TECHNICAL REPORT

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

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

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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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.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 tolerances[3] relevant to compliance checks on the yield information provided by manufacturers. They need to be combined with additional information on testing[4] and reporting as well as the inherent variability in the product associated with routine cigarette manufacturing. The statistical model given in this report is designed to incorporate all the relevant information to estimate compliance tolerances. The model is based upon the within and between laboratory standard deviations for tests of 100 cigarettes, together with additional terms to account for rounding the declared values and to include the product variability. A weakness in the model approach stems from the lack of data for estimating the terms relating to product variability, the only source of data being the UK Department of Health Survey, which is not specifically designed to provide such data. For this reason the model has been used in this report without including the product terms and the calculated tolerance values [$R_{100+rndg}$][5] compared with those from an alternative indirect prediction. Obviously, the $R_{100+rndg}$ values are lower than the true compliance tolerance since they do not include the product terms.

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

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1) Based on tests of 20 cigarettes.

2) Based on tests of 20 cigarettes.

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

4) See ISO 4387 and ISO 8243.

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

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

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

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

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

0.2 General Information

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

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

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

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

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

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

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

0.3 Sampling a population of cigarettes manufactured for sale

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

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

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

0.4 Development of smoking machines

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

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

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

1 Scope

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

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

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

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

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

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

$r = 2{,}8 * s_w$

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

where

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

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