

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

ICS 65.160

National foreword

This Published Document is the UK implementation of ISO/TR 22305:2008.

The UK participation in its preparation was entrusted to Technical Committee AW/40, Tobacco and tobacco products.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 December 2008

© BSI 2008

ISBN 978 0 580 57090 2

Amendments/corrigenda issued since publication

Date	Comments

TECHNICAL REPORT

**ISO/TR
22305**

First edition
2006-06-01

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



Reference number
ISO/TR 22305:2006(E)

AVA Documents
Document Sharing Hub
© ISO 2006

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2006

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Statistical functions for repeatability (r), reproducibility (R) of yield measurements and compliance tolerances for declared smoke constituent yields	1
2.1 Statistical functions for repeatability (r) and reproducibility (R)	1
2.2 A statistical model for compliance tolerances	2
3 Sources of data	3
3.1 International collaborative studies	3
3.2 UK Department of Health Cigarette Survey data	4
4 Comparison of 2003 CORESTA Collaborative Study data with those previously reported.....	4
4.1 General.....	4
4.2 Comparison of repeatability r_{20} values from CCS-03 with other collaborative studies	4
4.3 Comparison of reproducibility R_{20} values from CCS-03 with other collaborative studies	5
4.3.1 General.....	5
4.3.2 Relationship between reproducibility R_{20} and smoke constituent yield	5
4.4 Comparison of R_{100} reproducibility values from collaborative studies with measurement tolerances estimated from the UK Department of Health Cigarette Survey data	5
5 Review of information relevant to setting a compliance tolerance for carbon monoxide	6
5.1 General.....	6
5.2 Compliance data for current tolerances.....	6
5.3 Confidence intervals associated with yield measurements.....	6
5.4 Statistical models	7
5.5 Prediction of a tolerance for CO from the relative variability in their reproducibility values	8
6 Conclusions	8
7 Recommendations.....	8
Annex A (informative) Background considerations on the choice of sampling procedures	31
Annex B (informative) The determination of carbon monoxide in cigarette smoke — Problems in the evaluation of results	33
Annex C (informative) Proposals from the UK Tobacco Manufacturers Association for a practicable tolerance for verifying cigarette packet declarations of carbon monoxide (March, 2002)	41
Annex D (informative) Analysis of bias measurements from the UK Department of Health Cigarette Survey	59
Annex E (informative) ASIA COLLABORATIVE STUDY #11 2002/2003	71
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	86
Bibliography	146

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

Introduction

0.1 Summary

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

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

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

The measurement CIs represented by r_{20} and R_{20} provide a starting point for estimating the 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)

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	33	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
	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	r_{20} %	Yield mg	r_{20} mg	r_{20} %	Yield mg	r_{20} mg	r_{20} %
ACS10	5	15,35	0,86	5,6	1,25	0,080	6,4	NM	NM	NM
	1	1,24	0,31	24,9	0,11	0,021	18,6	1,99	0,38	18,9
	2	2,90	0,49	16,8	0,30	0,038	12,8	3,88	0,50	12,9
	3	4,71	0,53	11,1	0,45	0,042	9,4	8,85	0,78	8,9
	4	10,08	0,73	7,2	0,86	0,069	8,0	9,81	0,80	8,1
ACS11	5	15,16	0,83	5,5	1,23	0,075	6,1	14,79	0,94	6,4
	1	1,20	0,32	26,9	0,14	0,025	18,4	1,63	0,36	22,1
	2	2,95	0,42	14,2	0,28	0,036	12,7	3,63	0,45	12,3
	3	4,65	0,55	11,8	0,40	0,051	12,6	5,58	0,71	12,7
	4	9,40	0,75	8,0	0,83	0,091	11,0	8,85	0,98	11,1
CCS-03	5	14,26	0,85	6,0	1,30	0,081	6,2	13,09	0,88	6,7
	1	12,68	0,94	7,4	0,94	0,073	7,8	8,31	0,82	9,9
	2	14,06	0,74	5,3	1,30	0,078	6,0	13,23	0,87	6,6
	3	10,07	0,66	6,5	0,82	0,068	8,3	10,99	1,00	9,1
	4	11,56	0,71	6,2	0,85	0,059	7,0	11,85	0,95	8,0
	5	5,43	0,55	10,2	0,47	0,037	7,9	6,79	0,75	11,1
	6	10,04	0,64	6,4	0,79	0,063	7,9	9,55	0,74	7,8
	7	3,54	0,49	13,9	0,33	0,035	10,6	4,12	0,52	12,5
	8	9,83	0,72	7,3	0,88	0,071	8,1	11,45	0,97	8,5
	1	15,13	0,88	5,8	1,24	0,066	5,3	14,99	1,02	6,8
CAMG-01	1	15,15	0,74	4,9	1,22	0,069	5,7	14,48	0,74	5,1

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} mg	Yield mg	R_{20} mg	Yield mg	R_{20} mg	R_{20} %
ISO 4387	1	0,82	0,60	73,2	NA	NA	NA	NA	NA
	2	1,61	0,74	46,0	NA	NA	NA	NA	NA
	3	3,31	0,90	27,2	NA	NA	NA	NA	NA
	4	7,70	1,51	19,6	NA	NA	NA	NA	NA
	5	12,61	1,70	13,5	NA	NA	NA	NA	NA
	6	17,40	1,84	10,6	NA	NA	NA	NA	NA
ISO 10315	1	NA	NA	NA	0,09	0,069	NA	NA	NA
	2	NA	NA	NA	0,18	0,069	NA	NA	NA
	3	NA	NA	NA	0,33	0,076	NA	NA	NA
	4	NA	NA	NA	0,67	0,109	NA	NA	NA
	5	NA	NA	NA	0,84	0,142	NA	NA	NA
	6	NA	NA	NA	1,41	0,195	NA	NA	NA
ISO 8454	1	NA	NA	NA	NA	NA	3,45	1,18	34,2
	2	NA	NA	NA	NA	NA	3,56	1,03	28,9
	3	NA	NA	NA	NA	NA	9,89	2,22	22,4
	4	NA	NA	NA	NA	NA	13,80	3,00	21,7
	1	15,00	1,98	13,2	1,21	0,216	NM	NM	NM
	2	1,66	2,24	134,7	0,09	0,072	NM	NM	NM
ACS9	1	2,74	0,84	30,8	0,29	0,081	NM	NM	NM
	2	4,35	0,97	22,3	0,37	0,094	NM	NM	NM
	3	10,04	1,75	17,5	0,83	0,161	NM	NM	NM
	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 %
	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 — $R_{100+mdg}$ values for NFDPM and CO calculated from the 2003 CORESTA Collaborative Study data

Yield	$R_{100+mdg}$ 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		20 %	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	mg	$R_{100+mdg}$ value	
		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.

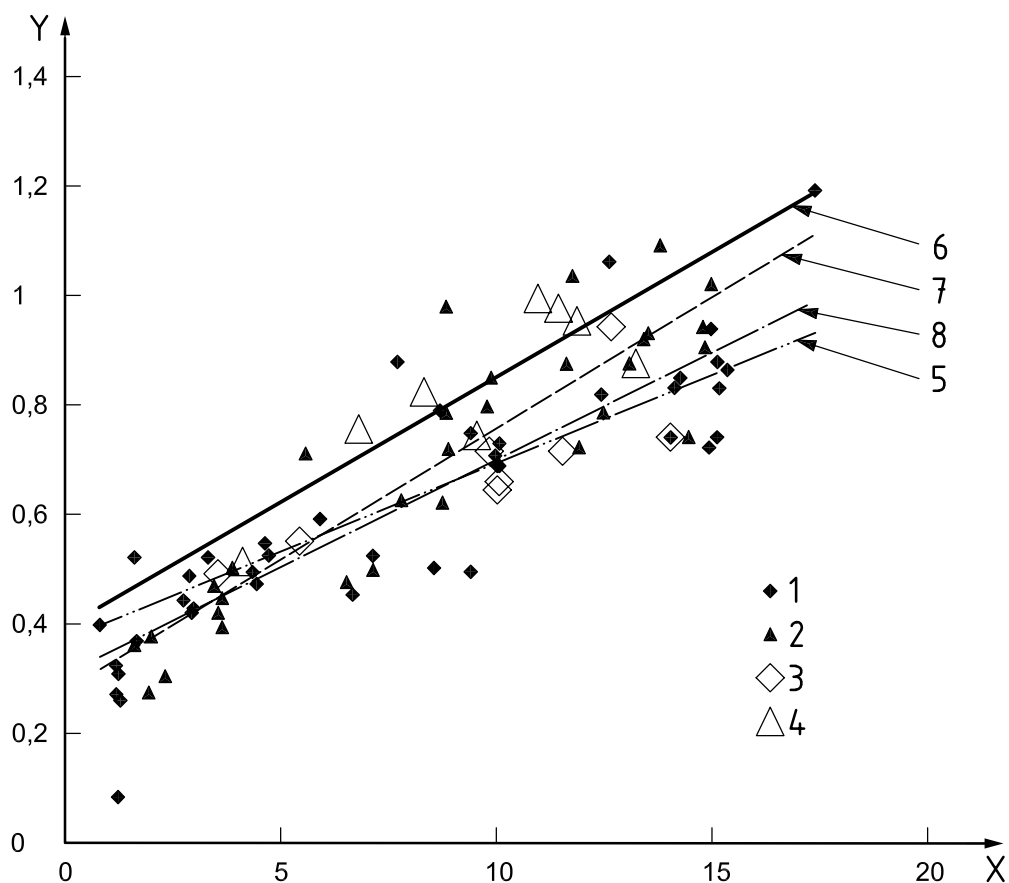
Table 8 — CO tolerance predicted from ISO 8243:2003 NFDPM tolerance and the CO/NFDPM ratio of the $R_{100+rdg}$ values from Table 6

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.

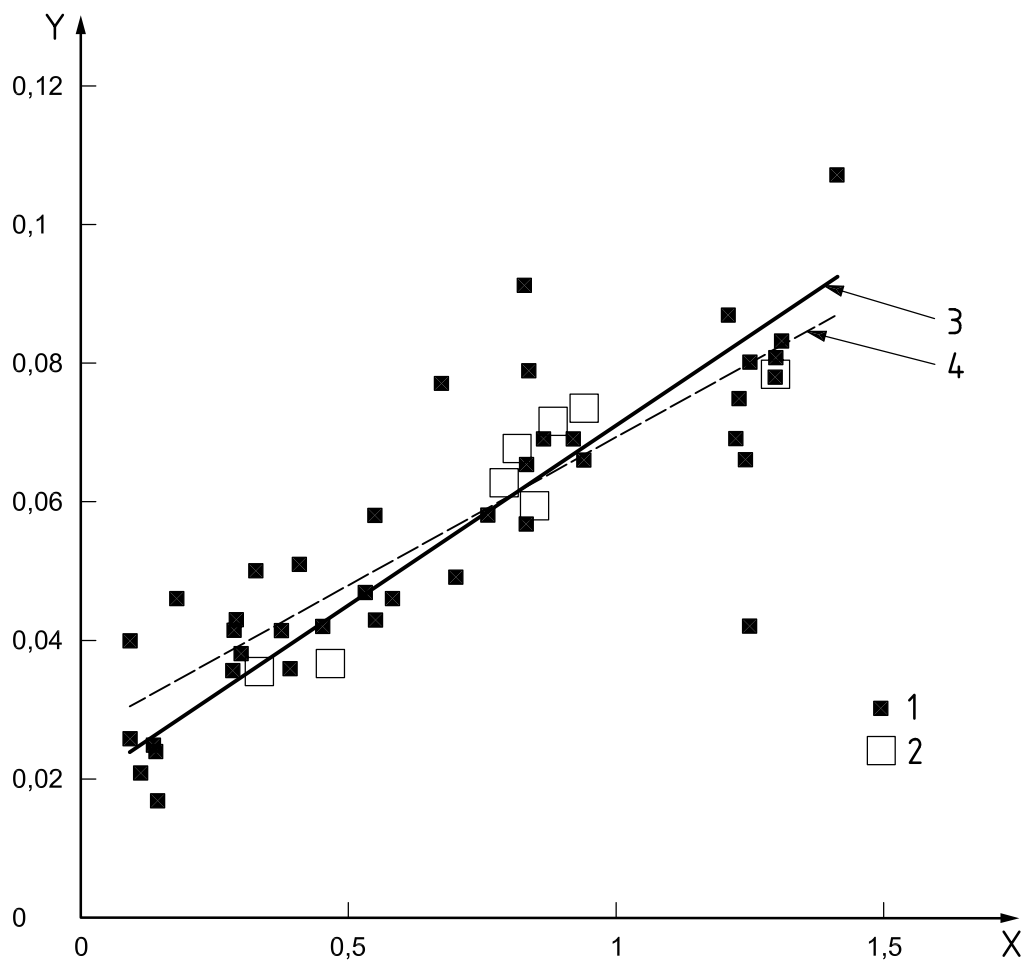


Key

- 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-NFPDM)

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

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

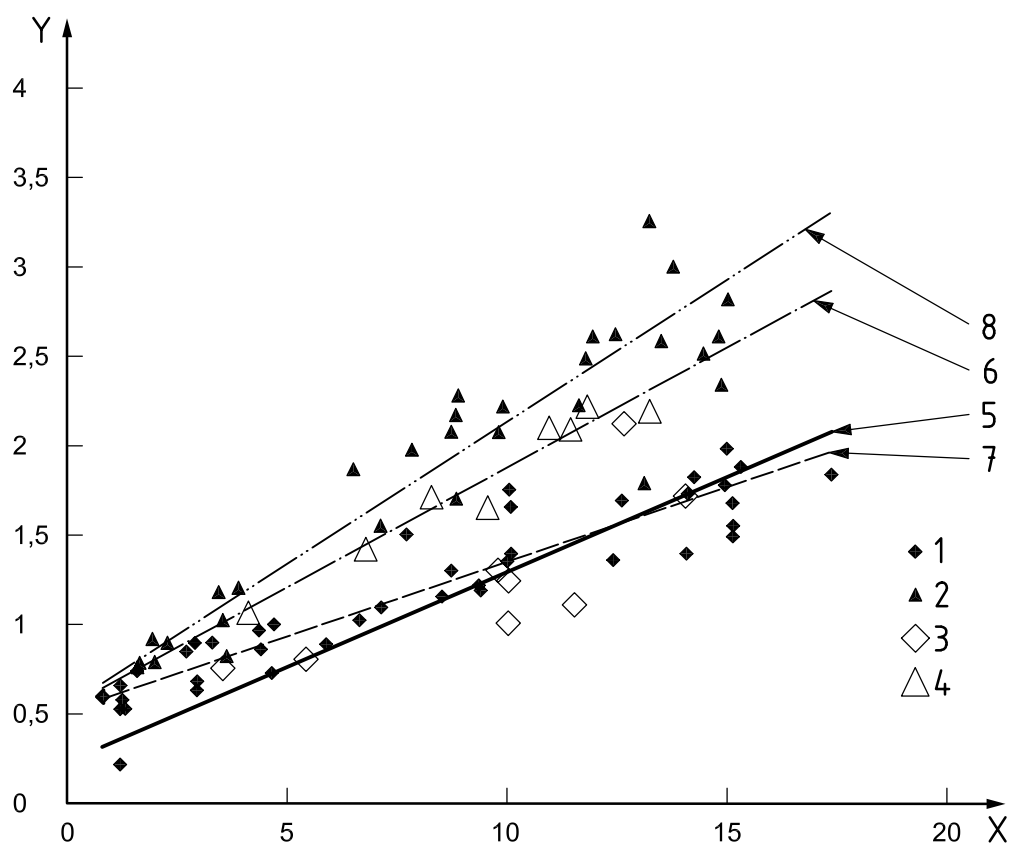


Key

- 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

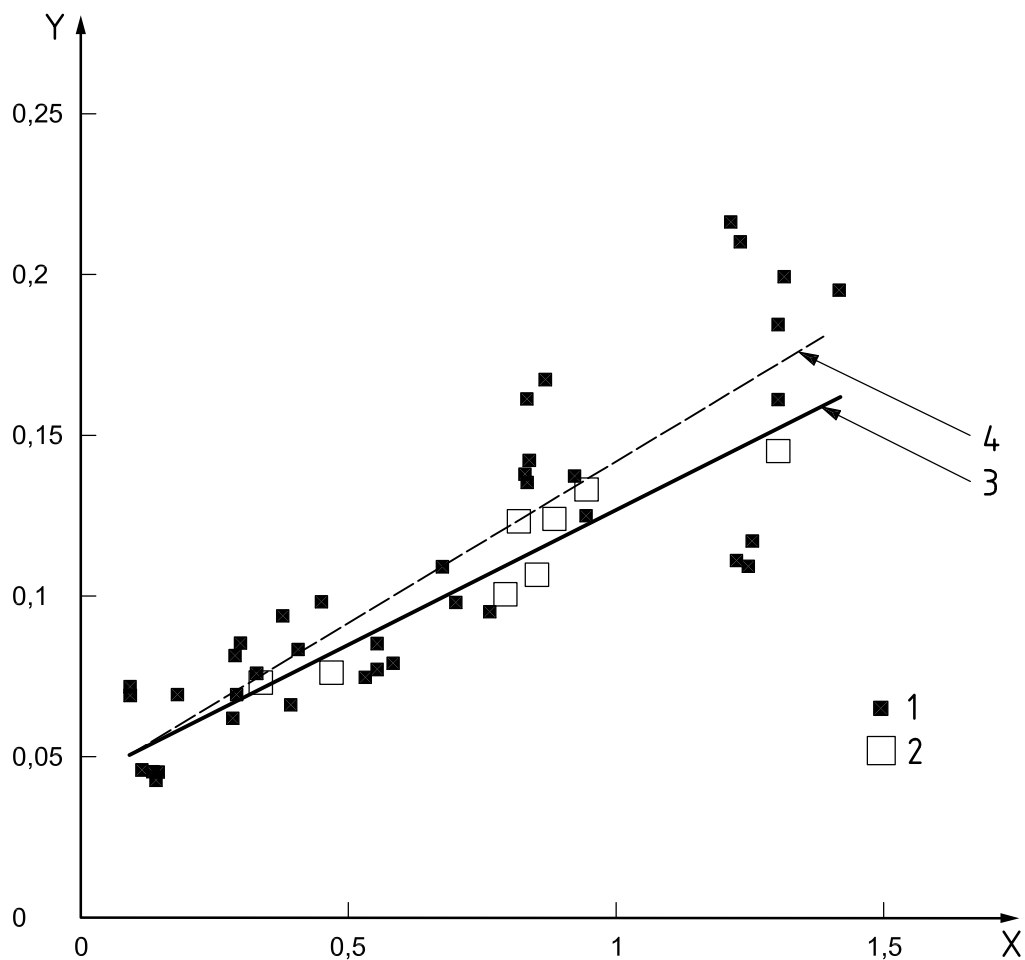


Key

- 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

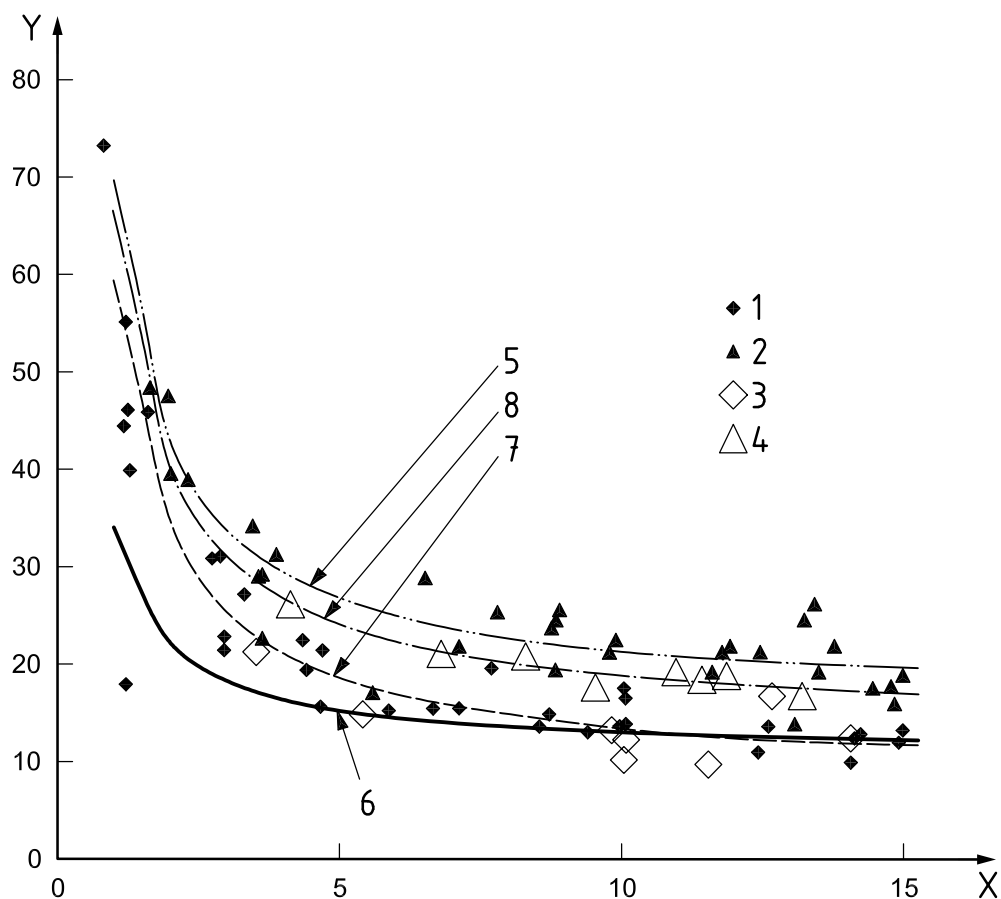


Key

- 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

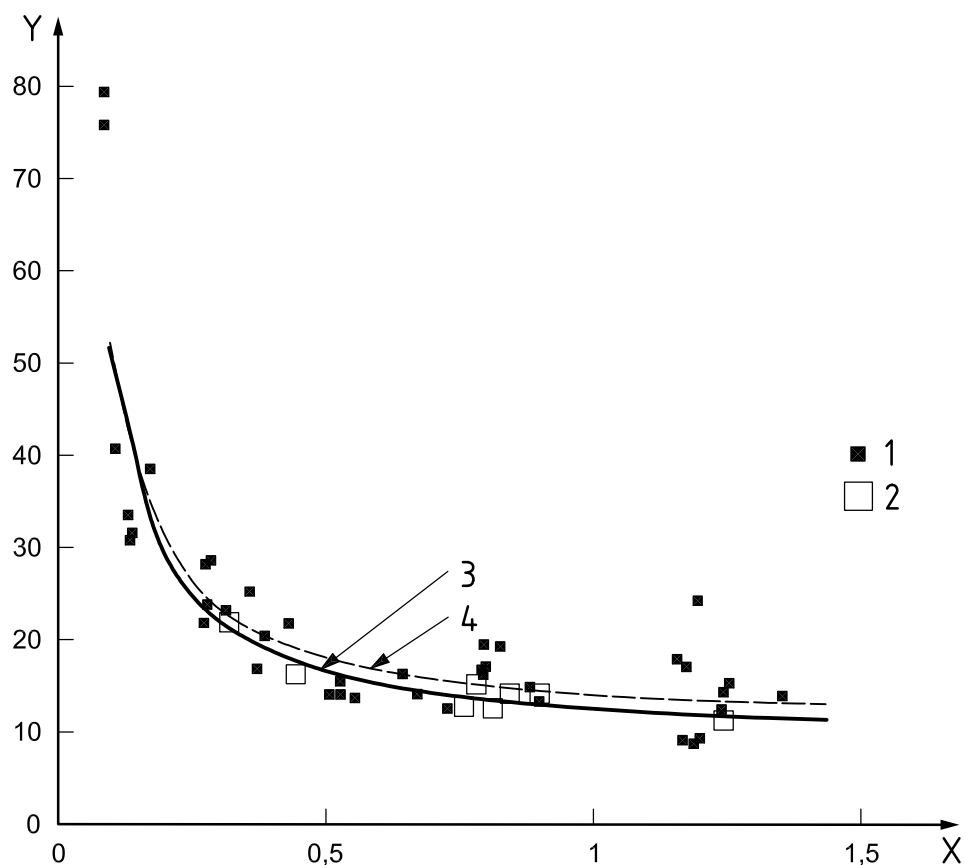


Key

- 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)

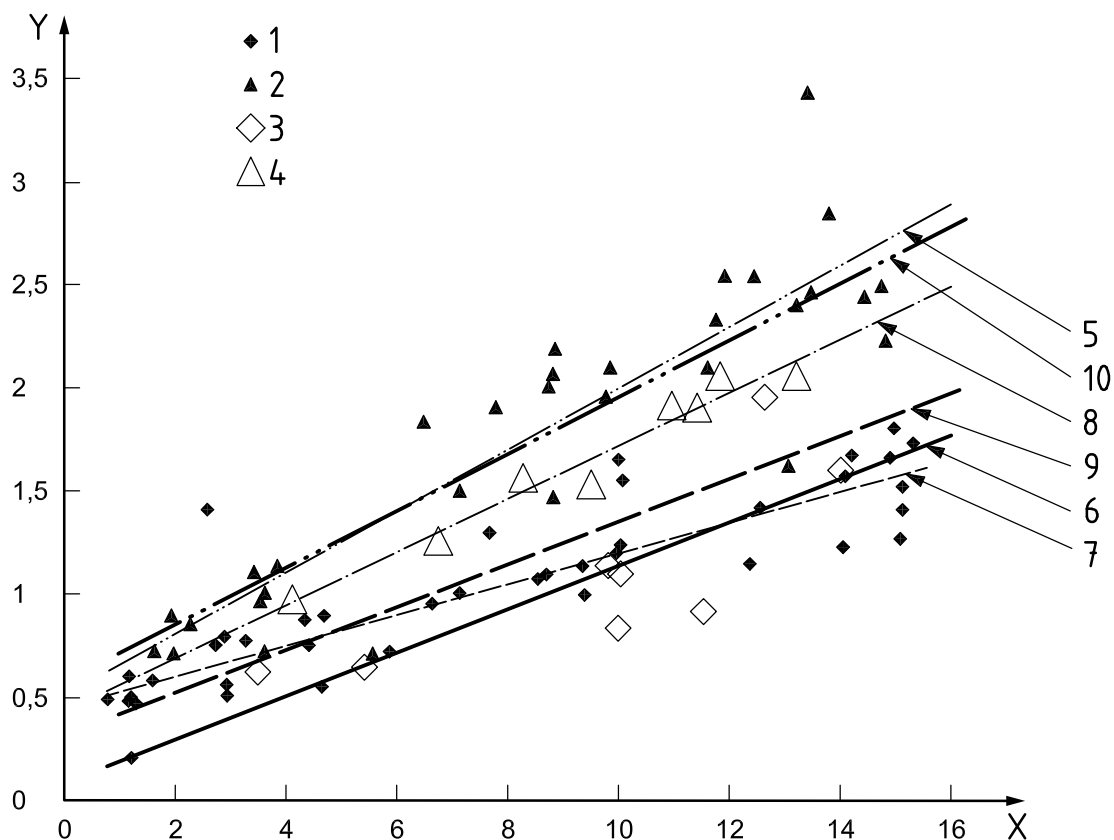


Key

- 1 other
- 2 CCS-03
- 3 CCS-03
- 4 other

X nicotine yield, mg
Y reproducibility R_{20} , %

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

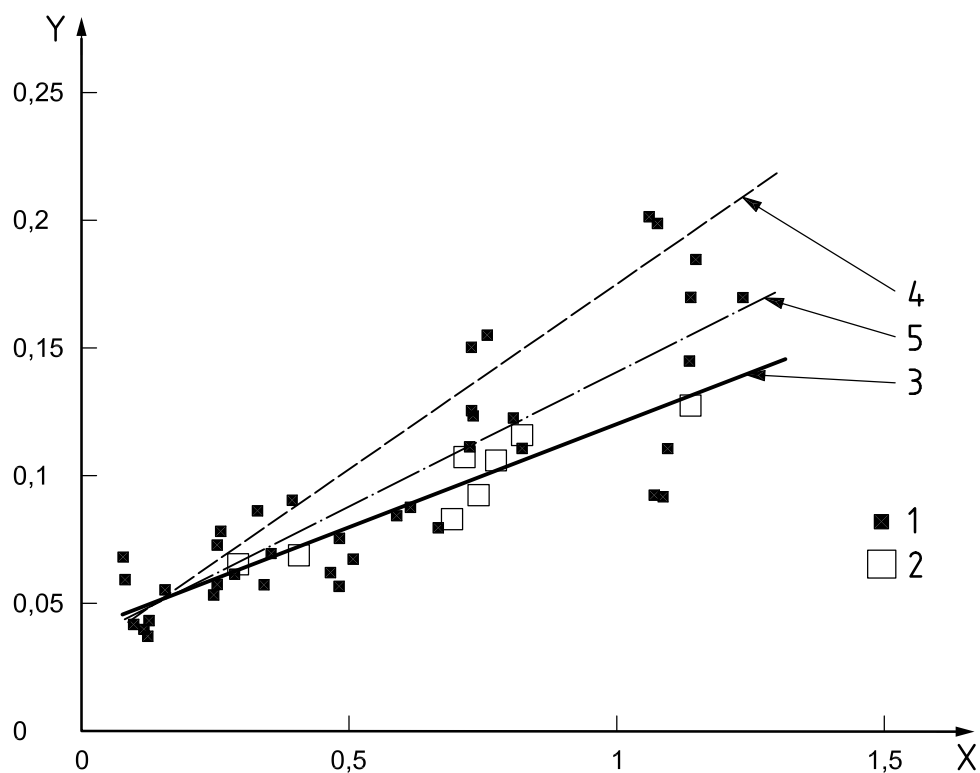


Key

- 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

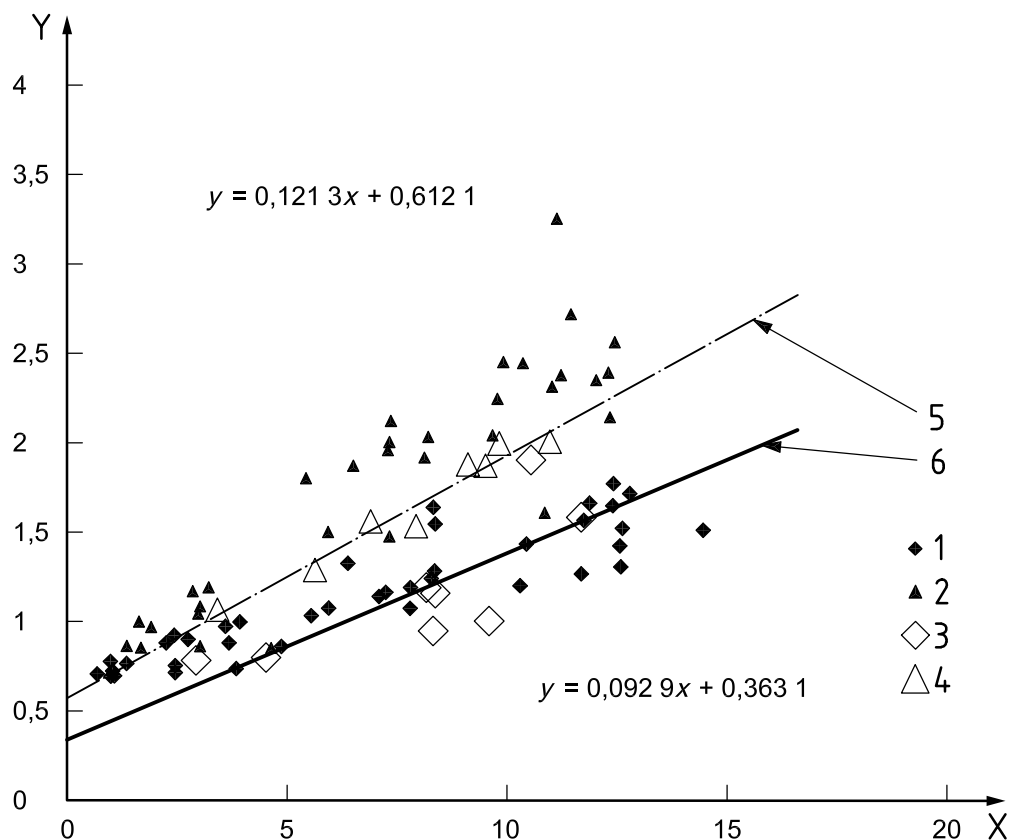
Figure 7 — R_{100} reproducibility values for NFDPM and CO



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



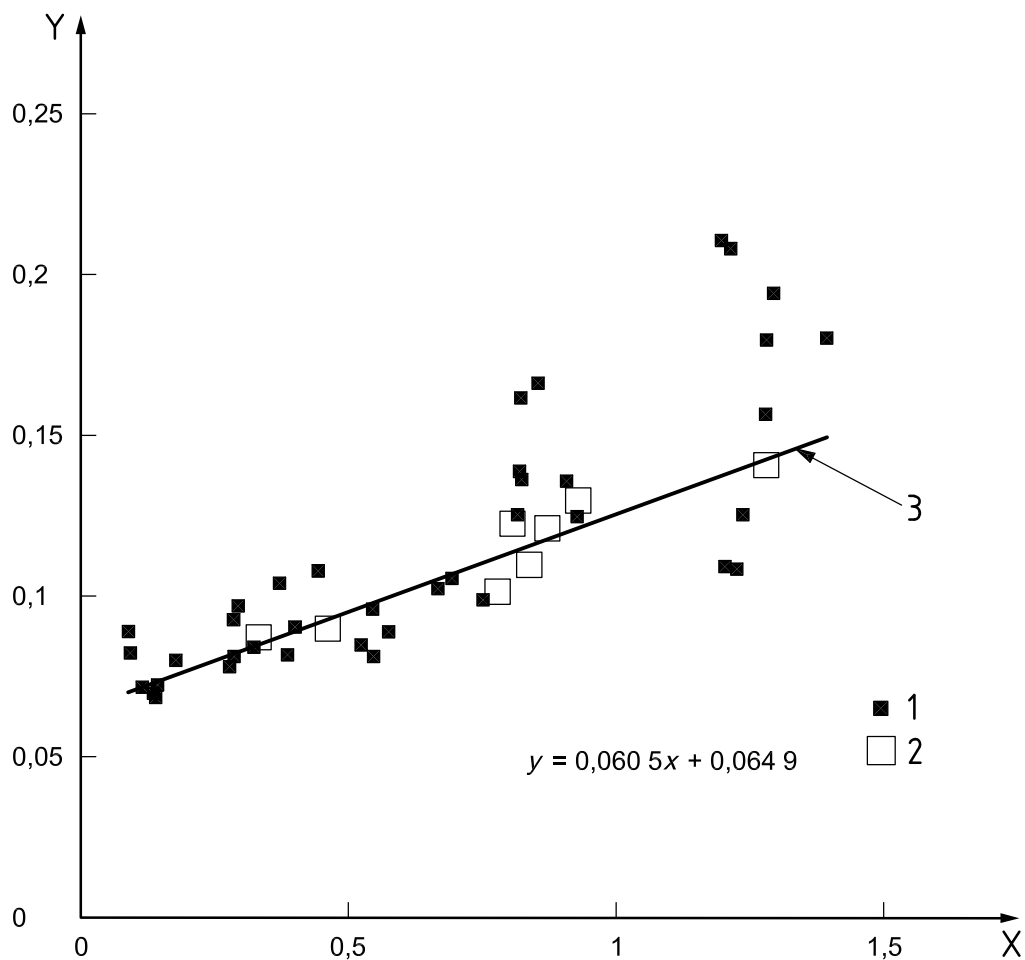
Key

- 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.

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.

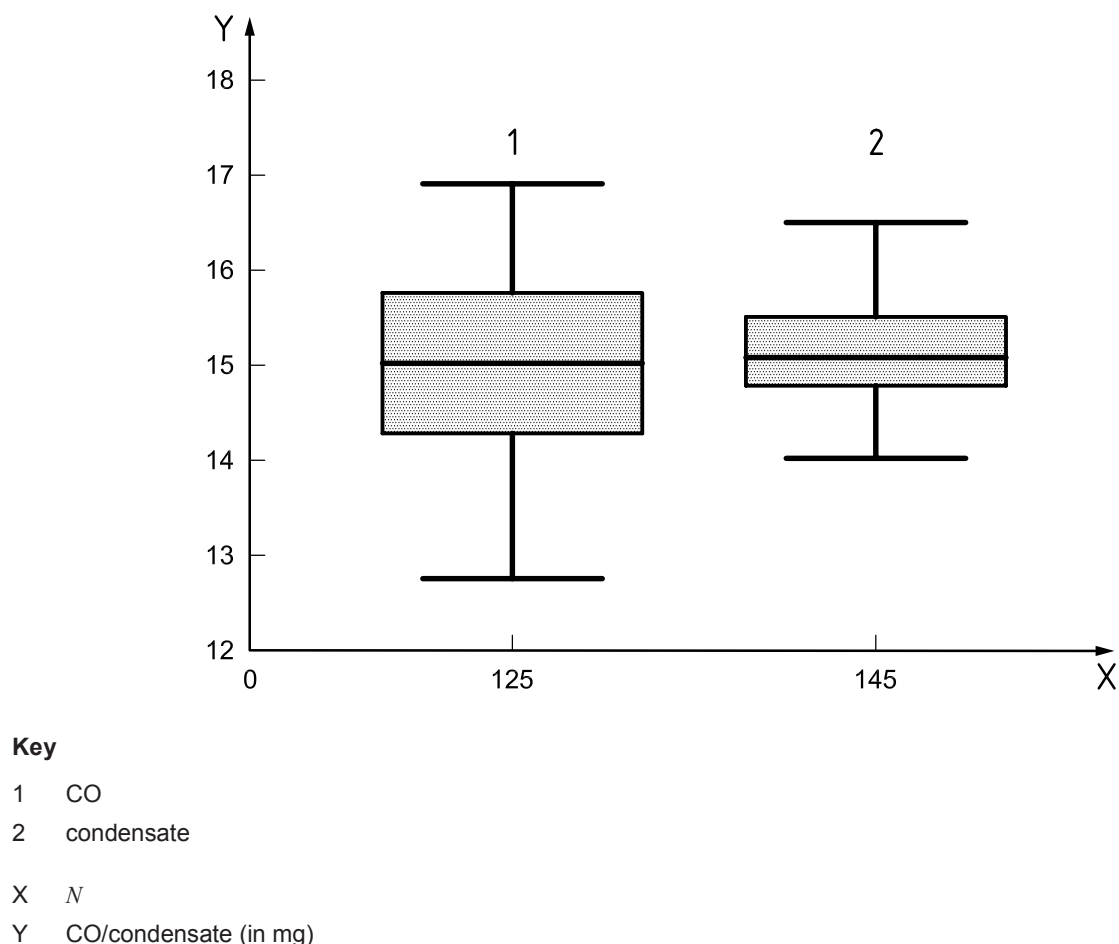
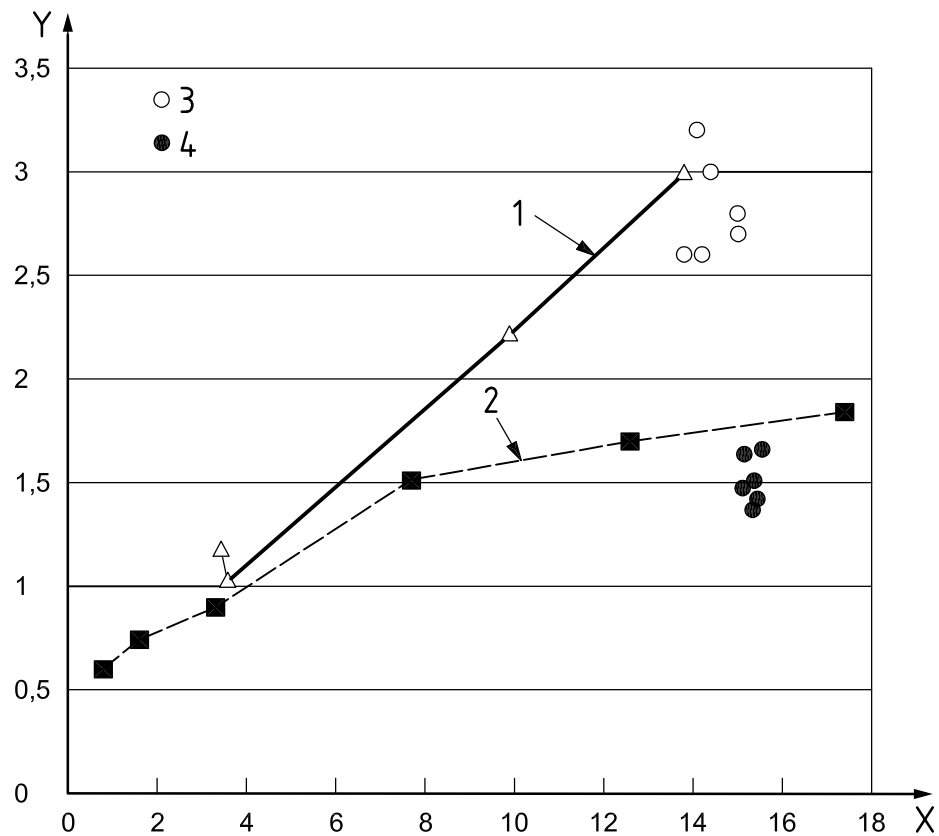


Figure B.1 — Distribution of CO and condensate (Data B - 2000)

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.

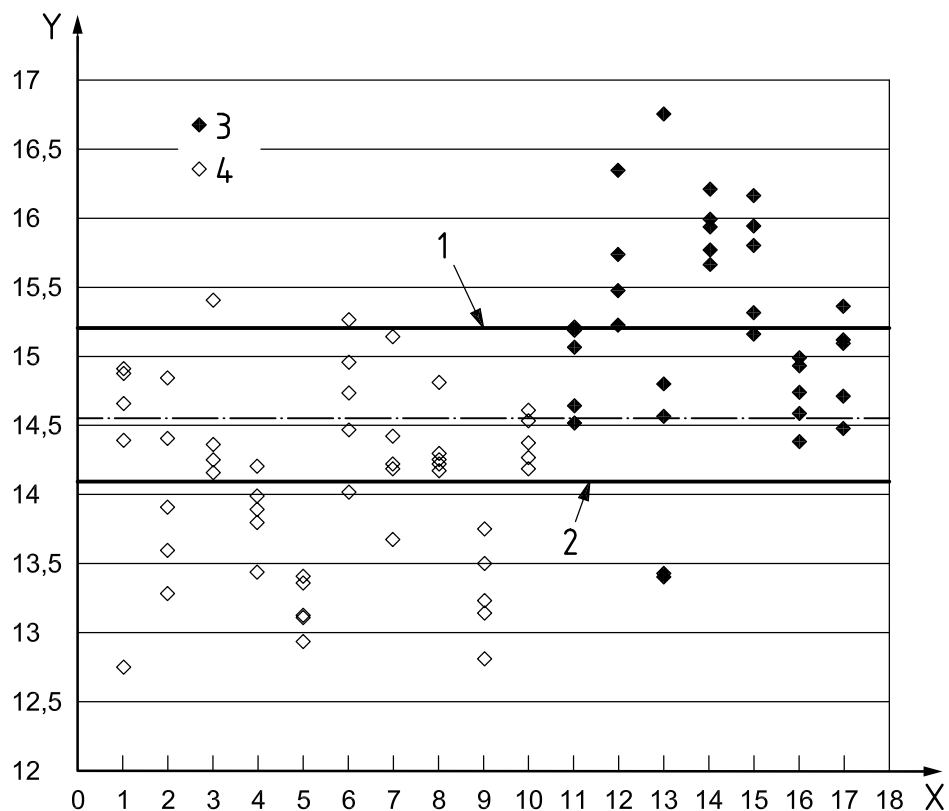


Key

- 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)