the role of the intermediate conditions and to bias reductions in the cases of the relevant analysis-of-variance models. Moreover, he gave important references to the point 'comparative studies' and supplemented the list by mentioning the analysis of degradation data and the evaluation of the coincidence between field performance and laboratory experiment.

7-5-2 Remark: There was no comment on an inquiry of the Chairman on whether or not multivariate calibration techniques should be put onto the list.

7-6 Preparatory activities and start of work:

7-6-1 Tasks for the Chairman: A revised and enlarged version of the background document has to be prepared for publication as a VAMAS Report. It was agreed that short reports on the Group and its intended working area should be prepared for the VAMAS Bulletin and similar journals of ISO and ASTM. Moreover, a formal application for seeking full approval of the Group and its objectives by the VAMAS chairman and the VAMAS Steering Committee has to be set up.

7-6-2 Remark: In the meantime, support on 7-6-1 was obtained from the Secretary of VAMAS in correspondence with a decision of the Steering Committee during its Berlin meeting.

- 7-6-3 Possible Contributions from the participants: A questionnaire on this subject was circulated during the meeting. The results of the inquiry are listed in an Appendix to these Minutes (see Chapter 14).
- 7-6-4 Handling of requests for cooperation
- 7-6-4-1 Service: Among the options of either delegating the complete business to a sufficient experienced member of the Group with access to appropriate computational facilities or giving details to all members of the Group in seeking sufficient experienced member for parts of the cooperation, priority was given to the first option.
- 7-6-4-2 Optimization: It was agreed on a solution according to the first option of 7-6-4-1.
- 7-6-4-3 Research: Organization and supervising of research was found to be a matter of the individual member of the Group. To support the development of this objective and to allow an exchange of scientists, the Chairman will try to move something on the side of funding.
- 7-6-4-4 Consulting: A sufficient experienced member of the Group has to be found in each case.
- 7-6-4-5 Remark: The foregoing subjects indicate the task of drafting Articles of the Group.
- 7-6-5 Informations from VAMAS: More detailed informations from VAMAS on forthcoming interlaboratory studies are necessary.
- 7-6-6 Start of work: It was agreed that the work should start after full approval of the Group in the fields Service, Research and Consulting. Optimization was found to be more critical and should be offered in advanced cases only after the initial phase.
- 7-6-7 Existing software: A list of software for collaborative studies is given in an Appendix to these Minutes (see Chapter 15).
- 7-7 Appendix: Two interlaboratory studies: To give examples on different kinds of interlaboratory studies occurring in the frame of VAMAS, to show the importance of a classification of such studies, and to allow an impression on the variety of helpful statistical approaches, the Chairman gave an outline on two VAMAS interlaboratory studies of different structure and purpose from the statistical point of view. The first study, a collaborative study, was conducted by TWA 1 'Wear Test Methods'. The second study was designed with the structure of a distributed study and conducted by TWA 10 'Materials Databanks'.
- 7-7-1 Collaborative study: Data on friction and wear were obtained from ball-on-disc specimens for dry sliding with different combinations of ball and disc materials. The Monte Carlo method was used to generate artificial data according to the experimental data. Monte Carlo distribution functions of repeatability and reproducibility led to an optimal experimental design as follows: 35 labo-

ratories, 2 observations per laboratory and materials combination, 4 ball materials and 4 disc materials, i.e., 32 specimens per laboratory. - Reference: unpublished report, BAM.

- 7-7-2 Distributed study: Computational models for the analysis of creep and fatigue data obtained from engineering steels and alloys were compared. The applied statistical approach was least squares, more precisely, regression - Reference: VAMAS Technical Reports 6 and 7, October 1990.

8 COMMENTS FROM NON-PARTICIPANTS

- 8-1 France: Mr. Amzallag reported that he forwarded copies of the background document to departments responsible for VAMAS related activities of important French enterprises. For example, Électricité de France (nuclear power) and SNECMA (aerospace). The reactions were constructive. One is now waiting for concrete activities. An additional contribution from this side should not be excluded.
- 8-2 UK: A short and constructive comment on the background document from Dr. M. Crowder, University of Surrey, was forwarded to the Chairman by Mrs. Wolstenholme. This comment raised again the important problem of financial sponsorship in connection with research to be conducted in the frame of the Group.
- 8-3 USA: An additional comment was announced by Mr. Horwitz.

9 FINAL RESOLUTION TO VAMAS

9-1 Preliminaries

The Chairman learned from a letter which he obtained from Mrs. Croarkin that the VAMAS chairman 'stressed the importance of the initial meetings for deciding whether or not existing protocols for interlaboratory tests will suffice for VAMAS studies and for settling upon new protocols as required'.

- 9-1-1 Existing protocols: It was agreed that a decision on whether or not existing protocols suffice for VAMAS studies depends to a great extent on the specific situation under consideration. Taking into account, moreover, the variety of different structures of VAMAS collaborative investigations and the importance of solid materials in the frame of VAMAS, an answer to this question on such a general level seemed to make no sense. It was rather found that a rational choice of an appropriate protocol or procedure for the design, execution and analysis of a specific interlaboratory study should be a matter of the working field 'service' according to the first objective of the Group.
- 9-1-2 New protocols: While in certain cases, in particular for collaborative studies, adjustment, modification and supplementation of existing protocols can be done in certain cases in the frame of the working field 'service', the development of new protocols or procedures is a task of the working field 'research' according to

the third objective of the Group.

9-2 Organization and Administration

- 9-2-1 Objectives: It was confirmed that the objectives of the group shall be Service, Optimization, Research and Consulting. For details it is referred to 7-4 and to the background document.
- 9-2-2 Name of the Group: Messrs. Amzallag and Muster pointed to the fact that the term 'Advanced Materials' in the provisional name of the Group 'Statistical Techniques for Advanced Materials Interlaboratory Studies' is an unnecessary restriction of the intended working area of the Group. This term is already contained in 'VAMAS' and should not be repeated. The name 'Statistical Techniques for Interlaboratory Studies and Related Projects' could solve this problem.
- 9-2-3 Designation of the Group: Up to now, the Chairman used a combination of the terms 'Technical Working Area' and 'Advisory Group' to allow a vote on this important subject to the participants of the initial meeting. Though the Steering Committee favoured the second term, this vote was in favour of the designation 'Technical Working Area'. It was found that the term 'Advisory Group' is not sufficient for a description of the full spectrum of activities expected from such a group. Nevertheless, one can point to the specific character of the Group by an appropriate position in the List of VAMAS TWAs.
- 9-2-4 Kinds of Membership: The Group is prepared to allow Regular and Corresponding Members.

9-3 Proposals

- 9-3-1 Standardization: VAMAS should exert its influence on standardization bodies to initiate the preparation of some kind of 'Standard Practice for Use of the Term Interlaboratory Study and Related Terms'. Helpful suggestions can be drawn from a report of Mr. Horwitz on the AOAC Interlaboratory Studies Committee (1989) and from papers according to the list of references to the background document.
- 9-3-2 VAMAS test methods: It was agreed that guidelines containing statistical and experimental requirements should be prepared which have to be met when establishing a VAMAS test method. These requirements should consider the different stages of test methods from initiating the development to continued maintenance.

9-4 Problems

- 9-4-1 Financial support: The problems of travelling expenses and research funds were considered as pressing needs. Communication and co-operation with other TWAs requires travelling, for example, to take part in annual meetings. The importance of research funds, for example, to finance guest researchers, was already stressed in Chapter 8-2.

9-4-2 Remark: The Chairman is prepared to raise the problem of 9-4-1 on other occasions, too.

10 ANY OTHER BUSINESS

There were no items raised from the participants. Therefore, supplementary tasks of the Group were discussed:

10-1 Workshops: The Group should prepare Workshops to introduce members of the experimental TWAs into the basic principles of statistics and optimization to improve the design, the analysis and the effectiveness of interlaboratory studies.

10-2 Conferences: A further task of the Group should be the organization and execution of scientific conferences. Example: 'Computing and Computers in Materials Research and Testing'. Emphasis could be given to collaborative research and to advanced materials and standards.- Mr. Muster proposed a slightly modified title.

10-3 Remark: It was agreed that members should mention their membership in a footnote to papers on interlaboratory studies and related subjects.

11 DATE AND VENUE FOR NEXT MEETING

There was interest in trying to have the next meeting organized for Friday, 26th November 1993 at the National Institute of Standards and Technology, Gaithersburg, USA, provided that this date is not affected by the Thanksgiving Day. The Chairman will try to find out whether corresponding arrangements are possible and whether travelling expenses can be refunded in certain cases.

12 APPENDIX: MEMBERSHIP LIST

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12-17 Remark: Hence, the Group meets a condition set up by the VAMAS chairman according to which membership should be balanced between those with expertise in statistical methods and those with expertise in materials science and engineering.

13 APPENDIX: APPLICATION FOR MEMBERSHIP

Mr. Doerffel indicated that the following scientists from non-G7 countries are interested in the subjects of the Group and in an appropriate kind of membership:

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13-5 Remark: The VAMAS chairman was informed. It is intended to provide for contact and information. A final decision should, however, be left to the next Meeting of the Steering Committee.

14 APPENDIX: POSSIBLE CONTRIBUTIONS OF PARTICIPANTS TO THE WORKING FIELDS OF THE GROUP

14-1 C. Amzallag

14-1-1 Service: Commercial packages on PC and VAX systems - SAS, BBN, Statgraphics - for collaborative studies and analysis-of-variance models.

14-1-2 Optimization: Finite element analyses; development of postprocessors to finite element codes: fatigue and fracture mechanics problems.

14-1-3 Research: Bayesian approaches.

14-1-4 Consulting: Statistical computing; materials testing.

14-2 K. Doerffel

14-2-1 Service: Specific problems in connection with interlaboratory studies; modified versions of classical analysis-of-variance models.

14-2-2 Optimization: ./.

14-2-3 Research: degradation failures.

14-2-4 Consulting: Statistics, analytical chemistry.

14-3 T. Fritz

14-3-1 Service: Collaborative Studies; analysis of variance models; experimental design; computer programs.

14-3-2 Optimization: Response surface methodology; Finite element applications; Monte Carlo Method; Supercomputing.

14-3-3 Research: Discrimination of test methods; Bayesian approaches.

14-3-4 Consulting: Mathematics; computational statistics; materials research and testing.

14-4 W. Gerisch

as 14-3.

14-5 J. Häyrynen

14-5-1 Service: Support for organization of programmes; design of experiments.

14-5-2 Optimization: Environmental effects; MC method.

14-5-3 Research: Personnel effects.

14-5-4 Consulting: Materials testing in extreme temperatures; complex mechanical testing; organization of programmes; specimen size effects.

14-6 W. Muster

14-6-1 Service: ./.

14-6-2 Optimization: Failure probability analysis of ceramic parts; threedimensional NDE high resolution techniques and imaging evaluation.

14-6-3 Research: Materials oriented statistical procedures; experimental methodology and statistical evaluation.

14-6-4 Consulting: ./.

14-7 K. Suzuki

14-7-1 Service: Statistical analysis; statistical computer simulation; analysis of degradation data.

14-7-2 Optimization: Methods of experimental design (classical and Taguchi); nonlinear optimization (random effects models).

14-7-3 Research: Methods of the analysis of degradation data; analysis of lifetime data.

14-7-4 Consulting: Computational statistics.

14-8 S. Steinborn

as 14-3.

14-9 T. Svensson

14-9-1 Service: Statistical modelling; statistical analysis; programs: MatLab, Statgraphics.

14-9-2 Optimization: ./.

14-9-3 Research: Statistical analysis of fatigue.

14-9-4 Consulting: Mathematics; statistics; materials testing.

15 APPENDIX: COMPUTER PROGRAMS ON COLLABORATIVE STUDIES

The following list is given on request of the participants:

15-1 ISO 5725: BSI, Precision of Test Methods, Guide for the Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests (BS 5497, Part 1: National Appendix), London: BSI 1987: Random Effects One-Way Layout: FORTRAN 77.

15-2 ISO 4259: L. J. Painter, Report to members of ISO/TC 28/WG2: Work accomplished and to be presented for review at ISO/TC 28/WG2 Meeting in Stavanger, Norway, 2-5 June 1986: Random Effects Two-Way Layout: SAS.

15-3 ASTM E 691: ASTM, ASTM Interlaboratory Data Analysis Software, E 691, User's Guide, Philadelphia: ASTM 1991: Random Effects One-Way Layout, FORTRAN 77.

15-4 see also Chapter 6-3.

16 APPENDIX: MANUSCRIPTS, PAPERS AND STANDARDS

In connection with the meeting the following documents were received by the Chairman:

16-1 F. Bastenaire, Role of statistics in the certification of standard reference materials, Geostandards Newsletter 3, 2 (1979) 115-120 (Amzallag).

16-2 J. P. Gauthier, C. Amzallag, J. A. Le Duff, E. Diaz, High cycle fatigue of austenite (316L) and ferritic (A508) steels under random loading, Fifth International Spring Meeting SF2M-ASTM-SAE, June 15-17, 1992, Paris, France (Amzallag).

16-3 C. Amzallag, J. A. Le Duff, C. Ribin, G. Mottet, Crack closure measurements and analysis of fatigue crack propagation under variable amplitude loading, Fifth International Spring Meeting SF2M-ASTM-SAE, June 15-17, 1992, Paris, France (Amzallag).

16-4 F. Faure P. Ould and P. Balladon, Effect on long term aging on the mechanical properties of stainless steel welds in P.W.R., Trends in Welding Research, 1-5 June 1992, Gatlinburg, TN, USA (Amzallag).

- 16-5 P. Balladon, B. Duquaire, J. M. Lardon and G. Gay, Prediction of maximum CTOD values of high strength offshore steels using a local approach, 24th National Symposium on Fracture Mechanics, ASTM 1992 (Amzallag).
- 16-6 G. Baundry, G. Giroud, G. Duplomb, J.H. Bulit, D. Girodin, G. Dudragne and G. Jacob, Fatigue property evaluation of bearing steels produced by rotary continuous casting, Impact of Improved Material Quality on Properties, Product Performance, and Design, ASME, MD-Vol. 28 (1991) 27-39 (Amzallag).
- 16-7 G. Baundry, G. Duplomb, G. Giroud, J. Saleil and G. Jacob, Use of rotary continuous cast steel for production of tubes for bearing applications, Creative Use of Materials for Rolling Contact Bearings, ASTM, November 6-8 (1991) San Diego, CA (Amzallag).
- 16-8 normalisation française, Produits métalliques: Pratique des essais de fatigue oligocyclique, A03-403, afnor 1990 (Amzallag).
- 16-9 normalisation française, Produits métalliques: Pratique des essais de vitesse de propagation de fissure en fatigue, A03-404, afnor 1991 (Amzallag).
- 16-10 normalisation française, Produits métalliques: Essais de fatigue, Traitement statistique des données, A03-405, afnor 1991 (Amzallag).
- 16-11 K. Doerffel, M. Schulze und S. Spauszus, Auswertung eines internationalen Gemeinschaftsversuchs auf dem Gebiet der Normalprobenanalytik, Neue Hütte 7 (1962) 377-379 (Doerffel).
- 16-12 K. Doerffel, Gemeinschaftsversuch unter Anwendung eines Probenpaares, Neue Hütte 12 (1967) 762-763 (Doerffel).
- 16-13 W. Gerisch and T. Fritz, Statistical and computational aspects of the discrimination of CEC test methods, On two computer programs, Fourth CEC International Symposium, Birmingham, UK, 1993 (Gerisch).
- 16-14 T. Fujimori, T. Miyazu and K. Ishikawa, Evaluation of analytical methods using signal-noise ratio as a statistical criterion, Microchemical Journal 19, (1974) 74-85 (Suzuki).
- 16-15 K. Suzuki and M. Ashitate, A necessary and sufficient condition for monotone procedure in a maintenance policy with monitors, Rep. Stat. Appl. Res. JUSE 38, 2 (1991) 11-18 (Suzuki).
- 16-16 K. Suzuki, Nonparametric estimation of lifetime distributions from a record of failures and follow-ups, Journ. Amer. Stat. Assoc. 80, 369 (1985) (Suzuki).
- 16-17 M. Crowder and L. Wolstenholme, Statistical methodology for inter-comparison studies, NPL (Ed.), Aspects of Materials Metrology and Standards for Structural Applications, Elsevier (Wolstenholme).
- 16-18 M. Crowder, Interlaboratory comparisons: Round robins with random effects, Appl. Statist. 41, 2 (1992) 409-425 (Wolstenholme).

APPENDIX B

THE TWA / ADVISORY GROUP IN BRIEF

OBJECTIVES

- To provide statistical and computational support in the phases of the design and the analysis of classical interlaboratory studies on the basis of existing guidelines, standards and computer programs (**Service**).
- To provide mathematical, statistical and computational support for optimizing specimens, processes, experiments, interlaboratory studies and test methods by approaches of experimental design and quality engineering (Taguchi), finite elements, and mathematical modelling and computer simulation (**Optimization**).
- To identify needs and undertake corresponding problem-oriented research and development on statistical models and computational approaches for collaborative projects of selected structure and purpose (**Research**).
- To offer consultation and help in mathematics, computational statistics, scientific computing, selected fields of chemometrics, and materials science and engineering (**Consulting**).

6-1 INITIATION AND MEMBERSHIP. The work of bringing together the Group in accordance with corresponding statements in the minutes of the Steering Committee meeting 14 (December 19-20, 1991, Tokyo: p. 20) and 15 (June 4-5, 1992, Berlin: pp. 5-6) has now come to a certain end so that the initial meeting of the Group could be held on 27 November 1992 in Berlin. At that time, the Group had sixteen members from eight countries. In addition, applications for membership from four additional countries were announced. Membership in the Group is balanced between those with expertise in the mathematical and computational fields, in particular in computational statistics, and those with expertise in materials science and engineering, where in both cases important fields of national and international pre-standards research are covered.

6-2 TASKS. Referring to the tasks of the Group, one has to observe first that the Group was designed to support collaborative projects of other TWAs by theoretical methods and numerical procedures of applied and computational statistics. This led to the spectrum of services to be offered at the TWA level, as given more detailed in the box. In addition to the scientific activities an-

nounced *ibidem* under the terms Service, Optimization, Research and Consulting, the minutes of the Steering Committee meeting 15 indicate that the Group will be asked to review, prior to submission to Co-ordinators and Steering Committee, all new project requests that contain testing programs.

6-3 INITIAL MEETING. On the agenda of the initial meeting were, essentially, short presentations of the participants on their institutes and work, the discussion of a background document on the situation in fields in which activities of the Group can be expected, an inquiry on possible contributions of the participants to these fields, the handling of requests for cooperation from other groups, and further concrete preliminary steps which have to be taken before the work of the Group can start.

Apart from the obvious fact that the statistical coverage of the structural variety of possible VAMAS collaborative projects by existing guidelines, standards and computer programs is unsatisfactory, the following needs were identified: (i) The use of the term Interlaboratory Study (and related terms) requires some kind of Standard Practice. Suggestions are contained in a report by W. Horwitz on the AOAC Interlaboratory Studies Committee (1989) and in the background document. (ii) A guideline on statistical and experimental requirements to be met when establishing a VAMAS test method should be set up.

6-4 BACKGROUND DOCUMENT. Observing comments and proposals made in connection with the initial meeting, the 1992 version of the background document was revised and enlarged, slightly. The new version is now published in the main part of the present BAM / VAMAS report.

6-5 FURTHER PLANS. To provide for a further dissemination of mathematical and computational means appropriate for the purposes of VAMAS and other research in materials science and engineering, the Group plans the preparation of workshops to introduce interested members of other TWAs or related institutions into corresponding fields of statistics and optimization. Moreover, the organization of scientific conferences 'Computational Materials Science and Engineering' was taken into consideration.

6-6 START OF WORK. After full approval by the Steering Committee, the Group is prepared to start its scientific work in the fields Service, Research and Consulting. The treatment of more advanced problems in Optimization can require additional preparatory activities and should be postponed to the time after the immediate starting phase.

Technical Reports of BAM published up to now

- Nr. 1/1968
Forschung und Entwicklung in der Bundesanstalt für Materialprüfung (BAM)
Rechenschaftsbericht für den Bundesminister für Bildung und Wissenschaft
- Nr. 2/1970 (vergriffen)
G. Andreae
Zum Problem des Feuchtigkeitsschutzes von Dehnungsmeßstreifen und Halbleitergebern
- Nr. 3/1970
J. Ziebs
Über das mechanische Verhalten von Aluminium-Stahl-Freileitungsseilen als Beispiel für Verbundbauteile
- Nr. 4/1970 (vergriffen)
A. Burmester
Formbeständigkeit von Holz gegenüber Feuchtigkeit – Grundlagen und Vergütungsverfahren
- Nr. 5/1971
N. Steiner
Die Bedeutung der Netzstellenart und der Netzwerkkettendichte für die Beschreibung der elastischen Eigenschaften und des Abbaus von elastomeren Netzwerken
- Nr. 6/1971
P. Schneider
Zur Problematik der Prüfung und Beurteilung des Luftschallschutzes von Bauelementen bei unterschiedlichen Einbaubedingungen
- Nr. 7/1971
H.-J. Petrowitz
Chromatographie und chemische Konstitution – Untersuchungen über den Einfluß der Struktur organischer Verbindungen auf das Verhalten bei der Dünnschicht-Chromatographie
- Nr. 8/1971
H. Veith
Zum Spannungs-Drehungs-Verhalten von Baustählen bei Wechselbeanspruchung
- Nr. 9/1971
K.-H. Möller
Untersuchung über die sichernde Wirkung poröser Massen in Acetylenflaschen
- Nr. 10/1972
D. Aurich, E. Martin
Untersuchungen über die Korngrößenbestimmung mit Ultraschall zur Entwicklung einer für die Praxis geeigneten zerstörungsfreien Meßmethode
- Nr. 11/1972
H.-J. Krause
Beitrag zur Kenntnis der Schnittriefenbildung und Schnittgütwerte beim Brennschneiden
- Nr. 12/1972
H. Feuerberg
Über Veränderungen von Nylon-6-Fasern beim Texturieren
- Nr. 13/1972
K.-H. Habig, K. Kirschke, W.-W. Maennig, H. Tischer
Festkörpergleitreibung und Verschleiß von Eisen, Kobalt, Kupfer, Silber, Magnesium und Aluminium in einem Sauerstoff-Stickstoff-Gemisch zwischen 760 und $2 \cdot 10^{-7}$ Torr
- Nr. 14/1972
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Untersuchungen zur Amplitudenabhängigkeit der Ultraschalldämpfung in Metallen bei 20 kHz
- Nr. 15/1972
H. Pohl
Studie und Probleme der chemischen Edelmetall-Analyse
- Nr. 16/1972
E. Knublauch
Über Ausführung und Assagefähigkeit des Normbrandversuches nach DIN 4102, Blatt 2, im Hinblick auf die Nachbildung natürlicher Schadensfeuer
- Nr. 17/1972
P. Reimers
Aktivierungsanalyse mit schnellen Neutronen, Photonen und geladenen Teilchen
- Nr. 18/1973
W. Struck
Das Spröbruchverhalten des Baustahles R St 37-2 N in geschweißten Konstruktionen, dargestellt mit Hilfe der Methode des Temperaturvergleiches
- Nr. 19/1973
K. Kaffanke, H. Czichos
Die Bestimmung von Grenzflächentemperaturen bei tribologischen Vorgängen
- Nr. 20/1973
R. Rudolphi
Brandrisiko elektrischer Leitungen und Installationen in Wänden
- Nr. 21/1973
D. Klaffke, W. Maennig
Die kontinuumsmechanische Erfassung des zeitlichen Ablaufs der elastisch-plastischen Dehnungen bei der Zerrüttung
- Nr. 22/1973
R. Rudolphi, E. Knublauch
Untersuchungen für ein Prüfverfahren zur Bemessung der Brandschutzbekleidung von Stahlstützen
- Nr. 23/1973
W. Ruske
Reichs- und preußische Landesanstalten in Berlin Ihre Entstehung und Entwicklung als außeruniversitäre Forschungsanstalten und Beratungsorgane der politischen Instanzen
- Nr. 24/1973
J. Stanke, E. Klement, R. Rudolphi
Das Brandverhalten von Holzstützen unter Druckbeanspruchung
- Nr. 25/1973
E. Knublauch
Über das Brandgeschehen vor der Fassade eines brennenden Gebäudes unter besonderer Berücksichtigung der Feuerbeanspruchung von Außenstützen
- Nr. 26/1974
P. Jost, P. Reimers, P. Weise
Der Elektronen-Linearbeschleuniger der BAM – Eigenschaften und erste Anwendungen
- Nr. 27/1974
H. Wüstenberg
Untersuchungen zum Schallfeld von Winkelpföcken für die Materialprüfung mit Ultraschall
- Nr. 28/1974
H. Heinrich
Zum Ablauf von Gasexplosionen in mit Rohrleitungen verbundenen Behältern
- Nr. 29/1974
P. Schneider
Theorie der dissipativen Luftschalldämmung bei einem idealisotropen porösen Material mit starrem Skelet für senkrechten, schrägen und allseitigen Schallfall
- Nr. 30/1974 (vergriffen)
H. Czichos, G. Salomon
The Application of Systems Thinking and Systems Analysis to Tribology
- Nr. 31/1975
G. Fuhrmann
Untersuchungen zur Klärung des Verhaltens thermoplastischer Kunststoffe bei Wechseldehnungsbeanspruchung
- Nr. 32/1975
R. Rudolphi, B. Böttcher
Ein thermo-elektrisches Netzwerkverfahren zur Berechnung stationärer Temperatur- und Wärmestromverteilungen mit Anwendungsbeispielen
- Nr. 33/1975
A. Wagner, G. Kieper, R. Rudolphi
Die Bestimmung der Temperaturleitfähigkeit von Baustoffen mit Hilfe eines nichtstationären Meßverfahrens
- Nr. 34/1976 (vergriffen)
H.-J. Deppe
Untersuchungen zur Vergütung von Holzwerkstoff Nr. 35/1976
E. Limberger
Der Widerstand von Platten, die als Beplankungsmaterial leichter Wände verwendet werden, gegenüber dem Aufprall harter Körper – Vorschlag für ein Prüfverfahren –
- Nr. 36/1976 (vergriffen)
J. Hundt
Wärme- und Feuchtigkeitsleitung in Beton unter Einwirkung eines Temperaturgefälles
- Nr. 37/1976
W. Struck
Die stoßartige Beanspruchung leichter, nichttragenden Bauteile durch einen mit der Schulter gegenprallenden Menschen – Vorschlag für ein Prüfverfahren –
- Nr. 38/1976
K.-H. Habig
Verschleißuntersuchungen an gas-, bad- und ionisiertem Stahl 42 CrMo 4
- Nr. 39/1976
K. Kirschke, G. Kempf
Untersuchung der viskoelastischen Eigenschaften von Flüssigkeiten (mit nicht-Newtonschem Fließverhalten) insbesondere bei höherer Scherbeanspruchung
- Nr. 40/1976
H. Hantsche
Zum Untergrundabzug bei energiedispersiven Spektren nach verschiedenen Verfahren
- Nr. 41/1976
B. Böttcher
Optische Eigenschaften cholesterinischer Flüssigkeiten
- Nr. 42/1976
S. Dietlen
Ermittlung der Mindestzündenergie brennbarer Gas in Mischung mit Luft
- Nr. 43/1976
W. Struck
Das Spröbruchverhalten geschweißter Bauteile aus Stahl mit zäh-sprödem Übergang im Bruchverhalten dargestellt mit Hilfe der Methode des Temperaturvergleiches
- Nr. 44/1976
W. Matthees
Berechnung von räumlichen, linear elastischen Systemen, die aus finiten Stab- und Balkenelementen zusammengesetzt sind, unter Verwendung des Programms „Stab-Werk“
- Nr. 45/1976
W. Paatsch
Untersuchung des Elektrodenverhaltens im Vakuum aufgedampfter Metallschichten
- Nr. 46/1977 (vergriffen)
G. Schickert, H. Winkler
Versuchsergebnisse zur Festigkeit und Verformung von Beton bei mehraxialer Druckbeanspruchung
Results of Test Concerning Strength and Strain of Concrete Subjected to Multiaxial Compressive Stresses
- Nr. 47/1977
A. Plank
Bautechnische Einflüsse auf die Tragfähigkeit von Kunststoffdübeln für Fassadenbekleidungen
- Nr. 48/1977
U. Holzlöhner
Setzung von Fundamenten infolge dynamischer Last angewendet auf die Fundamente einer geplanten Schnellbahn
- Nr. 49/1977
G. Wittig
Untersuchungen zur Anwendung von Mikrowellen in der zerstörungsfreien Prüfung
- Nr. 50/1978 (vergriffen)
N. Czaika, N. Mayer, C. Amberg, G. Magiera, G. Andreae, W. Markowski
Zur Meßtechnik für die Sicherheitsbeurteilung und -überwachung von Spannbeton-Reaktordruckbehältern
- Nr. 51/1978
J. Sickfeld
Auswirkung von chemischen und physikalisch-technologischen Einflußfaktoren auf das Beständigkeitsverhalten von Oberflächenbeschichtungen auf der Basis von Reaktionsbeschichtungstoffen
- Nr. 52/1978
A. Tomov
Zum Einfluß der Gleitgeschwindigkeit auf das tribologische Verhalten von Werkstoffen hoher Härte bei reiner Festkörperreibung
- Nr. 53/1978
R.-G. Rohrmann, R. Rudolphi
Bemessung und Optimierung beheizbarer Straßen- und Brückenbeläge
- Nr. 54/1978
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Magnetisches Verhalten dünner Eisenschichten bei mechanischer Wechselbeanspruchung
- Nr. 55/1978
D. Klaffke
Beobachtung und Orientierungsbestimmung der Oberflächenkristallite polykristalliner 99,999 %-Al-Proben bei Biegewechselbeanspruchung
- Nr. 56/1979
W. Brünner, C. Langlie
Stabilität von Sandwichbauteilen
- Nr. 57/1979
M. Stadthaus
Untersuchungen an Prüfmitteln für die Magnetpulverprüfung

- vestigations on Inspection-Media for Magnetic article-Testing
r. 58/1979
I. Struck
ermittlung des Bauteilwiderstandes aus Versuchsergebnissen bei vereinbartem Sicherheitsniveau
- r. 59/1979
J. Plauk
mittlung der Verformungen biegebeanspruchter tahlbetonbalken mit der Methode der Finiten Elemente unter besonderer Berücksichtigung des Ver- undes zwischen Beton und Stahl
- r. 60/1979
J. Spreckelmeyer, R. Helms, J. Ziebs
ntersuchungen zur Erfassung der Kaltformbarkeit on Feinblechen beim Strecken
- r. 61/1979
J. Richter
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I. Gerisch, G. Becker
eomagnetobiologisch bedingter Zusammenhang wischen der Fraßaktivität von Termiten und der Zahl er Sterbefälle
- r. 63/1979
J. Behrend, J. Ludwig
ntersuchungen an Stopfbuchsen von Ventilen und chiebern für Gase
- r. 64/1980
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- r. 65/1980
Schmidt, D. Aurich, R. Helms, H. Veith, J. Ziebs
ntersuchungen über den Einfluß des Spannungszu- tandes auf bruchmechanische Kennwerte
- r. 66/1980
I. Hattwig
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- r. 67/1980
V. Matthees
eitrag zur dynamischen Analyse von vorgespannten nd vorbelasteten Feder-Masse-Systemen mit ver- nderlicher Gliederung unter stoßartiger Beanspru- hung
- r. 68/1980
I. Petersohn
berflächenmeßverfahren unter besonderer Berück- ichtigung der Stereomeßtechnik.
ntwicklung eines volleuzentrischen Präzisions- ionimeters
- r. 69/1980
Buchhardt, P. Brandl
ntersuchungen zur Integrität des Liners von Reak- ortsicherheitshüllen (Containments) in Stahlbeton- nd Spannbetonbauweise
- r. 70/1980 (vergriffen)
I. Schickert
chwellenwerte beim Betondruckversuch
- r. 71/1980
V. Matthees, G. Magiera
ntersuchungen über durch den Boden gekoppelte ynamische Wechselwirkungen benachbarter Kern- raftwerksbauten großer Masse unter seismischen inwirkungen
- r. 72/1980
I. Rudolphi
bertragbarkeit der Ergebnisse von Brandprüfungen m Beispiel von Stahl- und Holzstützen
- r. 73/1980
J. Wegener
ergleichende Untersuchungen zum Tragverhalten on Klemmkupplungen für Stahlrohrgerüste nach be- stehenden deutschen Prüfvorschriften und geplan- ten europäischen bzw. internationalen Prüfnormen
- r. 74/1980
I. Rudolphi, R. Müller
LGOL-Computerprogramm zur Berechnung zweidi- ensionaler instationärer Temperaturverteilungen it Anwendungen aus dem Brand- und Wärmeschutz
- r. 75/1980
I.-J. Heinrich
eitrag zur Kenntnis des zeitlichen und örtlichen ruckverlaufs bei der plötzlichen Entlastung unter ruck stehender Behälter und Behälterkombinationen
- Nr. 76/1980
D. Klaffke, W.-W. Maennig
Deformationsverhalten von Rein- und Reinstalumi- num sowie Cu 99,9 und St 37 bei Biegewechselbean- spruchung im Rasterelektronenmikroskop
- Nr. 77/1981
M. Gierloff, M. Maulzsch
Untersuchung des Verhaltens von Lagerzementen
- Nr. 78/1981
W. Rücker
Dynamische Wechselwirkung eines Schienen- Schwelensystems mit dem Untergrund
- Nr. 79/1981
V. Neumann
Ein Beitrag zur Untersuchung der wasserstoffbeein- flußten Kaltrißneigung höherfester niedriglegierter Feinkornbaustähle mit dem Implantversuch
- Nr. 80/1981
A. Plank, W. Struck, M. Tzschätzsch
Ursachen des Teileinsturzes der Kongreßhalle in Ber- lin-Tiergarten
- Nr. 81/1981
J. Schmidt
Graphisch-rechnerisches Verfahren zum Erfassen der Zündhäufigkeit zündbarer Stoffe; Anwendung auf Datenmaterial aus dem Bereich der Statistik
- Nr. 82/1982
R. Helms, H.-J. Kühn, S. Ledworowski
Zur werkstoffmechanischen Beurteilung des Kerb- schlagbiegeversuches
Assessment of the mechanical behaviour of mate- rials in the notched bar impact test
- Nr. 83/1982
H. Czichos, P. Feinle
Tribologisches Verhalten von thermoplastischen Kunststoffen
- Kontaktdeformation, Reibung und Verschleiß, Ober- flächenuntersuchungen -
- Nr. 84/1982
R. Müller, R. Rudolphi
Übertragbarkeit der Ergebnisse von Brandprüfungen im Kleinprüfstand (Vergleichsversuche)
- Nr. 85/ISBN 3-88314-231-X/1982
H. Czichos
Technische Materialforschung und -prüfung
- Entwicklungstendenzen und Rahmenvorschläge für ein EG-Programm „Basic Technological Research“ - Materials Research and Testing
- Development Trends and Outline Proposals for a Community Programme “Basic Technological Re- search” -
- Nr. 86/ISBN 3-88314-232-8/1982
K. Niesel, P. Schimelwitz
Zur quantitativen Kennzeichnung des Verwitterungs- verhaltens von Naturwerksteinen anhand ihrer Gefü- gemerkmale
- Nr. 87/ISBN 3-88314-240-9/1982
B. Isecke, W. Stichel
Einfluß baupraktischer Umgebungsbedingungen auf das Korrosionsverhalten von Spannstählen vor dem Injizieren
- Nr. 88/ISBN 3-88314-254-9/1983
A. Erhard
Untersuchungen zur Ausbreitung von Longitudinal- wellen an Oberflächen bei der Materialprüfung mit Ul- traschall
- Nr. 89/ISBN 3-88314-263-8/1983
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- Nr. 90/ISBN 3-88314-264-6/1983
K. Brandes, E. Limberger, J. Herter
Kinetische Grenztragfähigkeit von stoßartig belaste- ten Stahlbetonbauteilen
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