
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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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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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 tolerances³⁾ relevant to compliance checks on the yield information provided by manufacturers. They need to be combined with additional information on testing⁴⁾ 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.

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 tolerances⁶⁾, 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
$R_{100+rdg}$	20 % with a minimum of 1,5 mg
	<u>or</u>
	25 % with a minimum of 1 mg
$R_{100+rdg}$ 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.

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 CORESTA⁷⁾ 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)

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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

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

$$r = 2,8 * s_w$$

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

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 is 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.

$$R_{100} = \pm 2 \{2 [s_w^2/5 + s_b^2]\}^{1/2} \quad (2)$$

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 $R_{100+rdg}$ 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.

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

period and include the longer term measurement variability not present in the 'at one point in time' collaborative studies. It can be seen from Figure 7 that the NFDPM and CO data corresponds closely with that from the collaborative studies, the regression lines being very close to those from the 'Other' studies and slightly higher than the CCS-03 lines. Concerning nicotine, the correspondance is not as good (see Figure 8), the UK data being much higher.

Overall the UK data, based on a substantial data set, sits fairly centrally within the 'Other' data for NFDPM and CO and is, therefore, slightly higher than the CCS-03 values. It is at the top end of the range of 'Other' values for nicotine and, therefore, much higher than the CCS-03 values.

5 Review of information relevant to setting a compliance tolerance for carbon monoxide

5.1 General

It should be noted that different tolerances are given in ISO 8243 for sampling at one point in time or over a period of time. Those for one point in time are greater, in order to account for the increased product variability associated with a sample which is less representative of the population manufactured for sale. The tolerances quoted in this section are for 'over a period of time' sampling and testing.

5.2 Compliance data for current tolerances

The only market for which long-term compliance data exists appears to be the UK. The Department of Health Survey data includes the NFDPM and nicotine yields declared on the packet for each of the brands for which duplicate tests were carried out by the LGC and the manufacturer. The infringement rates for NFDPM and nicotine based on the tolerances given in ISO 8243 can therefore be calculated. If the tolerance is correctly set at the 95 % level, the expectation would be for the declared yields of only 5 % of brands tested to be outside of the limits (i.e. the greater of ± 15 % or ± 1 mg for NFDPM, or $\pm 0,1$ mg for nicotine). This was found to be the case for NFDPM (see Table C.4), with 4 % outside the tolerance, but for nicotine the infringement rate was 11 %. The reason for this higher infringement rate is unclear, but is probably associated with the greater than expected measurement variability, illustrated by Figure 8, among the particular group of laboratories involved in the UK Study.

As manufacturers have only recently been required to declare the CO yields, compliance data is not yet available in the UK. The LGC and Manufacturer's measurements are, however, available in accordance with the same procedures as for NFDPM and nicotine. These have been used to predict that, for CO, 10 % of brands would be expected to violate the NFDPM tolerance (i.e. the greater of ± 15 % or ± 1 mg) whilst for NFDPM and nicotine, using the same procedure, 5 % and 11 % would be expected respectively, i.e. almost exactly as observed (see Table C.4). The NFDPM tolerance was applied to CO as the work was reported prior to the introduction of the current UK and ISO 8243 tolerance of the greater of ± 20 % or ± 1 mg.

Overall, the UK data indicates that the 15 %, or 1 mg, tolerance for NFDPM is appropriate, but that for CO it needs to be greater. The 15 %, or 0,1 mg, tolerance for nicotine does not seem to be working at the expected 95 % acceptance rate, but this is possibly due to the greater than expected measurement variability between the group of laboratories involved in the UK Study.

5.3 Confidence intervals associated with yield measurements

The confidence intervals, i.e. reproducibility values, associated with the measurement of NFDPM and CO yields in accordance with the procedures of ISO 4387 and ISO 8454 respectively, have been reviewed in both Annex B and this Technical Report. This Technical Report also reviews the R_{20} values for nicotine yield by ISO 10315.

The reproducibility data from ISO 4387 and ISO 8454, together with data from DIN ring trials and CORESTA inter-laboratory studies, were reviewed in Annex B. The conclusion, based on this data, was that a tolerance of ± 25 % or ± 1 mg was justified, but ± 20 % or ± 1 mg should be recommended on the basis of improved harmonization of smoking machines and future good quality control during manufacture.

The review of reproducibility values in this Technical Report is based upon a more extensive data set (see Table 1). In addition, the R_{20} values have been adjusted to represent the testing of 100 cigarettes (R_{100}), this being the basis upon which compliance checks based on ISO standards are carried out. The R_{100} values are combined for NFDPM and CO in Figure 7 and for nicotine in Figure 8, in both cases plotted against the respective smoke constituent yield.

In line with the conclusion drawn in 4.3, the CCS-03 data and the regression lines fitted to them are taken to best represent the current position on measurement variability. The linear regression equations for the CCS-03 data from Figure 7 have been used to calculate the R_{100} values for NFDPM and carbon monoxide for yields of 1 mg to 15 mg and these are given in Table 4 together with the CO/NFDPM ratio. The values are tabulated both in mg and percentage units, with the range of each column covered by the tolerances in ISO 8243 identified by the shading. It is clear that the R_{100} values for carbon monoxide are always significantly greater than those for NFDPM at matched yields. In addition for CO, the R_{100} value is greater than the minimum tolerance of 1 mg at a yield of 5 mg and the equal to the 20 % tolerance at 6 mg. The values for NFDPM are all below the ISO 8243 tolerances, as are those for nicotine given in Table 5.

Overall, it seems that the current CO tolerance in ISO 8243 is set below the minimum required value to allow for the variability in the yield measurement (R_{100}) using ISO 8454.

5.4 Statistical models

The statistical function for the reproducibility of smoke constituent yield measurements R_{20} (see 2.1) was extended to include additional components of variance relevant to calculating a compliance tolerance (see 2.2). This process can be broken down into three steps in terms of the level of certainty about the inclusion of each new term and the data from which to estimate it.

Step 1

$$R_{20} \rightarrow R_{100}$$

Step 2

$$R_{100} \rightarrow R_{100} + \text{Rounding Variance Term} = R_{100+\text{rndg}}$$

Step 3

$$R_{100+\text{rndg}} \rightarrow R_{100+\text{rndg}} + \text{Product Variance Terms} = \text{Compliance Tolerance}$$

The data in R_{100} form, i.e. 'Step 1', has already been considered in 5.3 above.

The data in $R_{100+\text{rndg}}$ form, i.e. 'Step 2', is reviewed below, but as it is only possible to estimate 'Step 3' values using product variance terms based upon the UK Department of Health Survey data, which is not specifically designed to provide such data, the 'Step 3' tolerance values are not, therefore, included in this report.

The derivation of the $R_{100+\text{rndg}}$ values is described in 2.2. They are plotted against the respective smoke constituent yield in Figure 9 for NFDPM and CO, and in Figure 10 for nicotine. The regression lines for the CCS-03 data were used to calculate the $R_{100+\text{rndg}}$ values for NFDPM and CO in Table 6 and nicotine in Table 7.

Again, the $R_{100+\text{rndg}}$ CO values are much greater than those for NFDPM, as demonstrated by the values for the CO/NFDPM ratio in Table 6, and exceed the ISO 8243 tolerance over the yield range of 4 mg to 7 mg. It should be noted that this range covers the cross-over point between the minimum and percentage tolerance values, thereby allowing the tolerance to be reset to an appropriate value by increasing either. It should also be noted that NFDPM and nicotine values are equal to or slightly greater than the ISO 8243 tolerances over the range of 6 mg to 7 mg, again the cross-over point between the minimum and percentage tolerance values.

Overall, the $R_{100+rdg}$ values show that the current ISO 8243 CO tolerance needs to be increased in order to avoid falsely failing brands during compliance checks, and there is evidence that this risk extends to NFDPM and nicotine at the cross-over point between the minimum and percentage tolerance values.

5.5 Prediction of a tolerance for CO from the relative variability in their reproducibility values

The CO/NFDPM ratio values in Table 6 can be used to estimate a CO tolerance on the basis of the ISO tolerance for NFDPM being valid and that for CO needing to reflect the relative values of reproducibility for the two smoke constituents. The tolerance for NFDPM is, therefore, 1 mg for yields of 1 mg to 6 mg and then 15 % for higher yields, and the corresponding CO tolerance is the product of the NFDPM value and the CO/NFDPM ratio. The values, thus calculated, are given in Table 8 both in mg and percentage units. The values indicate that a minimum tolerance of about 1,5 mg is required with a percentage tolerance of about 22 %.

Overall the CO/NFDPM ratio values indicate the current ISO 8243 minimum value for the CO tolerance needs to be increased to 1,5 mg.

6 Conclusions

The latest CCS-03 data gives similar estimates of repeatability and reproducibility to those previously reported, although the reproducibility is at the lower end of the range for CO. Although there is no hard evidence that the harmonization work on smoking machines has reduced the variability in CO yield measurements, the data from CCS-03 is 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 CCS-03 data shows measurements of carbon monoxide to be more variable than those for NFDPM, in line with previously reported collaborative data and with that provided by the UK Department of Health Survey. It therefore supports the need for a higher compliance tolerance for CO.

Whilst the reproducibility estimates, in the form of the R_{100} and $R_{100+rdg}$ values reported here, cannot be taken directly as compliance tolerances for checking declared values, no tolerance should be set at a lower value.

The R_{100} and $R_{100+rdg}$ values show that the CO tolerance in ISO 8243 needs to be increased to prevent too many brands being falsely failed in the yield range around the cross-over point between the minimum and percentage tolerance values.

The relative values of reproducibility for NFDPM and carbon monoxide indicate that a tolerance of 22 % with a minimum of 1,5 mg is required for the measurement of carbon monoxide yields when sampling and testing over a period of time.

7 Recommendations

The tolerance in ISO 8243 should be amended in line with the findings in Clause 5 and the conclusions drawn in Clause 6 of this report.

The most cautious approach would be to increase the minimum tolerance for CO for testing 'over a period of time' to 1,5 mg and leave the basic tolerance set at 20 %. Since the 'one point in time' tolerance should not be lower, the same minimum value should apply.

The tolerance values should be further reviewed when compliance rates are established from regulatory checks. It is probable that such data will only become available in the UK and would require 2 to 3 years to assemble.

Table 1 — Details of collaborative studies

Data source	Year of study	Number of labs	Number of cigarette brands/types	Number and type of smoking machines						Number of cigarettes tested	
				linear			rotary			linear	rotary
				NFDPM	CO		NFDPM	CO			
ISO 4387	1990	30	6	23	NR		7	NR		100	100
ISO 10315	1990	30	6	23	NR		7	NR		100	100
ISO 8454	1993	32	4	NR ^a	20		NR	13		100	100
ISO 15592-3	2001	20	1 (CM3) ^b		NR			NR		300	300
CAMG ^c	2000		1 (CM3) ^b	12	10		15	12		100	100
CAMG ^c	2001		1 (CM3) ^b	14	12		11	11		100	100
CAMG ^c	2002		1 (CM3) ^b	16	14		22	23		100	100
9 th ACS ^d	2000	42	5	38	NR		13	NR		120	120
10 th ACS ^d	2001	44	5	29	20		19	19		120	120
11 th ACS ^d	2002	42	5	30	20		19	19		120	120
1 st ASR ^e	2001	17	12	7	7		12	12		50	60
2 nd ASR ^e	2002		3	11	11		15	14		50	60
CCS ^f	2003	39	8	22	22		39	39		100	100

^a NR indicates the yield of the smoke constituent was not reported.
^b CM3 indicates the 3rd CORESTA Monitor was tested.
^c CORESTA Analytical Methods Group (tests of 3rd CORESTA monitor CM3).
^d Asia Collaborative Study.
^e CORESTA Alternative Smoking Regime Task Force.
^f CORESTA Collaborative Study.

Table 2 — Repeatability values from collaborative studies

Study	Cigarette	NFDPM			Nicotine			CO		
		Yield mg	r_{20} mg	r_{20} %	Yield mg	r_{20} mg	r_{20} %	Yield mg	r_{20} mg	r_{20} %
ISO 4387	1	0,82	0,40	48,8	NA	NA	NA	NA	NA	NA
	2	1,61	0,52	32,3	NA	NA	NA	NA	NA	NA
	3	3,31	0,52	15,7	NA	NA	NA	NA	NA	NA
	4	7,70	0,88	11,4	NA	NA	NA	NA	NA	NA
	5	12,61	1,06	8,4	NA	NA	NA	NA	NA	NA
	6	17,40	1,19	6,8	NA	NA	NA	NA	NA	NA
ISO 10315	1	NA	NA	NA	0,09	0,040	44,0	NA	NA	NA
	2	NA	NA	NA	0,18	0,046	25,7	NA	NA	NA
	3	NA	NA	NA	0,33	0,050	15,3	NA	NA	NA
	4	NA	NA	NA	0,67	0,077	11,4	NA	NA	NA
	5	NA	NA	NA	0,84	0,079	9,5	NA	NA	NA
	6	NA	NA	NA	1,41	0,107	7,6	NA	NA	NA
ISO 8454	1	NA	NA	NA	NA	NA	NA	3,45	0,47	13,6
	2	NA	NA	NA	NA	NA	NA	3,56	0,42	11,8
	3	NA	NA	NA	NA	NA	NA	9,89	0,85	8,6
	4	NA	NA	NA	NA	NA	NA	13,80	1,09	7,9
ISO 15592-3	1	15,00	0,94	6,3	1,21	0,087	7,2	NM	NM	NM
ACS9	1	1,66	0,37	22,2	0,09	0,026	28,6	NM	NM	NM
	2	2,74	0,44	16,2	0,29	0,042	14,4	NM	NM	NM
	3	4,35	0,50	11,4	0,37	0,041	11,1	NM	NM	NM
	4	10,04	0,69	6,9	0,83	0,065	7,9	NM	NM	NM

Table 2 (continued)

Study	Cigarette	NFDPM		Nicotine		CO	
		Yield mg	r_{20} mg	Yield mg	r_{20} mg	Yield mg	r_{20} mg
	5	15,35	0,86	1,25	0,080	NM	NM
ACS10	1	1,24	0,31	0,11	0,021	1,99	0,38
	2	2,90	0,49	0,30	0,038	3,88	0,50
	3	4,71	0,53	0,45	0,042	8,85	0,78
	4	10,08	0,73	0,86	0,069	9,81	0,80
	5	15,16	0,83	1,23	0,075	14,79	0,94
ACS11	1	1,20	0,32	0,14	0,025	1,63	0,36
	2	2,95	0,42	0,28	0,036	3,63	0,45
	3	4,65	0,55	0,40	0,051	5,58	0,71
	4	9,40	0,75	0,83	0,091	8,85	0,98
	5	14,26	0,85	1,30	0,081	13,09	0,88
CCS-03	1	12,68	0,94	0,94	0,073	8,31	0,82
	2	14,06	0,74	1,30	0,078	13,23	0,87
	3	10,07	0,66	0,82	0,068	10,99	1,00
	4	11,56	0,71	0,85	0,059	11,85	0,95
	5	5,43	0,55	0,47	0,037	6,79	0,75
	6	10,04	0,64	0,79	0,063	9,55	0,74
	7	3,54	0,49	0,33	0,035	4,12	0,52
	8	9,83	0,72	0,88	0,071	11,45	0,97
	1	15,13	0,88	1,24	0,066	14,99	1,02
	1	15,15	0,74	1,22	0,069	14,48	0,74

Table 2 (continued)

Study	Cigarette	NFDPM			Nicotine			CO		
		Yield mg	r_{20} mg	r_{20} %	Yield mg	r_{20} mg	r_{20} %	Yield mg	r_{20} mg	r_{20} %
CAMG-02	1	14,13	0,83	5,9	1,30	0,078	6,0	13,51	0,93	6,9
CASR2	1	1,19	0,27	22,9	0,14	0,017	11,7	1,94	0,28	14,3
	2	9,39	0,49	5,3	0,83	0,057	6,8	11,63	0,87	7,5
	3	14,07	0,74	5,3	1,31	0,083	6,3	13,26	2,47	18,7
CASR1	1	9,99	0,71	7,1	0,94	0,066	7,0	11,95	0,72	6,1
	2	12,42	0,82	6,6	0,92	0,069	7,5	13,43	0,92	6,8
	3	10,09	0,69	6,8	0,58	0,046	7,9	11,79	1,04	8,8
	4	8,56	0,50	5,9	0,70	0,049	7,0	8,76	0,62	7,1
	5	7,14	0,53	7,4	0,55	0,043	7,8	8,89	0,72	8,1
	6	6,66	0,46	6,8	0,55	0,058	10,5	7,81	0,63	8,0
	7	5,89	0,59	10,1	0,53	0,047	8,9	7,14	0,50	7,0
	8	4,43	0,47	10,7	0,39	0,036	9,2	6,52	0,48	7,3
	9	2,96	0,43	14,5	0,29	0,043	14,8	3,64	0,40	10,9
	10	1,30	0,26	20,0	0,14	0,024	17,1	2,30	0,30	13,2
	11	8,72	0,79	9,1	0,76	0,058	7,6	12,47	0,79	6,3
	12	14,94	0,72	4,8	1,25	0,042	3,4	14,85	0,91	6,1
a NA-Not Applicable and NM-Not Measured.										
b Values in shaded cells were considered suspect and not used in the report.										

Table 3 — Reproducibility values from collaborative studies

Study	Cigarette	NFDPM			Nicotine			CO		
		Yield mg	R_{20} mg	R_{20}	Yield mg	R_{20} mg	R_{20} %	Yield mg	R_{20} mg	R_{20} %
ISO 4387	1	0,82	0,60	73,2	NA	NA	NA	NA	NA	NA
	2	1,61	0,74	46,0	NA	NA	NA	NA	NA	NA
	3	3,31	0,90	27,2	NA	NA	NA	NA	NA	NA
	4	7,70	1,51	19,6	NA	NA	NA	NA	NA	NA
	5	12,61	1,70	13,5	NA	NA	NA	NA	NA	NA
	6	17,40	1,84	10,6	NA	NA	NA	NA	NA	NA
ISO 10315	1	NA	NA	NA	0,09	0,069	75,8	NA	NA	NA
	2	NA	NA	NA	0,18	0,069	38,5	NA	NA	NA
	3	NA	NA	NA	0,33	0,076	23,3	NA	NA	NA
	4	NA	NA	NA	0,67	0,109	16,2	NA	NA	NA
	5	NA	NA	NA	0,84	0,142	17,0	NA	NA	NA
	6	NA	NA	NA	1,41	0,195	13,8	NA	NA	NA
ISO 8454	1	NA	NA	NA	NA	NA	NA	3,45	1,18	34,2
	2	NA	NA	NA	NA	NA	NA	3,56	1,03	28,9
	3	NA	NA	NA	NA	NA	NA	9,89	2,22	22,4
	4	NA	NA	NA	NA	NA	NA	13,80	3,00	21,7
ISO 15592-3	1	15,00	1,98	13,2	1,21	0,216	17,9	NM	NM	NM
	1	1,66	2,24	134,7	0,09	0,072	79,4	NM	NM	NM
ACS9	2	2,74	0,84	30,8	0,29	0,081	28,3	NM	NM	NM
	3	4,35	0,97	22,3	0,37	0,094	25,1	NM	NM	NM
	4	10,04	1,75	17,5	0,83	0,161	19,4	NM	NM	NM

Table 3 (continued)

Study	Cigarette	NFDPM			Nicotine			CO		
		Yield mg	R ₂₀ mg	R ₂₀	Yield mg	R ₂₀ mg	R ₂₀ %	Yield mg	R ₂₀ mg	R ₂₀ %
	5	15,35	1,89	12,3	1,25	0,302	24,2	NM	NM	NM
ACS10	1	1,24	0,57	46,0	0,11	0,046	40,7	1,99	0,78	39,4
	2	2,90	0,90	31,1	0,30	0,085	28,6	3,88	1,21	31,2
	3	4,71	1,01	21,4	0,45	0,098	21,8	8,85	2,17	24,5
	4	10,08	1,40	13,9	0,86	0,167	19,3	9,81	2,08	21,2
	5	15,16	1,69	11,1	1,23	0,210	17,1	14,79	2,62	17,7
ACS11	1	1,20	0,66	55,0	0,14	0,045	33,5	1,63	0,79	48,2
	2	2,95	0,63	21,4	0,28	0,062	21,9	3,63	0,82	22,6
	3	4,65	0,73	15,7	0,40	0,083	20,5	5,58	0,95	17,0
	4	9,40	1,20	12,7	0,83	0,138	16,6	8,85	1,71	19,3
	5	14,26	1,83	12,8	1,30	0,184	14,2	13,09	1,79	13,7
CCS-03	1	12,68	2,12	16,7	0,94	0,133	14,1	8,31	1,72	20,7
	2	14,06	1,73	12,3	1,30	0,145	11,2	13,23	2,20	16,6
	3	10,07	1,24	12,3	0,82	0,123	15,1	10,99	2,11	19,2
	4	11,56	1,11	9,6	0,85	0,107	12,6	11,85	2,22	18,7
	5	5,43	0,80	14,8	0,47	0,076	16,3	6,79	1,42	20,9
	6	10,04	1,01	10,0	0,79	0,100	12,7	9,55	1,66	17,4
	7	3,54	0,75	21,3	0,33	0,073	21,8	4,12	1,08	26,1
	8	9,83	1,30	13,2	0,88	0,124	14,0	11,45	2,09	18,2
CAMG-00	1	15,13	1,49	9,8	1,24	0,109	8,8	14,99	2,82	18,8
CAMG-01	1	15,15	1,55	10,2	1,22	0,111	9,1	14,48	2,52	17,4

Table 3 (continued)

Study	Cigarette	NFDPM			Nicotine			CO		
		Yield mg	R_{20} mg	R_{20}	Yield mg	R_{20} mg	R_{20} %	Yield mg	R_{20} mg	R_{20} %
CAMG-02	1	14,13	1,73	12,2	1,30	0,161	12,4	13,51	2,59	19,2
CASR2	1	1,19	0,53	44,4	0,14	0,045	31,6	1,94	0,92	47,5
	2	9,39	1,21	12,9	0,83	0,135	16,3	11,63	2,23	19,2
	3	14,07	1,39	9,9	1,31	0,199	15,2	13,26	3,25	24,5
CASR1	1	9,99	1,35	13,5	0,94	0,125	13,3	11,95	2,62	21,9
	2	12,42	1,36	10,9	0,92	0,137	14,9	13,43	3,52	26,2
	3	10,09	1,66	16,5	0,58	0,079	13,6	11,79	2,50	21,2
	4	8,56	1,16	13,6	0,70	0,098	14,0	8,76	2,07	23,7
	5	7,14	1,10	15,4	0,55	0,085	15,5	8,89	2,28	25,6
	6	6,66	1,03	15,4	0,55	0,077	14,0	7,81	1,98	25,3
	7	5,89	0,89	15,1	0,53	0,075	14,2	7,14	1,56	21,8
	8	4,43	0,86	19,4	0,39	0,066	16,9	6,52	1,88	28,8
	9	2,96	0,68	22,9	0,29	0,069	23,8	3,64	1,06	29,1
	10	1,30	0,52	40,0	0,14	0,043	30,7	2,30	0,89	38,8
	11	8,72	1,30	14,9	0,76	0,095	12,5	12,47	2,63	21,1
	12	14,94	1,78	11,9	1,25	0,117	9,4	14,85	2,36	15,9
a NA-Not Applicable and NM-Not Measured.										
b Values in shaded cells were considered suspect and not used in the report.										

Table 4 — R_{100} values for NFDPM and CO calculated from the 2003 CORESTA Collaborative Study data

Yield	R_{100} value						
	NFDPM			CO			CO/NFDPM ratio
		8243	% of yield		8243	% of yield	
mg	mg	tol.		mg	tol.		
1	0,18	1 mg	18,3	0,55	1 mg	55,0	3,01
2	0,29		14,4	0,68		34,0	2,36
3	0,39		13,1	0,81		26,9	2,06
4	0,50		12,5	0,94		23,4	1,88
5	0,60		12,1	1,07		21,3	1,77
6	0,71	1 mg	11,8	1,19	20 %	19,9	1,69
7	0,81	15 %	11,6	1,32	20 %	18,9	1,63
8	0,92		11,5	1,45		18,2	1,58
9	1,02		11,4	1,58		17,6	1,54
10	1,13		11,3	1,71		17,1	1,51
11	1,23		11,2	1,84		16,7	1,49
12	1,34		11,2	1,97		16,4	1,47
13	1,44		11,1	2,10		16,1	1,45
14	1,55		11,1	2,22		15,9	1,44
15	1,65	15 %	11,0	2,35	20 %	15,7	1,42

^a The shaded sections indicate the ranges covered by the tolerances given in ISO 8243:2003 for 'over a period of time' sampling.

Table 5 — R_{100} values for nicotine calculated from the 2003 CORESTA Collaborative Study data

Yield	R_{100} value		
	mg	8243 tol.	% of yield
0,1	0,047	0,1 mg	46,6
0,2	0,054		26,8
0,3	0,061		20,2
0,4	0,068		26,9
0,5	0,075		15,0
0,6	0,082	0,1 mg	13,6
0,7	0,089	15 %	12,7
0,8	0,096		12,0
0,9	0,103		11,4
1,0	0,110		11,0
1,1	0,117		10,7
1,2	0,124		10,4
1,3	0,131		10,1
1,4	0,138		9,9
1,5	0,145	15 %	9,7

^a The shaded sections indicate the ranges covered by the tolerances given in ISO 8243:2003 for 'over a period of time' sampling.

Table 6 — $R_{100+rdg}$ values for NFDPM and CO calculated from the 2003 CORESTA Collaborative Study data

Yield	$R_{100+rdg}$ value						
	NFDPM			CO			CO/NFDPM ratio
		8243	% of yield		8243	% of yield	
mg	mg	tol.		mg	tol.		
1	0,46	1 mg	45,6	0,73	1 mg	73,3	1,61
2	0,55	1 mg	27,4	0,85	1 mg	42,7	1,56
3	0,64		21,4	0,98		32,5	1,52
4	0,73		18,4	1,10		27,4	1,49
5	0,83		16,6	1,22		24,4	1,47
6	0,92		15,3	1,34		22,3	1,46
7	1,01	15 %	14,5	1,46	20 %	20,9	1,44
8	1,11	15 %	13,8	1,58		19,8	1,43
9	1,20		13,3	1,70		18,9	1,42
10	1,29		12,9	1,83		18,3	1,41
11	1,39		12,6	1,95		17,7	1,41
12	1,48		12,3	2,07		17,2	1,40
13	1,57		12,1	2,19		16,8	1,39
14	1,66		11,9	2,31		16,5	1,39
15	1,76	15 %	11,7	2,43	20 %	16,2	1,38

NOTE The shaded sections indicate the ranges covered by the tolerances given in ISO 8243:2003 for 'over a period of time' sampling.

Table 7 — $R_{100+mdg}$ for nicotine calculated from the 2003 CORESTA Collaborative Study data

Yield	$R_{100+mdg}$ value		
	mg	8243 tol.	% of yield
0,1	0,071	0,1 mg	71,0
0,2	0,077		38,5
0,3	0,083		27,7
0,4	0,089		22,3
0,5	0,095		19,0
0,6	0,101	0,1 mg	16,9
0,7	0,107	15 %	15,3
0,8	0,113		14,2
0,9	0,119		13,3
1,0	0,125		12,5
1,1	0,131		12,0
1,2	0,138		11,5
1,3	0,144		11,0
1,4	0,150		10,7
1,5	0,156	15 %	10,4

NOTE The shaded sections indicate the ranges covered by the tolerances given in ISO 8243:2003 for 'over a period of time' sampling.

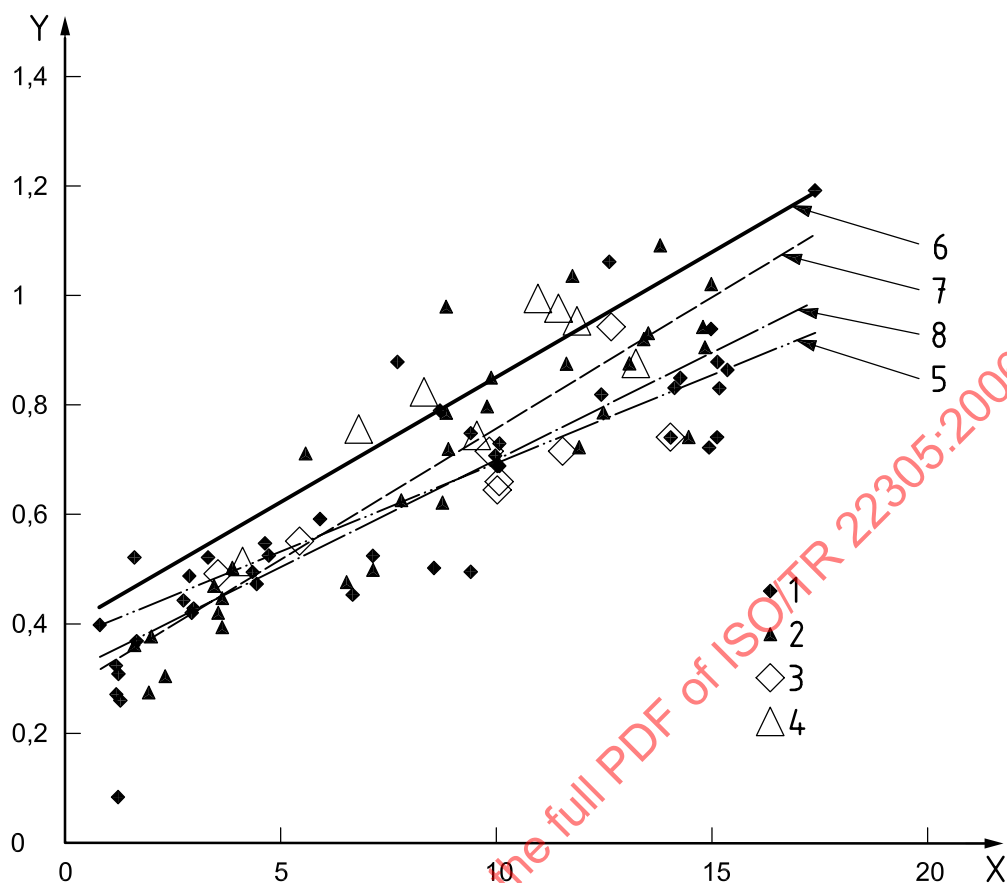
Table 8 — CO tolerance predicted from ISO 8243:2003 NFDPM tolerance and the CO/NFDPM ratio of the $R_{100+rdg}$ values from Table 6

Yield	NFDPM Tolerance value ^a ISO 8243:2003	CO/NFDPM ratio ($R_{100+rdg}$ values from Table 6)	Predicted CO tolerance ^b	Predicted CO tolerance ^c
mg	mg		mg	%
A	B	C	(B × C)	100 (B × C)/A
1	1,00	1,61	1,61	160,8
2	1,00	1,56	1,56	77,9
3	1,00	1,52	1,52	50,7
4	1,00	1,49	1,49	37,3
5	1,00	1,47	1,47	29,4
6	1,00	1,46	1,46	24,3
7	1,05	1,44	1,51	21,6
8	1,20	1,43	1,72	21,5
9	1,35	1,42	1,92	21,3
10	1,50	1,41	2,12	21,2
11	1,65	1,41	2,32	21,1
12	1,80	1,40	2,52	21,0
13	1,95	1,39	2,72	20,9
14	2,10	1,39	2,92	20,8
15	2,25	1,38	3,11	20,8

^a The tolerance specified in ISO 8243:2003 for 'over a period of time' sampling is 15 % of the NFDPM yield with a minimum value of 1 mg. The tolerance in mg is, therefore, 1 mg for yields of 1 mg to 6 mg, and 0,15 x Yield for yields of 7 mg and above.

^b The shaded section of the column indicates the yield range covered by the 1 mg minimum tolerance value for CO specified in ISO 8243:2003.

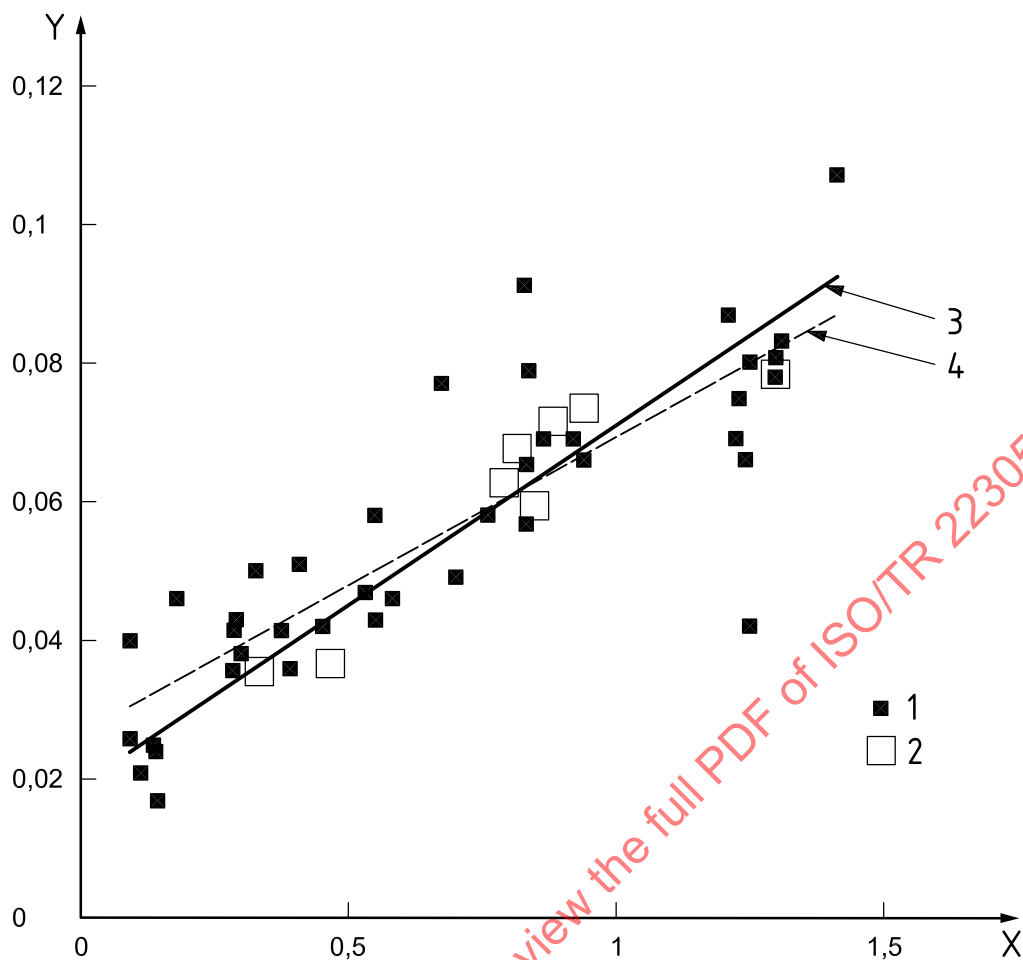
^c The shaded section of the column indicates the yield range covered by the 20 % tolerance value for CO specified in ISO 8243:2003 for 'over a period of time' sampling.

**Key**

- 1 other-NFDPM
- 2 other-CO
- 3 CCS-03-NFDPM
- 4 CCS-03-CO
- 5 linear (CCS-03-NFDPM)
- 6 linear (CCS-03-CO)
- 7 linear (other-CO)
- 8 linear (other-NFDPM)

X NFDPM or CO yield, mg
 Y repeatability r_{20} , mg

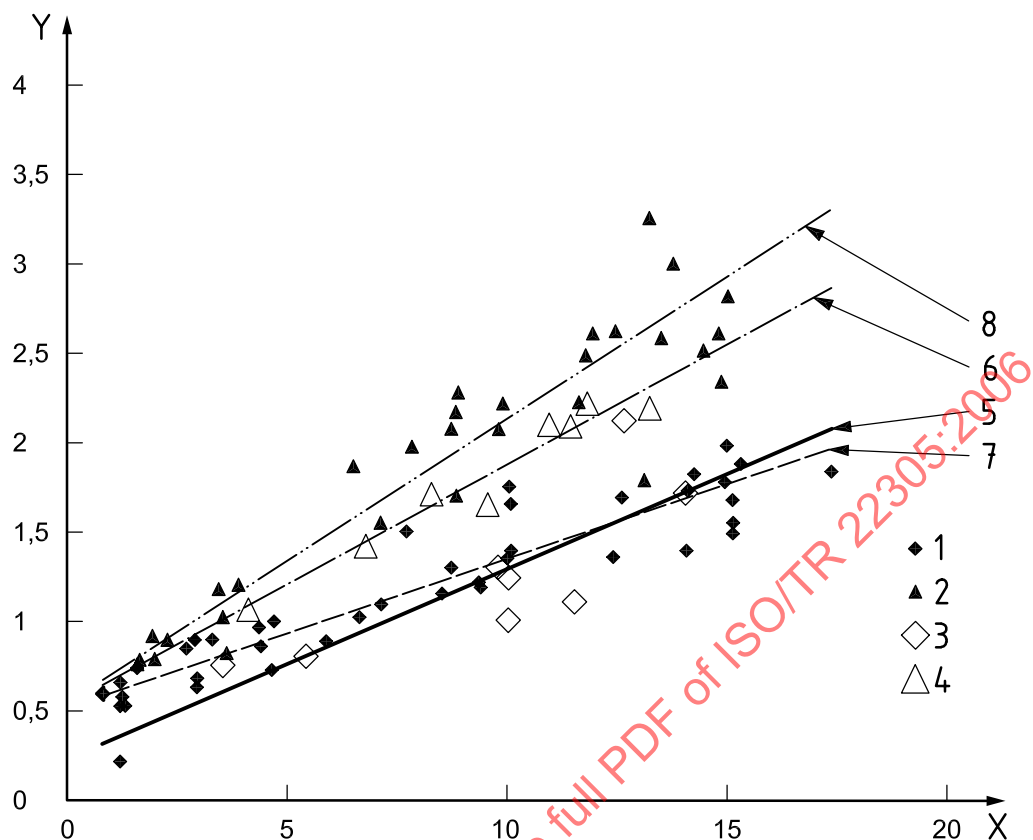
Figure 1 — r_{20} repeatability values for NFDPM and CO



Key

- 1 other
- 2 CCS-03
- 3 linear (CCS-03)
- 4 linear (other)
- X nicotine yield, mg
- Y reproducibility r_{20} , mg

Figure 2 — r_{20} repeatability values for nicotine

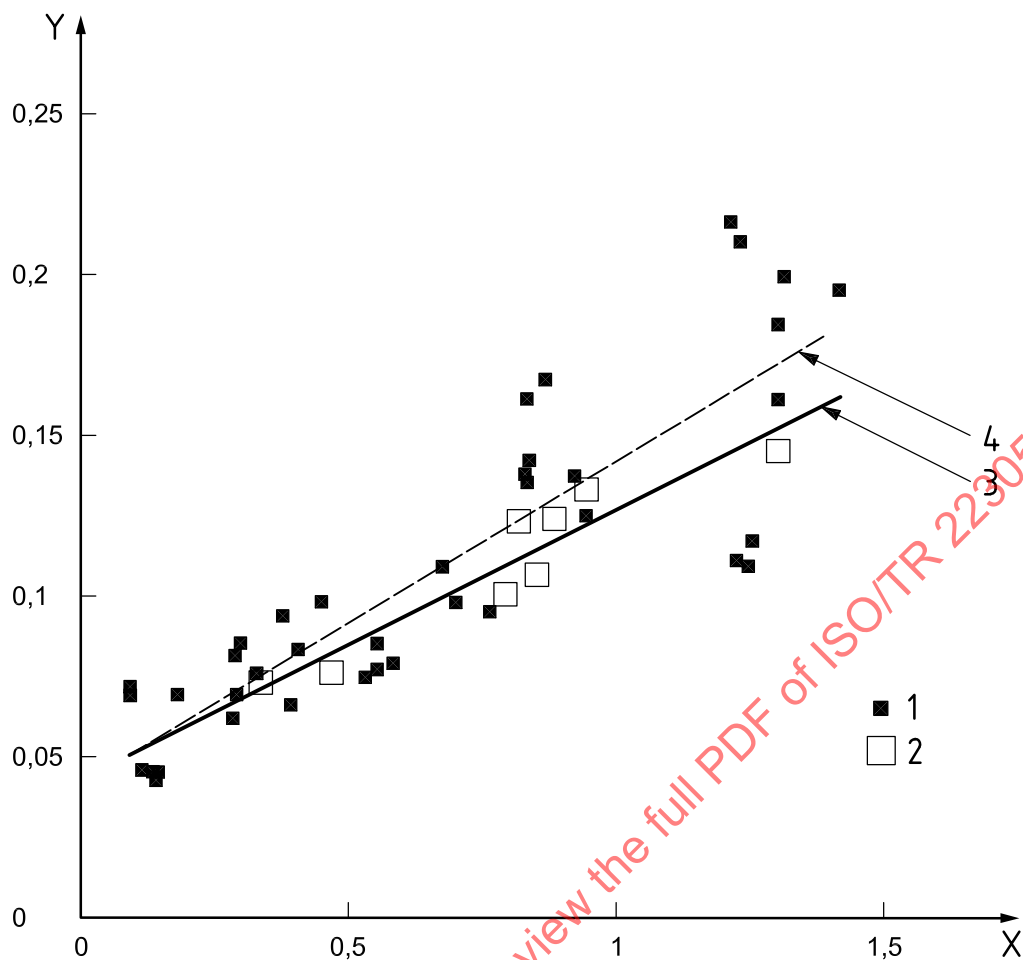


Key

- 1 other-NFDPM
- 2 other-CO
- 3 CCS-03-NFDPM
- 4 CCS-03-CO
- 5 linear (CCS-03-NFDPM)
- 6 linear (CCS-03-CO)
- 7 linear (other-NFDPM)
- 8 linear (other-CO)

X NFDPM or CO yield, mg
Y reproducibility R_{20} , mg

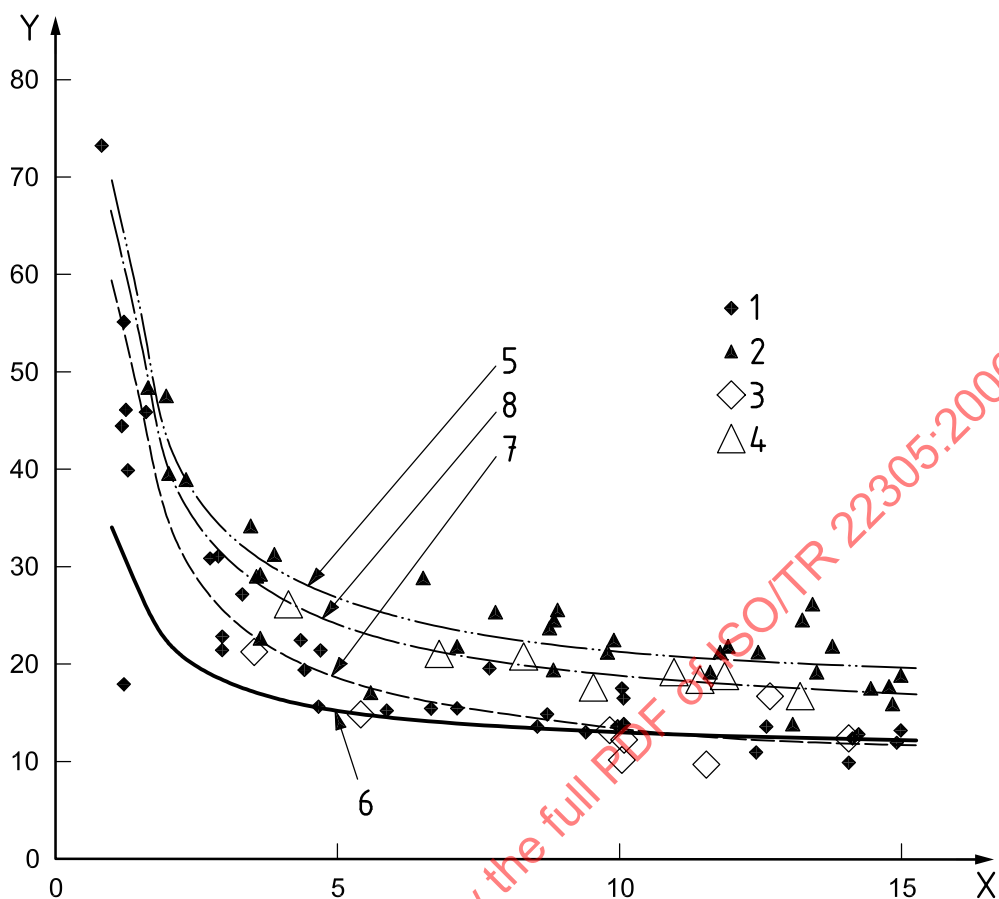
Figure 3 — R_{20} reproducibility values for NFDPM and CO



Key

- 1 other
 - 2 CCS-03
 - 3 linear (CCS-03)
 - 4 linear (other)
- X nicotine yield, mg
Y repeatability R_{20} , mg

Figure 4 — R_{20} reproducibility values for nicotine



Key

- 1 other-NFDPM
- 2 other-CO
- 3 CCS-03-NFDPM
- 4 CCS-03-CO
- 5 other-CO
- 6 CCS-03-NFDPM
- 7 other-NFDPM
- 8 CCS-03-CO

X NFDPM or CO yield, mg
Y reproducibility R_{20} , %

Figure 5 — R_{20} reproducibility values for NFDPM and CO (% of yield)

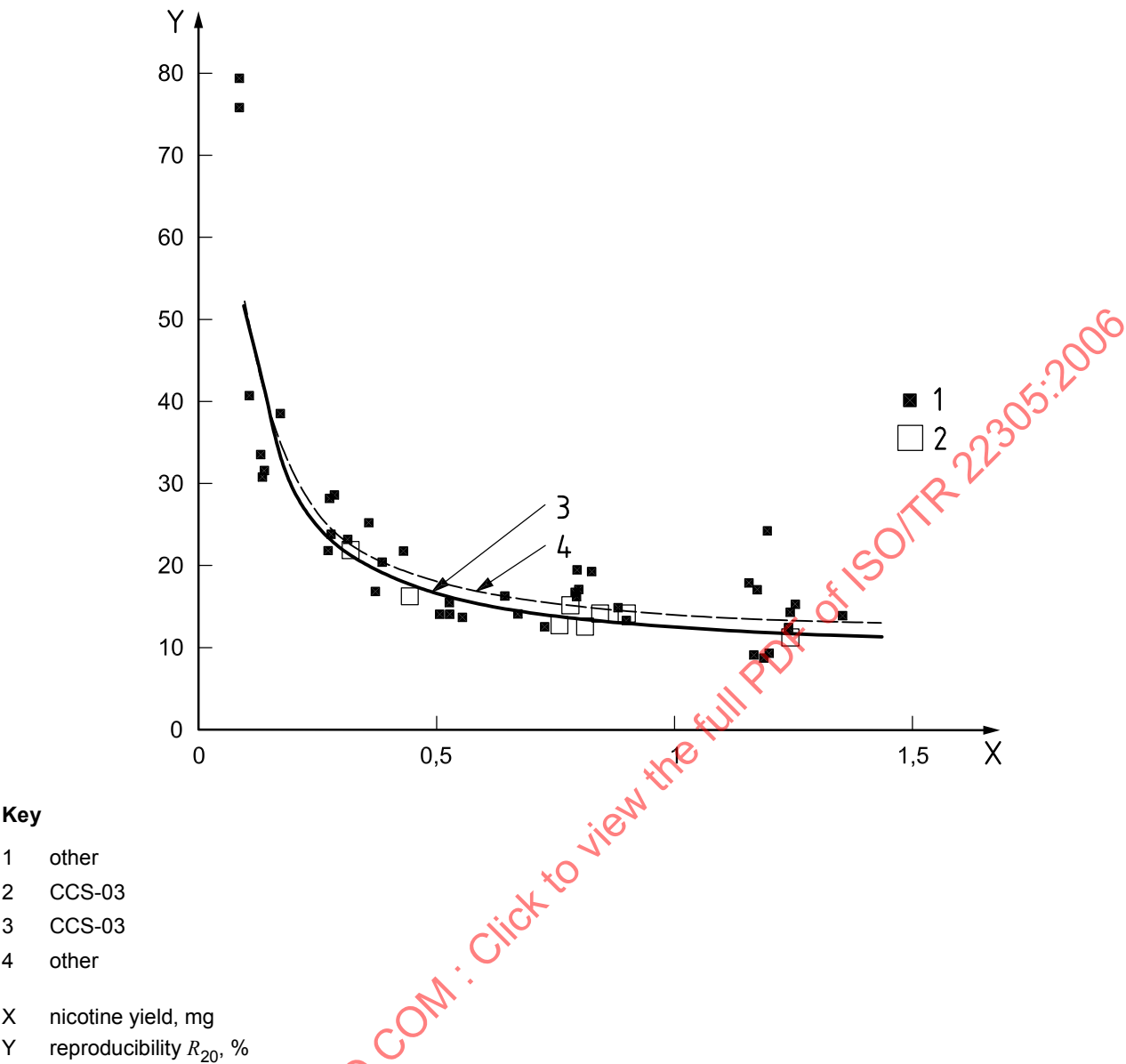
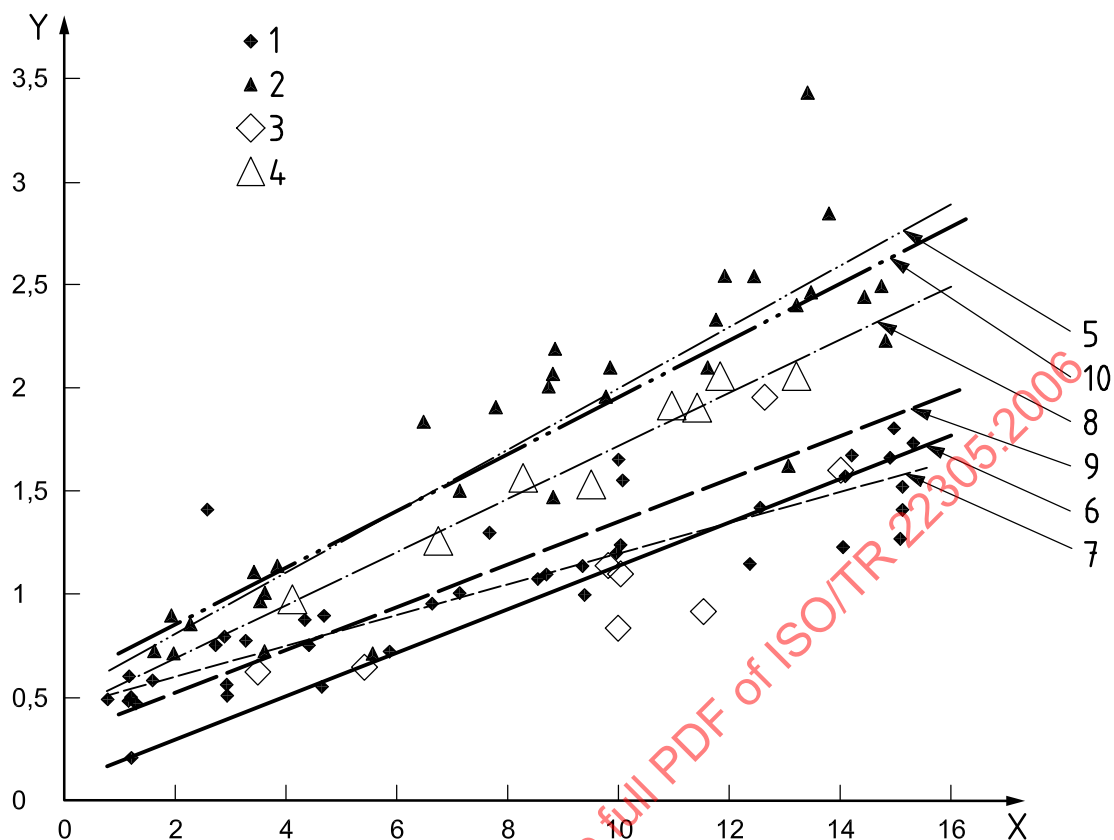


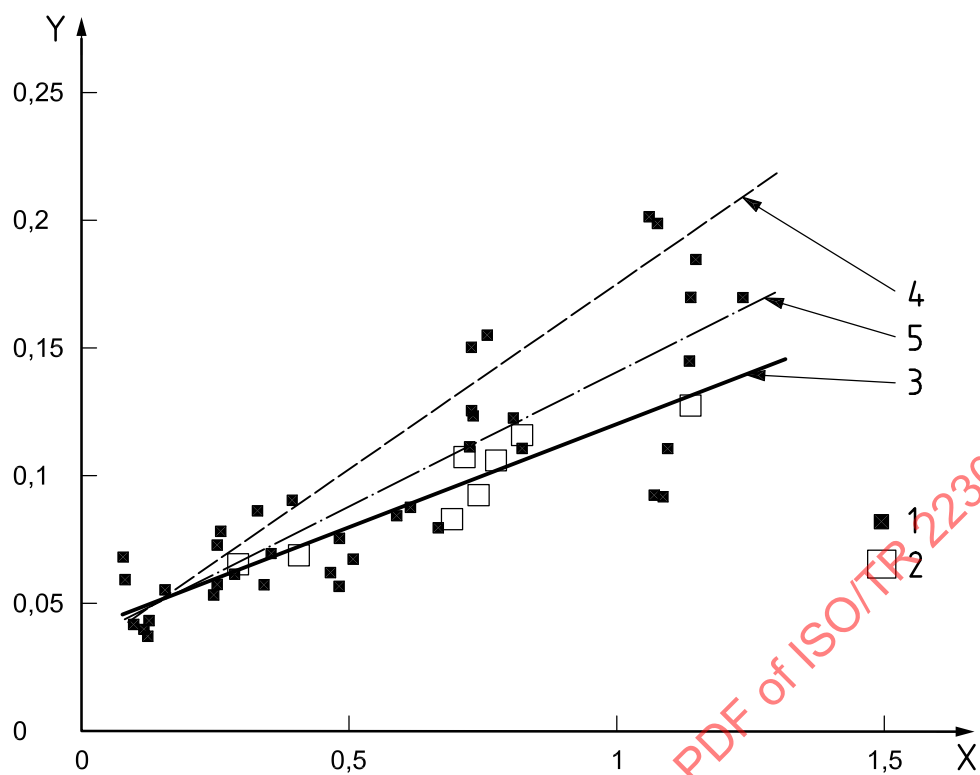
Figure 6 — R_{20} reproducibility values for nicotine (% of yield)

**Key**

- 1 other-NFDPM
- 2 other-CO
- 3 CCS-03-NFDPM
- 4 CCS-03-CO
- 5 linear (other-CO)
- 6 linear (CCS-03-NFDPM)
- 7 linear (other-NFDPM)
- 8 linear (CCS-03-CO)
- 9 UK-NFDPM
- 10 UK-CO

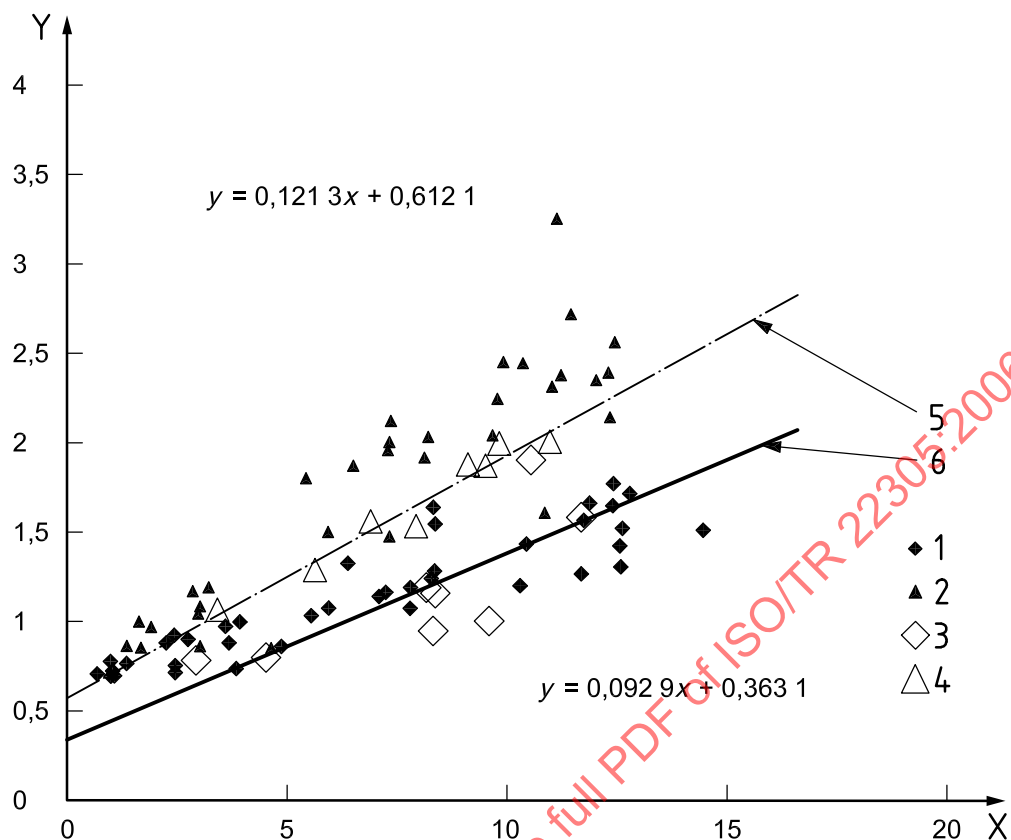
X NFDPM or CO yield, mg
 Y reproducibility R_{100} , mg

Figure 7 — R_{100} reproducibility values for NFDPM and CO

**Key**

- 1 other
- 2 CCS-03
- 3 linear (CCS-03)
- 4 UK
- 5 linear (other)
- X nicotine yield, mg
- Y reproducibility R_{100} , mg

Figure 8 — R_{100} reproducibility values for nicotine

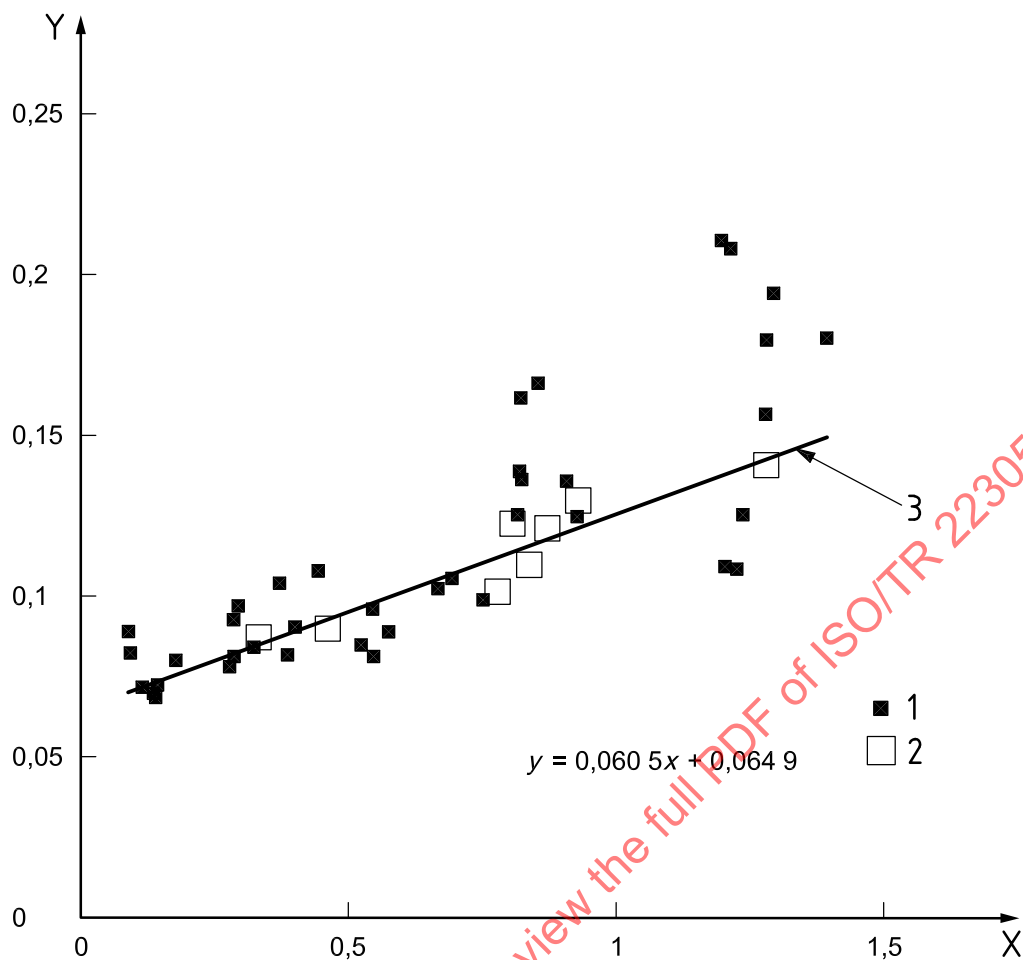
**Key**

- 1 other-NFDPM
- 2 other-CO
- 3 CCS-03-NFDPM
- 4 CCS-03-CO
- 5 linear (CCS-03-CO)
- 6 linear (CCS-03-NFDPM)

X NFDPM or CO yield, mg

Y reproducibility $R_{100+mdg}$ with rounding, mg

Figure 9 — $R_{100+mdg}$ reproducibility values for NFDPM and CO



Key

- 1 other
- 2 CCS-03
- 3 linear (CCS-03)
- X nicotine yield, mg
- Y reproducibility $R_{100+rndg}$ with rounding, mg

Figure 10 — $R_{100+rndg}$ reproducibility values for nicotine

Annex A (informative)

Background considerations on the choice of sampling procedures

A.1 Introduction

It is particularly difficult to recommend a general method of sampling cigarettes. The objective of sampling is, clearly, to provide a representative sample, but the problem arises because the specific purpose for which tests are required affects the recommendation.

A.2 Variability

Variability arises from the methods used to test cigarettes (e.g. see ISO 4387), but there are also appreciable contributions to the variability of the product as cigarette manufacture continues over a period of time. These are reflected in sources of variability described below.

- **Short-term variability** – It is impossible to control the weight of every cigarette precisely. The moisture content of the tobacco varies around its target value. Paper porosity contains similar variability. Tipping materials are also variable. Thus, the design characteristics of the cigarettes being manufactured at any one time vary in a random fashion around their target values, and these variations give rise to corresponding variations in smoke yields.
- **Medium-term variability** – Superimposed on the sources of short-term variability are the sources of medium-term variability such as batch-to-batch changes in materials (paper, tipping, filter papers, filter towels), grade substitutions in the blend, wear of machinery, etc.
- **Long-term variability** – In the long-term there are changes in the blend due to different crop years. Machinery replacement programmes and the upgrading of manufacturing processes can influence the product. Suppliers of non-tobacco materials (papers, tipping, etc.) may change. All these sources of long-term variability are added to both the short- and medium-term contributions.

These terms are described for practical convenience, but it should be remembered that these sources of variability operate as a continuum over time. Experience over numerous years has shown that when attempting to estimate a “true” overall mean (i.e. over all production runs), the contribution to the variability of medium-term effects is larger than that of short-term effects, with the influence of long-term effects being larger than either of these.

For samples taken according to ISO 8243:2003, A.3, the implications are that the 95 % confidence limits (for the mean of the smoke yields) calculated from the sample data reflect only short-term variability. Increasing the size of the sample taken at any one point in time can only reduce the effect of the short-term sources of variability on the precision of the mean of the sample. Thus, the mean of a sample taken at a single timepoint is of limited value in predicting the mean likely to be obtained from any later sample, no matter how big these samples might be. For samples taken according to ISO 8243:2003, A.4, the implications are that the 95 % confidence limits (for the mean of the smoke yields) calculated from the sample data reflect short-term and medium-term variability. In this case increasing the number of sub-period samples reduces the confidence limits. However, unless the sampling period is greatly extended, the calculated confidence limits will still not reflect the long-term variability. Experience of sampling at point of sale (see ISO 8243:2003, A.2) has shown that the data are often of little value. The rotation of stocks in retail outlets is often very poor so that old packs appear for sale on shelves and the conditions for storage are frequently far from ideal.

Determinations on a point of sale sample reflect the smoke yields from the product available to a buyer of that particular increment. However, a gross sample made up from point of sale increments may have a wider inherent variability (than those quoted in ISO 8243:2003, 7.3) arising from unspecified periods of manufacture, and may possibly include cigarettes manufactured before and after intentional design changes.

A.3 Recommendations

These factors lead to the strong recommendation that determinations be made on the population manufactured for sale, sampled at manufacturers' factories or importers' warehouses.

Because of the short-, medium- and long-term variations in cigarette manufacture, it is strongly recommended that the "Sampling over a period of time" procedure (ISO 8243:2003, A.4 and B.4) be used wherever possible.

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Annex B (informative)

The determination of carbon monoxide in cigarette smoke — Problems in the evaluation of results

B.1 Summary

The Directive of the European Parliament and the Council for the Harmonization of Legal and Administrative Regulations of the Member Countries concerning the Manufacture, Presentation/Packaging and Sale of Tobacco Products dated 5th June 2001 stipulates that the carbon monoxide content of cigarettes must be printed on the product pack. The values will be subject to control and monitoring by the food standards authorities. At present two different types of smoking machines, both of which comply with the standards of ISO 3308 are used for this purpose. The data on which the present study is based show a divergence in the values obtained when determining carbon monoxide content. In conclusion, it can be assumed that until the different types of smoking machine are harmonized, a greater range of variation in carbon monoxide content is to be expected in comparison to the determination of condensate.

Keywords: carbon monoxide, cigarette smoke, smoking machine, regulation.

B.2 Introduction

Carbon monoxide is a gas which is ubiquitous in our environment, slightly lighter than air and readily soluble in a number of organic solvents.

Carbon monoxide (CO) and carbon dioxide (CO₂) are the principal constituents of the gas phase of cigarette smoke and are formed through the thermal decomposition and pyrolysis of tobacco constituents (starch, cellulose, sugar, amino acids, etc.).

During the puffing on a cigarette approx. 30 % of carbon monoxide is formed by thermal decomposition and approx. 36 % by pyrolysis of tobacco constituents. Approx. 23 % is formed by the reduction of carbon monoxide at temperatures exceeding 650 °C in the burning cone (*Baker* in the literature^[12]).

The starting point of the present Technical Report is the Directive of the European Parliament and of the Council on the approximation of the laws, regulations and administrative provisions of the Member States concerning the manufacture, presentation and sale of tobacco products of 5th June 2001^[13]. This Directive prescribes that the carbon monoxide content of cigarettes should be printed on the packs, which will be controlled and monitored by the food standards authorities.

A working group was set up at the end of the 1980s to prepare an ISO Standard^[14] designed to harmonize the different standards that exist regarding the smoking machines used worldwide as well as the use of differing smoking machines. The goal of this group was formulated as follows: "to develop one set of standard methods which may be used world-wide, irrespective of smoking machine or other equipment". This had become necessary in order to achieve comparable yields for nicotine and condensate to be printed on packs. Based on an inter-laboratory study comprising 30 laboratories in 15 countries, estimated values for the variabilities were determined for these two parameters^[14].

This led to a great amount of information and experience in dealing with the smoking yields for nicotine and condensate. However this is not the case with respect to the CO yields that must now be printed on the packs.

Smoking machines were developed over a period of some 40 years. The design and construction of the various smoking machines which comply with ISO 3308 are widely divergent in some respects.

There are currently two different types of smoking machines which comply with ISO 3308 in use:

- the linear smoking machine, in which the position of the cigarette and the holder is fixed;
- the rotating smoking machine, in which the cigarette and the holder are movable.

The present Technical Report deals with the issue of CO determination in the mainstream smoke of cigarettes. In principle, however, the considerations presented here also apply to other tobacco products which can be mechanically smoked.

B.3 Methods and data

B.3.1 Method of determining CO

Determination of CO is carried out in three steps. The gas phase of the cigarette is sampled in a special container during the mechanical smoking of cigarettes. The container is either directly connected to a CO analyzer or is separate and can be subsequently connected to the CO analyzer. Carbon monoxide is determined as a percentage (Volume fraction) in the gas mixture in the CO analyzer. The third step is the calculation of the CO yield in milligram per cigarette.

The standard procedure for the determination of CO is to be found in the method stipulated under ISO 8454^[15]. According to this method, CO is determined in the gas phase of cigarette smoke on the basis of the NDIR (non-dispersing infrared analyzer) procedure.

B.3.2 Data used for this study

Data A: DIN/VdC monitor inter-laboratory study

Data B: Annual CORESTA inter-laboratory studies 1995 - 2000 for the monitor cigarettes CM2 and CM3^[16].

Data C: Data from ISO 4387^[5] and ISO 8454^[15]

The data sources for the present Technical Report are the results of comprehensive and extensive national and international inter-laboratory studies.

In addition, the first two data sources (Data A and Data B) are studies which are carried out at intervals of one year. This means that, besides enabling a cross-sectional comparison across the laboratories involved per year, it is also possible to make a longitudinal comparison over time, thus ensuring additional checks on consistency of findings.

B.4 Results

Figure B.1 shows the distribution of 125 CO and 145 condensate measurements for the monitor CM3 obtained in the inter-laboratory study 2000 in 17 laboratories (Data B). It is clearly apparent that the values for the two parameters lie in the same range of approx. 13 to 17 mg/cigarette. Condensate distribution shows a much narrower scattering however ($s = 0,52$) than that for CO ($s = 0,99$). Since there is a good correlation between the CO and condensate measurements across the entire range represented on the market, the question arises as to the reasons for the relatively higher variation in the CO values measured (Figure B.1).

Figure B.2 shows that with respect to data from the data sources B and C in comparison with the results of Figure B.1, higher variations for CO are found across the entire range than for condensate, indicated by the reproducibility R . While the R values for condensate reach a maximum of 1,8 in the upper range for condensate, the corresponding CO values can reach more than $R = 3$. Since the reproducibility R is largely determined by the divergences between the measurements from different laboratories, it can be assumed that the use of different smoking machines in the individual laboratories could be responsible for the higher variation in CO.

In Figure B.3, the results of the inter-laboratory study 2000 for the CORESTA monitor CM3 (Data B) are broken down into data obtained from linear and from rotating smoking machines. It is apparent from Figure B.3 that the values measured when using rotating smoking machines lie considerably above those obtained using linear smoking machines.

In Figure B.4, the CO measurements obtained from linear and rotating smoking machines from data sets A and B are set against each other in a scatter plot. One data point represents the mean values from at least 30 smoking analyses from at least 6 laboratories. The measurements obtained from rotating smoking machines lie substantially above those from linear smoking machines across the entire range from 6 mg to 16 mg. Adjustment by regression reveals through its gradient a value which is on average 8 % higher for the values obtained using a rotating smoking machine.

B.5 Discussion

Figures B.3 and B.4 support the assumption based on Figures B.1 and B.2 that a divergence in yields between linear and rotating smoking machines is responsible for the high variability of CO measurements in inter-laboratory studies. If the variations of the CO values are assessed separately according to the type of machine used, i.e. for rotating machines only and for linear machines only, the variation is found to be roughly comparable to that of condensate values.

According to Article 6 of the EU Directive of 5th June 2001, carbon monoxide content must be reported to the Member Countries by the manufacturers/importers, together with further product information such as condensate and nicotine, categorized for each brand name and type, by 31st December 2002. The Member Countries will then ensure that this data is published in an appropriate form (Figures B.2 and B.3).

As a consequence, governmental control bodies will be obliged to control the CO values submitted by the manufacturers/importers from 31st December 2002 onwards. German monitoring laboratories use rotating smoking machines exclusively. Due to the divergence in values between linear and rotating smoking machines this means that differences may be found between the reported CO values and those measured by the supervising institutions. The following example may serve to illustrate this: the manufacturer/importer has used a linear smoking machine for determining the carbon monoxide value to be printed on the pack. The analytical data for CO show an estimated mean value of 9,2 mg per cigarette and a standard deviation of 0,5. The circles in Figure B.5 demonstrate the possible range of the measured values. In view of the statistical findings, the manufacturer/importer can therefore state 9 mg CO per cigarette for the product.

The control institution will use a rotating smoking machine to check the CO value given on the pack in this case. However, as shown by the evaluation of the data sources mentioned above when using rotating smoking machines the mean value of CO determinations is 8 % higher than when a linear smoking machine is used (Figure B.4). Assuming the same standard deviations as found with linear smoking machines, the fictitious measurements are symbolized by circles in Figure B.5. Four possible values measured when using rotating smoking machines are found to exceed the 9 mg CO per cigarette stated on the pack by a margin ranging from 1,3 mg to more than 2 mg.

The next thing that has to be considered is to what extent the high reproducibility R may affect the overall tolerance for CO.

If an additive model (without correlation of the two tolerance values) is taken as a basis for the interaction between product variability (ISO 8243^[17]) and R , an overall tolerance (depending on the range of values) of approx. 22 % is found at an assumed product variability of 15 %, and an overall tolerance of between 25 % and 26 % at an assumed product variability of 20 %. These overall tolerance values are clearly above those which can be calculated for condensate in comparison.

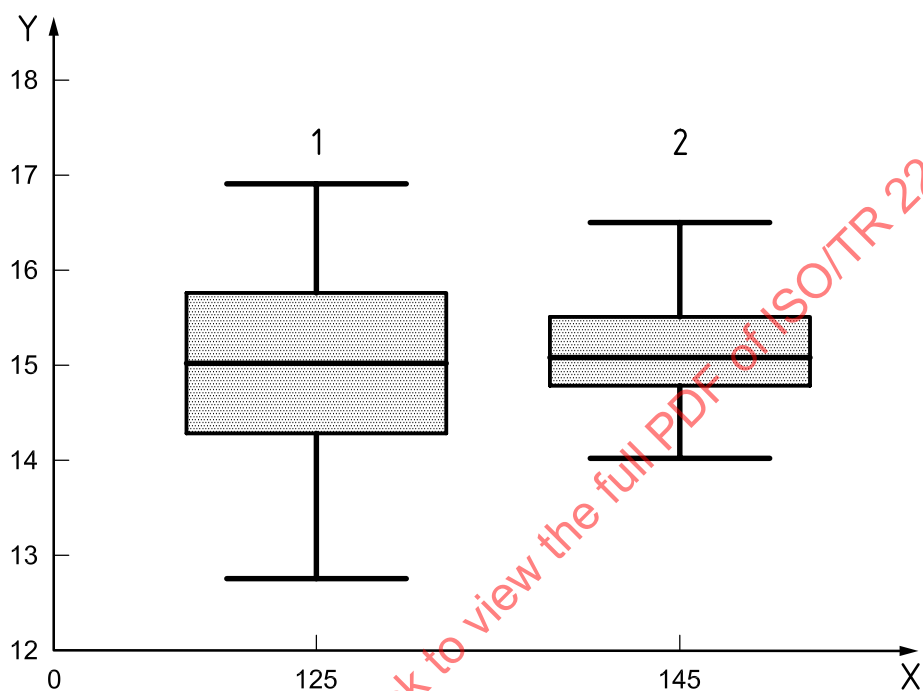
B.6 Conclusions

A formally precise method should permit a scatter range of $> \pm 25$ % for CO. However, in order to ensure that consumer information is not confusing or misleading a scatter range of this order of magnitude should be avoided.

Assuming that during production the entire product variability range is kept as low as possible, it is suggested that a variability range (according to the kind of sampling as per ISO 8243^[17]) of $\pm 20\%$ or $\pm 25\%$ respectively – but not less than 1 mg CO/cig. – should be applied.

In view of the divergence in values in CO determination when using different smoking machine types (linear or rotating type) as demonstrated above, the ISO authority must be called upon to give priority to the harmonization of smoking machines in terms of the quantitative determination of carbon monoxide in smoke¹⁾.

NOTE The authors are indebted to the DIN working group “EU Richtlinie für Tabakprodukte” (EU Directive for tobacco products) for their excellent support and the many valuable discussions.

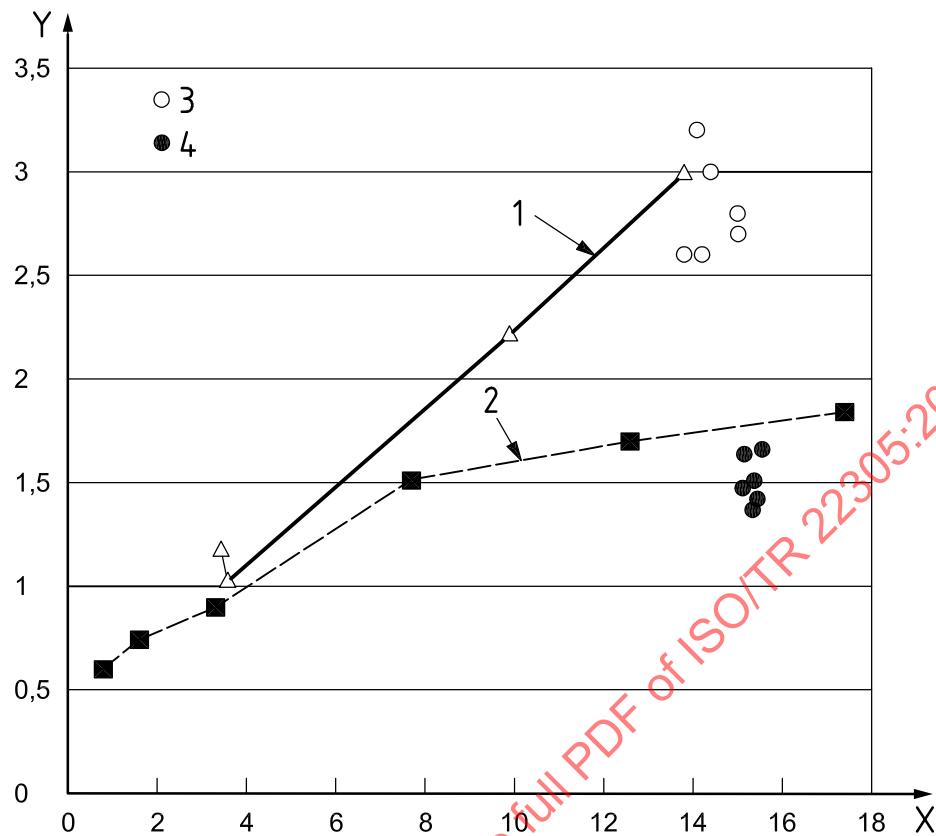


Key

- 1 CO
- 2 condensate
- X *N*
- Y CO/condensate (in mg)

Figure B.1 — Distribution of CO and condensate (Data B - 2000)

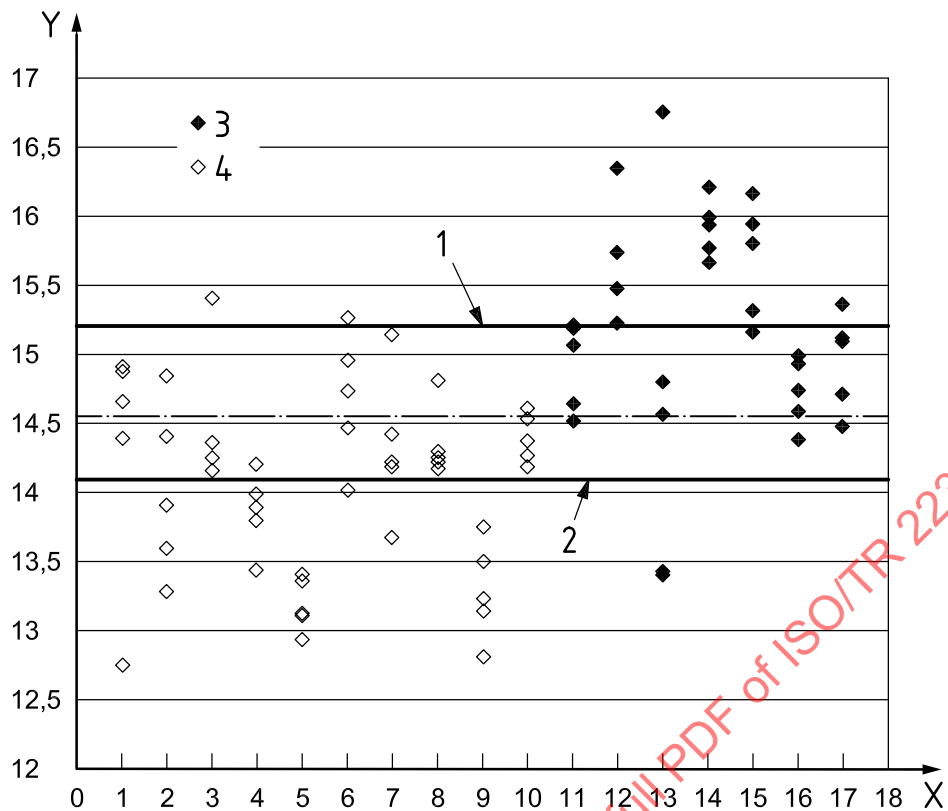
1) During the meeting of ISO/TC 126 on June 19 and 20, 2001 in Berlin it was decided that the ISO/TC 126 will deal with the problems involved in the determination of carbon monoxide in cigarette smoke.

**Key**

- 1 CO (mg/cig): ISO 8454
- 2 condensate (mg/cig): ISO 4387
- 3 CO: CM2/CM3 CORESTA Ring Trial
- 4 condensate: CM2/CM3 CORESTA Ring Trial

X CO/condensate (in mg)
 Y reproducibility

Figure B.2 — R in relation to CO and condensate (Data from B and C)

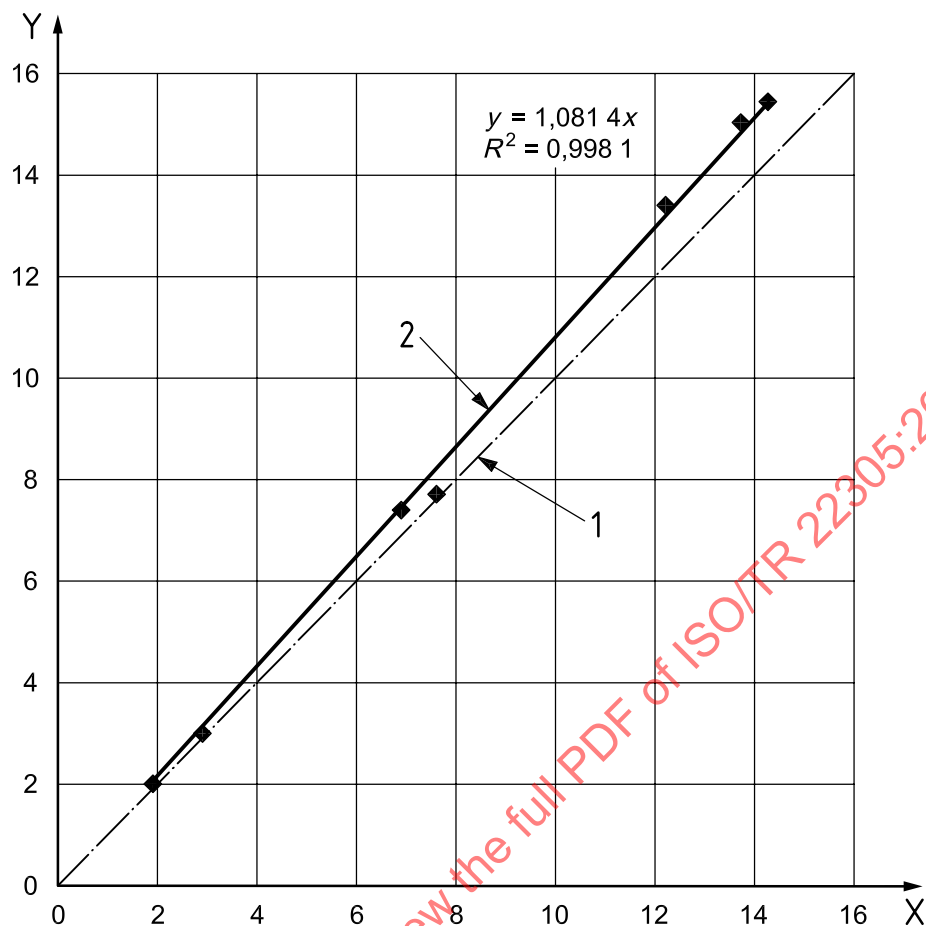


Key

- 1 rotating (mean value)
- 2 linear (mean value)
- 3 rotating
- 4 linear

X lab number
Y CO (in mg/cig)

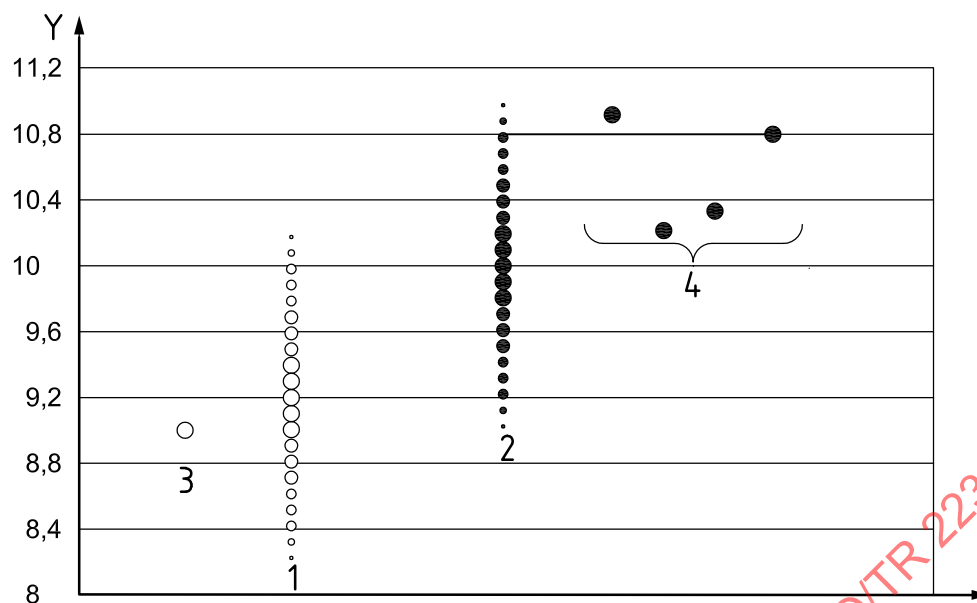
Figure B.3 — Comparison “linear - rotating smoking machines” (Data B - 2000)

**Key**

- 1 diagonal
- 2 regression line

X CO (mg/cig) linear smoking machine
 Y CO (mg/cig) rotating smoking machine

Figure B.4 — CO (mg/cig) linear vs rotating smoking machine (RM20/CSR)



Key

- 1 linear smoking machine
 - 2 rotating smoking machine
 - 3 value on package
 - 4 4 measurements to verify the value on the package
- Y CO (mg/cig)

Figure B.5 — Possible comparison constellations

Annex C (informative)

Proposals from the UK Tobacco Manufacturers Association for a practicable tolerance for verifying cigarette packet declarations of carbon monoxide (March 2002)

C.1 Background

Nicotine free dry particulate matter (NFDPM) and smoke nicotine values have been declared on cigarette packets in EC countries since the early 1990s. In the UK, NFDPM is shown as an integer value, with smoke nicotine rounded to one decimal place. The tolerances specified in ISO 8243 when sampling over a period of time and adopted by the Department of Health/Laboratory of the Government Chemist (LGC) for verifying declared values are $\pm 15\%$, with a minimum of ± 1 mg for NFDPM or ± 0.1 mg for smoke nicotine. The tolerance increases to 20 % when sampling takes place at one point in time. No tolerance is specified for carbon monoxide.

As a consequence of a recent EC Directive (2001/37/EC), cigarette packets will have to include carbon monoxide values (rounded to the nearest integer in most member states); this is to be introduced at the end of 2002. It is also relevant to note that in 2004, NFDPM and carbon monoxide declarations must not exceed 10 mg, with an upper limit of 1 mg for smoke nicotine.

In anticipation of these changes, a statistical study of recent smoke yield data was carried out with the main aim of estimating a practicable tolerance for verifying on-pack values of carbon monoxide. A re-appraisal of NFDPM and smoke nicotine tolerances was also carried out.

A summary of the main findings from this study is given below, preceded by a brief description of the basis of statistical analysis. Although much of the data used in the analysis is from UK Department of Health (DoH) Cigarette Surveys, data from the ISO methods for NFDPM, smoke nicotine and carbon monoxide are also key to the analysis. The findings are considered to be relevant on an international basis and not limited to the UK.

C.2 Basis of statistical analysis

There are two major sources of statistical variation in cigarette smoke yields – the laboratory measurement (analytical) and the product itself. These two sources can be further divided into time and place dependent components. That is, both the product and the measurements made on it fluctuate over time (both in the short and longer term), whilst analytical measurements also vary between laboratories, even for matched samples of cigarettes.

In the context of packet labelling, cigarette samples from production are laboratory-tested by a manufacturer and rounded smoke yield averages determined; it should be noted that rounding also adds to the overall variation in the declared values. Under regulations for most EU states, including the UK, NFDPM is rounded to the nearest 1 mg and smoke nicotine to 0.1 mg. Verification of the packet declarations by a regulatory laboratory such as the LGC can only occur at a later date, after the manufacturer has determined the yield and printed it on the packet. The interval between the determination of the declared values and their independent verification will, therefore, always cover a period of many months. Clearly, in such a comparison of results (e.g. 'Rounded Manufacturer Average – LGC Mean from later production'), the statistical variation includes both product and analytical components, for both the short and longer periods of time, and involves separate laboratories.

In the UK, the DoH Cigarette Survey procedure forms the basis for verifying the packet declarations. Each Survey takes place over a calendar year and consists of bi-monthly sampling (from the factory or importer's warehouse) and laboratory testing over a 12-month period, with four channels of five cigarettes being smoked per sampling occasion. The official assessment of packet declarations compared with the LGC averages is carried out after the completion of each Survey and is based upon the average LGC value for at least five occasions of sampling and testing (ISO 8243 sets a minimum requirement of five sampling periods).

Concurrent with the DoH Survey, each manufacturer carries out its own testing according to the same format and on 'matched' samples from the same 200-outers. These data serve two purposes. They are regularly compared with corresponding data from the LGC as a check on laboratory biases. They are also used to monitor the ongoing performance of individual brands relative to the NFDPM and smoke nicotine yields declared on the packet.

The statistical problem, therefore, is how to quantify this variation in the measured smoke yields and represent it in the form of tolerances such that violations (due to uncontrollable sources) occur very infrequently, that is, on 5 % of occasions. To this end, four different approaches were taken.

- a) **Approaches 1 and 2** were based on simple modelling of the components of variation noted earlier. For both, it was assumed that the laboratories (chosen at random from the population of laboratories used by potential manufacturers/importers) use standard ISO methods and procedures.

The analytical variability between laboratories on one occasion in time, and the short term product and analytical variation within a laboratory, were obtained from the values of reproducibility (R) and repeatability (r) given in the International Standards ISO 4387 (NFDPM), ISO 10315 (Smoke nicotine) and ISO 8454 (Carbon monoxide). These values, based upon data from international collaborative studies of 30 laboratories, must be considered to be both robust and generally applicable.

Approach 1 was simply to note the R values from the ISO standards. These values represent 95 % confidence limits on the difference between mean values obtained by two laboratories for the same brand produced on one occasion in time and tested under repeatability conditions. This approach did not, of course, include all of the product related components of variation, most notably those over longer periods of time. Nevertheless, it was considered that this would provide an initial indication of the tolerances required for carbon monoxide relative to NFDPM and smoke nicotine.

In order to gain a better representation of the practical situation in which many and varied brands are manufactured and laboratory tested over a period of time, Approach 2 extended the model to include additional components of product variation not included in the ISO R values. In particular, components were included to represent product variation over time and the interaction between measurements of different brands by separate laboratories. These estimates were provided by a statistical analysis of LGC and manufacturers' data for 44 brands from the DoH Survey for which the declared NFDPM and Nicotine yields were unchanged within a survey. Subsets of these data were analysed so that the product variation across surveys could be estimated without being invalidated by the effects of 'major' specification changes.

In general there are two reasons for changing the specification of a brand. Most frequently, 'minor' specification changes are made to counter uncontrollable variation in materials and manufacturing and maintain the declared NFDPM and Nicotine yields; it is noted that these changes must reduce the effect of the inherent product variability on smoke yields. 'Major' changes to alter the declared NFDPM and Nicotine yields are made much less frequently.

This extended model also assumed the UK situation such that a packet declaration is compared with the LGC unrounded average from each 12-month Survey which follows its introduction or ongoing use. An allowance was therefore made for the additional variability due to the rounding of the manufacturers' averages.

- b) **Approaches 3 and 4** were more direct but may not fully reflect the population of laboratories (implicit in the ISO estimates of r and R) that could be used by potential manufacturers/importers.

Approach 3 was based on end of survey summaries provided by the LGC, and the differences 'Packet Declaration – LGC Survey Average' across UK selling brands for Surveys 39 to 44 (1995 to 2000) were studied. Where declared NFDPM or Nicotine yields changed within a Survey, the data for these brands were excluded from the analysis. This resulted in a total of 537 comparisons across the six surveys. It is noted that, in the absence of on-pack declarations of carbon monoxide, this could only be done for NFDPM and smoke nicotine.

In order to provide comparable information for carbon monoxide, **Approach 4** attempted to mimic the above by defining declared values (for all three analytes) on the basis of the manufacturers' rounded averages for the period of a survey. This was done for individual brands and, similar to above but across consecutive pairs of surveys, the differences 'Packet Declaration (mimicked for Survey n) – LGC Average (for Survey $n + 1$)' were compared for $n = 39$ to 43. These comparisons were restricted to those brands for which the declared NFDPM and smoke nicotine values were unchanged for the duration of any two consecutive surveys under study; this resulted in a total of 334 comparisons.

For both of these approaches, it was of interest to note the percentages of 'violations' with respect to the present tolerances for NFDPM and smoke nicotine. For comparison purposes, under Approach 4, this was also done for carbon monoxide using the ISO tolerance for NFDPM.

C.3 Data analysed

The R and r values from the International Standards ISO 4387 (NFDPM), ISO 10315 (Smoke nicotine) and ISO 8454 (Carbon monoxide), used in Approach 1 are given in Table C.8.

It is important to note that the data analysed from the DoH Surveys were received from, and validated by, the LGC. This included confirmation of the manufacturers' data, together with the declared packet values of NFDPM and smoke nicotine. It should also be noted that any data-selection carried out to ensure that 'major' specification changes would not invalidate the analysis was based upon LGC guidance on changes to packet declared yields of NFDPM and smoke nicotine.

C.4 Main results

Approach 1 – Direct comparison of ISO reproducibility (R) values

Based on the R values taken from the ISO Standards referenced earlier (see Table C.8), the following values were calculated after fitting straight line regression equations (see Figure C.1).

Table C.1 — 95 % Confidence limits

Mean yield mg	NFDPM		Smoke nicotine		Carbon monoxide	
	mg	%	mg	%	mg	%
2	0,8	41	0,07	36	0,8	41
4	1,0	24	0,09	23	1,2	30
6	1,1	19	0,11	19	1,6	26
8	1,3	16	0,13	17	1,9	24
10	1,4	14	0,15	15	2,3	23
12	1,6	13	0,17	14	2,7	22

(For smoke nicotine, the 'Mean yield' in Table C.1 should be divided by 10.)

It is noted from this table that whereas there is a very close agreement in the confidence limits expressed as a percentage of the mean yield for NFDPM and smoke nicotine, the corresponding values for carbon monoxide are appreciably higher for yields above 4 mg/cig (see Figure C.2). Quantitatively, in the region 6 to 12 mg/cig for NFDPM, the carbon monoxide confidence limits as a percentage of the mean yield are of the order of 25 % compared with about 15 % for NFDPM and smoke nicotine.

Given that the present tolerance for NFDPM (i.e. 15 %) has worked successfully in the UK, a corresponding effective tolerance for carbon monoxide would need to be of the order of 25 %.

Approach 2 – Estimation of tolerances from an extended statistical model

The following table shows the confidence limits estimated from the statistical model outlined earlier and detailed in C.7.1. The brands were grouped in bandings of 0 mg to 6 mg and ≥ 7 mg for NFDPM and carbon monoxide, and 0 mg to 0,6 mg and $\geq 0,7$ mg for smoke nicotine. These classifications were based on rounded individual-brand averages (manufacturers' measurements) over the six surveys and chosen to correspond with the break points between 15 % and 0,1 mg (Smoke nicotine) and 15 % and 1 mg (NFDPM).

Table C.2 — 95 % Confidence limits

NFDPM			Smoke nicotine			Carbon monoxide		
Mean mg	No. Brands	95 % CL mg	Mean mg	No. Brands	95 % CL mg	Mean mg	No. Brands	95 % CL mg
4,7	10	1,2	0,47	11	0,11	4,3	7	1,4
10,8	34	1,6	0,91	33	0,14	10,8	37	2,6

NOTE In the statistical model underlying these results, a major factor was the assumption that measurement variability by both the LGC and the manufacturer should be included. It is important to acknowledge that discussions with the LGC had raised a query about this assumption; the suggestion being that the variability associated with the manufacturers' measurements ceases to be relevant once a declared packet value has been established. This is discussed further in C.5.

Applying the present ISO tolerances to the DoH Survey data used in the extended model for NFDPM (also applied to carbon monoxide) and smoke nicotine, the following overall percentage-violations were obtained.

Table C.3 — Violations (%)

NFDPM	Smoke nicotine	Carbon monoxide
5,1	5,4	19,5

It is noted that whilst these results indicate percentage-violations close to 5 % for both NFDPM and smoke nicotine, the figure for carbon monoxide is much higher. A comparable percentage for carbon monoxide (4,5 %) is obtained by applying a tolerance of ± 25 %. Further discussion on what tolerances would be practicable, for NFDPM and smoke nicotine as well as for carbon monoxide, is given later.

Approaches 3 and 4 – Observed and predicted violations of ISO tolerances based on DoH Survey data

From **Approach 3** described in C.2 b) above, Figures C.3 and C.4 (NFDPM and smoke nicotine respectively) show the differences for individual brands between the LGC measurements (survey averages) and the values declared on packets by the manufacturers.

From **Approach 4** also described in C.2 b) above, Figures C.5, C.6 and C.7 (NFDPM, smoke nicotine and carbon monoxide respectively) show the differences for individual brands between the LGC measurements (survey averages) and the manufacturers' measurements (rounded survey averages) from the previous survey.

Figures C.3 to C.6 also show the individual differences relative to the present ISO tolerances for NFDPM and smoke nicotine. Figure C.7 shows the individual differences for carbon monoxide when applying the ISO tolerance specified for NFDPM. A summary of the overall percentages of brands exceeding these tolerances is given in Table C.4.

Table C.4 — Violations of tolerances (%)

	NFDPM	Smoke nicotine	Carbon monoxide
<u>Approach 3</u> 'Packet Declaration-LGC Survey Average'	3,7	11,4	NA
<u>Approach 4</u> Mimic of 'Packet Declaration – LGC Average'	4,5	10,5	10,2

(Where "NA" denotes not available – the survey data analysed covers the period 1995 to 2000 and carbon monoxide was not declared on packets until 2003.)

These results indicate the following:

- a close agreement between Approaches 3 and 4 for NFDPM and smoke nicotine, a comparison not being possible for carbon monoxide: since Approach 4 makes comparisons directly with manufacturers' measurements, this finding confirms that, in general, the violations are not due to falsely declared values;
- a reasonable agreement between the above figures for NFDPM and those obtained using the statistical model (i.e. Table C.3).

A much higher percentage of violations of the tolerance for smoke nicotine compared with that indicated by Table C.3 (more than double). This would seem to suggest a case for widening the product tolerance of $\pm 15\%$ (minimum 0,1 mg) for smoke nicotine. A possible explanation for the inconsistency is a long-term measurement bias between the LGC and all manufacturers' laboratories. This was checked for Approach 3 (see C.7.2) for NFDPM and smoke nicotine and, although violations were reduced for smoke nicotine (11,4 % to 9,1 %), it was insufficient to bring it in line with the statistical model. Another suggested explanation is that this may stem, at least in part, from NFDPM having been traditionally regarded as the primary control variable. That is, 'minor' amendments to brand specifications in order to maintain NFDPM declared values could lead to unintended secondary effects to smoke nicotine. It is also noted that such secondary effects on carbon monoxide could differ since it is a component of the vapour phase of cigarette smoke rather than a particulate component as with both NFDPM and smoke nicotine.

A substantially lower percentage of violations of the ISO NFDPM tolerance when applied to carbon monoxide compared with that shown by the model approach. Nevertheless, the 10,2 % of occasions for which the laboratory differences exceeded the $\pm 15\%$ tolerance is more than double what would be practicable.

These results not only reiterate the need for a wider tolerance than $\pm 15\%$ for carbon monoxide, but also suggest a case for increasing the existing tolerance for smoke nicotine. Further discussion on this is given in the following section.

C.5 Further discussion

It was noted in C.4 that discussions with the LGC about the formulation of a statistical model and access to the required DoH Survey data had raised some doubt about the suggested model as finalised in the current Approach 2. In particular, it was suggested that no allowance should be made for the statistical variability in the manufacturers' measurements since, once a smoke yield is declared (i.e. on the cigarette packet) it is 'fixed' and 'error-free'.

However, this does not seem reasonable since declared on-pack values have been based upon prior measurements by a manufacturer's laboratory and are therefore subject to measurement 'error' and variability in the product itself; that is, the declared values merely represent a manufacturer's estimates. Furthermore, the ongoing maintenance of a brand at the intended smoke yield requires continual specifications-adjustment to compensate for variations from many sources. These are recognised and described in Annex A of this Technical Report together with their potential impact on measurement variability. Again, the manufacturer's laboratory results are relevant since any checks made on the effects of any 'tweaking' of a brand's specification will be clouded by the imprecise nature of the product and its smoke yield measurements. The practical difficulties of maintaining a brand are further compounded when production occurs at more than one factory.

For the record, if the model did not make allowance for 'error' in the manufacturer's measurements, or for the variability associated with the product, the following percentage-violations would be predicted by the extended model when applying the existing ISO tolerances (NFDPM tolerance applied to CO).

Table C.5 — Violations (%)

NFDPM	Smoke nicotine	Carbon monoxide
1	1	8

Clearly all three figures in this table grossly underestimate the percentage-violations as indicated by, not only Table C.3 which included in the model an allowance for the manufacturers' measurements, but also Table C.4 which reflected the practical experience in the UK during recent years. Even these figures indicate that a much higher tolerance is needed to align the percentage-violations for carbon monoxide with NFDPM and smoke nicotine.

In addition to the above, the effect of making the dual-comparison was not considered when the tolerances were originally set for NFDPM and smoke nicotine. That is, the tolerances of $\pm 15\%$ (minimum ± 1 mg and $\pm 0,1$ mg) were set such that 5 % of violations due to each of NFDPM and smoke nicotine would be expected to occur purely due to chance variability. Inevitably, the consequence of this is that more than 5 % violations will occur (by 'chance') per brand due either to NFDPM and/or smoke nicotine unless differences in measurements between two laboratories are perfectly correlated. It should be noted that, although the smoking process is common, subsequent analytical procedures for the three analytes are completely independent. It is not unreasonable, therefore, to expect inter laboratory differences to be independent, and this is clearly illustrated by the poor correlations between the NFDPM and smoke nicotine biases, and NFDPM and carbon monoxide biases, in Figures C.8 and C.9 respectively.

If, therefore, it is intended that no more than a 5 % violation rate (due to chance variability) should occur on a per-brand basis, it is clear that the individual tolerances for NFDPM and smoke nicotine (and in the future carbon monoxide) would need to be widened. That is, based on the results from Approach 4, whereas the average violation-rate for the individual analytes is 8,3 % (see Table C.4), this is more than doubled to 19,5 % as determined by any one or more of the three analytes exceeding their individual tolerances.

When considered in this way, even allowing for a proportion of the observed violations being 'real', there would seem to be a case for adopting tolerances of $\pm 15\%$ (NFDPM), $\pm 20\%$ (Smoke nicotine) and $\pm 25\%$ (Carbon monoxide). Applying these limits to the results under Approach 4 indicates an overall brand-violation rate of approximately 8 %.

C.6 Conclusions / Recommendations

The longer term objective is to provide relevant input for the review and updating of ISO 8243 by including a tolerance for carbon monoxide. The above statistical analysis and discussion has indicated the practical need to set a tolerance of the order of $\pm 25\%$ for the verification of carbon monoxide, with a minimum of ± 1 mg.

The current ISO tolerance for NFDPM has been found to be appropriate, but the same analysis indicates the tolerance for smoke nicotine would more appropriately be $\pm 20\%$.

It should be noted that the $\pm 15\%$ tolerances specified in ISO 8243 for NFDPM and smoke nicotine did not allow for joint comparisons. Clearly, if a sensible and workable arrangement is to be reached with regulators it is crucial to clarify the issue of multiple comparisons. In particular, should the 5 % failure-rate be set on the basis of individual brand or individual brand and analyte?

It is clear that the extended statistical model should be based upon the comparison of data from a regulator's laboratory with that from the manufacturer's laboratory, rather than a fixed declared value.

C.7 Additional data

C.7.1 Statistical model

$$95\% \text{ CL} = \pm 2 \left\{ 2 \left[(P + \text{Residual}/4) / 6 + L + P_L \right] + \text{Rounding} \right\}^{1/2}$$

where

P	is the variance due to product variability over time;
L	is 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;
<i>Residual</i>	is the variance to the basis of 5 cigarettes;
<i>Rounding</i>	is the variance associated with rounding tar to 1 mg and nicotine to 0,1 mg and is 0,083 for tar and 0,00083 for nicotine.

Source of data for each variance:

- P from joint LGC/Manufacturer's Survey data;
- L from reproducibility values in ISO methods;
- P_L from joint LGC/Manufacturers' Survey data;
- *Residual* from repeatability values in ISO methods;
- *Rounding* from a standard function.

NOTE In the above model, it is assumed that the Survey averages are based upon the measurement of 4 replicate determinations, each of 5 cigarettes, on 6 separate occasions.

C.7.2 Adjustment of data used in Approach 3 for long-term LGC bias

The measurement bias was calculated for the brands used in Approach 3 for NFDPM and smoke nicotine. The bias for all the matched brands in the DoH Surveys, as reported by the LGC, is also included in the table below for comparison. There is little difference between the two biases, indicating that the brands used in Approach 3 are a reasonably representative sample.

Table C.6 — Measurement bias (Manufacturer-LGC)

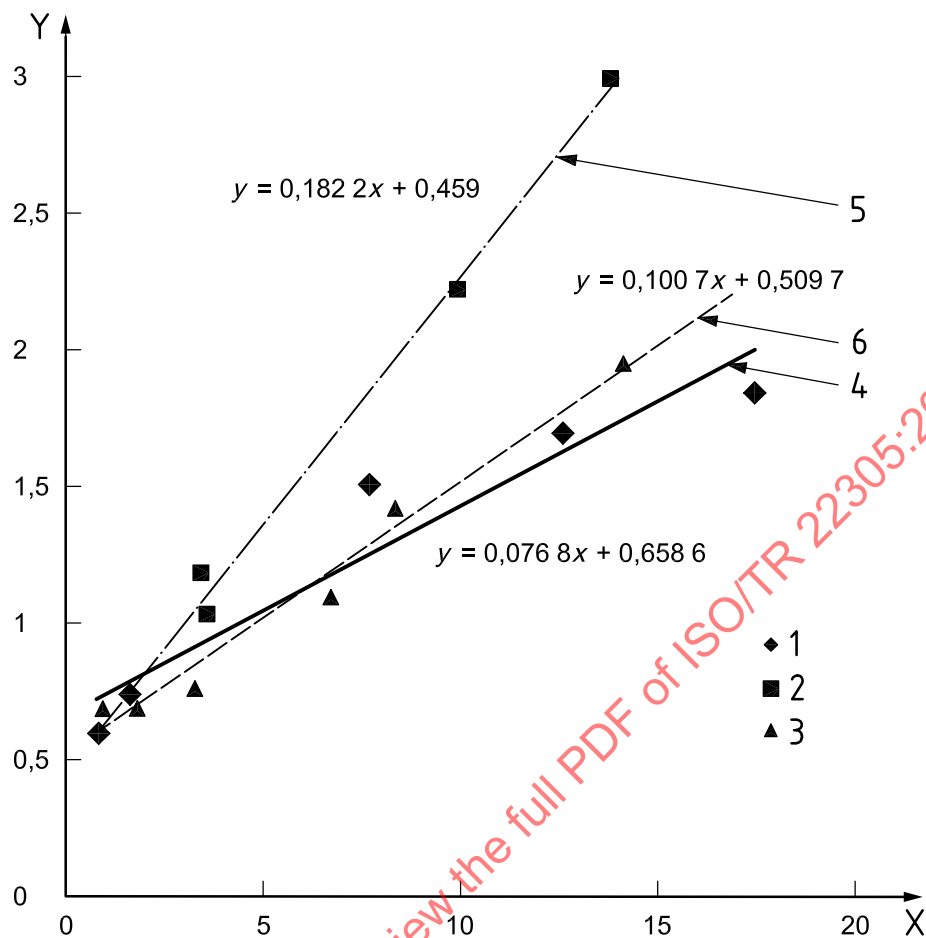
Survey	Approach 3 Brands		All matched brands	
	NFDPM (mg)	Nicotine (mg)	NFDPM (mg)	Nicotine (mg)
39	– 0,151	+ 0,038	– 0,17	+ 0,03
40	– 0,261	+ 0,026	– 0,25	+ 0,03
41	– 0,198	+ 0,033	– 0,19	+ 0,03
42	– 0,256	+ 0,020	– 0,27	+ 0,02
43	– 0,327	– 0,007	– 0,33	– 0,01
44	– 0,016	– 0,014	– 0,02	– 0,01
Mean	– 0,202	+ 0,016	– 0,205	+ 0,015

For each brand, the previously calculated difference between the manufacturer's declared value and the LGC measured value was corrected using the above biases on an individual survey basis. The number of brands outside the ISO tolerances for NFDPM and smoke nicotine were then estimated and are given in Table C.7.

Table C.7 — Violations of ISO tolerances

		Before bias adjustment		After bias adjustment	
		Number	%	Number	%
NFDPM	— Total	20	3,7	20	3,7
	— Positive	8	1,5	13	2,4
	— Negative	12	2,2	7	1,3
Nicotine	— Total	61	11,4	49	9,1
	— Positive	51	9,5	27	5,0
	— Negative	10	1,9	22	4,1

It is noted that the bias adjustment makes no difference to the total violations for NFDPM and has little effect on the positive/negative split. In comparison the total violations for smoke nicotine are reduced by 2,3 % and the positive/negative split is much better balanced.

**Key**

1 NFDPM

2 CO

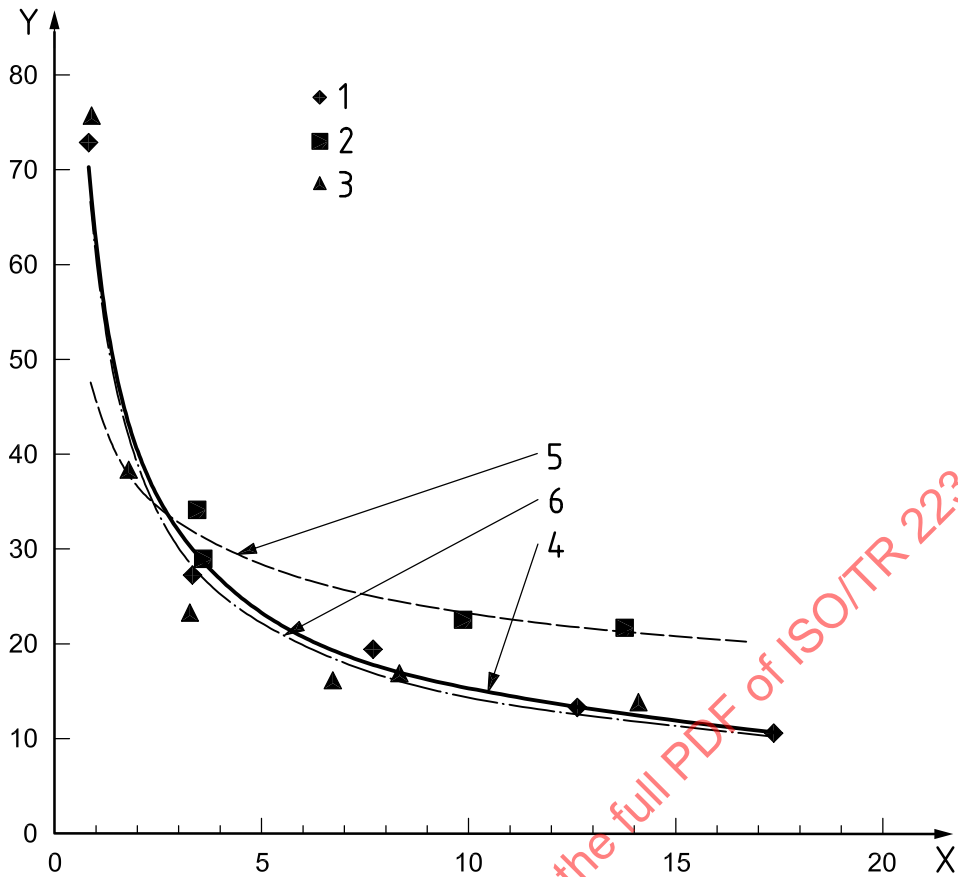
3 nicotine

4 linear (NFDPM)

5 linear (CO)

6 linear (nicotine)

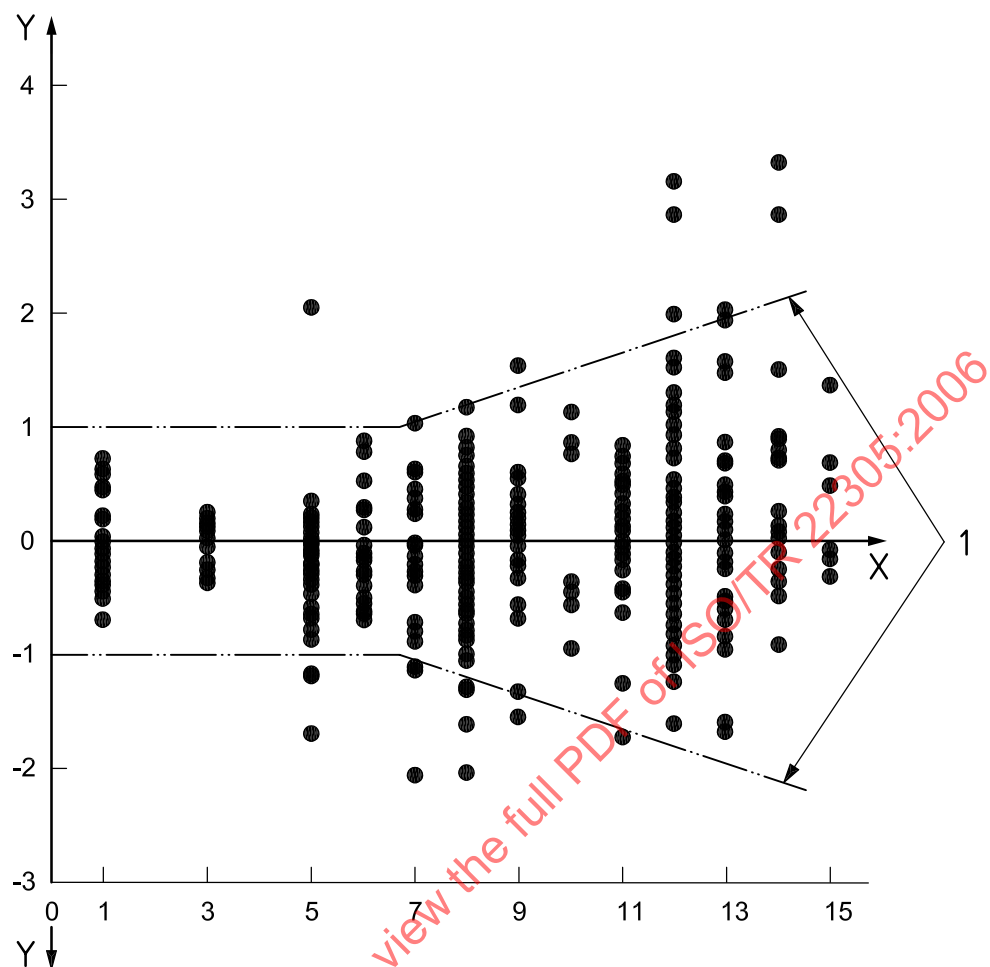
X yield ($\times 10$ for nicotine) mgY R ($\times 10$ for nicotine) mg**Figure C.1 — ISO reproducibility R**



- Key
- 1 NFDPM
 - 2 CO
 - 3 nicotine
 - 4 power (NFDPM)
 - 5 power (CO)
 - 6 power (nicotine)

X yield ($\times 10$ for nicotine) mg
Y $(R/Yield) \times 100$ %

Figure C.2 — ISO reproducibility R (% of yield)

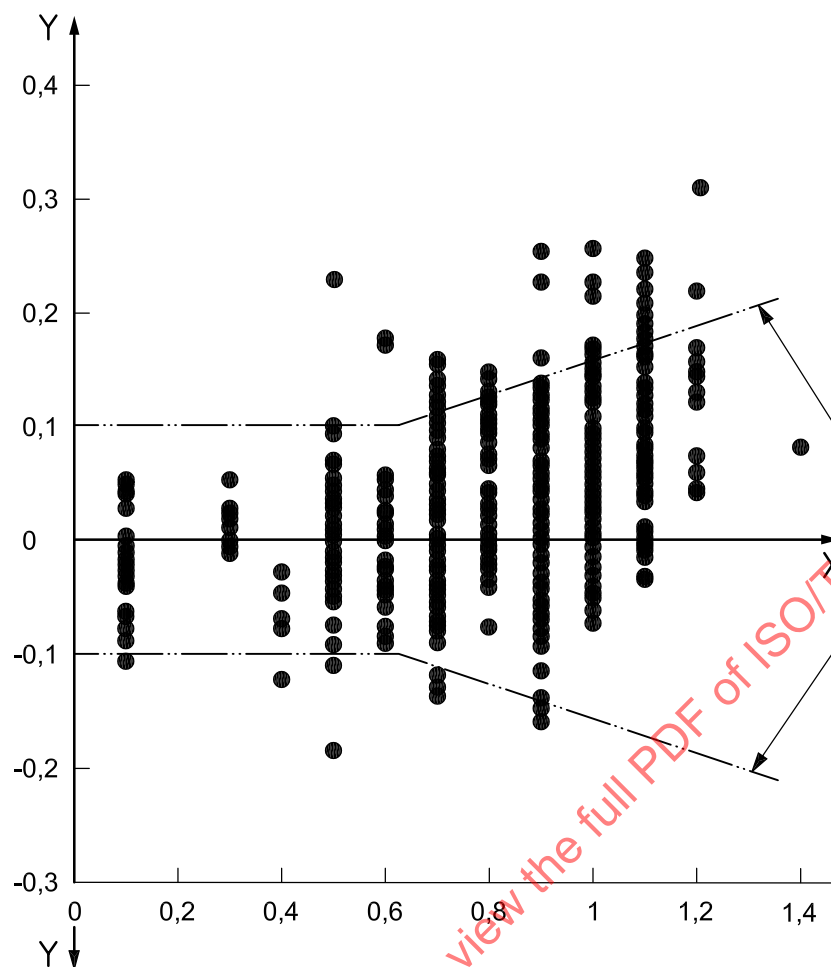
**Key**

1 ISO tolerance

X declared NFDPM (mg)

Y declared-LGC NFDPM (mg)

Figure C.3 — Difference between declared and LGC NFDPM



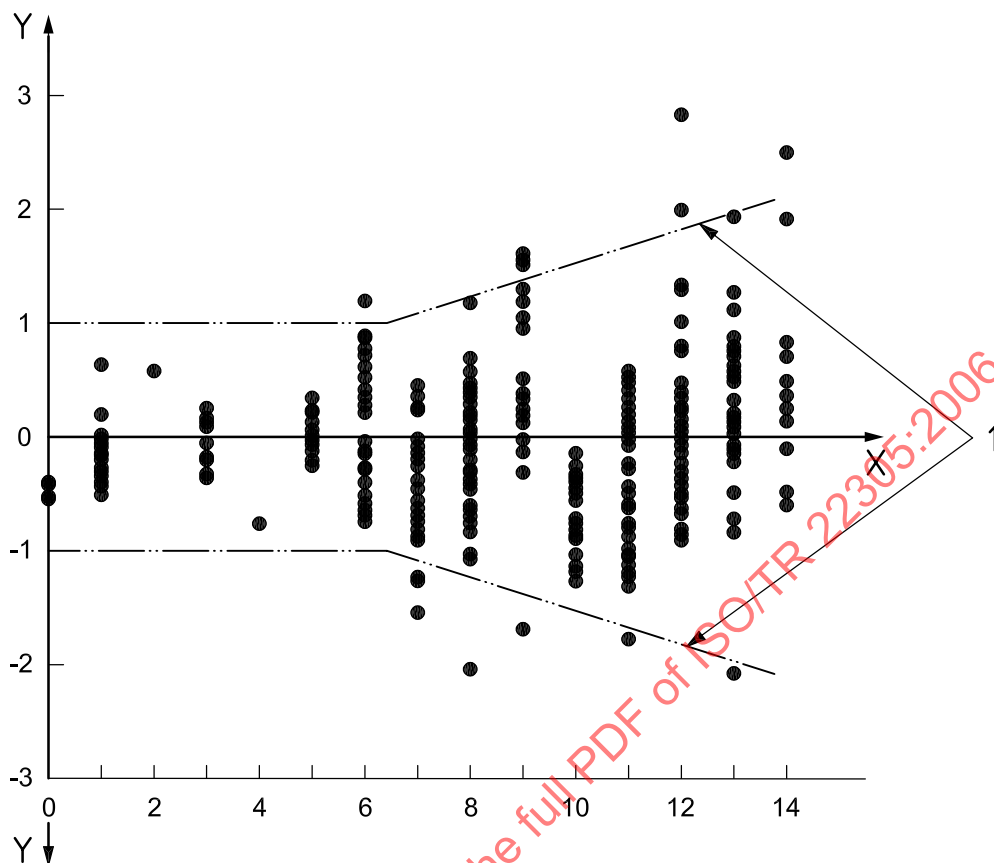
Key

1 tolerance

X declared nicotine (mg)

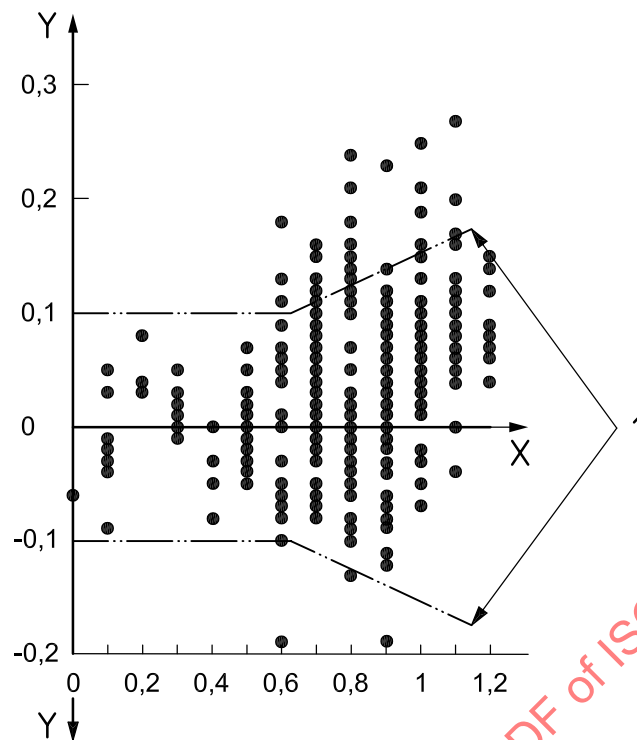
Y declared-LGC nicotine (mg)

Figure C.4 — Difference between declared and LGC nicotine

**Key**

- 1 tolerance
- X manufacturer rounded NFDPM for survey n (mg)
- Y manufacturer rounded mean for survey n (mg) – LGC NFDPM for survey n+1 (mg)

Figure C.5 — NFDPM
Manufacturer rounded mean (Survey n)-LGC mean (Survey n+1)



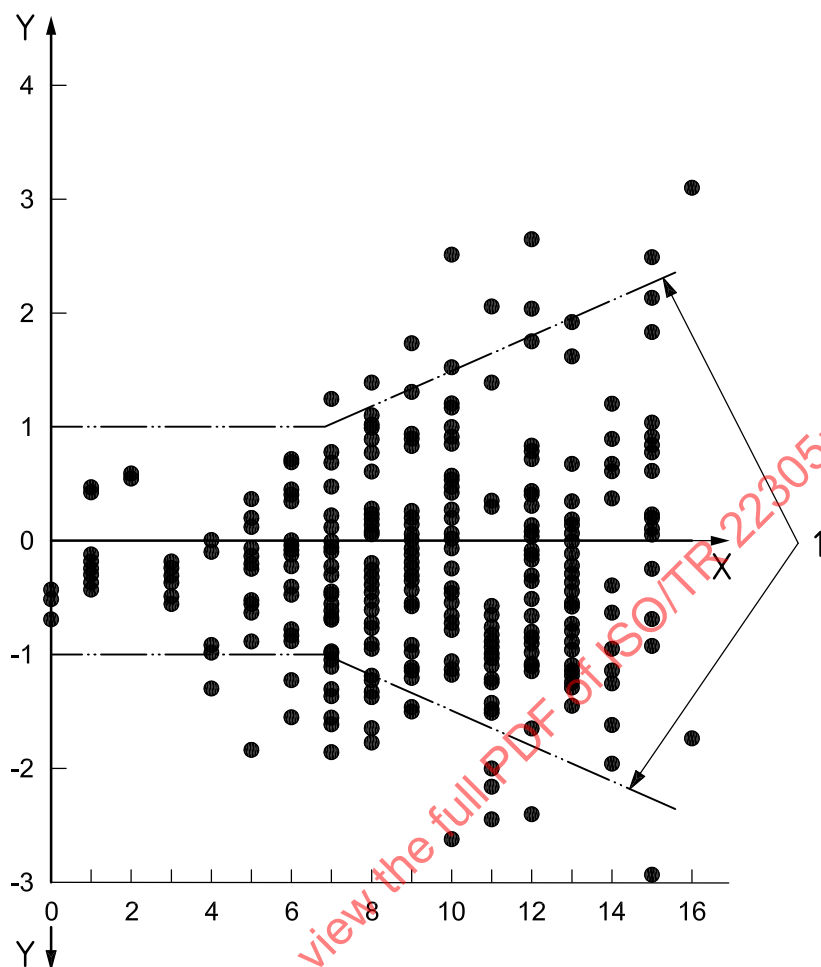
Key

1 tolerance

X manufacturer rounded nicotine for survey n (mg)

Y manufacturer rounded mean for survey n-LGC nicotine for survey n+1 (mg)

Figure C.6 — Nicotine
Manufacturer rounded mean (Survey n)-LGC mean (Survey n+1)

**Key**

1 tolerance

X manufacturer rounded CO for survey n (mg)

Y manufacturer rounded mean for survey n-LGC CO for survey n+1 (mg)

Figure C.7 — Carbon monoxide
Manufacturer rounded mean (Survey n)-LGC mean (Survey n+1)

Table C.8 — ISO repeatability and reproducibility values for NFDPM, smoke nicotine and carbon monoxide

NFDPM values from ISO 4387		
Yield mg	Repeatability	Reproducibility
0,82	0,40	0,60
1,61	0,52	0,74
3,31	0,52	0,90
7,70	0,88	1,51
12,61	1,06	1,70
17,40	1,19	1,84
Smoke nicotine values from ISO 10315		
Yield mg	Repeatability	Reproducibility
0,091	0,040	0,069
0,179	0,046	0,069
0,326	0,050	0,076
0,673	0,077	0,109
0,835	0,079	0,142
1,412	0,107	0,195
Carbon monoxide values from ISO 8454		
Yield mg	Repeatability	Reproducibility
3,45	0,47	1,18
3,56	0,42	1,03
9,89	0,85	2,22
13,80	1,09	3,00

Repeatability (r) = $2,8 \sigma_r$

Reproducibility (R) = $2,8 \sqrt{(\sigma_L^2 + \sigma_r^2)}$

where

σ_r^2 is within laboratory variance, and

σ_L^2 is between laboratory variance.

NOTE All values are based upon testing 20 cigarettes.

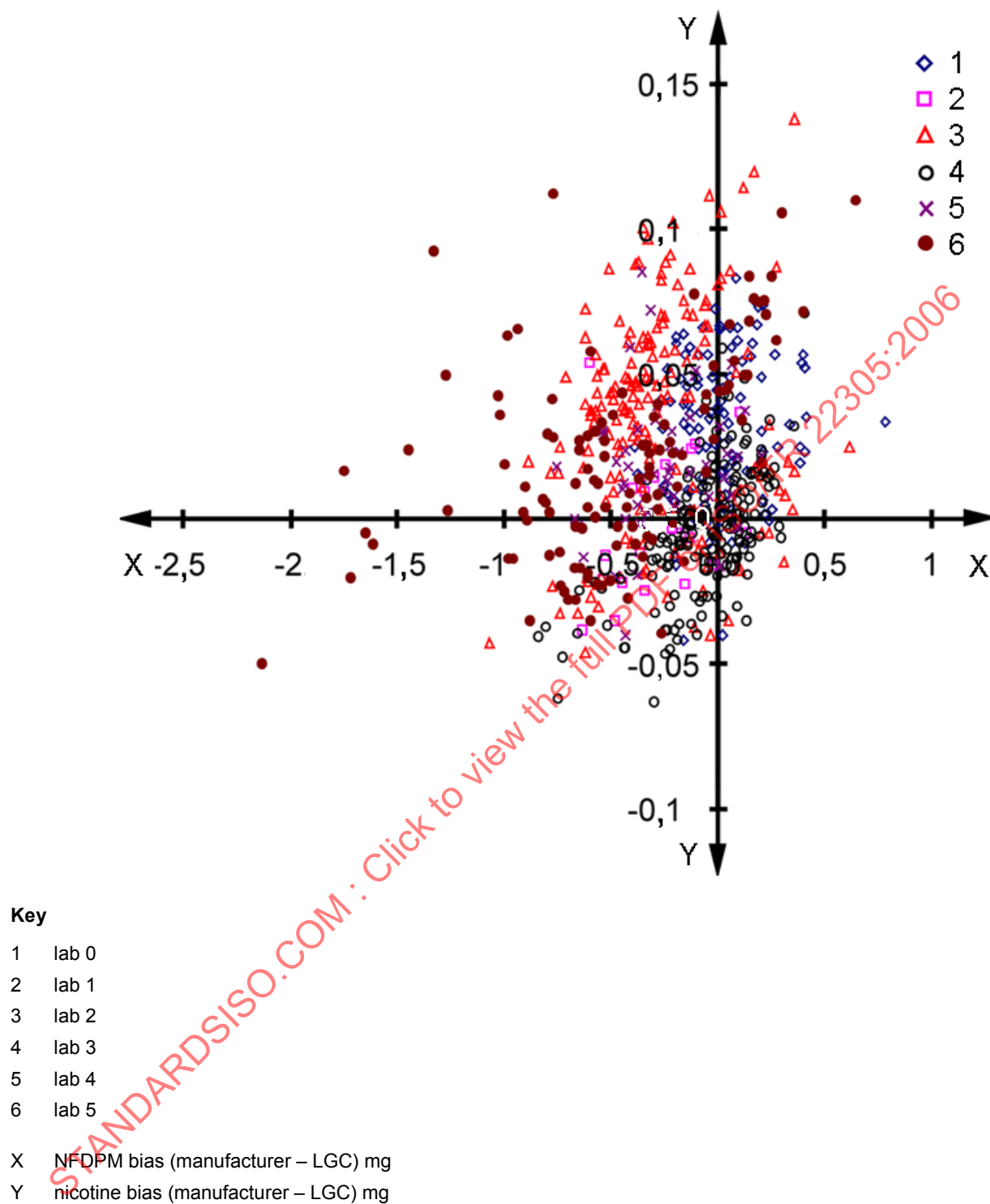


Figure C.8 — Tar vs nicotine
Bias plot for individual brands from DoH surveys 39 to 44

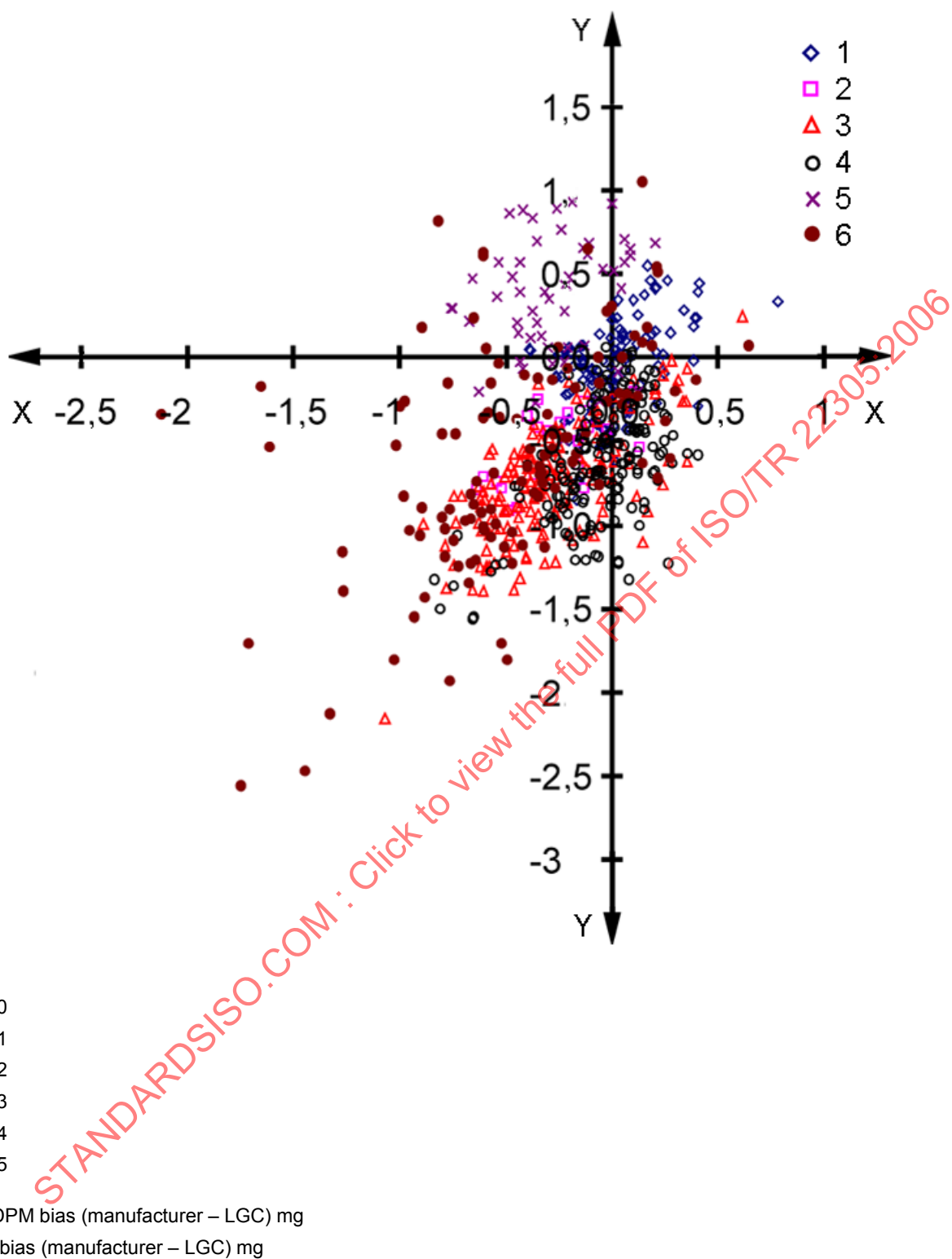


Figure C.9 — Tar vs carbon monoxide
Bias plot for individual brands from DoH surveys 39 to 44

Annex D (informative)

Analysis of bias measurements from the UK Department of Health Cigarette Survey

The Department of Health Cigarette Survey procedure currently forms the basis for verifying the packet declarations in the UK. It was originally set up to monitor the tar reduction program included in voluntary agreements between the UK Government and Tobacco Industry.

The surveys started in 1972 and were divided into 6 sampling periods of 1 month, during which 4 separate sub-samples were taken. A total of 20 cigarettes were tested for each of the 6 sampling periods, 5 from each sub-sample. A gap of one month was left between surveys so that there was a period of seven months between the start of successive surveys.

The duration of each survey was changed to a calendar year in 1995. Since then each survey has involved bi-monthly sampling (from the factory or importer's warehouse) and laboratory testing over a 12-month period, again with four channels of five cigarettes being smoked per sampling occasion. The official assessment of packet declarations by comparison with the Laboratory of the Government Chemist (LGC)¹⁾ yield measurements, is carried out after the completion of each Survey and is based upon the average LGC value for at least five occasions of sampling and testing.

Concurrent with the Department of Health Survey, each manufacturer's laboratory carries out its own testing according to the same format and on 'matched' samples from the same 200-outers. These data 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) is calculated for each brand. These types of checks are essential for all laboratories to ensure their procedures are under control and their measurements are valid.

The measurement biases for NFDPM, nicotine, carbon monoxide, water and puffs are shown for each manufacturer's laboratory in Figures D.1, D.2, D.3, D.4 and D.5 respectively. The period covered is from 1991 immediately after the publication of the revised ISO smoking standards and the bias values are the average for the brands tested from each manufacturer.

If the measurements from the laboratories did not vary with time, the laboratory biases would be constant. This is obviously not the case, the Figures show the biases for all laboratories vary from survey to survey, sometimes randomly and sometimes in a common direction. When the latter occurs, it indicates the measurements from the 'reference' laboratory have altered.

Particular points to note are as follows.

- The smoking hood on the linear machines used by most laboratories was being modified to comply with the introduction of a fixed airflow over the cigarettes during the period of the first 2 or 3 surveys. This was coupled with the setting up of calibration procedures for the anemometers needed to measure the airflow. Bias changes in this period probably resulted from this change.
- The biases for NFDPM are predominantly negative whereas those for nicotine are positive. Although it might be expected that differences in these two particulate phase constituents would be linked, measurement differences between the LGC and other laboratories are driving them in opposite directions.

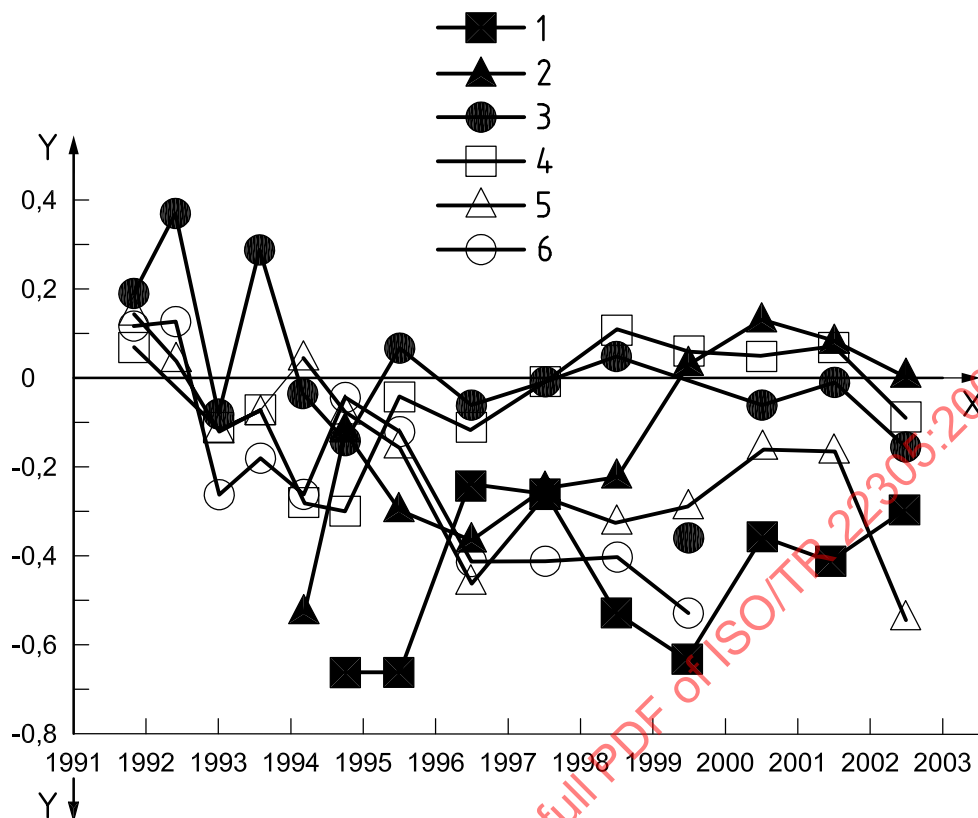
1) Since December 2002 the survey has been carried out by Arista Laboratories Europe.

- The universal rise in the nicotine bias between 2000 and 2001 occurred when the LGC changed their GCs and would, therefore, seem to be due to an unexplained difference between the old and new equipment.
- The increased positive bias for carbon monoxide and water in 1997 for laboratory 4 was associated with the introduction of a new automated smoking machine, and the decrease in these biases in 2001 with the modification of the smoking hood.

The biases for individual brands from 6 surveys, covering the years from 1995 to 2000, are plotted against the LGC yield in Figures D.6, D.7 and D.8, for NFDPM, nicotine and carbon monoxide, respectively. Also included in each Figure are lines fitted to the ± 2 standard deviation points calculated for groups of brands divided into a number of yield ranges. A measurement tolerance was calculated for each of the 3 smoke constituents from the difference between the $+ 2 s$ and $- 2 s$ lines. These are shown as linear plots in Figure D.9 and numerical values in Table D.1. The yield ranges for which the tolerances in ISO 8243 operate on the basis of percentage or mg values, are shaded in the table.

It is of interest to note that the clear banding of brands for tar yield apparent in Figure D.1 is not replicated for nicotine and carbon monoxide. This is due firstly to the brands' being designed primarily to achieve a given tar yield and secondly because tar, nicotine and carbon monoxide cannot be independently controlled.

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**Key**

- 1 lab 5
- 2 lab 1
- 3 lab 3
- 4 lab 0
- 5 lab 4
- 6 lab 2

X year

Y bias (manufacturer-LGC) mg/cig

Figure D.1 — Tar bias (Mean for each manufacturer)

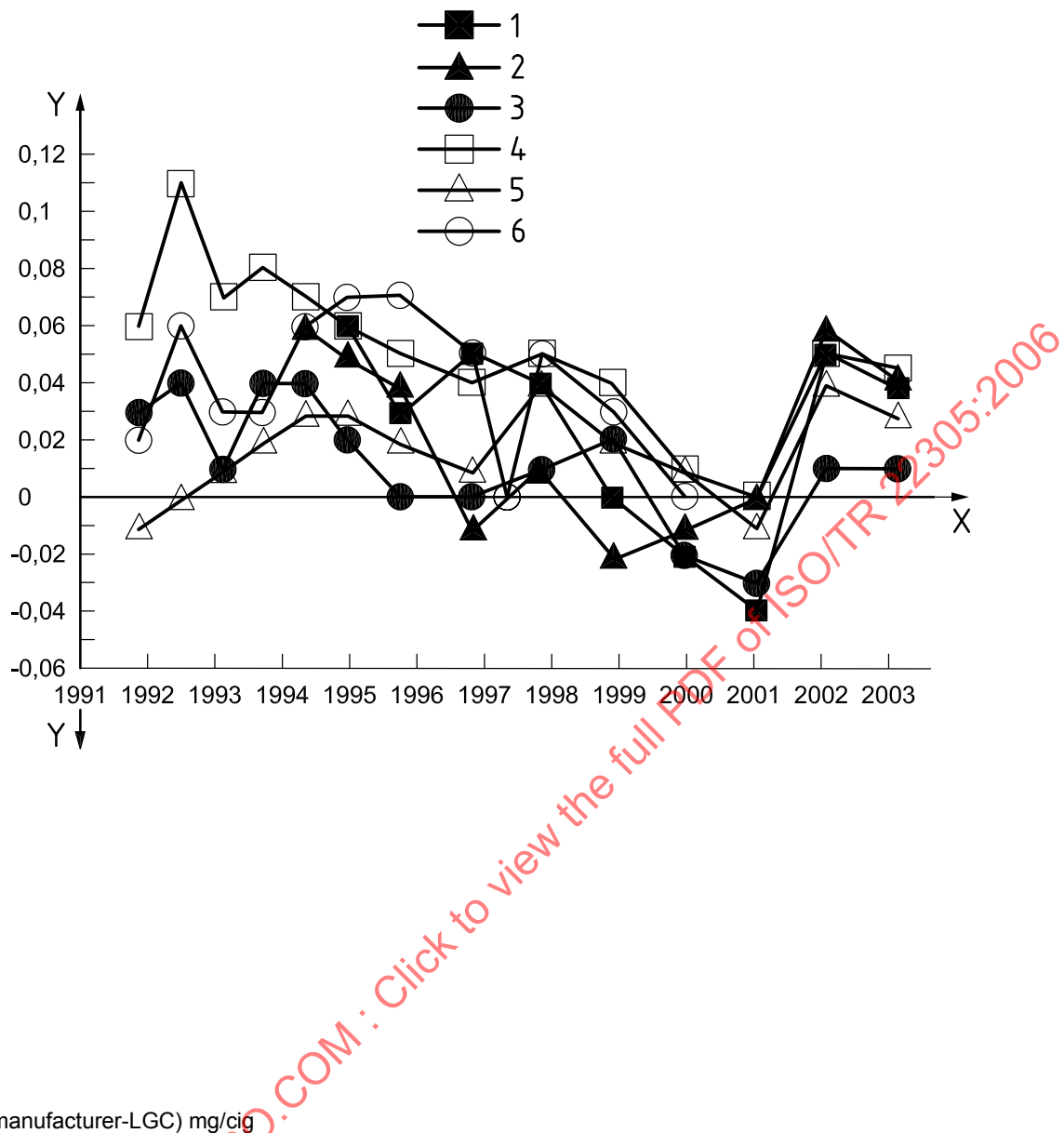


Figure D.2 — Nicotine bias (Mean for each manufacturer)

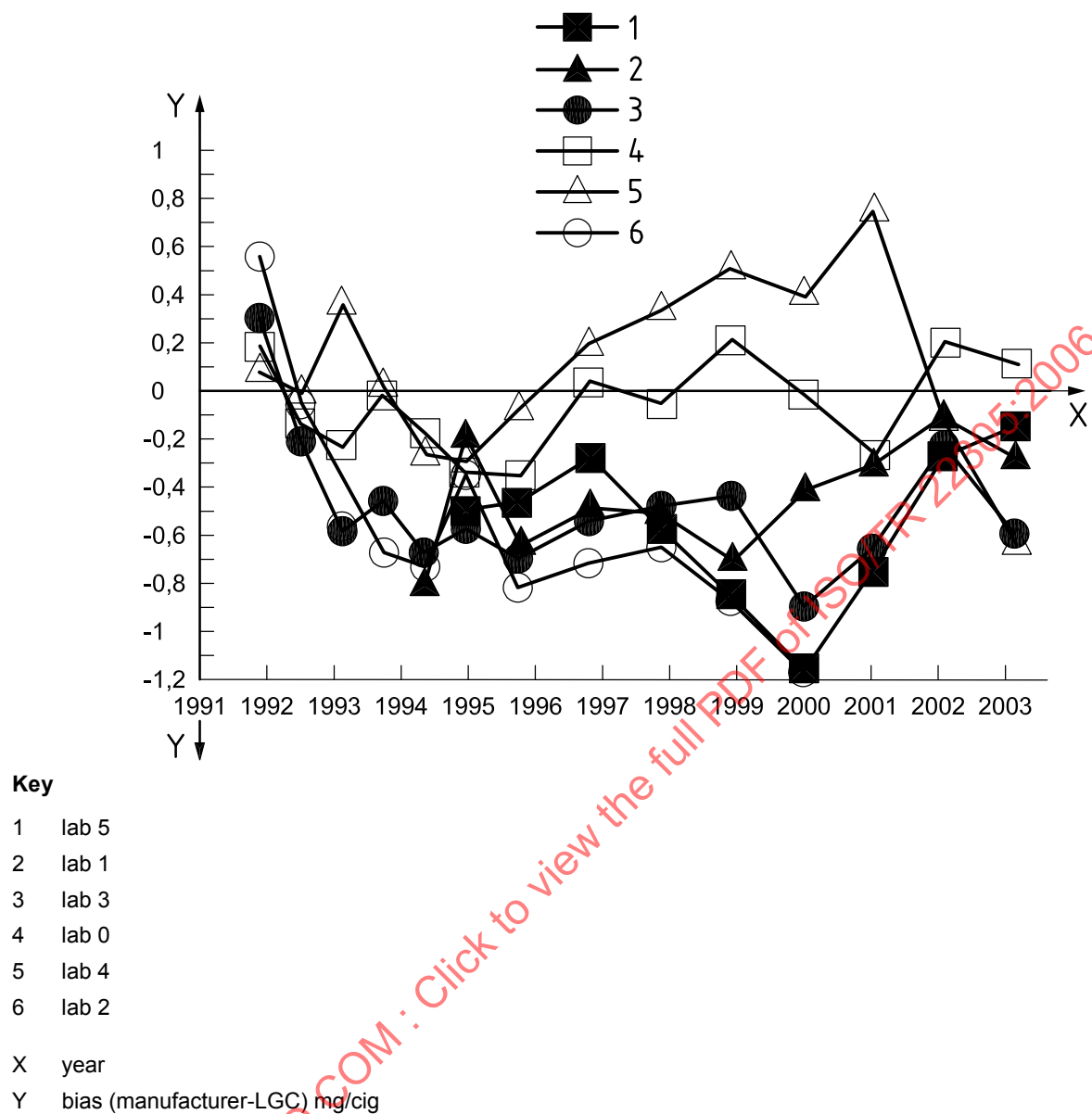


Figure D.3 — CO bias (Mean for each manufacturer)

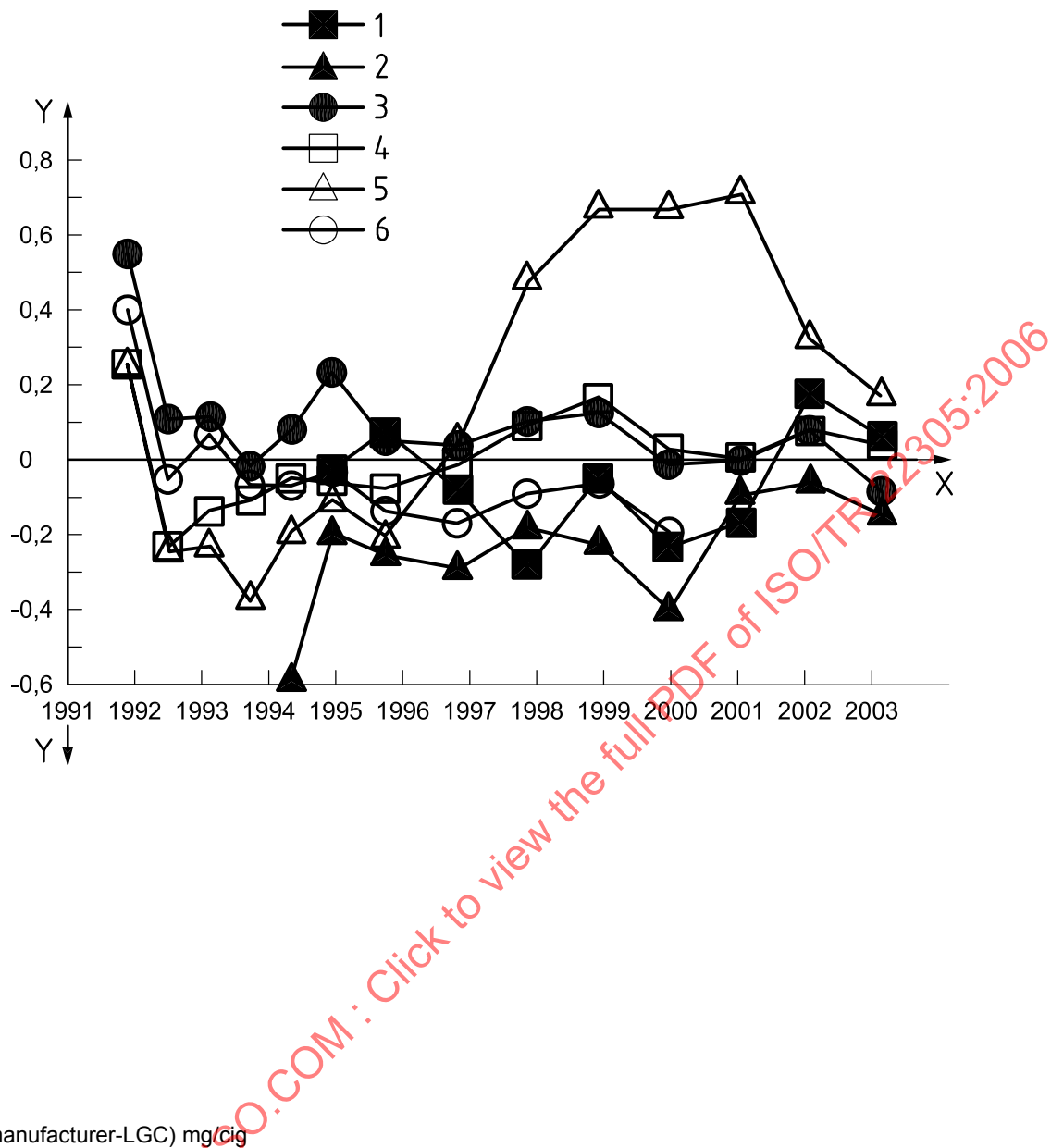
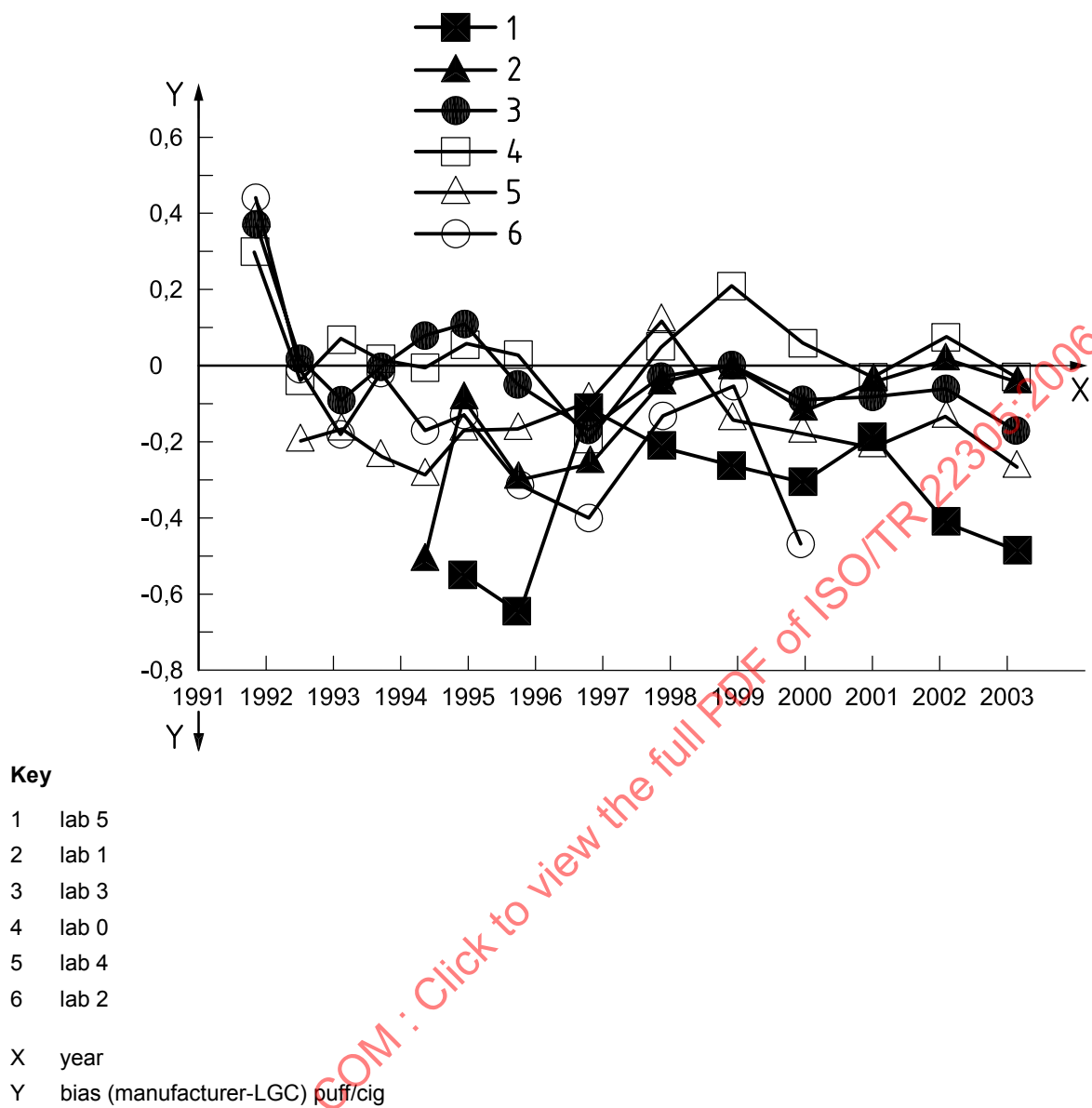
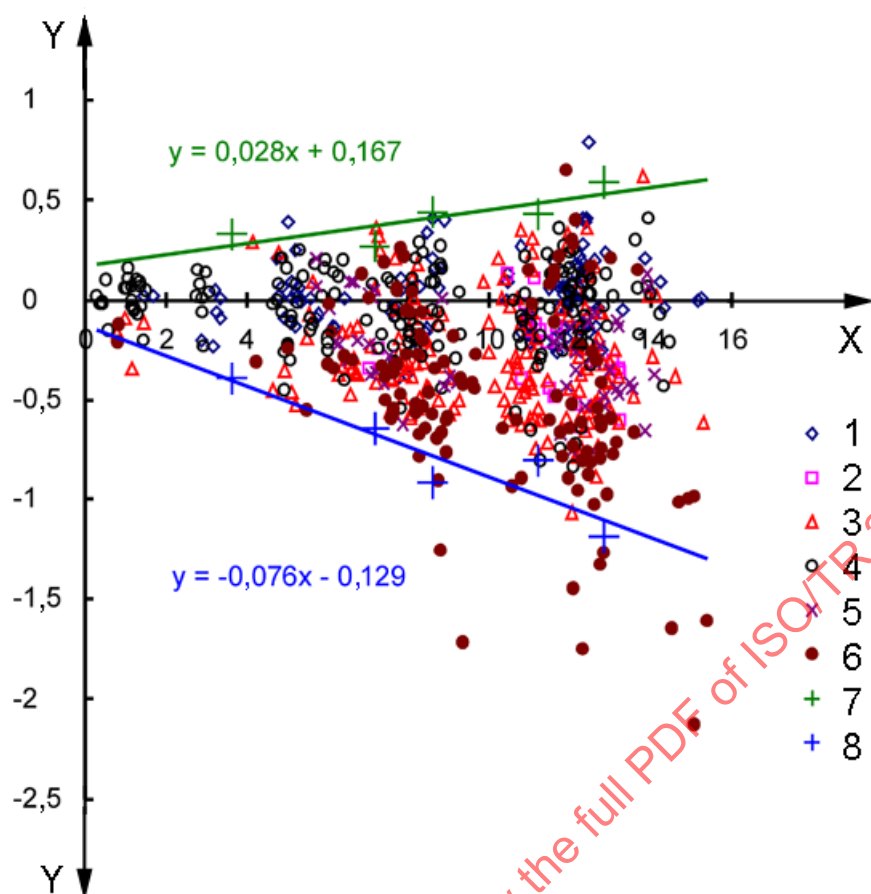


Figure D.4 — Water bias (Mean for each manufacturer)



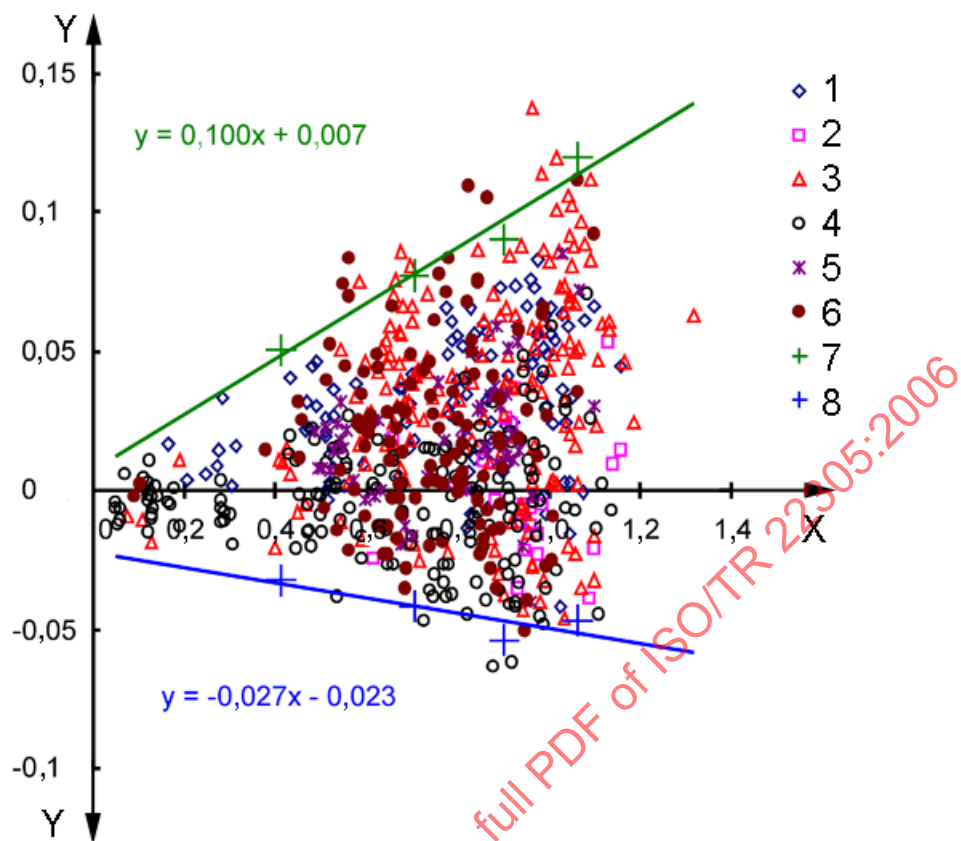


Key

- 1 lab 0
- 2 lab 1
- 3 lab 2
- 4 lab 3
- 5 lab 4
- 6 lab 5
- 7 +2SD
- 8 -2SD

X LGC tar yield, mg
Y bias (manufacturer-LGC), mg

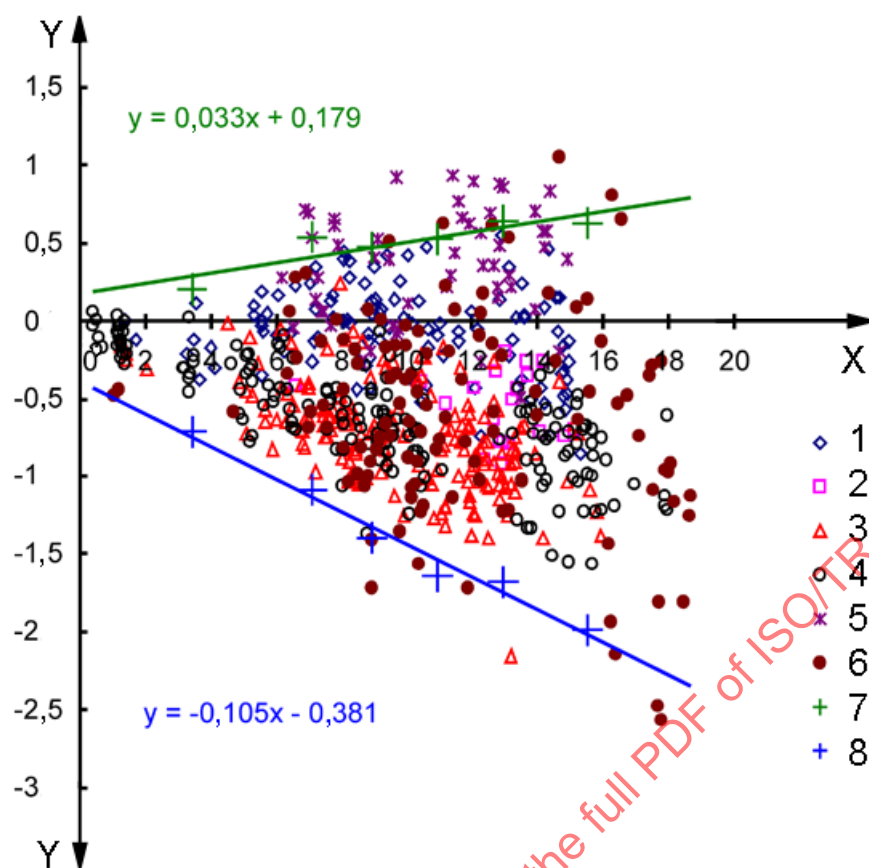
Figure D.6 — Tar biases (mg)

**Key**

- 1 lab 0
- 2 lab 1
- 3 lab 2
- 4 lab 3
- 5 lab 4
- 6 lab 5
- 7 +2SD
- 8 -2SD

X LGC nicotine yield, mg
 Y bias (manufacturer-LGC), mg

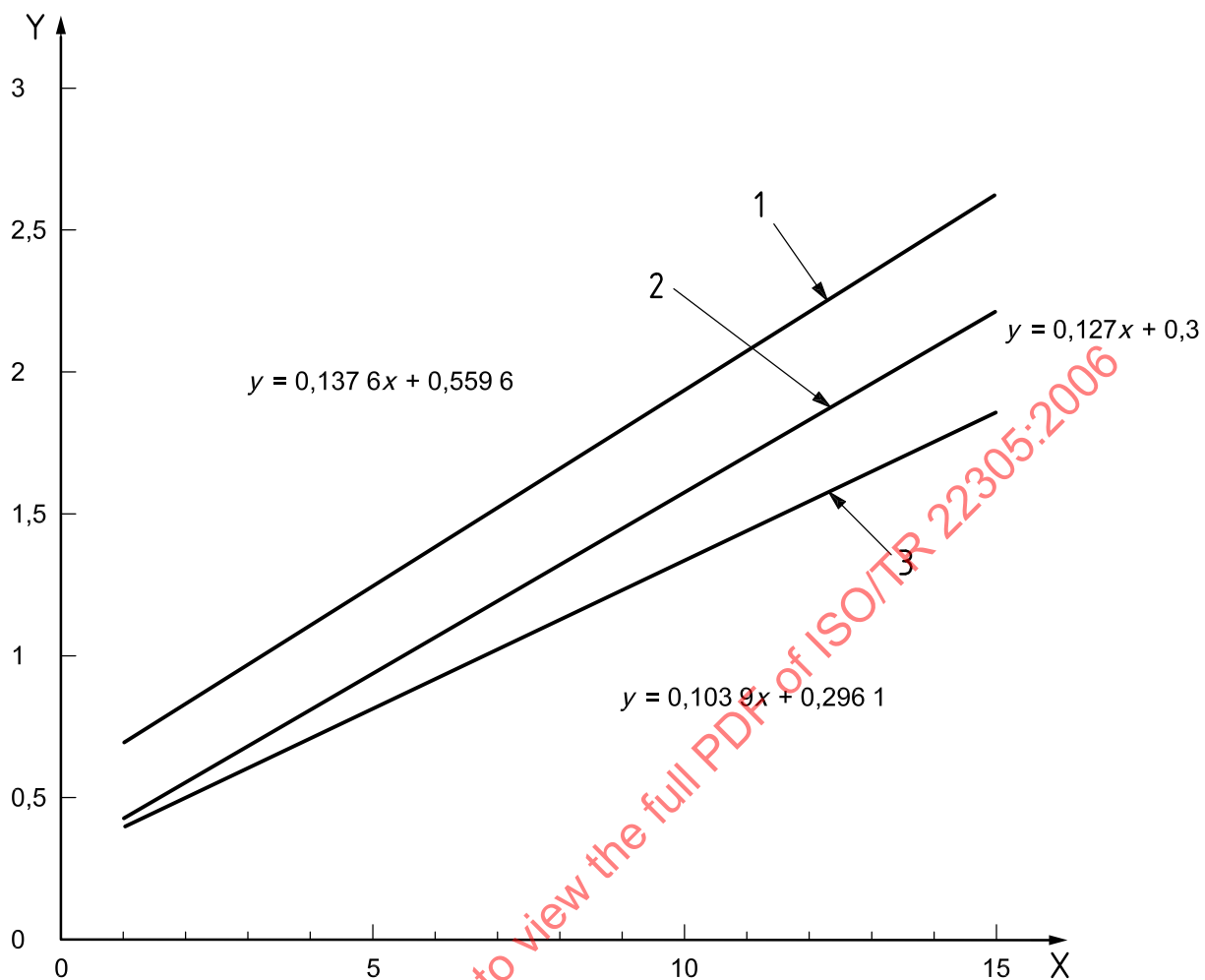
Figure D.7 — Nicotine biases (mg)

**Key**

- 1 lab 0
- 2 lab 1
- 3 lab 2
- 4 lab 3
- 5 lab 0
- 6 lab 5
- 7 +2SD
- 8 -2SD

X LGC CO yield, mg
Y bias (manufacturer-LGC), mg

Figure D.8 — Carbon monoxide biases (mg)

**Key**

- X LGC yield, mg
 Y bias range (manufacturer-LGC), mg
- 1 CO
 2 nicotine
 3 NFDPM

Figure D.9 — T, N and CO bias ranges (mg)
 (Yield and bias ×10 for nicotine)

Table D.1 — Measurement tolerance based on ± 2 standard deviations of bias measurements

Yield ^a mg	NFDPM		Carbon monoxide		Nicotine	
	mg	%	mg	%	mg	%
1	0,40	40,0	0,70	69,7	0,04	42,7
2	0,50	25,2	0,83	41,7	0,06	27,7
3	0,61	20,3	0,97	32,4	0,07	22,7
4	0,71	17,8	1,11	27,8	0,08	20,2
5	0,82	16,3	1,25	25,0	0,09	18,7
6	0,92	15,3	1,39	23,1	0,11	17,7
7	1,02	14,6	1,52	21,8	0,12	17,0
8	1,13	14,1	1,66	20,8	0,13	16,5
9	1,23	13,7	1,80	20,0	0,14	16,0
10	1,34	13,4	1,94	19,4	0,16	15,7
11	1,44	13,1	2,07	18,8	0,17	15,4
12	1,54	12,9	2,21	18,4	0,18	15,2
13	1,65	12,7	2,35	18,1	0,20	15,0
14	1,75	12,5	2,49	17,8	0,21	14,8
15	1,85	12,4	2,62	17,5	0,22	14,7

^a Divide by 10 for nicotine.

Annex E (informative)

ASIA COLLABORATIVE STUDY #11 2002/2003

E.1 General information

E.1.1 Introduction

This report presents and analyses the results of Asia Collaborative Study #11, conducted in 2002 by Lakson Tobacco Company Limited, Pakistan, on the determination of nicotine-free dry particulate matter (NFDPM), nicotine and CO in mainstream cigarette smoke, in accordance with ISO standards.

This study is conducted once every year on cigarette samples having different tar levels and the meeting provides opportunities for an open exchange of technical information in ISO smoking methodology and related subjects.

NOTE Minor editorial changes have been made to the original study report to bring it in line with the required ISO editorial format, and to more easily include it in this Technical Report.

E.1.2 History

A collaborative study was requested by government chemists within the Asia region who were responsible for providing reliable data to their regulatory agencies regarding the ISO tar of cigarettes sold in Asia. The parties concerned within the Asia region voluntarily initiated the Asia Collaborative Study in 1992. The number of participants in the study increased progressively from 9 in 1992 to 44 in 2001, although it decreased slightly to 42 in the last (ACS #11) study conducted during 2002 (see Table E.1).

Table E.1 — Basic study description 1992-2002

	Year	Participants	Country	Tar levels	Location and year of meeting	Host
1 st	1992	9	6	4	Singapore, 1993	PM USA
2 nd	1993	22	14	8	Hong Kong, 1994	RJR USA
3 rd	1994	24	15	5	Tokyo, 1995	JT
4 th	1995	28	16	4	Taejon, 1996	KGTRI
5 th	1996	29	16	4	Bangalore, 1997	ITC
6 th	1997	37	16	5	Shanghai, 1998	STMA
7 th	1998	39	17	5	Tokyo, 1999	JT
8 th	1999	40	17	5	Kuala Lumpur, 2000	CMTM
9 th	2000	42	17	5	Bali, 2001	Sampoerna
10 th	2001	44	18	5	Tokyo, 2002	JT/Lakson
11 th	2002	42	19	5	Singapore, 2003	T.A. S'pore

E.1.3 Benefits

- **Government agencies:** To obtain precise data for the judicious application of regulations.
- **Manufacturers:** To obtain assurance that their products are within the specific limits.
- **Consumers:** To obtain reliable information upon which to make their selection.

E.2 Outline of the present study

E.2.1 Objectives

To determine the degree of agreement in NFDPM, nicotine and CO among the participants.

E.2.2 Participants

Forty-two laboratories participated in the present study, with Japan Tobacco Inc., Philip Morris USA, Philip Morris GmbH Germany, Filtrona Technology Center, Reemtsma GmbH & Co., British American Tobacco Australia and PT HM Sampoerna Tbk. contributing two data sets. Out of 42 participating laboratories, 32 sent analysis data of CO. The participants are listed in Table E.2 in alphabetical order of country.

NOTE 1 Separate code numbers (1 to 42) were assigned to each participating laboratory in confidence. For laboratories sending 2 sets of data (linear and rotary, 2 linear type or 2 rotary type), an alphabetical code is added to assigned lab code to differentiate the type of machine.

NOTE 2 JT International–Canada, CTR-India, National QC Laboratory–Indonesia and BAT–UK either did not send their results or sent them very late and hence are not included in ACS #11.

NOTE 3 Laboratory(ies) marked with *) are new participants in the 11th Asia Collaborative Study.

NOTE 4 Laboratory(ies) marked with **) provided two sets of data (either using linear and rotary, or two linear smoking machines or two rotary smoking machines).

NOTE 5 Laboratory #6 did not measure water and calculated the same by multiplying TPM by 0,11. Resultant data is, therefore, excluded in the water and NFDPM analysis in this report.

Table E.2 — Participants 2002

No.	Name of Participants	Country
1	Philip Morris Ltd. Australia	Australia
2	British American Tobacco Australia	Australia**)
3	Shanghai Tobacco Quality Supervision and Test Station	China
4	China National Tobacco Quality Supervision and Test Centre	China
5	Guangdong Tobacco Quality Supervision and Test Station	China
6	Fujian Tobacco Quality Supervision and Test Station	China
7	Yunnan Tobacco Quality Supervision and Test Station	China
8	Hubei Tobacco Test Center	China*)
9	China American Tobacco Co. Ltd.	China
10	Hong Kong Government Lab	H.K.
11	LTR Industries	France
12	Phillip Morris GmbH	Germany**)
13	JT International Germany GmbH	Germany
14	Borgwaldt Technik GmbH	Germany
15	Reemtsma GmbH & Co.	Germany**)
16	Philip Morris Holland B.V.	Holland
17	Godfrey Philips India Ltd.	India
18	ITC Limited	India
19	PT HM Sampoerna	Indonesia**)
20	PT. Bentoel Prima	Indonesia
21	PT. Djarum	Indonesia
22	Japan Tobacco Inc.	Japan**)
23	Tobacco Institute of Japan	Japan
24	Royal Scientific Society	Japan*)
25	Central Research Institute, KT & G Corp.	Korea
26	Quality and Technology Bureau, KT & G Corp.	Korea
27	British American Tobacco (Malaysia) Bhd.	Malaysia
28	JT International Tobacco Sdn Bhd	Malaysia
29	PT. Sampoerna JL (M)	Malaysia
30	Lakson Tobacco Company, Ltd.	Pakistan
31	British American Tobacco (Singapore) Ltd.	Singapore
32	Health and Sciences Authority	Singapore
33	Philip Morris Europe R & D	Switzerland
34	Taiwan Tobacco & Wine Board	Taiwan
35	National Laboratories of Foods and Drugs	Taiwan
36	Thailand Tobacco Monopoly	Thailand
37	LGC (Teddington) Ltd.	UK
38	Filtrona Technology Centre	UK**)
39	Brown & Williamson Tobacco Corp.	USA
40	Philip Morris U.S.A	USA**)
41	RJ Reynolds Tobacco Co.	USA
42	Arista Laboratories Inc.	USA
NOTE * and **: see E.2.2, Notes 3 and 4.		

E.2.3 Cigarette samples

Five different brands of cigarettes were used in this study. Each participant received 2 cartons each of the 5 different brands coded A to E. Details of the samples are given in Table E.3.

Table E.3 — Details of samples

Code	Brands	Supplier	Intended ISO tar (mg/cig)
A	Winston One 100's Box	JTI Germany	1
B	PM Extra Lights KS Box	PM USA	3
C	Mild Seven Super Lights Box	JT	5
D	Kent Mild KS Box	B & W USA	9
E	CORESTA CM4	Borgwaldt	15

E.2.4 Testing items

Each laboratory was requested to determine the total particulate matter, water, nicotine, nicotine-free dry particulate matter (NFDPM) in smoke and puff count using a routine analytical smoking machine in accordance with the ISO standards. CO measurement was considered as optional for laboratories having CO measurement facilities.

A test protocol for the Asia Collaborative Study #11 was sent to the participants in September 2002. The tests required to be performed by the participants are detailed in Table E.4.

Table E.4 — Study parameters

Smoking machine	Smoke characteristics	Units of measurement	Number of tests	Total data Sets/Sample
Linear 20-port (Filtrona Type)	TPM, water, nicotine, CO	mg/cigarette	6 runs, 4 port/run 5 cig/port	6 (24 data points)
	Puff count	Puffs/cigarette		
	ISO tar (NFDPM)	mg/cigarette	ISO tar = TPM - nicotine - water	
Rotary 20-port (Borgwaldt Type)	TPM, water, nicotine, CO	mg/cigarette	6 runs of 20 cig for each sample	6
	Puff count	Puffs/cigarette		
	ISO tar (NFDPM)	mg/cigarette	ISO tar = TPM - nicotine - water	

E.3 Survey of test equipment and ambient conditions

E.3.1 Test equipment

Details of type and model of test equipment employed by the participants for the study are given in Appendix 6 of the original study description which has been given to the participants and can be made available via the Secretariat.¹⁾

A brief summary of the type of equipment is as follows:

a) Smoking machines

- | | |
|---|---------|
| — Linear 20-port (Filtrona Type) | 30 labs |
| (2 labs submitted 2 data sets of SM 350/400 and ASM500) | |
| — Rotary 20-port (Borgwaldt Type) | 19 labs |
| (2 labs submitted data of Borgwaldt RM20CS and RM200) | |

b) Gas chromatographs

- All labs used gas chromatograph, except lab #6

c) CO analyzer

- 32 labs sent data for CO analysis
- 2 labs sent 2 data sets of Filtrona and Borgwaldt
- 1 lab sent 2 data sets of Filtrona SM400 and ASM500
- 1 lab sent 2 data sets of Filtrona SM400 and RM200
- 2 lab sent 2 data sets of Borgwaldt RM20CS and RM200
- 1 lab sent 2 data sets of Filtrona SM350/400 Upgrade 20-port linear

d) Air flow meters

- | | |
|---------------------------------------|---------|
| — Schiltknecht Anemometer (any types) | 27 labs |
| — Filtrona/Cerulean | 8 labs |
| — Lambrecht Anemometer 642 | 3 labs |
| — Borgwaldt | 2 labs |
| — TSI | 1 lab |
| — JJS - 1 | 1 lab |

1) Whilst Annex E contains the main part of the report circulated to participants in the Asian Collaborative Study, some parts are not directly relevant to this Technical Report and are not included.

E.3.2 Ambient conditions

ISO 3402 and ISO 3308 specify the ambient conditions for the conditioning and testing of cigarette samples as follows:

- Conditioning atmosphere: 22 ± 1 °C and RH (60 $\pm$ 3) %
- Testing atmosphere: 22 ± 2 °C and RH (60 $\pm$ 5) %
- Average air velocity at the cigarette levels: 200 $\pm$ 30 mm/sec

All laboratories that participated in the 11th Asia Collaborative Study met the above required conditions.

E.4 Directions for data analysis

E.4.1 Anomalous data and missing data

All test data reported by the participants is summarized in Appendix 7 of the original study description.

The results were screened for anomalous data and missing data.

If a particular data point for a given parameter was missing, the associated parameters were also eliminated for that port and run combination.

Negative water values were substituted with zeros before the application of statistical tests.

E.4.2 Definitions of the means and standard deviations in this analysis

One test result was defined as the average yield obtained from 20 cigarettes in a single run. The concept should be applied to both linear and rotary smoking machines. With regard to test data obtained by linear smoking machines, the means and standard deviations should be calculated as follows:

e.g.) Laboratory #1, Sample B, NFDPM

Run 1-1	2,56	(data 1)
Run 1-2	3,05	(data 2)
Run 1-3	2,47	(data 3)
Run 1-4	3,18	(data 4)



Run 1	Test results 1	2,82
Run 2	Test results 2	2,44
Run 3	Test results 3	2,47
Run 4	Test results 4	2,63
Run 5	Test results 5	2,49
Run 6	Test results 6	2,27
	Mean =	2,52

$$S = 0,187$$

Average yield = 2,82

E.4.3 Statistical analysis methodologies

E.4.3.1 Exploratory data analyses

E.4.3.2 Mean plots

Mean plots demonstrate both the differences in variation and the level of parameters tested.

The central point in the plots represents the mean and the vertical line represents twice the standard deviation of each set of test data reported by participants.

E.4.3.3 Z-Scores

Z-Scores are being used more and more frequently in inter-laboratory comparisons around the world. A z-score is a normalized value which gives a “score” to each result, relative to the other numbers in the group. A z-score close to zero means that the result corresponds well with those from the other laboratories.

A “classical” z-score, based on mean and standard deviation, is:

$$Z = \frac{\text{result} - \text{mean}}{\text{standard deviation}}$$

A robust z-score simply uses the median and normalized interquartile range (IQR) in place of the mean and standard deviation, so:

$$Z = \frac{\text{result} - \text{median}}{\text{normalized IQR}}$$

Once z-scores have been calculated on a data-set they may be interpreted in the following way:

$$\begin{aligned} |z| &\leq 2 \text{ satisfactory} \\ 2 < |z| < 3 &\text{questionable} \\ |z| &\geq 3 \text{ unsatisfactory} \end{aligned}$$

i.e. if a result has a z-score value greater than 3 or less than -3, it is classified as unsatisfactory.

E.4.3.4 Ranking

- Ranking table
- Ranking graphs

A rank is used to indicate the relationship between an individual and the group.

For individuals that are tied, the arithmetic mean is used to indicate their rank. A rank of 1 is assigned to the largest amount, rank 2 to the next largest, etc.

For measuring parameters, if the scores received by a laboratory fall outside the limits, the laboratory shows a pronounced systematic error on the determination of the parameter.

E.4.3.5 Reproducibility limits (R) and repeatability limits (r)

R and r tables

R and r graphs

Reproducibility and repeatability are two measures of precision and both are used to describe the variability of a test method.

Two sets of data were analyzed for the R and r , i.e. with and without removal of outliers. The Box-and-Whisker plots analysis were used to remove extreme outliers' values prior to further statistical analysis of the data.

E.4.4 Reproducibility limits (R)

Definition: The closeness of agreement between the tests results obtained under reproducibility conditions.

On average, single results from matching cigarette samples reported by two laboratories will differ by more than the reproducibility limit (R) not more than once in 20 cases, if the normal and correct operation of the method is followed.

E.4.5 Repeatability limits (r)

Definition: The closeness of agreement between mutually independent test results obtained under repeatability conditions.

On average, the difference between two single results from matching cigarettes samples obtained by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit (r) not more than once in 20 cases, if the normal and correct operation of the method is followed.

In other words, R and r can be defined as an established value at a given parameter level below which the absolute difference between two inter-laboratory test results may be expected to fall with a specified probability (0,05).

E.4.6 R and r calculations

$$R = 2,8 s_R$$

$$r = 2,8 s_r$$

where

s_r is the repeatability standard deviation between laboratories as expressed by $\sqrt{\sum s^2/p}$;

s_R is the reproducibility standard deviation among laboratories as expressed by the larger of [s_r and $\sqrt{(s_{\text{xavg}})^2 + (s_r)^2 + (s_r)^2 (n-1)/n}$];

s is the standard deviation of an individual laboratory;

p is the number of laboratories;

s_{xavg} is the standard deviation of the laboratories' averages as expressed by

$$\sqrt{\sum_{1}^p d^2 / (p-1)}$$

d is the difference of the individual laboratory's average from the overall average of the laboratories.

E.5 Summary of statistical analyses

E.5.1 Exploratory data analysis

E.5.1.1 Mean plots

The plots of the means and standard deviations, in ascending order of the mean, are shown in Appendix 1 of the original study description.

Table E.5 lists the laboratories having the highest or lowest mean values.

Table E.5 — Laboratories with the highest or lowest mean values

	Sample	A	B	C	D	E
TPM	High	41	18	18	18	40A
	Low	17	17	27	27	30
Water	High	41	41	41	40A	40A
	Low	3	3	3	3	35
Nicotine	High	7	39	39	16	25
	Low	6	6	6	6	6
NFDPM	High	37	18	18	18	35
	Low	17	17	27	27	27
Puff count	High	17	17	17	17	17
	Low	34	41	32	32	32
CO	High	39	4	39	25	25
	Low	27	27	27	27	5

Table E.6 lists the laboratories having the highest standard deviation for each sample, parameter-wise.

Table E.6 — Laboratories with the highest standard deviation

Sample	A	B	C	D	E
TPM	6	6	41	11	31
Water	30	30	30	11	18
Nicotine	7	7	7	35	21
NFDPM	41	41	41	11	31
Puff count	4	25	7	5	35
CO	23A	26	41	35	35

E.5.2 Z-Scores

The z-scores calculation for the three parameters nicotine, NFDPM and CO of each laboratory data are shown in Appendix 2 of the original study description.

Table E.7 lists the laboratories having questionable and unsatisfactory data for each sample.

Table E.7 — Laboratories with questionable and unsatisfactory data

Parameter	Nicotine									
Sample	A		B		C		D		E	
z-scores	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust
Questionable	6, 7, 17, 27	37, 38	39	38, 39	6, 37, 39	11, 15, 18, 37, 38	4, 16	9, 15, 16, 25, 37, 38	25, 40A	4, 7, 15, 32, 37
Unsatisfactory	—	7, 6, 17, 27	6	6	—	6, 39	6	4, 6	6	6, 25, 40A

Table E.7 — (continued)

Parameter	NFDPM									
Sample	A		B		C		D		E	
z-scores	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust
Questionable	—	37	17, 27	—	3, 17, 27	40, 24	18	3, 17, 32, 35	27	27
Unsatisfactory	17	17	18	17, 18, 27	18	3, 17, 18, 27, 35	27	27, 18	—	—

Parameter	CO									
Sample	A		B		C		D		E	
z-scores	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust	Classic.	Robust
Questionable	21, 27, 39	21, 27, 39	4	4, 23, 25, 39	25, 27, 39	4, 8, 31, 40A	27	23	—	5, 23
Unsatisfactory			27	27	—	5, 20, 23, 25, 27, 35, 39	25	25, 27	25	25

E.5.3 Ranking

E.5.3.1 Ranking tables

The ranking scores for each laboratory for the five parameters measured are shown in Appendix 3 of the original study description.

For the ranking score calculations, one laboratory (#6) was excluded from water and NFDPM ranking, i.e. the data from 48 laboratories was used for ranking score calculations for water and NFDPM, while the data from 49 laboratories was used for calculating the ranking score for TPM, nicotine and puff count only.

In the present study of 5 “samples” and 49 “laboratories” for TPM, nicotine and puff count, the approximate 5 % two-tail limits for the ranking scores are 30 - 220.

In the present study of 5 “samples” and 48 “laboratories” for water and NFDPM, the approximate 5 % two-tail limits for the ranking scores are 30 - 215.

In the present study of 5 “samples” and 39 “laboratories” for CO, the approximate 5 % two-tail limits for ranking scores are 25 - 175.

Among 49 “laboratories” for TPM, nicotine and puff count, 48 “laboratories” for water and NFDPM, and 39 “laboratories” for CO, the following showed significantly high or low scores (see Table E.8):

Table E.8 — Laboratories with significantly high or low measurements

Ranking	TPM	Water	Nicotine	NFDPM	Puff count	CO
Significantly high	17, 27	1, 3, 5, 10A, 16	6, 38	17, 27, 30	20, 30, 32	5, 20, 23, 27
Significantly low	18	42A	7, 11, 37, 39	3, 18	10A, 17, 22, 26A, 27, 35	4, 25, 39

E.5.3.2 Ranking tables

The ranking graphs are shown in Appendix 3 of the original study description.

E.5.4 Precision statistics

E.5.4.1 R and r tables

The values for reproducibility and repeatability for each measured parameter were calculated in accordance with ISO 5725-2:1994. The values are shown in Table E.9.

Table E.9 — Repeatability and reproducibility

	TPM	
Ave. (mg/cig)	r Repeatability	R Reproducibility
1,48	0,37	0,83
3,51	0,49	0,98
5,56	0,63	1,18
11,67	0,98	1,62
17,73	1,07	1,93

	Water	
Ave. (mg/cig)	r Repeatability	R Reproducibility
0,170	0,150	0,353
0,294	0,187	0,337
0,483	0,251	0,425
1,458	0,464	0,929
2,167	0,492	1,177

	Nicotine	
Ave. (mg/cig)	r Repeatability	R Reproducibility
0,132	0,029	0,062
0,281	0,035	0,072
0,404	0,051	0,092
0,821	0,094	0,157
1,301	0,082	0,239

Table E.9 (continued)

	NFDPM	
Ave. (mg/cig)	<i>r</i> Repeatability	<i>R</i> Reproducibility
1,184	0,326	0,724
2,939	0,432	0,854
4,678	0,539	1,058
9,404	0,774	1,411
14,244	0,843	1,915

	Puff Count	
Ave. (mg/cig)	<i>r</i> Repeatability	<i>R</i> Reproducibility
10,62	0,51	1,64
7,65	0,43	1,19
7,48	0,38	1,11
7,47	0,36	1,10
9,10	0,41	1,31

	CO	
Ave. (mg/cig)	<i>r</i> Repeatability	<i>R</i> Reproducibility
1,63	0,39	0,95
3,63	0,45	0,97
5,55	0,75	1,46
8,85	0,96	2,06
13,09	0,88	2,33

The values without outlier are shown in Table E.10

Table E.10 — Repeatability and reproducibility without outlier

	TPM	
Ave. (mg/cig)	<i>r</i> Repeatability	<i>R</i> Reproducibility
1,48	0,37	0,84
3,50	0,49	0,81
5,56	0,63	0,93
11,65	0,98	1,63
17,72	1,08	1,94

Table E.10 (continued)

	Water	
Ave. (mg/cig)	r Repeatability	R Reproducibility
0,135	0,143	0,206
0,278	0,182	0,279
0,483	0,249	0,365
1,450	0,464	0,929
2,131	0,500	1,190

	Nicotine	
Ave. (mg/cig)	r Repeatability	R Reproducibility
0,135	0,025	0,045
0,283	0,036	0,062
0,404	0,051	0,083
0,827	0,091	0,138
1,298	0,081	0,184

	NFDPM	
Ave. (mg/cig)	r Repeatability	R Reproducibility
1,203	0,324	0,662
2,953	0,420	0,633
4,654	0,548	0,730
9,400	0,749	1,196
14,257	0,850	1,826

	Puff count	
Ave. (mg/cig)	r Repeatability	R Reproducibility
10,56	0,50	1,43
7,60	0,43	0,95
7,43	0,38	0,88
7,39	0,36	0,77
9,00	0,37	0,94

Table E.10 (continued)

	CO	
Ave. (mg/cig)	r Repeatability	R Reproducibility
1,63	0,36	0,79
3,63	0,45	0,82
5,58	0,71	0,95
8,85	0,98	1,71
13,09	0,88	1,79

E.5.4.2 R and r plots

Plots of the reproducibility values and repeatability values for the method are shown in Appendix 4 of the original study description. Values of reproducibility and repeatability of the ISO method, as given in ISO 4387:2000, ISO 10362-1:1999 and ISO 10315:2000 for yield of NFDPM, water and nicotine respectively, were also plotted for comparison.

Water

		<u>with outliers</u>	<u>without outliers</u>
r	Higher than those described in ISO	Nil	Nil
R	Higher than those described in ISO	Sample A, D	Sample D

Nicotine

		<u>with outliers</u>	<u>without outliers</u>
r	Higher than those described in ISO	Sample D	Sample D
R	Higher than those described in ISO	Sample D, E	Sample D, E

NFDPM

		<u>with outliers</u>	<u>without outliers</u>
r	Higher than those described in ISO	Nil	Nil
R	Higher than those described in ISO	Sample C, D, E	Sample E

E.5.5 Comparison of linear and rotary smoking machines

We made a comparison between linear and rotary smoking machines using data of the Asia Collaborative Study #4 - 11. The number of laboratories that used linear or rotary in Asia Collaborative Study #4 - 11 is listed in Table E.11.

Table E.11 — Number of linear and rotary smoking machines in different Asia Collaborative Studies

Type	4 th	5 th	6 th	7 th	8 th	9 th	10 th	11 th
Linear	24	24	23	30	29	33	29	30
Rotary	5	5	8	9	15	13	19	19
TOTAL	29	29	31	39	44	46	48	49

The comparison of linear and rotary for each parameter is shown in Appendix 5 of the original study description.

There seemed to be very few differences in the results of TPM, water, nicotine, NFPDM yields and puff count for all samples.

Sample E (higher tar cigarettes) yielded significantly higher water results and lower TPM, NFDPM on rotary smoking machines compared to those obtained on linear smoking machines.

Rotary smoking machines also yielded higher CO results than those of the linear machines. However, the differences in study #11 are significantly lower than those observed in earlier studies.

NOTE 1 Lakson Tobacco Company Limited thank all the participants who provided data for this collaborative study, and Dr. Muramatsu of TIOJ Laboratory, Japan, and Dr. Mochammad Sholichin of P.T.H.M. Sampoerna Tbk., Indonesia, for reviewing the final draft copy of this report and providing useful feedback.

NOTE 2 Special thanks to the following members of Lakson for their tireless efforts, support and commitment in producing the ACS #11 report within a short span of time: Mr. Syed Umer Afraz Sharjeel, Senior Officer, Group Quality Assurance, Mr. Muhammad Arshad Ghaznavi, Web Designer, and Miss Tabassum Zaidi, Research Officer.

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Annex F (informative)

2003 CORESTA Collaborative Study Report

CORESTA study for the estimation of the repeatability and reproducibility of the measurement of nicotine-free particulate matter, nicotine and CO in smoke using the ISO smoking methods, September 2003

F.1 Introduction

The CORESTA CO Sub-Committee was set up in May 1999 by the CORESTA Scientific Committee "to address the issue of different CO results obtained with rotary and linear smoking machines. The sub-committee should make recommendations on how to resolve the issue."

The CO Sub-Committee decided to perform this study in order to confirm the successful conclusion of the work undertaken.

Basically the CO Sub-Committee has studied a series of parameters that could influence the smoke yields from the smoking machines. The lighting energy, the lighting method, the distance between the lighter and the cigarette end and the position of the side seam during smoking has been studied. However, the most important parameter is the airflow around the cigarettes. A much better understanding of the importance of this parameter, both velocity and direction, and a better control has been obtained. Many comparisons with a limited number of participants have been performed, and the rotary smoking machines RM200 and RM20 have been modified while modification of the rotary smoking machine RM20CSR was discussed but found not to be necessary.

To establish the results of the harmonization the sub-committee decided to perform a study of 8 products including 7 commercial brands and the CORESTA monitor CM4. The choice of products for the study was made to encompass the influence of different cigarette designs on the smoke yields balanced with the need to limit the study to a "reasonable" size for the participating laboratories. This meant that "normal" cigarettes with different levels of filter ventilation, cigarette length and cigarettes without filter could be included while preventing to inclusion of cigarettes with more special construction. As mentioned it was a priority to keep the study at a "reasonable" size as it was seen as more important to have a large number of participants smoking several brands than to try to include all possible parameters into a large-scale study with the risk of having (too) few participants.

Along with the CORESTA work the carbon monoxide standard ISO 8454 was up for revision and at the same time the *European Directive on the manufacture, presentation and sale of tobacco products* introduced the declaration of carbon monoxide yields from 1st January 2004 which led to a need for the introduction of tolerances for carbon monoxide values in the sampling standard (ISO 8243). The present CORESTA study contributes with analytical results to facilitate the evaluation of the necessary tolerances for carbon monoxide.

During the revision of ISO 8454 and ISO 8243 the ISO/TC 126 expressed interest in the participation of non-CORESTA members in the study and the CORESTA Board and Scientific Commission agreed to this. Consequently 5 regulatory laboratories along with other non-CORESTA members participated in the study.

The study was carried out with the participation of 39 laboratories reporting data from a total of 61 smoking machines. A list of the participants and the smoking machines used is given in F.7.8.

The protocol for the study and an example of the reporting format are part of the detailed study results and available from CORESTA.²⁾

F.2 Statistical evaluation

The data were scrutinised for printing errors and missing data by the study coordinator before the data were sent for statistical evaluation.

The outlier removal was performed as closely as possible to the guidelines given in ISO 5725.

The results were treated as one data set thus the outlier removal was not performed for each smoking machine type individually. No rank sum test was performed. The overall principle was to keep as many results, and not to remove as many as possible.

The full description of the statistical evaluation including the procedures for outlier removal is given in F.8 the Statistical report for the CORESTA study.

Two of the participating laboratories, each reporting one data set, were late for the statistical analysis and could not be included. The quality of the two "late" laboratories though was fully concordant with the results from the other participants after the statistical removal of outliers.

F.3 Discussion

To evaluate the results of the harmonization the smoke yields before and after the harmonization have to be compared.

When the sub-committee was set up in 1999 the smoke yields from the annual smoking of the CORESTA monitor CM2 from the different smoking machine types were as shown in Table F.1.

**Table F.1 — Smoke yields from the CORESTA annual smoking of CM2
Filtrona 400, RM200 and RM20CSR**

Study	Harmonization	Smoking machine	NFDPM	Nicotine	CO	Puff number
Spring 1999	"Before"	"Filtrona 400"	15,43	1,266	14,33	8,96
		RM200	14,71	1,224	15,97	8,75
		RM20CSR	15,11	1,262	15,29	9,11
		"400-200"	0,72	0,042	−1,64	0,21
		"400-20CSR"	0,32	0,004	−0,96	−0,15

It can be noted that the difference between the CO yields for the Filtrona 400 and the RM200 is larger than 10 %.

The latest results from the annual smoking of the CORESTA monitor CM4 shows the smoke yields given in Table F.2.

2) Whilst Annex F contains the main part of the report circulated to participants in the CORESTA Collaborative Study, some parts are not directly relevant to this Technical Report and are not included.

**Table F.2 — Smoke yields from the CORESTA
annual smoking of CM4 Filtrona 400, RM200 and RM20CSR**

Study	Harmonization	Smoking machine	NFDPM	Nicotine	CO	Puff number
Spring 2002	“Before”	“Filtrona 400”	14,53	1,316	12,67	9,19
		RM200	13,61	1,25	14,46	8,67
		RM20CSR	14,1	1,28	13,8	9,07
		“400-200”	0,92	0,066	−1,79	0,52
		“400-20CSR”	0,43	0,036	−1,13	0,12
Spring 2003	“Before”	“Filtrona 400”	14,53	1,29	13,09	9,24
		RM200	13,57	1,28	(13,42) ^a	8,89
		RM20CSR	14,1	1,29	13,59	9,08
		“400-200”	0,96	0,01	–	0,35
		“400-20CSR”	0,43	0	−0,5	0,16
Autumn 2003	“After”	“Filtrona 400”	14,52	1,31	12,38	9,15
		RM200	13,54	1,28	13,45	8,92
		RM20CSR	14,09	1,3	13,73	9,01
		“400-200”	0,98	0,03	−1,07	0,23
		“400-20CSR”	0,43	0,01	−1,35	0,14

^a The CO value from Spring 2003 should not be used as some of the smoke results may be from harmonized machines.

Table F.2 shows clearly that the results for NFDPM, nicotine or puff number have been stable or come closer together over the harmonization period. This means that the harmonization of the RM200 smoking machine has had no adverse influence on the results for NFDPM, nicotine or puff number.

The table also shows that the difference in carbon monoxide results from the SM400 and the RM200 smoking machines has been considerably reduced without any practical influence on other smoke yields from the two smoking machine types. That was our primary aim.

The sub-committee also discussed the need to harmonize the RM20CSR smoking machine. At a certain time it was considered logical that this would be necessary to obtain a similar improvement of the airflow (reduced turbulence) in the RM20CSR as in the RM200 smoking machine.

A modification of the RM20CSR was in fact studied and tested. The physical construction of the smoking machine, especially the position of the butt length detector, makes a modification, using the same principles as for the RM200, very difficult. It was thought that the airflow in the RM20CSR would show a similar turbulent pattern as the RM200 before the modification, and therefore also require a similar modification. But a study in fact showed that the turbulence in the RM20CSR was far less than expected. In light of this and the practical difficulties in modifying the physical construction of the RM20CSR it was decided to study the necessity to modify the RM20CSR by the present study.

The results in the table above show that the NFDPM, nicotine and puff number for CM4 from RM20CSR are "between" the corresponding results for RM200 and SM400, and that the difference between the CO values from RM200 and RM20CSR is so small that it may have no practical consequence. The relatively "high" CO value for the RM20CSR may nonetheless be a sign for caution. The present study comprises smoke yields from 7 commercial brands so the practical consequence of the above indicated CO differences can be studied for each of the 7 brands.

The report from the study contains all the analytical results but to facilitate a general conclusion we have generated the mean for the results of all the products for each parameter – that means we produced the results for an “average product”. These results are shown in Table F.3.

Table F.3 — Smoke yields from CORESTA study autumn 2003 – “average product”

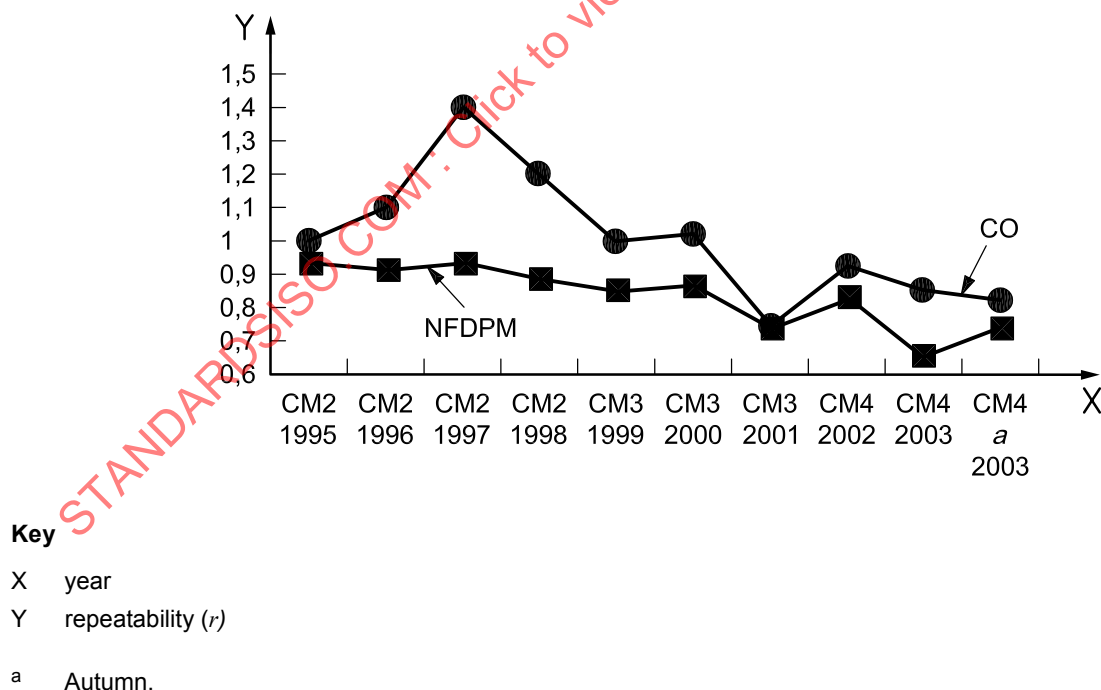
	“Filtrona 400”	RM200	RM20CSR
NFDPM	9,77	9,37	9,61
Nicotine	0,79	0,79	0,80
CO	9,10	9,68	9,84
Puff number	8,15	7,96	8,08

The CO values for the “average product” confirm (when combined with the variability between laboratories) that it is not needed to modify the RM20CSR.

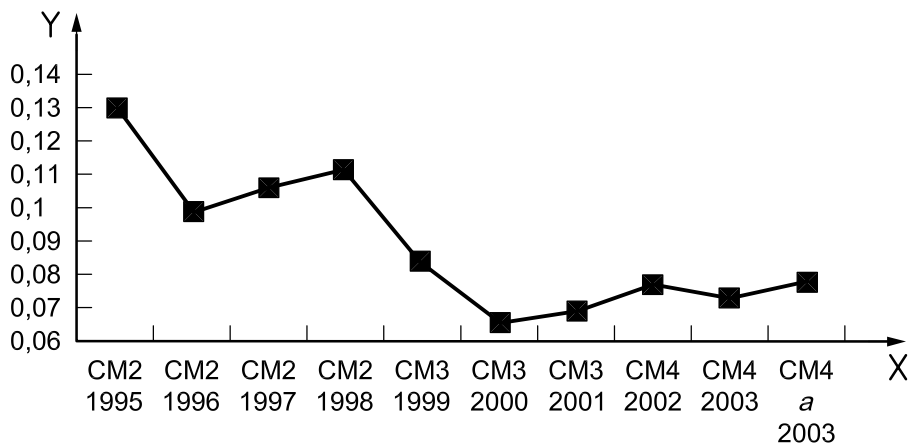
F.4 Variability of smoke yields from the annual smoking of the CORESTA monitor

The CORESTA monitor products CM2, CM3 and CM4 have been smoked annually since 1995 and the results give an impression of the development of the variability in the smoking and analytical process in the analytical laboratories from the tobacco industry.

It should be noticed that the results from year to year can have been influenced by changing in number and combination of participants and it is certainly a fact that the present study has a very much wider and larger participation than any previous CORESTA study.



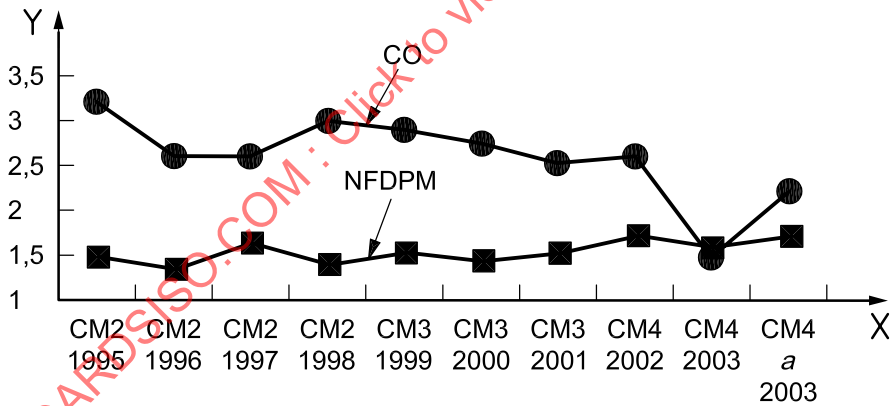
**Figure F.1 — CORESTA Monitor
Repeatability (r)
NFDPM and CO**



Key
X year
Y repeatability (*r*)
^a Autumn.

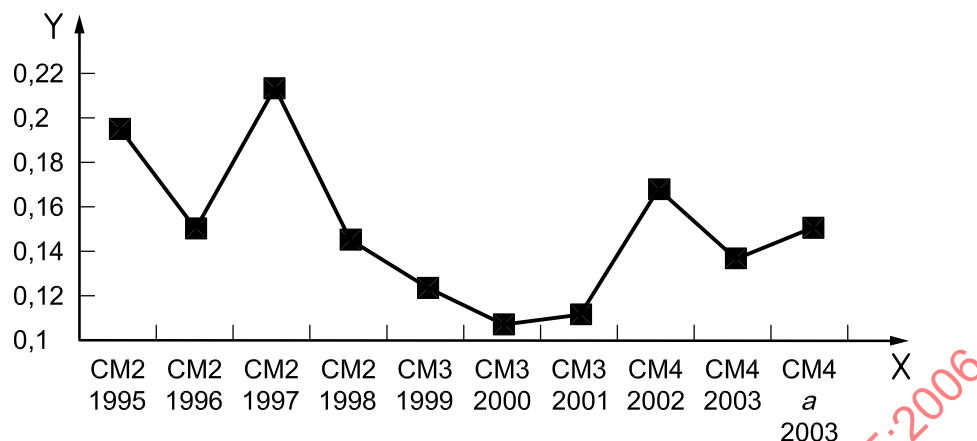
Figure F.2 — CORESTA Monitor
Repeatability (*r*)
Nicotine

The graphs show a general improvement of the repeatability for the smoke yields.



Key
X year
Y reproducibility (*R*)
^a Autumn.

Figure F.3 — CORESTA Monitor
Reproducibility (*R*)
NFDPM and CO

**Key**

X year

Y reproducibility (*R*)^a Autumn.

**Figure F.4 — CORESTA Monitor
Reproducibility (*R*)
Nicotine**

The positive development that we have seen for the reproducibility is also seen for the repeatability (with the above mentioned reservation for the value from spring 2003 for CO).

The overall conclusion of the graphs representing the development of repeatability and reproducibility of the smoke yields from the annual studies of the CORESTA monitor confirms that there has been a general improvement which in no way has been adversely influenced by the harmonization of the RM200 smoking machine, nor seem to indicate a necessity to modify the RM20CSR smoking machine.

F.5 Tolerances for CO

The estimated values for *R* for the 8 products analysed (relative to the mean of the individual parameter) are shown in Table F.4.

Table F.4 — Repeatability

<i>R</i> %	NFDPM	Nicotine	CO
Camel	16,7	14,1	20,7
CM4	12,3	11,2	16,6
Ducados	12,3	15,1	19,2
Marlboro	9,6	12,6	18,7
Marlboro Lights	14,8	16,3	20,9
Pall Mall 100	10,0	12,7	17,4
Philip Morris Super Lights	21,2	21,8	26,1
Regal	13,2	14,0	18,2

The values seem to indicate that there is a need for wider tolerances for the CO values than for the corresponding nicotine and tar values.

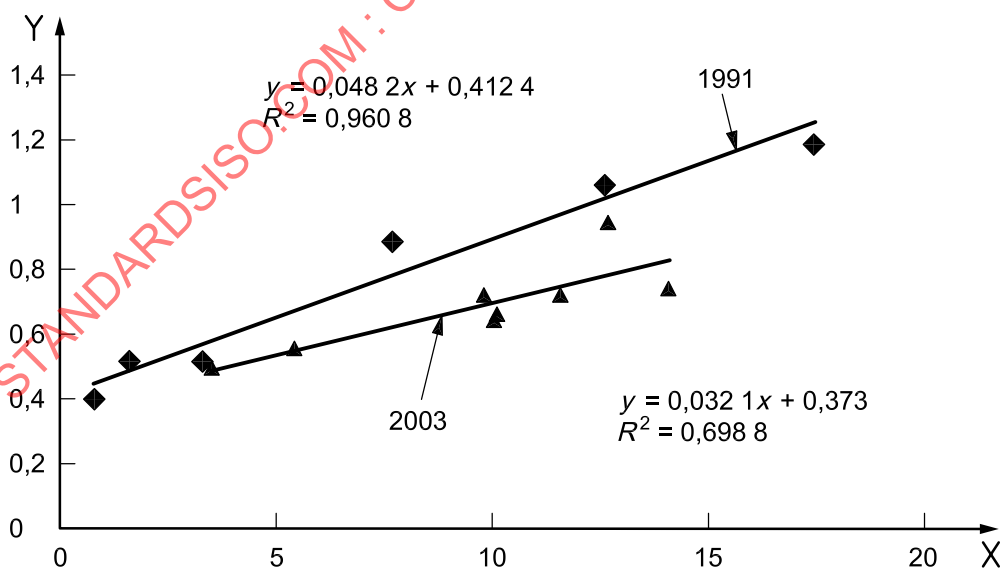
F.6 General variability of the smoking methods

The present study presents results from 39 laboratories for 8 products and it may be reasonable to compare the estimates for the variability (r and R) with the estimates obtained during the revision of the CORESTA and ISO smoking methods in 1991 and at the development of the CO method in 1993.

It must be emphasized that the regression lines which are calculated for the graphical presentation of the estimates are only added to "ease" the comparison and in no way implicate any functional relationship between the mean and the variability. The reduced fit to the regression line in 2003 may be attributed to the wider range of products analysed in the present study.

Table F.5 — Variability of NFDPM in two studies

Harmonization (1991)			Study Autumn 2003		
Mean	" r "	" R "	Mean	" r "	" R "
0,82	0,40	0,60	3,54	0,49	0,75
1,61	0,52	0,74	5,43	0,55	0,80
3,31	0,52	0,90	9,83	0,72	1,30
7,70	0,88	1,51	10,04	0,64	1,01
12,61	1,06	1,70	10,07	0,66	1,24
17,40	1,19	1,84	11,56	0,71	1,11
			12,68	0,94	2,12
			14,06	0,74	1,73

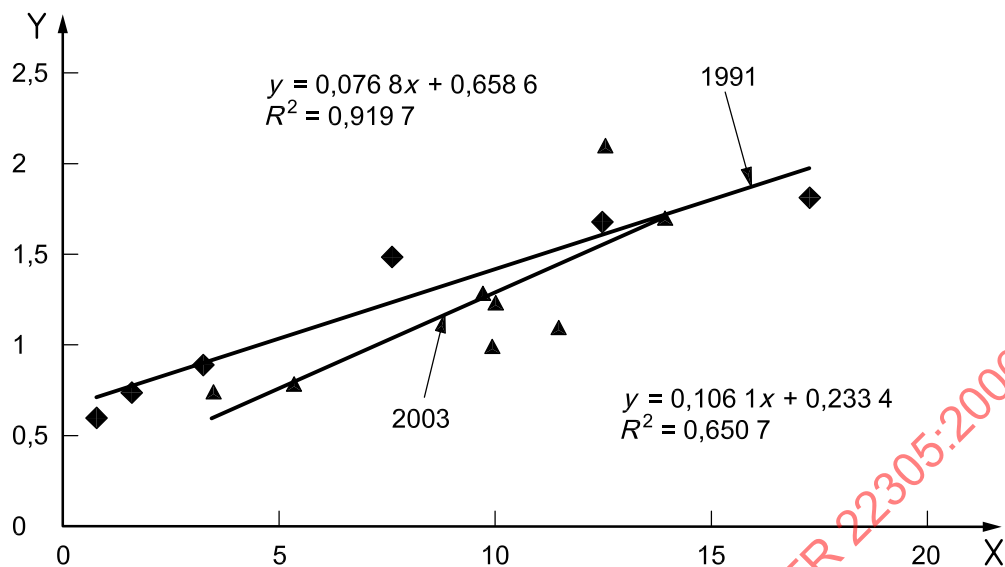


Key

X mean

Y repeatability (r)

Figure F.5 — NFDPM – r

**Key**

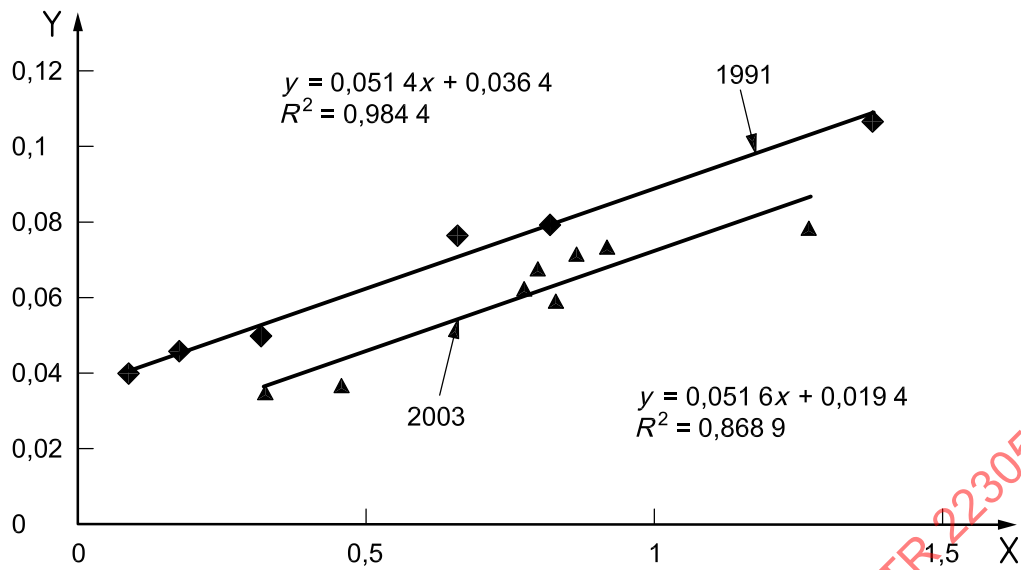
X mean

Y reproducibility (*R*)**Figure F.6 — NFDPM – *R***

The estimates indicate that the variability for NFDPM in 2003 is similar or reduced compared to 1991. It should be noted that the very high estimate for the tar value 12,68 (Camel plain) influences the regression very much. But as the regressions are only included to indicate the general trend we have not intended to perform any outlier test for this estimate.

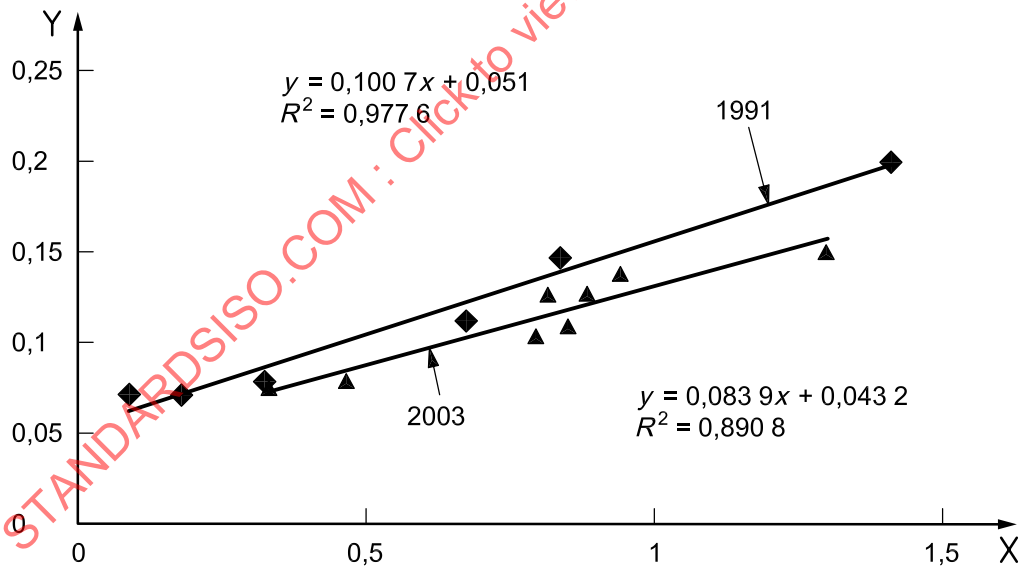
Table F.6 — Variability of nicotine in two studies

Harmonization (1991)			Study Autumn 2003		
Mean	" <i>r</i> "	" <i>R</i> "	Mean	" <i>r</i> "	" <i>R</i> "
0,091	0,040	0,069	0,33	0,04	0,07
0,179	0,046	0,069	0,47	0,04	0,08
0,326	0,050	0,076	0,79	0,06	0,10
0,673	0,077	0,109	0,82	0,07	0,12
0,835	0,079	0,142	0,85	0,06	0,11
1,412	0,107	0,195	0,88	0,07	0,12
			0,94	0,07	0,13
			1,30	0,08	0,15



Key
X mean
Y repeatability (*r*)

Figure F.7 — Nicotine — *r*



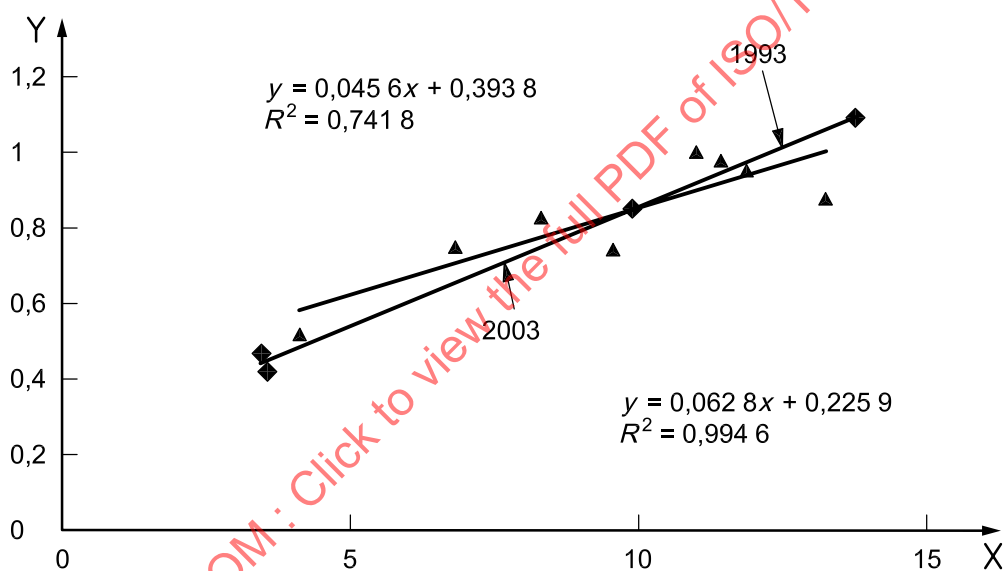
Key
X mean
Y reproducibility (*R*)

Figure F.8 — Nicotine — *R*

The estimates show that the variability for nicotine in 2003 is reduced compared to 1991.

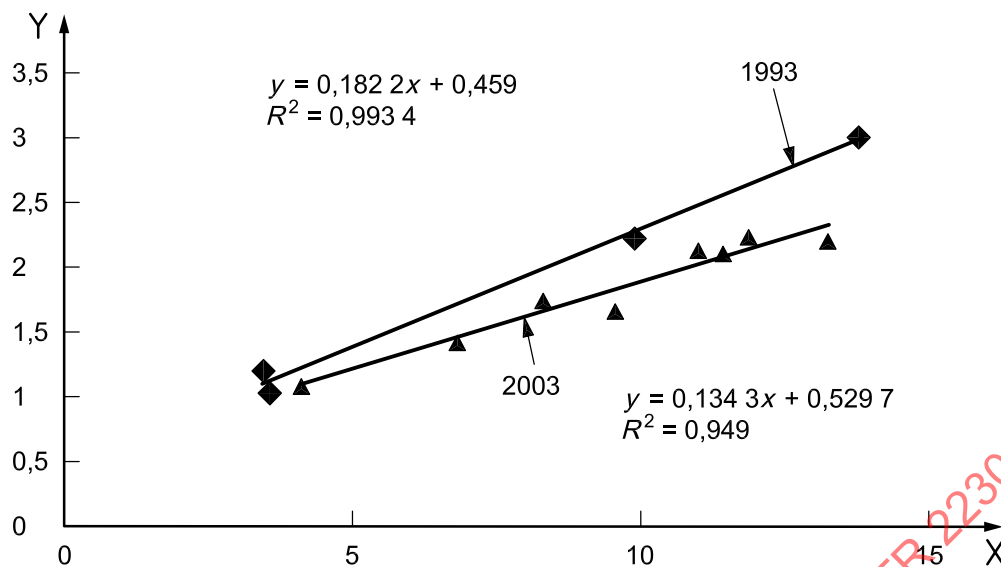
Table F.7 — Variability of CO in two studies

CO study (1993)			Study Autumn 2003		
Mean	"r"	"R"	Mean	"r"	"R"
3,45	0,47	1,18	4,12	0,52	1,08
3,56	0,42	1,03	6,79	0,75	1,42
9,89	0,85	2,22	8,31	0,82	1,72
13,80	1,09	3,00	9,55	0,74	1,66
			10,99	1,00	2,11
			11,45	0,97	2,09
			11,85	0,95	2,22
			13,23	0,87	2,20

**Key**

X mean
Y repeatability (r)

Figure F.9 — CO — r

**Key**

- X mean
- Y reproducibility (R)

Figure F.10 — CO — R

The estimates show that the variability for CO in 2003 is similar or reduced compared to 1993.

F.7 Conclusion

The present study demonstrates that:

- the differences in CO yields have been reduced without adverse influence on the smoke yields for NFDPM and nicotine;
- a modification of the RM20CSR smoking machine is not necessary;
- the estimates of variability for the smoking methods have generally been reduced in comparison to the estimates from 1991 (for CO from 1993);
- the relative variability for CO determination is larger than for the determination of NFDPM and nicotine.

F.8 Statistical report for the CORESTA study**F.8.1 Objectives**

- The identification of outlying results and laboratories, attributable to both abnormal average levels and excessive levels of variation.
- To establish the average levels of NFDPM, nicotine, CO, puff number, TPM and water for products CORESTA Monitor CM4, Camel Regular, Ducados, Marlboro, Marlboro Lights, Pall Mall 100's, Philip Morris Super Lights and Regal King Size.
- The determination of repeatability [r] and reproducibility [R] statistics for these six parameters, for each product and for specific subsets of machines.
- A comparison across products/laboratories of the conditioned weight of the products.

F.8.2 Results available

The full list of results is tabulated in Form A of the original study description.

F.8.3 Missing data

Laboratory AX (RM200) only reported two runs for Camel. Laboratory G1 (RM20CSR) only reported four runs for Regal and CM4. Laboratory M (ASM516) only reported four runs for CO for Camel. Laboratory AV only reported four runs for CO for Pall Mall 100 and three runs for CO for Camel.

F.8.4 Analysis

The statistical analysis follows as closely as possible the methods provided by ISO 5725-2 (1994).

A visual inspection of the results in Form A does not reveal any obviously erroneous data (as defined in 7.6.2 of ISO 5725-2). A graphical representation of the results in Form A is provided in 12 figures of the original study description (not included in this report).

F.8.5 Outliers

When scrutinising the results for outliers the following convention is adopted:

- If the TPM result is declared an outlier and excluded, then the nicotine, water and NFDPM results are excluded.
- If the nicotine result is declared an outlier and excluded, then the NFDPM result is excluded.
- If the water result is declared an outlier and excluded, then the NFDPM result is excluded.

The average level for each product, laboratory and parameter is shown in Form B (see Table F.17). The amount of spread within each cell, measured by the standard deviation between five runs, is shown in Form C (see Table F.18).

To assess the within laboratory variability, Cochran's test was applied to the results in Form C (see Table F.18).

In the first iteration of Cochran's test the following outliers and stragglers were identified.

Table F.8 — Outliers and stragglers

Product	Laboratory	Machine	Parameter	Variance	Cochran's	Grubb's
Camel	M	ASM516	TPM	1,363	Outlier	Outlier
Camel	M	ASM516	Water	0,304	Outlier	Outlier
Camel	AC	SM400	TNA	0,019	Outlier	Outlier
Camel	AY	RM20	CO	0,928	Outlier	Straggler
CM4	B	ASM516	TPM	2,067	Outlier	Outlier
CM4	AY	RM20	Water	0,194	Straggler	
CM4	AW	RM20	TNA	0,011	Outlier	
CM4	B	ASM516	NFDPM	1,97	Outlier	Outlier
CM4	AAA	RM200	CO	0,487	Straggler	
Ducados	AY	RM20	Puff number	0,085	Straggler	
Ducados	AY	RM20	TPM	1,039	Outlier	
Ducados	AY	RM20	Water	0,249	Outlier	
Ducados	AW	RM20	TNA	0,011	Outlier	
Ducados	AY	RM20	NFDPM	0,867	Outlier	
Marlboro	G1	RM20CSR	Puff number	0,135	Outlier	
Marlboro	AY	RM20	Water	0,43	Outlier	
Marlboro	AW	RM20	TNA	0,013	Outlier	
Marlboro	AY	RM20	NFDPM	1,056	Outlier	Straggler
Marlboro	AF	RM20CSR	CO	1,369	Outlier	Straggler
Marlboro Lights	AY	RM20	Puff number	0,147	Outlier	
Marlboro Lights	AY	RM20	Water	0,234	Outlier	
Marlboro Lights	AW	RM20	TNA	0,002	Outlier	
Marlboro Lights	AY	RM20	NFDPM	0,296	Outlier	
PM Super Lights	G1	RM20CSR	Puff number	0,154	Outlier	
PM Super Lights	AJ	SM400	TPM	0,207	Outlier	Straggler
PM Super Lights	AY	RM20	Water	0,214	Outlier	
PM Super Lights	AC	SM400	TNA	0,015	Outlier	Outlier
PM Super Lights	AJ	SM400	NFDPM	0,205	Straggler	Straggler
PM Super Lights	AJ	SM400	CO	0,307	Outlier	
Pall Mall 100	B	RM20	Puff number	0,983	Outlier	Straggler
Pall Mall 100	B	RM20	TPM	3,941	Outlier	Outlier
Pall Mall 100	AT	RM20CSR	Water	0,31	Outlier	Straggler
Pall Mall 100	AW	RM20	TNA	0,017	Outlier	Outlier
Pall Mall 100	B	RM20	NFDPM	2,117	Outlier	Outlier
Pall Mall 100	B	RM20	CO	1,662	Outlier	Outlier
Regal	AY	RM20	Water	0,202	Outlier	
Regal	AW	RM20	TNA	0,029	Outlier	
Regal	AK	SM400	CO	0,601	Straggler	

To determine if this relatively high level of variation can be explained by a single result, Grubb's test was applied to each set of five individual results. This test found outlying results, which were excluded from the analysis. The Cochran's outliers identified in red in the above table whose high level of variation could not be explained by a single result were then excluded from further analysis for the respective parameters, products and laboratories. Where water results were excluded, NFDPM results for the same laboratory and product were excluded. Where nicotine results were excluded, NFDPM results for the same laboratory and product were excluded. Where TPM results were excluded from further analysis, nicotine, water and NFDPM results were also excluded. Any results excluded using the above convention are shown in shaded boxes in Forms B and C (see Tables F.17 and F.18).

The second iteration of Cochran's test identified the outliers and stragglers given in Table F.9.

Table F.9

Product	Laboratory	Machine	Parameter	Variance	Cochran's
Camel	AY	RM20	Water	0,152	Outlier
Camel	AF	RM20CSR	Nicotine	0,006	Outlier
Camel	B	ASM516	TPM	0,844	Straggler
CM4	AC	SM400	Nicotine	0,010	Outlier
CM4	AAA	RM200	TPM	0,667	Straggler
CM4	AY	RM20	Water	0,194	Straggler
CM4	AAA	RM200	CO	0,487	Straggler
Ducados	AW	RM20	Water	0,226	Outlier
Ducados	AS	RM20	Nicotine	0,004	Outlier
Ducados	AY	RM20	Puff	0,085	Straggler
Ducados	AF	RM20CSR	TPM	0,512	Straggler
Marlboro	AF	RM20CSR	Water	0,213	Outlier
Marlboro	B	RM20	Nicotine	0,005	Outlier
Marlboro Lights	G2	RM20CSR	Puff	0,129	Outlier
Marlboro Lights	AY	RM20	Nicotine	0,001	Outlier
Marlboro Lights	C	RM20	NFDPM	0,221	Straggler
Pall Mall 100	AY	RM20	Puff	0,171	Outlier
Pall Mall 100	AC	SM400	Nicotine	0,007	Outlier
Pall Mall 100	M	RM20CSR	CO	1,366	Outlier
PM Super Lights	AT	SM400	Puff	0,112	Outlier
PM Super Lights	AF	RM20CSR	Water	0,092	Outlier
PM Super Lights	AY	RM20	Nicotine	0,001	Straggler
PM Super Lights	AN	RM200	CO	0,193	Straggler
Regal	G1	RM20CSR	Water	0,171	Outlier
Regal	AK	SM400	CO	0,601	Straggler

Grubb's test on the above results failed to identify any outliers. The outliers identified in red from Cochran's test were thus eliminated from further analysis. Where water or nicotine was identified as an outlier, NFDPM was also excluded for that product and laboratory.

The third iteration of Cochran's test identified the outliers and stragglers given in Table F.10.

Table F.10

Product	Laboratory	Machine	Parameter	Variance	Cochran's	Grubb's
CM4	AAA	RM200	TPM	0,6670	Straggler	
CM4	AAA	RM200	CO	0,4870	Straggler	
CM4	AY	RM20	WATER	0,1940	Straggler	
CM4	R	SM400	NICOTINE	0,0045	Straggler	
Camel	B	ASM516	TPM	0,8440	Straggler	
Camel	G2	RM20CSR	WATER	0,1411	Outlier	Straggler
Camel	AW	RM20	NICOTINE	0,0041	Straggler	
Ducados	AY	RM20	PUFF	0,0855	Straggler	
Ducados	AF	RM20CSR	TPM	0,5123	Straggler	
Marlboro	AS	RM20CSR	NICOTINE	0,0048	Outlier	
Marlboro light	G1	RM20CSR	PUFF	0,1176	Outlier	
Marlboro light	AJ	SM400	NFDPM	0,2205	Straggler	
PM Super Lights	AY	RM20	PUFF	0,0680	Straggler	Straggler
PM Super Lights	C	RM200	WATER	0,0405	Straggler	
PM Super Lights	AY	RM20	NICOTINE	0,0009	Straggler	Straggler
PM Super Lights	AN	RM200	CO	0,1935	Straggler	Straggler
Pall Mall 100	S	RM200	CO	0,8343	Outlier	
Regal	H	SM400	WATER	0,1131	Straggler	
Regal	AK	SM400	CO	0,6012	Straggler	

Grubb's test on the five individual results for these laboratories identified four stragglers. The outliers identified in red from Cochran's test were thus eliminated from further analysis. Where water or nicotine was identified as an outlier, NFDPM was also excluded for that product and laboratory.

The fourth iteration of Cochran's test identified the stragglers given in Table F.11.

Table F.11

Product	Laboratory	Machine	Parameter	Variance	Cochran's	Grubb's
CM4	AAA	RM200	TPM	0,667	Straggler	
CM4	AY	RM200	WATER	0,19398	Straggler	
CM4	R	SM400	TNA	0,00447	Straggler	
CM4	AAA	RM200	CO	0,487	Straggler	
Camel	B	ASM516	TPM	0,84403	Straggler	
Camel	AW	RM20	TNA	0,00405	Straggler	
Ducados	AY	RM20	PUFF	0,08547	Straggler	
Ducados	AF	RM20CSR	TPM	0,5123	Straggler	
Marlboro	AU	RM20CSR	TNA	0,21905	Straggler	Straggler
Marlboro Lights	AJ	SM400	NFDPM	0,22052	Straggler	
PM Super Lights	AY	RM20	PUFF	0,06803	Straggler	
PM Super Lights	C	RM200	WATER	0,04053	Straggler	
PM Super Lights	AY	RM20	TNA	0,00087	Straggler	
PM Super Lights	AN	RM200	CO	0,19348	Straggler	
Regal	H	SM400	WATER	0,11313	Straggler	
Regal	AK	SM400	CO	0,60117	Straggler	

Grubb's test on the five individual results for these laboratories identified one straggler. The stragglers identified were retained (ISO 5725-2:1994, 7.6.9).

Identified in Forms B and C (see Tables F.17 and F.18) are those results declared stragglers or outliers by Cochran's test. All stragglers were retained.

Following the completion of Cochran's test, Grubb's test for between laboratory variation was applied to the laboratory averages in Form B (see Table F.17).

Grubb's test identified the outliers and stragglers given in Table F.12.

Table F.12

Product	Parameter	Laboratory	Machine	Grubb's mean	Grubb's
CM4	Puff number	AJ	SM400	11,36	Outlier
CM4	TPM	AW	RM20	14,85	Outlier
Camel	Nicotine	AW	RM20	0,72	Outlier
Camel	Puff number	AJ	SM400	9,26	Outlier
Camel	Nicotine	B	SM400	1,13	Straggler
Ducados	Puff number	AJ	SM400	8,74	Outlier
Marlboro	Puff number	AJ	SM400	9,94	Outlier
Marlboro	TPM	AW	RM20	12,18	Outlier
Marlboro Lights	Puff number	AJ	SM400	9,44	Outlier
PM Super Lights	NFDPM	A	RM20CSR	2,56	Outlier
PM Super Lights	Puff number	C	RM200	9,77	Outlier
PM Super Lights	CO	C	RM200	5,30	Straggler
PM Super Lights	CO	R	SM400	2,98	Straggler
Pall Mall 100	CO	AJ	SM400	12,44	Outlier
Pall Mall 100	Nicotine	B	SM400	0,93	Outlier
Pall Mall 100	Puff number	AJ	SM400	12,44	Outlier
Regal	Puff number	AJ	SM400	9,20	Outlier

The outliers identified from Grubb's test highlighted in red above were removed from further analysis. Where nicotine or water was identified as outliers, NFDPM was also removed for that product and laboratory. Where TPM was identified as an outlier nicotine, water and NFDPM were also removed for that product and laboratory.

The second iteration of Grubb's test for between-laboratory variation identified the outliers and stragglers given in Table F.13.

Table F.13

Product	Laboratory	Machine	Parameter	Grubb's	
Camel	B	SM400	Nicotine	1,134	Outlier
PM Super Lights	AJ	SM400	Puff	9,768	Outlier
PM Super Lights	C	RM200	CO	5,30	Straggler
PM Super Lights	R	SM400	CO	2,98	Straggler

The outliers identified from Grubb's test highlighted in red above were removed from further analysis. Where nicotine was identified as an outlier, NFDPM was also removed for that product and laboratory. The stragglers identified were retained (ISO 5725-2:1994, 7.6.9).

F.8.6 Average levels, repeatability and reproducibility

After the exclusion of outlying results, the average levels and r and R statistics for each parameter were calculated. Table F.15 illustrates these figures calculated using all available results for each parameter and product. Table F.19 illustrates these figures calculated for each product and parameter according to smoking machine type.

F.8.7 Conditioned weight of the products

Two figures in the detailed report and not shown here illustrate the weights of each of the products for each laboratory.

To assess the within laboratory variability, Cochran's test was applied to the weight results in Form A of the original study description (not included in this report). The results of this test are shown in Table F.14.

Table F.14 — Outlier with respect to the within variability

Product	Laboratory	Machine	Parameter	Variance	Cochran's
CM4	R	SM400	Weight	529,5	Outlier
Pall Mall 100	C	RM200	Weight	204,9	Outlier
Regal	AY	RM20	Weight	436,1	Straggler

To determine if this relatively high level of variation can be explained by a single result, Grubb's test was applied to each set of five individual results. This test did not find any stragglers or outliers with the five results. The two outliers were thus excluded from further analysis. Table 2 of the original study description presents the average weight and standard deviation for each product/laboratory/machine.

F.8.8 Participants in CORESTA study Autumn 2003

In total 39 laboratories shown in Table F.16 participated in the study and results were reported from 61 smoking machines.

Table F.15 — Average levels, r and R

Parameter	Product	Average	r	R
CO	Camel	8,31	0,82	1,72
	CM4	13,23	0,87	2,20
	Ducados	10,99	1,00	2,11
	Marlboro	11,85	0,95	2,22
	Marlboro Lights	6,79	0,75	1,42
	Pall Mall 100	9,55	0,74	1,66
	PM Super Lights	4,12	0,52	1,08
	Regal	11,45	0,97	2,09
NFDPM	Camel	12,68	0,94	2,12
	CM4	14,06	0,74	1,73
	Ducados	10,07	0,66	1,24
	Marlboro	11,56	0,71	1,11
	Marlboro Lights	5,43	0,55	0,80
	Pall Mall 100	10,04	0,64	1,01
	PM Super Lights	3,54	0,49	0,75
	Regal	9,83	0,72	1,30
Nicotine	Camel	0,940	0,073	0,133
	CM4	1,298	0,078	0,145
	Ducados	0,817	0,068	0,123
	Marlboro	0,848	0,059	0,107
	Marlboro Lights	0,465	0,037	0,076
	Pall Mall 100	0,790	0,063	0,100
	PM Super Lights	0,334	0,035	0,073
	Regal	0,883	0,071	0,124
Puff	Camel	7,42	0,39	0,79
	CM4	9,06	0,37	0,93
	Ducados	7,33	0,34	0,62
	Marlboro	7,97	0,33	0,73
	Marlboro Lights	7,66	0,35	0,83
	Pall Mall 100	9,86	0,42	1,11
	PM Super Lights	7,88	0,33	0,80
	Regal	7,33	0,32	0,76
TPM	Camel	15,38	1,19	2,23
	CM4	17,62	1,01	1,77
	Ducados	12,08	0,88	1,67
	Marlboro	14,55	1,10	1,88
	Marlboro Lights	6,52	0,60	0,97
	Pall Mall 100	11,96	0,82	1,27
	PM Super Lights	4,27	0,49	0,85
	Regal	12,23	0,96	1,87
Water	Camel	1,79	0,37	0,90
	CM4	2,26	0,53	1,26
	Ducados	1,22	0,40	0,84
	Marlboro	2,12	0,49	1,21
	Marlboro Lights	0,63	0,29	0,51
	Pall Mall 100	1,13	0,37	0,75
	PM Super Lights	0,39	0,25	0,38
	Regal	1,52	0,43	0,98

Table F.16 — Participating laboratories

	ASM 516	SM 400	RM 200	RM 20	RM 20CSR	Other
Altadis France		1				
Altadis Spain			1	1		
Amer Tobacco			1			
Arista Europa		1				
Arista USA	1		1			
Austria Tabak			1		1	
BAT Germany			1		1	
BAT Netherlands			1			
BAT Southampton			1			
Borgwaldt			1			
Cigatam (Philip Morris Mexico)		1				
Compañía Colombiana de Tabaco		1				
ETI - Monital	1					
Filtrona	1	1		1		
Gallaher	1	1			1	
Chemische Landesuntersuchungsanstalt Sigmaringen				1		
Heinz van Landewyck					2	
House of Prince		1				
Inspectorate for Health Protection - Netherlands				1		
Institute of Public Health of the Republic of Slovenia				1		
Japan Tobacco			1			
Japan Tobacco International - Germany		1	1		1	
JL Tiedemans					1	
KC automation						1
Labstat	1					
Le Tabac Reconstitue				1	1	
Laboratoire National d'Essais (LNE), France		1				
Massalin Particulares - Philip Morris Argentina	1	1				
Philip Morris – Switzerland		1	1		1	
Philip Morris Australia			1			
Philip Morris Germany			2		1	
Philip Morris Holland			1			
Philip Morris Portugal				1		
Reemtsma			1		1	
RJ Reynolds		1				
Rothmans Benson & Hedges	1	1				
Sampoerna		1	1		1	
Tabacos de Canarias (CITA)				1	1	
Tvornica duhana Rovinj, Croatia			1			
Total	7	14	18	8	13	1

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Table F.17 — Form B

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Camel	A	Rotary	RM200	7,13	15,74	2,00	1,010	12,74	8,79
Camel	A	Rotary	RM20CSR	7,20	14,97	1,89	0,992	12,09	8,35
Camel	A	Linear	SM400	7,23	15,50	1,61	0,984	12,91	8,32
Camel	AAA	Rotary	RM200	7,35	14,70	1,83	0,900	11,96	8,84
Camel	AAB	Linear	ASM516	7,66	15,85	1,18	0,920	13,75	7,96
Camel	AAC	Rotary	RM200	7,01	15,94	1,72	1,032	13,20	8,46
Camel	AAD	Rotary	RM20	7,60	16,06	2,22	0,994	12,84	7,84
Camel	AC	Linear	SM400	7,47	16,44	2,02	1.054**A(1)	13,37	8,72
Camel	AF	Rotary	RM20CSR	7,06	15,40	2,02	0.877**V(2)	12,48	8,63
Camel	AG	Linear	ASM516	7,30	15,52	1,32	0,964	13,24	8,25
Camel	AJ	Linear	SM400	9,26**	15,56	1,60	0,920	13,05	9,26
Camel	AK	Linear	ASM516	7,38	15,77	1,35	0,934	13,49	8,25
Camel	AK	Linear	SM400	7,46	16,62	1,79	0,966	13,86	8,19
Camel	AN	Linear	ASM516	7,72	16,79	1,37	0,998	14,42	8,82
Camel	AP	Rotary	RM20CSR	7,36	15,48	2,24	0,984	12,30	8,25
Camel	AQ	Linear	ASM516	7,29	14,82	1,22	0,932	12,67	7,97
Camel	AQ	Linear	SM400	7,43	15,46	1,43	0,994	13,04	7,94
Camel	AS	Rotary	RM20	7,19	15,65	2,06	0,924	12,67	9,48
Camel	AS	Rotary	RM20CSR	7,39	14,67	1,85	0,948	11,93	8,65
Camel	AT	Rotary	RM200	7,34	13,78	2,05	0,852	10,87	7,53
Camel	AT	Rotary	RM20CSR	7,27	14,81	2,12	0,904	11,79	8,18
Camel	AT	Linear	SM400	7,78	16,40	1,72	0,946	13,74	7,67
Camel	AU	Rotary	RM20CSR	7,80	16,65	1,98	0,930	13,74	8,96
Camel	AV	Rotary	RM200	7,78	15,30	2,05	0,866	12,39	8,87
Camel	AW	Rotary	RM20	6,75	13,93	1,51	0,720*V(3) #	11,70	7,83
Camel	AX	Rotary	RM200	7,58	15,98	2,17	0,980	12,83	9,02
Camel	AY	Rotary	RM20	7,15	14,43	1,77**V(2)	0,884	11,78	8,36*A(1)
Camel	AZ	Linear	SM400	7,20	14,66	1,23	0,894	12,54	7,52
Camel	B	Linear	ASM516	7,80	15,15*V(2)	1,26	0,886	13,00	7,92

#Also an outlier due to mean level

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Camel	B	Rotary	RM20	7,82	14,99	1,54	0,928	12,52	8,43
Camel	B	Linear	SM400	7,52	16,29	1,64	1,134***	13,52	7,82
Camel	C	Rotary	RM20	7,80	15,66	2,14	0,918	12,46	8,90
Camel	C	Rotary	RM200	7,88	15,89	2,31	0,926	12,65	9,88
Camel	D	Linear	SM400	7,46	16,63	1,91	0,920	13,80	7,88
Camel	E	Rotary	RM200	7,16	14,95	1,91	0,932	12,11	8,72
Camel	F	Rotary	RM200	7,56	15,53	2,04	0,944	12,54	8,29
Camel	F	Rotary	RM20CSR	7,52	15,95	2,28	0,974	12,70	8,64
Camel	F	Linear	SM400	7,55	16,71	1,78	0,986	13,95	7,87
Camel	G1	Rotary	RM20CSR	7,41	14,51	1,92	0,980	11,60	7,94
Camel	G2	Rotary	RM20CSR	7,32	14,94	2,00**V(3)	0,998	11,94	8,06
Camel	H	Linear	SM400	7,32	16,12	1,49	1,008	13,62	8,58
Camel	I	Rotary	RM200	7,09	14,99	2,02	0,906	12,06	8,14
Camel	L	Rotary	RM20	7,38	15,79	1,86	0,974	12,96	8,64
Camel	L	Rotary	RM20CSR	7,02	14,80	1,79	0,910	12,10	8,28
Camel	M	Linear	ASM516	7,66	15,56**A(1)	1,74**A(1)	0,902	12,92	7,65
Camel	M	Rotary	RM20CSR	7,41	14,78	1,82	0,898	12,07	7,61
Camel	M	Linear	SM400	7,43	15,23	1,58	0,870	12,78	7,70
Camel	N	Rotary	RM20CSR	7,86	15,27	1,89	0,934	12,45	8,62
Camel	N1	Rotary	RM200	7,36	14,99	1,90	0,918	12,18	7,61
Camel	N2	Rotary	RM200	7,62	14,88	1,84	0,926	12,11	8,21
Camel	Q	Rotary	RM20	7,41	15,45	1,93	0,914	12,60	8,84
Camel	R	Linear	SM400	7,90	14,37	1,26	0,922	12,19	6,87
Camel	S	Rotary	RM200	7,27	15,19	1,86	0,936	12,39	7,92
Camel	S	Rotary	RM20CSR	7,66	14,71	1,82	0,912	11,98	7,66
Camel	V	Rotary	RM200	7,31	15,01	1,92	0,938	12,16	9,02
Camel	V	Rotary	RM20CSR	7,22	14,67	1,69	0,956	12,02	8,20
Camel	Z	Rotary	RM200	7,15	15,55	2,17	0,928	12,45	8,86
CM4	A	Rotary	RM200	8,83	17,48	2,41	1,330	13,75	14,11

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
CM4	A	Rotary	RM20CSR	8,86	18,11	2,75	1,342	14,02	14,48
CM4	A	Linear	SM400	9,12	17,38	1,90	1,340	14,14	12,88
CM4	AAA	Rotary	RM200	9,07	17,94 ^{*V(2)}	2,58	1,284	14,08	14,38 ^{*V(1)}
CM4	AAB	Linear	ASM516	9,70	17,93	1,50	1,332	15,10	13,18
CM4	AAC	Rotary	RM200	8,67	16,77	1,97	1,342	13,45	12,58
CM4	AAD	Rotary	RM20	9,29	17,60	2,34	1,338	13,93	13,00
CM4	AC	Linear	SM400	9,00	17,77	2,14	1,276 ^{**V(2)}	14,35	12,76
CM4	AF	Rotary	RM20CSR	8,76	18,08	2,81	1,243	14,03	14,09
CM4	AG	Linear	ASM516	9,13	17,22	1,52	1,338	14,37	13,45
CM4	AJ	Linear	SM400	11,36 ^{**}	18,03	1,84	1,302	14,89	11,36
CM4	AK	Linear	ASM516	9,26	17,55	1,75	1,334	14,47	12,81
CM4	AK	Linear	SM400	9,22	17,76	2,00	1,328	14,43	12,81
CM4	AN	Linear	ASM516	9,64	18,54	1,63	1,412	15,50	13,53
CM4	AN	Rotary	RM200	8,77	17,30	2,11	1,334	13,86	12,77
CM4	AP	Rotary	RM200	8,72	17,22	2,49	1,248	13,49	13,64
CM4	AQ	Linear	ASM516	9,31	16,98	1,51	1,364	14,11	13,19
CM4	AQ	Linear	SM400	9,42	17,57	1,75	1,380	14,44	13,28
CM4	AS	Rotary	RM20	8,89	17,65	2,46	1,310	13,76	13,74
CM4	AS	Rotary	RM20CSR	8,93	17,85	2,46	1,310	14,09	14,02
CM4	AT	Rotary	RM200	9,27	17,31	2,92	1,260	13,14	12,87
CM4	AT	Rotary	RM20CSR	9,17	18,13	3,00	1,312	13,82	13,49
CM4	AT	Linear	SM400	9,37	18,25	2,06	1,280	14,91	11,03
CM4	AU	Rotary	RM20CSR	9,18	18,35	2,32	1,234	14,97	13,67
CM4	AV	Rotary	RM200	9,32	17,41	2,45	1,252	13,71	14,10
CM4	AW	Rotary	RM20	8,30	14,85 ^{**}	1,67	1,112 ^{**V(1)}	12,06	11,94
CM4	AX	Rotary	RM200	8,72	16,82	2,20	1,284	13,34	13,47
CM4	AY	Rotary	RM20	8,49	16,34	2,07 ^{*V(1)}	1,204	13,06	12,80
CM4	AZ	Linear	SM400	8,97	17,53	1,70	1,252	14,54	12,68
CM4	B	Linear	ASM516	9,66	18,17 ^{**A(1)}	1,66	1,312	15,20 ^{**A(1)}	12,74

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
CM4	B	Rotary	RM20	9,30	18,01	2,46	1,292	14,26	14,25
CM4	B	Linear	SM400	9,02	17,68	1,62	1,296	14,76	12,22
CM4	C	Rotary	RM20	9,45	17,30	2,51	1,258	13,53	13,88
CM4	C	Rotary	RM200	9,26	17,40	2,58	1,278	13,60	13,68
CM4	D	Linear	SM400	9,17	18,45	2,40	1,316	14,73	12,24
CM4	E	Rotary	RM200	8,75	16,86	2,28	1,288	13,29	13,88
CM4	F	Rotary	RM200	9,08	17,55	2,60	1,276	13,68	13,36
CM4	F	Rotary	RM20CSR	9,18	18,71	3,03	1,320	14,36	13,68
CM4	F	Linear	SM400	9,26	18,49	2,20	1,340	14,95	12,21
CM4	G1	Rotary	RM20CSR	8,99	17,71	2,91	1,368	13,44	13,39
CM4	G2	Rotary	RM20CSR	8,90	18,34	3,04	1,390	13,91	13,51
CM4	H	Linear	SM400	9,03	18,09	1,80	1,362	14,93	13,37
CM4	I	Rotary	RM200	8,69	17,29	2,43	1,238	13,62	12,94
CM4	L	Rotary	RM20	8,98	17,71	2,26	1,316	14,13	13,86
CM4	L	Rotary	RM20CSR	8,72	18,17	2,58	1,278	14,32	13,79
CM4	M	Linear	ASM516	9,72	17,98	1,89	1,332	14,76	12,95
CM4	M	Rotary	RM20CSR	8,90	18,39	2,68	1,266	14,44	13,62
CM4	M	Linear	SM400	8,87	17,44	2,17	1,210	14,06	12,59
CM4	N	Rotary	RM20CSR	9,48	17,77	2,37	1,310	14,09	14,04
CM4	N1	Rotary	RM200	9,08	17,19	2,35	1,268	13,57	13,14
CM4	N2	Rotary	RM200	9,16	16,97	2,41	1,248	13,32	13,64
CM4	Q	Rotary	RM20	8,97	17,77	2,42	1,266	14,09	14,06
CM4	R	Linear	SM400	9,64	16,13	1,44	1,268 ^{*V(3)}	13,42	11,56
CM4	S	Rotary	RM200	8,95	17,16	2,33	1,270	13,56	13,18
CM4	S	Rotary	RM20CSR	9,31	17,81	2,67	1,258	13,89	13,13
CM4	V	Rotary	RM200	8,81	17,28	2,47	1,268	13,55	13,82
CM4	V	Rotary	RM20CSR	8,72	17,39	2,39	1,298	13,70	13,50
CM4	Z	Rotary	RM200	8,53	16,79	2,42	1,230	13,14	13,08
Ducados	A	Rotary	RM200	7,19	12,26	1,30	0,848	10,12	11,57

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Ducados	A	Rotary	RM20CSR	7,30	12,69	1,47	0,856	10,37	12,21
Ducados	A	Linear	SM400	7,21	11,92	1,13	0,876	9,92	11,00
Ducados	AAA	Rotary	RM200	7,36	12,35	1,42	0,812	10,12	11,36
Ducados	AAB	Linear	ASM516	7,21	12,05	0,71	0,766	10,57	10,96
Ducados	AAD	Rotary	RM20	7,67	12,04	1,23	0,886	9,93	10,02
Ducados	AC	Linear	SM400	7,21	12,28	1,16	0,804	10,32	10,65
Ducados	AF	Rotary	RM20CSR	7,26	12,96 ^{*V(2)}	1,63	0,826	10,51	12,77
Ducados	AG	Linear	ASM516	7,16	11,60	0,89	0,852	9,86	11,15
Ducados	AJ	Linear	SM400	8,74 ^{**}	11,47	0,79	0,766	9,92	9,31
Ducados	AK	Linear	ASM516	7,26	11,76	0,89	0,828	10,05	10,48
Ducados	AK	Linear	SM400	7,22	12,16	1,06	0,826	10,27	10,62
Ducados	AN	Linear	ASM516	7,48	12,52	0,86	0,876	10,79	11,20
Ducados	AN	Rotary	RM200	7,24	11,83	1,13	0,850	9,85	10,24
Ducados	AP	Rotary	RM200	7,20	11,73	1,24	0,792	9,70	11,02
Ducados	AQ	Linear	ASM516	7,21	11,16	0,76	0,812	9,59	10,45
Ducados	AQ	Linear	SM400	7,32	11,96	0,94	0,850	10,16	10,66
Ducados	AS	Rotary	RM20	7,21	12,06	1,50	0,782 ^{**V(2)}	9,78	11,57
Ducados	AS	Rotary	RM20CSR	7,24	12,63	1,60	0,798	10,23	11,92
Ducados	AT	Rotary	RM200	7,55	12,09	1,71	0,776	9,61	10,53
Ducados	AT	Rotary	RM20CSR	7,44	12,57	1,79	0,810	9,98	11,34
Ducados	AT	Linear	SM400	7,74	12,84	1,33	0,790	10,72	9,80
Ducados	AV	Rotary	RM200	7,35	11,57	1,15	0,756	9,66	11,22
Ducados	AW	Rotary	RM20	6,90	10,68	1,12 ^{**V(2)}	0,650 ^{**V(1)}	8,91	9,75
Ducados	AX	Rotary	RM200	7,21	11,27	1,04	0,814	9,41	10,86
Ducados	AY	Rotary	RM20	6,91 ^{*V(1)}	11,50 ^{**V(1)}	1,31 ^{**V(1)}	0,772	9,41 ^{**V(1)}	11,16
Ducados	AZ	Linear	SM400	7,30	12,05	0,77	0,830	10,44	10,55
Ducados	B	Linear	ASM516	7,54	12,07	0,86	0,794	10,42	10,48
Ducados	B	Rotary	RM20	7,76	12,16	1,12	0,858	10,18	12,36
Ducados	B	Linear	SM400	7,40	12,85	1,02	0,854	10,98	11,02
Ducados	C	Rotary	RM20	7,60	11,78	1,42	0,766	9,59	11,22

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Ducados	C	Rotary	RM200	7,54	11,69	1,29	0,792	9,61	11,14
Ducados	D	Linear	SM400	7,40	13,09	1,52	0,846	10,72	10,32
Ducados	E	Rotary	RM200	7,17	11,60	1,23	0,798	9,57	11,12
Ducados	F	Rotary	RM200	7,38	11,83	1,26	0,800	9,77	10,71
Ducados	F	Rotary	RM20CSR	7,41	12,63	1,59	0,818	10,22	11,29
Ducados	F	Linear	SM400	7,28	12,84	1,22	0,844	10,78	10,25
Ducados	G1	Rotary	RM20CSR	7,49	12,83	1,71	0,890	10,23	11,76
Ducados	G2	Rotary	RM20CSR	7,42	12,64	1,57	0,880	10,19	11,60
Ducados	H	Linear	SM400	7,09	12,01	0,86	0,818	10,34	11,05
Ducados	I	Rotary	RM200	7,05	11,98	1,29	0,818	9,87	11,00
Ducados	L	Rotary	RM20	7,47	11,96	1,12	0,842	10,00	11,32
Ducados	L	Rotary	RM20CSR	7,24	12,58	1,33	0,848	10,39	11,64
Ducados	M	Linear	ASM516	7,45	12,34	1,05	0,812	10,48	11,09
Ducados	M	Rotary	RM20CSR	7,36	12,80	1,58	0,812	10,41	11,40
Ducados	M	Linear	SM400	7,16	12,14	1,17	0,764	10,21	10,34
Ducados	N	Rotary	RM20CSR	7,56	12,00	1,21	0,814	9,97	11,84
Ducados	N1	Rotary	RM200	7,38	11,65	1,28	0,794	9,58	10,80
Ducados	N2	Rotary	RM200	7,58	11,94	1,38	0,818	9,74	11,84
Ducados	Q	Rotary	RM20	7,23	11,78	1,30	0,734	9,75	11,26
Ducados	R	Linear	SM400	7,74	10,59	0,66	0,696	9,24	9,36
Ducados	S	Rotary	RM200	7,25	11,74	1,16	0,812	9,77	10,40
Ducados	S	Rotary	RM20CSR	7,51	12,14	1,34	0,816	9,99	11,03
Ducados	V	Rotary	RM200	7,11	11,95	1,23	0,798	9,92	11,24
Ducados	V	Rotary	RM20CSR	7,15	12,39	1,27	0,836	10,29	11,36
Ducados	Z	Rotary	RM200	7,16	11,71	1,31	0,804	9,59	10,74
Marlboro	A	Rotary	RM200	7,81	15,01	2,53	0,910	11,58	12,85
Marlboro	A	Rotary	RM20CSR	7,78	14,91	2,17	0,894	11,85	12,96
Marlboro	A	Linear	SM400	8,00	14,64	1,98	0,868	11,80	11,70
Marlboro	AAA	Rotary	RM200	8,06	14,78	2,45	0,838	11,49	12,26

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Marlboro	AAB	Linear	ASM516	8,06	14,00	1,42	0,814	11,77	11,88
Marlboro	AAC	Rotary	RM200	7,72	14,95	2,01	0,944	12,00	11,70
Marlboro	AAD	Rotary	RM20	8,13	14,53	1,91	0,900	11,71	10,82
Marlboro	AC	Linear	SM400	7,95	14,45	2,04	0,858	11,56	11,64
Marlboro	AF	Rotary	RM20CSR	7,86	15,47	2,67**V(2)	0,815	11,97	12,86**A(1)
Marlboro	AG	Linear	ASM516	7,72	13,71	1,45	0,862	11,40	11,95
Marlboro	AJ	Linear	SM400	9,94**	14,44	1,62	0,844	11,97	9,94
Marlboro	AK	Linear	ASM516	8,07	14,04	1,47	0,852	11,72	11,67
Marlboro	AK	Linear	SM400	8,05	14,70	1,92	0,864	11,92	11,79
Marlboro	AN	Linear	ASM516	8,21	14,45	1,52	0,868	12,05	12,02
Marlboro	AN	Rotary	RM200	7,83	14,33	2,04	0,878	11,41	11,54
Marlboro	AP	Rotary	RM200	7,81	14,44	2,44	0,838	11,17	12,02
Marlboro	AQ	Linear	ASM516	7,88	13,14	1,27	0,850	11,02	11,29
Marlboro	AQ	Linear	SM400	8,04	13,95	1,60	0,872	11,48	11,52
Marlboro	AS	Rotary	RM20	7,82	14,87	2,30	0,844	11,80	12,58
Marlboro	AS	Rotary	RM20CSR	7,96	15,25	2,11	0,814**V(3)	12,33	12,86
Marlboro	AT	Rotary	RM200	8,28	14,86	2,73	0,856	11,27	11,84
Marlboro	AT	Rotary	RM20CSR	8,14	15,43	2,87	0,880	11,68	12,33
Marlboro	AT	Linear	SM400	8,52	15,23	2,29	0,842	12,10	10,16
Marlboro	AU	Rotary	RM20CSR	8,14	15,52	2,53	0,824**V(4)	12,04	12,60
Marlboro	AV	Rotary	RM200	8,24	14,44	2,31	0,802	11,33	12,70
Marlboro	AW	Rotary	RM20	7,30	12,18**	1,46	0,620**V(1)	10,10	9,82
Marlboro	AX	Rotary	RM200	7,77	13,95	2,02	0,826	11,10	12,03
Marlboro	AY	Rotary	RM20	7,44	13,63	2,01**V(1)	0,790	10,83**V(1)	12,00
Marlboro	AZ	Linear	SM400	7,87	14,03	1,66	0,800	11,58	11,03
Marlboro	B	Linear	ASM516	8,26	14,25	1,68	0,812	11,76	11,50
Marlboro	B	Rotary	RM20	8,32	14,93	2,48	0,846**V(2)	11,80	12,74
Marlboro	B	Linear	SM400	7,74	14,21	1,66	0,786	11,76	10,78
Marlboro	C	Rotary	RM20	8,37	14,12	2,19	0,822	11,10	12,42

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Marlboro	C	Rotary	RM200	8,40	14,73	2,49	0,852	11,39	12,50
Marlboro	D	Linear	SM400	7,91	15,10	2,31	0,822	11,98	11,06
Marlboro	E	Rotary	RM200	7,74	14,24	2,22	0,856	11,17	12,44
Marlboro	F	Rotary	RM200	7,90	14,22	2,21	0,836	11,17	11,58
Marlboro	F	Rotary	RM20CSR	7,96	15,28	2,65	0,870	11,76	12,18
Marlboro	F	Linear	SM400	8,05	15,42	2,26	0,868	12,29	11,08
Marlboro	G1	Rotary	RM20CSR	8,26**V(1)	15,69	3,13	0,934	11,63	12,48
Marlboro	G2	Rotary	RM20CSR	7,98	15,16	2,76	0,916	11,49	12,08
Marlboro	H	Linear	SM400	7,79	14,19	1,58	0,846	11,77	11,81
Marlboro	I	Rotary	RM200	7,73	14,48	2,28	0,832	11,38	11,65
Marlboro	L	Rotary	RM20	7,98	15,01	2,24	0,860	11,91	12,60
Marlboro	L	Rotary	RM20CSR	7,79	14,97	2,32	0,840	11,81	12,16
Marlboro	M	Linear	ASM516	8,04	13,74	1,75	0,812	11,18	11,23
Marlboro	M	Rotary	RM20CSR	7,94	15,13	2,47	0,858	11,81	12,01
Marlboro	M	Linear	SM400	7,82	14,48	2,15	0,806	11,52	11,34
Marlboro	N	Rotary	RM20CSR	8,30	14,80	2,24	0,844	11,72	12,82
Marlboro	N1	Rotary	RM200	7,98	14,15	2,10	0,840	11,21	11,80
Marlboro	N2	Rotary	RM200	8,08	13,93	2,00	0,834	11,10	11,96
Marlboro	Q	Rotary	RM20	7,75	14,49	2,17	0,818	11,51	12,38
Marlboro	R	Linear	SM400	8,58	12,99	1,37	0,828	10,80	10,68
Marlboro	S	Rotary	RM200	7,94	14,27	2,11	0,846	11,32	11,36
Marlboro	S	Rotary	RM20CSR	8,09	14,41	2,25	0,824	11,33	11,67
Marlboro	V	Rotary	RM200	7,81	14,34	2,19	0,848	11,30	12,22
Marlboro	V	Rotary	RM20CSR	7,80	14,63	2,19	0,872	11,57	12,14
Marlboro	Z	Rotary	RM200	7,84	14,72	2,53	0,846	11,34	12,14
Marlboro Lights	A	Rotary	RM200	7,37	6,71	0,75	0,506	5,46	7,30
Marlboro Lights	A	Rotary	RM20CSR	7,42	6,59	0,69	0,496	5,41	7,18
Marlboro Lights	A	Linear	SM400	7,68	6,24	0,52	0,474	5,25	6,66
Marlboro Lights	AAA	Rotary	RM200	7,77	7,34	0,95	0,492	5,89	7,76

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Marlboro Lights	AAB	Linear	ASM516	7,90	6,49	0,38	0,454	5,66	6,77
Marlboro Lights	AAC	Rotary	RM200	7,12	6,37	0,41	0,506	5,45	6,36
Marlboro Lights	AAD	Rotary	RM20	7,75	6,69	0,32	0,514	5,86	6,24
Marlboro Lights	AC	Linear	SM400	7,69	6,48	0,57	0,460	5,45	6,67
Marlboro Lights	AF	Rotary	RM20CSR	7,64	6,58	0,68	0,392	5,44	7,17
Marlboro Lights	AG	Linear	ASM516	7,51	6,01	0,51	0,482	5,02	6,71
Marlboro Lights	AJ	Linear	SM400	9,44**	6,56	0,54	0,410	5,62**V(3)	5,88
Marlboro Lights	AK	Linear	ASM516	7,62	6,21	0,56	0,446	5,21	6,52
Marlboro Lights	AK	Linear	SM400	7,69	6,40	0,51	0,456	5,43	6,83
Marlboro Lights	AN	Linear	ASM516	7,92	6,60	0,42	0,466	5,72	6,53
Marlboro Lights	AN	Rotary	RM200	7,37	6,45	0,54	0,484	5,43	6,13
Marlboro Lights	AP	Rotary	RM200	7,34	6,45	0,65	0,462	5,34	6,74
Marlboro Lights	AQ	Linear	ASM516	7,51	6,15	0,59	0,474	5,08	6,60
Marlboro Lights	AQ	Linear	SM400	7,73	6,25	0,48	0,478	5,30	6,51
Marlboro Lights	AS	Rotary	RM20	7,44	6,82	0,90	0,488	5,43	7,20
Marlboro Lights	AS	Rotary	RM20CSR	7,83	6,70	0,94	0,502	5,26	7,63
Marlboro Lights	AT	Rotary	RM200	7,69	6,80	1,10	0,458	5,24	6,64
Marlboro Lights	AT	Rotary	RM20CSR	7,79	6,67	0,90	0,464	5,30	7,06
Marlboro Lights	AT	Linear	SM400	8,06	6,39	0,70	0,452	5,24	6,29
Marlboro Lights	AU	Rotary	RM20CSR	7,83	6,79	0,60	0,440	5,75	7,32
Marlboro Lights	AV	Rotary	RM200	7,75	6,62	0,65	0,442	5,54	7,14
Marlboro Lights	AW	Rotary	RM20	6,91	5,78	0,43	0,382**V(1)	4,97	6,17
Marlboro Lights	AX	Rotary	RM200	7,42	6,32	0,60	0,474	5,25	6,74
Marlboro Lights	AY	Rotary	RM20	7,33**V(1)	6,53	0,85**V(1)	0,464**V(2)	5,22**V(1)	7,10
Marlboro Lights	AZ	Linear	SM400	7,72	6,24	0,36	0,464	5,40	6,32
Marlboro Lights	B	Linear	ASM516	7,96	6,50	0,42	0,462	5,58	6,74
Marlboro Lights	B	Rotary	RM20	7,80	7,15	0,82	0,472	5,86	7,49
Marlboro Lights	B	Linear	SM400	7,82	7,04	0,66	0,464	5,92	6,78
Marlboro Lights	C	Rotary	RM20	8,02	6,44	0,67	0,436	5,34**V(2)	6,86

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Marlboro Lights	C	Rotary	RM200	8,02	6,73	0,75	0,460	5,52	7,80
Marlboro Lights	D	Linear	SM400	7,59	6,26	0,67	0,432	5,15	6,16
Marlboro Lights	E	Rotary	RM200	7,30	6,34	0,58	0,456	5,30	6,94
Marlboro Lights	F	Rotary	RM200	7,62	6,48	0,64	0,466	5,37	6,54
Marlboro Lights	F	Rotary	RM20CSR	7,76	6,84	0,75	0,488	5,60	6,86
Marlboro Lights	F	Linear	SM400	7,79	6,62	0,55	0,470	5,60	6,19
Marlboro Lights	G1	Rotary	RM20CSR	8,21 ^{**V(3)}	6,67	0,79	0,496	5,38	7,12
Marlboro Lights	G2	Rotary	RM20CSR	7,82 ^{**V(2)}	6,87	0,83	0,512	5,53	7,10
Marlboro Lights	H	Linear	SM400	7,57	6,60	0,42	0,482	5,70	7,02
Marlboro Lights	I	Rotary	RM200	7,35	6,84	0,73	0,474	5,65	6,95
Marlboro Lights	L	Rotary	RM20	7,44	6,73	0,79	0,464	5,48	7,14
Marlboro Lights	L	Rotary	RM20CSR	7,32	6,50	0,65	0,456	5,40	6,92
Marlboro Lights	M	Linear	ASM516	8,13	6,50	0,51	0,468	5,52	6,87
Marlboro Lights	M	Rotary	RM20CSR	7,94	6,63	0,73	0,456	5,45	6,82
Marlboro Lights	M	Linear	SM400	7,53	6,11	0,53	0,424	5,15	6,24
Marlboro Lights	N	Rotary	RM20CSR	8,14	6,43	0,62	0,466	5,35	7,11
Marlboro Lights	N1	Rotary	RM200	7,74	6,39	0,65	0,464	5,28	6,53
Marlboro Lights	N2	Rotary	RM200	7,86	6,25	0,64	0,458	5,15	6,63
Marlboro Lights	Q	Rotary	RM20	7,53	6,76	0,70	0,458	5,60	7,42
Marlboro Lights	R	Linear	SM400	8,24	5,79	0,47	0,412	4,91	5,58
Marlboro Lights	S	Rotary	RM200	7,48	6,67	0,60	0,466	5,61	7,03
Marlboro Lights	S	Rotary	RM20CSR	8,10	6,09	0,75	0,434	4,91	6,41
Marlboro Lights	V	Rotary	RM200	7,41	6,52	0,66	0,472	5,39	6,84
Marlboro Lights	V	Rotary	RM20CSR	7,55	6,45	0,63	0,478	5,34	7,06
Marlboro Lights	Z	Rotary	RM200	7,18	6,52	0,71	0,468	5,35	6,78
Pall Mall 100	A	Rotary	RM200	9,66	12,56	1,34	0,832	10,39	10,34
Pall Mall 100	A	Rotary	RM20CSR	9,71	12,11	1,22	0,822	10,07	10,11
Pall Mall 100	A	Linear	SM400	9,87	11,93	0,91	0,840	10,18	9,43
Pall Mall 100	AAA	Rotary	RM200	9,72	12,47	1,59	0,772	10,10	10,10

Table F.17 (continued)

Product	Laboratory	Type	Machine	Puff number	TPM	Water	Nicotine	NFDPM	CO
Pall Mall 100	AAB	Linear	ASM516	10,48	12,15	0,70	0,820	10,63	9,50
Pall Mall 100	AAC	Rotary	RM200	9,12	11,75	0,84	0,846	10,07	9,04
Pall Mall 100	AAD	Rotary	RM20	10,03	12,23	1,26	0,830	10,15	9,00
Pall Mall 100	AC	Linear	SM400	9,71	11,73	0,95	0,778**V(2)	10,00	9,15
Pall Mall 100	AF	Rotary	RM20CSR	9,64	12,05	1,39	0,702	9,96	10,12
Pall Mall 100	AG	Linear	ASM516	9,93	11,47	0,84	0,810	9,82	9,22
Pall Mall 100	AJ	Linear	SM400	12,44**	12,11	0,91	0,760	10,43	12,44**
Pall Mall 100	AK	Linear	ASM516	10,13	11,65	0,87	0,794	9,99	9,06
Pall Mall 100	AK	Linear	SM400	10,01	11,79	0,96	0,810	10,01	9,23
Pall Mall 100	AN	Linear	ASM516	10,29	11,89	0,71	0,828	10,36	9,22
Pall Mall 100	AN	Rotary	RM200	9,27	11,50	1,01	0,790	9,70	8,99
Pall Mall 100	AP	Rotary	RM200	9,39	12,12	1,25	0,794	10,08	9,82
Pall Mall 100	AQ	Linear	ASM516	10,10	11,55	0,84	0,840	9,87	9,39
Pall Mall 100	AQ	Linear	SM400	9,97	11,54	0,87	0,838	9,83	9,21
Pall Mall 100	AS	Rotary	RM20	9,31	12,40	1,42	0,792	9,98	9,90
Pall Mall 100	AS	Rotary	RM20CSR	9,69	11,89	1,31	0,796	9,78	10,13
Pall Mall 100	AT	Rotary	RM200	9,62	11,96	1,66	0,808	9,49	9,22
Pall Mall 100	AT	Rotary	RM20CSR	9,95	11,75	1,72	0,808	9,23	9,15
Pall Mall 100	AT	Linear	SM400	10,33	11,71	1,17*A(1)	0,796	9,75	8,98
Pall Mall 100	AU	Rotary	RM20CSR	10,03	12,42	1,14	0,770	10,51	10,22
Pall Mall 100	AV	Rotary	RM200	10,16	11,92	1,18	0,752	9,99	10,53
Pall Mall 100	AW	Rotary	RM20	8,84	10,86	0,81	0,678**A(1)	9,38	8,79
Pall Mall 100	AX	Rotary	RM200	9,66	11,83	1,06	0,776	10,00	9,94
Pall Mall 100	AY	Rotary	RM20	9,20**V(2)	11,97	1,24	0,758	9,97	9,92
Pall Mall 100	AZ	Linear	SM400	9,83	11,76	0,81	0,740	10,21	9,18
Pall Mall 100	B	Linear	ASM516	10,46	11,70	0,76	0,804	10,14	9,04
Pall Mall 100	B	Rotary	RM20	10,86*A(1)	12,76**A(1)	1,20	0,836	10,72**A(1)	10,57**A(1)
Pall Mall 100	B	Linear	SM400	10,06	12,21	0,92	0,932**	10,36	9,24
Pall Mall 100	C	Rotary	RM20	10,12	12,19	1,45	0,770	9,97	10,08