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English Version

**Technical report of the interlaboratory study for the
determination of Besatz in common wheat, rye and durum wheat**

Rapport technique relatif à l'essai interlaboratoire portant
sur la détermination du pourcentage d'impuretés dans le
blé tendre, le seigle et le blé dur

Technischer Bericht des Ringversuchs zur Bestimmung
von Besatz in Weizen, Roggen und Hartweizen

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Foreword

This document (CEN/TR 16324:2012) has been prepared by Technical Committee CEN/TC 338 "Cereal and cereal products", the secretariat of which is held by AFNOR.

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Introduction

The term "Besatz" applies to all components of a grain sample that differ from the normal basic cereal. The principle of the determination of Besatz is to separate all the groups of Besatz from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. The amount of Besatz and its constituent groups is important for health, cleaning, milling and further processing aspects. For these reasons Besatz is a part of contracts in grain trade and also of the grain intervention system of the EU. At present a European Standard for the determination of Besatz (EN 15587) has been developed.

An international interlaboratory test for the determination of Besatz, according to EN 15587 and involving 15 laboratories in 11 countries, was carried out with two common wheat, two rye and two durum wheat samples. Statistical analysis was possible for all Besatz fractions with the exception of grains overheated during drying, bunted grains, and impurities of animal origin.

Very high interlaboratory variation was found for the fractions mottled grains (including grains in which the germ is discoloured), unsound grains, and grains damaged by pests.

The lowest interlaboratory variation was found for the fractions extraneous matter, extraneous seeds, and ergot.

The coefficient of variability in most cases depended clearly on the amount of the respective Besatz group, whereby the amount of the respective Besatz group was in a normal range. As a result the coefficient of intralaboratory variability for the total Besatz was not higher than 10 %. The coefficient of interlaboratory variability for the total Besatz was not higher than 20 %, provided the amount of mottled grains or unsound grains or insect-damaged grain is not higher than 1 % (w/w).

1 Scope

The term "Besatz" applies to all components of a grain sample that differ from the normal basic cereal. It includes the following groups: broken grains, shrivelled grains, other cereals, grains damaged by pests, grains with discoloured germ, grains overheated during drying, sprouted grains, extraneous seeds, unsound grains, ergot, bunted grains, extraneous matter, husks and impurities of animal origin. The amount of Besatz and its constituent groups is important for health, cleaning, milling and further processing aspects. For these reasons Besatz is a component of contracts in grain trade and also of the grain intervention system of EU. The principle of the determination of Besatz is to separate all the groups of Besatz from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. There are various problems in the determination of Besatz: Firstly, the identification of the different groups of Besatz depends strongly on the experience and the knowledge of the investigator. Also experienced investigators can differ in their characterization of grains. Finally, one is faced with the fact that grain, even after mixing, is rarely homogenous. In other words, if a sample was divided by a sample divider into a number of portions, the amount of a specific group of Besatz in each portion could be different, even if absolutely no human or machine error occurred in each determination. These problems will result in variation of the results of the determination.

An international interlaboratory trial for the determination of Besatz in common wheat, durum wheat and rye was accomplished with 15 laboratories in order to get information on the intra- and interlaboratory variability of the determination of Besatz.

The Technical Report here describes the preparation and evaluation of the results of this interlaboratory test.

2 Design of the study

Each laboratory received two kinds of sample sets.

The **sample set A** consisted of six samples which had a mass of not less than 50g. The participants received two samples of common wheat, rye and durum wheat. The sample set A was prepared from basic cereal of unimpaired quality. Certain amounts of the 14 different Besatz groups ¹⁾ were added to each sample of the set A, in the way that the two samples of the same cereal had the same composition. The composition of the spiked samples was chosen to ensure that i) laboratories obtaining outlying results would be identified and ii) the influence of sample division on the precision of the method would be recognizable.

The **sample set B** consisted of six samples which had a mass of not less than 1kg. The participants received two samples of common wheat, rye and durum wheat. The sample set B was prepared from clean basic cereal, which contained small amounts of Besatz ²⁾. One bulk sample of slightly more than 40kg of each cereal (wheat, rye, and durum) was used. After careful mixing to make the bulk sample as homogeneous as possible it was divided using a sample divider to obtain 40 samples. Twenty of them were directly sealed in moisture proof plastic bags. The other 20 samples were spiked with certain Besatz groups, individually mixed and the sealed in moisture proof plastic bags. In this way we achieved different concentration levels of the various Besatz groups. The concentration levels were selected to represent the range of Besatz commonly found in grain samples. The results obtained for the sample set B were used for the calculation of the precision data given in Annex D of EN 15587:2008.

The participants were instructed to analyse the samples of set A only once and the samples of set B in duplicate. They had to divide the samples of set B to obtain the required size, before testing.

1) The added material of the various Besatz groups comes from numerous Besatz-investigations of our own laboratory. The material was reviewed independently by three persons with regard to the unique assignment to a Besatz group.

2) The use of basic cereal of unimpaired quality was impossible, because the cleaning of 120 kg of grain material by manual selection was not realisable.