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Cigarete - Merjenje proste suhe snovi nikotina, nikotina, vode in ogljikovega monoksida v cigaretnem dimu - Analiza podatkov primerjalnih ġtudij s poroḷ anjem razmerij med ponovljivostjo, obnovljivostjo in tolerancami

Cigarettes - Measurement of nicotine-free dry particulate matter, nicotine, water and carbon monoxide in cigarette smoke - Analysis of data from collaborative studies reporting relationships between repeatability, reproducibility and tolerances

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Cigarettes — Measurement of nicotine-free dry particulate matter, nicotine, water and carbon monoxide in cigarette smoke — Analysis of data from collaborative studies reporting relationships between repeatability, reproducibility and tolerances

Cigarettes — Détermination de la matière particulaire anhydre et exempte de nicotine, de la nicotine, de l'eau et du monoxyde de carbone dans la fumée de cigarette — Analyse des données provenant d'études collectives et traitant des relations entre la répétabilité, la reproductibilité et les tolérances

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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ISO/TR 22305 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Introduction

0.1 Summary

The purpose of this Technical Report is to review the smoke yield data provided to Working Group ISO/TC 126/WG 8 “Confidence intervals for the determination of carbon monoxide” and to use it as the basis for proposing a tolerance for checks of declared carbon monoxide yields.

There are many laboratories around the world routinely measuring the nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide yield of cigarette brands. They can, in general, be divided into two types: those run by cigarette manufacturers for quality monitoring and those run or contracted by regulators to check the yield information provided by manufacturers.

These laboratories need to assess their performance against others to ensure the reliability of their measurements. Their wide geographical spread limits such assessments on a national basis, so that international collaborative studies provide the most practical means and generate data sets on a regular basis. In addition to allowing individual laboratories to rank their measurements relative to others, the studies also establish confidence intervals (CIs) for the repeatability (r_{20})[1] of the measurements in a single laboratory and reproducibility (R_{20})[2] in different laboratories. The reported r and R values from each study have been used in isolation but when combined, as in this report, provide a means of assessing if newly reported values are outside the expected range. The values from the latest 2003 CORESTA study are compared in this way and found to be within the previously reported range of values but at the lower end. There is no hard evidence, therefore, that the harmonization work on smoking machines has reduced the variability in CO yield measurements, but the data have been shown to be as good as the best previously reported. For this reason, and because it was a large study including all current designs of smoking machine, it provides the most appropriate data for estimating compliance tolerances.

The measurement CIs represented by r_{20} and R_{20} provide a starting point for estimating the tolerances[3] relevant to compliance checks on the yield information provided by manufacturers. They need to be combined with additional information on testing[4] and reporting as well as the inherent variability in the product associated with routine cigarette manufacturing. The statistical model given in this report is designed to incorporate all the relevant information to estimate compliance tolerances. The model is based upon the within and between laboratory standard deviations for tests of 100 cigarettes, together with additional terms to account for rounding the declared values and to include the product variability. A weakness in the model approach stems from the lack of data for estimating the terms relating to product variability, the only source of data being the UK Department of Health Survey, which is not specifically designed to provide such data. For this reason the model has been used in this report without including the product terms and the calculated tolerance values [$R_{100+rndg}$][5] compared with those from an alternative indirect prediction. Obviously, the $R_{100+rndg}$ values are lower than the true compliance tolerance since they do not include the product terms.

The simplest indirect way of predicting a CO tolerance is from the measurement variability relative to NFDPM, for which an accepted tolerance exists. The ratio of the $R_{100+rndg}$ values calculated from the CORESTA 2003 Study data was used for this purpose.

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1) Based on tests of 20 cigarettes.

2) Based on tests of 20 cigarettes.

3) ISO 8243 has always included tolerances for NFDPM and nicotine but an interim CO tolerance was added in 2003 whilst ISO/TC 126/WG 8 considered a permanent value.

4) See ISO 4387 and ISO 8243.

5) Based on tests of 100 cigarettes with allowance for rounding the declared value.

ISO 8243 provides procedures, and tolerances [6)], for sampling both 'over a period of time', which is recommended, and 'at one point in time'. Tolerances derived from both the statistical model and ratio methods for 'over a period of time' sampling are summarized below.

Parameter evaluated	Carbon monoxide tolerance
$R_{100+\text{rndg}}$	20 % with a minimum of 1,5 mg or 25 % with a minimum of 1 mg
$R_{100+\text{rndg}}$ ratio (CO/NFDPM)	22 % with a minimum of 1,5 mg

It is recommended that the compliance tolerance for CO be set at 20 % for 'over a period of time' sampling, and 25 % for 'at one point in time' sampling, with a minimum value of 1,5 mg. This recommendation implies a corresponding amendment of ISO 8243.

It is further recommended that the tolerances and minimum values are reviewed when compliance rates are established from regulatory checks. It is possible that such data may only become available in the UK and may take two or three years to assemble.

0.2 General Information

Methods of measurement specified in ISO Standards require estimates of repeatability (r) and reproducibility (R). These are normally derived from a collaborative study conforming to the guidelines in ISO 5725-1 [1] and ISO 5725-2 [2] involving as many laboratories as possible.

There is a particular problem in obtaining estimates when the measurement results in the destruction of the product sample, for example, cigarettes or fuel for internal combustion engines. If laboratories are measuring the physical dimensions of, say, metal nuts and their bolts, measurements can be made on the same items by one operator within a laboratory (repeatability) and by different operators in many laboratories (reproducibility). In this example it is always the same set of nuts and bolts which is measured throughout the experiment.

For cigarette smoke constituent determinations, the situation is entirely different. The cigarettes, once sampled and smoked, produce a set of smoke constituent estimates, each of which is perfectly valid (provided that the standard methods have been followed) but which cannot be repeated or confirmed. The only possible check between data is to compare them with an accepted range of yield measurements.

A series of ISO Standards exists to condition the cigarettes [3], to specify the smoking machine [4] for routine analytical smoking [5] and to measure smoke nicotine [6], smoke water [7] [8] and smoke carbon monoxide (CO) [9].

Variation in the final yield of smoke constituent arises from all these procedures but also from manufacture of the product (see Annex A) and from the methods of sampling. These factors require the use of special procedures in collaborative tests on cigarette products. Product variability is minimized by the testing of matched samples, usually taken from a single small batch production, in each participating laboratory. The samples, therefore, do not include the normal product variability and are not representative of any individual brand.

The r and R values from collaborative studies are thus essentially estimates of measurement variability on near identical samples. They cannot be used directly as a tolerance for compliance checks of cigarette brands where other sources of variability must be taken into account.

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6) The 'over a period of time' tolerances are 15 % for NFDPM and nicotine, and 20 % for CO. The tolerances when sampling at 'one point in time' are increased to 20 % for NFDPM and nicotine and 25 % for CO. In both cases, a minimum value of 1 mg applies to NFDPM and CO and 0,1 mg nicotine.

0.3 Sampling a population of cigarettes manufactured for sale

ISO 8243 [10] specifies methods for sampling a population of cigarettes manufactured for sale. It also includes the expected tolerances when cigarettes brands are so sampled and when smoke components are measured using the standards detailed above.

Increasing international interest and in particular the EU Directive 2001/37/EC requiring the declaration of CO yield on cigarette packs showed that revision of this standard was urgent. ISO/TC 126 therefore decided in 2003 to set up a working group WG 8 with the task of first making a revision to add a tolerance for CO to the 1991 edition of the standard, and then to continue to revise and if possible, simplify the text of the standard. The first task was accomplished and ISO 8243 was published in 2003 as a minor revision. The tolerance for CO was included on the basis of existing studies showing the need for a higher tolerance than for NFDPM. However, further collaborative studies were conducted concurrently and the purpose of this Technical Report is to record the data from these studies and to compare them with other sources of data not previously reported in the ISO domain.

Any further revision will then have the most comprehensive data upon which to specify the tolerances for nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide.

0.4 Development of smoking machines

Pressures on laboratory efficiency and the need for greater flexibility in changing smoking parameters and types of smoke traps, have led to the development of smoking machines of differing designs, although meeting the requirements of ISO 3308. Evidence based on reproducibility values in ISO standards and other sources (see Annexes B, C, D) has shown that CO measurements are more variable than NFDPM (a smoke constituent of a similar level of yield). The various members of CORESTA[7] have assisted the manufacturers of smoking machines to better harmonize the operating conditions of the machines by evaluating the effect of modifications through collaborative studies. Such development has been found necessary to improve the agreement between smoke determinations on matched samples of cigarettes from different designs (all within the ISO 3308 specification) of smoking machines, a procedure which has been called 'harmonization'. As a final check on the harmonization a CORESTA Collaborative Study was set up in 2003, the details of which are given in Annex F.

7) CORESTA: Cooperation Centre for Scientific Research Relative to Tobacco (Centre de Coopération pour les Recherches Scientifiques Relatives au Tabac)

Cigarettes — Measurement of nicotine-free dry particulate matter, nicotine, water and carbon monoxide in cigarette smoke — Analysis of data from collaborative studies reporting relationships between repeatability, reproducibility and tolerances

1 Scope

This Technical Report records the data and conclusions from a review of international collaborative studies to establish the tolerance for checks of the carbon monoxide yields declared by cigarette manufacturers for their products, as specified in ISO 8243.

2 Statistical functions for repeatability (r), reproducibility (R) of yield measurements and compliance tolerances for declared smoke constituent yields

2.1 Statistical functions for repeatability (r) and reproducibility (R)

ISO 5725-1 and ISO 5725-2, present the general principles for collaborative tests and give methods for the determination of r and R.

In the present context, a collaborative test essentially entails the recruitment of as many laboratories as possible (8 - 15 is common to provide a reasonable level of confidence in r and R, according to ISO 5725-1:1994; 6.3.4), using ISO standard methods and procedures under repeatability conditions, to measure matched cigarette samples covering the normal range (normally 5 different samples, according to ISO 5725-1:1994; 6.4.1) obtained from a short production run in order to minimize the product variability ('If different items are to be used in different laboratories, then they shall be selected in such a way as they can be presumed to be identical for practical purposes.', ISO 5725-1:1994, 6.4.2).

As noted earlier, ISO requires that estimates of r and R shall be included in each standard which details a measurement procedure. In the present standards for the determination of NFDPM (ISO 4387), nicotine (ISO 10315) and carbon monoxide (ISO 8454), the r and R values are calculated as

$$r = 2{,}8 * s_w$$

and $$R = 2{,}8 * [s_b^2 + s_w^2]^{1/2}$$

where

s_w is the repeatability standard deviation between mean values of 20 cigarettes, with $\pm r$ representing 95 % confidence intervals on the difference between two mean values (of 20 cigarettes), determined in one laboratory from matched samples by one operator using the same equipment within the shortest feasible period of time;

s_b is the standard deviation between laboratories, with $\pm R$ representing 95 % confidence intervals on the difference between mean values (again, of 20 cigarettes from matched samples), determined in two different laboratories by different operators using different equipment.

NOTE For reasons of statistical validity, it is necessary that these statistics be calculated from replicate data points, each based on mean values of a fixed number of cigarettes for both linear and rotary smoking machines: 20 in this instance. For a linear smoking machine, therefore, a single mean value is formed by averaging over the results from smoking 4 channels, of 5 cigarettes, on the same smoking run. For a rotary machine, this equates to one smoking run. Repeatability and reproducibility values based on the testing of 20 cigarettes are designated by r_{20} and R_{20}, respectively.

It should also be noted that prior to the final calculations to produce estimates of s_b and s_w, the data should be screened for possible 'outliers'; that is, extremely high or low results relative to the large majority of the data which, if retained, would erroneously inflate the values of r and R. Various approaches for identifying outliers within a laboratory data set are specified in ISO 5725-2:1994 and certain techniques are recommended. However, the standard does not recommend tests for identifying outlying laboratories, but recognises the need for informed judgement. Clause 7.2.5 states 'This part of ISO 5725 does not provide a statistical test by which suspected laboratories may be judged. The primary decision should be the responsibility of the statistical expert, ….'. Obviously suspect data is best removed if confirmed to be technically suspect by the reporting laboratory. The consequence of removing too many results would be to erroneously reduce the estimates of r and R; and most crucially, R would be under-estimated if results for complete laboratories were unnecessarily removed. There is obviously a need for a cautious approach of this nature, which can result in suspect values being reported; and some are highlighted in Tables 2 and 3.

2.2 A statistical model for compliance tolerances

It is important to appreciate that the 95 % confidence intervals based on r_{20} and R_{20} alone, would be too low if applied in the context of checking on-pack declarations of NFDPM, nicotine and carbon monoxide. Two main components are missing:

— that due to the effects of rounding, to declare the on-pack values for NFDPM and carbon monoxide to the nearest integer and nicotine to one decimal place, and

— the component associated with the product (namely, longer-term product variability and the possible interaction between different product designs and their measurement by separate laboratories).

In the case of rounding, this can be calculated by assuming that the 'errors' follow a rectangular distribution. For example, if a mean value is to be corrected to the nearest integer, the errors would be evenly distributed between – 0,5 and + 0,5. It follows (from mathematical analysis of this distribution-function) that a variance of 0,083 needs to be included for NFDPM and carbon monoxide when rounding is to the nearest integer, with a variance of 0,00083 for nicotine when rounded to 1 decimal place.

Obtaining estimates of the additional product-related components of variance is not so straight forward. Ideally, the collaborative studies carried out to estimate r_{20} and R_{20}, would have been replicated on numerous and separate production runs of the brands tested. In the absence of this, the only data available for gaining insight into the additional product–related statistical variation is that from the UK Department of Health Survey, for which sampling and testing take place over a 12-month period and conform with ISO 8243. Results from this survey are discussed in 3.2 and 4.4 of this document and a related technical paper is provided in Annex C.

In Annex C, a statistical model is presented to extend the calculated reproducibility value to include the additional variance components due to rounding of the on-pack declared values of NFDPM, nicotine and carbon monoxide, and those related to the product itself. This is reproduced below, firstly to illustrate the way in which the different components of variance are combined for the purpose of estimating 95 % confidence intervals for checking on-pack declarations and, secondly, to indicate the need for wider intervals when sampling and testing occurs at one point in time rather than on a number of occasions over a period of time.

$$95\ \%\ CI = \pm 2\ \{2\ [(P \pm s_w^2)/5 + s_b^2 \pm P_L] + \text{Rounding}\}^{1/2} \quad (1)$$

where

P is the variance due to product variability over time;

s_b^2 s the variance due to between laboratory differences for individual brands;

P_L is the variance due to interaction between measurements by separate laboratories of different brands;

s_w^2 is the repeatability variance to the basis of 20 cigarettes.

Rounding is the variance associated with rounding.

NOTE This model assumes that the mean values obtained by a manufacturer (for determining the packet declaration) and by a would-be regulator (for checking purposes) are each based on the results from machine-smokings of 100 cigarettes, i.e. data from the smoking of 20 cigarettes on samples obtained on each of 5 separate occasions of production.

If the additional product-related variance components are removed from (1), the model represents the reproducibility R_{100} for tests of 100 cigarettes.

$$R_{100} = \pm 2 \{2 [s_w^2/5 + s_b^2]\}^{½} \quad (2)$$

The effect of including the additional variance due to rounding in (2) above can be seen by comparing the R_{100} values (Tables 4 and 5) with the $R_{100+rndg}$ values (Tables 6 and 7). The increase due to rounding, whilst being of practical importance, is small in comparison to the measurement variability. Its greatest impact is at the low end of the yield range, as it diminishes with increasing yield.

The above statistical model (1) should be seen as a simplification of the full relationship for estimating the tolerance. With a more extensive data-set, involving more laboratories than take part in the UK Survey, it may well be shown that additional components of variance relating to measurements and the product over a period of time should be included in this model. Even so, it does serve to show the way in which separate key elements can be combined and indicates that when 'spot-checks' are made on packet declarations, the 95 % confidence intervals will be higher than when sampling and testing is carried out on a number of occasions over a period of time. The component P (and other possible time-related components not included in the above model) would not be divided by 5 (i.e. the number of separate occasions of sampling and testing).

3 Sources of data

3.1 International collaborative studies

International collaborative studies take place on a regular basis, both to provide r and R values and to allow laboratories to assess their performance against others. The latter is an essential part of validating the output of smoke-testing laboratories (a requirement of ISO 17025 for laboratory accreditation), which is a need clearly demonstrated by Annex D.

The most recent data comes from the 2003 CORESTA Collaborative Study (CCS-03), but in recent years there have been a number of collaborative studies carried out both on an annual and an ad hoc basis. Two annual studies are those performed by members of the CORESTA Analytical Methods Group (CAMG) on the CORESTA Monitor and participants in the Asia Collaborative Study (ACS) on five commercial brands. An outline of the studies for which data are reviewed is given in Table 1, and the full report of the latest CORESTA Collaborative Study is attached (Annex F). Also included in Table 1 are the studies from which the r and R values reported in ISO 4387:2000, ISO 10315:2000 and ISO 8454:1995 were derived.

The number of linear and rotary smoking machines used in each study is indicated in Table 1. It should be noted that both the linear and rotary descriptions cover a range of designs which have changed over the period covered by the studies. To take one example, 7 of the linear machines used for the ISO 4387:2000 study were 8-channel machines, and the remainder were the normal 20-channel machines. No 8-channel machines were used in the CCS-03 study, although 7 linear 16-channel machines were included.

The repeatability (r_{20}) and reproducibility (R_{20}) values based on the testing of 20 cigarettes are given in Tables 2 and 3 respectively, for the measurement of NFDPM, nicotine and carbon monoxide yield.

3.2 UK Department of Health Cigarette Survey data

The UK is the only market for which a substantial set of compliance data exists. The Department of Health Cigarette Survey procedure forms the basis for verifying the packet declarations. Originally a survey covered a 6-month period with monthly sampling, but since 1995 they take place over a calendar year. Also since 1995, each survey has involved bi-monthly sampling (from the factory or importer's warehouse) and laboratory testing over a 12-month period, with 4 channels of 5 cigarettes being smoked per sampling occasion. The official assessment of packet declarations compared with the Laboratory of the Government Chemist (LGC)[8] averages is carried out after the completion of each Survey and is based upon the average LGC value for at least 5 occasions of sampling and testing.

Concurrent with the Department of Health Survey, some manufacturers carry out their own testing according to the same format and on 'matched' samples from the same 200-outers (cartons). These data serve two purposes. They are regularly compared with corresponding data from the LGC as a check on between laboratory measurement differences; the measurement bias (Manufacturer's yield – LGC yield) being calculated for each brand. They are also used to monitor the ongoing performance of individual brands in relation to the NFDPM and smoke nicotine yields declared on the packet.

The average bias of all brands made or imported by a manufacturer during each survey can be easily calculated and provides a simple way of comparing the performance of laboratories. The plots of mean laboratory biases in Figures D.1, D.2 and D.3 show that differences between laboratories are not fixed but change with time. Usually the shift between successive surveys is small, but sometimes it can be large (Figure D.2 for nicotine). A common change in the direction of the bias between successive surveys indicates a measurement change by the reference laboratory (the LGC). The longer-term changes reflected by bias changes of this nature are not, of course, included in 'at one point in time' collaborative studies.

4 Comparison of 2003 CORESTA Collaborative Study data with those previously reported

4.1 General

The latest CORESTA Collaborative Study provides values of r_{20} and R_{20} for a number of brands tested on a range of smoking machines, including all the currently available designs, following considerable effort by their manufacturers to harmonize the operating conditions in order to minimize differences in the measurement of CO yields.

The points of interest are, therefore, whether these latest r_{20} and R_{20} values differ greatly from those previously reported, and whether the R_{20} values for CO are still greater than those for NFDPM.

To simplify the comparison of the CCS-03 data with that from the other collaborative studies, the other studies are shown as a combined data set in the graphs discussed below. It should be noted that the 'Other' data set represents the testing of many brands by many laboratories using different designs of smoking machines over a period of several years.

4.2 Comparison of repeatability r_{20} values from CCS-03 with other collaborative studies

The prime interest is in the reproducibility values but, as these are influenced by the value of the repeatability standard deviation (see 2.1), it is relevant to review the r_{20} values given in Table 2. The NFDPM and CO values, plotted against their respective yields, are combined in Figure 1 to provide a direct comparison between these smoke constituents, whilst the nicotine values are shown separately in Figure 2.

It can be seen from Figure 1 that the values for NFDPM and CO overlap and the CCS-03 data are within the ranges previously reported. The regression lines indicate the CCS-03 and 'Other' data to be similar, on average, for NFDPM, with the CO values being slightly higher for CCS-03.

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8) Since December 2002 the survey has been carried out by Arista Laboratories Europe.

It can be seen from Figure 2 that the CCS-03 nicotine values are also within the ranges previously reported, and the regression lines are almost identical.

The overall conclusion is, therefore, that the CCS-03 repeatability values for the three smoke constituents are not different to previously reported values.

4.3 Comparison of reproducibility R_{20} values from CCS-03 with other collaborative studies

4.3.1 General

The reproducibility values given in Table 3 are shown plotted against the corresponding smoke constituent yield in Figure 3, for NFDPM and CO, and Figure 4, for nicotine.

Again, the distribution of the individual data points in these plots show the CCS-03 values to be within the range previously reported, although at the lower end for NFDPM and CO (see Figure 3). The CCS-03 data is, therefore, in line with the best previously reported. The CO values for all the data, both from the CCS-03 and other studies, are clearly greater than those for NFDPM. This is clearly illustrated by the separation between the corresponding regression lines for NFDPM and CO.

It should be noted that, although the NFDPM value for cigarette type 1 deviated considerably from the line fitted to the CCS-03 data, it was not considered to be sufficiently extreme to be treated as an outlier. It does serve, however, as an illustration of how much these values can vary from brand to brand, even within well-run collaborative studies.

The CCS-03 nicotine data (see Figure 4) corresponds closely with that previously reported, as illustrated by the regression lines.

The overall conclusion is that the recent work to better harmonize the operating conditions of smoking machines has not resulted in a clear reduction in the reproducibility of yield measurements. The CCS-03 data is, however, at the lower end of the reported range for CO and, therefore, in line with the best previously reported. As the study also included a mix of all current smoking-machine types, it must be considered to best represent the current level of measurement variability.

4.3.2 Relationship between reproducibility R_{20} and smoke constituent yield

It is interesting to note that the regression lines for all three smoke constituents show a linear increase in R_{20} with yield from a relatively large intercept value. The use of a simple percentage tolerance is, therefore, only viable if used in conjunction with a minimum value. Figures 5 and 6, with R_{20} re-plotted as a percentage of the smoke constituent yield, clearly illustrate this point. At very low constituent yields, the percentage R_{20} value is very high (i.e. infinite at zero yield) but decreases to become an almost fixed percentage at much higher yields.

The overall conclusion is that it is necessary to qualify the percentage tolerance with a minimum value for all three smoke constituents.

4.4 Comparison of R_{100} reproducibility values from collaborative studies with measurement tolerances estimated from the UK Department of Health Cigarette Survey data

As highlighted by the statistical model in section 2.2, the r and R values are traditionally based upon tests of 20 cigarettes and need to be adjusted when tests involve a different number. The ISO standards require 100 cigarettes to be tested, either as a single sample or as 5 separate samples of 20 cigarettes. The UK Department of Health Cigarette Survey is based upon testing 20 cigarettes from each of 6 sampling periods, but results are only reported if samples from at least 5 periods are tested. It is, therefore, more appropriate when comparing data from the collaborative studies discussed in section 4 with UK data, and considering its relevance to compliance tolerances, to use reproducibility (R_{100}) values based on 100 cigarettes.

Figures 3 and 4 showing R_{20} values plotted against the respective smoke constituent yield, are duplicated for R_{100} values in Figures 7 and 8, but with the UK measurement tolerance from Table D.1 also included. Although the UK data is based upon only 6 laboratories, the measurements cover many brands over a 6-year