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Report on

Asia-Oceania
I.S.O. 5660 Cone Calorimeter
Inter-laboratory trials

Prepared by

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September 1995

Coordinators' Preface

Heat release rate is becoming considered as the most important property for the fire hazard assessment of materials. The oxygen consumption principle has led to the development of heat release measurement for the different scales and configurations under simulated fire environments during the 1980's. Especially the Cone Calorimeter, currently under the process of international standardization as ISO 5660, is becoming a bench-scale laboratory standard for reaction to fire assessments.

However, the Cone Calorimeter is a sophisticated apparatus in comparison with conventional fire test apparatuses. Heat release rate of a material under specific irradiance is obtained only after the combination of very different measurements, namely gas concentrations, gas flow rate and heat flux. The oxygen consumption principle needs fairly complicated instrumentation and calculation, many of the procedure and other technical components of which are not yet standardized in practical way. Calibration of the heating conditions with heat flux gages needs careful treatment and maintenance of the instrumentation, for which, nevertheless, most of fire laboratories are not yet able to check the results against real values of irradiance.

In view of the importance of the Cone Calorimeter as a basic tool for fire hazard assessment and as a candidate for the future key regulatory reaction-to-fire test, several interlaboratory trials have been conducted in the late 1980's. Most of the participants of the Cone Calorimeter round robin were from Western Europe and North America since there were very few laboratories using the Cone Calorimeter equipments in Asia-Oceania until around the beginning of 1990's. However, many of the Cone Calorimeter users in this district suffer from technical difficulties of the equipments and from difficulties in establishing confidence in their own data.

In 1993, the Ministry of Construction of the Japanese Government started a five-year research & development programme for the possible renovation of the fire test methods. As part of this programme, interlaboratory trials with ISO reaction-to-fire tests were initiated first with ISO 5657 Ignitability test in 1993. The interlaboratory trial was extended to include the ISO 5660 Cone Calorimeter in 1994. Considering the growing interest in international fire research coordination in the Asia-Oceania, the invitation to the Cone Calorimeter round robin was circulated to all fire laboratories using Cone Calorimeter available in time for the round robin in this district. Also fire laboratories outside the district who were interested in the participation were similarly invited. Ten fire laboratories including two from outside Asia-Oceania finally decided to participate in the round robin. The participation from outside Asia-Oceania is appreciated not only because their own experiences have led to the improvement of the quality of the interlaboratory trials but also because their participation could be a germ of a wider international framework for cooperation of fire laboratories which is believed to be a key for a world-wide coordination of the fire-growth testing in the future.

Relation and connection in the participating laboratories in this round robin, or even participating countries, are believed to be far weaker than those in the previous Cone Calorimeter round robins which were run within some more formal framework as ASTM or ISO. This fact has resulted in some difficulties in keeping consistent testing conditions in the participants, and this difficulty could be reflected in the result of the round robin. However, this difficulty is considered rather an essential characteristic in Asia-Oceania, and is a potential problem to be overcome when a wider inter-district round robin is intended in the future. The coordinators wish that the experience through this project become a basis for further cooperation of the participating laboratories, and also wisdom and ideas for a wider interlaboratory cooperations be derived from this experience. A new Cone Calorimeter round robin with wider scope is already planned on the basis of the experience of this round robin as a CIB W14 (Fire) subprogramme.

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Acknowledgment

The coordinators would like to acknowledge technical support of Dr. M.L. Janssens, American Forest and Paper Association, and Dr. V. Babrauskas, Fire Science and Technology Inc., throughout the round robin projects and the efforts of Dr. A. Marchal, STA fellow at Building Research Institute, who made the major contribution to the analysis and is the substantial author of this report. The coordinators thank all participants and the committee members of this project. The international Coordinator especially thanks Prof. W.K. Chow, the Hong Kong Polytechnic University, for the arrangement of the international participants meeting in Hong Kong in March 1995. For the majority of the round robin participants, it was the first opportunity to meet together.

Table of Contents

Introduction

Presentation

Purpose

As part of a large inter-laboratory calibration program initiated by the Ministry of Construction of the Japanese Government, the I.S.O. 5660 Cone Calorimeter has been submitted to an international round robin, including mainly Asian countries. If different cooperation programs were ever launched, through EU and USA, to bring a harmonization of fire tests, Asian laboratories still stayed out from such calibration platform. This round robin, conducted under the auspices of the Building Research Institute of Japan, aims to improve the measurements' quality of the participating laboratories and develop the reliability and accuracy of fire tests data.

The present document reports a statistical analysis of the first I.S.O. 5660 Cone Calorimeter inter-laboratory trials in Asia-Oceania, carried out in 1994-1995. The main objectives which have been sought were :

- To check the consistency of the Cone Calorimeter's outputs between the participants.

- To point out the major deviations and troubleshootings which could appear through the data processing, try to identify and to link them to their potential origins.

- To assess statistically suitable statements of the repeatability and the reproducibility achievable by the method.

- To enable recommendations to the participating laboratories in order to improve their operating experience in conducting Cone Calorimeter tests and to design some quality control procedures.

During the execution and the analysis of this round robin, but unluckly after its initiation, data and related reports about previous similar inter-laboratory trials on Cone Calorimeter (by A.S.T.M.) and on other fire tests (I.S.O. 5657) became available to the coordinators. In addition to the exposed initial objectives, those documents allow us to test another important item, the coherence and consistency between Cone Calorimeter round robins results or more specifically between some fire parameters.

Definition

The definition of the principles used in this report can be found in Section 3.1 of I.S.O. 5725 titled "Precision of test method". The most important ones are :

- *Coefficient of variation* : The standard deviation (of a particular statistic), divided by the mean value (of the same statistic).

- *Repeatability conditions* : Conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

- *Repeatability value (r)* : The value below which the absolute difference between two single test results obtained under repeatability conditions may be expected to lie with a probability of 95%.

- *Reproducibility conditions* : Conditions where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment.

- *Reproducibility value (R)* : The value below which the absolute difference between two single test results obtained under reproducibility conditions may be expected to lie with a probability of 95%.

Plan

Participants

Invitation for participation was circulated by August, 1994 to all fire laboratories in Asia-Oceania using the Cone Calorimeter equipment available in time for the round robin. Fire laboratories outside Asia-Oceania interested in this project were also invited to the round robin. As the result, the 10 fire laboratories listed below decided to participate in this round robin.

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Summary and analysis of the data have been prepared using the data from 8 laboratories out of the 10 participants which became available to the secretariat by the end of May, 1995. In this report, participants are only identified as alphabetical characters as Laboratory A, B, and so on. The identifications do not reflect the order of the names in the list above.

Planning and coordination of the round robin program were conducted by the executive committee consisting of M. Yoshida*, Y. Hasemi*, T. Harada, S. Kikuchi, K. Yoshida, Alec M.Y. Lei, I. Nakaya, Y. Tanaike (Japan Testing Center for Construction Materials) and S. Tasaka (General Building Research Corporation of Japan). Messrs Tanaike and Tasaka are not Cone Calorimeter users, but were invited to the project as experienced fire test experts. Dr. A Marchal (STA fellow at Building Research Institute) joined the project in 1995.

* Respectively General & International Coordinator, Building Research Institute.

Specimen

The following materials were selected as the specimens. All specimens of each material were arranged by Building Research Institute using the products from a same lot directly shipped by factories. 100mm x 100mm specimens were sent to the participants. Thickness depends on the material.

- PMMA sheet *(referred to as* PMMA*)*
  transparent, 10mm thick,
  density 1180 kg/m^3.
  Product from Mitsubishi-Kasei, Ltd. Japan, identified as "*Aclylite*".

- Medium Density Fiber board *(referred to as* MDF*)*
  untreated (natural wood color), 12mm thick,
  density 640 kg/m^3.
  Produced from tropical wood through arrangement of the Forestry and Forest Product Research Institute, Japan.

- polyVinyl chloride Coated Steel Plate *(referred to as* VCSP*)*
  black, 0.6mm thick,
  average density 6870 kg/m^3.
  Product from Toyo Kohan Ltd., Japan.

- Gypsum Board *(referred to as* GB*)*
  covered by paper sheets on both sides, 9.5mm thick,
  average density including the paper sheets 650 kg/m^3, paper 230~240 kg/m^2.
  Product from Yoshino Gypsum Ltd., Japan, identified as "*Tigerboard*".
  Graded as "*quasi-noncombustible material*" in the Japanese Building Standard Law.

- PolyVinyl Chloride sheet *(referred to as* PVC*)*
  white, 2mm thick,
  average density 1420 kg/m^3.
  Product from Takiron, Ltd., Japan, identified as "*Takiron Plate*".

Instructions

Two different irradiance levels ($30kW/m^2$ and $50kW/m^2$) were chosen. Only horizontal orientation has been selected. At least three replicate tests were to be conducted for each heat flux level for each material in randomized order for different materials. A two seconds interval was suggested for data sampling. Medium density fiber board specimens were to be conditioned before test. Calibration of heat release rate was to be made for two levels of heat release using methanol (100cc and 200cc) without radiation. Calibration of heat flux gage was to be done against either virgin heat flux gage or appropriate calibration furnace*.

* All Japanese participants were able to compare their own heat flux gage with a reference heat flux gage. It was regretful that this circulation was impossible for other participants because of the relatively tight schedule and potential import-custom problems.

Reported parameters

The following data were to be reported on each test.

- Initial mass and total mass loss during each test.
- Time to ignition.
- Heat release rate as a function of time.
- First peak of heat release rate and time to first peak.
- Average heat release rate at 180s.
- Total heat release at 180s, 300s and over the burning period.
- Average effective heat of combustion over the burning period.
- Burning period.

Schedule

September 1994	Shipping of specimens and instructions
December 1994	First deadline for data submission (Four laboratories delivered data)
January 1995	Committee meeting, preliminary analysis of the data, delivery of summarized data to participants
March 1995	Participants meeting
May 1995	Final deadline for data submission

Analysis of the data

Analysis method

The I.S.O. 5725 "Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests" has been adopted to calculate and analyze the data. The report to A.S.T.M. on Cone Calorimeter inter-laboratory trials has been used as a guideline as well. All the definitions and calculations used in this report can be found in those two documents. This method has been chosen here for two main reasons. First of all, the I.S.O 5725 provides a very important guidance about the data analysis procedures and especially recommendations on how to deal with the outlying observations (this point will be discussed later).

The second goal which has been aimed by choosing this standard concerns the comparison ability between the different round robins which have already been published on Cone Calorimeter purpose. The A.S.T.M. and the I.S.O. inter-laboratory trials have actually been proceeded by the same data analysis procedure and so, even if the chosen parameters as well as the products are not exactly the same, overall results can be compared.

Bias

The general average of a material's property, called "level of the test property", is not necessarily equal to the "true" value. The difference between these two data, when it exists, is called the "bias of the test method". In the case of Cone Calorimeter, bias seems difficult to report insofar that an independent true assessment of a variable's value remains hardly reachable. However, all data associated with heat release measurement (first peak of heat release rate, average heat release rate, partial heat evolved, total heat evolved, and so on) are reduced using the $\Delta h_c/r_o$ constant which is given with ±5% of uncertainty. Thus, a bias of around ±5% can be expected for these heat release related variables.

Irregularities

As prescribed in the I.S.O. 5725, gross errors have to be removed from the data analyses. Even if, as they have to be purged, they will not enter in consideration in the further data treatment, the presence of such important deviation must be staved off by an accurate operating protocol and a careful attention must be paid on the obtained observations. We could propose, as it has been written in the 'Memorandum for US Laboratories participating in the Cone Calorimeter round-robin testing', dated from April 28, 1988 that if any of the mean heat release values for the first 60, 180, 300 seconds after ignition, among the three replicates of one material at one set of conditions, differs by more than 10% from the average for the three replicates, an additional series of three tests has to be executed. The data that does not conform to the 10% variability should, of course, be identified and returned to the round-robin's organization.

This measure must decrease the numbers of gross errors detected and, on the same way, improve the reliability of the data together with the repeatability values. During the present round robin, this instruction may not have been applied.

In spite of the one-test-gross-errors, three general kinds of 'curiosities' have been noticed and are reported below.

Ignition of PVC and GB

Mat.	Irr.	Laboratory reference							
	kW/m²	B	D	E	F	G	H	I	J
GB	30	NI	-	-	NI	-	-	NI	NI
PVC	30	NI	NI	-	NI	-	-	NI	NI
	50	-	NI	-	-	-	-	NI	NI

Table I. PVC & GB's ignition occurrence (NI : No Ignition).

The results of the ignition 'success' are quite confusing to analyze since the ratio of 'no-ignition laboratories' on 'ignition-laboratories' could not lead to discard one or the another laboratory. The explanation of such discordant data should be inquired. The interpretation of the definitions of 'flash ignition' and 'sustained surface ignition' could be a key in the comprehension of this phenomenon.

Moreover one of the main factor influencing ignition time, and therefore, flame outbreak, that is to say just 'ignition', is by far the irradiance level. Some miscalibrations of the heat flux gage leading to an overestimate of the irradiance and consequently to an operating weaker radiant exposure, could thus be suspected. That potential overestimate may explain the scattering of ignition results, especially in the case of the 30 kW/m^2 runs.

In order to realize the importance of this ignition occurrence variability, it should be noticed that in the scope of the Asian inter-laboratory trials on I.S.O. 5657 'Ignitability test' conducted on exactly the same products, PVC under an exposure of 30 kW/m² has always been ignited and, thus, must obviously always be able to be ignited at higher heat fluxes (50 kW/m² for instance in our case)..

Concerning GB, just one among the six laboratories taking part in the I.S.O. 5657 'Ignitability test' round robin did not state any 'ignition time', defined as 'sustained surface ignition', for the gypsum board sample at 30 kW/m². However, its values of 'flash ignition' for that specimen agreed perfectly with the ignition time reported by the five other laboratories. This means that, even this 'outlying' laboratory succeeds somehow in igniting the GB sample under a 30 kW/m^2 irradiance, and makes a drastic contrast with the present results.

The heating process being quite analogous in both standards, the high percentage of no-ignition obtained here could appear as a major irregularity, and consequently must be carefully watched over. In the following study, the lack of data due to no-ignition runs will just be considered as missing data, without any statistical processes.

Another irregularity, or inconsistency rather, linked to the ignition occurrence variability lies in the fact that some laboratories reported different parameters (such as first peak of heat release rate or total heat evolved) even if they did not state any ignition time for the same run. If these data are consistent with the other laboratories results, that is to say in reproducibility condition, they will be processed as regular data, assuming that the lack of ignition time data was just due to a visual difficulty of the operator to assess the proper instant of flame appearance. If not, they will be discarded. However, as the use of such kind of observations could lend itself to suspicion, it will always be mentioned in the following analyses.

Missing data

Despite the ignition/no-ignition problem, just two laboratories computed the all ten parameters requested by the round robin organization as it is indicated in Table II. The others just skip over some variables, even if these data could be reachable (no physical or technical troubles with samples were notified). As there are no visible reasons which could explain this lack of data, the 'empty' parameters will be just regarded as missing data.

	Laboratory reference							
	B	D	E	F	G	H	I	J
N° of unstudied para.	3	0	0	3	2	3	7	6

Table II. Number of variables which have never been studied (Total requested : 10).

Nevertheless, this default of answers is unfortunate for several reasons, especially for the accuracy of the analysis and the relevancy of the conclusions. The comparison, for instance, between the performances of laboratory I or J and laboratory D or E becomes actually very critical, even in relative figures, insofar that the statistical analysis will not recover by far the same amount of entries.

Definition of the parameters

In the case of MDF's runs (at both levels of irradiance), laboratory H showed some clearly outlying values of first heat release rate peak and especially of time to first heat release rate peak (between 9 and 15 times larger than the average time value of the other laboratories). Looking more closely to their heat release curves, a misunderstanding of the parameter's definition has been pointed out. The given results were not the coordinates from the 'first' heat release rate peak, but the one from the 'maximum' heat release rate peak. Such errors did not occur for the other materials tested because first peaks were also maximum peaks. So, even if the parameter's definition was misunderstood, the results were however correct.

Considering that there was no reasons to keep these erroneous data, since they did not correspond to the requested parameters, they have been removed and replaced by the values we could have read from the heat release rate profiles. The graphical readings were, of course, not as accurate as data acquisition unit could be and it is the reason why these data must be marked and used carefully.

If other parameters definitions misunderstanding could be suspected (concerning mainly times and calculations), this is the only one which have been proved and corrected.

Nomenclature

According to the I.S.O. 5725 data analysis method and taking into account the previous remarks, the values obtained are analyzed in Tables III to XII (at the end of the present report), corresponding to the 10 requested parameters on the 5 materials tested at two levels of irradiance. The nomenclature used is :

- Mass loss: Remaining mass of the sample at the end of the test expressed in percentage of the initial mass (%).
- T_{ig} : Time to ignition (sustained flaming), expressed in seconds (s).

- f.h.r.r peak: First heat release rate peak (first extreme value of heat release rate), expressed in kilowatt per square meter (kW/m^2).

- T_f: Time to first heat release rate peak (time of occurrence of the first extreme value of heat release rate), expressed in seconds (s).

- PHE_{180}: Total heat evolved during the first 180 seconds after ignition (integration of the heat release rate profile from time to ignition to 180 seconds), expressed in megajoule per square meter (MJ/m^2).

- AHR_{300}: Average heat release rate computed over the period starting at time to ignition and ending at 300 seconds, (kW/m^2).

- PHE_{300}: Total heat evolved during the first 300 seconds after ignition (cf. PHE_{180}).

- THE: Total heat evolved during the entire test period, (MJ/m^2).

- e.h.c. : Effective heat of combustion (ratio of heat release rate on corresponding mass loss rate), expressed in megajoule per kilogram (MJ/kg).

- T_{end}: Test ending time, the end of the test being defined as when the mass loss rate drops below 2.5 g/s and the average mass loss rate over the next minute does not exceed 2.5g/s.

- No. of Labs: Number of laboratories from whom data analyses have been completed, that is to say, from whom data was available and *after* removing the statistical outliers (This last point differs from the definition used in A.S.T.M. inter-laboratory trials in which this number includes the statistical outliers).

- m, r, R: The mean (or 'level of the test property'), repeatability (inside one laboratory) and reproducibility (between laboratories) respectively, and as defined before.

- (a3;b3) : the coefficients of a regression line to fit a possible functional relationship between the repeatability r and the mean m ($r = a3 + b3*m$). From a statistical viewpoint, the fitting of a straight line is complicated by the fact that both r and m are estimates and thus subject to error. The I.S.O. 5725 recommends an iterative procedure to calculate the coefficients of such an equation. (a3;b3) are the results of the three steps' calculation (Most of time, a two step's calculation could be considered as the final result).

- (A3;B3) : the coefficients of a regression line to fit a possible functional relationship between the reproducibility R and the mean m ($R = A3 + B3*m$). These are the corresponding coefficients to (a3;b3) for the reproducibility.

- Ave. r, R: the overall averages of repeatability and reproducibility values for the same parameter. In term of fitting with the r (or R) versus m curves, this represents horizontal lines and gives then an overall repeatability and reproducibility values of the studied parameter. These values have to be used when the model for error is considered as independent of the mean.

• V.C. : the overall average by laboratory of the variation coefficient defined previously as the ratio of the standard deviation on the mean value of the same statistic

Results

After purging the gathered data from obvious irregularities (or inconsistencies) inside their three replicates runs, relative means and their standard deviations, Cochran's one-sided outlier's test was used to determine outlying laboratories in repeatability at each level. Dixon's test was used, at each level as well, for the reproducibility. For both tests, I.S.O. 5725 describes two types of outlying observations, based on their probabilities of occurring P:

- $P > 5\%$ (i.e. Cochran's or Dixon's test statistic is less than its 5% critical value) the item tested is accepted as correct.
- $5 > P > 1\%$ (i.e. the test statistic lies between its 5% and 1% critical values) the item tested is called ***Straggler*** and have to be marked. Stragglers could be retained as correct.
- $1\% > P$ (i.e., Cochran's or Dixon's test statistic is greater than its 5% critical value) the item tested is called ***Outlier*** and has to be marked. Outliers should be discarded.

Both types of outlying data are notified for each level. Stragglers have been retained as correct, while outliers have been removed from further calculations. Once an outlying entry has been identified, tests have been reconducted on the remaining data in order to check the presence of 'hidden' outlying observations.

Calculations of the mean, repeatability and reproducibility have been then computed for all levels and on each parameter, except when the number of retained set of data (initial data minus irregularities and outliers for one particular level of one variable) was less than 3. In such a case, it was considered that too few data were available to produce a relevant statistical analysis, and results have been replace by a horizontal stroke in the tables.

Once the mean, repeatability and reproducibility values have been assessed with regard to the previous remarks, a functional relationship between them (i.e. $r = f(m)$ and $R = f(m)$) has been sought. The fitting equations have been proceeded by a 3 iterative steps' method, as prescribed in the I.S.O. 5725, part 15.6 and have been plotted for each studied parameters. In addition to the coefficients of these regression lines, the averages of repeatability and reproducibility values over the whole set of materials for each variable have been indicated in order to choose, according the plot of r (and R) against m, the best satisfactory final estimation of repeatability and reproducibility, if ever.

Tables of overall coefficient of variation (V.C.) has been added for each parameter. Main notes have been made on each page, but some comments could be added.

Comments

Raw data

Mass loss (Table III)

The shape of the diagram r,R = *f*(m) leads us to prescribe a constant average value for the repeatability and the reproducibility instead of choosing the regression lines. The negative second coefficients of the latest (b3 and B3) confirm that choice. It is actually very seldom to get such negative slopes, the repeatability and reproducibility increasing, logically and most of time, with the mean. A constant value of 0.03 for the repeatability and 0.11 for the reproducibility could therefore be proposed.

Remembering the typical figures of the load cell accuracy (around ±1%), the obtained results are very good. However, the weight range of the tested products (starting from about 30g for the PVC samples to nearly 120g for the PMMA samples) might be too narrow to interpolate safely these precision figures to heavier (thicker) samples.

Time to ignition (Table IV)

A graphical good agreement is observed between the regression line and the calculated repeatabilities and reproducibilities. However, the very high value of the reproducibility (151% of the mean) of PVC under a 50 kW/m^2 irradiance has to be noticed (point far from the R3 line). During the I.S.O. 5657 round robin an analogous behavior of this same product has already been pointed out and was suspected to be due to the numerous transient flaming that PVC's ignition can produce.

As it has been done in these Ignitability test inter-laboratory trials, a best-estimate relationship between R and m may be obtained by removing that point. A more complete comparison between the two standards will be report in next chapter.

First peak of h.r.r *(Table V)*

As first remark, if the figures of repeatability could be considered as correct (around 10% of the mean) for most of materials, the reproducibility values stay very high. And this is of first importance knowing that heat release rate is the major data given by the Cone Calorimeter and somehow one of the most useful data to handle with fire behavior of materials (entry data for several fire propagation and flame spread models). It is thus hardly prejudicial for models harmonization and corroboration to obtained so large difference in the h.r.r assessment between laboratories.

Such a difference between repeatability and reproducibility may be reduced by improving the accuracy of the calibration factor (C). In fact, this factor, calculated by a methane calibration, intervene in h.r.r calculations as a constant multiplicative factor inside a laboratory and, thus, has quite no influence on repeatability conditions but could be an important item on reproducibility conditions (between laboratories). A harmonization of the calibration factor calculation process (methane specification, heat flux gage, ...) should be a clue to decrease the reproducibility values.

An other way to improve the consistency of h.r.r results between laboratories could be the harmonization of the drying agent located on the oxygen gas sampling line. Previous studies have shown that according to the nature and the quality of that chemical filter, some variations could occur in the oxygen concentration evaluation and thus in the h.r.r data.

These remarks are valid for all materials except PVC for which both repeatability and reproducibility are almost intolerable (around 40% of the mean for the repeatability and 260% in reproducibility!). Together with the previous statistical analysis of ignition time results from that specimen, we can already conclude that a Cone Calorimeter agreement on PVC inside and between this round robin participating laboratories is a failure, as, from many point of view, ignition time and rate of heat release are the main parameters which are looking for during a Cone Calorimeter experiment.

The statistical results of first peak of heat release rate of GB could be compared with the PVC's ones. That point could lead us to say that materials with a single peak shape of h.r.r profile give some difficulties to be correctly tested. But in the case of VCSP which presents a single narrow peak of h.r.r as well, the results, although high, are much smaller, in term of repeatability and reproducibility, than the two previous materials. We should thus emphasize on the ignition's occurrence variability (*cf. Irregularities*) that several laboratories encountered to ignite GB and PVC. This phenomenon could explain a further instability in the heat release profiles and consequently in the first peak value.

Even if its r and R values are, in average, the smallest of the whole set of materials, not so low reproducibility figures can surprisingly be observed for PMMA at both levels of irradiance. 'Surprisingly' because PMMA, and that is due to its degradation mode, is often said to be one of the best calibration material. However, these results are, by far, much more better than the analogous figures obtained during the A.S.T.M. round robin (R around 58% of the mean for the American inter-laboratory trials versus 28% for the present one).

Time to first peak of h.r.r *(Table VI)*

For that parameter, roughly the same remark than the previous one concerning the h.r.r peak parameter of PVC and GB, should be brought, i.e large reproducibility values certainly due to the ignition troubles. However, the repeatability range of that time variable lies in correct borders (around 11% of the mean in average), even better than the repeatability range of the other important timing data, the ignition time (around 15% of the mean in average).The comparison between these two times is quite interesting insofar that if we can consider that, at the first glance, ignition time is mainly governed by the external heat flux, the time to first peak of h.r.r also included an evaluation of oxygen analyzer response latency.

As for the calibration factor (C), the analyzer response delay time could be considered as a specification of each Cone Calorimeter device and so should not have any tremendous effect on the repeatability conditions. This fact could explain the good repeatability results we obtained, but we can wonder why they are even better, in average, than the ignition time repeatability values. This difference might prove that it is more delicate to judge the exact moment of the outbreak of the flame than to draw a vertical line on the heat release rate curve. We should thus emphasize on the main and sensible importance of an accurate definition of "Ignition Time". A second hypothesis could be that this time to first heat release peak might be more 'stable', more featuring, less subject to fluctuations than the previous one. However, the repeatability improvement between the two parameters is definitively not huge enough to secure such an assumption.

If the delay time must not have any influence on repeatability conditions, figures show, as we can expect, that this is not the case for reproducibility conditions. We can notice that, in the case of PMMA, where the first peak of heat release occurs at a late stage of the manipulation (mean time > 300s), the R coefficients are the best of the all studied materials, even better than the analogous figures concerning ignition time (in percentage of the mean), but it is the only case. On the opposite, for early stage peak occurrence, that is to say for all the other samples, results are worse. The PMMA's heat release curve does not present a real triangle peak shape as the other one (at the beginning of the manipulation at least) and one could wonder if the characteristic physical or chemical phenomena conducting to that particular and smooth shape, phenomena which must be featuring from PMMA degradation process, could explain such good results. But, as a matter of fact, this idea has to be given up insofar that, as we previously mentioned, the repeatability values of any of the samples are very good and thus do not allow any assumption on the 'stability' of one or the other material degradation processes.

In other words, and as it is logical to think, the oxygen analyzer delay time decreases heavily the between-laboratories' agreement for 'rapid' peak, i.e. during the initial stage of the run, but its effect fades for 'late' peak, just as if it was diluted by the time of occurrence. This observation could lead us to conclude that, as for the calibration factor and in the same time (during the same calibration), a mutual, and especially accurate, process of oxygen analyzer response latency assessment should be brought in order to decrease the reproducibility values.

***Partial and average heat release** (Table VII to IX)*

For that series of parameters, attention must be paid on the fact that four laboratories, maximum, took part in the study. Thus, results could not have the same "weight" for drawing conclusions. Nevertheless, for the three studied variables, the distributions of repeatability and reproducibility values in respect to the mean seem to abide by the regression lines. Moreover the slope coefficients of their respective equations lie in the same range of order (around 0.05 in repeatability and 0.25 in reproducibility).

The concentration of outlying data for the two partial cumulative parameters (*total h.r.r until 180 seconds & total h.r.r until 300 seconds*) from D laboratory is noticeable. In addition, this same laboratory has been identified three times as straggler for the average heat release parameter. Except E which have been identified straggler just one time, D is the only laboratory for these three parameters in which outlying data have been found (and 9 times!). That observation could point out laboratory D as a total 'outlying laboratory' as described in Par. 11.2.3.d of the I.S.O. 5725, and thus lead us to discard its whole data. Laboratory D has, nevertheless, been kept in this study.

***Total heat release** (Table X)*

Laboratory D has still been identified twice as outlier. Together with the previous remark, it can be guessed that D has undoubtedly some troubles with its integrated data. Being already identified twice outlier on 'time to first peak of h.r.r' and twice as well on 'h.r.r peak', these bad performances seem, at least, to make sense. In the case of laboratory H, three times outlier on that parameter but never on parameters which could be related to total heat release, the results are quite puzzling. For both laboratories, the explanation of such discordant data should be inquired.

If the 'calculated' points do not seem to follow with a good agreement the proposed regression lines, this is certainly due to the VCSP and GB specimens. Both of these materials do not include a large proportion of 'combustible' elements as it is proved by their mass loss (just around 5% for the first one and 15% for the '*quasi-noncombustible*' second one). Their resulting total heat releases are thus very small and involve the curve to pass by the origin.

It is allowed to think that a so low total heat release may give rise to some irrelevancies. GB and VCSP show actually some very large values of repeatability and reproducibility in term of percentage in respect to the mean. Moreover, as we mentioned previously in the case of GB and PVC, the ignition troubles can still be suspected to affect that parameter. Removing these points, (GB and VCSP) the slope coefficients of the fitting equation become very close to the A.S.T.M. results (b3 = 0.06 and B3 = 0.08 for the present study without GB and VCSP, versus 0.056 and 0.075 for the A.S.T.M. round robin).

Gathering all data about cumulative heat release, that is to say total heat release until 180 seconds, 300 seconds and over the total burning period, an overall graphic can be drawn in order to see how repeatability and reproducibility are evolving. If the regression coefficients of that line lie in the same range of order than the split data, no featuring evolution has been found.

Effective heat of combustion (Table XI)

Just three laboratories computed that value. If the VCSP's run is excepted, the values of repeatability and reproducibility seem to be constant around 1.6 and 2 respectively (disregarding the VCSP value), results in good agreement with the I.S.O. inter-laboratory trials in which constant bounds are obtained as well. The 'out of average' statistics of VCSP might be explained by a perturbation due to the steel plate (incombustible and which could act as a heat reflector) included in the samples.

The effective heat of combustion must not vary, theoretically, according to the external heat flux if degradation modes are constant (same energies of activation). For each of studied materials we can presume, according to the literature, that, at 30 kW/m^2 and 50 kW/m^2 levels of irradiance, their respective degradation processes have to be identical. Looking at the means, we can notice that this law is perfectly respected and this is a good indication of the reliability of the computed results, in repeatability conditions at least.

Test end time (Table XII)

If the repeatability value of that last parameter could be considered as acceptable in the majority of the case, the reproducibility values overtake quite always the mean. It seems that there is no reachable reproducibility value at all for that parameter. The regression lines attempts fail (with some negative coefficients) and an average value of repeatability or reproducibility is, according to the plot, impossible to accept.

Moreover, the results from Laboratories D and E seem definitively suspicious insofar that for all the runs of one same level, a constant value have been found out. Remembering how looks like a Cone Calorimeter run, this behavior is very improbable. Some confusions in the meaning of the ending test time could be suspected, and an accurate definition of the burning period (when the mass loss rate drop below 2.5 g/s and the average mass loss rate over the next minute does not exceed 2.5g/s) should be used and precisely respected.

Outlying entries distribution

The distribution of the outlying entries, in others words 'where' are the outliers and stragglers, has been studied in order to focus on the difficulties the participating laboratories encountered. This should bring some keys to understand the major causes of troubles and point out the improvements that should be done.

Distribution according to the type of data

In table XIII (next page), the distribution of the outliers and stragglers are given according to the type of data. The set of the ten requested parameters of the present round robin could be divided into three main groups : the 'simple', corresponding to a single apparatus, the 'cumulative', or integrated data, and the 'combined', or calculated data. Each main group could then be subdivided according to the dimension of each parameter (time, mass, heat, ...).

The row and the column called 'N° of part' indicate the number of manipulations which have been made (row : by laboratory, column : by parameter). The dashes denote that the parameter has not been studied at all by the laboratory. As the number of manipulations constantly varies, the outlying observations 'concentration', or 'ratio', have to be expressed in respect to the total amount of runs (two last rows and columns titled 'Tot rel.'). No distinction has been brought between repeatability outlying entries and reproducibility outlying entries. This point will be discussed later.

The last two rows could be, somehow, considered as a "reliability" ranking of the laboratories. E, F and G seem to give the best result, while laboratory D (with 13.6% of outliers) stays far away from the average percent of the seven other laboratories.

We also could notice that laboratory H would considerably increase its performances by solving its integration troubles (3/4 of its outlying entries). Unluckily, no data from its partial cumulative heat release were available. It is thus difficult to assess the cause of such outlying data. These unstudied parameters could have guide us to check if the only integration calculation process could be involved in the underestimates of total heat release, or if some other reasons have to be sought (burning time period definition, h.r.r base line, ...).

B laboratory seems to have difficulties with the time parameters and especially with ignition time. As its heat related variables remain correct, we can assume that it may not just be due to a cone irradiance misadjustment. However, no outlying values in a parameter assessment do not imply a perfect evaluation of that parameter.

The major part of outlying values of laboratory E concern time variables as well. However half of laboratory E's outliers come from 'Ending time', parameter which seems, according to the previous chapter, highly difficult to control. Moreover, there are some reasons to think that Laboratory E may have use an 'unusual' definition of that parameter insofar that for the three replicates of each run, exactly the same value to within a second has been found. These outliers must easily be eliminated using the proper definition of the burning period.

So few parameters have been computed by I and J laboratories that it is quite impossible to analyze further their results, even in percent. Nevertheless their number of outlying values is not negligible and some additional data should be provided to assume their possible causes of occurrence.

Type of data		Lab. ref	B		D		E		F		G		H		I		J		Tot		Outlier	Straggler
		State	o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	Tot rel (%)	
		N° of part	58		88		100		58		75		70		27		33		509			
Simple	Mass Loss	77							1					1	1	1			2	2	2.60	2.60
	Tig	68	3				1					1			1				5	1	7.35	1.47
	T peak	57	1		2	1		1	1						-		-		4	2	7.02	3.51
	T end	47	-				2		-		1		1		-		1		5	0	10.64	0.00
	Peak	76			2				1		1	1				1	1		5	2	6.58	2.63
Cumul.	Tot 180	35			3						-		-		-		-		3	0	8.57	0.00
	Tot 300	31			2	1					-		-		-		-		2	1	6.45	3.23
	Tot rhr	57			2	1							3		-		-		5	1	8.77	1.75
Combi.	Ave 300	33	-			3		1	-						-		-		0	4	0.00	12.12
	ehc	28	-		1	2			-				-		-		-		1	2	3.57	7.14
Total		509	4	0	12	8	3	2	3	0	2	2	4	1	2	2	2	0	32	15	6.29	2.95

Tot rel. (%)		B	D	E	F	G	H	I	J	Tot	A.S.T.M. results
	Outlier	6.90	13.64	3.00	5.17	2.67	5.71	7.41	6.06	6.29	***1.26***
	Straggler	0.00	9.09	2.00	0.00	2.67	1.43	7.41	0.00	2.95	***18.40***

Distribution of the outliers and stragglers according to the type of data.

Table XIII.

Concerning the two last columns, that is to say the relevancy of each parameter, as it has been guessed, the ending test time shows the worst results. Surprisingly, best percentages are found for mass loss and, to a lower degree, e.h.c. (linked to mass loss) although the regular accuracy of the load cell is around ± 1%.

Two of the most important parameters of the Cone Calorimeter, ignition time and especially peak of heat release, lie around 7% of outlying runs. This figure seems too high, even if the major influence of the numerous outliers from D on h.r.r peak and those from B on ignition time can be felt. However, no outlying variable (a parameter which would implicate a large number of outliers) has be found.

Distribution according to the different levels.

In table XIV, we tried to check the distribution of outliers and stragglers according to the levels of irradiance. We could suspect that for low heat flux, as ignition may be more delicate, the number of outlying data would increase.

Lab. ref		B		D		E		F		G		H		I		J		Tot			
State		o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	Tot. rel.	
Flux	N° part	58		88		100		58		75		70		27		33		509		Out.	Strag.
30	242			5	4	1	1	2		1	1	4		1	1			14	7	5.79	2.89
50	267	4		7	4	2	1	1		1	1		1	1	1	2		18	8	6.74	3.00
Tot	509	4	0	12	8	3	2	3	0	2	2	4	1	2	2	2	0	32	15	6.29	2.95
Outlier		6.90		13.64		3.00		5.17		2.67		5.71		7.41		6.06		6.29			
Straggler		0.00		9.09		2.00		0.00		2.67		1.43		7.41		0.00		2.95			

Table XIV. Distribution of outlying values according to the external heat flux irradiance.

It seems to be the case for Laboratory H (4 outliers), but we already know that 3/4 of its outlying data derived from the total heat release parameter and so are unlikely to be due to any ignition troubles. Moreover, the table XIV's two last columns shows an opposite overall behavior. Overall behavior which might lead us to conclude, on the contrary of our first idea, that the higher heat flux, the larger number of outliers.

This assumption could make sense insofar that higher the irradiance, quicker the manipulation, so smaller the time variables are (which represent 1/3 of the studied parameters) and consequently more difficult are their assessments. Laboratory B could be a good example of this presumed phenomenon with all its outliers occurring during the 50 kW/m^2 runs and, as we previously noticed, all for time variables.

However the results expressed in percent just differ from one point and, thus, a statistical distribution between the two levels can be guessed. According to these figures, it appears that external irradiance must not have any effect on the outlying values concentration.

In Table XV (next page), the above table XIV has been detailed by including the different type of specimen. The distribution of the outliers and stragglers is so given according to the products and the irradiance they were submitted to (in other words, the different levels). The same nomenclature with the previous tables has been used. The two last rows are, of course and obviously, exactly identical in the three tables.

Products		Lab. ref	B		D		E		F		G		H		I		J		Tot		Outlier	Straggler
		State	o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	o	s	Tot rel (%)	
		N° of part	58		88		100		58		75		70		27		33		509			
PMMA	30	56										1			1	1			1	2	1.79	3.57
	50	56			1			1											1	1	1.79	1.79
MDF	30	56			4	1													4	1	7.14	1.79
	50	56				1	1					1				1			1	3	1.79	5.36
VCST	30	54				2					1								1	2	1.85	3.70
	50	54	1		3	1								1					4	2	7.41	3.70
GB	30	44			1				2				3						6	0	13.64	0.00
	50	55			2	1									1		1		4	1	7.27	1.82
PVC	30	32				1	1	1					1						2	2	6.25	6.25
	50	46	3		1	1	1		1		1						1		8	1	17.39	2.17
Total		509	4	0	12	8	3	2	3	0	2	2	4	1	2	2	2	0	32	15	6.29	2.95

Tot rel. (%)		B	D	E	F	G	H	I	J	Tot	A.S.T.M. results
	Outlier	6.90	13.64	3.00	5.17	2.67	5.71	7.41	6.06	6.29	***1.26***
	Straggler	0.00	9.09	2.00	0.00	2.67	1.43	7.41	0.00	2.95	***18.40***

Distribution of the outliers and stragglers according to the products.

Table XV.

Some precision can be added, now, to the previous comment about H's way to improve its results. They must actually check not only their integration process, but also their gypsum board samples (with still 3/4 of its outliers). This concentration of outlying values could lead us to think that, as all the samples were given to be issued from the same batches, the conditioning operating system of GB samples in laboratory H has to be verified. Previous studies have shown that Cone Calorimeter's answers of materials as GB and MDF could be very sensitive to moisture contents and thus to prior test conditioning.

On the same point of view, laboratory B concentrates its outliers on PVC samples. And that makes sense with the previous remarks concerning its difficulties to assess properly the time variables. We already mentioned that PVC is inclined to give some transient flaming which could perturb the timing evaluation, and especially ignition time.

From the laboratory D columns of results, we can notice that there is no particular product on which outlying observations are focused, meaning that the cause of the D's outliers generation is unlikely to be linked to one or another product. A general inquiry must so be brought more on the experimental device and its whole operating conditions than on any particular result.

Despite what has been said previously about reproducibility values of some PMMA's parameters, PMMA shows in this table the best overall results and that could somehow justify its designation as 'calibration material'.

On the other hand, GB and, especially PVC take a large part to increase the outlying concentration of these inter-laboratory trials. We have already touched on the reasons for which most of the participants had difficulties to test them. All together, PVC and GB represent about 2/3 of the total amount of outliers found out during this round robin.

At last, on tables XIII and XV, A.S.T.M. Cone Calorimeter inter-laboratory trials overall results about outlying entries concentration have been indicated to allow a comparison. Without analyzing too far the figures (the A.S.T.M. round robin using different parameters and different products), two major points can be seen. The outliers concentration of the present round robin is much more higher than the A.S.T.M. one. But, on the other hand, stragglers percentages are in the opposite balance.

This comparison tends to prove that the results of the present study are much more scattered around the mean values, while A.S.T.M. observations stay centralized around them, as two concentric circles. It is authorized to think that a kind of 'auto-control' as described in the chapter '*Irregularities*' of the present report and which have been applied during the A.S.T.M. round robin would painlessly reduce the number of outliers and thus reduce the diameter of the 'scattering circle' to more acceptable one.

However, if we consider laboratory D as an outlying laboratory (causing 2/3 of the amount of outlying values) and, so, if we discard all its data, this percentage jumps from 6.29 to 3.93. Keeping laboratory D, but removing all the PVC runs, the outliers concentration falls to 4.32. In both cases, it is obviously still more than three times the 1.26% of A.S.T.M., but these calculations show that a lot of improvements could be easily done.

Distribution according repeatability and reproducibility

No distinction has been brought between repeatability and reproducibility outlying observations since now. This categorization may not be of first general importance insofar that outlying entries have anyhow to be removed from further analyses. On the other hand, it could be useful to examine, according to laboratories, if improvements efforts have to be done just inside the laboratory or if the variation of apparatus, operating experiences and so on, weaken the consistency of the results.

Lab. ref.		B	D	E	F	G	H	I	J	Tot.	partial total
Reprod.	outlier	1	0	0	1	1	1	0	0	4	12.50% of outliers
	straggler	0	0	0	0	1	1	1	0	3	20.00% of stragglers
repeat.	outlier	3	12	3	2	1	3	2	2	28	87.50% of outliers
	straggler	0	8	2	0	1	0	1	0	12	80.00% of stragglers
Total Reprod.		1	0	0	1	2	2	1	0	7	
Total repeat.		3	20	5	2	2	3	3	2	40	

Table XVI. Distribution of outlying values according to the inside or between laboratories correlation.

As shown in the above Table XVI, the great majority of the outlying observation are due to a lack of repeatability, meaning agreement inside one laboratory (repeatability condition). The case of Laboratory D is exemplary, all its 20 outlying data holding in repeatability and confirm our previous recommendation to check their Cone Calorimeter apparatus and their operating conditions.

On that point, we are on total agreement with the American round robin during which most of the outlying data were due to repeatability condition as well. So, all the classical advice to improve repeatability of any tests can here be made (*cf. Irregularities*). But, we have to be aware that improvements of repeatability will not necessarily decrease the reproducibility figures.

Data dispersion

The four graphs of Figure I are the representations of the ratio between the laboratory average value and the overall mean of several types of parameters regrouped by dimension (for instance, the Time data dispersion graph gathers all the timing data : ignition time, time to first peak of h.r.r, ending test time). The aim of such diagram is to point out the general tendency of any laboratories to under- or over-estimate data. Outlying values have been removed from the data used here.

Time related data

This graph has been drawn in order to determine and assess the presence of 'rapid' or 'slow' laboratories. The standard deviation observed is mainly due to the ending test time, data included in the calculation except for B, F and I laboratories which did not provide with that parameter. Moreover, the laboratory I representative point must not be directly compared to the others insofar that its average point and standard deviation line represent just the ignition time variable, the only timing parameter he provided.

According to the diagram, Laboratory F seems to be a 'slow' laboratory (standard deviation line does not cross the 100% line). On the opposite, even if its standard deviation interval crosses the average line, Laboratory G seems to be a quite 'rapid' laboratory. The operator's ability to detect instantaneously the moment of ignition may not be involved in such 'rapid/slow' categorization, but, as we previously mentioned, it is likely to be just a question of cone irradiance adjustment. Without paying attention to outlying values, a more accurate calibration of the heat flux could be necessary in order, for F and G laboratories, to reduce their deviation tendency. As we previously mentioned, moisture contents and thus samples conditioning may affect ignition time assessment as well, especially in the case of MDF and GB specimens. It is why, along with the heat flux calibration, conditioning system has to be checked as another possible cause of discrepancy.

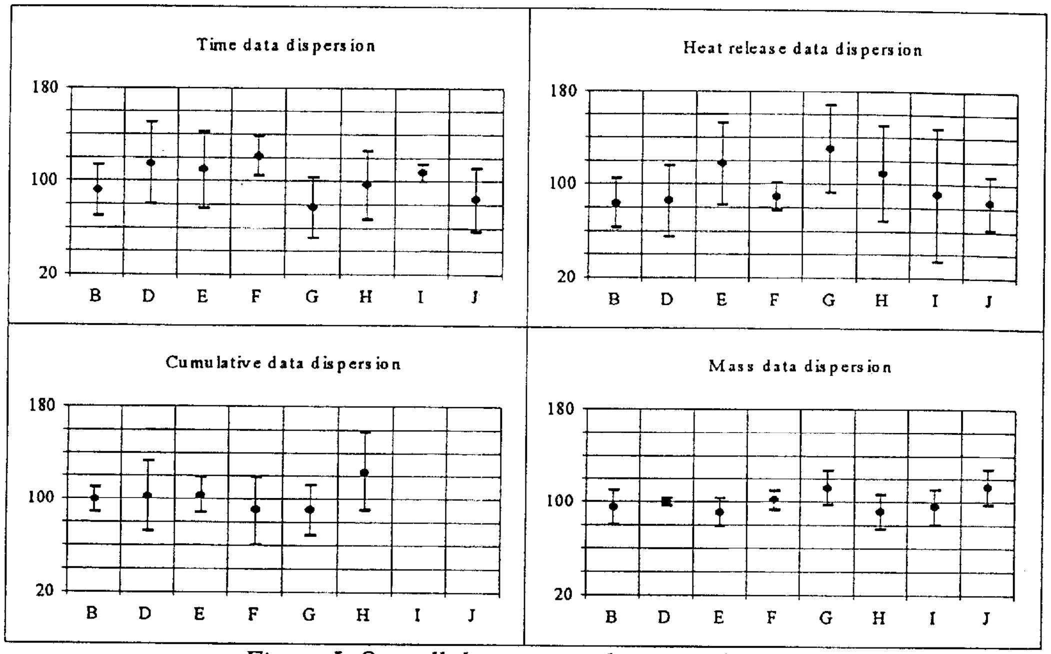


Figure I. Overall deviation to the general mean.

h.r.r data

All the standard deviation intervals are crossing the 100% line, but, however, G laboratory seems to overvalue heat release in average and F underevaluate it. If we compare these two last results with the two previous one concerning the time variables, we notice that they are going in opposite sense (underevaluation of time/overevaluation of h.r.r and vice-versa). Laboratories F and G are not the only laboratories in such configuration. Five out of the eight participating laboratories find their results in a 'symmetrical' location about the 100% line.

That remark could actually confirm our hypothesis about the cone heater calibration effect on the time variable and, in addition on heat release. In the range of studied irradiance levels, if the heat flux is set higher than prescribed, it will obviously decrease the ignition time ($1/Tig^{½}$ prop. to external heat flux) and increase the h.r.r peak value (materials answer to the thermal stress). And an overevaluation of the heater calibration will increase ignition time (and definitively other related time variables) and decrease the first maximum h.r.r values. That 'symmetrical' behavior thus makes sense and points out the major role of the thermal calibration for the consistency of Cone Calorimeter data. Some almost analogous reasoning could be drawn for conditioning system variation, insofar that moisture contents' vaporization could be considered to act as a "heat remover", but this effect could occur only for few of the chosen specimens.

In the case of B and E laboratories, for which the respective representative plots of time data and heat release data are on the same side of the 100% line, additional hypotheses have to be sought. Nevertheless, even if outlying data were not taken into account in plotting the figure I, we have to remember that these two laboratories encounter the main part of their difficulties in assessing the time variables (*cf. Table XIII*).

Another important remark which has to be done on that diagram is the huge interval line of I laboratory which indicates a wide sharing out of its h.r.r results. It has to be explained, at least, and consequently to be reduced. A so large standard deviation interval is hardly acceptable, h.r.r being one of the most important data being searched by using Cone Calorimeter.

integrated data

This graph gathers all data concerning total heat release (total heat release up to 180s, 300s and total heat release). Except for H laboratory, for which remarks about its integrated data have already been made, the concentration of the results around the 100% line is better than in the two previous diagrams. It can be guessed that the fact to integrate data fades somehow their disparity and balances their average values. No clear relation between the cumulative data dispersion graph and the time and heat release data dispersion graphs have been found.

Load cell data

The diagram just confirms our previous conclusions about the load cell data which appears as one of the most reproducible and repeatable variable among the set of parameters which have been requested.

Effect of the equipment

The laboratories participating in this round robin have used different styles of Cone Calorimeters. Some characteristics of the apparatuses which have been used are sum up in table XVII.

Laboratory ref.		B	D	E	F	G	H	I	J
Cone environment	wall	1 wall	1 wall	4 open sides	1 wall		1 wall	1 wall	1 wall
	glasses	3	3		3		3	3 s	free
O_2 Analyzer	range	0-25	0-21	0-21	0-25		0-25	0-25	
	delay time	35	30	15	54		40	18	
	Initial				*		*	**	
Heat gage		A	A	A	A		A	B	
Remarks					#		##	##	

* : Average for 60 seconds before the start of measurement.
** : Oxygen concentration assumed as 20.95%.
# : Own software.
: Software computed by the manufacturer, equations unknown.

Table XVII. Some characteristics of the laboratories' Cone Calorimeter.

Despite the different attempts made to link any of these characteristics to the outlying concentration or the deviation tendency of one or the other laboratories, no clear relations have been found out. However, we should notice that this absence of simple relation does not imply that there are no possible links between apparatuses' specifications and inaccuracy or potential systematic deviation as, as a matter of fact, the number of tests points we computed was quite restricted. The only thing which could be noticed, but just as a remark, is that F laboratory which has got the larger oxygen analyzer delay time (54 seconds) has also the bigger time data deviation (around 120% of the overall general mean). Nevertheless this fact does not represent any law neither tendency.

Comparison

Inter-laboratory Trials on I.S.O. 5657 Ignitability test.

A large number of inter-laboratory trials have been conducted on various fire tests. Unluckily, for most of them, data will be difficult to compare since different analysis methods have been employed. However, the I.S.O. Ignitability of building products, I.S.O. 5657, is an example that is directly comparable. Moreover, this test is especially interesting to compare since it uses a conical heater somewhat similar to the one used on Cone Calorimeter, and the I.S.O. 5657 round robin has been proceeded by the same I.S.O. 5725 data analysis method.

Time to ignition

Since that test is only a test for ignitability, only one variable is examined, the ignition time. As the same materials were used in both round robins, a five points comparison can be made. Unluckily, among the two levels of irradiance which were used, just one was common.

	PMMA		MDF		VCSP		GB		PVC	
	Igni.	Cone	Igni.	Cone	Igni.	Cone	Igni.	Cone	Igni.	Cone
N° labs	5	7	5	8	7	8	6	4	7	2
m	78.50	72.87	85.69	82.18	29.67	23.9	127.95	100.7	138.354	-
r	7.18	10.7	10.34	8.95	6.19	4.94	15.71	13.81	50.6483	-
R	19.12	32.41	18.20	42.14	8.07	7.51	33.16	36.84	74.7523	-

Table XVIII. Mean and related data of Ignition time in both I.S.O. 5657 and I.S.O. 5660.

The above table XVIII compares the ignition time obtained during the I.S.O. 5657 round robin of 1994-95 and the present Cone Calorimeter inter-laboratory trials, under 30 kW/m^2. For the three first products (PMMA, MDF, VCSP) a very good correlation is shown in terms of mean and repeatability. We can notice that average ignition times obtained from Cone Calorimeter seem always be a little bit lower than the one obtained by the ignitability test.

Concerning gypsum board, even if the reproducibility intervals overlap one to another, the agreement between the mean value stays poor. This could be explained by the difficulties that 4/8 laboratories encounter to ignite GB during this Cone Calorimeter round robin. The same remark has to be done in the case of PVC. The dashes do not mean no results, but just not enough results to process a statistical analysis.

We could propose, in order to reduce the uncertainty and transient flaming that some materials could give during Cone Calorimeter experiments, to let the spark ignition device during 10 seconds after the first flame outbreak in order to ensure ignition.

Repeatability & reproducibility

As the results of the I.S.O. 5657 trials were analyzed according to the same model for error as used to analyze the Cone Calorimeter data, the same kind of relation between r (or R) and m have been established in both round robins (figure II), and thus can be compared.

It can be seen that if the repeatability curves are quite close, the reproducibility for Ignitability test is substantially better (smaller) than for the Cone Calorimeter.

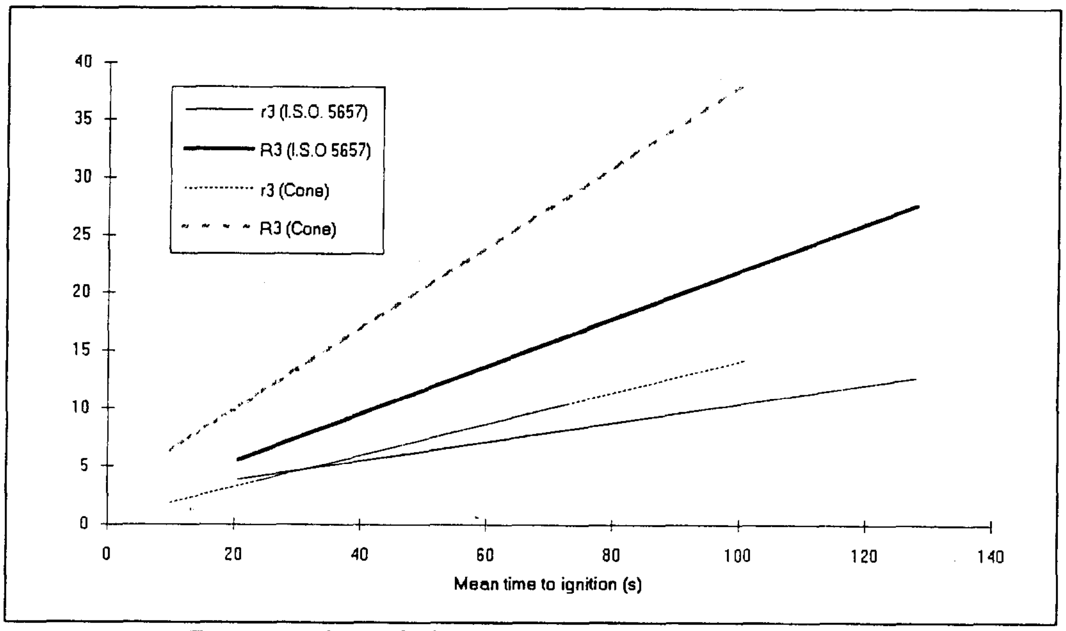


Figure II. Cone Calorimeter & Ignitability test results.

Inter-laboratory Trials on Cone Calorimeter from A.S.T.M. & I.S.O.

Along with 'other' fire tests, it seems logical to compare our results with some very similar inter-laboratory trials which have been published. As mentioned previously, two Cone Calorimeter round robins have already been conducted, both under the auspices of I.S.O, one directly by I.S.O. and the second one by A.S.T.M.. Since the protocol being tested was quite the same, the data should be comparable.

Common materials

The Table XIX provides a comparison of some parameters of particle board and PMMA in the A.S.T.M. and present round robins. Unluckily, MDF is the only material tested in both inter-laboratory trials with some similar characteristics (material, thickness and density). In the case of PMMA, the respective samples did not have the same thickness neither the same color, nevertheless some parameters which are given to be unaffected by thickness could be compared.

		Tig		h.r.r peak*		Total heat		e.h.c.	
		A.S.T.M.	Present	A.S.T.M.	Present	A.S.T.M.	Present	A.S.T.M.	Present
MDF	N° labs	6	8	6	8	6	6	6	3
	m	24.3	30	256.1	257.1	88.7	79.6	12.6	11.98
	r	6.83	7.06	58.3	18.86	18.6	10.17	1.36	1.88
	R	14.9	16.3	90.6	101.1	22.4	37.54	2.79	1.95
PMMA	N° labs	6	8	6	8	incomparable		6	3
	m	20.3	28.4	*1125*	*943*			24.8	23.1
	r	1.97	3.87	*191*	*127.1*			2.85	1.31
	R	8.22	10.39	*667*	*214*			3.95	2.314

Table XIX. Comparison of some variables in A.S.T.M. and the present round robin.

The chosen comparative parameters are the only common in both round robins. The star on the head of the h.r.r peak column means that this variable is actually not defined on the same way in both trials. A.S.T.M. prescribed to report the maximum h.r.r peak and the present study asked for the first h.r.r peak. But, as a matter of fact, for these two materials, the shape of the heat release curves makes the definition similar and thus comparable.

Even if the particle boards do not have the same origin neither the same batch, a very good agreement is observed for each common parameter which has been studied. The agreement does not only concern the mean values, but also repeatability and reproducibility.

For PMMA, the comparison is more delicate as samples did not have the same size, which, for instance, forbids the comparison of total heat release, and may be prejudicial for the h.r.r peak variable. However, ignition time and effective heat of combustion should not be affected. If e.h.c. parameters are in good agreement, the reproducibility interval of ignition time overlaps at the last end. A better correlation could have been expected even if the difference of color may disturb the comparison.

Repeatability & reproducibility

A direct comparison with the ISO round robin on materials runs, as previously made with the A.S.T.M. inter-laboratory trials, was impossible since the only available data were just given on repeatability and reproducibility fitting equations terms.

As for all the round robins which have been taken into reference in this report, the two antecedent Cone Calorimeter trials were analyzed according to the same I.S.O. 5725 model for error, and thus can be compared.

	Tig			Total heat			e.h.c.		
	A.S.T.M.	I.S.O.	Present	A.S.T.M.	I.S.O.	Present	A.S.T.M.	I.S.O.	Present
a3	4.5	2.6	0.53	11	3.9	0.7	1.02	2*	1.6*
b3	0.158	0.102	0.14	0.056	0.07	*0.07*	0.064		
A3	7.0	7.4	2.88	16.3	7.4	3.5	2.06	3.4*	2.0*
B3	0.247	0.196	0.35	0.075	0.092	*0.20*	0.07		

Table XX. Comparison of the coefficients of the fitting equations r,R =f(m) in the three Cone Calorimeter round robins.

The table above allows a comparison between the different coefficients of the r (or R) and m regressions in the 3 round robins. The h.r.r peak coefficients are not compared here since the definition may not be always comparable (according to the product and their heat release curves).

If the values of the intercept (a3 or A3) do not match very well, most of time, the slope (b3 or B3) lies in the same range of order, though always slightly higher in the present trials. The slope coefficients of the total heat release parameter (in italic) computed during this round robin could appear as an exception to the above remark, but as we mentioned in the 'comments' chapter, GB and VCSP are suspected to distort these figures (*cf. Comments. Total heat release rate*) and removing these data, the slope coefficients of the fitting equation become very close to the A.S.T.M. and I.S.O. results (b3 = 0.06 and B3 = 0.08), and thus confirm our assumptions about the potential irrelevancies generate by products which produce very low total heat release.

The stars denote the fact that for the e.h.c criterion, the best estimates fitting equations which have been chosen during I.S.O. trials as well as during the present one, are a horizontal line (constant values of repeatability and reproducibility), independent of the mean. We gave here as figures for the present round robin, the values of repeatability and reproducibility computed without the VCSP specimen (*cf. Comments. Effective Heat of Combustion*). By mean of that calculation, results of the present trials are altogether comparable, and even better than the I.S.O.'s one.

Conclusions

Among the purposes of these first Asia-Oceania I.S.O. 5660 Cone Calorimeter inter-laboratory trials data analyses, one of the major intention was to underline the delicate points and potential pitfalls of the test procedure that participating laboratories met or could meet. The data processing was instructive from several points of view and enables us to provide some recommendations in order to improve accuracy, reliability and consistency of Cone Calorimeter test operating protocol and consequently of computed data. Without entering any audit of particular laboratory case, three main general conclusions can be derived from this report.

First of all, the relative concentration of outliers needs definitely to be reduced. Even when laboratories which could be suspected to take a large part in that value are removed, the concentration of outlying entries stays very high, especially in repeatability conditions. A general attention must be paid on that problem. A kind of 'auto-control', or more precisely the establishment of a criterion according to which additional testing is required should be brought.

We can propose, as a simple auto-regulation, that if any of the mean heat release values for the first 60, 180, 300 seconds after ignition, among the three replicates of one material at one set of conditions, differs by more than 10% from the average for the three replicates, an additional series of three tests has to be executed. That procedure must remove rapidly and easily, most of the numerous outliers we found out.

Secondly, it is guessed that some misreading or misunderstanding of parameters' definition could be the cause of defective results. Main troubles were found for time parameters, and mostly for ignition time and test ending time. The definition of 'ignition time' is from great importance since most of the calculations, inside Cone Calorimeter applications and outside, take it in reference. As it is now common to say, the term of 'ignition' should be understood, for cone purpose, as 'sustained flaming'. Concerning test end time, the definition related to the mass loss rate as given in the chapter 'Nomenclature' must be the more appropriate to ensure data consistency.

And thirdly, the tremendous impact of a right and accurate calibration of the irradiance level has been pointed out throughout the report, especially in reproducibility conditions and for data deviation tendency. All the governing factors leading to calibrate or to set the external heat flux, from the temperature controller to the heat flux gage, should be carefully checked and harmonized. Moreover, as it has been prescribed during the ASTM round robin, the need for a reference heat flux meter, distinct from the one used for daily calibrations seems obvious.

It is thought that a specific attention must be paid on the calibration factor assessment (C) and on oxygen analyzer response time, by means of methane purity specification, flow meters accuracy, drying agent harmonization, and so on.

Finally, the aim to reach, now, is to encourage the laboratories to increase their general reliability and to improve their methodology in order to prepare a second round robin. As we previously mentioned, it can be guessed that with few efforts great improvement can be expected.

References

- Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory test, I.S.O. 5725. International Organization for Standardization, Geneva, 1986.

- A.S.T.M. Task Group E5.21, T.G. 60, "Report to A.S.T.M. on Cone Calorimeter Inter-laboratory Trials.", January, 1990.

- Fire tests - Reaction to Fire - Ignitability of building products, International Standard I.S.O. 5657, International Organization for Standardization, Geneva, 1986.

- Proposed Test Method for Heat and Visible Smoke Release Rates for Materials and Products using an Oxygen Consumption Calorimeter, E-5 Proposal P 190, 1986 Annual Book of A.S.T.M. Standards, Vol. 04.07, pp. 1221-1237, 1986.

- Babrauskas V., Grayson S.J., "Heat Release in Fires", Elsevier Science Publishers Ltd, Barking, England, 1992.

- Asia-Oceania I.S.O. 5657 - Ignitability of building products Test - Inter-laboratory Trials Report to Building Research Institute, Building Research Institute, Tsukuba, Japan, 1995.

- Fire tests - Reaction to Fire - Rate of Heat Release from building products, Draft International Standard I.S.O. 5660, International Organization for Standardization, Geneva 1991.

Annex

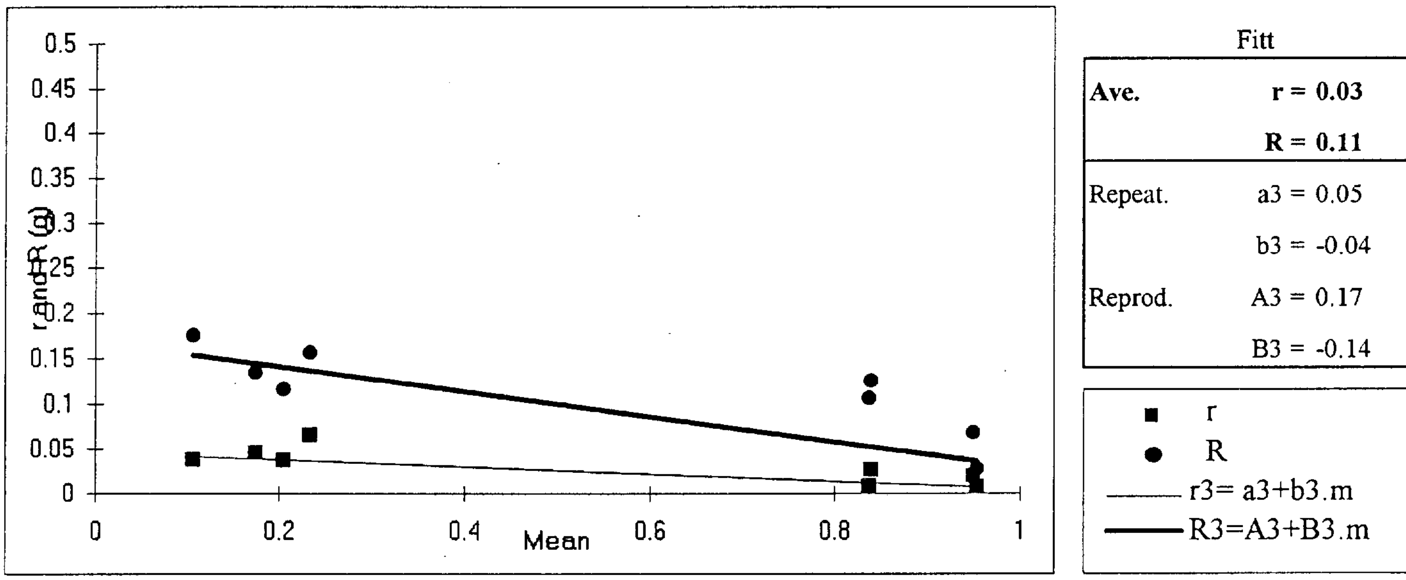
Statistical Analysis Results

Tables III to XII
Repeatability & Reproducibility Raw Results.

Table III

Mass loss (Mass initial-Mass final)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	0	0	8	8	8	8	8	7	6	6
Notes.	a	a	-	b	-	c	-*	d	e*	f*
mean (m)	-	-	0.205	0.174	0.953	0.949	0.840	0.837	0.233	0.106
repeatability (r)	-	-	0.038	0.046	0.008	0.020	0.027	0.008	0.066	0.038
Reproducibility (R)	-	-	0.116	0.134	0.028	0.069	0.125	0.106	0.156	0.175



Fitt

Ave.	**r = 0.03**
	R = 0.11
Repeat.	a3 = 0.05
	b3 = -0.04
Reprod.	A3 = 0.17
	B3 = -0.14

Lab.	B	D	E	F	G	H	I	J
V.C.	1.4391	0.5656	1.0294	1.4692	1.2832	1.4456	3.59	2.60

Notes.

* These figures must be handled with precaution insofar that for 4 of the 9 laboratories no ignition was reported, but a mass loss was recorded.

a : PMMA being totally burned out, there is no mass left at the end of the manipulation. Results are so uninterpretable and were not taken into account.

b : Laboratory I was identified as straggler (repeatability)

c : Laboratory H was identified as straggler (reproducibility)

d : Laboratory I was removed as outlier (repeatability)

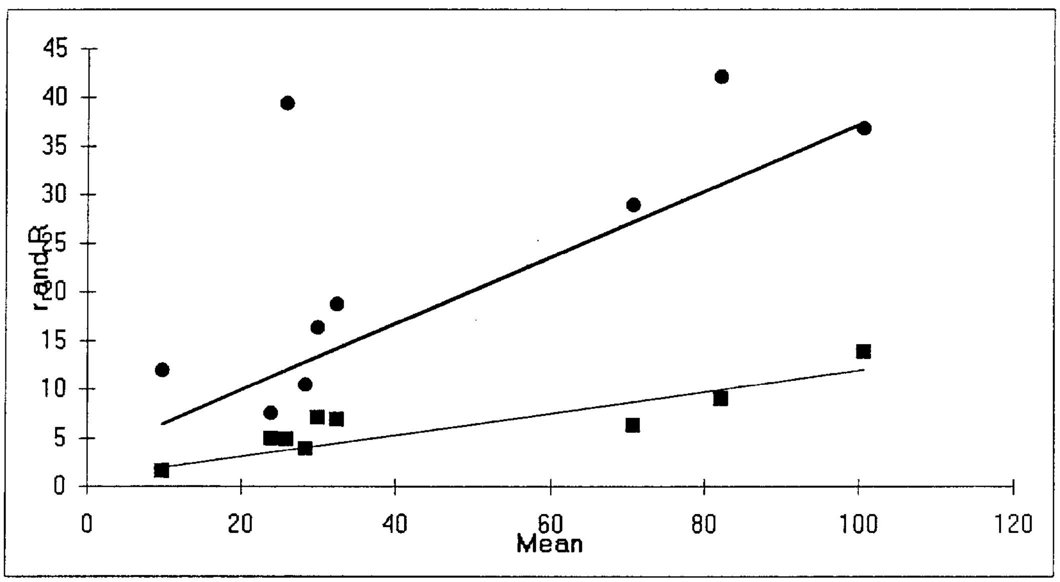
e : Missing data from Laboratories D & F (No ignition)

f : Missing data from laboratory D (1 ignition/ 3 trials) and Laboratory F was removed as outlier (repeatability)

Table IV

Time to Ignition (s)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	7	8	8	8	8	7	4	8	2	5
Notes.	a	-	-	b	-	c	-*	-	d**	e***
mean (m)	70.752	28.367	82.183	29.996	23.904	9.7833	100.74	32.496	-	25.906
repeatability (r)	6.2918	3.875	8.9518	7.0565	4.9361	1.5976	13.815	6.8678	-	4.8077
Reproducibility (R)	28.911	10.392	42.138	16.302	7.5079	11.883	36.841	18.686	-	39.36



Fitt

Ave.	r = 6.47
	R = 23.56
Repeat.	a3 = 0.88
	b3 = 0.11
Reprod.	A3 = 3.11
	B3 = 0.34

■ r
● R
—— r3= a3+b3.m
—— R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	5.2384	5.5399	4.5182	3.8366	4.0303	3.9253	7.2604	4.8478

Notes.

* : Missing data from laboratory B, F, I, J (No ignition)

** : Missing data from laboratory B,D, F, I, J (No ignition) : No data enough, results disgarded.

*** : Missing data from laboratory I, J (No ignition)

a : laboratory I was removed as outlier (repeatability)

b : laboratory G was identified as straggler (reproducibility)

c : laboratory B was removed as outlier (repeatability)

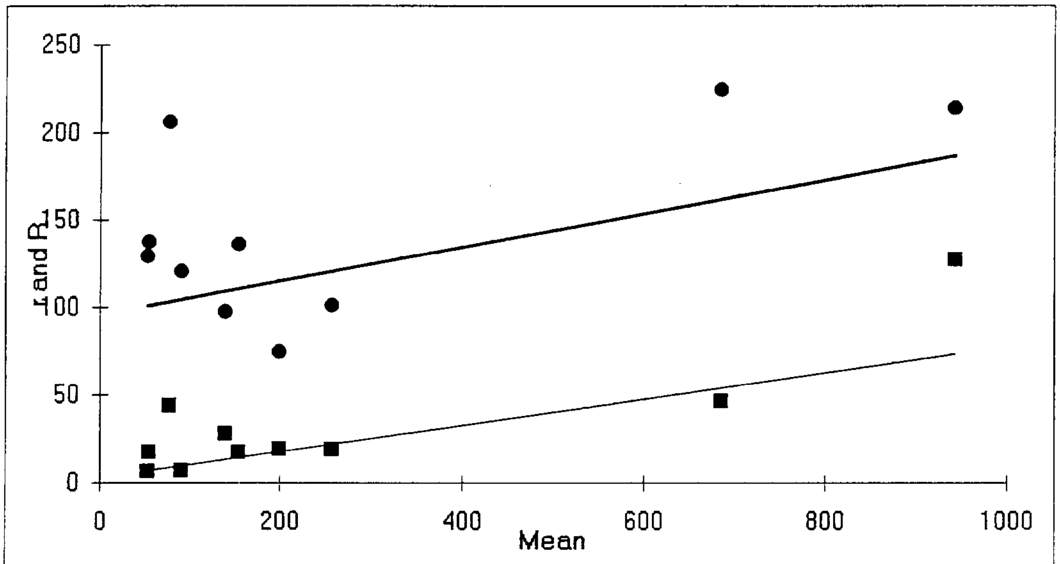
d : laboratory E was removed as outlier (repeatability)

e : laboratory B was removed as outlier (repeatability & reproducibility)

Table V

First peak of R.H.R. (kW/m²)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	8	8	8	8	8	8	5	7	5	6
Notes.	a	b	c	c	-	-	d	e*	-*	f
mean (m)	685.37	942.7	199.26	257.14	139.3	153.95	53.193	90.208	76.68	54.658
repeatability (r)	46.485	127.08	19.37	18.856	28.06	17.458	6.6041	6.8108	44.054	17.511
Reproducibility (R)	224.44	214.21	74.475	101.1	97.344	135.84	128.93	120.32	206.06	137.34



Fitt

Average	r =	33.2284
	R =	144.006
Repeat.	a3 =	3.07179
	b3 =	0.07452
Reprod.	A3 =	95.492
	B3 =	0.09696

■ r
● R
—— r3= a3+b3.m
—— R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	2.6416	3.7546	5.043	3.8814	3.475	3.6821	4.6239	1.7617

Notes.

* : Missing data due to non-ignition

a : laboratory G was identified as straggler (repeatability)

b : laboratory I was identified as straggler (repeatability)

c : With corrected data from Laboratory H (first peak/maximun peak)

d : laboratory D was removed as outlier (repeatability)

laboratory F was removed as outlier (repeatability), data without ignition.

e : laboratory D was removed as outlier (repeatability)

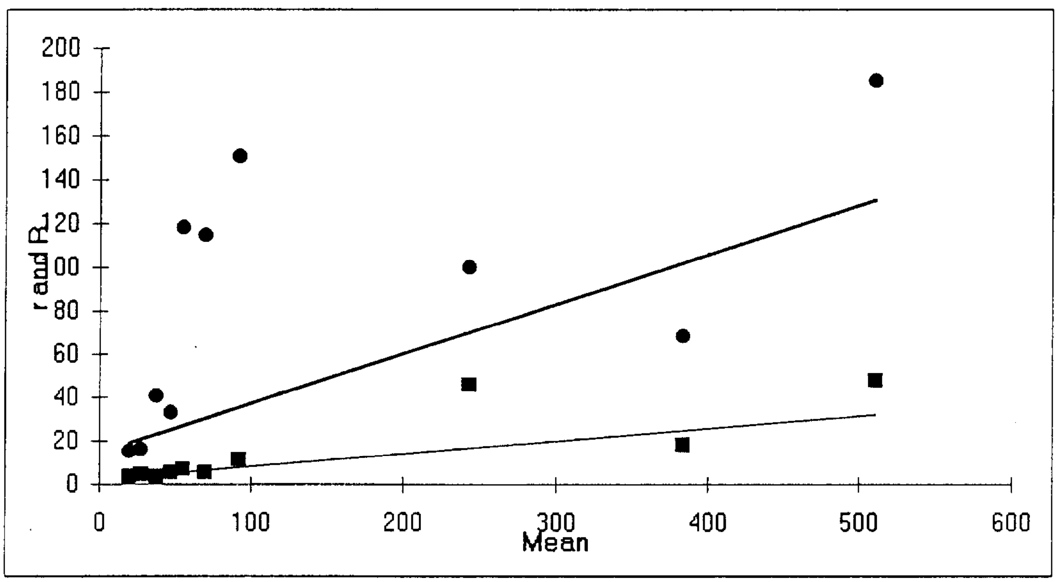
f : laboratory G was removed as outlier (reproducibility), 4 times the averall average!

laboratory J was removed as outlier (repeatability), data without ignition

Table VI

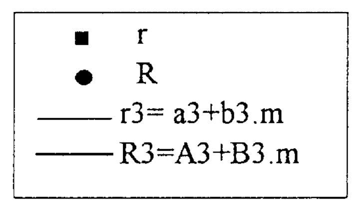
Time to first peak of R.H.R. (s)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	6	5	5	6	6	6	4	6	4	5
Notes.	-*	a*	b*'	-*'	-*	-*	c*	-*	d*	e*
mean (m)	510.94	383.8	69.778	47.111	27.222	19.667	92	37.389	243.1	55.059
repeatability (r)	47.935	18.432	5.465	5.6358	4.7024	3.6425	11.582	3.8569	46.065	6.9459
Reproducibility (R)	185.14	68.349	114.73	33.206	16.176	15.386	150.64	40.76	100.1	118.41



Fitt

Average	r =	15.4263
	R =	84.2908
Repeat.	a3 =	2.50116
	b3 =	0.05848
Reprod.	A3 =	14.5898
	B3 =	0.22764



Lab.	B	D	E	F	G	H	I	J
V.C.	4.5989	5.7729	1.4341	2.8257	1.0866	3.7761	-	-

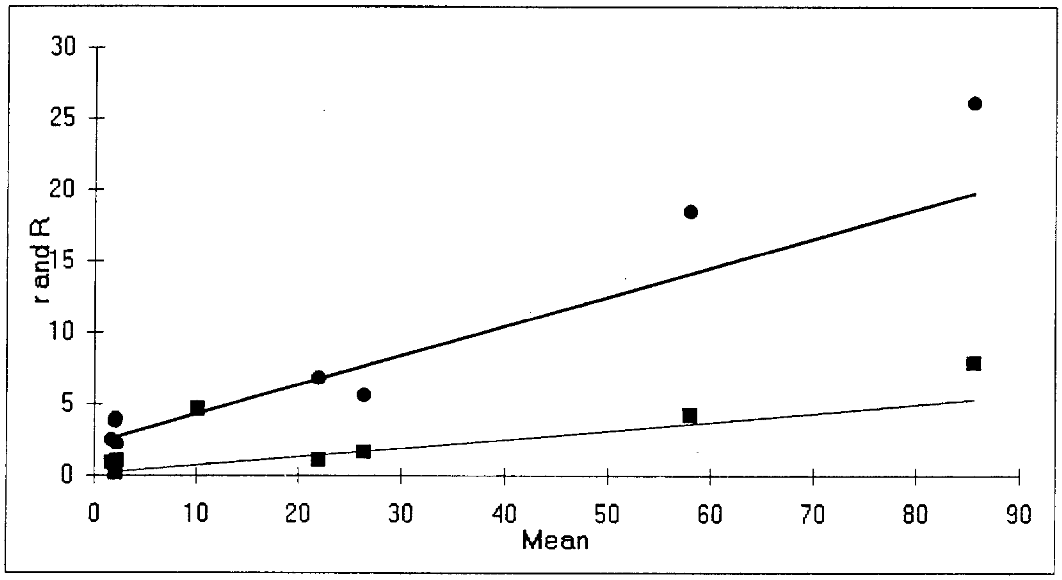
Notes.

- * : Missing data from I, J laboratories, no obvious reasons
- ' : with corrected data from laboratory H (first peak/maximun peak)
- a : Laboratory D was removed as outlier (repeatability)
- b : Laboratory D was removed as outlier (repeatability)
- c : Laboratory F was removed as outlier (reproducibility)
- d : Laboratories D & E was identified as straggler (repeatability)
- e : Laboratory B was removed as outlier (repeatability)

Table VII

Total Heat Release from Tig to 180s (MJ/m²)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	4	4	3	4	4	3	3	3	1	3
Notes.	-*	-*	a*	-*	-*	b*	c*	d*	e*	f*
mean (m)	58.025	85.558	21.978	26.383	2.2167	2.025	1.7	2.0917	-	10.125
repeatability (r)	4.2327	7.8884	1.0896	1.6291	1.0337	0.1771	0.9063	0.2892	-	4.6452
Reproducibility (R)	18.465	26.135	6.7917	5.5954	2.2259	3.7901	2.4797	3.9396	-	4.6575



Fitt		
Average	r =	2.43235
	R =	8.2311
Repeat.	a3 =	0.11818
	b3 =	0.06043
Reprod.	A3 =	2.28033
	B3 =	0.2042

■ r
● R
—— r3= a3+b3.m
—— R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	3.9811	2.7588	4.656	1.4213	-	-	-	-

Notes.

* : Missing data from G, H, I, J laboratories, no obvious reasons.

a : Laboratory D was removed as outlier (repeatability)

b : Laboratory D was removed as outlier (repeatability)

c : Missing data from Laboratory B, no ignition.

d : Laboratory D was removed as outlier (repeatability)

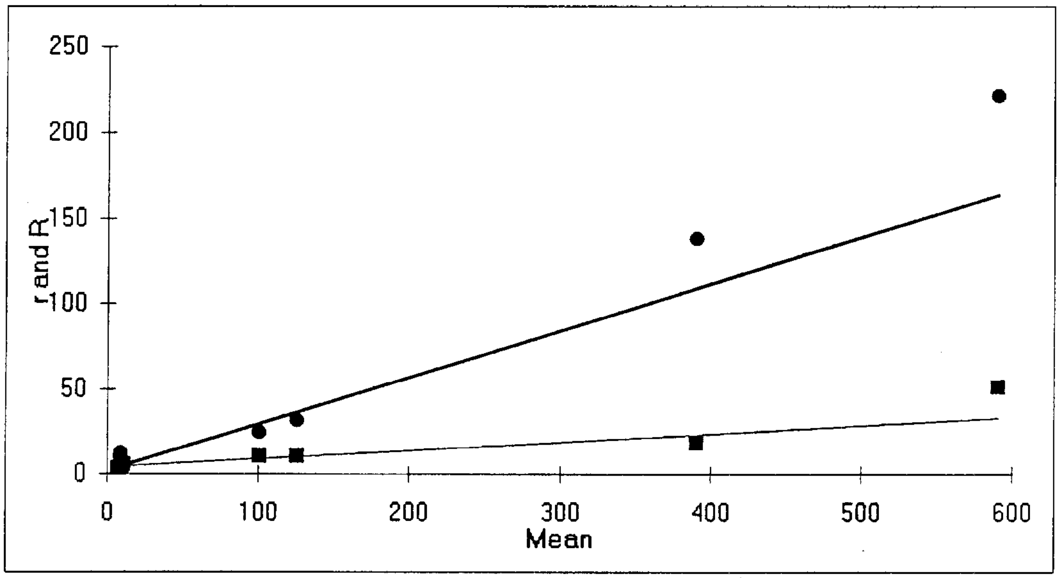
e : Missing data from Laboratories B, C, F, no ignition : data discarded.

f : Missing data from D laboratory

Table VIII

Average Heat release from Tig to 300s (kW/m²)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	4	4	4	4	3	3	3	4	2	2
Notes.	-*	a*	b*	-*	-*	c*	-*	-*	d*	e*
mean (m)	390.38	590.53	101.12	125.84	8.95	10.444	7.1889	9.15	-	-
repeatability (r)	18.158	51.039	10.333	10.536	6.8896	5.4712	3.4532	4.52	-	-
Reproducibility (R)	138.14	221.49	24.545	31.451	12.104	6.8765	3.5281	3.9435	-	-



Fitt

Average	r =	13.7999
	R =	55.2589
Repeat.	a3 =	4.10373
	b3 =	0.04865
Reprod.	A3 =	2.07228
	B3 =	0.27371

■ r

● R

—— r3= a3+b3.m

—— R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	-	3.4548	4.7063	-	1.9628	1.582	-	-

Notes.

* : Missing data from B, F, I, J laboratories, no obvious reasons

a : Laboratoy E was identified as straggler (repeatability)

b : Laboratoy D was identified as straggler (repeatability)

c : Laboratoy D was identified as straggler (repeatability)

d : Missing data from D, G laboratories, not enough data to analyse : level discarded.

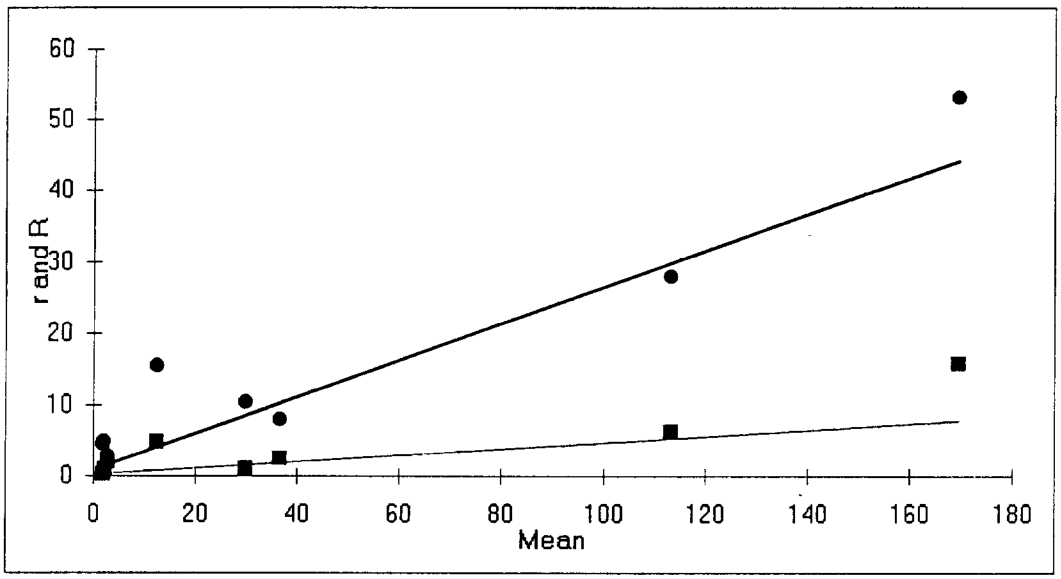
e : Laboratoy D was identified as straggler (repeatability)

Missing data from D, G laboratories, not enough data to analyse: level discarded.

Table IX

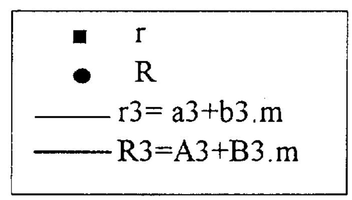
Total Heat Release from Tig to 300s (MJ/m²)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	4	4	3	4	3	2	2	2	1	3
Notes.	-*	-*	a*	-*	b*	c*	d*	e*	f*	g*
mean (m)	113.24	169.43	29.878	36.633	2.8111	-	-	-	-	12.367
repeatability (r)	6.3384	15.868	1.0441	2.467	1.9206	-	-	-	-	4.832
Reproducibility (R)	28	53.213	10.424	8.007	2.8365	-	-	-	-	15.469



Fitt

Average	r =	5.41161
	R =	19.6582
Repeat.	a3 =	0.23584
	b3 =	0.04511
Reprod.	A3 =	0.92367
	B3 =	0.25532



Lab.	B	D	E	F	G	H	I	J
V.C.	3.2263	2.697	4.7079	1.0713	-	-	-	-

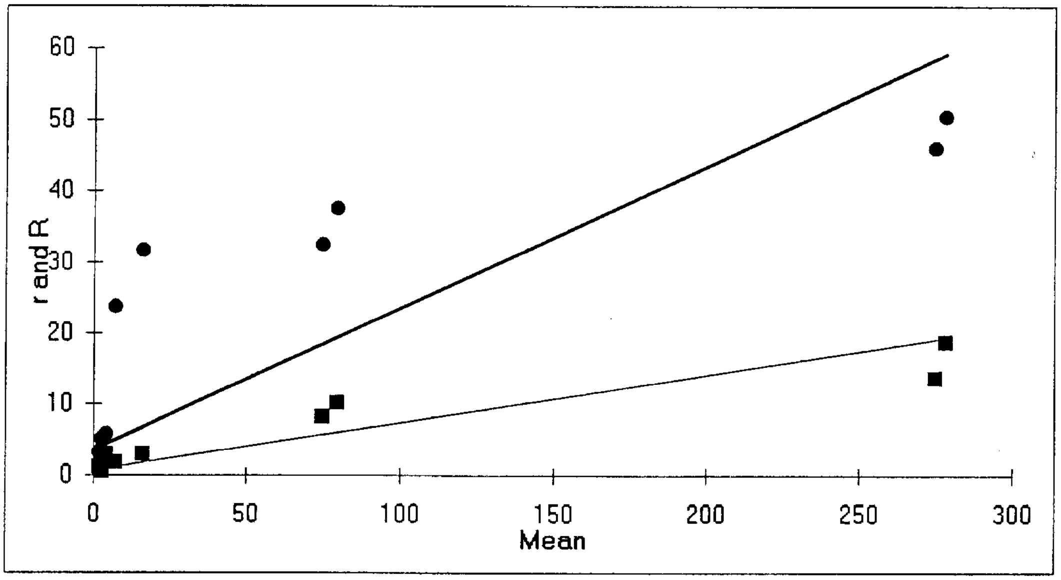
Notes.

- * : Missing data from G, H, I, J laboratories, no obvious reasons
- a : Laboratory D was removed as outlier (repeatability)
- b : Missing data from F laboratory
- c : Missing data from F laboratory &
  Laboratory D was removed as outlier (repeatability) : no enough data : leveldiscarded.
- d : Missing data from F, B laboratories
- e : Laboratory D was identified as straggler (repeatability)
- f : No data except from Laboratory E : level discarded.
- g : Missing data from D laboratory

Table X

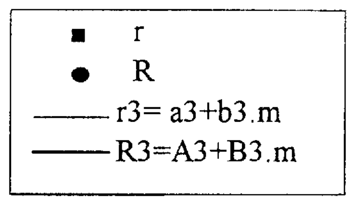
Total Heat release (kW/m²)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	6	6	6	6	6	5	4	6	3	5
Notes.	-*	-*	-*	-*	a*	b*	c*	-*	d*	e*
mean (m)	278.28	274.94	74.756	79.606	2.8	2.6944	1.92	4.1667	7.1231	16.1
repeatability (r)	18.753	13.64	8.2093	10.169	1.8421	0.5533	1.1297	2.9428	1.7816	2.9271
Reproducibility (R)	50.461	46.096	32.418	37.539	3.1828	5.0675	3.1653	5.82	23.728	31.647



Fitt

Average	r =	6.19468
	R =	23.9125
Repeat.	a3 =	0.70908
	b3 =	0.06703
Reprod.	A3 =	3.50246
	B3 =	0.20013



Lab.	B	D	E	F	G	H	I	J
V.C.	1.9129	3.8855	2.1664	1.2135	4.2559	1.9402	-	-

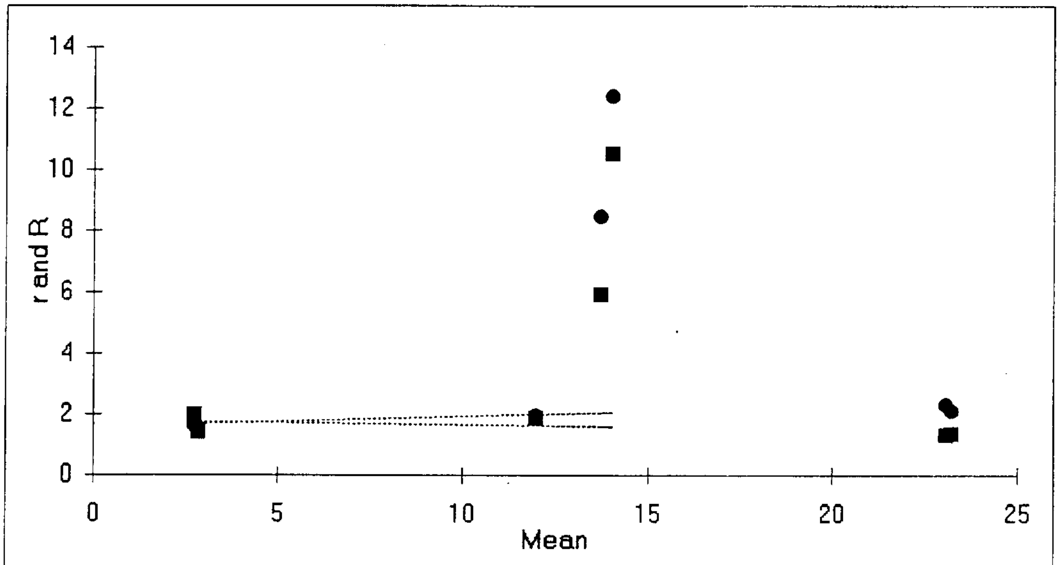
Notes.

- * : Missing data from I, J laboratories, no obvious reasons.
- a : Laboratory D was identified as straggler (repeatability)
- b : Laboratory D was removed as outlier (repeatability)
- c : Laboratory H was removed as outlier (repeatability & reproducibility)
- d : Laboratory H was removed as outlier (repeatability)
- e : Laboratory D was removed as outlier (repeatability)

Table XI

Effective Heat of Combustion (MJ/kg)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	3	3	2	3	3	3	3	3	2	2
Notes.	-*	-*	a*	b*	c*	-*	-*	-*	d*	d*
mean (m)	23.233	23.089	-	11.978	14.033	13.722	2.8556	2.7444	-	-
repeatability (r)	1.3442	1.3048	-	1.8793	10.53	5.905	1.3879	1.9818	-	-
Reproducibility (R)	2.098	2.3139	-	1.948	12.403	8.4614	1.5611	1.6244	-	-



Fitt

Average	**r =**	**3.47609**
	R =	**4.34429**
Repeat.	a3 =	1.79994
	b3 =	-0.01512
Reprod.	A3 =	1.57582
	B3 =	0.035

■ r

● R

.... r3= a3+b3.m

.... R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	-	14.116	4.863	-	4.2788	-	-	-

Notes.

* : Missing data from B, F, H, I ,J laboratories, no obvious reasons.

a : Laboratory D was removed as outlier (repeatability), no data enoughto analyze : level discarded

b : Laboratory D was identified as straggler (repeatability)

c : Laboratory D was identified as straggler (repeatability)

d : Missing data from D laboratory, no data enough to analyze: level discarded

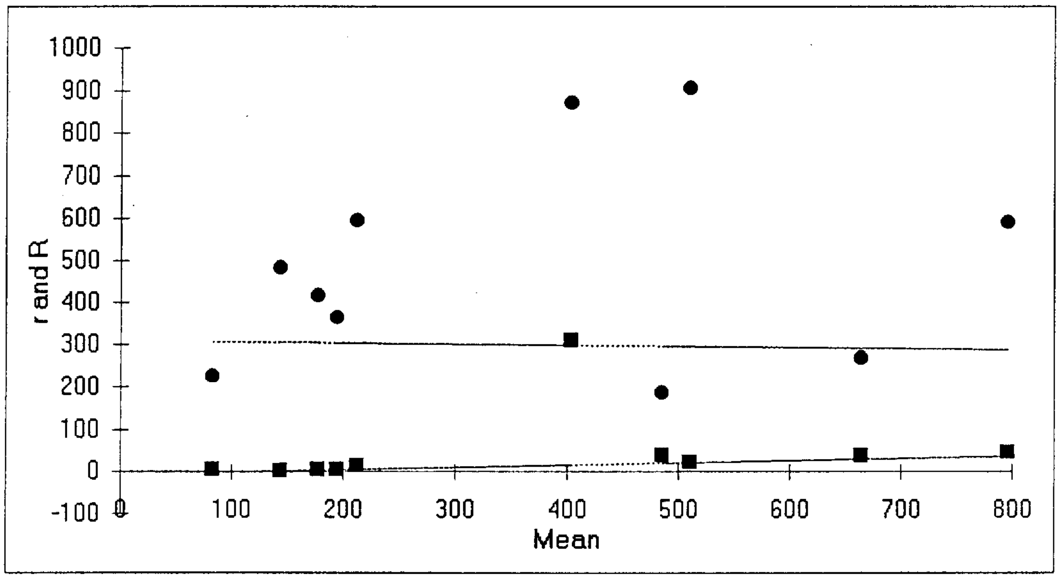
Rq. The maximun number of laboratories taking part in that analysis is only three.

Results must so be handle with precaution.

Table XII

Test End Time (s)

Products	PMMA		MDF		VCST		GB		PVC	
Flux (kW/m²)	30	50	30	50	30	50	30	50	30	50
Number of labs.	5	5	5	4	4	5	3	4	4	3
Notes.	-*	-*	-*	a*	b*	-*	c*	d*	-*	e*
mean (m)	664.73	484.8	795.8	510.07	143.67	82.8	177.33	194.67	403.42	212.42
repeatability (r)	37.661	38.626	47.302	23.033	1.3199	4.4673	4.3976	5.4422	310	13.793
Reproducibility (R)	269.15	186.29	591.23	907.24	482.71	225.35	417.05	365.33	872.29	595.4



Fitt

Average	r =	48.6037
	R =	491.204
Repeat.	a3 =	-5.59274
	b3 =	0.05308
Reprod.	A3 =	309.011
	B3 =	-0.02705

■ r

● R

——— r3= a3+b3.m

——— R3=A3+B3.m

Lab.	B	D	E	F	G	H	I	J
V.C.	-	2.9149	5.5953	-	3.0286	2.4034	-	2.8483

Notes.

* : Missing data fromB, F, I, no obvious reasons.

a : Laboratory E was removed as outlier (repeatability)

b : Laboratory G was removed as outlier (repeatability)

c : Laboratory H was removed as outlier (repeatability)

d : Laboratory J was removed as outlier (repeatability)

e : Laboratory E was removed as outlier (repeatability)

Rq. Results seem to be unstable, no fitt possible.

Puzzling results obtained by D & E laboratories (no dispersion of the results).