ASSESSMENT OF VARIANCE IN MEASUREMENT OF HECTOLITRE MASS OF WHEAT AND MAIZE, USING EQUIPMENT FROM DIFFERENT GRAIN PRODUCING AND EXPORTING COUNTRIES

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Declaration

I, the undersigned, hereby declare that the work contained in this thesis is my own original work and that I have not previously in its entirety or in part submitted it at any university for a degree.

	22/02/08
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Abstract

South Africa as well as other grain producing and exporting countries' grading systems strongly relies on hectolitre mass (HLM) as a guide to grain quality. It is known that these countries use either one of two types of HLM equipment. These devices consist of either a funnel or a cylindrical device (chondrometer) with a measuring cylinder of known volume underneath which is then filled with grain in a controlled manner. Subsequently the HLM devices from Australia, Canada, France, Germany, South Africa, the United Kingdom (UK) and the United States of America (USA) were compared using impurity free mixed wheat, single South African cultivars as well as maize samples.

Very little variation in HLM measurements within the HLM devices was observed with intra-class correlation (ICC) agreement values close to one. Comparing the actual HLM values obtained with the respective devices showed that the results obtained with the Australian device was significantly ($P < 0.05$) higher, and those obtained with the South African devices significantly ($P < 0.05$) lower compared to the other devices. As would be expected the devices showed better overall ICC agreement when the HLM tests were performed with the single cultivar samples (ICC agreement = 0.762) as opposed to the mixed wheat samples (ICC agreement = 0.523). However, the HLM values obtained with all the devices correlated well with each other (ICC consistency >0.90) indicating that correction factors can therefore be developed to convert the HLM results between devices.

When ten South African devices were compared statistical differences were observed, but the overall ICC agreement (0.975) and consistency (0.993) values indicated that the differences would not be significant in practice. Hectolitre mass determinations performed on samples prior to and after impurities have been removed revealed that the removal of impurities resulted in a significant ($P < 0.05$) increase in HLM. The effect of operator was shown to be significant ($P < 0.05$) when operators with three levels of competency, i.e. skilled, semi-skilled and unskilled, were investigated.

The effect of wet and dry cycles on the HLM measurements was investigated and the results showed that wetting and drying could change the integrity of the wheat. Moisture correction factors cannot be applied to convert the HLM values of grain that underwent moisture changes as different samples responded differently to the moisture treatments.

Comparing the respective devices with mixed maize samples (impurities not removed) very little variation in HLM measurements within each device was observed. The comparison of the devices revealed that the HLM measurements obtained with the Australian and French devices were significantly ($P < 0.05$) higher and that obtained from the Canadian device significantly ($P < 0.05$) lower compared to those obtained with the other devices. Again it was shown that the devices correlate well (ICC consistency > 0.97) and that correction factors can be applied to convert HLM results between devices. An alternative to the use of correction factors could be the replacement of the South African device with the German device for both wheat and maize.

The removal of impurities from the maize samples significantly ($P < 0.05$) increased the HLM values. Therefore, it is likely that correction factors can be used to convert HLM values of maize samples before and after removal of impurities.

Uittreksel

Suid-Afrika sowel as ander lande wat graan produseer en uitvoer gebruik hektolitermassa (HLM) om die algemene kwaliteit van graan te bepaal. Een van twee tipes apparate word gewoonlik gebruik om HLM te bepaal; die tregter metode wat toegerus is met ‘n maatbeker van bekende volume of die kolom metode wat bestaan uit ‘n silindriese apparaat met ‘n maatbeker van bekende volume onderaan geheg. Die apparate van Australië, Duitsland, Frankryk, Kanada, Suid-Afrika, die Verenigde Koninkryk en die Verenigde State van Amerika is met mekaar vergelyk deur gebruik te maak van gemengde koring, enkel Suid-Afrikaanse kultivars asook mieliemonsters.

Die intra-klas korrelasie (IKK) ooreenstemming het aangetoon dat byna geen variasie in HLM waardes binne die apparate voorkom nie. Toe die werklike HLM waardes, soos verkry met die verskillende apparate, met mekaar vergelyk is, is gevind dat die HLM waardes soos verkry met die apparaat van Australië beduidend ($P < 0.05$) hoër en die wat verkry was met die Suid-Afrikaanse apparaat beduidend ($P < 0.05$) laer was in vergelyking met die ander apparate. Soos verwag, het die apparate beter IKK ooreenstemming getoon toe die HLM toetse met enkel kultivars (IKK ooreenstemming = 0.762) bepaal was in teenstelling met die HLM toetse wat met gemengde koringmonsters (IKK ooreenstemming = 0.523) bepaal is. Die HLM waardes wat met die verskillende apparate verkry is, het egter goed met mekaar gekorreleer (IKK konsekwensie > 0.90). Dit is ‘n aanduiding dat korreksiefaktore bereken kan word om die HLM resultate tussen apparate om te skakel.

Statistiese verskille is waargeneem toe tien Suid-Afrikaanse HLM apparate met mekaar vergelyk is, maar die IKK ooreenstemming (0.975) en die IKK konsekwensie (0.993) het aangetoon dat die verskille nie betekenisvol in die praktyk sal wees nie. HLM bepalings voor en na die verwydering van onsuiwerhede het aangetoon dat die verwydering van onsuiwerhede ‘n beduidende ($P < 0.05$) toename in HLM waardes teweeggebring het. Die effek van operateurs, met drie vlakke van opleiding, i.e. opgelei, semi-opgelei en onopgelei, op HLM waardes was beduidend ($P < 0.05$).

Nat- en droogsiklusse het die digtheid en integritiet van die koringmonsters verander. Daar is gevind dat elke monster verskillend reageer op verskillende vogbehandelings. Dit is dus onmoontlik om korreksiefaktore vir vog te bereken vir HLM waardes van koringmonsters wat aan vog veranderinge blootgestel was.

Min variasie in HLM waardes binne apparate is waargeneem toe die verskillende HLM apparate met mieliemonsters (onsuiwerhede nie verwyder) getoets is. Die vergelyking van apparate het getoon dat die HLM waardes verkry met die apparate van Australië en Frankryk beduidend ($P < 0.05$) hoër was en dié verkry met die apparaat van Kanada beduidend ($P < 0.05$) laer as dié van die ander apparate. Al die apparate het weer eens goed met mekaar gekorreleer (IKK konsekwensie > 0.97) dus kan korreksiefaktore bereken word om HLM waardes tussen apparate om te skakel. Die alternatief tot korreksiefaktore is om die huidige Suid-Afrikaanse HLM apparaat met die Duitse apparaat te vervang vir beide koring en mielies.

Die verwydering van onsuiwerhede uit mielies het 'n beduidende ($P < 0.05$) toename in HLM teweeggebring. Korreksiefaktore kan dus moontlik toegepas word om HLM waardes om te skakel tussen mieliemonsters voor en na die verwydering van onsuiwerhede.

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Abbreviations

Anon	Anonymous
ARC	Agricultural Research Council
ca.	*circa* (about)
e.g.	*exempli gratia* (for example)
et al.	*et alibi* (and elsewhere)
Fig.	Figure
$g.cm^{-3}$	Gram per cubic centimetre
HLM	Hectolitre mass
i.e.	*id est* (that is)
ICC	Intra-class correlation
IKK	Intra-klas korrelasie
$kg.hL^{-1}$	Kilogram per hectolitre
L	Litre
$lb.bu^{-1}$	Pounds per bushel
mL	Millilitre
n	Number of samples
r	Correlation coefficient
RANOVA	Repeated analysis of variance
SA	South Africa
sd	Standard deviation
se	Standard error
SEM	Average error
UK	United Kingdom
USA	United States of America

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Language and style used in thesis are in accordance with the requirements of the International *Journal of Food Science and Technology*. This thesis represents a compilation of manuscripts where each chapter is an individual entity and some repetition between chapters has, therefore, been unavoidable.

CHAPTER 1

Introduction

Introduction

Hectolitre mass (HLM), also referred to in some countries as test weight or bushel weight, is the weight of a standard volume of wheat and is a function of the density of wheat (Lockwood, 1960; Pushman & Bingham, 1975; Donelson *et al.*, 2002). It is one of the oldest specifications used in wheat grading and serves as a guide to a combination of characteristics, including wheat flour yield (Posner & Hibbs, 2005). Many researchers investigated the effectiveness of HLM as a guide to flour yield (Mangels & Sanderson, 1925; Shuey, 1960; Barmore & Bequette, 1965; Pushman & Bingham, 1975; Dexter *et al.*, 1987). In one of the earliest studies on HLM determinations high correlation coefficients were obtained between HLM and flour yield determined over seven crop years, i.e. 1916 (r = 0.72) and 1919 to 1924 (r = 0.77) (Mangels & Sanderson, 1925). The 1916 crop year was included in the study because many of the samples from this crop had very low HLM values. In contrast to this study Shuey (1960) observed a poor correlation between HLM and flour yield for three consecutive crop years. Furthermore, HLM was also shown to be a poor indicator of flour yield potential when Pacific Northwest (PNW) white wheat was used (Barmore & Bequette, 1965). Despite these contradictory results HLM determination is still one of the most popular wheat quality tests. This can be ascribed to the fact that the equipment is cheap, the test is easy to perform and above all, it gives reliable results in a simple numerical manner (Lockwood, 1960).

The South African grading system strongly relies on HLM as a guide to wheat grain quality (Anon., 1998; Sierk Ybema, Senwes, Klerksdorp, South Africa, personal communication, 2006). During wheat marketing, a numerical value or grade is allocated to the grain based on the results of several tests performed, of which HLM determination is one of the more important tests. In South Africa wheat producers are remunerated firstly according to the HLM value of their wheat before other factors, i.e. protein content is taken in consideration (Anon., 1998; Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). An increase in HLM results in a higher allocated grade and subsequently in a higher price per ton of wheat; unless other grade determining factors such as protein content will negatively impact the grade (Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). The HLM values of sound wheat normally varies from 70 to 80 $kg.hl^{-1}$, but can be higher or lower due to several factors i.e., environmental conditions and insect damage (Troccoli & Di Fonzo, 1999; Sierk Ybema, Senwes, Klerksdorp, South Africa, personal communication, 2006).

Climatic influences such as severe drought during grain filling and weather conditions conducive to rapid disease spread and or lodging can lead to shrivelled grain, which lowers HLM through reduced packing efficiency (Weibel & Pendleton, 1964; Gooding & Davies, 1997). Immature wheat or badly shrivelled wheat, as a result of drought or disease, usually has low HLM values and corresponding poor flour yields (Halverson & Zeleny, 1988). Shrivelling caused by fungal diseases may result in a decrease in both the starch and protein contents of the wheat kernel, leading to less dense kernels with low HLM values (Phil Williams, PDK Grains, Nanaimo, British Columbia, Canada, personal communication, 2007). Because shrivelled kernels contain proportionately more bran than endosperm compared with plump, well-filled grain, HLM is used as a rough indication of flour yield (Gooding & Davies, 1997).

As HLM is a function of packing efficiency (the percentage of a bulk volume occupied by grain) and kernel density (Yamazaki & Briggle, 1969; Gooding & Davies, 1997) it is influenced by shrivelled kernels which impairs the HLM through reduced packing efficiency (Gooding & Davies, 1997). The packing efficiency of wheat is also influenced by kernel shape and surface characteristics such as presence of brush hairs and surface condition, which can be rough or smooth (Barmore & Bequette, 1965). Roughening of the bran coat occurs during cycles of wetting and drying which causes a decrease in the density of the kernels (Swanson, 1946; Pool *et al.*, 1957). Handling the grain, on the other hand, tends to polish the kernels and allows them to pack tighter in the test container (Shuey, 1960).

It is clear that HLM determination of wheat has been widely studied. This, however, does not hold true for the HLM determination for maize. It has been shown earlier that it is of more value to determine HLM for wheat than for maize, as there is a high correlation between HLM and flour yield for wheat as opposed to the low or zero correlation between HLM and maize grits for maize (USDA, 1933). It has, however, been shown that in order to guarantee a good milling quality in maize, it is desirable for maize cultivars to have high HLM values (USDA, 1933). Immature maize may result in very low HLM values, but feeding tests performed on high and low HLM maize, respectively, showed that low HLM maize was sometimes of superior feeding value due to its relatively higher protein quality (USDA, 1933).

In Europe few grain specialists considered HLM for maize of any value since it was shown not to relate to other quality factors (Hall & Hill, 1973). It was suggested at the time that HLM for maize either be proved of some value or rather be removed from the standards. Dorsey-Redding *et al.* (1991) reported that a need for simple, rapid and reliable tests that could relate maize quality to product yields in various end uses still exists. Although HLM, as used in the United States maize standards is not a precise indicator of any specific grain quality attribute, it is regarded as an indication of grain soundness (Dorsey-Redding *et al.*, 1991). As HLM does comply with the criteria as stated above it can be utilised in the maize industry as a rapid quality test. Maize with low HLM often has a lower percentage of hard endosperm and consequently, produces a lower yield of prime, large grits when milled (Rutledge, 1978) indicating that HLM can indeed be used as a quality indicator of maize.

Hectolitre mass is clearly an important quality indicator in wheat quality analysis. However, it has been observed that HLM values acquired from the same wheat consignment differ when determined in one country compared to another (Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). Different countries around the world tend to have their own HLM devices and methods of determination. It is therefore likely that the differences in HLM values observed for the same wheat consignment as determined in different countries can be attributed to the different HLM equipment being used. This statement stems from the fact that some role players in the wheat industry suspected that the South African HLM device results in different HLM values compared to its international counterparts.

Until now, no studies have been executed to compare different HLM equipment and the available literature on HLM devices also fails to address differences in HLM likely to be obtained in different countries. As a result consensus was reached, amongst role players in the South African wheat industry, that this needed further investigation. Furthermore, this study was extended to include the determination of HLM on maize because the South African Maize Industry currently has no official or standard method in place, for determining HLM on maize.

The outcome of this study will be useful in determining the status of HLM in the current South African grading system and to ensure that the South African wheat industry reaches its full potential. This study will verify whether the SA HLM device does give different results in comparison to its international counterparts. If the South African device indeed gives different HLM values, this could influence grade determining specifications for wheat imports. South African HLM determinations might subsequently need to be brought in line with international standards. As a result, the SA wheat and maize grading systems will be comparable with international standards with positive effect on the economy in the long run.

The objectives of this study were therefore to:

- evaluate HLM equipment as used in Australia, the United Kingdom, Canada, France, Germany and the United States of America in comparison to the South African HLM device using wheat samples of varying HLM values;
- evaluate HLM equipment as used in Australia, United Kingdom, Canada, France, Germany and the United States of America in comparison to the South African HLM device using maize samples of varying HLM values;
- compare HLM values obtained from ten respective South African HLM devices;
- evaluate the effect of impurities on HLM determinations;
- evaluate the effect of operator on HLM determinations; and
- investigate the effect of consecutive drying and wetting cycles on HLM determinations using wheat samples of varying HLM values.

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CHAPTER 2

Literature review

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Literature review

1. Introduction

Hectolitre mass (HLM) is the mass of a specific volume of grain and the result is reported in $kg.hL^{-1}$ (Anon., 1998a). Hectolitre mass is often also referred to as bushel weight, test weight or specific weight (Pushman, 1975; Hook, 1984). In South Africa the terminology hectolitre mass is used and this term will henceforth be used throughout this thesis and will replace older terminology used in earlier studies.

Although HLM determination is a common measurement used as part of wheat grading, not many studies have been performed to evaluate the method or the factors influencing it. This literature review will give an overview of research that has been performed on HLM determination and related aspects. Issues that will be covered in this chapter include: wheat and flour quality; maize quality; the importance of HLM as a grain quality indicator, especially in relation to flour milling yield; and factors likely to influence the HLM determination of cereal grains.

2. Wheat and flour quality

2.1 Background

Wheat quality has a different meaning for each intended end-use of the grain or flour (Oleson, 1994). Wheat, whether hard or soft, is milled to flour and the flour is used to produce consumables such as breads, biscuits and pasta whether in bakeries or in a household kitchen (Oleson, 1994). Physical wheat quality is usually performed by means of several tests including: visual grading; HLM determination; density and hardness (Anon., 1995; Ohm *et al*., 1998). Additionally, several other quality tests, e.g. rheology measurements, have to be performed on the flour as well. Rheology is the study of the deformation and flow of matter under the influence of an applied stress (Anon., 2007). The performance of the dough during the baking process can be predicted once the rheology results of the flour have been studied.

2.2 Wheat grading

According to the South African grading regulations commercial wheat is classified into different classes by means of a grading system (Appendix 1) (Anon., 1998a). These classes include bread wheat, durum wheat and biscuit wheat (Anon., 1998a). Each class is further divided into sub-classes or grades, i.e. bread wheat is classified into grades B1, B2, B3, B4 and utility grade and a grade referred to as class other wheat (wheat that does not comply with the standards specified for the other five classes).

In order to grade or classify the wheat, certain characteristics have to be evaluated. The analysis is done by a professional wheat grader who evaluates characteristics such as HLM, protein content, moisture content and α-amylase activity, as determined according to the Hagberg falling number procedure (Bass, 1988; Anon. 1998a; AACC, 2004). Other evaluations include visual determination of damage from heat and insects, as well as presence of immature and sprouted kernels, foreign material, other grains and live insects (Anon., 1998a). Wheat kernels are also inspected for the presence of possible fungal growth. Fungi can grow on the wheat kernel during growth in the field or during storage due to improper storage conditions (Anon., 1998a). The wheat grader must also inspect a 10 kg sample of wheat for noxious seeds, i.e. *Convolvulus spp. and Ipomoea purpurea* which is harmful for humans when consumed (Anon., 1998a). The grading characteristics, as evaluated for each consignment of wheat, must comply with specified criteria in order to be allocated a certain grade; the higher the grade, the better the quality of the wheat and the higher the remuneration.

2.3 Flour milling

Wheat is delivered to the mill via road and/or railway transportation. Once at the mill, the wheat is graded according to the grading regulations, cleaned and stored in silos, according to grade, under controlled conditions (Bass, 1988; Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). It is further conveyed to intermediate storage bins (Bass, 1988), from which it can be blended with other grades to obtain a desired blend or grist (Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). A grist or blend of wheat is usually made up of different grades of wheat in order to obtain a required quality in the flour. Blending is, in most cases, based on flour protein and different levels of different grades of wheat are blended to obtain the required protein content required for the end product. Blending of different types of wheat flours is also done when durum flour is used in baking of flatbreads. Important factors to consider when durum flour is used for flatbread baking are durum strength, starch damage and pigment content (Dick & Matsuo, 1988). Successful baking is usually not accomplished at the 100% durum level because of the weak gluten strength and high starch damage and pigment content. As a result durum flour is often blended with a medium dough-strength, non-durum bakery flour to produce bread products with the desired texture and colour (Dick & Matsuo, 1988).

Cleaned wheat from the storage bins are conditioned to 15.5-16% moisture content (Ohm *et al.*, 1998; Phil Williams, PDK Grains, Nanaimo, British Columbia, Canada, personal communication, 2007) with the appropriate amount of water for 8-24 hours to prepare the wheat for milling (Inglett & Anderson, 1973; Bass, 1988; Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). The wheat is then conveyed to the mill to undergo the grinding process, which is basically performed with three sets of rolls; break rolls, sizing rolls and reduction rolls (Bass, 1988). During this process the kernels are broken, the endosperm scraped off from the bran and the endosperm reduced or ground to flour. After each grinding process the material is sifted to remove the flour. At the end of the milling process the original wheat kernels are separated into three types of material, namely pure endosperm, composites of endosperm plus bran and relatively pure bran

(Bass, 1988). It is essential to recover as much endosperm as possible from the kernel in order to conduct an efficient milling process.

2.4 Flour quality

Wheat flour quality is determined by subjecting the flour to different quality tests. Most important are those tests performed with rheological devices, e.g. Mixograph, Farinograph, Alveograph and Extensigraph to measure the rheological characteristics of the dough. Dough rheology is based upon the unique property of wheat dough, namely its viscoelasticity (able to stretch and easily change shape) (Mailhot & Patton, 1988). Flour obtains this property from gluten that consists of the two proteins glutenin and gliadin. Glutenin is responsible for the strength and cohesiveness of gluten whilst gliadin contributes towards the extensibility trait.

Rheological methods characterise gluten by measuring characteristics such as extensibility and resistance to extension of the dough; hydration time; maximum development time; and tolerance to breakdown at a predetermined consistency during mechanical mixing (Mailhot & Patton, 1988). The Chopin Alveograph (AACC 54-30A) or Extensigraph (AACC 54-10) are generally used to measure the resistance to extension and the extensibility of the fully developed dough (Mailhot & Patton, 1988). The Brabender Farinograph (AACC 54-21*)* and Mixograph (AACC 54-40A) measures properties such as optimum mixing time and stability of the dough during the mixing process.

Quantitative quality analysis on flour include protein quantification using the protein combustion method (AACC 46-30), the Kjeldahl method (AACC 46-10) and/or near infrared (NIR) spectroscopy (AACC 39-11); moisture analysis using the oven method (AACC 44-15A) or NIR spectroscopy; starch damage (AACC 76-30A); α-amylase activity using the Hagberg falling number method (AACC 56-81B) and colour determination with the Satake Colour Grader (previously known as the Kent-Jones Colour Grader) (FTP Method No. 0007/3, 7/1991) (SAGL, 2001). These methods are commonly used in the South African cereal industry. Other suitable, reliable, certified testing methods can also be used and can be accredited as in-house developed methods.

2.5 The breadmaking process

The required ingredients for breadmaking, i.e. flour, water, yeast, salt and pre-mixes are mixed in a mixing bowl and transformed to a homogeneous dough mass which includes tiny foam-like air bubbles (Bloksma & Bushuk, 1988). The dough is proofed to allow fermentation to take place (Bloksma & Bushuk, 1988). During fermentation the development of the dough continues and the yeast converts sugar to carbon dioxide and ethanol. The carbon dioxide is responsible for the expansion of the dough and the fully developed gluten structure for the retention of the gas (Bloksma & Bushuk, 1988). When the dough has reached the desirable volume, the loaves are baked at *ca.* 240˚C. During the baking process the dough expands even further (oven rise) as the volume of the gas increases. The dough is turned into breadcrumb and crust and the foam structure is transformed into a more porous structure (Bloksma & Bushuk, 1988).

3. Maize quality

3.1 Background

Evaluation of maize quality is generally limited to visual inspection and laboratory milling tests (Watson, 1987). Visual tests include inspection of the kernels for blemishes and insect damage. The hardness of maize kernels is also determined because of its importance during the maize milling process as it determines the milling performance. Hectolitre mass is commonly measured for maize in the United States (Watson, 1987), but in South Africa there is no formal standard or regulation in place for HLM determination of maize. End-user determined tests, i.e. vitamin A content, fat content, ash and particle size determination are performed on the maize flour (Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006). Additionally, producers of maize meal strive to produce a product that is as white as possible without adding bleaching agents; this adds to the importance of the colour test (Arie Wessels, Sasko Strategic Services, Paarl, South Africa, personal communication, 2006).

3.2 Maize grading

The South African maize grading system has grading standards for class white maize (WM) and class yellow maize (YM), which is respectively classified into three grades, i.e. WM1, WM2 and WM3 and YM1, YM2 and YM3 (Appendix 2) (Anon., 1998b). The grading system allows standards for the following deviations for class white maize: a) foreign material (0.3%); b) defective kernels above and below the 6.35 mm round hole sieve (7%); c) other coloured maize kernels (3%); d) collective deviations for a, b and c (8%), provided that the deviations are individually within their specified limits; and pink maize kernels (12%) (Anon., 1998b). A consignment of maize, which does not comply with the standards, specified for class white or class yellow maize is classified as class other maize and no standards are determined for this class (Anon., 1998b). No official HLM determination method for maize exists in South Africa, however, if measured the lower platform of the South African HLM device is used. In the US standards, provision are made for three classes of maize i.e., yellow maize, white maize and mixed maize (FGIS, 1984a) and unlike in South Africa a well established procedure of HLM determination exists, utilising the funnel and quart cup method. The results, however, are reported in pounds per bushel (Watson, 1987).

3.3 Maize milling

The major end product of the dry milling process is maize meal (Alexander, 1987) and that of wet milling, highly refined starches and sweeteners for the food industry (May, 1987). Both these processes will only be described briefly.

3.3.1 Dry maize milling

The maize is cleaned in two steps i.e., dry cleaning and wet cleaning (Alexander, 1987). During dry cleaning material such as metal, pieces of cob and broken kernels are removed whilst during wet cleaning surface dirt, dust and rodent excreta are removed from the kernel. After the wet cleaning process the maize is conditioned to 12 to 15% moisture content in tempering bins (Alexander, 1987; Fowler, 1993). The bran and germ is subsequently removed from the endosperm (leaving the samp intact) with a Beall degerminator (Alexander, 1987; Fowler, 1993). The endosperm is subsequently dried, cooled and sifted. Part of it is isolated as large flaking grits, whilst the rest is conveyed to the roller mills for reduction into smaller sizes (Alexander, 1987, Fowler, 1993). The bran and germ is further processed as the 'through stock' stream to produce other by-products of the dry milling process.

3.3.2 Wet maize milling

The maize is cleaned on vibrating screens to remove both coarse and fine material (May, 1987). The milling process involves softening of the maize in water (steeping) under controlled conditions in terms of temperature, time, sulphur dioxide and lactic acid contents. Subsequently the maize is transferred to coarse grinding mills to separate the germ from the rest of the kernel (May, 1987). After the removal of the germ the maize slurry is screened to separate fibre from the starch and gluten, whilst the gluten is removed from the starch by means of centrifuging. Finally the starch slurry is washed with water in a counter current manner to purify the starch (May, 1987).

3.4 Maize for distilled products

Maize is used in the production of whiskeys and other alcoholic drinks (Watson, 1987). Distillers are interested in obtaining the highest yield of whiskey consistent with acceptable flavour. Therefore, only high starch hybrids are sought after. Naturally ear-dried maize is preferred but, they will accept low-temperature dried maize if it is of superior quality. The absence of mouldy kernels is important, as well as high HLM and a low level of stress cracks (Watson, 1987). Maize quality relating to high HLM values is therefore of importance for production of distilled products.

4. Hectolitre mass

4.1 Background

Hectolitre mass determination is believed to have been performed as early as the 17th or 18th centuries and to be of British origin (Greenaway *et al.*, 1977). The first reported HLM determination was, however, performed in 1858 and the result was used as a grading factor for spring wheat in Milwaukee, Wisconsin (Phillip *et al.*, 1936). The Chicago Board of Trade adopted this measurement as a grading factor for spring wheat in 1859 (Phillip *et al.*, 1936). However, little is known about the design of early HLM devices or the procedure used to perform the tests.

It is recommended by the International Organisation of Legal Metrology (OIML) that an HLM instrument capable of receiving 20 L of grain in its measuring receptacle is used as a national standard (Anon., 1974). National standards can be made and used in accordance with the specifications described in the International Recommendation of the OIML R 15, Edition 1974 (E). The South African grading system uses a funnel equipped HLM device that provides uniform packing in a 0.5 L container. The excess grain is levelled with a wooden scraper (10 mm thick, 40 mm in width, at least 100 mm long and one edge must be rounded) and the mass of the grain in grams is divided by five to convert it to kilogram per hectolitre ($kg.hL^{-1}$) (Anon., 1998). Similar devices and methodologies are uitilised in other countries such as Canada and North America. Another type of HLM device consists of a cylindrical device (chondrometer) where one column of grain is isolated with a cutter from another column underneath (known volume) which is then filled with grain in a controlled manner. Chondrometers are utilised in countries such as Australia, United Kingdom, France and Germany. The grain collected in the cylinder of known volume is weighed and converted to $kg.hL^{-1}$ using appropriate conversion tables.

The continued use of HLM as a grading factor over the years implies that it reflects to some extend useful information regarding the milling quality of wheat (Mangels & Sanderson, 1925). However, few grain specialists consider HLM for maize of value since it does not seem to relate to quality factors to the same extend as it does to wheat (Hall & Hill, 1973). Hall and Hill (1973) suggested that HLM determination for maize should either be removed from the standards or proof should be given that HLM is of value regarding maize quality.

4.2 Relationship between hectolitre mass and the milling yield of flour

Hectolitre mass is most frequently associated with the indication or prediction of flour yield (Mangels & Sanderson, 1925; Barmore & Bequette, 1965; Anon., 1998a). Flour yield is defined as the percentage of straight grade flour obtained from a given mass of wheat after cleaning and scouring, but before tempering has occurred (Mangels & Sanderson, 1925).

It has been shown that heavy, plump wheat will yield more flour than light, shrivelled wheat (Mangels & Sanderson, 1925; Barmore & Bequette, 1965; Anon., 1998a), implicating that the flour yield potential is expected to increase with increasing HLM values and *vice versa*. In addition, it has been shown that immature or shrivelled kernels, that are less dense with lower endosperm to bran ratios and consequently decreased HLM, will also have reduced flour yield (Halverson & Zeleny, 1988). It has also been pointed out that above a HLM value of 73.4 kg.hL^{-1} the HLM of wheat has little influence on the milling yield, but that at lower HLM values the milling yield will decrease rapidly as the HLM decreases (Halverson & Zeleny, 1988).

Contradictory results have been published over the years regarding the relationship between HLM and flour yield. A high positive correlation was found between HLM and flour yield for seven crop years, 1916 and 1919-1924, with a correlation coefficient of 0.72 for the 1916 crop and an average correlation coefficient of 0.77 for the years 1919-1924 (Mangels & Sanderson, 1925). When the correlation between HLM and milling yield was reported for the averages of three crop years (1956-1958), the individual results showed poor correlation (Shuey, 1960). It was also observed that some wheat cultivars inherently have low HLM values but yield more flour than cultivars with similar or higher HLM values (Shuey, 1960). Conversely, cultivars can have an increase in HLM values, due to handling, without having changed in their flour yields (Shuey, 1960). It has been shown by Shuey (1960) that cultivars can differ in HLM by as much as 13.1 kg.hL^{-1} without differing in flour yield (Shuey, 1960). In a study of 28 Canadian Western Amber Durum (CWAD) wheat samples, a correlation coefficient of 0.86 was found between semolina yield and HLM values (Irvine, 1964). A low correlation coefficient (r = 0.32) was found between HLM and flour yield for soft red wheat cultivars (Gaines, 1991) and a correlation coefficient of 0.50 (P < 0.01) was found between micro HLM testing (70 g) and flour yield (Ohm *et al.*, 1998). A significantly high correlation (r = 0.82; P < 0.05) between HLM and potential flour yield was reported in a later study done with Argentine triticale (a cross between durum wheat and rye) (Aguirre *et al.,* 2002).

Several studies have, however, also been done which showed that no correlation exists between HLM and flour yield. Barmore & Bequette (1965) found that HLM was a poor estimate for flour yield for Pacific Northwest White (PNW) wheat and that this wheat had lower HLM values than common white cultivars but still had a higher flour yield regardless of cultivar, area of production, crop year or HLM. It was pointed out that these factors should be considered when grading PNW white wheat (Barmore & Bequette, 1965). Similar results were later obtained with soft white wheat cultivars where no correlation (r = 0.09) was observed (Gaines, 1991). Low correlations between HLM and flour yield for soft (r = 0.17) and hard (r = 0.41) wheat were, respectively, found in later research (Hook, 1984) while Schuler *et al.* (1995) found no correlation between HLM and flour yield (r = -0.24).

Although many researchers have explored the correlation between HLM and flour yield it seems that no general conclusion has been reached (Hook, 1984). It has been suggested by some researchers that HLM cannot be used as an indicator of flour yield as the correlation seems to be higher within a single cultivar than between cultivars (Lockwood, 1960; Greenaway *et al.*, 1977).

Hectolitre mass as a quality indicator for maize has not been proven very useful (Dorsey-Redding *et al.*, 1991). However, it has been shown that maize with low HLM has lower percentage of hard endosperm and therefore produces lower yield when milled (Rutledge, 1978); thus HLM can be useful as an indicator of milling yield of maize.

4.3 Relationship between hectolitre mass and thousand kernel weight

Thousand kernel weight (TKW) is the average weight of a kernel, with a factor of 1000 included to provide the necessary precision of the measurement (Hlynka & Bushuk, 1959). Thousand kernel weight is a function of kernel size and density (Halverson & Zeleny, 1988, Dorsey-Redding *et al.*, 1991) and it gives the miller important information regarding the flour yield of wheat considering that large, dense kernels normally have higher ratio of endosperm to bran than smaller, less dense kernels (Halverson & Zeleny, 1988). Thousand kernel weight (TKW) is correlated with kernel size, as large kernels will weigh more than small kernels, but there is no correlation between kernel size and HLM (Hlynka & Bushuk, 1959; Yamazaki & Briggle, 1969). Results of 30 cultivars cultivated at seven locations showed a highly significant correlation ($r = 0.75$; $P < 0.01$) between kernel weight and HLM results, suggesting that factors which has an influence on kernel weight also has an influence on HLM (Ghaderi & Everson, 1971). A significant ($P < 0.01$) correlation ($r = 0.47$) was also found between HLM and kernel weight by Ohm *et al.* (1998). The positive correlation between HLM and kernel weight was reported to be due to environmental effects (Ghaderi & Everson, 1971). Little genetic (varietal) correlation was, however, found between kernel weight and HLM, suggesting that there were no genes in common controlling kernel weight and HLM in the cultivars studied (Ghaderi & Everson, 1971). Poor correlations between HLM and TKW are probably influenced by differences in cultivars, especially in TKW (Phil Williams, PDK Grains, Nanaimo, British Columbia, Canada, personal communication, 2007). It was later reported that TKW correlated with milling yield of semolina. The TKW ranged from 18-54 g with a corresponding range in HLM from 72-86 $kg.hL^{-1}$ (Matsuo & Dexter, 1980). Thus a clear correlation of kernel weight with HLM was indicated. Dexter *et al.* (1987) pointed out that samples with low kernel weight also tend to give low HLM results. Dorsey-Redding *et al.* (1991) found no correlation between TKW and HLM for maize in a study done over two crop (1987-1988) years.

Despite the fact that some correlation may exist between HLM and TKW, the latter cannot be used as an alternative to HLM to predict flour yield, as no correlation ($r = 0.02$) was found between TKW and flour yield (Hook, 1984). It was also pointed out that the correlation of TKW with flour yield is not very high, especially at high values and between cultivars (Gooding & Davies, 1997). Furthermore, the TKW measurement is not as convenient to perform compared to HLM determination (Gooding & Davies, 1997).

5. Factors influencing hectolitre mass

5.1 Background

Various factors have an effect on HLM determinations; the most important being those that occur when the wheat is still growing in the field. Environmental effects such as drought and rainfall can have a huge effect on the HLM determination of grain whilst damage due to insect infestation or fungi can be detrimental to this measurement. Additionally the shape and size of the kernel can influence the way the kernels pack into the HLM test container. For example kernels with an irregular shape do not allow close packing of the kernels in the container and hence the HLM value will be reduced.

5.2 Environmental conditions

Environmental conditions can cause changes to occur in the grain kernels before harvest e.g., periods of drought during grain filling (Weibel & Pendleton, 1964), alternate wetting and drying cycles (Yamazaki & Briggle, 1969) and inclement weather causing delayed harvest (Czarnecki & Evans, 1986). The added moisture due to rainfall will decrease the density of the endosperm and as a result the HLM will be lower (Lockwood, 1960). The moisture on the bran coat may cause it to roughen and in turn this will have a negative effect on the packing properties of the wheat and subsequently the HLM values, which will be lower (Milner & Shellenberger, 1953). Occurrence of rain during the last stages of maturity will not affect the mass of the individual kernels significantly but might result in kernel expansion and consequently, low HLM values (Ghaderi & Everson, 1971). Hall (1972) reported that maize samples that were harvest later had a lower HLM than samples that were harvest earlier. He observed that weathering in the field were responsible for the lower HLM values obtained in the samples that were harvested at the later stage.

Results from a study carried out over two years showed average HLM values to decrease for five cultivars cultivated in Canada due to moderate rainfall (Czarnecki & Evans, 1986). The cultivars differed in HLM reduction, with the cultivar Neepawa showing the largest reduction of 2.9 and 4.2 $kg.hL^{-1}$ in successive years. Rain-induced field sprouting will also reduce the HLM of wheat (Donelson *et al.*, 2002). On the other hand drought will have a shrivelling effect on the kernels which will cause the HLM values to decrease (Halverson & Zeleny, 1988; Donelson *et al.,* 2002).

When HLM values of cultivars grown at different localities were compared, results indicated that drier climates produced wheat with higher HLM values than cultivars grown in more humid localities (Gaines *et al.*, 1996). It was found that the wheat grown in the humid locality was softer than the wheat grown in the drier localities and that the drier, harder wheat had better flour yield potential than the softer wheat. Wheat cultivated in drier localities, therefore, had better overall quality. The effect of humidity and altitude on HLM determinations of wheat samples, grown at the same locality has also been observed (Jannie Hanekom, Sasko Strategic Services, Paarl South Africa, personal communication, 2006). Hectolitre mass determinations performed on the same wheat sample at different altitudes also resulted in different HLM values.

5.3 Packing efficiency

Packing efficiency (the percentage of bulk volume occupied by grain) has an effect on the HLM values of grain (Yamazaki & Briggle, 1969; Ghaderi *et al.*, 1971; Greenaway *et al.,* 1977; Dorsey-Redding *et al.*, 1991). Irvine (1961) reported that hard red spring wheat samples with a constant kernel density of 1.43 $g.cm^{-3}$ and the same moisture content delivered HLM values ranging from 67-84 $kg.hL^{-1}$. He came to the conclusion that HLM is influenced mostly by packing quality, which is affected by the size and shape of the kernel. In other words when moisture and density were kept constant another factor brought about the changes in HLM values and Irvine (1961) concluded it to be the packing quality of the kernels. Cultivar consistency in packing efficiency has been shown in soft wheat, i.e. high HLM cultivars had high packing efficiency and low HLM cultivars low packing efficiency (Yamazaki & Briggle, 1969). Cleaned grain showed a higher packing efficiency than unclean grain (Yamazaki & Briggle, 1969) with subsequent higher expected HLM values. It has been found that packing efficiency may be reduced by broken, split, flattened or shrivelled grain (Yamazaki & Briggle, 1969). Kernels with an unusual shape allows for spaces between the kernels and therefore also result in loose packing of the kernels (Lockwood, 1960). Shrivelled kernels inhibit uniform packing and bring about different responses in HLM through the presence of planar and concave surfaces mixed with normal convex contours of the intact kernel (Yamazaki & Briggle, 1969; Ghaderi & Everson, 1971). The removal of badly shrivelled and damaged grain improved the HLM values by as much as 3.6 $kg.hL^{-1}$ (Schuler *et al.*, 1994). Large kernels result in higher HLM than small shrivelled kernels. Plump kernels pack more uniformly and result in higher HLM values, whereas small kernels, usually more elongated, pack more randomly and loose to give rise to lower HLM values (Dick & Matsuo, 1988). Trocolli and Di Fonzo (1999) reported a significant high correlation (r = 0.98; P = 0.001) between HLM and packing efficiency and concluded that differences in packing efficiency, and therefore HLM, is affected by kernel shape and not necessarily size which was in agreement with Yamazaki & Briggle (1969).

Another characteristic of the kernels, which influence packing efficiency, is the smoothness of the kernel which is affected by hairs on the kernels and the condition of the kernel surface (Barmore & Bequette, 1965). Hair on the surface of the kernels does not allow close packing and thus result in lower HLM. The smoothness of the kernel is dependent on cultivar, wetting and drying after maturation and the amount of handling it undergoes (Swanson, 1946). Frequent handling and moving of the grain may polish the bran coats and causes the HLM to increase because less surface friction is present between the kernels, causing them to pack more closely in the test container (Swanson, 1946; Shuey, 1960; Halverson & Zeleny, 1988). On the other hand, kernels with a rough texture can cause the HLM to decrease, because the weathered surface of the kernels does not allow close packing of kernels (Barnes, 1989; Schuler *et al.*, 1994).

It is clear that HLM is influenced by the way that kernels pack in a container. The shape of a kernel, rather than its size, can influence the packing efficiency of wheat. The removal of shrivelled kernels will, therefore, improve packing efficiency and increase the HLM value of wheat. The smoother the kernel surface the more efficient the packing will be in the measuring container and the higher the HLM value.

5.4 Kernel density

Density of grain is normally measured with a pycnometer (Yamazaki & Briggle, 1969; Ohm *et al.*, 1998; Troccoli & Di Fonzo, 1999). A method for the determination of density as described by Yamazaki & Briggle (1969) is as follows: Grain is poured into the cup of a Beckman Air Comparison Pycnometer to overflow it from a funnel suspended above; an arrangement similar to that for HLM determination. The excess grain is evened with the top of the cup and the content of the cup is transferred to the pycnometer to determine the volume where after the grain is weighed. Density ($g.mL^{-1}$) values are determined from the volume and weight obtained as described.

Hectolitre mass per definition is the measure of the density of wheat and many researchers have delved into the relationship between density and HLM (Lockwood, 1960). The density of the kernel can influence the HLM of the grain, i.e. wheat that is dense has a high HLM and oats for example that is less dense than wheat have a lower HLM (Hlynka & Bushuk, 1959; Halverson & Zeleny, 1988). It is also true that variation in density of the same grain may be sufficient to be reflected in the HLM values (Hlynka & Bushuk, 1959). A relationship was found between HLM and density of grain in a study of ten UK winter wheat cultivars (Pushman & Bingham 1975). Additionally it was found that HLM was related to grain density rather than to TKW and flour yield (Pushman & Bingham 1975). Pomeranz *et al.* (1986) illustrated a positive correlation between HLM and the density of maize. Significant correlations (r = 0.78 and 0.80; P = 0.0001) were also found between density and the HLM of maize for two crop years (Dorsey-Redding *et al.*, 1991).

As density of wheat is determined by the environment whilst growing (Yamazaki & Briggle, 1969; Halverson & Zeleny, 1988), kernels that have matured under adverse conditions may not fill out normally and thus have lower density than usual (Hlynka & Bushuk, 1959; Yamazaki & Briggle, 1969). Low HLM is associated with low grain density due to air-filled spaces in the endosperm or the separating layers of the pericarp (Bayles, 1977). Alternate wetting and drying through weathering also play a significant role in the determination of density in wheat (Yamazaki & Briggle, 1969). A correlation coefficient of 0.57 (P = 0.05) was found between average HLM and density values for seven cultivars (Yamazaki & Briggle, 1969). The biological structure of the grain and the chemical composition, including its moisture content, also has an influence on the density (Halverson & Zeleny, 1988). At moisture contents higher than 12% the bulk density of the grain decreased as such that HLM also decreased (McLean, 1987). A correlation coefficient of 0.70 (P = 0.01) between HLM and kernel density was reported in a later study (Troccoli & Di Fonzo, 1999). Low correlation coefficients (r = 0.17 & r = 0.3; P = 0.06) were obtained between HLM and density in studies done by

Ghaderi *et al.* (1971) and Schuler et al. (1994), respectively, whilst no correlation was reported in a later study (Schuler *et al.*, 1995).

5.5 Protein content

The density of starch (1.51) is higher than the density of wheat gluten (1.29) (Ghaderi *et al.*, 1971), but it has been shown that increased protein content may lead to higher kernel density. This is due to the packing of protein into the spaces between the starch granules in the endosperm, thus increasing the HLM (Pushman & Bingham, 1975). Flour protein was shown to correlate moderately with HLM (r = 0.56; P = 0.05) and it was found that when flour protein content increased so did the HLM (r = 0.56; P = 0.004) (Schuler *et al.*, 1994). A moderate correlation (r = 0.55; P = 0.01) was also found between HLM and protein content in 20 UK wheat cultivars recommended in 1995 (Anon., 1995). The correlation (r = 0.64) was higher in 25 cultivars grown in the absence of nitrogen fertiliser and agrochemicals (Thompson, 1995).

Low HLM is associated with low density and low protein content in soft wheat because of the mealyness, which is a result of air spaces (Yamazaki & Briggle, 1969). It was reported that low HLM maize was of superior feeding quality over high HLM maize due to the relatively higher protein levels in the low HLM maize (USDA, 1933). In a later study low correlations (r = 0.2 and 0.15; P = 0.05) were found, for the 1987 (n = 183) and 1988 (n = 195) crop years, between the HLM of maize and protein content (Dorsey-Redding *et al.*, 1991). A low correlation (r = 0.11) was obtained between HLM and kernel protein within low protein wheat samples (Ghaderi *et al*, 1971) while HLM was not significantly correlated (r = -0.103; P < 0.05) with flour protein content of Argentine triticale (Aguirre *et al.*, 2002). No relationship was found between HLM and protein content when Neepawa Canada Western Red Spring (CWRS) wheat (Tipples *et al.*, 1977) or several durum wheat cultivars (Dexter *et al.*, 1982) were grown under a wide range of nitrogen fertiliser levels. Dexter *et al.* (1987) found a negative relationship between HLM and protein content of Canada Western Amber Durum (CWAD) during the 1984 (r = -0.95; P < 0.01) as well as the 1985 (r = -0.91; P < 0.01) crop years. A strong negative response (r = -0.75; P < 0.05 and r = -0.93; P < 0.01) was found between HLM and protein in two cultivars of Canada Prairie Spring wheat, over three crop years (1989-1991) (Preston *et al.*, 1995). It was suggested that the decrease in HLM with increased protein content may be due to environmental stress (drought) rather then a direct response to protein content, because the kernels seemed to be less plump at high protein content (Preston *et al.*, 1995). Gaines (1991) observed that protein content can be either positively or negatively associated with HLM.

5.6 Moisture content

An increase in moisture content results in low HLM values and wheat of low moisture content is generally high in HLM (Hlynka & Bushuk, 1959). An increase in moisture content will cause the HLM to decrease, as the density of water is 1.0 whereas that of starch is 1.51 (Lockwood, 1960). The presence of water causes the grain to swell in size therefore, reducing the number of kernels that can fit into the test container (Lockwood, 1960). Consequently, the HLM value of the grain will be lower and the damper the wheat the greater the effect. It has already been recommended earlier that the HLM should be done immediately on wet grain because HLM increases with drying (Boerner & Ropes, 1922).

Drying of shelled maize caused an increase in HLM as dry maize has a lower coefficient of friction than wet maize which causes it to pack closer together compared to wet maize (Hall, 1972). The increase was rapid at the earlier stages of drying but decreased during the later stages. It was observed that the decrease was greater at lower temperatures when four different temperature (21, 49, 77 and 104°C) levels were investigated (Hall, 1972). The HLM value declined after a maximum had been reached, thus emphasising the importance not to over dry the maize. It was suggested that HLM for maize may be of value as a measurement of kernel density if all samples are tested at a constant moisture content of, e.g. 15.5% (Hall, 1972).

The HLM of a mixed wheat sample increased with 0.34 $kg.hL^{-1}$ for each 1% decrease in moisture content, when it was dried from approximately 20% to 12.5% moisture content (Pushman, 1975). The effects of wetting compared to drying were studied in four cultivars where samples with initial moisture content of 16% were either dried or wetted to produce a range of moisture contents from 11 to 20%. It was found that one cultivar had a different response to changes in moisture content than the other cultivars had (Pushman, 1975). The addition of water reduced the average HLM of the four cultivars with *ca.* 1.65 $kg.hL^{-1}$ per 1% increase in moisture content and during drying the increase in the average HLM was 0.28 $kg.hL^{-1}$ per 1% decrease in moisture content (Pushman, 1975).

The effects of wetting were further investigated with two cultivars, where each cultivar was mixed and divided into sets of two samples each (Pushman, 1975). One sample of a set was treated to give a range of moisture contents as described above and the second sample was dried to approximately 11% moisture content (Pushman, 1975). Water was then added to bring this latter sample to approximately the moisture content at which the corresponding sample had been tested. Samples that were dried followed by wetting showed a marked hysteresis effect and were lower in HLM than the samples that were wetted directly to moisture contents between 11 and 17 % (Pushman, 1975). Thus, samples that were dried and then wetted resulted in a greater reduction in HLM than samples that was only subjected to wetting.

In a later study the increase in moisture content reduced the mean HLM of four cultivars by approximately 1.33 $kg.hL^{-1}$ per 1% moisture content increase and for drying the increase in average HLM was 0.27 $kg.hL^{-1}$ per 1% decrease in moisture content (Hook, 1984). When the previously dampened wheat was dried, and the previously dried wheat was dampened, the situation was reversed (Hook, 1984). In this case drying had a greater effect on the HLM than wetting. Successive wetting and drying of wheat can occur in the field, during storage, in transit and at the mill and when HLM is tested to give an indication of flour yield it may be different from the initial value because of the various moisture changes (Hook, 1984). The relationship between moisture content and HLM differs between cultivars and HLM values at certain moisture contents can alter depending on whether the grain was wetted or dried (Mclean, 1987).

It has been suggested that wheat that has been wetted can never return to its original state and therefore the subsequent change in HLM values (Jannie Hanekom, Sasko Strategic Services, Paarl, South Africa, 2006). The wheat kernel seems to swell and increase in size and the endosperm is separated from the bran coat. When the kernel is dried there will still be spaces between the endosperm and bran and due to that the HLM will be lower. The bran coat also roughens when wet and this prevents close packing of the kernel in the test container and therefore the HLM will be lower. Due to the known lower HLM of wet wheat compared to dry wheat conversion tables were developed for wet wheat in order to determine the minimum HLM of the wet wheat after drying (Sierk Ybema, Senwes, Klerksdorp, South Africa, personal communication, 2006). Earlier studies have shown that adjustment of HLM values to constant moisture content cannot be comparable unless the previous treatment of the grain is known (Pushman, 1975). Care should be taken when correction factors are applied to HLM values in order to convert the value to a fixed moisture content base, because different moisture correction coefficients would apply to each type of wheat (Hook, 1984). It is not known why different rates of change occur, within cultivars, with changes in moisture content but speculation leaves possibilities of the differences in the ratio of hard to floury endosperm and environmental differences, *per se*.

5.7 Impurities

The amount of impurities such as seeds and chaff can have an influence on HLM. Small round seeds fit into the spaces between the kernels and can therefore increase the HLM (Lockwood, 1960). Light and bulky impurities such as chaff and straw decrease the HLM by preventing close packing of the kernels in the container (Lockwood, 1960). Wheat that underwent screening and aspiration also showed an increase in HLM (Lockwood, 1960). Screening polishes the wheat and allows it to pack more closely in the container and aspiration removes the impurities (Lockwood, 1960). The consequential increase in HLM by applying the latter processes can be as much as 4 $kg.hL^{-1}$. An increase of up to 2.07 $kg.hL^{-1}$ was obtained in a later study, after the removal of dockage (Greenaway *et al*., 1971). In the South African grading system, HLM is measured on dirty wheat (wheat where the screenings or dirt has not been removed with the standard grading sieve) and the grade allocated accordingly (Anon., 1998a). In the South African grading system the term "screenings" refers to all material that passes through the standard grading sieve (a grooved hand sieve made from 1 mm stainless steel with openings 1.8 mm wide and 12.7 mm long, mounted in a plastic frame that fits in an aluminum pan with a solid base of 330.2 – 333 mm in diameter) (Anon., 1998a). Some silo owners, however, determine HLM on wheat received from the farmer on a cleaned sample. The sample is cleaned prior to grading by cleaning it with a laboratory cleaner (Sierk Ybema, Senwes, Klerksdorp, South Africa, personal communication, 2007).

5.9 Operator

Highly significant differences (P = 0.01) were found between two operators for five different grains (Greenaway *et al*., 1971). However, one operator was more skilled and experienced in performing HLM tests than the other one. It was, further, found that the operators' techniques were not consistent and that the results would be better if one or both of the operators' skills were improved. This is an indication that operators should receive sufficient training and develops adequate skill in order to be more consistent when performing HLM tests. In a later study highly comparable results were obtained between two operators who performed HLM tests on a HLM and bushel device, respectively (Greenaway *et al*., 1977). Correlation coefficients for tests done with wheat on the HLM and bushel device, by the two operators, were 0.998 and 0.997, respectively. For maize correlation coefficients of 0.986 and 0.987 were obtained with the HLM and bushel devices, respectively (Greenaway *et al*., 1977). Although the tests were performed with other grains as well only data relevant to this study is reported.

5.10 Comparing different devices

In a study done with an automatic (a simulation of the manual operations usually done by a grader e.g. the zigzag motions used to strike off the excess grain from the measuring cup prior to weighing) and a manual (standard weight per bushel) apparatus respectively, results showed that HLM for five cereal grains agreed to within 1.55 $kg.hL^{-1}$ or less (Greenaway *et al*., 1971). These results were within the tolerance of ±1.55 $kg.hL^{-1}$ as required for the automatic device. However, a slight difference was noted for flaxseed and soybeans, which exceeded the 1.55 $kg.hL^{-1}$ tolerance. The differences in HLM for flaxseed and soybeans were found to be attributable to the automatic device and it was suggested that research must continue until the results obtained on the automatic device are in better agreement with the manual device. Variability within the samples also played a role in the disagreement between the two devices (Greenaway *et al*., 1971). Automatic devices of 1971 and the equipment available today are technically substantially different from each other. The new devices are able to give excellent results, which is even better than the manual method, since the human error factor is absent (Sierk Ybema, Senwes, Klerksdorp, South Africa, personal communication, 2007).

A study was done to compare a 1 L HLM (most commonly used in Europe) device and a standard USDA bushel device in order to convert test weight to HLM (Greenaway, *et al*., 1977). It was reported that the correlation coefficients between the HLM and bushel devices were high. Tests done with wheat and maize delivered the correlation coefficients of 0.986 and 0.992, respectively (Greenaway, *et al*., 1977). Theoretical correction factors as cited in Greenaway *et al.* (1977) for converting HLM to test weight and *vice versa* are 0.7768 and 1.2873, respectively. According to the results reported the correction factors for converting HLM to test weight, for wheat and maize, were 0.7539 and 0.7877, respectively. The correction factors to convert test weight to HLM were 1.3264 and 1.2695 for wheat and maize, respectively. The differences between the theoretical correction factors and those obtained by Greenaway *et al.* (1977) were suggested to be due to the physical characteristics of the grain seed crops. As only one HLM and bushel device was studied the authors suggested that other devices and methods should be evaluated before official factors can be established.

6. Conclusion

The use of HLM as a grading factor is justified by its correlation to flour yield, in spite of HLM being reported to be a poor estimate of flour yield. This stems from the fact that the physical properties of wheat are influenced by factors such as moisture content, packing efficiency and environmental conditions which alters its HLM. For example the HLM of wheat that underwent wetting, decreased and did not reflect the real flour yield of that wheat. Moisture correction factors have been suggested but studies have shown that a single conversion factor cannot be used to adjust HLM values to a constant moisture basis; unless the moisture treatment history of the grain is known.

The following general conclusions can be made from the foregoing literature review.

- Hectolitre mass can be used as a rough indicator of flour yield.
- There is no correlation between HLM and 1000 kernel weight.
- Thousand kernel weight cannot be used as a substitute to HLM to predict flour yield.
- Environmental conditions such as drought and rainfall influence HLM.
- Grain free of impurities and shrivelled kernels has a better packing efficiency, thus increasing the HLM.
- Smoothness of the kernel surface and shape of the kernel influences the packing efficiency.
- Density of the grain is reflected by its HLM value.
- Protein can either be positively or negatively correlated with HLM.
- HLM decrease as the moisture content increases and vice versa.
- Cultivars respond differently to the increase or decrease in moisture content.
- Correction factors cannot be used to correct for moisture changes in HLM.
- Impurities have an influence on HLM, therefore, it is suggested to use clean grain to perform HLM tests.
- Training of the operator is essential to obtain consistent HLM results.
- Hectolitre mass devices correlate well with pounds per bushel devices.

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CHAPTER 3

Assessment of variance in measurement of hectolitre mass of wheat, using equipment from different grain producing and exporting countries

Assessment of variance in measurement of hectolitre mass of wheat, using equipment from different grain producing and exporting countries.

Abstract

Hectolitre mass (HLM) is the weight of a standard volume of grain and is regarded as an indication of the density and soundness of the grain. Different wheat producing countries use either one of two types of hectolitre mass equipment. The respective devices consist of either a funnel that provides uniform packing in a container of known volume or of a cylindrical device where a column of grain is isolated with a cutter (metal blade which is slit through a column of wheat to separate one column of grain from another) from a cylinder of known volume underneath, which is then filled with grain in a controlled manner. Devices similar to the latter type are used in Australia, United Kingdom, France and Germany. Hectolitre mass devices equipped with a funnel are used for HLM measurements in South Africa, United States of America (USA) and Canada. The effect of these different types of devices on the variance in HLM values has been investigated in this study. It has been found that the South African device results in HLM values significantly lower ($P < 0.05$) than that of the other devices. The device currently used in Australia resulted in HLM values significantly higher ($P < 0.05$) compared to the other devices. Nevertheless it has been found that the devices correlate (ICC consistency >0.90) well with one another and correction factors can be calculated to convert between devices. The effect of several factors such as impurities, operator and moisture content on HLM measurements has also been investigated. A significant ($P < 0.05$) increase has been found in HLM values after the removal of impurities and it has been shown to be device dependent. Although a statistically significant ($P < 0.05$) operator effect has been observed it would be insignificant in practice as ICC agreement values >0.92 were obtained. When ten respective South African HLM devices were compared, statistically significant differences were found, but in practice would be insignificant (ICC agreement >0.98). Consecutive wetting and drying cycles significantly ($P < 0.05$) influenced the HLM determinations.

Keywords: wheat; hectolitre mass; hectolitre mass devices; impurities; operator; moisture content

Introduction

Hectolitre mass (HLM), also referred to in some countries as bushel-, specific- or test weight (Hook, 1984) is the weight of a standard volume of grain and it is generally believed to be a measure of the density and soundness of grain. This measurement is a very important indicator of the physical quality of wheat (Hlynka & Bushuk, 1959; Hook, 1984; Ohm *et al.*, 1998) and has long been recognised as a general indicator of the flour yield of wheat (Barmore & Bequette, 1965). Therefore, it is understandable that it is the most important wheat grading factor in South Africa (Anon., 1998) and other countries such as the United States of America (USA) and Canada (Hook, 1984). In South

Africa HLM is determined with a two-level HLM device and for wheat the measurement is performed with the measuring cup positioned on the higher of the two platforms. For other cereals such as maize, oats and sorghum HLM are measured with the measuring cup on the lower platform. The result is expressed in kilogram per hectolitre ($kg.hL^{-1}$).

Several factors could influence the HLM value of wheat, i.e. repeated analysis of the same wheat sample (Shuey, 1960); presence or absence of impurities (Lockwood, 1960); effect of different operators (Greenaway *et al.*, 1971) and the effect of change in moisture content (Pushman, 1974; Hook, 1984). It is believed that the HLM of wheat increases with sample handling because the kernels scour against each other causing polishing of the bran coat (Shuey, 1960). As a result the kernels pack more closely in the test container, which means that more kernels can fit into the container and a higher HLM measurement is obtained. HLM is also influenced by the presence or absence of impurities, which can have a negative or positive effect on the HLM value of wheat (Lockwood, 1960). The presence of impurities such as chaff can lower the HLM value as it does not allow for the close packing of wheat in the container and it is lighter in weight and more bulky than wheat (Lockwood, 1960; Greenaway *et al.,* 1971). Other impurities such as small round seeds can increase the mass of wheat in a container because they fill the little air spaces that naturally form between the wheat kernels (Lockwood, 1960). It is thus advisable to perform HLM measurements on clean, impurity free wheat.

In a study performed by Greenaway *et al.* (1971) it was shown that highly significant ($P < 0.05$) differences exist between HLM values obtained by different operators, due to inconsistent execution of the HLM measurements by an unskilled operator. It was, however, suggested that improved agreement between results could be achieved if the technique of one or both of the operators was improved (Greenaway *et al.*, 1971).

The influence of change in moisture content on the HLM of wheat is somewhat more technical than the other factors previously discussed. Wheat on the land is often exposed to wet and dry cycles due to rainfall and sunshine. When the wheat gets wet from rain the kernel expands and is less dense therefore the HLM decreases (Pushman, 1974). When it is exposed to sunlight or warm weather afterwards, the kernel will shrivel but will never return to its initial shape and density and although the HLM will be higher than that of the previously wet grain it will still be lower than that of the original wheat before wetting (Jannie Hanekom, Sasko Strategic Services, Paarl, personal communication, 2007).

One of two types of HLM equipment is being used in different countries and the South African grading system uses a device equipped with a funnel that provides uniform packing in a 500 mL measuring cup. The excess grain in the measuring cup is levelled with a wooden scraper and the mass of the grain in grams is divided by five to convert it to kilogram per hectolitre (Anon., 1998). Similar devices and methodologies are utilised in the USA and Canada. The other type of HLM equipment consists of a cylindrical device (chondrometer) where a column of grain is isolated with a cutter (metal blade which is slit through a column of wheat to separate one column of grain from another) from a cylinder of known volume underneath, which is then filled with grain in a controlled manner. Similar devices are used in Australia, United Kingdom (UK), France and Germany. The grain collected in the cylinder of known volume is weighed and converted to $kg.hL^{-1}$ using appropriate conversion tables. The effect of these different types of devices on the variance in HLM measurements has not been investigated to date.

The objectives of this study were therefore to evaluate:

- HLM equipment as used in Australia, Canada, France, Germany, UK and the USA in comparison to the South African device using mixed wheat and single South African wheat cultivar samples which covered a range of HLM values;
- HLM values obtained from ten respective South African HLM devices;
- effect of impurities on HLM determinations;
- effect of operator on HLM determinations; and
- effect of consecutive drying and wetting cycles on HLM determinations.

Materials and Methods

Wheat samples and hectolitre mass equipment

Wheat samples, obtained from wheat flour mills (Ruto Mills, Sasko Grains, Tiger Milling) and wheat breeding institutes (ARC-Small Grain Institute, Monsanto, Panar Seed), included South African wheat, imported wheat as well as single South African wheat cultivars. The samples were selected to cover a HLM range of *ca*. 10 $kg.hL^{-1}$ (Table 1). All the samples were stored at room temperature and fumigated regularly with pyrethroid insecticides to prevent infestation. The HLM equipment evaluated included devices from Australia (Graintec Pty Ltd., Peregian Beach, Queensland, Australia), Canada (Dimo's Tool & Die Ltd., Winnipeg, Canada), France (Chopin Technologies, Villeneuve-la-Garenne Cedex, France), Germany (KERN & Sohn GmbH, Barlingen-Frommern, Germany), USA (Seedburo Equipment Co., Chicago, USA), United Kingdom (Farm-Tec, Whitby, North Yorkshire, UK) and South Africa (Table 2). Two South African HLM devices were included in the evaluations. During this study all the HLM measurements were performed by the same operator, apart from Experiment 7 where the effect of different operators was evaluated. In Experiment 7 operator 1 refers to the operator who has performed the HLM measurements in this study and is referred to as the skilled operator.

Table 1 The HLM values (determined using South African HLM device) of the wheat samples before and after the removal of impurities, respectively

Sample number	Type of wheat	Origin/Cultivar	HLM (kg.hL^{-1}) before removal of impurities	HLM (kg.hL^{-1}) after removal of impurities
1	Mixed wheat (imported)	Argentina	78.58	79.18
2	Mixed wheat (imported)	Argentina	81.42	81.28
3	Mixed wheat (imported)	USA soft wheat	77.18	78.39
4	Mixed wheat (imported)	USA DNS* wheat	80.52	80.69
5	Mixed wheat (imported)	Australia	80.53	80.77
6	Mixed wheat (imported)	Ukraine	79.12	79.00
7	Mixed wheat (local)	Northern Cape	79.06	79.31
8	Mixed wheat (local)	Northern Cape	78.63	78.34
9	Mixed wheat (local)	Northern Cape	76.62	76.37
10	Mixed wheat (imported)	Germany	73.41	74.24
11	Mixed wheat (local)	Western Cape	82.49	81.66
12	Mixed wheat (local)	SST 88	78.24	78.51
13	Mixed wheat (local)	SST 036	74.55	75.45
14	South African cultivar	Elands	77.21	77.05
15	South African cultivar	Gariep	77.14	74.89
16	South African cultivar	Kariega	78.34	78.12
17	South African cultivar	Baviaans	77.72	77.92
18	South African cultivar	Olifants	80.95	81.15
19	South African cultivar	SST 399	73.43	73.35
20	South African cultivar	Carnia 826	74.45	74.75
21	South African cultivar	SST 322	70.85	71.24
22	South African cultivar	SST 806	74.90	74.88
23	South African cultivar	SST 027	79.82	79.96
24	South African cultivar	SST 88	79.99	79.84
25	South African cultivar	SST 015	77.29	77.84
26	South African cultivar	Pannar 3377	73.96	72.90
27	South African cultivar	SST 88	73.88	73.46
28	Mixed wheat (local)	Western Cape	74.27	75.26
29	Mixed wheat (local)	Western Cape	78.42	79.17
30	Mixed wheat (local)	Western Cape	77.31	78.27
31	Mixed wheat (local)	Western Cape	75.96	77.18
32	Mixed wheat (local)	Western Cape	77.18	77.58
33	Mixed wheat (local)	Western Cape	76.06	77.21
34	Mixed wheat (local)	Western Cape	77.33	79.18
35	Mixed wheat (local)	Western Cape	75.61	76.91
36	Mixed wheat (local)	Western Cape	76.51	77.14
37	Mixed wheat (local)	Western Cape	72.85	73.45

* Dark Nothern Spring

Table 2 Illustration and a short description of the HLM devices

Country	Description of HLM devices
 **Australia**	Aluminium 500 mL measure with filler and cutter bar.
 **Canada**	Ohaus 500 mL measure with Cox Funnel and round wooden striker. 500 mL measure supplied with certificate of calibration (calibrations performed traceable to national standard).
 **France**	Niléma Litre with filling hopper and cutter bar. Designed in accordance with the AFNOR NF *V 03*-719 (1996) standard and standardised to a 50 L French reference.
 **Germany**	Kern 220/222 Grain Sampler with filler and cutter bar. Compliant to ISO 7971-2:1995 standard.
 **SA**	South African two-level HLM device with funnel and 500 mL measuring container and wooden scraper.
 **UK**	Easi-Way Portable Hectolitre Test Weight Kit with cutter bar. Matched to EC 20 L volume (Directive 71/347/EC) and conforms to ISO 7971-2:1995 and BS 4371 Part 23 standards.
 **USA**	Seedburo 151 Filling Hopper with quart cup and strike-off stick.

Hectolitre mass equipment operating procedures

Australian Aluminium 500 mL measure

Position the container with the hole in its centre on top of the receiving container (500 mL). Do not insert the cutter through the slit as yet. Fill both containers through the filler hole. Insert the cutter through the slit. Discard the grain above the cutter. Weigh the grain in the bottom of the container using an automatic balance (Model, *PB1502-S Mettler-Toledo*, Scwarzenbach, Switzerland*)*. Convert the mass of the grain to $kg.hL^{-1}$, in order to determine the HLM of the grain, by dividing it by five.

Canadian Ohaus 500 mL measure and Cox funnel

Insert the slide into the Cox funnel to close the opening of the funnel. Fill the Ohaus measuring container (500 mL) with grain to be tested until it overflows. Pour the grain in the measuring container, plus an extra hand full, into the Cox funnel. Position the funnel on top of the measuring container in such a way that the notched legs of the funnel fits securely onto the rim of the container. Remove the slide from the opening of the funnel in one quick motion to ensure that the grain drops evenly into the measuring container. Carefully remove the funnel from the container while taking care not to disturb the grain in the measuring container. Any jarring at this stage will result in compaction of the grain and inaccurate results will be obtained. Position the round wooden striker on the rim of the container and scalp off the excess grain by means of three full-length zigzag motions. Determine the mass of the grain in the Ohaus measuring container in grams using an automatic balance. Convert the mass of the grain to $kg.hL^{-1}$ using the HLM conversion chart supplied with the device (Appendix 3).

French Niléma Litre

Place the hopper on top of the one litre container. Fill the hopper to the upper edge with even flowing grain. Open the valve of the hopper to release the grain into the container. Carefully insert the cutter into the slit. Hold the container firmly during insertion, to avoid vibration and settling of the grain. Remove the hopper from the container and weigh the grain in the container. Divide the weight of the grain (in grams) by 10 to convert the mass of the grain to $kg.hL^{-1}$.

German KERN 220/222 Grain Sampler

Fill the pre-filling measure with grain up to the level mark. Ensure that the piston is positioned on the straightedge (levelling blade) before filling the filling hopper. The piston falls smoothly down the measuring container and drives air through the exit holes in the base of the container. It controls the rate of fall and ensures the smooth flow of grain from the filling container into the measuring container. Pour the grain into the filling hopper, from a height of 3-4 cm above the hopper. Pull the straightedge from the slit in one quick motion, without jarring the device. Insert the straightedge into the slit after the grain has fallen into the measuring container (1 L) and push it through the grain. Do not knock, shake or jar the apparatus during the procedure as it could lead to erroneous results. Discard the excess grain on top of the straightedge and remove the filling hopper and straightedge. Weigh the grain (in grams) in the container and read the HLM in $kg.hL^{-1}$, corresponding to the weight of the grain, from the conversion chart supplied with the device (Appendix 4).

South African hectolitre mass device

Fill the funnel with enough grain to allow it to overflow and scrape off the excess grain, with the round edge of the wooden scraper, at an angle of 90º. Put the measuring container underneath the funnel on the raised platform of the device and open the valve to release the grain into the measuring container (500 mL). Move the funnel to the left of the kettle. Position the wooden scraper at an angle of 90º on the rim of the container and scalp off the excess grain in one quick, smooth motion. Determine the weight of the remaining wheat in grams. Convert the weight in grams to $kg.hL^{-1}$ by dividing it by 5.

UK Easi-Way Portable Hectolitre Test Weight Kit

Insert the cutter through the slit in the container and drop the plunger weight (piston) onto the cutter. Fill the cylinder with grain from a height of approximately 25 mm above the opening. Remove the cutter from the cylinder to allow the weight, together with the grain to descend into the lower chamber (600 mL). Re-insert the cutter through the column of grain to isolate the sample in the lower chamber. Discard the excess grain from the cylinder and remove the cutter. Determine the weight of the remaining grain in grams. Read the HLM in $kg.hL^{-1}$, corresponding to the weight, from the conversion chart supplied with the device (Appendix 5).

USA Seedburo 151 Filling Hopper

Assemble the device as described in the user manual supplied with the device. Fill the funnel with enough grain to overflow the kettle (measuring container, quart cup = 1100 mL). Open the valve to release the grain into the kettle. Move the funnel to the left of the kettle to provide space on top of the kettle. Position the wood striker on the rim of the container and scalp off the excess grain by means of three full-length zigzag motions. Determine the weight of the grain in the measuring container in grams. Convert the weight in grams to pounds per bushel ($lb.bu^{-1}$) and from $lb.bu^{-1}$ to $kg.hL^{-1}$ as indicated in the HLM conversion chart supplied with the device (Appendix 6).

Experimental Procedures

A schematic layout of the sequence of the experiments performed is depicted in Fig. 1 and the detailed layouts of each respective experiment in Figs. 2-9.

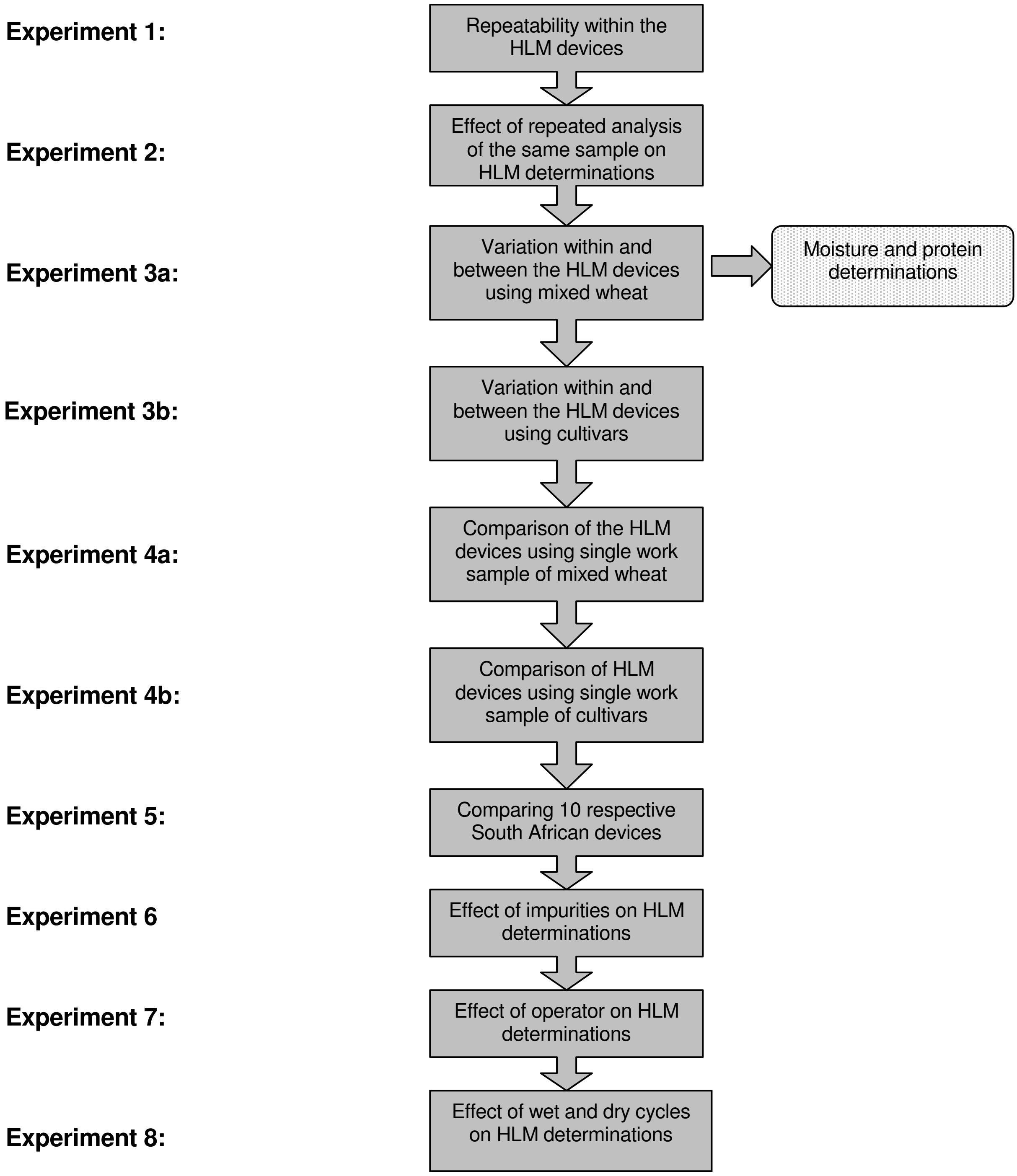

Figure 1 Schematic layout of sequence of experiments performed.

Experiment 1: Repeatability within the HLM devices (Fig. 2)

A 16 kg sample of wheat (Table 1; sample 6; HLM = 79.00 $kg.hL^{-1}$) was cleaned from impurities using a Carter Day Dockage Tester (Seedburo Equipment Co., Chicago, USA). The settings of the Dockage Tester were as laid out in Table 3. Subsequently the wheat was thoroughly mixed in a laboratory mixer (Model MR10L, Chopin Technologies, Villeneuve-la-Garenne Cedex, France) for 15 minutes. The wheat was divided into eight, two kilogram samples; two kilograms of wheat to be tested on each respective HLM device. The order of the devices, on which the HLM tests were performed, was randomly selected. Ten repetitions were performed on each device and between each repetition the two kilogram sample was mixed by pouring it, five times, from one bucket to another. The HLM was determined on each device according to the operating procedures described earlier.

Table 3 Settings of Carter Day Dockage Tester

			Sieves		
Air	**Feed**	**Riddle**	**Top**	**Middle**	**Bottom**
4	6	#2	#3	#6	#1

Experiment 2: Effect of repeated analysis of the same wheat sample on its HLM (Fig. 3)

Three different wheat samples (Table 1; 16 kg of each of samples 8, 11 & 13), with a difference of 6.21 $kg.hL^{-1}$ between the highest and lowest sample, were used. The impurities were removed from the samples using a Carter Day Dockage Tester. After removal of impurities each sample was mixed by pouring it three times through a Boerner Divider (Seedburo Equipment Co., Chicago, USA). Each sample was subsequently divided into eight, two kilogram samples; individual samples to be tested on each of the HLM devices. Ten consecutive repetitions were executed on each HLM device using the respective individual samples. However, after the first test only the amount of wheat that was needed to do the test was used for the following nine repetitions. The other two samples were analysed similarly.

Experiment 3a: Variation within and between the HLM devices using mixed wheat samples (Fig. 4)

Ten wheat samples (Table 1; 48 kg each of samples 1-5, 7-8 & 10-12), with a difference of 7.04 $kg.hL^{-1}$ between the highest and lowest sample, were used. The samples were cleaned with a Carter Day Dockage Tester to rid them of impurities and each wheat sample was subsequently poured three times through a Boerner Divider in order to obtain a well-mixed sample. Each 48 kg sample was divided into eight sub-samples of six kilograms each (individual samples to be tested on each device). These sub-samples were further divided into three, two kilogram sub-sub samples. Each of the three sub-sub samples was tested in duplicate on each HLM device (the order of samples and devices was randomly selected).

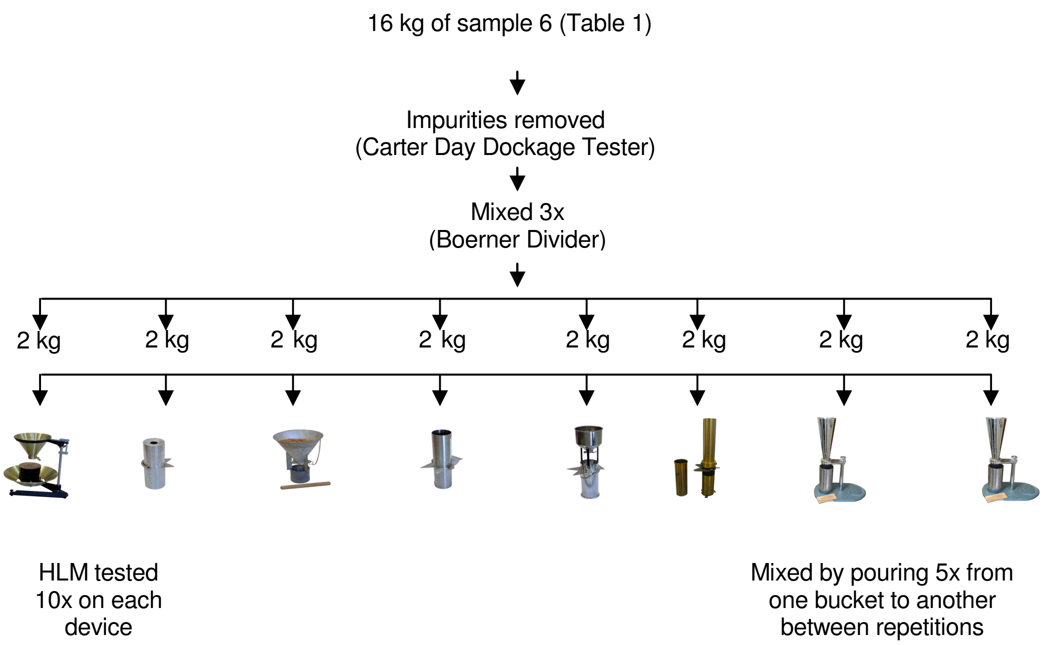

Figure 2 Schematic layout of Experiment 1: repeatability within the respective hectolitre mass (HLM) devices.

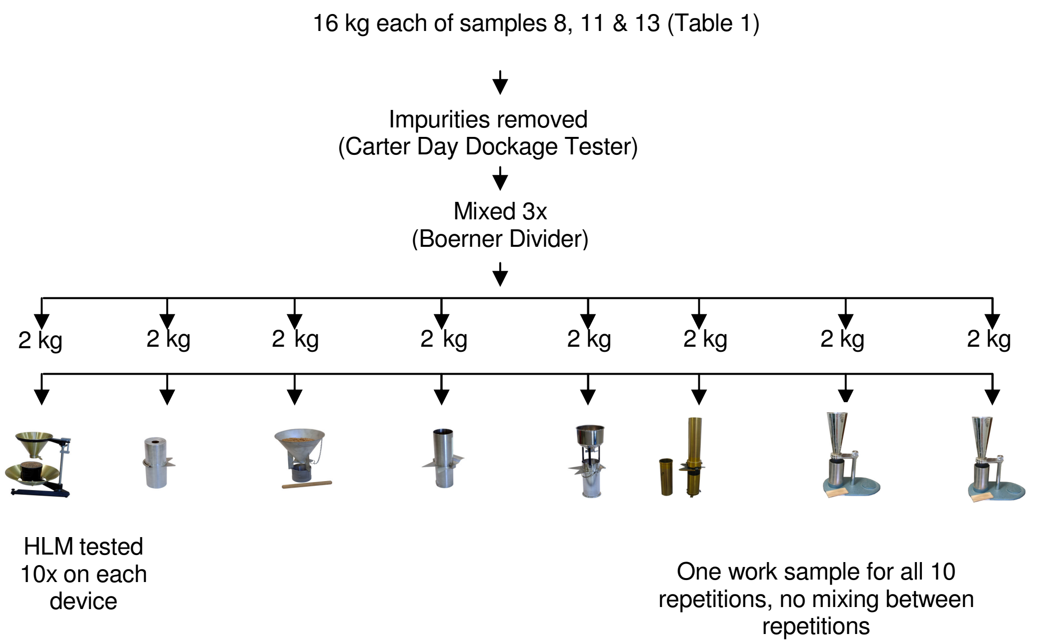

Figure 3 Schematic layout of Experiment 2: effect of repeated analysis of the same wheat sample on its hectolitre mass (HLM).

After each test was done the work sample was mixed back with the rest of the sample (poured from one bucket to another 5 times), where after the second test (duplicate) was performed. By completion of the experiment each sample resulted in six HLM values (two repetitions of three samples) for each of the HLM devices. All of the samples were analysed in a similar way.

To confirm the efficiency of the mixing and dividing of the samples, the moisture and protein contents were determined in duplicate for each of the sub-sub-samples (2 kg sample), respectively. A 50 g sample was used to analyse the moisture and protein contents of each of the 2 kg samples. The samples for testing were obtained by reducing each 2 kg with the Boerner Divider. One half of the sample was continuously poured through the Boerner Divider to obtain the 50 g work sample. The work sample was only removed from the sub-sub samples after the HLM determinations were performed.

Moisture determination

Moisture determinations were performed according to an adapted version of the AACC 45-15A method (AACC, 2004). The 50 g work sample was ground using a laboratory hammer mill (Model 3100, Perten, Huddinge, Sweden). The ground sample was transferred to a sample container, mixed thoroughly with a spatula and covered with an airtight seal. The weight of the moisture dish with lid was recorded (accurate to at least 0.001 g). After the balance was tared 5 g of ground wheat meal was weighed into the moisture dish and the exact weight recorded (accurate to 0.001 g). Subsequently, the uncovered dish (lid beneath the dish) was placed into an air oven (Model EM10, Chopin, Villeneuve-la-Garenne Cedex, France) at 130˚C and left to dry for 60 minutes. The dish was removed from the oven, covered and placed into a desiccator to cool. The mass of the covered dish was determined after 45 minutes and the weight recorded (accurate to 0.001 g). The following equation was used to determine the loss in weight as moisture:

$$A/B \times 100$$

where A = (weight of the moisture dish with original sample) – (weight of the moisture dish with dried sample) and B = (weight of the moisture dish with original sample) – (weight of empty moisture dish).

Protein determination

The protein content (as is) was analysed using the Dumas combustion method with a nitrogen analyser (Model TruSpec® N Elemental Determinator, Leco, St. Joseph, Michigan). The special tin foil sample cup was placed onto the balance and was tared. A small amount (0.05 g, accurate to 0.001 g) of the EDTA standard (Carbon = 40.99%; Nitrogen = 9.57%; Hydrogen = 5.56%) (supplied by Leco Africa, Kempton Park, South Africa) was weighed into the cup, where after it was twisted into an enclosed capsule (hands must be as dry as possible). The capsule was placed into the carousel loading head of the device and the rest of the test was performed as described in the TruSpec® User Manual (Anon., 2004). The same procedure was followed for the wheat meal sample except that 0.35 g of sample was weighed into the tin foil (accurate to 0.001 g). The instrument determines the total amount of nitrogen in a whole wheat flour sample and the factor of 5.7 (AACC method 46-30) (AACC, 2004) was used to convert the nitrogen to protein and it was expressed on a 12% moisture basis.

<u>Experiment 3b: Variation within and between the HLM devices using South African wheat cultivars (Fig. 4)</u>

Experiment 3a was repeated but this time using single South African wheat cultivars (Table 1; 10 x 48 kg of each of samples 14-21, 24 & 26) with a HLM range of 9.91 $kg.hL^{-1}$.

<u>Experiment 4a: Comparison of the HLM devices using a single work sample of mixed wheat samples (Fig. 5)</u>

Ten samples of wheat (Table 1; 6 kg each of samples 1-5, 7-8 & 10-12) with a difference of 7.04 $kg.hL^{-1}$ between the highest and lowest sample were obtained. The samples were cleaned from impurities with a Carter Day Dockage Tester. After the impurities were removed, the respective samples were poured consecutively three times through a Boerner Divider in order to obtain well-mixed samples. Each 6 kg sample was divided into three, 2 kg sub-samples to be tested on the respective devices. The experiment started with HLM determinations on the American device, as this device requires more wheat to perform the measurement than the other devices, whilst the testing order of the other devices was randomly chosen. The work sample obtained from the American device was further used to test the HLM of the other devices. Duplicate measurements were performed on each HLM device using the same initial work sample. Once the first round of tests was performed on all of the devices, the order of the devices was again randomly changed, and the second sequence of tests was carried out. All samples were tested similarly.

<u>Experiment 4b: Comparison of the HLM devices using a single work sample of South African wheat cultivars (Fig. 5)</u>

Experiment 4a was repeated but this time using single South African wheat cultivars (10 x 6 kg each of samples 14-21, 24 & 26; Table 1) with a difference of 9.91 $kg.hL^{-1}$ between the highest and lowest sample.

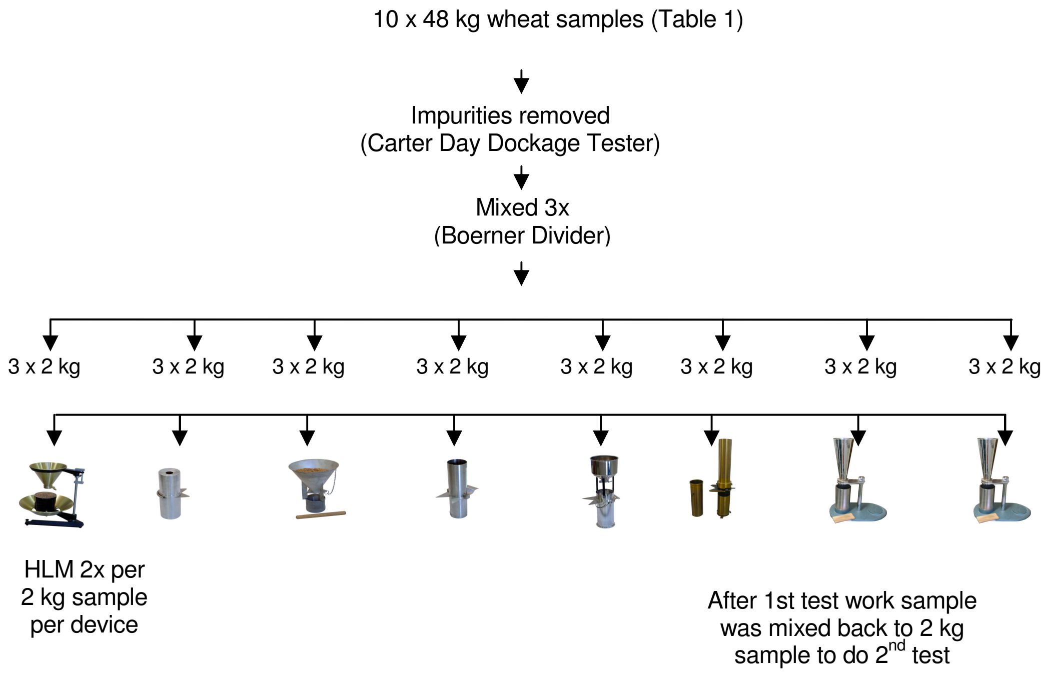

Figure 4 Schematic layout of Experiments 3a and 3b: determination of variation in hectolitre mass (HLM) within and between HLM devices.

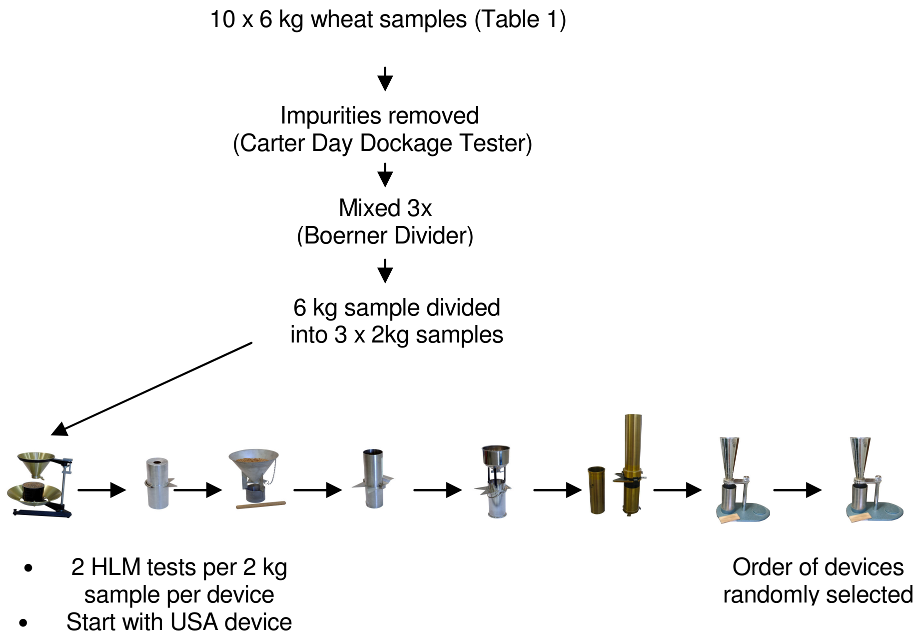

Figure 5 Schematic layout of Experiments 4a and 4b: comparison of hectolitre mass (HLM) devices using a single work sample.

Experiment 5: Comparison of ten South African HLM devices (Fig. 6)

Ten different wheat samples (Table 1, 2.5 kg each of samples 28-37), with a range of 5.73 $kg.hL^{-1}$, were selected from the Western Cape region to evaluate the repeatability of ten respective South African HLM devices. The respective samples were cleaned from impurities using a Carter Day Dockage Tester, where after it was thoroughly mixed two times with a Boerner Divider. Ten respective flour-mills and silo depots made their HLM devices available for execution of the HLM determinations. Each, of the ten samples, was tested in duplicate on each of the ten devices. The work sample obtained after doing the first test, on the first device, was used for the second test (duplicate). This work sample was subsequently kept for measurement of the HLM on the other devices as well. The remaining samples were tested similarly.

Experiment 6: Effect of impurities on HLM determinations (Fig. 7)

Ten different wheat samples (Table 1; 2.5 kg each of samples 28-37), with a range of 5.73 $kg.hL^{-1}$, were obtained. The respective samples were not cleaned from impurities and were thoroughly mixed with a Boerner Divider (twice). The first HLM determination was performed on the USA device, as this device needs the largest wheat sample compared to the other devices. The order of the other devices was randomly selected. The work sample obtained from the USA device was subsequently used to perform the HLM measurements on the other devices as well. Duplicate measurements of the same work sample were executed on each HLM device, however, after the first repetition the order of the devices was randomly changed again to complete the second test. The rest of the samples were tested similarly.

After completion of the first series of duplicate measurements, the impurities were removed from the 2.5 kg samples using a Carter Day Dockage Tester. The samples were mixed with a Boerner Divider (twice) and the HLM measurements conducted as described earlier.

Experiment 7: Effect of operator on HLM determinations (Fig. 8)

Ten respective wheat samples (Table 1; 2.5 kg each of samples 28-37), with a range of 5.73 $kg.hL^{-1}$, were obtained. The respective samples were cleaned from impurities using a Carter Day Dockage Tester, where after it was thoroughly mixed twice with a Boerner Divider. Three different operators, with three levels of competency (skilled, semi-skilled and unskilled), conducted HLM measurements using the same sample of wheat. The tests were conducted on two South African HLM devices and the work sample obtained from doing the first measurement with each sample by the first operator was used for testing the other device and was kept for the other operators to perform the HLM measurements. Each operator performed ten repetitions on each sample, but for each repetition the samples were randomly selected.

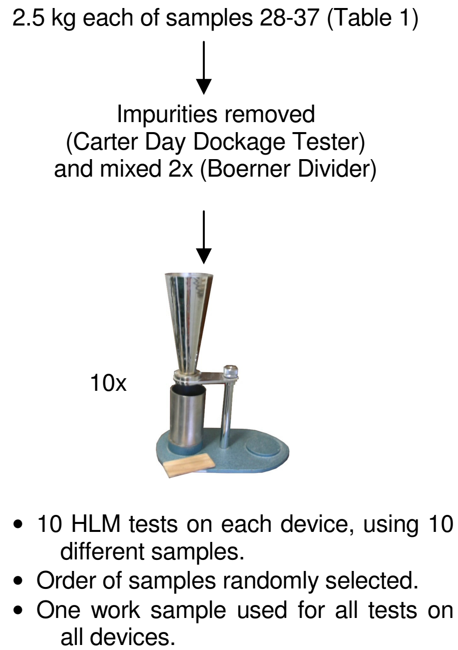

Figure 6 Schematic layout of Experiment 5: comparison of ten respective South African hectolitre mass (HLM) devices.

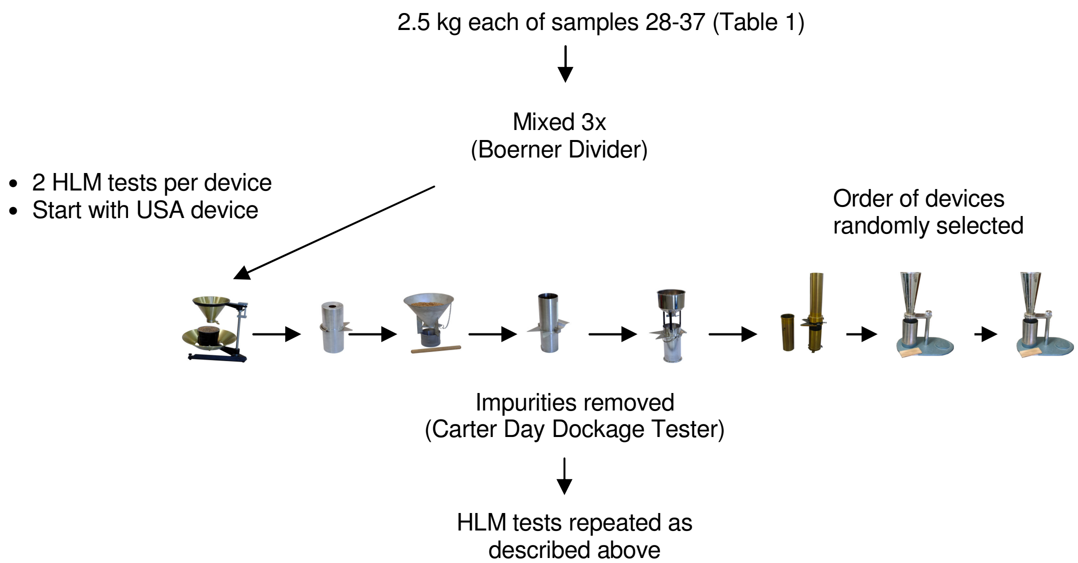

Figure 7 Schematic layout of Experiment 6: effect of impurities on hectolitre mass (HLM) determinations.

Experiment 8: Effect of wet-dry cycles on HLM determinations (Fig. 9)

Four samples (Table 1; 8 kg each of samples 5, 8, 13 & 17), with a range of 5.32 kg.hL^{-1}, were selected. The four respective bulk samples were divided into 4 sub-samples of 2 kg each. For each of the four samples, three of its four sub-samples were conditioned to moisture contents of *ca.* 14%, 16% and 18%, respectively, whilst the remaining sample was kept at its original moisture content. The samples were conditioned by adding the appropriate amount of water to the samples in order to obtain each of the respective moisture contents. The samples were mixed on a rotary mixer for 20 minutes and left to equilibrate for a further 24 hours. After 24 hours the samples were mixed again for 5 minutes on the rotary mixer and kept at 4˚C for another 24 hours. The samples were removed from cold storage and were allowed to equilibrate to room temperature for two hours after which the HLM determinations were immediately performed.

The HLM of all 16 sub-samples were measured in duplicate on each of the eight different devices. The first measurement was again performed on the USA device and the work sample obtained was used for measuring the HLM of the samples on the other devices. Subsequently all 16 sub-samples were dried in a forced circulation oven (Model FSOE8, Labcon Pty. Ltd., Roodepoort, South Africa) at 35˚C to a moisture content of *ca.* 10%. The moisture contents of the dried samples were confirmed according to the one-hour oven method as described earlier. The HLM values were determined as described above after which the samples were conditioned again to their moisture contents before drying, i.e. original, 14%, 16% and 18% respectively. The moisture content and the HLM of each of the 16 sub-samples were measured again. The moisture contents of the samples after the respective moisture treatments are displayed in Table 4.

Statistical analysis

Statistical analyses were performed and graphs compiled using Statistica version 7.1 (StatSoft, Inc., Tulsa, OK, USA). Repeated measures analysis of variance (RANOVA) was performed to compare average measurements between instruments to determine absolute differences. The bar around the average represents the 95% confidence interval for the average measurements. Bonferroni and Fisher least significant difference (LSD) *post-hoc* testing was used. All references to significant differences indicate statistical differences. In summary tables the standard errors were reported with the means. The standard error (se) is related to the standard deviation (sd) in the following way: $\mathrm{se} = \mathrm{sd}/\sqrt{n}$, where n is the sample size.

Additionally the intra-class correlation (ICC) coefficients were determined as the ICC agreement that correlates measurements with each other, while taking into account the differences in absolute values of the respective measurements, and the ICC consistency that only correlates measurements. All ICC calculations were done using the R statistical programming language.

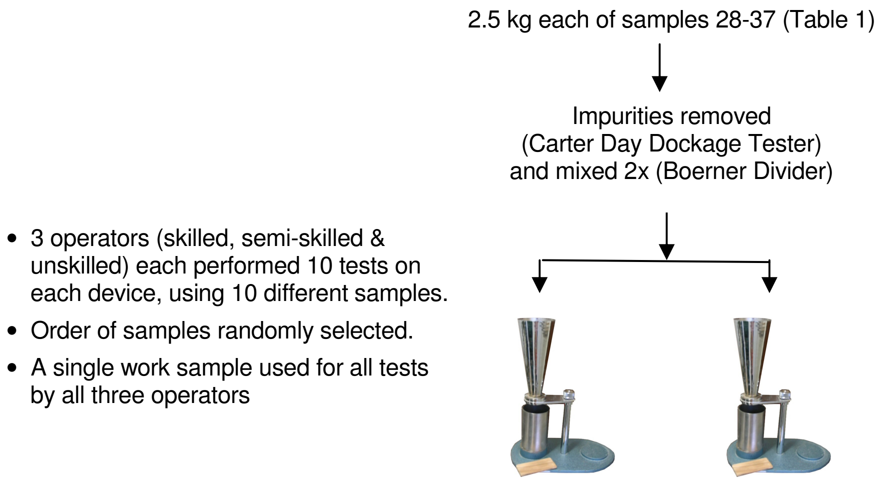

Figure 8 Schematic layout of Experiment 7: effect of operator on hectolitre mass (HLM) measurements.

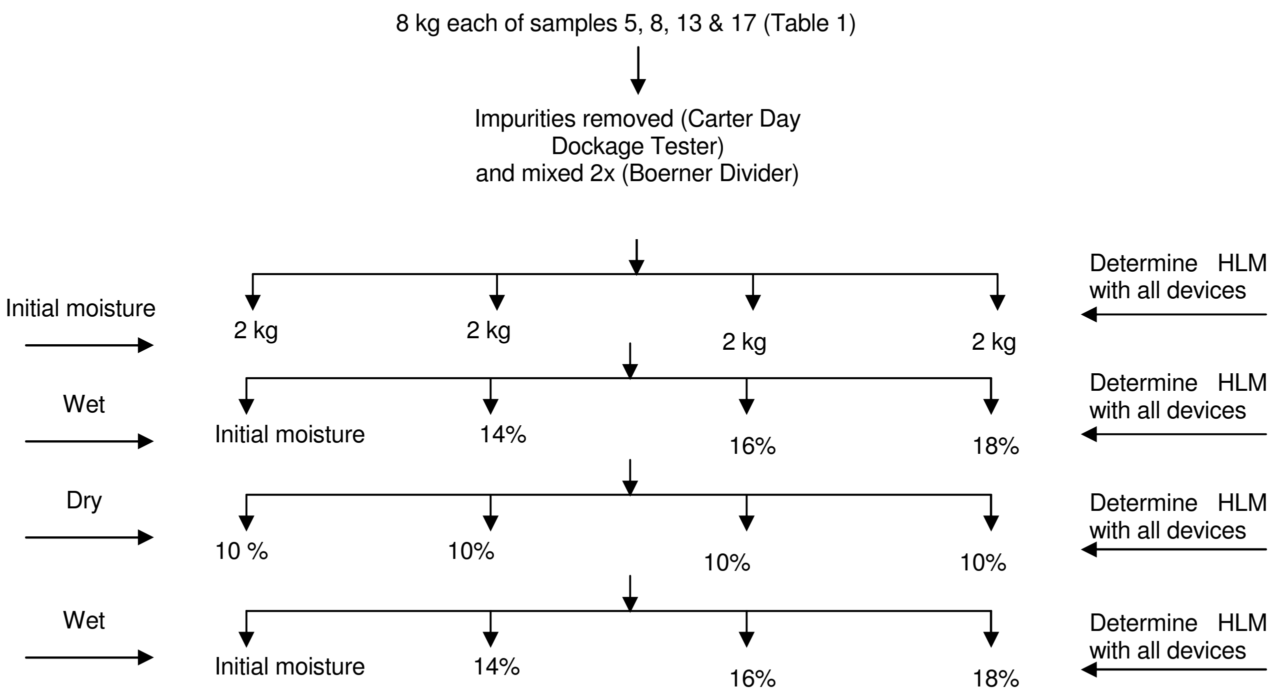

Figure 9 Schematic layout of Experiment 8: effect of wet and dry cycles on hectolitre mass (HLM) values.

Table 4 Moisture changes of wheat samples after respective moisture treatments

Moisture treatments	Changes in moisture content (%)			
	Sample 5	**Sample 8**	**Sample 17**	**Sample 13**
Initial moisture	11.2	12.1	11.8	11.3
Tempered to *ca*. 14%	14.1	14.7	13.8	14.1
Tempered to *ca*. 16%	15.7	16.1	15.3	15.7
Tempered to *ca*. 18%	16.7	17.7	16.7	17.0
Drying initial to *ca*. 10%	9.8	10.6	10.4	10.2
Drying 14% to *ca*. 10%	10.2	9.5	9.4	9.9
Drying 16% to *ca*. 10%	9.9	10.8	10.6	10.8
Drying 18% to *ca*. 10%	10.8	9.9	9.5	9.8
Tempered back to initial moisture	11.2	11.7	11.6	11.0
Tempered back to *ca*. 14%	_	14.5	13.6	13.7
Tempered back to *ca*. 16%	_	15.7	16.1	15.2
Tempered back to *ca*. 18%	_	16.9	17.3	17.0

Results

Experiment 1: Repeatability within the HLM devices

(Detailed results in Appendix 7, Table 7.1)

The HLM measurements within each HLM device were highly repeatable with the least repeatable results obtained with the Australian device having the highest standard deviation of 0.57 (Table 5). The devices from France and USA showed the highest degree of repeatability (lowest standard deviation).

Experiment 2: Effect of repeated analysis of the same wheat sample on its HLM value

(Detailed results in Appendix 7, Table 7.2)

The HLM values of the ten repetitions performed with sample 11 using the Australian HLM device decreased as the repetitions increased (Fig. 10). Figure 11 compares the averages of the first and last four repetitions, respectively, and shows a significant difference ($P < 0.05$). This phenomenon was, however, not observed for the other two samples also analysed on the Australian device.

On the other hand a significant ($P < 0.05$) increase in HLM values was noted with increasing repetitions when HLM measurements were performed with the second South African device using sample 13 (Fig. 12). Figure 13 compares the averages of the first and last four repetitions, respectively, and shows a significant difference ($P < 0.05$). Again HLM results obtained for the other two samples, also analysed on the second South African device, did not show this observed trend. Neither was this trend observed for the first South African device.

Table 5 Hectolitre mass (HLM) values (mean ± standard deviation (sd)) displaying the repeatability within the HLM devices

HLM device	Mean ± sd	*n*
Australia	81.95 ± 0.570	10
Canada	81.44 ± 0.207	10
France	80.42 ± 0.115	10
Germany	80.67 ± 0.148	10
SA 1	79.29 ± 0.164	10
SA 2	78.69 ± 0.292	10
UK	80.84 ± 0.280	10
USA	80.21 ± 0.119	10

Experiment 3a: Variation within and between the HLM devices using mixed wheat samples (Detailed results in Appendix 7, Tables 7.3.1 and 7.3.1.1)

The intra-class correlation (ICC) agreement was determined to express variation in HLM measurements within the respective HLM devices in terms of actual values. The ICC agreement (Fig. 14) revealed little variation within the devices with all the ICC agreement values higher than 0.94, indicating the devices to produce highly repeatable results. The ICC consistency also showed that the HLM tests done with the respective sub-sub-samples, within the devices, are correlated (ICC consistency >0.94). The fact that little variation existed within the devices proves that sample variation between the sub-sub samples was also low. Additionally the results of moisture and protein determinations performed on the sub-sub-samples verified efficient mixing, dividing and reduction of the sub-sub-samples as no significant differences have been found between the respective moisture and protein contents of the sub-samples (Appendix 7, Table 7.3.1.1). Therefore, it can be said with confidence that the mixing and dividing of the bulk samples have been done in such a way to produce well mixed sub-samples divided in such a way as to be efficiently representative of the original sample.

The RANOVA results (Fig. 15) show that the average HLM measurements obtained on the German, Canadian, French, USA and British devices were not significantly different ($P > 0.05$). The two South African devices gave similar average HLM values ($P > 0.05$) to each other and the Australian device resulted in average HLM measurements that were significantly different ($P < 0.05$) to all the other devices. It is clear from Fig. 15 that the South African devices resulted in significantly lower and the Australian device in significantly higher HLM measurements in comparison to the other devices.

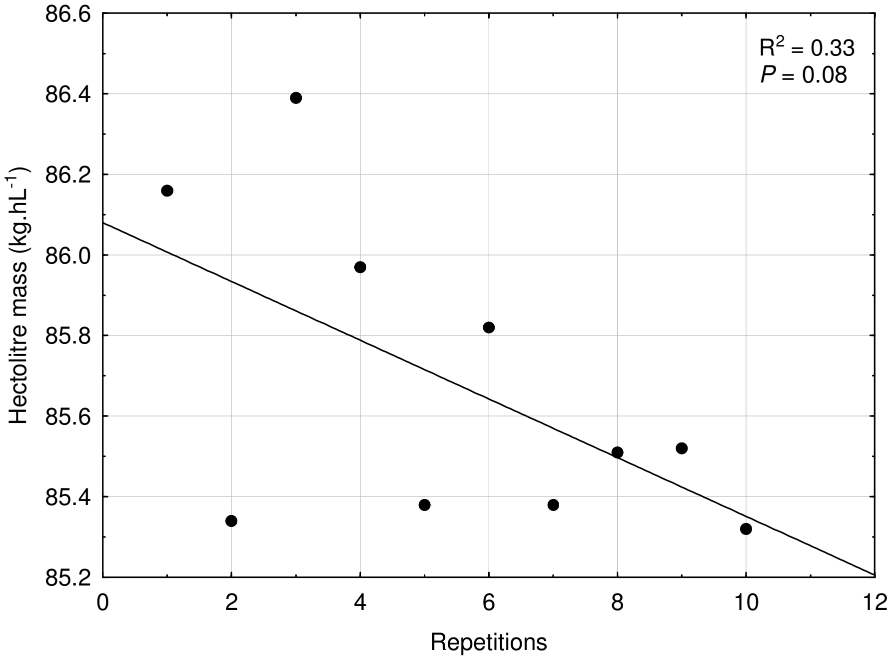

Figure 10 Regression scatter plot showing the decrease in the ten successive repetitions of measurements obtained with the Australian device using sample 11.

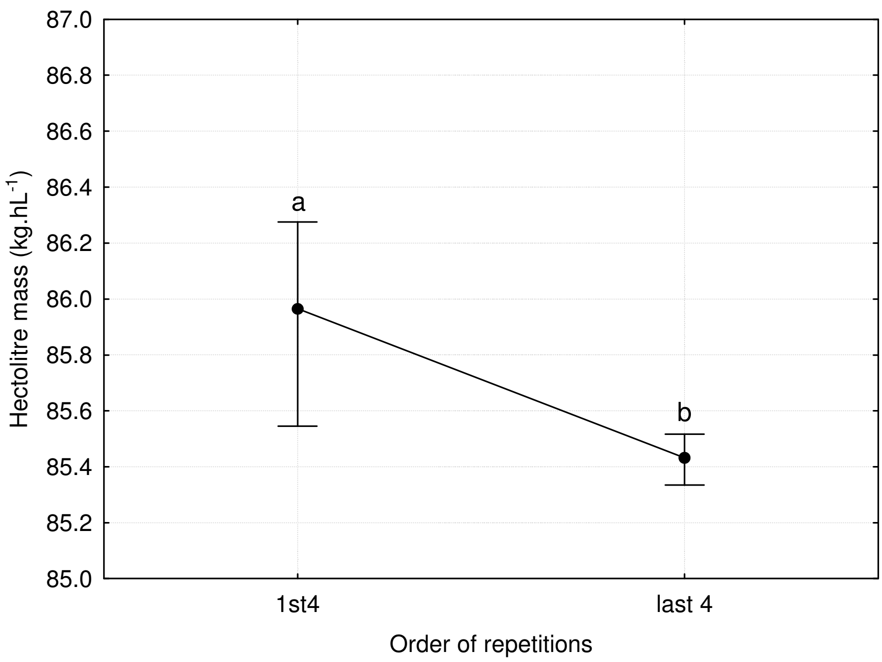

Figure 11 The averages of the first and last four repetitions of measurements obtained with the Australian device using sample 11.

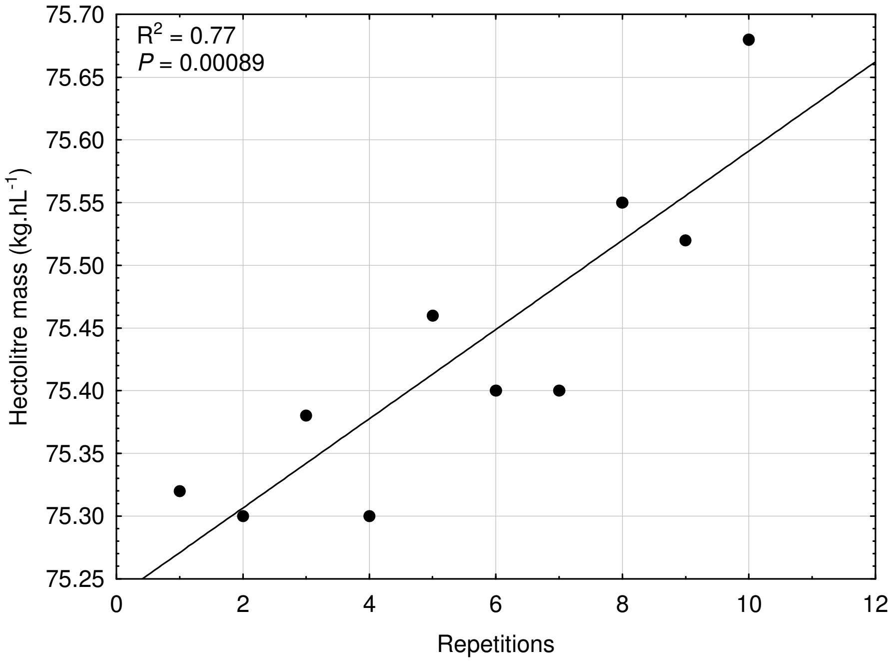

Figure 12 Regression scatter plot of the ten successive repetitions of measurements obtained with the second South African device using sample 13.

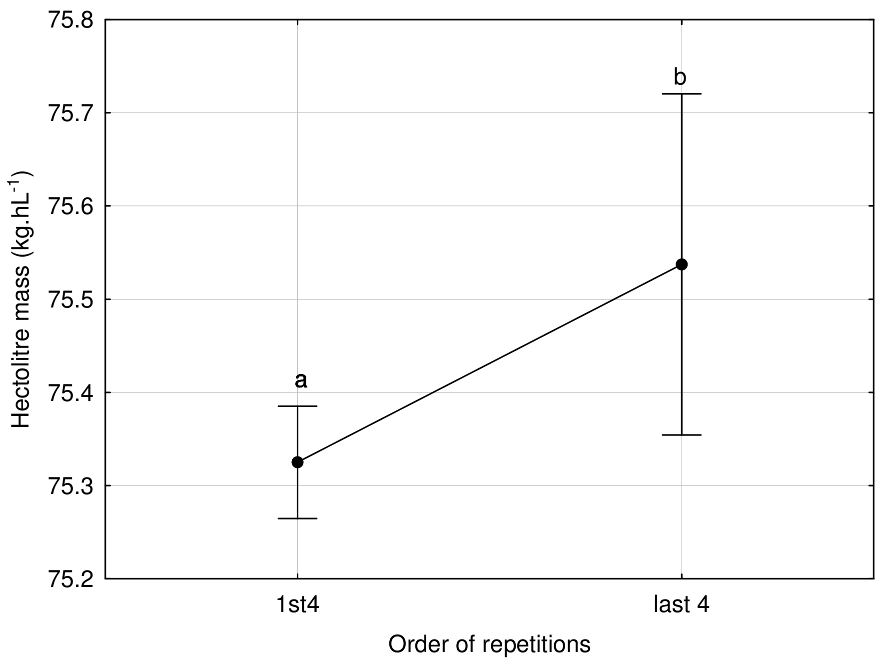

Figure 13 The average values of the first and last four repetitions of measurements obtained with the second South African device using sample 13.

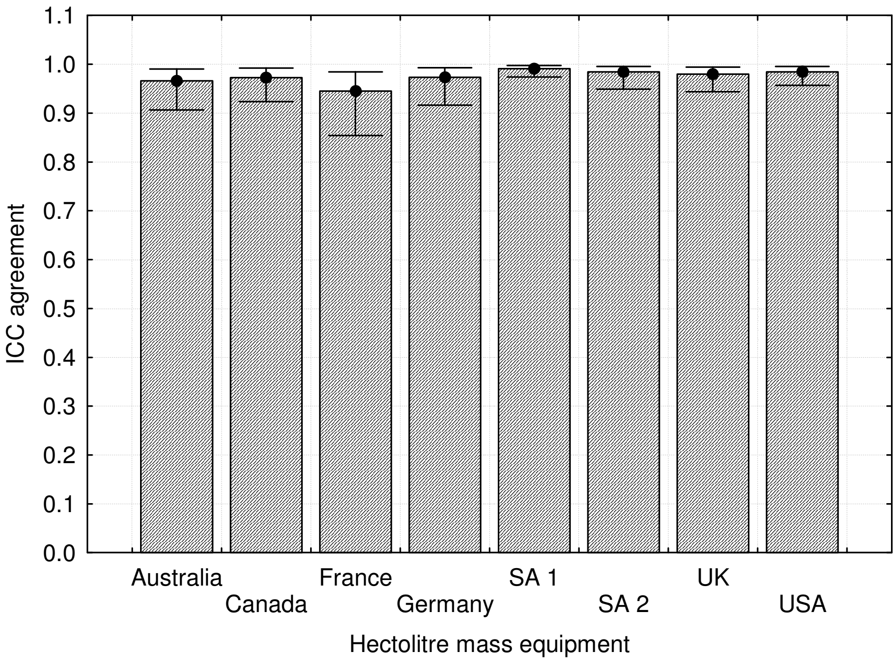

Figure 14 Intra-class correlation (ICC) agreement showing the variation in terms of actual hectolitre mass (HLM) values within HLM devices as determined with mixed wheat samples. Error bars denote 0.95 confidence intervals.

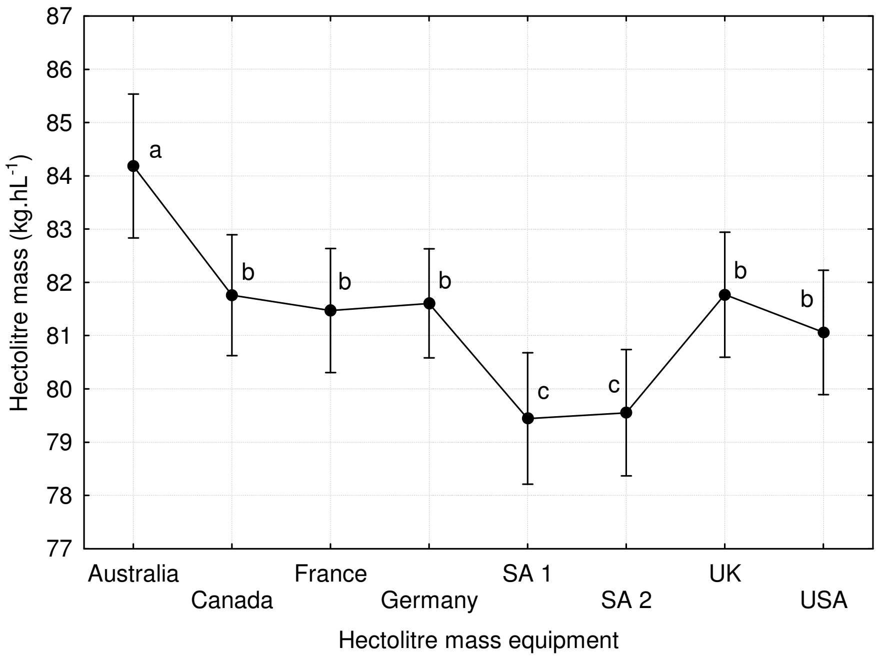

Figure 15 Differences between the average hectolitre mass (HLM) values obtained with the HLM devices using mixed wheat samples as determined with repeated analysis of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Bonferroni *post-hoc* analyses.

The ICC agreement was also determined to express the variation between the respective devices in terms of actual HLM values. An overall ICC agreement of 0.523 was observed between the devices, indicating that the average HLM measurements of the devices did not agree well with each other. The average measurements of the devices from France, USA, Canada, UK and Germany gave similar results, the two South African devices agreed with each other and the Australian device agreed the least with any of the other devices (Fig. 16). The highest ICC agreement value (0.69) was observed between the USA and the two South African devices. In spite of the low ICC agreement, it indicates that in terms of actual HLM measurements the USA device resulted in most similar results to the two South African devices compared to any of the other devices (Table 6). The second device most similar to the South African HLM devices in terms of actual measurements was the French device (0.58) and the lowest agreement was obtained with the Australian device (0.20). The ICC consistency (Fig. 17), however, shows that in spite of the HLM devices differing from one another in terms of the actual HLM values, the devices did correlate well with each other with only the device from Australia having an ICC consistency value of less than 0.9.

Experiment 3b: Variation within and between the HLM devices using South African wheat cultivars (Detailed results in Appendix 7, Table 7.3.2)

Similar results were obtained with the single wheat cultivars as with the mixed samples. The Boerner Divider was efficient in providing well mixed and representatively divided sub-samples as indicated by the within device ICC agreement (Fig. 18). Highly correlated HLM values were obtained when the HLM values of each sub-sample, within each device, was correlated with the use of ICC consistency (ICC consistency >0.96). It can also be deduced that due to working with single wheat cultivars very little sample variation was present that could have influenced the repeatability of the respective devices. The HLM results obtained from the devices proved to be more repeatable, within each device, when using single wheat cultivars compared to the mixed wheat samples.

Results obtained with RANOVA (Fig. 19) showed that the average HLM measurements of the Australian device were significantly higher ($P < 0.05$) and those of the South African devices significantly lower ($P < 0.05$) compared to HLM measurements obtained with the other devices. The between device ICC agreement (Fig. 20) shows that the devices agreed better, in terms of actual values, with each other when HLM was determined with single wheat cultivars. The overall ICC agreement improved from 0.524 with the mixed wheat samples (Table 6) to 0.768 with the single wheat cultivars (Table 7). As before the highest average ICC agreement (0.882) was found between the two South African devices and the USA device (Table 7). The agreement in terms of actual values between all the devices is displayed in Table 7.

The ICC consistency (Fig. 21) shows an improved correlation between the devices when the HLM measurements were performed using single South African wheat cultivars.

Table 6 Intra-class correlation (ICC) agreement showing the agreement, in terms of actual values, between the hectolitre mass (HLM) devices using mixed wheat samples

HLM equipment		ICC agreement	ICC consistency
Australia	Canada	0.438	0.855
Australia	France	0.405	0.879
Australia	Germany	0.390	0.847
Australia	SA 1	0.203	0.901
Australia	SA 2	0.200	0.877
Australia	UK	0.458	0.880
Australia	USA	0.327	0.834
Canada	France	0.969	0.983
Canada	Germany	0.969	0.972
Canada	SA 1	0.496	0.980
Canada	SA 2	0.509	0.979
Canada	UK	0.953	0.948
Canada	USA	0.889	0.971
France	SA 1	0.567	0.981
France	SA 2	0.588	0.988
France	UK	0.956	0.969
France	USA	0.950	0.979
Germany	France	0.958	0.958
Germany	SA 1	0.494	0.952
Germany	SA 2	0.516	0.968
Germany	UK	0.942	0.942
Germany	USA	0.885	0.935
SA 1	SA 2	0.990	0.991
SA 1	UK	0.500	0.977
SA 1	USA	0.675	0.985
SA 2	UK	0.516	0.981
SA 2	USA	0.693	0.982
UK	USA	0.896	0.978
Overall		0.524	0.943

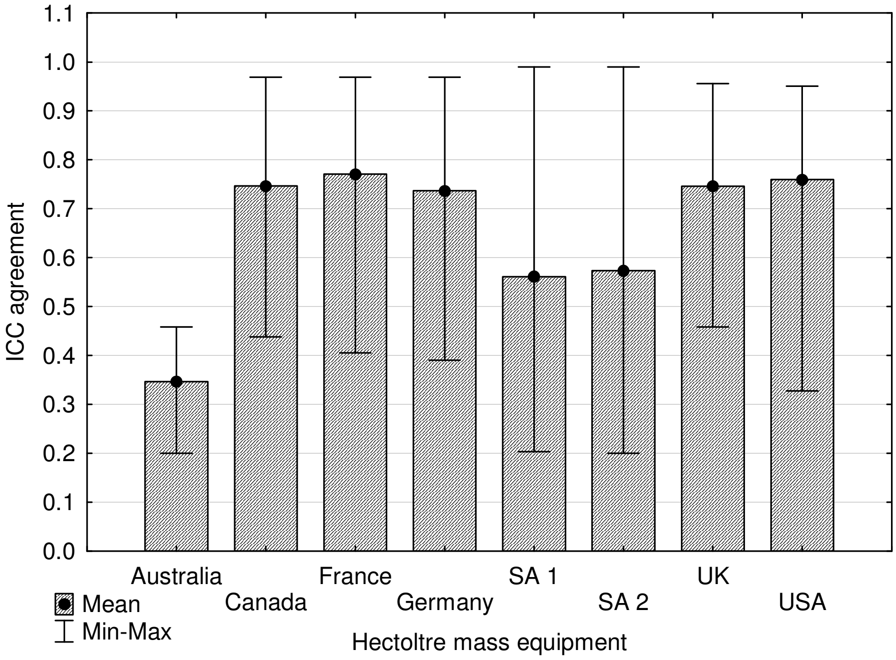

Figure 16 Intra-class correlation (ICC) agreement (averaged as calculated from Table 6) showing the variation in terms of actual hectolitre mass (HLM) values between HLM devices as determined using mixed wheat samples.

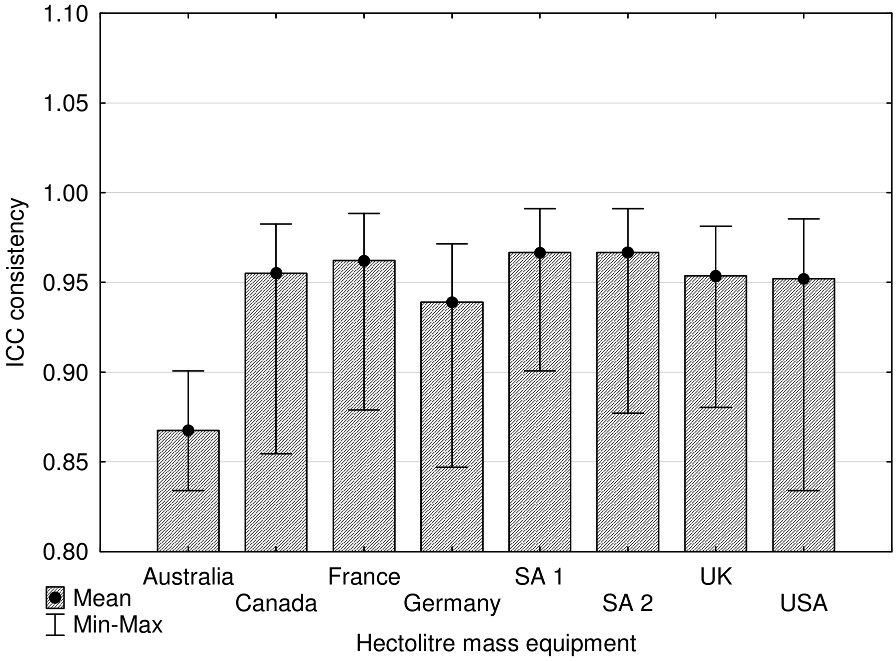

Figure 17 Intra-class correlation (ICC) consistency (averaged as calculated from Table 6) of the hectolitre mass (HLM) values between HLM devices as determined using mixed wheat samples.

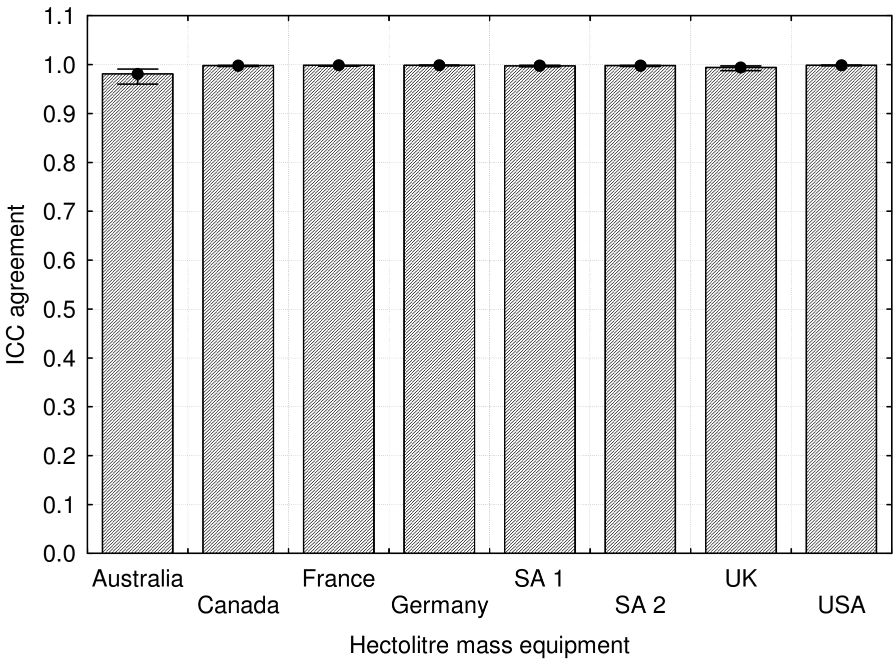

Figure 18 Intra-class correlation (ICC) agreement showing the variation in terms of actual hectolitre mass (HLM) values within HLM devices as determined using South African wheat cultivars. Error bars indicate 0.95 confidence intervals.

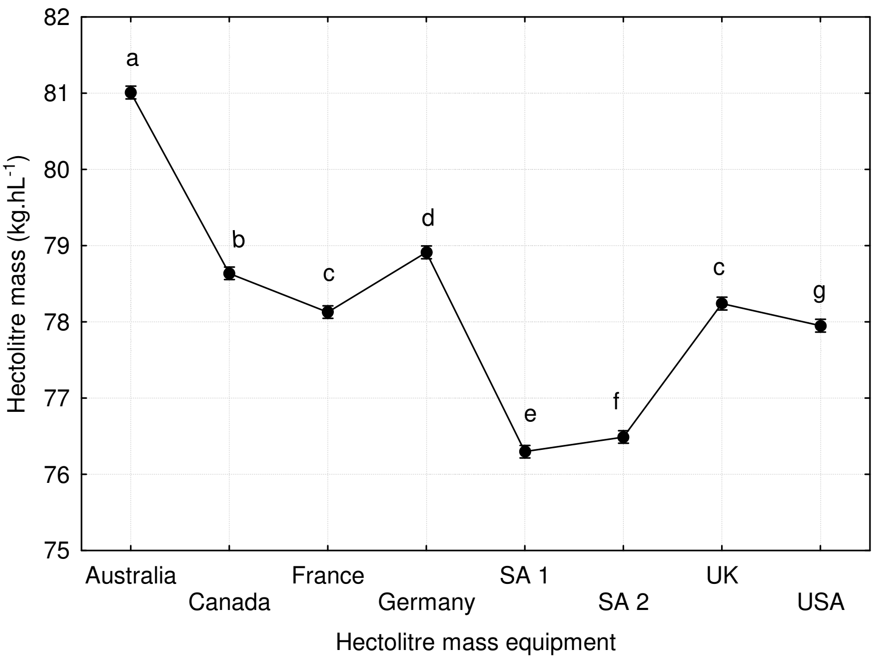

Figure 19 Differences between the average hectolitre mass (HLM) values obtained with the HLM devices using South African wheat cultivars as determined with repeated analysis of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Fisher least significance difference (LSD) *post-hoc* analyses.

Table 7 Intra-class correlation (ICC) agreement showing the agreement, in terms of actual values, between the hectolitre mass (HLM) devices using South African wheat cultivars

HLM equipment		ICC agreement	ICC consistency
Australia	Canada	0.745	0.983
Australia	France	0.667	0.985
Australia	Germany	0.766	0.983
Australia	SA 1	0.442	0.975
Australia	SA 2	0.462	0.986
Australia	UK	0.658	0.983
Australia	USA	0.637	0.982
Canada	France	0.980	0.994
Canada	Germany	0.973	0.977
Canada	SA 1	0.778	0.995
Canada	SA 2	0.800	0.992
Canada	UK	0.972	0.981
Canada	USA	0.969	0.993
France	Germany	0.940	0.974
France	SA 1	0.849	0.996
France	SA 2	0.871	0.994
France	UK	0.986	0.986
France	USA	0.996	0.998
Germany	SA 1	0.692	0.962
Germany	SA 2	0.725	0.973
Germany	UK	0.958	0.988
Germany	USA	0.921	0.972
SA 1	SA 2	0.991	0.993
SA 1	UK	0.802	0.976
SA 1	USA	0.871	0.995
SA 2	UK	0.831	0.980
SA 2	USA	0.893	0.994
UK	USA	0.983	0.988
Overall		0.768	0.986

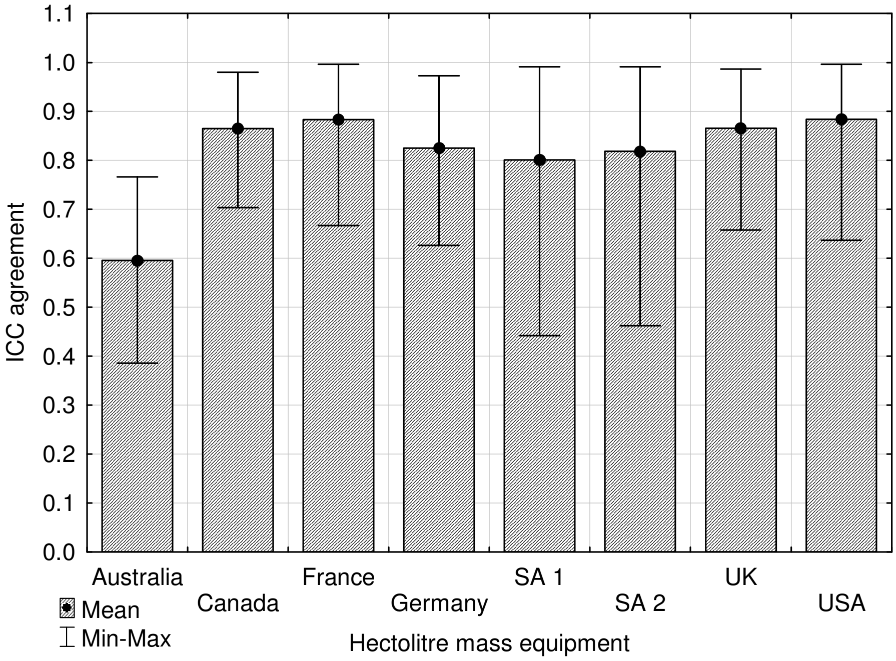

Figure 20 Intra-class correlation (ICC) agreement (average values calculated from Table 7) showing the variation in terms of actual hectolitre mass (HLM) values between HLM devices as determined using South African wheat cultivars.

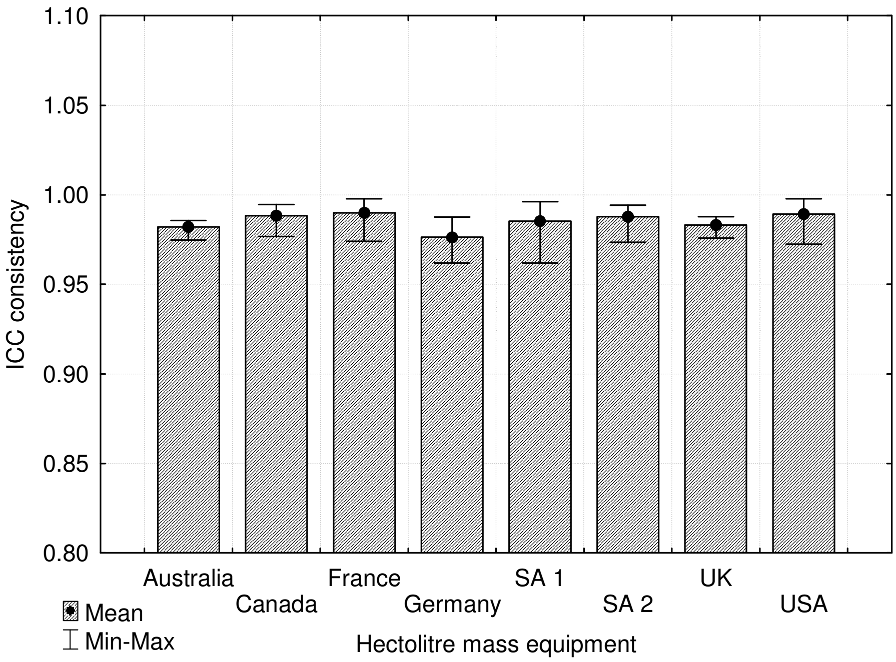

Figure 21 Intra-class correlation (ICC) consistency (average values calculated from Table 7) of the hectolitre mass (HLM) values between HLM devices as determined using South African wheat cultivars.

Experiment 4a: Comparison of different HLM devices using a single work sample of mixed wheat samples

(Detailed results in Appendix 7, Table 7.4.1)

In an attempt to eliminate the possible effect of internal sample variation Experiment 3 was repeated but this time each of the ten bulk samples were each divided into three respective sub-samples from which three work samples were removed and duplicate HLM measurements were performed with each of the three work samples, on all of the devices.

The comparison of the devices with RANOVA (Fig. 22) showed that the average measurements obtained with the two South African devices and the Australian device were significantly ($P < 0.05$) different from the other devices. The South African devices delivered the lowest average measurements, whilst the Australian device delivered the highest.

An overall ICC agreement coefficient of 0.420 was obtained and it was once again observed that the actual average measurements of the two South African devices and the USA device were most similar, with an ICC agreement of 0.613 (Table 8). The agreement in terms of actual HLM values between the respective devices is displayed in Table 8.

The ICC agreement results show (Fig. 23) that the average measurements of all the HLM devices agreed to a lesser extent than observed earlier. The Australian device especially had a very low ICC agreement of 0.245. Despite of the decreased agreement the devices still correlated (Fig. 24) well with each other with ICC consistency values of above 0.9.

Experiment 4b: Comparison of different HLM devices using a single work sample of South African wheat cultivars

(Detailed results in Appendix 7, Table 7.4.2)

The comparison of the HLM obtained from the respective devices, using RANOVA (Fig. 25) showed that the average measurements obtained with the two South African devices and the Australian device were, as observed earlier, significantly different ($P < 0.05$) from the other devices with the South African devices resulting in the lowest average measurements whilst the Australian device resulted in the highest.

An overall ICC agreement coefficient of 0.762 was determined and again it was found that the average measurements of the two South African devices agreed most with the average measurements of the USA device indicated by an average ICC agreement of 0.879 (Table 9). The ICC agreement between the respective devices is shown in Table 9.

Again the actual HLM measurements of all the devices were similar (Fig. 26), except for the Australian device which had the lowest ICC agreement of 0.580. Nevertheless, all of the devices correlated (Fig. 27) well with each other with ICC consistency values of higher than 0.95.

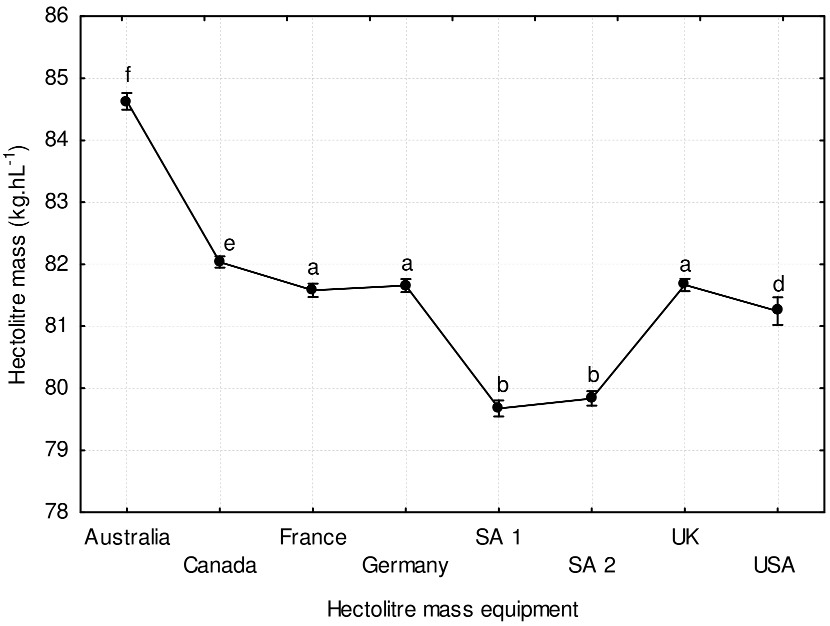

Figure 22 Differences between the average hectolitre mass (HLM) values obtained with the HLM devices using a single work sample of mixed wheat samples as determined with repeated analysis of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Bonferroni *post-hoc* analyses.

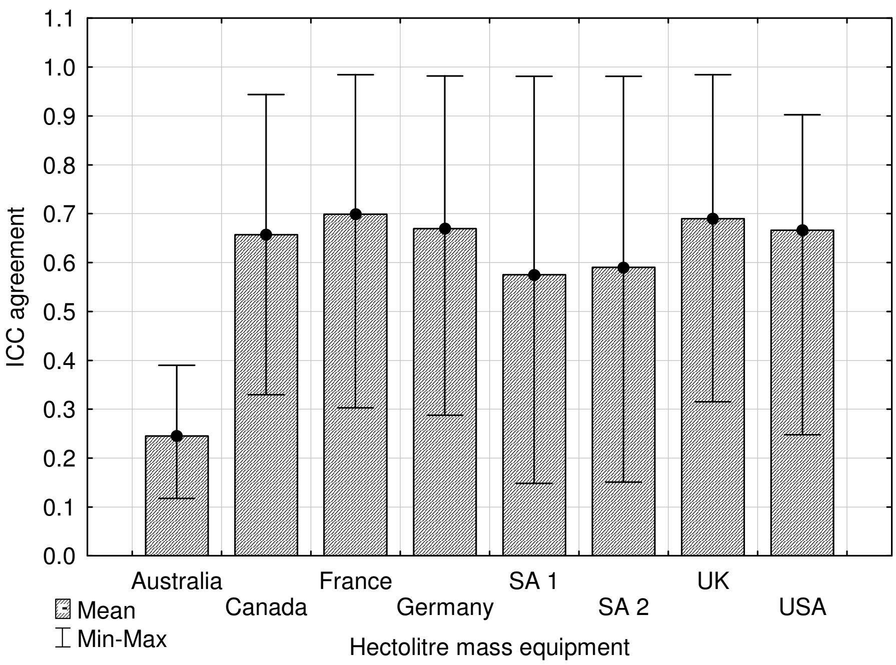

Figure 23 Intra-class correlation (ICC) agreement showing the variation in terms of actual hectolitre mass (HLM) values between HLM devices as determined using a single work sample of mixed wheat samples.

Table 8 Intra-class correlation (ICC) agreement showing the agreement, in terms of actual values, between the hectolitre mass (HLM) devices using a single work sample of mixed wheat samples

HLM equipment		ICC agreement
Australia	Canada	0.390
Australia	France	0.303
Australia	Germany	0.288
Australia	SA 1	0.148
Australia	SA 2	0.151
Australia	UK	0.315
Australia	USA	0.248
Canada	France	0.94
Canada	Germany	0.92
Canada	SA 1	0.451
Canada	SA2	0.476
Canada	UK	0.944
Canada	USA	0.807
France	Germany	0.976
France	SA 1	0.537
France	SA 2	0.565
France	UK	0.985
France	USA	0.902
Germany	SA 1	0.484
Germany	SA 2	0.516
Germany	UK	0.982
Germany	USA	0.845
SA 1	SA 2	0.981
SA 1	UK	0.513
SA 1	USA	0.594
SA 2	USA	0.632
UK	SA 2	0.543
UK	USA	0.865
Overall		0.420

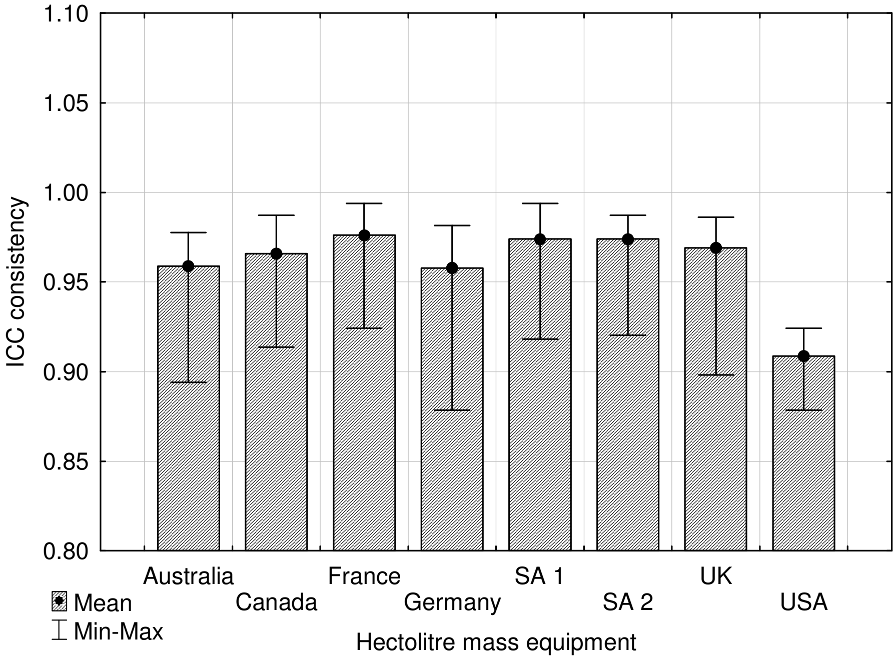

Figure 24 Intra-class correlation (ICC) consistency showing the correlation between the hectolitre mass (HLM) values as determined on the HLM devices using single work samples of mixed wheat.

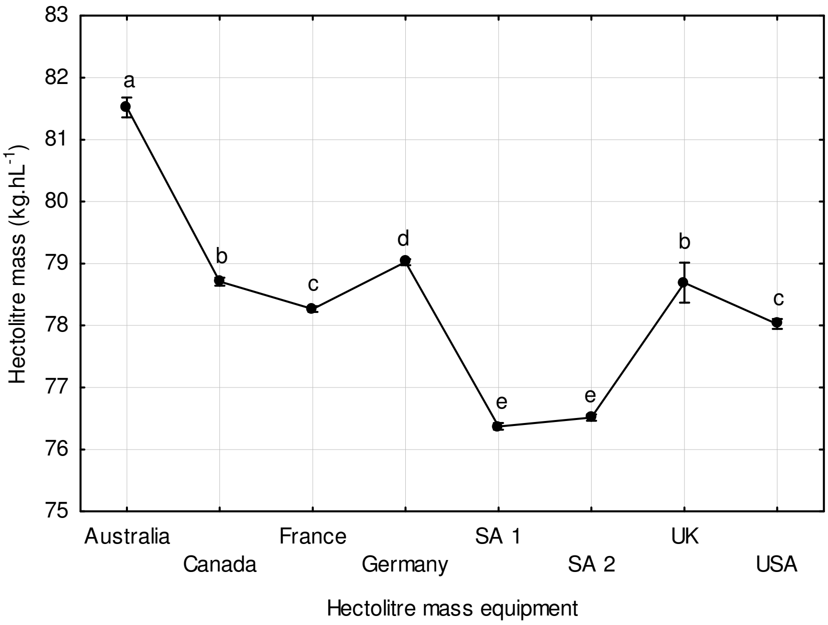

Figure 25 Differences between the average hectolitre mass (HLM) values obtained with the HLM devices using a single sample of South African wheat cultivars as determined with repeated analysis of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Bonferroni *post-hoc* analyses.

Table 9 Intra-class correlation (ICC) agreement showing the agreement, in terms of actual values, between the hectolitre mass (HLM) devices using a single work sample of South African wheat cultivars

HLM equipment		**ICC agreement**
Australia	Canada	0.689
Australia	France	0.627
Australia	Germany	0.693
Australia	SA 1	0.406
Australia	SA 2	0.424
Australia	UK	0.644
Australia	USA	0.581
Canada	France	0.989
Canada	Germany	0.964
Canada	SA 1	0.783
Canada	SA 2	0.807
Canada	UK	0.96
Canada	USA	0.972
France	Germany	0.938
France	SA 1	0.845
France	SA 2	0.866
France	UK	0.949
France	USA	0.993
Germany	SA 1	0.685
Germany	SA 2	0.71
Germany	UK	0.932
Germany	USA	0.915
SA 1	SA 2	0.998
SA 1	UK	0.739
SA 1	USA	0.869
SA 2	UK	0.762
SA 2	USA	0.888
UK	USA	0.938
Overall		0.763

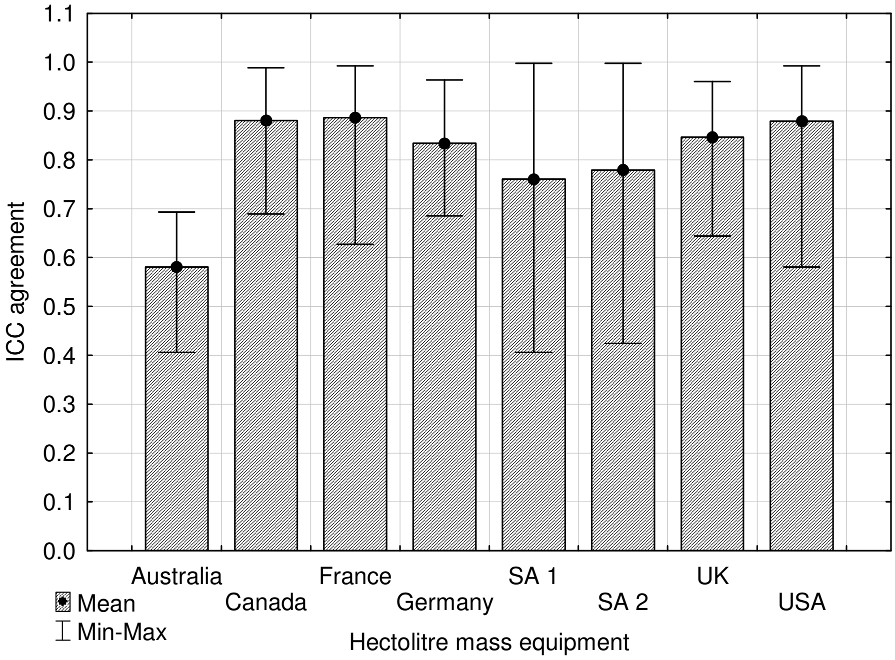

Figure 26 Intra-class correlation (ICC) agreement showing the variation in actual hectolitre mass (HLM) values between the HLM devices as determined using a single work samples of South African wheat cultivars.

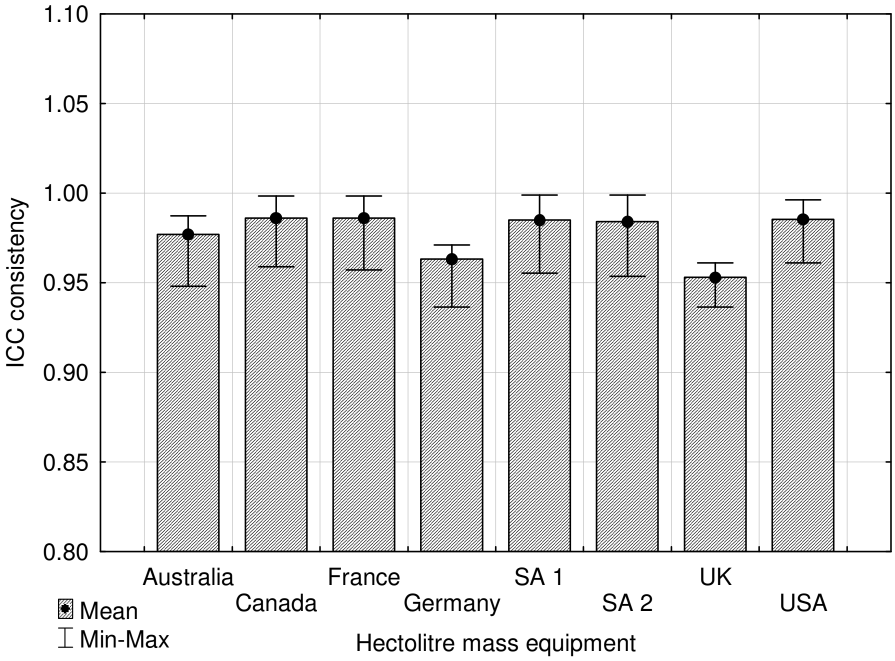

Figure 27 Intra-class correlation (ICC) consistency showing the correlation between the hectolitre mass (HLM) values as determined on the HLM devices using a single work sample of South African wheat cultivars.

Experiment 5: Comparing ten respective South African HLM devices in commercial use

(Detailed results in Appendix 7, Table 7.5)

Although statistical differences ($P < 0.05$) were observed between the HLM results obtained from some of the ten respective South African devices (Fig. 28) the differences were really small as reflected in the overall ICC agreement and consistency coefficients of 0.975 and 0.993, respectively. The average error (SEM) determined between the respective devices was 0.12 $kg.hL^{-1}$ which confirmed that the significant differences observed would not be significant in practice. The descriptive statistics of the experiment are displayed in Table 10.

Experiment 6: Effect of impurities on HLM determination

(Detailed results in Appendix 7, Table 7.6)

The results (Fig. 29) showed that there was a significant increase ($P = 0.00035$) in HLM values with the removal of impurities. However, the increase was not consistent for all the devices indicating that the increase in HLM is dependent on the device ($P < 0.00057$) being used (Fig. 30). The average increase in HLM for the two South African devices was 0.98 $kg.hL^{-1}$ and 0.94 $kg.hL^{-1}$ (Table 11), respectively; indicating that similar devices resulted in similar increases in HLM after the removal of impurities. The HLM increases for the other devices are also displayed (Table 11).

Experiment 7: Effect of operator on HLM determination

(Detailed results in Appendix 7, Table 7.7)

The investigation on the effect of different operators on HLM determinations on two South African devices showed that a significant ($P < 0.05$) operator effect existed between three operators with different levels of skill and experience (Fig. 31). The average HLM measurements for the operators are displayed in Table 12. The lowest ICC agreement (0.920) and consistency (0.916) values were observed for the least skilled operator. However, these values did increase with increasing experience and were 0.947 (ICC agreement) and 0.945 (ICC consistency) for the results obtained on the second South African device.

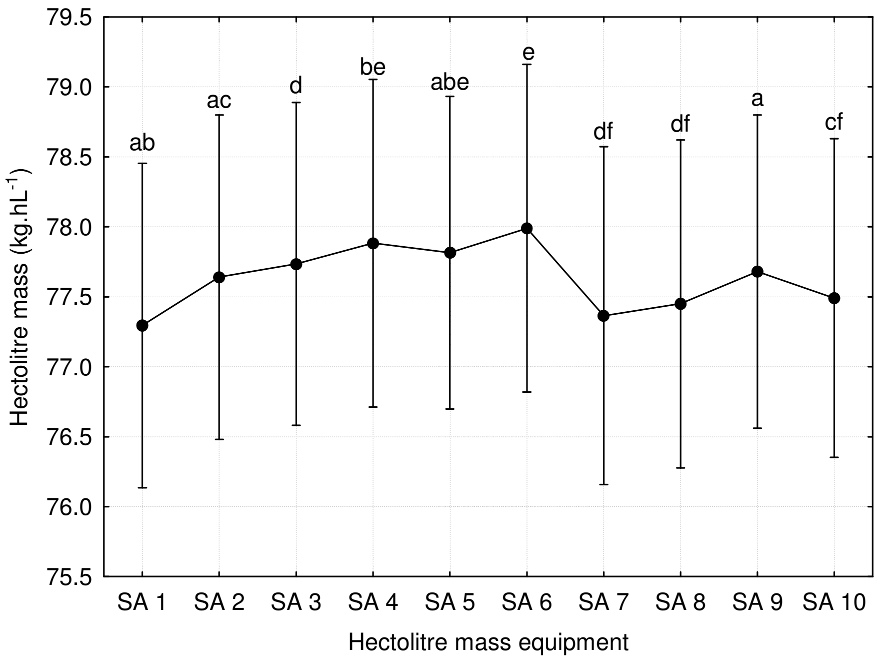

Figure 28 The evaluation of ten South African hectolitre mass (HLM) devices currently in commercial use. Error bars denote 0.95 confidence intervals.

Table 10 Hectolitre mass (HLM) values (mean ± standard error (se)) of ten South African devices using mixed wheat samples

HLM Device	**Mean ± se**	***n***
SA 1	77.29 ± 0.512	10
SA 2	77.64 ± 0.512	10
SA 3	77.73 ± 0.510	10
SA 4	77.88 ± 0.518	10
SA 5	77.82 ± 0.494	10
SA 6	77.99 ± 0.518	10
SA 7	77.37 ± 0.534	10
SA 8	77.45 ± 0.518	10
SA 9	77.68 ± 0.495	10
SA 10	77.49 ± 0.503	10

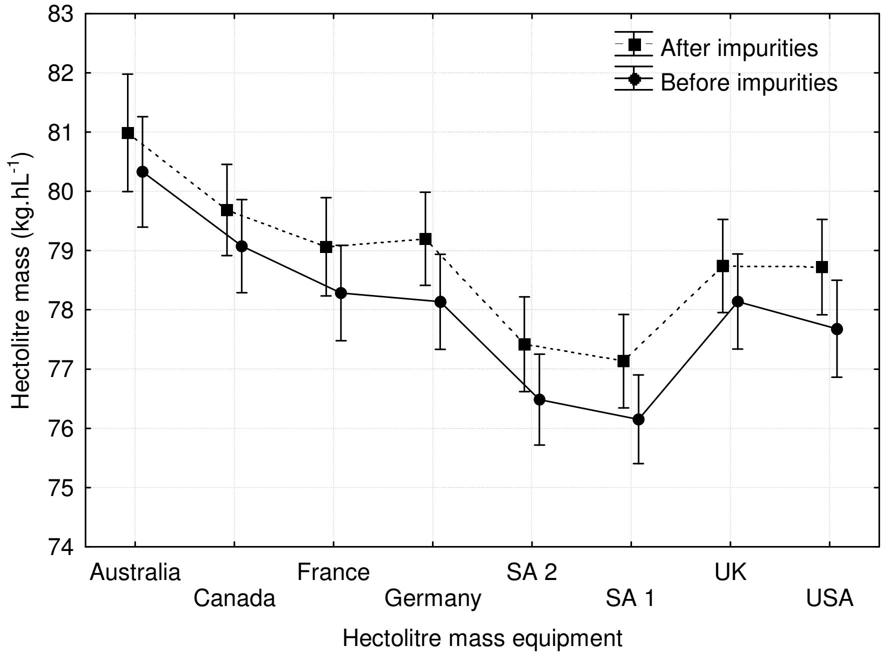

Figure 29 Evaluation of hectolitre mass (HLM) devices before and after impurities have been removed. Error bars denote 0.95 confidence intervals. The interaction effect was significant ($P < 0.01$) which indicate that differences before and after the removal of impurities are device dependent.

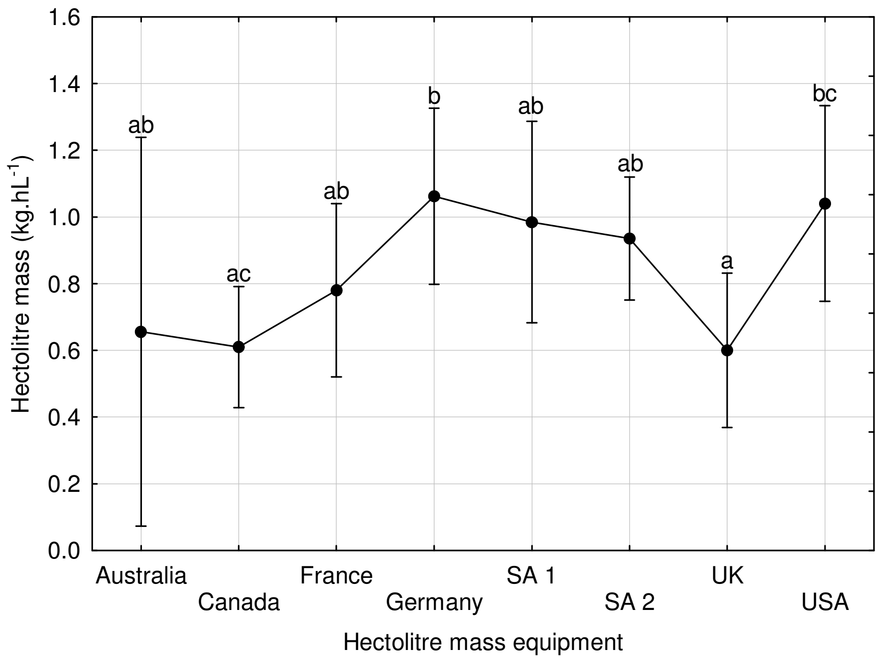

Figure 30 Increase in hectolitre mass (HLM) values observed for the HLM devices after impurities have been removed. Error bars denote 0.95 confidence intervals.

Table 11 Hectolitre mass (HLM) values (mean ± standard error (se)) after the removal of impurities

HLM Device	**Mean ± se**	***n***
Australia	0.66 ± 0.257	10
Canada	0.61 ± 0.080	10
France	0.78 ± 0.115	10
Germany	1.06 ± 0.117	10
SA 1	0.98 ± 0.133	10
SA 2	0.94 ± 0.082	10
UK	0.60 ± 0.102	10
USA	1.04 ± 0.130	10

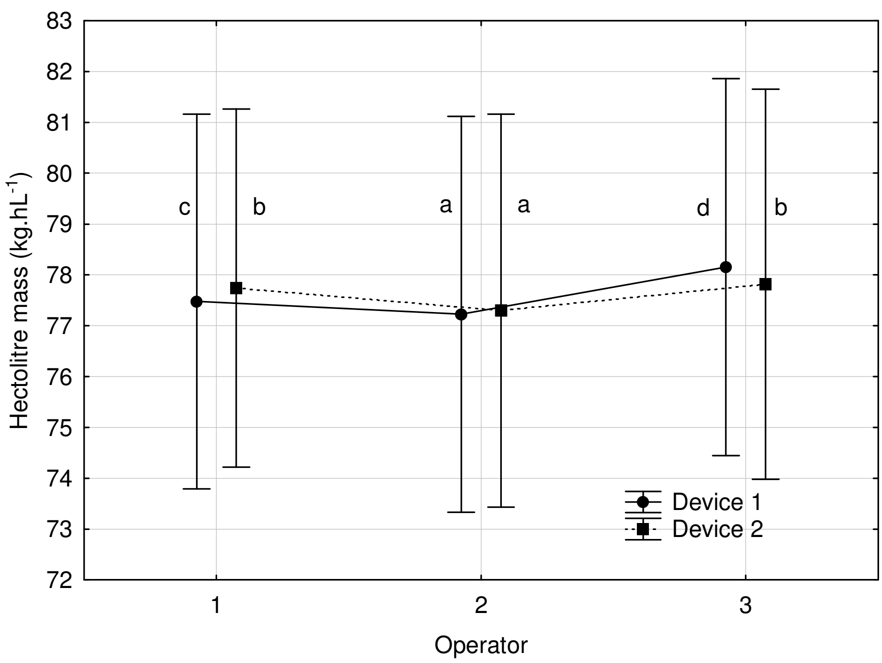

Figure 31 The effect of different operators on two South African hectolitre mass (HLM) devices. Error bars denote 0.95 confidence intervals.

Table 12 Hectolitre mass (HLM) values (mean ± standard error (se)) of obtained with two SA HLM devices by three operators using mixed wheat samples

HLM device	Operator	Mean ± se	*n*
SA 1	1	77.48 ± 1.629	10
SA 1	2	77.22 ± 1.721	10
SA 1	3	78.15 ± 1.638	10
SA 2	1	77.74 ± 1.557	10
SA 2	2	77.30 ± 1.708	10
SA 2	3	77.81 ± 1.696	10

Experiment 8: Effect of moisture on HLM determinations

(Detailed results in Appendix 7, Table 7.8)

The HLM values significantly ($P < 0.05$) decreased as the moisture content increased (Fig. 32). The different moisture treatments subjected to the samples can be seen in Table 4. The results in Fig. 33 illustrated the effect of the different moisture treatments on the average HLM values of the samples. After the first wetting the samples were dried to *ca.* 10% where after the samples were conditioned back to their moisture contents before they were dried, i.e. control, 14%, 16% and 18%, respectively (Table 4). It was found (Fig. 33) that the HLM of the samples that had been conditioned to 14% and 16% moisture content and dried to *ca.* 10% moisture content did not decrease below their initial HLM values when conditioned back to *ca.* 14% and 16% moisture content. The HLM values of the samples that were conditioned to 18% decreased below their initial HLM values after being dried and conditioned back to what their moisture contents were before drying. It therefore seems that the more moisture is added and removed, the more severe the effect on HLM measurements. The control samples (Fig. 34), which did not receive any moisture treatment before drying, did not significantly ($P > 0.05$) increase in HLM values when dried (*ca.* 0.39 $kg.hL^{-1}$ average for all the samples). When the dried control samples were conditioned back to their starting moisture content the average HLM significantly ($P < 0.05$) decreased with *ca*. 0.78 $kg.hL^{-1}$ and 1.2 $kg.hL^{-1}$ below their initial HLM and the HLM they were after drying, respectively.

The changes in HLM values brought about by the different moisture treatments were significantly ($P < 0.05$) different for the devices (Fig. 35). The average HLM values of the samples had a severe drop in HLM after receiving the first wetting. The samples that were dried after the first wetting was still lower than the control samples. Whereas, the second wetting (after drying) decreased the average HLM values of the samples even further. It is clear that wetting and drying cycles change the density and integrity of the wheat and that drying will not restore the wheat to its original HLM.

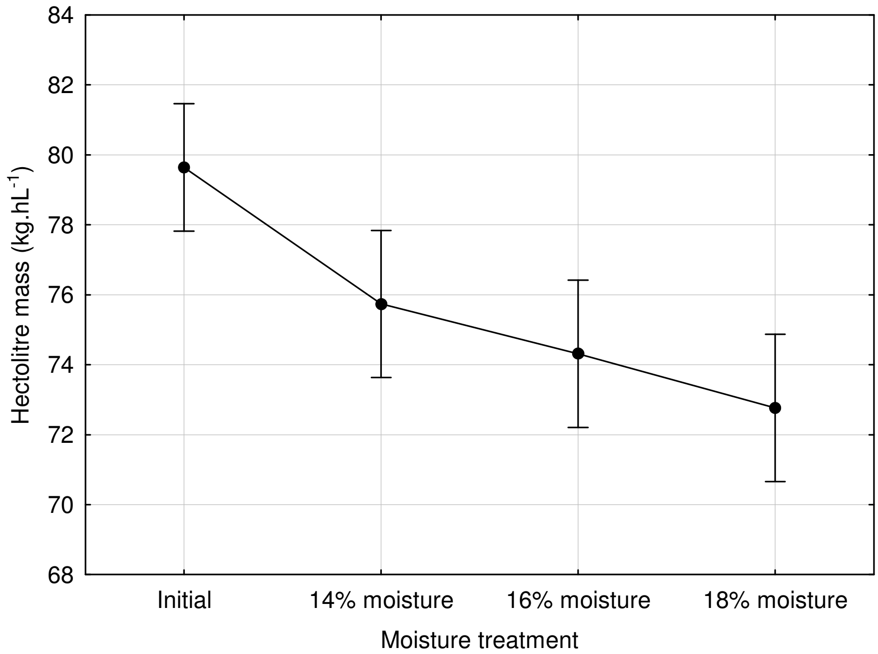

Figure 32 Effect of moisture content on the mean hectolitre mass (HLM) values as determined after conditioning of wheat samples. Error bars denote 0.95 confidence intervals.

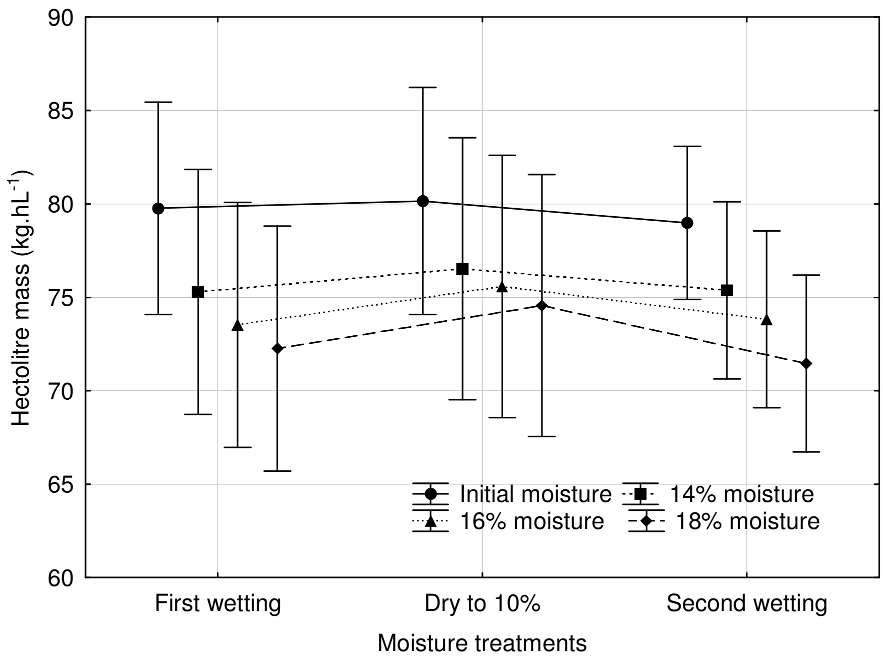

Figure 33 Effect of change in moisture content on mean hectolitre mass (HLM) values evaluated in terms of the effect of wetting (treatments 1 & 3) and drying (treatment 2) cycles. Error bars denote 0.95 confidence intervals.

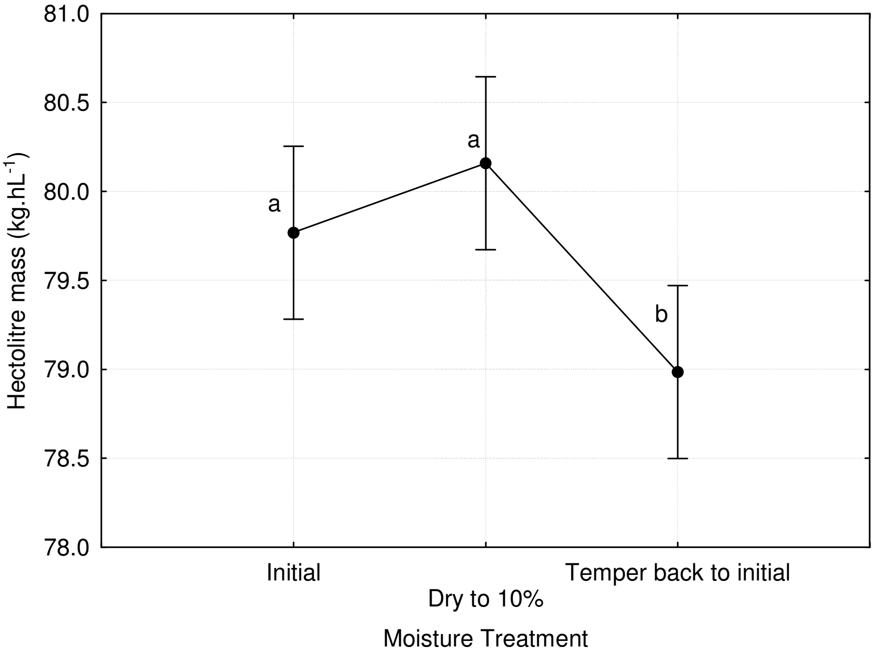

Figure 34 Effect of moisture treatment on the mean hectolitre mass (HLM) values of the control samples. Error bars denote 0.95 confidence intervals.

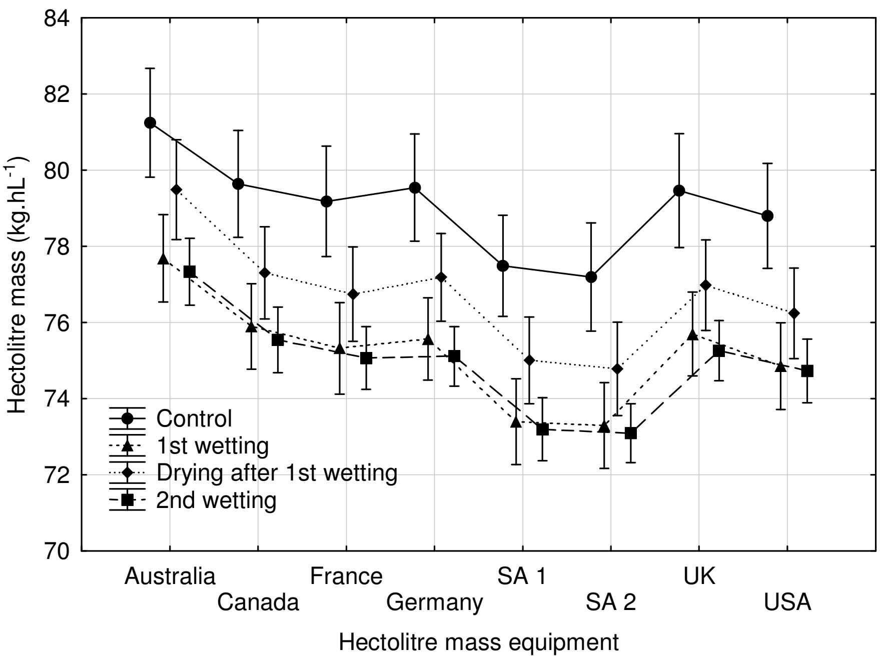

Figure 35 The effect of wetting and drying on hectolitre mass (HLM) measurements obtained with the different HLM devices. Error bars denote 0.95 confidence intervals.

Discussion

Apart from the Australian device the HLM measurements performed on all the other HLM devices resulted in HLM values that were highly repeatable within each device (Table 5). The way the Australian device is operated can probably be held accountable for the less repeatable HLM values and possibly for the significant ($P < 0.05$) decrease in HLM values obtained when sample 11 was measured ten times consecutively (Fig. 10). During the filling process of the measuring container there is no controlled flow of the wheat into the receiving cylinder, such as is the case with the devices that use either a funnel or pre-filling column with or without a piston. This significant ($P < 0.05$) decrease could also have been coincidental, as it was not observed for the other two samples analysed. In contrast, a significant ($P < 0.05$) increase in the HLM values of sample 13 measured repeatedly (10 times) with the SA 2 device was observed (Fig. 12). This trend was, however, not observed in the HLM measurements obtained for the other samples also measured on this device, or any of the samples measured with the SA1 device. This significant increase could have been coincidental as no operational variation or errors were observed during the execution of this experiment, as was the case with the Australian device.

It has been reported by Shuey (1960) that frequent handling and moving of the grain might polish the bran coat of the kernels and causes the HLM to increase. In contrast, the current study revealed that HLM values did not change significantly with increasing measurements. The samples used in this study were received from flour mills where they were most likely frequently handled. In addition the samples have been cleaned, mixed and divided during this study. It could be assumed that the samples underwent maximum polishing and would not have changed further during HLM determinations. Therefore, the differences observed between the measurements would be due to the different devices being used and not due to the alteration of the sample when the tests were performed.

The repeatability of the HLM measurements within the respective devices was confirmed when HLM measurements were performed with two sets of ten respective wheat samples each, i.e. ten mixed wheat samples (Fig. 14) and ten South African wheat cultivars (Fig. 18). The lack of variation in the HLM values obtained within the respective devices is also an indication that the reduction of the bulk sample into sub- and then sub-sub samples has been done efficiently. The bulk samples were mixed and divided with a Boerner Divider to obtain homogenously reduced and representative sub-sub samples of the bulk wheat sample. This confirms the findings of Petersen *et al.* (2004) that the Boerner Divider is most efficient in reducing samples. The mixing and sub-sampling procedures subjected to the bulk samples were therefore successful in producing homogeneous, well mixed and representatively reduced sub-samples as sample variation did not seem to have influenced the HLM values obtained.

From the RANOVA results, for the mixed wheat (Fig. 15) and the single cultivars (Fig. 19), it was observed that the Australian device resulted in significantly higher ($P < 0.05$) and the South

African devices in significantly lower ($P < 0.05$) average HLM measurements, whilst all of the other devices gave average measurements that were not statistically different from one another ($P > 0.05$). The significant differences in HLM values observed between the respective devices could possibly have been explained in terms of the different ways in which the devices are operated and the different volumes of grain used. This is apart from the Australian device where the significant ($P < 0.05$) difference could be explained due to the lack in controlled flow when filling the receiving cylinder. The difference observed between the devices (Fig. 19) was due to the use of the more relaxed LSD as indicator of significance as opposed to the conservative method of Bonferroni (Fig. 15), which is less prone to indicate small significant differences between objects. When the ICC agreement was determined to evaluate the differences in HLM measurements, in terms of actual values between the respective devices, it was found for both the mixed samples (Fig. 16) and single wheat cultivars (Fig. 20) that the Australian device resulted in HLM values that were most different, compared to the other devices. The South African devices also resulted in different HLM values compared to the other devices but not to the same extend as the Australian device. It was found that the HLM values obtained from the respective devices agreed better with each other when HLM measurements were performed with the single South African wheat cultivars (higher ICC agreement values) as opposed to the mixed wheat samples (lower ICC agreement values). This was expected because the South African cultivar samples consist of a single cultivar whereas the mixed wheat may have consisted of a mixture of cultivars; consequently more variation could have been expected within the latter samples. The assumption can, therefore, be made that when the HLM of mixed wheat samples are determined, more variation can be expected in the HLM measurements due to the variation within the samples.

The ICC agreement of the USA device agreed most with the South African devices, which means that their actual average measurements were most similar (ICC agreement = 0.594-0.893). The French device related second best to the South African devices in terms of average actual measurements (ICC agreement = 0.537-0.871) whilst the Australian device related the least (ICC agreement = 0.148-0.462). The HLM devices currently used in Canada, France, Germany and the United Kingdom had similar ICC agreement values and consequently the HLM values obtained from these devices were most similar. It would have been ideal to compare the respective devices with a 20 L volume as described in the International Recommendation (Anon., 1974). However, it is known that the device from UK has been matched to an EC 20 L volume and conforms to ISO 7971-2:1995. Similarly the device from Germany is known to be compliant to the ISO 7971-2:1995 standard. These two devices could, therefore, be assumed to give the most acceptable and presumably accurate results.

High ICC consistency values were determined between all the HLM devices for HLM measurements executed with both mixed wheat samples (Fig. 17) and single cultivars (Fig. 21). As expected it was observed that the ICC consistency values determined for the single wheat cultivars were higher and more consistent than those determined for the mixed wheat samples. The Australian device again was found to correlate the least (ICC consistency = 0.86 for mixed wheat and 0.98 for single cultivar samples) with the other HLM devices. It would, however, be possible that the HLM results obtained from the respective devices could be made comparable by means of correction factors. Hectolitre mass results obtained on the respective devices can be made comparable by converting the HLM results of a specific HLM device, as necessary, to match that of another using an appropriate calculated correction factor.

Comparing the devices (with RANOVA), using a single work sample indicated that the Australian device resulted in the highest, and the South African devices in the lowest average measurements for the mixed wheat (Fig. 22) and for the single cultivars (Fig. 25). When the actual measurements (ICC agreement) were compared it was found that the American device related the best and the French device the second best to the South African devices for both the mixed wheat (Fig. 23) and the cultivars (Fig. 26). Nevertheless, all the devices correlated highly with each other (Fig. 24 and Fig. 28 for mixed wheat and single cultivars, respectively) indicating again that the possible use of correction factors could allow direct comparisons between the different HLM devices.

Due to difficulties to get access to multiple devices from the other countries, comparison between ten of each of the international devices was not possible. As these devices were newly purchased, it was expected that the device obtained from each country would give repeatable results. The ten South African devices were old and are not currently manufactured, therefore it was crucial to determine whether they delivered repeatable results. It was found that the ten South African devices differed significantly ($P < 0.05$) from one another (Fig. 28). However, the ICC agreement showed that in terms of actual measurements, the average HLM measurements of the respective devices were similar, as an overall ICC agreement factor of 0.975 was obtained. Furthermore, the ICC consistency showed that the HLM measurements obtained from the respective devices were highly correlated (ICC consistency = 0.994). Therefore, it can be accepted that all the devices resulted in the same HLM measurement for the same sample of wheat. However, slight differences between the volumes of the South African 500 mL measuring containers exist, and could have influenced the HLM values. The small variation present in the HLM results could also have been due to the fact that some of the devices were not in as good condition as would have been expected. Some of the South African devices did not have the correct scraper, some closing valves did not close properly, some hoppers were not properly aligned (hanging loose) and some measuring cups were dented. Furthermore, HLM measurements should be done in an environment free from vibrations as vibrations can result in compaction of the wheat and consequently an incorrect HLM result. At one location such vibrations were experienced due to the elevators in the mill and that could have influenced the results obtained at that specific location. All these factors could have had a detrimental influence on the HLM results and if all the devices were in good condition the results could have been better. Maintenance of the devices is vital to ensure that the devices are always in proper working condition.

It has been observed that the removal of impurities from the wheat resulted in an increase in HLM values. The increase in HLM values was significantly different ($P < 0.05$) for the respective devices indicating a significant device dependency (Figure 30). The results obtained from the two respective South African devices were, however, similar. Previous research showed an average increase obtained in HLM using a bushel device, after the dockage had been removed from a sample of wheat, to be 2.07 kg.hL^{-1} (Greenaway *et al.*, 1971). The results obtained in the current study using a bushel device (i.e., the device from the USA) indicated about half the increase in HLM compared to results obtained by Greenaway *et al.* (1971). The method of dockage removal as used by Greenaway *et al.* (1971) might have been different from the method used in this study and therefore likely to be the reason for the discrepancy in the results.

Significant differences were found between HLM values when different operators, using the South African devices, performed the measurements. The average differences in HLM of 0.5 and 0.6 kg.hL^{-1} when 10 different samples had been analysed on the two respective devices by the skilled and unskilled operators are, however, not significant in practice. The HLM values obtained by the semi-skilled operator differed significantly from those obtained by both the skilled and unskilled operators. As there was reason to believe that these results were not reliably obtained it was not considered suitable to include them in further discussions. In an earlier study, significant differences were also found between operators, one was less skilled and experienced than the other, for five grains tested (Greenaway *et al.*, 1971). It was pointed out by Greenaway *et al.* (1971) that there was place for improvement in the results if one or both of the operators' techniques were to improve. Operator 3, in the current study, had never performed the HLM test previously and still delivered fairly good results. Nevertheless, if Operator 3 had the skill and experience, as also pointed out by Greenaway *et al.* (1971), of the skilled operator the results would have been more agreeable.

The mean HLM values of the samples that have been conditioned to 14, 16 and 18% moisture contents, respectively, showed a remarkable decrease in mean HLM values as the moisture content increased (Fig. 32). This confirms similar results from previous studies, which also showed that HLM values decreased as moisture contents increased (Lockwood, 1960; Pushman, 1975; Hook, 1984). The lower HLM values obtained after the samples have been wetted reflected the poor packing efficiency and lower density of the wetted wheat, which was also pointed out by Pushman (1975). The swelling of the kernel and the roughening of the bran coat may be responsible for the decrease in HLM, as was also observed by Pushman (1975). The fact that the density of the dry wheat (starch) is 1.51 g.cm^3 as opposed to that of 1 g.cm^3 of water may also have resulted in the wetter wheat being lighter and in the lower HLM values compared to the dry wheat (Hlynka & Bushuk, 1959). However, it has been observed earlier that changes in surface friction played a greater role in altering HLM values than changes in grain density (Scott, 1951). Samples that were wetted, then dried and wetted again to their moisture contents of the first respective wettings did not reach the HLM values as have been obtained after the first wetting (Fig. 33). It is thus clear that wet and dry cycles change the integrity and density of the wheat and the HLM of the initial sample will never be obtained again once the sample has been wetted or dried. Correction factors cannot be applied to samples that has been wetted or dried as it has been noticed that different wheat samples responded differently to the increase or decrease in moisture content (results not shown); the higher the HLM the bigger the decrease in HLM values when wetted.

Conclusions

The following general conclusions can be made from this study:

- The HLM values obtained were shown to be very repeatable within each respective device.
- The repeated analyses of the same work sample did not scour the wheat kernels to such an extent that the HLM values were significantly affected.
- Clean, impurity free wheat resulted in higher HLM values compared to unclean wheat and device dependency was observed.
- Different operators did not have a significant effect on the HLM measurements in terms of practical applications.
- Different wheat samples responded differently to the addition or removal of moisture hence the assumption that correction factors cannot be applied to wheat that has been subjected to wet and dry cycles.
- Significant statistical differences were obtained, in average HLM values, when ten respective SA devices were compared, but these differences would not be significant in practice.
- Comparison of the different HLM devices revealed that the Australian device resulted in the highest HLM values and the South African devices the lowest.
- The HLM values obtained with the devices from Canada, France, Germany and the UK did not differ significantly.
- High correlations observed between the HLM values obtained from the respective devices indicated the possible use of correction factors to convert HLM determinations between devices.

Correction factors could, therefore, be used to convert the HLM determinations between the respective devices. Alternatively the South African wheat industry could choose to replace the South African device currently in use with one of the international devices. A suitable replacement would be one of the devices that are compliant to the ISO 7971-2:1995 standard, i.e. the devices from Germany and the UK. The device from Germany could be a suitable replacement as it is a very stable device and less prone to jarring than the other devices. Furthermore, it is accompanied with a pre-filling measure that allows for consistent filling of the one litre measuring cylinder. Additionally it is equipped with a piston that controls the rate of fall and ensures a smooth flow of grain from the filling hopper into the measuring cylinder.

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CHAPTER 4

Assessment of variance in measurement of hectolitre mass of maize, using equipment from different grain producing and exporting countries

Assessment of variance in measurement of hectolitre mass of maize, using equipment from different grain producing and exporting countries

Abstract

The South African maize industry does not have an official method for hectolitre mass (HLM) determination. Hectolitre mass determinations are currently unofficially performed on the lower platform of the two-leveled South African device. Hectolitre mass devices from different countries, including the device from South Africa, were evaluated and compared in this study in order to suggest a possible device for utilisation in the maize milling industry. All the devices were found to produce repeatable HLM results within each device and repeated analysis on the same sample of maize did not increase the HLM values. It was found that the devices from Australia and France resulted in significantly ($P < 0.05$) higher and the device from Canada in significantly ($P < 0.05$) lower HLM values compared to the other devices. The devices from the United States of America (USA), the United Kingdom (UK), Germany and South Africa, on the other hand, resulted in HLM values not significantly ($P > 0.05$) different to each other. The French device, equipped with a funnel hopper, did not allow the maize kernels to flow freely into the measuring cylinder and it is advised not to use this device for the HLM determination of maize. Nevertheless all the devices correlated (ICC consistency > 0.97) well with one another and correction factors can be calculated, if required, to convert between the HLM values obtained with the respective devices. Hectolitre mass values determined before and after the removal of impurities showed a significant ($P = 0.0324$) increase after the removal of impurities. The device from Germany is suggested to be the most suitable device to be used for the HLM determination of maize in South Africa.

Keywords: hectolitre mass; hectolitre mass devices; impurities; maize; wheat

Introduction

No official method for hectolitre mass (HLM) determination of maize has been established in the South African maize grading standards. This is probably due to the contradicting results regarding the relationship between HLM and maize quality, especially in terms of milling index. One of the reasons for this is believed to be the density of the maize kernels and the way the kernels pack into the measuring container of the HLM device (Rutledge, 1978). As large spaces are often present between the maize kernels in the measuring cup the HLM does not reflect the true mass of the maize per volume in the cup. It is believed that maize has an average void volume (space between the kernels in a bulk) of 42.3% (Thompson & Isaacs, 1967). In an attempt to reduce the amount of air spaces and to ensure closer packing in the cup, the HLM determination is unofficially performed on the lower level of the two-leveled South African device.

HLM determination is, however, used in the United States of America (USA) maize standards but not as a precise indicator of any specific grain quality attribute (Dorsey-Redding *et al.*, 1991). It is regarded as an indication of grain soundness and is used to determine storage space (Watson, 1987). It has been reported that maize with low HLM often has a lower percentage of hard endosperm and consequently, produces a lower yield of prime, large grits when milled (Rutledge, 1978). High correlation coefficients were reported between HLM and density (r = 0.936) as well as HLM and hardness (r = 0.72) for yellow dent maize hybrids (Pomeranz *et al.*, 1986), indicating the value of HLM as an index to maize quality. Significant correlations between HLM and density (r = 0.78 and 0.80; P = 0.0001) and HLM and hardness (r = 0.69 and 0.67; P = 0.0001) were also established in a study over two crop years (1987 and 1988) (Dorsey-Redding *et al.*, 1991). A need for simple, rapid and reliable tests that will relate maize quality to end-product yield was highlighted by Dorsey-Redding *et al.* (1991). As the measurement of HLM is rapid and easy to perform, it can be utilised in the maize industry as a rapid quality test.

Studies performed on wheat suggested that the HLM would increase as sample handling increased (Shuey, 1960; Halverson & Zeleny, 1988). It is believed that handling polishes the wheat and thus allows closer packing of the wheat in the container. Similar studies have not been performed on maize as yet. The effect of impurities on the HLM of wheat has been widely studied and it was found that the removal of impurities would increase the HLM value (Lockwood, 1960; Greenaway *et al.,* 1971). HLM determination for maize is done on dirty (impurities not removed) maize. As yet it has not been reported whether the impurities present can have a negative effect on the HLM values of maize.

The objectives of this study were therefore:

- To develop a suitable method of HLM determination for maize in South Africa by evaluating HLM equipment as used in Australia, the UK, Canada, France, Germany and the USA, compared to duplicate South African devices, using maize samples of varying HLM values; and
- To determine the effect of impurities on the HLM value of maize.

Materials and Methods

Maize samples and hectolitre mass equipment

Maize samples were obtained from maize mills in South Africa that covered a range of *ca.* 72-79 kg.hL^{-1} (Table 1). The samples were stored at room temperature with regular periods of fumigation with pyrethroid insecticides to prevent infestation. The HLM equipment evaluated included devices from Australia (Graintec Pty Ltd., Peregian Beach, Queensland, Australia), Canada (Dimo's Tool & Die Ltd., Winnipeg, Canada), France (Chopin Technologies, Villeneuve-la-Garenne Cedex, France), Germany (KERN & Sohn GmbH, Barlingen-Frommern, Germany), South Africa, United Kingdom (Farm-Tec, Whitby, North Yorkshire, UK) and USA (Seedburo Equipment Co., Chicago, USA). The description and operating procedures of the respective HLM devices are described in detail in Chapter 3. The same operator performed all the HLM measurements.

Table 1 The HLM values (determined with the South African device) of the maize samples, before and after the removal of impurities

Sample number	HLM ($kg.hL^{-1}$) before removal of impurities	HLM ($kg.hL^{-1}$) after removal of impurities
1	77.13	76.81
2	77.89	77.05
3	76.59	77.82
4	77.85	78.58
5	74.39	75.12
6	77.48	77.51
7	78.03	78.89
8	79.45	80.10
9	76.36	77.58
10	72.08	72.51
11	77.33	77.99

*Impurities were removed with a standard South African maize grading sieve (6.35 mm round-hole sieve).

Experimental Procedures

A schematic layout of the sequence of the experiments performed is depicted in Figure 1 and the detailed layout of each respective experiment in Figs. 2-6.

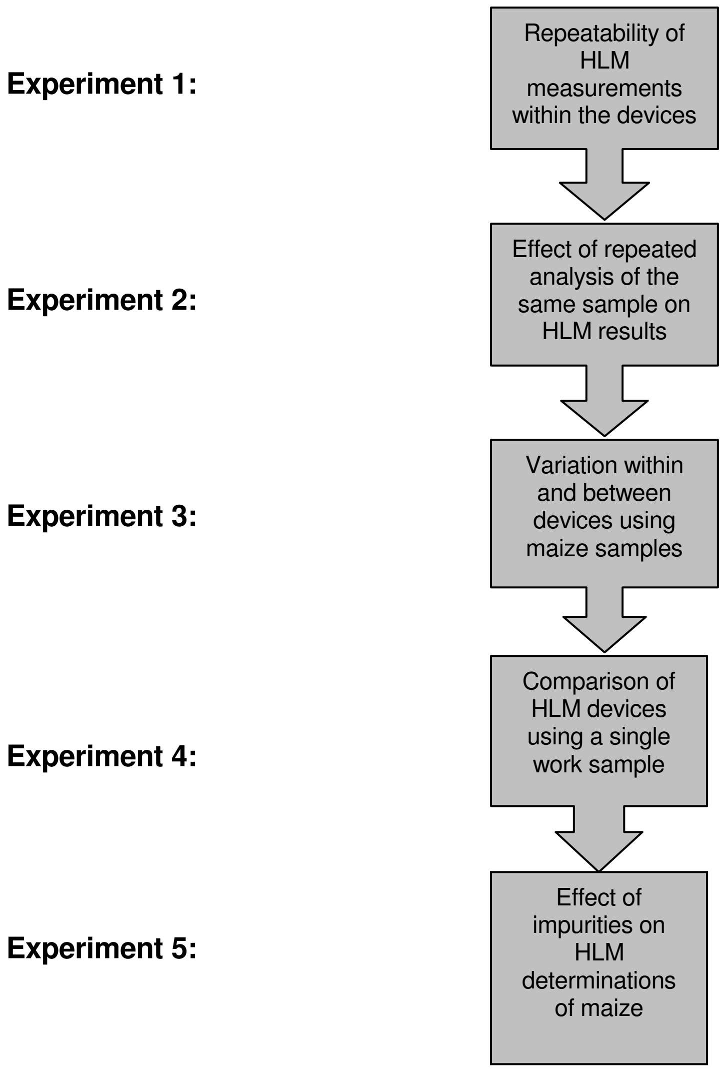

Figure 1 Schematic layout of the sequence of the respective experiments performed.

Experiment 1: Repeatability within the HLM devices (Fig.2)

A 12 kg maize sample (Table 1; sample 2; HLM = 77.89 $kg.hL^{-1}$) was obtained and thoroughly mixed with a laboratory mixer (Model MR10L, Chopin, Villeneuve-la-Garenne Cedex, France) for 15 minutes. Subsequently it was divided into eight, one and a half kilogram samples; one individual sample to be analysed on each device. The order of the devices, on which the HLM values were determined, was randomly selected. Ten repetitions were performed on each device and between each repetition the one and a half kilogram sample were mixed by pouring it, five times, from one bucket to another. The HLM measurements were performed on each device according to the operating procedures as described in detail in Chapter 3.

Experiment 2: Effect of repeated analysis of the same maize sample on the HLM (Fig. 3)

Three maize samples (12 kg each) were obtained (Table 1; samples 3-5; HLM = 76.59; 77.85 & 74.39 $kg.hL^{-1}$). The samples were poured through a Boerner Divider (Seedburo, Chicago, USA) three times in order to obtain a homogenously mixed sample. Each sample was divided into eight (one sample for each device) one and a half kilogram samples. The HLM determinations were performed according to the operating procedures as described in Chapter 3. Ten consecutive repetitions were executed on the first maize sample with each HLM device, but after the first measurement only the amount of maize that was needed to perform the measurement (work sample) was kept for the following nine repetitions. The other samples were analysed similarly.

Experiment 3: Variation within and between the HLM devices using maize samples (Fig. 4)

Ten maize samples of 36 kg each (Table 1; samples 1-3 & 5-11; HLM = 72.08-79.45 $kg.hL^{-1}$) were obtained. The respective samples were poured through a Boerner Divider three times in order to obtain a well-mixed sample. Each 36 kg sample was divided into eight sub-samples of 4.5 kilograms each. These sub-samples were further divided into three, one and a half kilogram sub-sub samples. Each of the three sub-sub samples was tested in duplicate on each HLM device (the order of the maize samples and HLM devices was chosen randomly). After the first determination was performed the work sample was mixed back (poured five times from one bucket to another) with the rest of the sample, where after the second measurement (repetition) was done. The remaining samples were measured similarly.

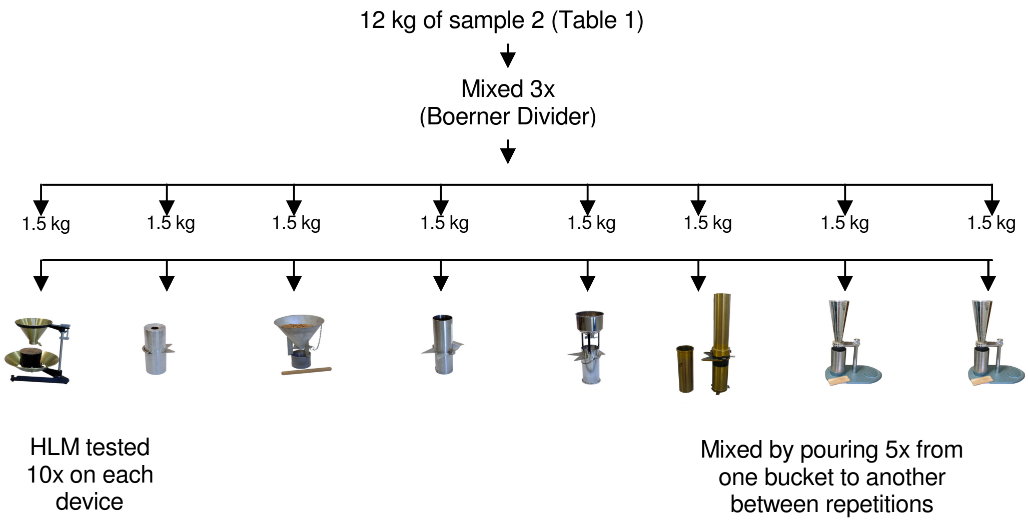

Figure 2 Schematic layout of Experiment 1: repeatability within the hectolitre mass (HLM) devices.

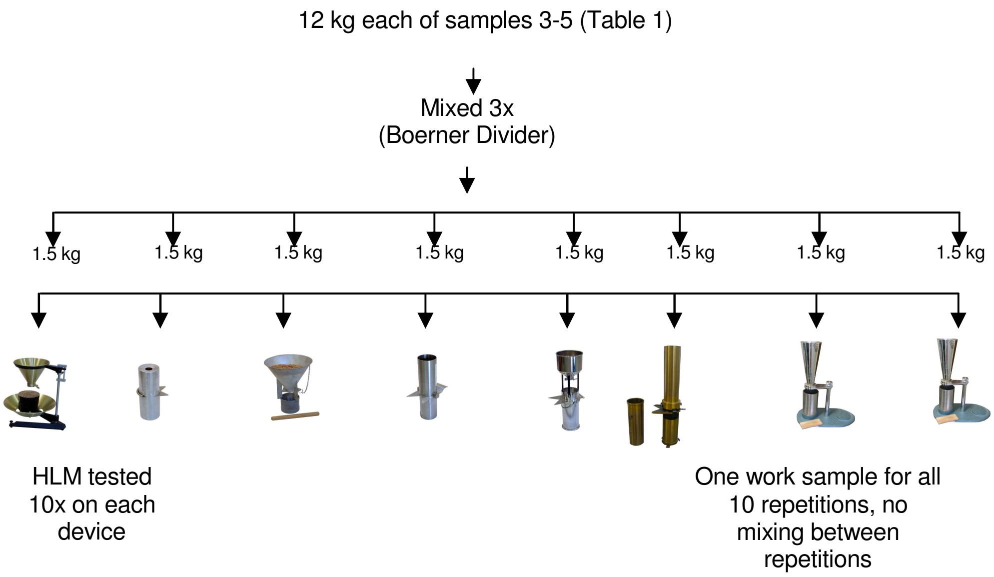

Figure 3 Schematic layout of Experiment 2: effect of repeated analysis on the same maize sample on its hectolitre mass (HLM).

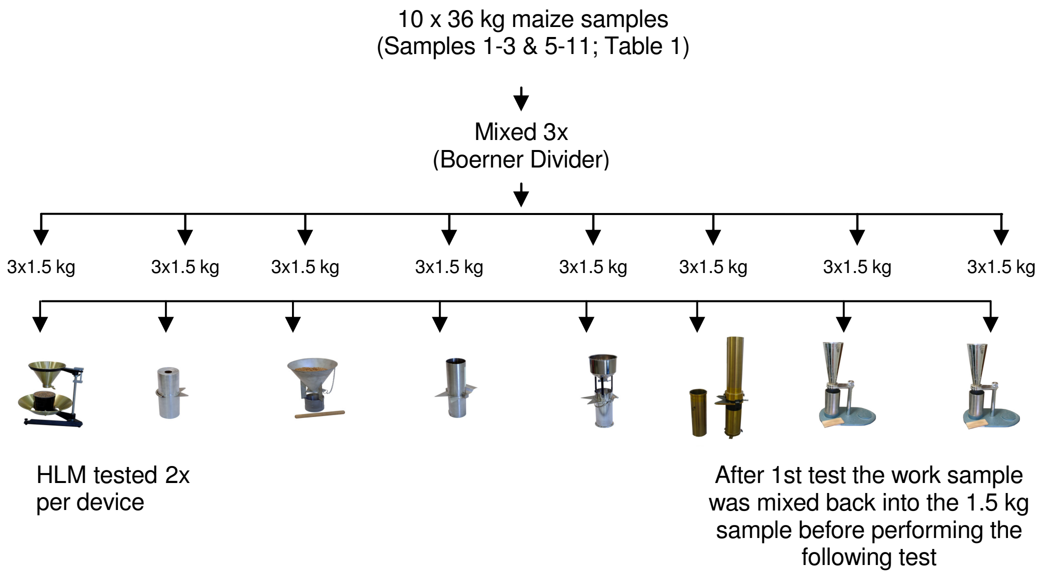

Figure 4 Schematic layout of Experiment 3: determination of variation in hectolitre mass (HLM) within and between devices.

Experiment 4: Comparison of HLM devices using a single work sample of maize (Fig. 5)

Ten respective maize samples of 4.5 kg each (Table 1; Samples 1-3 & 5-11; HLM = 72.08-79.45 $kg.hL^{-1}$) were obtained. The samples were poured through a Boerner Divider three times in order to obtain a well-mixed 4.5 kg sample. Each sample was divided into three, one and half-kilogram sub-samples. The experiment started with HLM determinations on the USA device, as this device requires a larger maize sample than the other devices. The testing order of the remaining devices was randomly chosen. The work sample obtained from the USA device was subsequently used to measure the HLM of the samples on the other devices, as well. Two repetitions were executed on each HLM device with the same work sample (from USA device), however, after the first measurement the order of the devices was randomly chosen. Similar procedures were followed for the other two sub-samples and the remaining samples.

Experiment 5: Effect of impurities on HLM determinations (Fig. 6)

Ten maize samples of 1.5 kg each (Table 1; Samples 1-3 & 5-11; HLM = 72.08-79.45 $kg.hL^{-1}$) were obtained. The first sample was poured through a Boerner Divider twice in order to obtain a well-mixed sample. The experiment started with HLM determinations on the USA device, as this device requires a larger maize sample than the other HLM devices. The work sample obtained from the USA device was subsequently used to measure the HLM on the remaining devices. Two repetitions were executed on each HLM device with the same work sample (from USA device), however after the first measurement the order of the devices was randomly chosen. The experiment was repeated with the other samples. The impurities were removed from all the samples (10 x 1.5 kg) by removing the broken and small maize kernels with a standard maize grading sieve (6.35 mm round-hole sieve). The larger foreign matter on top of the sieve was manually removed. Where after the samples were mixed once with a Boerner Divider and the HLM measurements conducted as described before.

Statistical analysis

Statistical analyses were performed and graphs compiled using Statistica version 7.1 (StatSoft, Inc., Tulsa, OK, USA). Repeated measures analysis of variance (RANOVA) was performed to compare average measurements between instruments to determine absolute differences. The bar around the average represents the 95% confidence interval for the average measurements. Bonferroni, Fisher least significant difference (LSD) and Tukey high significant difference (HSD) *post-hoc* testing was used. In summary tables the standard errors were reported with the means. All references to significant differences indicate statistical differences. The standard error (se) is related to the standard deviation (sd) in the following way: $\mathrm{se} = \mathrm{sd}/\sqrt{n}$, where n is the sample size. Additionally the intra-class correlation (ICC) coefficients were determined as the ICC agreement that correlates measurements with each other, while taking into account the differences in absolute values of the respective measurements, and the ICC consistency that only correlates measurements. All ICC calculations were done using the R statistical programming language.

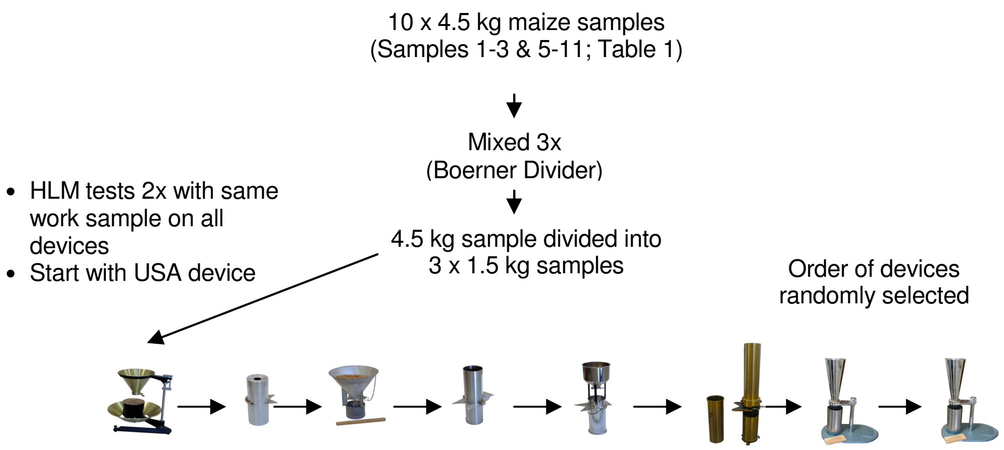

Figure 5 Schematic layout of Experiment 4: comparison of the hectolitre mass (HLM) devices using a single work sample.

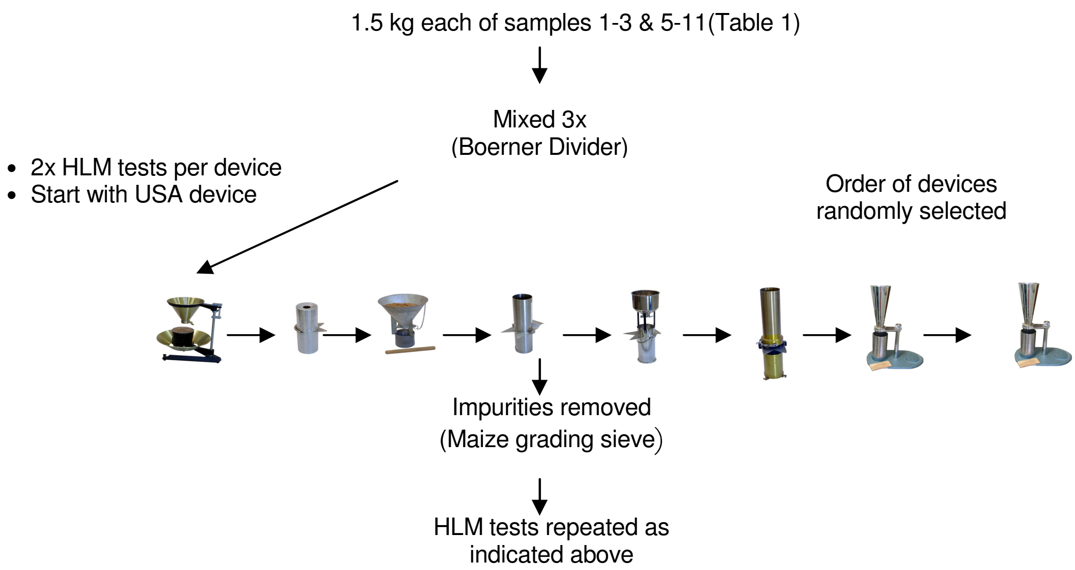

Figure 6 Schematic layout of Experiment 5: effect of impurities on hectolitre mass HLM determinations.

Results

Experiment 1: Repeatability within the respective HLM devices

(Detailed results in Appendix 8, Table 8.1)

The standard deviations of the HLM values obtained with the HLM devices (Table 2) show that the SA 1 and UK devices resulted in the least repeatable HLM results, whilst the devices from France and SA 2 resulted in the highest repeatability within devices.

Experiment 2: Effect of repeated analysis of the same maize sample on the HLM

(Detailed results in Appendix 8, Table 8.2)

The β-coefficients (slope of the regression line) and the P-values (describing the null-hypothesis i.e., that the slope of the regression line must be equal to zero) obtained by means of regression analysis are shown in Table 3. It was found that the HLM of the respective maize samples did not increase with repeated analysis as no significant slope was observed. There was, however, one exception as sample 3 measured on the SA 1 device showed an increase in HLM with increasing repetitions (β-coefficient = 0.73; $P = 0.02$). This could have been coincidental as the other two samples measured on the same device did not result in a significant slope.

Experiment 3: Variation within and between the HLM devices using maize samples

(Detailed results in Appendix 8, Table 8.3)

The ICC agreement indicates the repeatability of the measurements within each device in terms of actual values. The ICC agreement was high (ICC agreement > 0.96) for all the devices indicating little variation between the HLM values obtained when measurements were performed on the same sample within a particular device (Fig. 7). This also indicates efficient mixing and sub-sampling which ensured a representative sample. The bulk sample has been thoroughly mixed and divided with a Boerner Divider to produce the sub-samples for each device. This is in agreement with findings by Petersen et al. (2004), which showed that the Boerner Divider is the most efficient in providing a reduced sample that is representative of the bulk sample. The ICC consistency additionally showed that the repetitions within each device were highly correlated (ICC consistency > 0.96).

The RANOVA results showed that the average HLM measurements obtained on the German, South African, UK and USA devices did not differ significantly ($P > 0.05$) (Fig.8). The average HLM results obtained from the devices from Australia and France were significantly ($P < 0.05$) higher than those obtained with the other devices and the HLM results obtained with the Canadian device significantly ($P < 0.05$) lower.

Additionally the ICC agreement was determined to express the variation between the respective devices in terms of actual HLM values. An overall ICC agreement of 0.52 was obtained between the respective devices. The German, South African, UK and USA devices gave most similar results in terms of actual HLM values (Fig. 9). The Australian and French devices resulted in HLM values most different from the other devices followed by the Canadian device. The ICC agreements between the two South African devices and the other devices are displayed in Table 4. The ICC consistency showed that in spite of the low overall ICC agreement value the respective devices did correlate well with each other (ICC consistency > 0.986) (Fig. 10).

Table 2 Hectolitre mass (HLM) values (mean ± standard deviation (sd)) of the HLM devices

HLM device	Mean ± sd
Australia	81.65 ± 0.253
Canada	76.60 ± 0.244
France	81.45 ± 0.193
Germany	77.38 ± 0.216
SA 1	77.30 ± 0.304
SA 2	77.78 ± 0.182
UK	78.04 ± 0.295
USA	77.53 ± 0.229

Table 3 *β*-Coefficient and *P*-values showing the effect of repeated analysis on HLM values

	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
				Sample 3				
β-coefficient	0.23	0.058	0.41	0.27	0.73	0.073	-0.41	0.03
P-value	0.47	0.87	0.24	0.45	0.02	0.84	0.23	0.94
				Sample 4				
β-coefficient	-0.05	0.067	0.18	-0.26	0.02	0.12	0.07	-0.1
P-value	0.88	0.85	0.61	0.48	0.95	0.74	0.86	0.77
				Sample 5				
β-coefficient	0.36	-0.17	0.28	0.26	0.35	0.19	-0.29	0.45
P-value	0.31	0.64	0.44	0.47	0.32	0.59	0.42	0.19

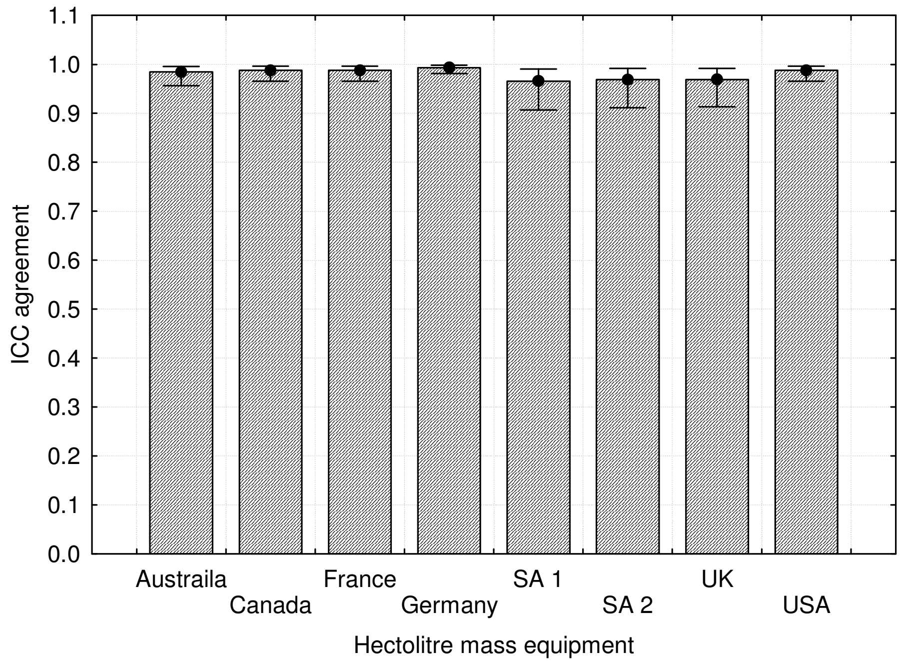

Figure 7 Intra-class correlation (ICC) agreement showing the variation in terms of actual hectolitre mass (HLM) values within HLM devices as determined with maize samples. Error bars denote 0.95 confidence intervals.

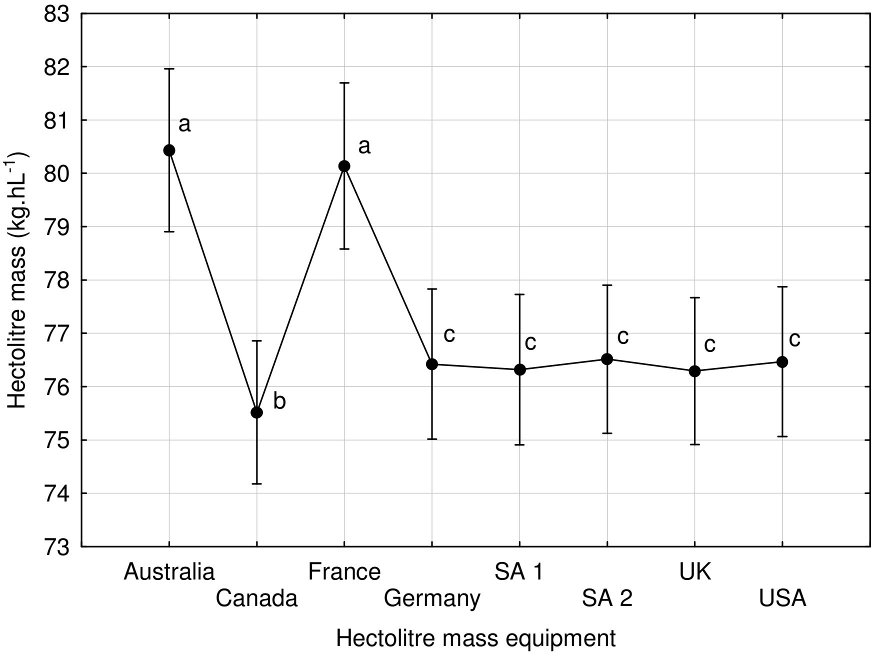

Figure 8 Differences between the average hectolitre mass (HLM) values obtained with the different HLM devices as determined by means of repeated analyses of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Tukey high significant difference (HSD) post-hoc analyses.

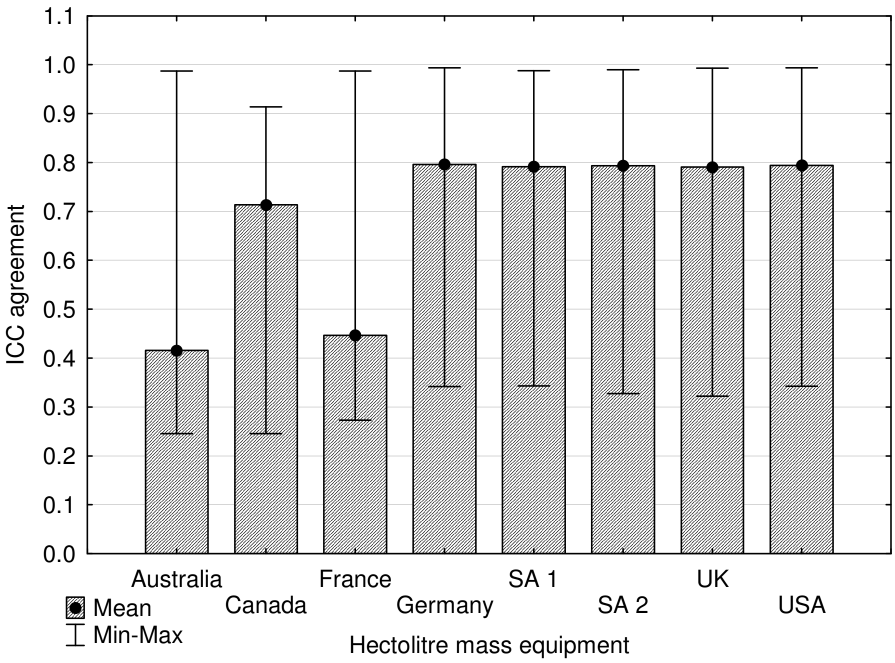

Figure 9 Intra-class correlation (ICC) agreement (average values calculated from Table 4) showing the variation in terms of actual hectolitre mass (HLM) values between HLM devices as determined with maize samples.

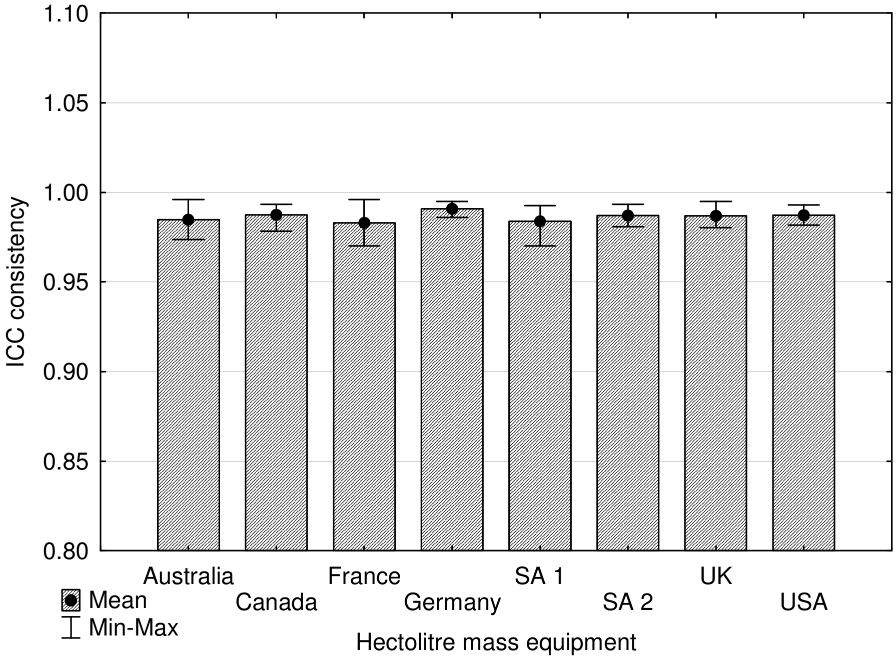

Figure 10 Intra-class correlation (ICC) consistency (average values calculated from Table 4) showing the variation in terms of actual hectolitre mass (HLM) values between HLM devices as determined with maize samples.

Table 4 Intra-class correlation (ICC) agreement and consistency showing the agreement, in terms of actual hectolitre mass (HLM) values of maize samples between the devices

HLM equipment		ICC agreement	ICC consistency
Australia	Canada	0.245	0.978
Australia	France	0.987	0.996
Australia	Germany	0.342	0.993
Australia	SA 1	0.327	0.982
Australia	SA 2	0.343	0.974
Australia	UK	0.322	0.988
Australia	USA	0.342	0.982
Canada	France	0.273	0.979
Canada	Germany	0.891	0.989
Canada	SA 1	0.914	0.993
Canada	SA 2	0.874	0.993
Canada	UK	0.913	0.989
Canada	USA	0.884	0.991
France	Germany	0.379	0.989
France	SA 1	0.365	0.981
France	SA 2	0.382	0.970
France	UK	0.356	0.980
France	USA	0.383	0.985
Germany	SA 1	0.990	0.990
Germany	SA 2	0.986	0.986
Germany	UK	0.993	0.995
Germany	USA	0.993	0.993
SA 2	SA 2	0.987	0.991
SA 1	UK	0.982	0.987
SA 1	USA	0.988	0.987
SA 2	UK	0.986	0.984
SA 2	USA	0.986	0.988
UK	USA	0.983	0.985
Overall		0.520	0.986

Experiment 4: Comparison of different HLM devices using a single work sample of maize
(Detailed results in Appendix 8, Table 8.4)

The ICC agreement (Fig. 11) indicates that little variation was observed in HLM values obtained by the measurements performed on the same sample within a particular device. Again it is an indication of efficient mixing and reducing of the bulk sample, using the Boerner Divider, resulting in representative sub-samples. Furthermore, the ICC consistency shows that the repetitions performed within each device were highly correlated (ICC consistency > 0.96).

The average HLM measurements obtained by means of RANOVA showed that the German, SA 2 and UK devices did not differ significantly ($P > 0.05$) (Fig. 12). Similarly it was observed that the average HLM values obtained with the SA 1, SA 2 and USA devices did not differ significantly ($P > 0.05$) (Fig. 12). The HLM results obtained with the devices from Australia and France were significantly ($P < 0.05$) higher than those obtained from the other devices. The results obtained with the Canadian device were significantly ($P < 0.05$) lower compared to the other devices.

Differences were observed between the actual HLM measurements obtained with the respective devices as expressed by means of the ICC agreement (Fig. 13). Again it was shown that the actual HLM values obtained with the devices from Australia and France were most different from those obtained with the USA, UK, German and South African devices followed by the device from Canada. From Table 5 it is clear that the devices from Germany, UK and USA devices resulted in HLM values most similar to that obtained by the two South African devices, in terms of actual values (ICC agreement). The HLM values obtained with the Australian and French devices differed the most from the two South African devices (Table 5). The ICC consistency (> 0.93), on the other hand, shows that all the devices correlated well with each other in spite of not having resulted in similar HLM values (Fig. 14).

Experiment 5: Effect of impurities on HLM determinations
(Detailed results in Appendix 8, Table 8.5)

A significant ($P = 0.0324$) increase was found in HLM after the impurities were removed (Fig. 15). The average increase in HLM obtained with the devices, after the impurities were removed, is displayed in Table 6.

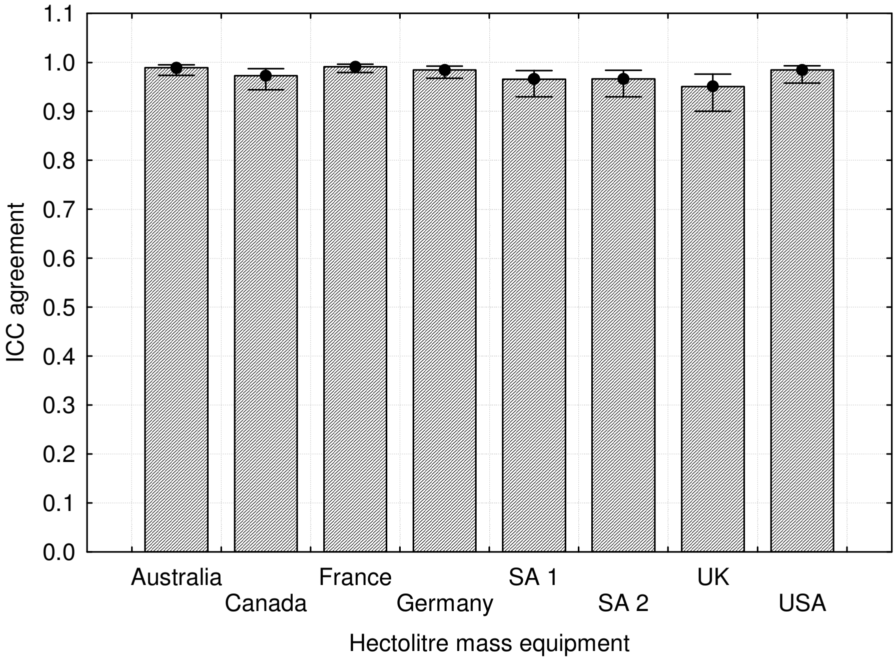

Figure 11 Intra-class correlation (ICC) agreement showing the variation in terms of actual hectolitre mass (HLM) values within HLM devices as determined using a single work sample of maize. Error bars denote 0.95 confidence intervals.

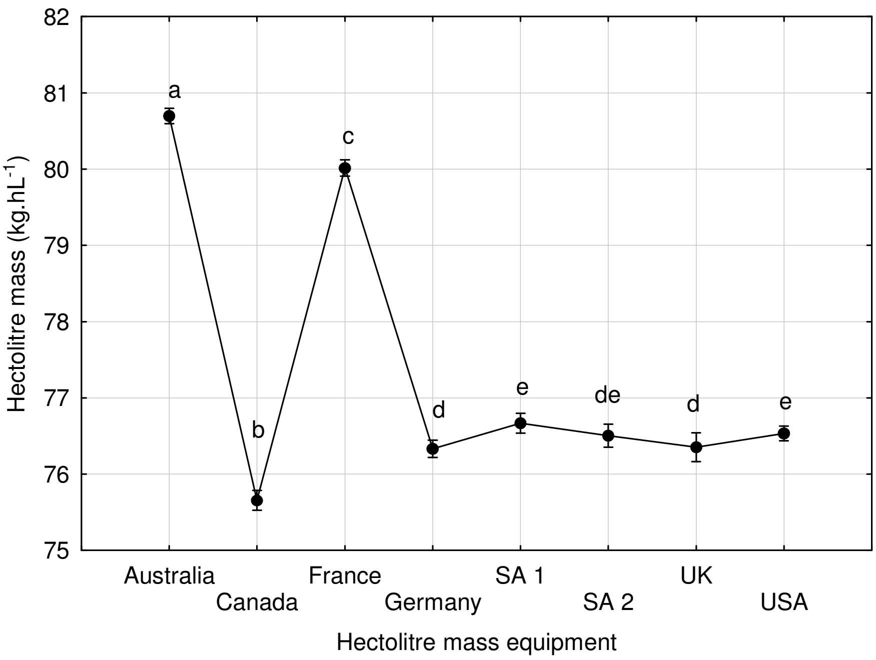

Figure 12 Differences between the average hectolitre mass (HLM) values obtained with the different HLM devices as determined by means of repeated analyses of variance (RANOVA). Error bars denote 0.95 confidence intervals. Different letters indicate significant differences obtained from Bonferroni post-hoc analyses.

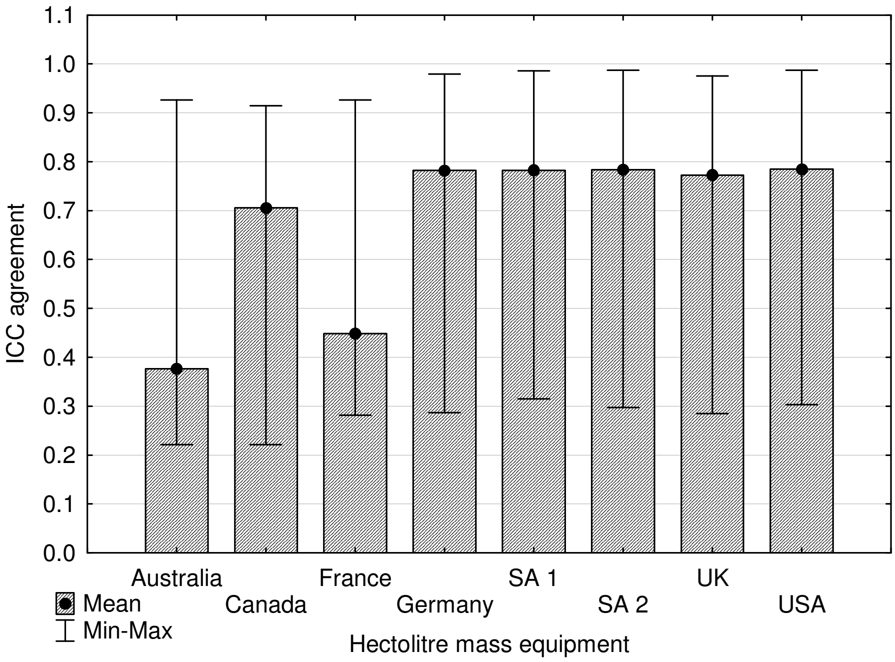

Figure 13 Intra-class correlation (ICC) agreement showing the differences, in terms of actual values, between the hectolitre mass (HLM) devices using a single work sample of maize.

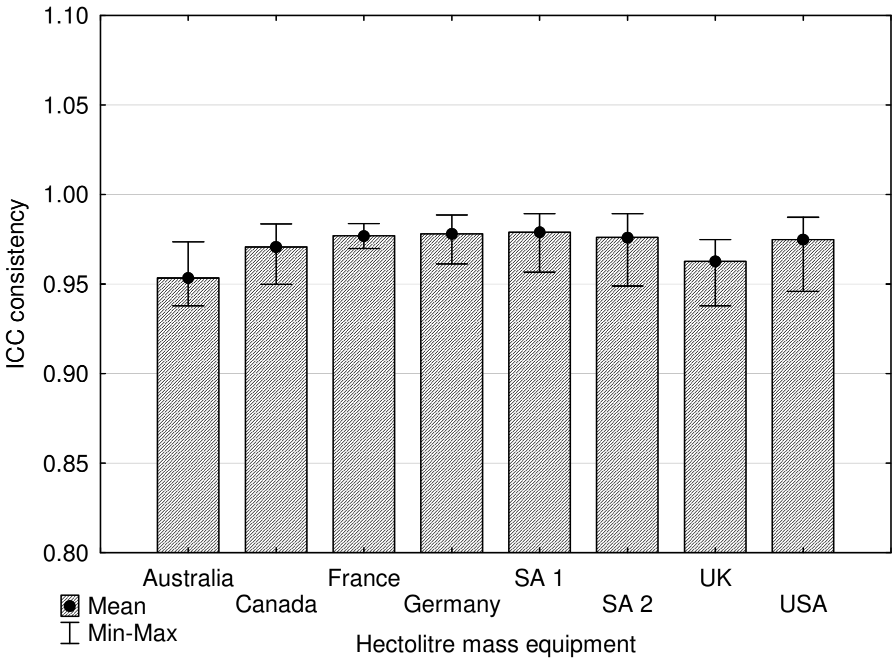

Figure 14 Intra-class correlation (ICC) consistency showing the correlation between the respective hectolitre mass (HLM) devices using a single work sample of maize.

Table 5 Intra-class correlation (ICC) agreement showing the agreement, in terms of actual hectolitre mass (HLM) values of maize samples, between the devices

HLM equipment		**ICC agreement**
Australia	Canada	0.221
Australia	France	0.926
Australia	Germany	0.287
Australia	SA 1	0.315
Australia	SA 2	0.297
Australia	UK	0.285
Australia	USA	0.303
Canada	France	0.282
Canada	Germany	0.915
Canada	SA 1	0.851
Canada	SA 2	0.886
Canada	UK	0.898
Canada	USA	0.886
France	Germany	0.368
France	SA 1	0.410
France	SA 2	0.387
France	UK	0.370
France	USA	0.396
Germany	SA 1	0.974
Germany	SA 2	0.980
Germany	UK	0.976
Germany	USA	0.976
SA 1	SA 2	0.986
SA 1	UK	0.958
SA 1	USA	0.983
SA 2	UK	0.962
SA 2	USA	0.988
UK	USA	0.960
Overall		0.500

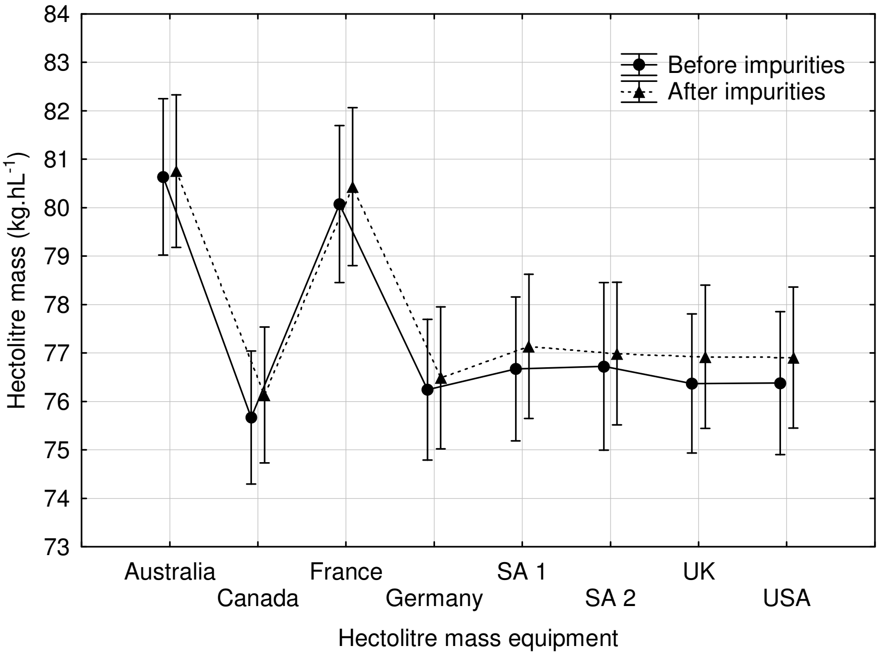

Figure 15 Average HLM values before and after the removal of impurities with a standard maize grading sieve. Error bars denote 0.95 confidence intervals.

Table 6 Hectolitre mass (HLM) values (mean ± standard error (se)) before and after the removal of impurities

HLM device	Impurities	Mean ± se	*n*
Australia	Before	80.64 ± 0.714	10
	After	80.75 ± 0.696	10
Canada	Before	75.67 ± 0.606	10
	After	76.13 ± 0.620	10
France	Before	80.07 ± 0.716	10
	After	80.43 ± 0.720	10
Germany	Before	76.24 ± 0.642	10
	After	76.49 ± 0.648	10
SA 1	Before	76.67 ± 0.656	10
	After	77.14 ± 0.658	10
SA 2	Before	76.72 ± 0.764	10
	After	76.99 ± 0.650	10
UK	Before	76.37 ± 0.635	10
	After	76.92 ± 0.654	10
USA	Before	76.38 ± 0.653	10
	After	76.90 ± 0.643	10

Discussion

The SA 1 and UK devices had the lowest within device repeatability of HLM values, and the devices from France and SA 2 the highest repeatability (Table 2). The presence of defective maize kernels and foreign matter in the samples may have had an influence on the repeatability, therefore the lack of similarity between the two South African devices. The differences might also have been a result of slight differences in the devices. Years of use may have altered the technical properties of the devices, resulting in differences in repeatability which might have had a bigger influence than the physical state of the samples. The results obtained from the French device were unreliable as the small opening in the funnel of the device caused blockage and had to be poked in order for the kernels to flow into the measuring container. This device was designed to determine HLM on small grains and is not really suitable to test HLM of maize samples, especially not South African maize known for its bigger kernels.

The respective HLM devices resulted in repeatable HLM results within each device (Fig. 7). The lack of variation within the devices is an indication of the efficient reduction of the bulk sample to sub-samples. Therefore, it can be deduced that the mixing and sub-sampling subjected to the bulk samples were successful in producing homogenous, well mixed and reduced sub-samples. The samples were mixed and divided with a Boerner Divider, which has been shown to be the most efficient apparatus in providing well reduced samples (Petersen *et al.*, 2004). Some sample variation will, however, always be present in maize samples due to the impurities present in the samples as HLM is usually determined on maize samples before removal of impurities.

Contrary to previous studies performed with wheat (Shuey, 1960), the maize samples did not show an increase in HLM with sample handling (Table 3). Shuey (1960) found that repeated handling of the wheat scours the bran coat, which leads to an increase in HLM values. Apart from one exception, obtained with the first South African device and sample 3, the HLM values of the respective maize samples did not increase with subsequent repetitions of the same sample as determined with the different HLM devices. The overall assumption can therefore be made that repeated measurements of the same sample would not have a significant effect on the HLM value thereof. Furthermore, the samples used in this study were received from maize mills where they underwent frequent handling and in addition they have been mixed and divided for this study. It can therefore be deducted that the samples underwent maximum change and could not have changed further during the execution of this study. Therefore, the differences observed between the measurements would most likely be due to the different devices and not due to the alteration of the sample when the tests were performed.

Significant ($P < 0.05$) differences were, however, observed between the HLM values obtained with some of the devices (Fig. 8). It was expected that the HLM values obtained with the French device would differ from the values obtained with the other devices because the funnel outlet was too small for the maize kernels to flow freely. Consequently the kernels had to be poked to fall into the measuring cylinder. A possible reason for the Australian device resulting in significantly higher results might have been due to its different way of filling the measuring cylinder. It has no designated flow "controller", i.e. a funnel or pre-filling container with a piston (weight) resulting in inconsistent packing of the maize kernels. In the case of the Canadian device it is likely that the height of the measuring container might have been responsible for the different HLM results obtained. The measuring container is shallow compared to the measuring containers of the other devices; therefore, less compaction of the maize kernels is obtained as opposed to the other devices. Another possible explanation for the differences between the devices could be due to the way the conversion factors were determined for each respective HLM device. The variation between repetitions (as denoted by large error bars) can be due to the various impurities present in the maize samples. Each of the sub-sub-samples could have contained different amounts and types (i.e. maize cob, broken kernels and leaves) of impurities.

Although differences were observed in the ICC agreement values (Fig. 9), indicating actual differences in the HLM values obtained with the respective HLM devices, the ICC consistency values were found to be high for all devices (Fig. 10). This is an indication that correction factors can be determined to convert between the HLM results obtained with the respective devices.

Further comparison of the devices, using a single work sample, indicated similar results as previously found in terms of absence of variation within the respective devices (Fig 11). The smaller error bars obtained indicated the reduction of variation within the samples due to using a single work sample. A disadvantage to using a single work sample was that the sample changed over time, resulting in small differences between the devices (Fig. 12) compared to when each device was tested with its own sub-sample (Fig. 8). The ICC agreement also revealed differences between the respective devices in terms of their actual HLM values (Fig. 13). Correlating the devices by means of the ICC consistency (Fig. 14) showed that the devices correlate well with each other.

A significant (P = 0.0324) increase in HLM values was found after the impurities have been removed from the respective maize samples (Fig. 15). Maize contains impurities that are often large and very light and therefore has a negative effect on the HLM. As a result some large impurities found in maize will decrease the HLM due to the larger size of the impurities that would take up more space, but would be lower in mass compared to the maize kernels. In South Africa HLM determinations are currently unofficially performed on maize samples from which the impurities are not removed. As the removal of impurities has a significant increase on the HLM values, it is advisable that the HLM measurements be performed on maize samples after the removal of impurities. An alternative could be to calculate correction factors to convert HLM values before and after removal of impurities as no device dependency was observed.

Conclusion

The following general conclusions can be made from this study:

- The removal of impurities with a standard South African maize grading sieve increased the HLM of maize significantly.
- The respective HLM devices all resulted in repeatable measurements within devices.
- The HLM obtained from the respective devices differed in actual HLM values.
- The HLM devices from the different countries, correlates well with one another.
- Correction factors could be applied to convert the HLM measurements obtained with one device to that of another.
- The devices from Canada, Germany, South Africa, UK and USA operated well with maize.

It is advisable that a device without a funnel is used to determine the HLM of maize to prevent that the large maize kernels block the funnel opening. The German device would be the most suitable device to use as it does not have a funnel and it is a very stable device which is less prone to jar when the test is performed. It is also compliant to the International Standard (ISO 7971-2:1995) and therefore, has an accurate and detailed description of its operational procedure. It would be of great value if research could be performed to determine if HLM values (specifically determined with the German device) correlate with the milling index of maize or other related maize quality parameters.

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CHAPTER 5

General discussion and conclusions

General discussion and conclusions

The wheat industry in South Africa strongly relies on hectolitre mass (HLM) as a guide to wheat grain quality. This is also the case in other grain producing and exporting countries. Either one of two types of HLM equipment are usually used. These devices consist of either a funnel and measuring cup of known volume or a cylindrical device (chondrometer) with a measuring cylinder of known volume underneath which is then filled with grain in a controlled manner. As to date no comprehensive study, to compare these respective devices, has been done. The HLM devices from Australia, Canada, France, Germany, United Kingdom (UK), the United States of America (USA) and South Africa were compared using mixed wheat, single South African cultivar as well as maize samples. A study which compared a 1 L HLM device (most commonly used in Europe) and a standard bushel device found high correlation coefficients between the HLM and bushel devices (Greenaway, *et al.*, 1977).

The respective HLM devices resulted in HLM values that were highly repeatable within each device. The less repeatable HLM values obtained within the Australian devices could be due to the way the device operates. The device do not allow for controlled flow of grain into the measuring container as is the case with the other devices.

An overall ICC agreement value of 0.523 was obtained between the respective devices when the tests were performed with the mixed samples. The overall ICC agreement between the devices increased to 0.762 when the tests were performed with the single cultivar wheat samples. This was expected as the single cultivars consists of only one cultivar and less sample variation is present as compared to the mixed wheat where more than one cultivar were often present and more variation could have been expected. The ICC consistency revealed that the devices were highly correlated and this suggested, therefore, that correction factors could be developed that would allow conversion of HLM values between devices.

When ten South African HLM devices were compared, significant ($P < 0.05$) differences were found between the devices. These differences would, however, not be significant in practice as indicated by the high ICC agreement (0.975) and consistency (0.993) values. Some of the devices were in bad overall condition and the results might have been more comparable if the devices were in a better working condition. It is advised that regular maintenance be performed on the HLM devices. It should also be ensured that the correct wooden scraper is used when executing the test. It is also advised that HLM tests be performed in an environment free from vibrations as this could lead to compaction of the grain and therefore incorrect results.

It has been observed that the removal of impurities from the wheat resulted in a significant ($P < 0.05$) increase in HLM values ranging from *ca.* 0.6-1.1 $kg.hL^{-1}$ depending on the device. The increase in HLM values was significantly different ($P < 0.05$) for the respective devices indicating significant device dependency. The results obtained from the two respective South African devices were similar. A previous study showed an average increase of 2.07 $kg.hL^{-1}$ in HLM values after the dockage had been removed from a sample of wheat, using a bushel weight device (Greenaway *et al.*, 1971). The results obtained in the current study using a bushel device (USA device) delivered about half the increase in HLM compared to results obtained by Greenaway *et al.* (1971). The method of dockage removal as used by Greenaway *et al.* (1971) might have been different from the method used in this study and therefore likely to be the reason for the different results obtained.

The investigation of the effect of operators on HLM values revealed significant differences between operators. In an earlier study, significant differences were also found between operators, one was less skilled and experienced than the other, for five grains tested (Greenaway *et al.*, 1971). It was pointed out by Greenaway *et al.* (1971) that there was place for improvement in the results if one or both of the operators' techniques were to improve. The training of operators is necessary in order to obtain consistent HLM values that portray the real quality of the grain tested.

The mean HLM values of the samples that have been conditioned to higher moisture contents showed a remarkable decrease in HLM values as the moisture content increased. This confirms similar results from previous studies which also showed that HLM values decreased as moisture contents increased (Lockwood, 1960; Pushman, 1975; Hook, 1984). The lower HLM values obtained after wetting reflected the poor packing efficiency and lower density of the wetted wheat, which was also pointed out by Pushman (1975). Pushman (1975) observed that the swelling of the kernel and the roughening of the bran coat might also be responsible for the decrease in HLM values. The fact that the density of the dry wheat (starch) is 1.51 $g.cm^3$ as opposed to that of 1 $g.cm^3$ of water may also have resulted in the wetter wheat being lighter and in the lower HLM values compared to the dry wheat as discussed by Hlynka & Bushuk (1959). However, it has been observed earlier that changes in surface friction played a greater role in altering HLM values than changes in grain density (Scott, 1951). Samples that were wetted, than dried and wetted again to their moisture contents of the first respective wettings did not reach the HLM values obtained after the first wetting. It is thus clear that wet and dry cycles change the integrity and density of the wheat and the HLM of the initial sample will never be obtained once the sample has been wetted or dried. Correction factors cannot be applied to compensate for change in moisture content as it has been noticed that different wheat samples responded differently to an increase or decrease in moisture content.

The evaluation of the respective devices using maize samples showed that the devices from Australia and France delivered HLM values significantly ($P < 0.05$) higher compared and the device from Canada HLM values significantly ($P < 0.05$) lower than the other devices. The reason for the differences in HLM values obtained with the Australian device could again have been due the reason discussed earlier. The French device, which resulted in highly repeatable HLM values when tested with wheat, was unsuitable for HLM determinations with maize. The small opening of the funnel through which the samples flows into the measuring cup did not allow the larger maize kernels to flow freely. The kernels had to be poked in order for them to flow into the measuring cup leading to erroneous results. The shallow measuring cup of the Canadian device might be responsible for the lower HLM values obtained with the bigger maize kernels. The ICC consistency (>0.97) however showed that the devices were all highly correlated and that correction factors could be developed to convert the HLM results of one device to that of another, as was the case for wheat.

An alternative to the use of correction factors could be the replacement of the South African device with the German device for both wheat and maize. The German device is accompanied with a secondary filling beaker to fill the device with and a convenient conversion table is supplied with the device. More importantly is a very stable device and less prone to jar like the other devices did. The American bushel device also worked well with wheat and maize, but the measurements are in pounds per bushel and must therefore be converted to the metric kilogram per hectolitre, unless an automatic conversion balance is used.

The removal of impurities from maize resulted in a significant (P = 0.0324) increase of the HLM values and it is advised that the HLM for maize is determined after the removal of impurities. An alternative could be to calculate correction factors to convert HLM values before and after removal of impurities as no device dependency was observed.

In spite of the controversy regarding the relationship between HLM of grain and milling performance, HLM determination is firmly established as a quality criterion of grains and will be used as a quality indicator and remain a major pricing factor in future.

Results obtained in this study confirmed the benefit of HLM determination for both the wheat and maize industries and encourage further research. Research to determine the effect of wetting and drying on HLM values of single wheat cultivars would be advantageous to re-evaluate the use of conversion factors. The effect of wetting after drying and drying after wetting on HLM values will also be useful to determine which of the two scenarios have the greatest effect on HLM determinations. The incorporation of HLM determination into the South African maize grading system is a strong possibility and future studies could include the evaluation of the relationship between the milling index of maize and HLM.

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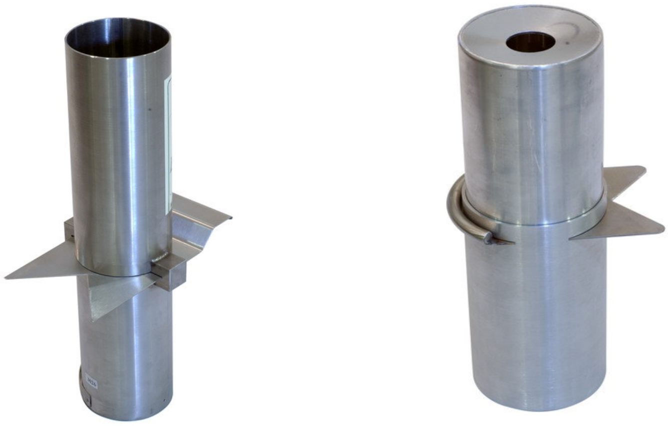
APPENDICES



Appendix 1

Table 1 The South African bread wheat grading table (2006/2007)

	Minimum			Maximum permissible percentage deviations (m/m)									
				A	B	C	D	E	F	G	H	I	J
Grade	**Hectoliter mass ($kg.hl^{-1}$)**	**Falling number (seconds)**	**Protein content (%)**	**Heavily frost damaged kernels**	**Field mould infected kernels**	**Storage mould infected kernels**	**Screenings**	**Other grain and unthreshed ears**	**Gravel, stones, turf and glass**	**Foreign matter + F**	**Heat damaged kernels**	**Damaged kernels + H**	**Combined deviations (D+E+G+I)**
Grade 1	77	220	12	5	2	0.5	3	1	0.5	1	0.5	2	5
Grade 2	76	220	11	5	2	0.5	3	1	0.5	1	0.5	2	5
Grade 3	74	220	10	5	2	0.5	3	1	0.5	1	0.5	2	5
Grade 4	72	200	9	5	2	0.5	3	1	0.5	1	0.5	2	5
Utility grade	70	150	8	10	2	0.5	10	4	0.5	3	0.5	5	10
Class other wheat	<70	<150	<8	>10	>2	>0.5	>10	>4	>0.5	>3	>0.5	>5	>10
Sample size (min)	1kg	300 g cleaned wheat	Device instruction	25 g screened	25 g screened	25 g screened	500 g unscreened	50 g screened	100 g screened	100 g screened	100 g screened	25 g screened	–

Adapted from source: Anon. (1998a)

Appendix 2

Table 2 The South African maize grading table

Deviation	Maximum permissible deviation (%)					
	White maize			Yellow maize		
	WM1	WM2	WM3	YM1	YM2	YM3
1. Foreign matter	0.3	0.5	0.75	0.3	0.5	0.75
2. Defective maize kernels above and below the 6.35 mm round-hole sieve	7	13	30	*	*	*
3. Defective maize kernels that can pass through the 6.35 mm round-hole sieve	*	*	*	4	10	30
4. Defective maize kernels that cannot pass through the 6.35 mm round-hole sieve	*	*	*	9	20	30
5. Other colour maize kernels	3	6	10	2	5	5
6. Deviations referred to in 1, 2, 3, 4 and 5 collectively: Provided that the deviations are individually within the specified limits	8	16	30	9	20	30
7. Pink maize kernels	12	12	12	12	12	12

* Not specified

Adapted from source: Anon. (1998b)

Appendix 3

Table 3 Gram to hectolitre mass conversion chart for wheat of the Ohaus 500 mL measure and Cox funnel*

g.0.5 L^{-1}	kg.hL^{-1}	g.0.5 L^{-1}	kg.hL^{-1}
340	70.0	378	77.5
341	70.1	379	77.7
342	70.3	380	78.0
343	70.5	381	78.1
344	70.7	382	78.3
345	71.0	383	78.5
346	71.1	384	78.7
347	71.3	385	79.0
348	71.5	386	79.1
349	71.7	387	79.3
350	72.0	388	79.5
351	72.1	389	79.7
352	72.3	390	80.0
353	72.5	391	80.1
354	72.7	392	80.3
355	73.0	393	80.5
356	73.1	394	80.7
357	73.3	395	81.0
358	73.5	396	81.1
359	73.7	397	81.3
360	74.0	398	81.5
361	74.1	399	81.7
362	74.3	400	82.0
363	74.5	401	82.1
364	74.7	402	82.3
365	75.0	403	82.5
366	75.1	404	82.7
367	75.3	405	83.0
368	75.5	406	83.1
369	75.7	407	83.3
370	76.0	408	83.5
371	76.1	409	83.7
372	76.3	410	84.0
373	76.5	411	84.1
374	76.7	412	84.3
375	77.0	413	84.5
376	77.1	414	84.7
377	77.3	415	85.0

*To convert gram to kg.hL^{-1} for maize the following equation can be used: gram/500 mL measuring container x 0.191932 + 2.13.

Appendix 4

Table 4 Gram to hectolitre mass conversion chart of the Kern 220/222 Grain Sampler

$g.L^{-1}$	$kg.hL^{-1}$	$g.L^{-1}$	$kg.hL^{-1}$	$g.L^{-1}$	$kg.hL^{-1}$	$g.L^{-1}$	$kg.hL^{-1}$
694	70.07	734	74.08	774	78.08	814	82.09
695	70.17	735	74.18	775	78.19	815	82.19
696	70.27	736	74.28	776	78.29	816	82.29
697	70.37	737	74.38	777	78.39	817	82.39
698	70.47	738	74.48	778	78.49	818	82.49
699	70.57	739	74.58	779	78.59	819	82.59
700	70.67	740	74.68	780	78.69	820	82.69
701	70.77	741	74.78	781	78.79	821	82.79
702	70.87	742	74.88	782	78.89	822	82.89
703	70.97	743	74.98	783	78.99	823	82.99
704	71.07	744	75.08	784	79.09	824	83.09
705	71.17	745	75.18	785	79.19	825	83.20
706	71.27	746	75.28	786	79.29	826	83.30
707	71.37	747	75.38	787	79.39	827	83.40
708	71.47	748	75.48	788	79.49	828	83.50
709	71.57	749	75.58	789	79.59	829	83.60
710	71.67	750	75.68	790	79.69	830	83.70
711	71.77	751	75.78	791	79.79	831	83.80
712	71.87	752	75.88	792	79.89	832	83.90
713	71.97	753	75.98	793	79.99	833	84.00
714	72.07	754	76.08	794	80.09	834	84.10
715	72.17	755	76.18	795	80.19	835	84.20
716	72.27	756	76.28	796	80.29	836	84.30
717	72.37	757	76.38	797	80.39	837	84.40
718	72.47	758	76.48	798	80.49	838	84.50
719	72.57	759	76.58	799	80.59	839	84.60
720	72.67	760	76.68	800	80.69	840	84.70
721	72.77	761	76.78	801	80.79	841	84.80
722	72.87	762	76.88	802	80.89	842	84.90
723	72.97	763	76.98	803	80.99	843	85.00
724	73.07	764	77.08	804	81.09	-	-
725	73.18	765	77.18	805	81.19	-	-
726	73.28	766	77.28	806	81.29	-	-
727	73.38	767	77.38	807	81.39	-	-
728	73.48	768	77.48	808	81.49	-	-
729	73.58	769	77.58	809	81.59	-	-
730	73.68	770	77.68	810	81.69	-	-
731	73.78	771	77.78	811	81.79	-	-
732	73.88	772	77.88	812	81.89	-	-
733	73.98	773	77.98	813	81.99	-	-

Appendix 5

Table 5 Gram to hectolitre mass conversion chart of the Easi-Way Portable Hectolitre mass Test Weight Kit

Gram	$kg.hL^{-1}$
346	70.0
348	70.4
350	70.8
352	71.2
354	71.6
356	72.0
358	72.4
360	72.8
362	73.2
364	73.6
366	74.0
368	74.4
370	74.8
372	75.2
374	75.6
376	76.0
378	76.4
380	76.8
382	77.2
384	77.6
386	78.0
388	78.4
390	78.8
392	79.2
394	79.6
396	80.0
398	80.4
400	80.8
402	81.2
404	81.6
406	82.0
408	82.4
410	82.8
412	83.2
414	83.6
416	84.0
418	84.4
420	84.8
422	85.2

Appendix 6

Table 6 Gram to test weight (lb.bu^{-1})* conversion chart of the Seedburo 151 Filling Hopper with quart cup

Gram	lb.bu^{-1}	Gram	lb.bu^{-1}	Gram	lb.bu^{-1}	Gram	lb.bu^{-1}
758	53.5	778	54.9	798	56.3	818	57.7
758.5	53.5	778.5	54.9	798.5	56.3	818.5	57.7
759	53.5	779	55.0	799	56.4	819	57.8
759.5	53.6	779.5	55.0	799.5	56.4	819.5	57.8
760	53.6	780	55.0	800	56.4	820	57.8
760.5	53.7	780.5	55.1	800.5	56.5	820.5	57.9
761	53.7	781	55.1	801	56.5	821	57.9
761.5	53.7	781.5	55.1	801.5	56.5	821.5	58.0
762	53.8	782	55.2	802	56.6	822	58.0
762.5	53.8	782.5	55.2	802.5	56.6	822.5	58.0
763	53.8	783	55.2	803	56.6	823	58.1
763.5	53.9	783.5	55.3	803.5	56.7	823.5	58.1
764	53.9	784	55.3	804	56.7	824	58.1
764.5	53.9	784.5	55.3	804.5	56.8	824.5	58.2
765	54.0	785	55.4	805	56.8	825	58.2
765.5	54.0	785.5	55.4	805.5	56.8	825.5	58.2
766	54.0	786	55.5	806	56.9	826	58.3
766.5	54.1	786.5	55.5	806.5	56.9	826.5	58.3
767	54.1	787	55.5	807	56.9	827	58.3
767.5	54.1	787.5	55.6	807.5	75.0	827.5	58.4
768	54.2	788	55.6	808	75.0	828	58.4
768.5	54.2	788.5	55.6	808.5	75.0	828.5	58.4
769	54.3	789	55.7	809	57.1	829	58.5
769.5	54.3	789.5	55.7	809.5	57.1	829.5	58.5
770	54.3	790	55.7	810	57.1	830	58.6
770.5	54.4	790.5	55.8	810.5	57.2	830.5	58.6
771	54.4	791	55.8	811	57.2	831	58.6
771.5	54.4	791.5	55.8	811.5	57.2	831.5	58.7
772	54.5	792	55.9	812	57.3	832	58.7
772.5	54.5	792.5	55.9	812.5	57.3	832.5	58.7
773	54.5	793	55.9	813	57.4	833	58.8
773.5	54.6	793.5	56.0	813.5	57.4	833.5	58.8
774	54.6	794	56.0	814	57.4	834	58.8
774.5	54.6	794.5	56.1	814.5	57.5	834.5	58.9
775	54.7	795	56.1	815	57.5	835	58.9
775.5	54.7	795.5	56.1	815.5	57.5	835.5	58.9
776	54.7	796	56.2	816	57.6	836	59.0
776.5	54.8	796.5	56.2	816.5	57.6	836.5	59.0
777	54.8	797	56.2	817	57.6	837	59.0
777.5	54.8	797.5	56.3	817.5	57.7	837.5	59.1

Appendix 6 continue

Gram	$lb.bu^{-1}$	Gram	$lb.bu^{-1}$	Gram	$lb.bu^{-1}$	Gram	$lb.bu^{-1}$
838	59.1	858	60.5	878	61.9	898	63.4
838.5	59.2	858.5	60.6	878.5	62.0	898.5	63.4
839	59.2	859	60.6	879	62.0	899	63.4
839.5	59.2	859.5	60.6	879.5	62.0	899.5	63.5
840	59.3	860	60.7	880	62.1	900	63.5
840.5	59.3	860.5	60.7	880.5	62.1	900.5	63.5
841	59.3	861	60.7	881	62.2	901	63.6
841.5	59.4	861.5	60.8	881.5	62.2	901.5	63.6
842	59.4	862	60.8	882	62.2	902	63.6
842.5	59.4	862.5	60.8	882.5	62.3	902.5	63.7
843	59.5	863	60.9	883	62.3	903	63.7
843.5	59.5	863.5	60.9	883.5	62.3	903.5	63.7
844	59.5	864	61.0	884	62.4	904	63.8
844.5	59.6	864.5	61.0	884.5	62.4	904.5	63.8
845	59.6	865	61.0	885	62.4	905	63.8
845.5	59.6	865.5	61.1	885.5	62.5	905.5	63.9
846	59.7	866	61.1	886	62.5	906	63.9
846.5	59.7	866.5	61.1	886.5	62.5	906.5	64.0
847	59.8	867	61.2	887	62.6	907	64.0
847.5	59.8	867.5	61.2	887.5	62.6	907.5	64.0
848	59.8	868	61.2	888	62.6	908	64.1
848.5	59.9	868.5	61.3	888.5	62.7	908.5	64.1
849	59.9	869	61.3	889	62.7	909	64.1
849.5	59.9	869.5	61.3	889.5	62.8	909.5	64.2
850	60.0	870	61.4	890	62.8	910	64.2
850.5	60.0	870.5	61.4	890.5	62.8	910.5	64.2
851	60.0	871	61.4	891	62.9	911	64.3
851.5	60.1	871.5	61.5	891.5	62.9	911.5	64.3
852	60.1	872	61.5	892	62.9	912	64.3
852.5	60.1	872.5	61.6	892.5	63.0	912.5	64.4
853	60.2	873	61.6	893	63.0	913	64.4
853.5	60.2	873.5	61.6	893.5	63.0	913.5	64.4
854	60.2	874	61.7	894	63.1	914	64.5
854.5	60.3	874.5	61.7	894.5	63.1	914.5	64.5
855	60.3	875	61.7	895	63.1	915	64.6
855.5	60.4	875.5	61.8	895.5	63.2	915.5	64.6
856	60.4	876	61.8	896	63.2	916	64.6
856.5	60.4	876.5	61.8	896.5	63.2	916.5	64.7
857	60.5	877	61.9	897	63.3	917	64.7
857.5	60.5	877.5	61.9	897.5	63.3	917.5	64.7

* To convert test weight ($lb.bu^{-1}$) to hectolitre mass ($kg.hL^{-1}$) the following formulas must be used for all types of wheat except durum wheat: ($lb.bu^{-1}$ x 1.292) + 1.419 and for all other grains: $lb.bu^{-1}$ x 1.287.

Appendix 7

Table 7.1 Experiment 1: repeatability within the respective hectolitre mass (HLM) devices

Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
81.90	81.20	80.50	80.29	79.06	79.34	80.60	80.23
81.07	81.20	80.35	80.59	79.39	78.64	80.60	79.97
80.87	81.60	80.46	80.69	79.17	78.18	80.60	80.10
82.56	81.40	80.59	80.69	79.42	78.86	80.80	80.23
82.07	81.80	80.43	80.69	79.34	78.52	80.80	80.36
82.28	81.20	80.46	80.79	79.46	78.62	80.60	80.23
81.93	81.40	80.32	80.69	79.29	78.76	81.20	80.10
82.60	81.60	80.30	80.69	78.98	78.66	81.00	80.23
82.03	81.60	80.57	80.79	79.43	78.74	81.40	80.36
82.19	81.40	80.25	80.79	79.35	78.58	80.80	80.23

Table 7.2 Experiment 2: effect of repeated analysis of the same wheat sample on its hectolitre mass (HLM)

			Sample 11				
Australia	**Canada**	**France**	**Germany**	**SA 1**	**SA 2**	**UK**	**USA**
86.16	84.80	84.03	84.10	82.17	82.30	85.60	83.72
85.34	84.40	84.31	83.90	82.08	82.56	85.40	83.85
86.39	84.40	84.19	83.70	82.05	82.46	85.60	83.85
85.97	84.40	84.21	83.90	81.94	82.82	86.20	83.72
85.38	84.20	84.13	83.80	82.20	82.50	85.80	83.85
85.82	84.20	84.05	83.80	82.15	82.84	85.60	83.72
85.38	84.80	84.08	83.70	82.20	82.44	85.80	83.72
85.51	84.60	83.87	83.80	81.98	82.52	85.80	83.72
85.52	84.60	84.31	83.80	82.17	82.44	85.60	83.72
85.32	84.60	84.02	83.70	82.19	82.44	85.40	83.85
			Sample 8				
84.02	81.00	80.79	81.09	78.92	79.06	82.60	80.62
83.42	81.00	80.69	80.99	78.93	79.38	82.20	80.62
83.50	81.00	80.73	80.99	78.96	78.96	82.80	80.75
83.73	80.70	80.83	80.99	79.02	79.04	82.80	80.62
84.51	80.70	80.77	81.19	78.92	79.28	82.80	80.62
83.22	80.50	80.63	81.09	79.38	79.12	81.80	80.62
82.39	80.70	80.78	81.09	79.27	79.28	82.20	80.75
83.95	80.70	80.77	80.99	78.89	79.30	82.40	80.62
83.90	80.70	80.65	81.09	79.03	79.08	82.20	80.62
83.51	80.70	80.73	81.09	78.89	79.28	82.40	80.62
			Sample 13				
78.97	77.10	76.82	77.28	75.25	75.32	77.20	77.39
78.90	77.30	77.01	77.28	75.25	75.30	77.60	77.39
80.21	77.50	77.05	77.38	75.37	75.38	77.60	77.39
78.54	77.00	76.98	77.48	75.29	75.30	77.80	77.26
79.21	77.00	76.88	77.38	75.33	75.46	77.60	77.26
80.14	77.50	77.07	77.68	75.24	75.40	77.60	77.26
78.80	77.50	76.86	77.38	75.38	75.40	77.40	77.13
79.22	77.30	77.05	77.38	75.28	75.55	77.60	77.39
79.00	77.30	77.09	77.38	75.31	75.52	77.40	77.26
79.02	77.30	76.94	77.48	75.35	75.68	77.60	77.39

Table 7.3.1 Experiment 3a: determination of variation in hectolitre mass (HLM) within and between HLM devices using mixed wheat samples

Sample	Sub-	Rep	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
1	a	1	85.21	81.80	81.18	81.49	79.19	79.10	81.00	80.75
	a	2	84.09	81.40	81.31	81.19	79.14	79.22	80.80	81.01
	b	1	83.92	81.20	81.16	81.19	79.56	79.18	81.60	80.75
	b	2	83.40	81.20	81.42	80.99	79.35	79.44	81.20	80.75
	c	1	83.62	81.40	81.05	81.29	79.23	79.30	80.80	80.88
	c	2	83.90	81.40	81.08	81.39	79.17	79.48	81.40	81.01
2	a	1	85.60	83.60	82.79	83.80	81.20	81.24	83.60	82.82
	a	2	85.26	83.40	83.06	83.70	81.28	81.51	83.40	82.56
	b	1	84.80	84.00	82.85	83.60	81.37	81.40	83.40	82.69
	b	2	85.37	83.60	83.18	83.70	81.18	81.27	83.20	82.69
	c	1	84.99	83.60	82.95	83.60	81.15	81.55	83.80	82.69
	c	2	85.48	83.60	83.01	83.60	81.24	81.27	83.60	82.82
3	a	1	82.56	80.70	80.65	80.99	79.32	79.02	81.60	81.01
	a	2	83.06	80.70	80.73	81.09	79.34	79.06	81.60	81.14
	b	1	83.05	81.20	81.05	80.89	79.24	79.18	81.80	80.88
	b	2	82.49	81.20	81.07	81.09	79.04	79.19	81.40	81.01
	c	1	83.37	81.20	80.71	80.59	78.97	79.61	81.60	80.75
	c	2	83.57	81.40	80.77	80.69	78.85	79.41	81.20	80.75
4	a	1	85.92	83.00	82.28	82.39	80.76	79.86	82.60	82.30
	a	2	86.20	83.00	81.85	81.79	80.38	80.03	82.80	82.04
	b	1	85.01	83.00	82.31	82.19	80.92	79.89	82.60	82.04
	b	2	85.69	82.80	82.57	81.79	80.39	80.12	82.20	81.91
	c	1	85.28	82.60	82.02	82.19	80.87	80.86	83.00	82.04
	c	2	86.18	82.60	81.77	81.89	80.43	80.40	82.40	81.78
5	a	1	84.87	83.20	82.99	82.59	80.53	80.62	82.20	81.91
	a	2	84.34	83.40	83.11	82.69	80.55	80.74	82.40	82.04
	b	1	85.25	83.40	83.28	82.69	80.50	80.64	82.20	82.30
	b	2	84.71	83.00	82.79	82.89	80.52	80.48	82.40	82.30
	c	1	84.35	83.60	82.80	82.59	80.63	80.88	82.40	82.30
	c	2	85.19	83.20	82.76	82.69	80.62	80.78	82.40	82.30

Table 7.3.1 continued

7	a	1	86.65	81.00	82.46	82.29	80.05	79.68	82.20	80.23
	a	2	86.42	81.80	82.20	83.30	79.73	79.84	81.00	80.62
	b	1	86.39	81.80	82.45	82.19	80.36	79.99	82.80	81.01
	b	2	86.72	81.80	82.63	82.29	79.63	79.63	82.40	80.62
	c	1	86.18	83.00	80.49	81.69	79.50	80.52	82.80	81.14
	c	2	85.35	82.20	80.58	81.39	79.39	80.30	81.20	80.75
8	a	1	82.89	81.40	81.24	81.29	78.55	79.25	81.20	80.75
	a	2	82.65	81.20	81.25	81.19	78.41	79.02	81.60	80.75
	b	1	81.76	81.60	81.19	81.19	78.94	79.40	81.60	80.75
	b	2	83.16	81.60	81.16	81.19	78.94	79.33	81.60	80.75
	c	1	82.80	81.40	81.02	81.09	78.95	79.40	81.40	80.62
	c	2	82.70	81.00	81.07	80.99	79.05	79.26	81.20	80.49
10	a	1	81.33	79.00	78.48	78.99	76.07	76.14	79.00	77.91
	a	2	81.74	78.70	78.59	78.89	76.16	76.16	78.40	77.91
	b	1	80.89	78.70	78.37	79.09	75.93	76.16	79.00	78.03
	b	2	81.09	79.00	78.38	78.79	76.08	76.32	78.40	77.78
	c	1	80.73	79.00	78.86	78.79	76.18	76.58	78.80	78.03
	c	2	80.68	79.00	78.74	78.89	75.91	76.18	78.60	78.03
11	a	1	86.30	83.60	83.84	83.09	81.66	81.66	84.40	83.72
	a	2	86.45	83.60	83.87	83.20	81.78	81.76	84.40	83.85
	b	1	87.26	83.20	83.59	83.20	81.53	81.52	84.20	83.59
	b	2	86.89	83.60	83.70	83.40	81.57	81.70	84.00	83.46
	c	1	86.69	83.20	83.88	82.79	81.66	81.62	84.60	82.94
	c	2	87.01	83.20	83.77	82.89	81.34	81.70	84.80	82.82
12	a	1	82.08	79.70	79.10	80.29	77.64	77.71	80.00	79.07
	a	2	82.30	80.00	79.05	80.19	77.31	77.76	79.60	79.33
	b	1	81.67	79.70	79.43	79.99	77.66	77.76	80.20	79.20
	b	2	82.41	79.50	79.23	80.29	77.29	77.41	80.00	79.20
	c	1	82.47	80.00	79.49	80.09	77.25	77.25	80.00	78.94
	c	2	82.58	79.70	79.70	80.09	77.26	77.44	80.20	79.07

Table 7.3.1.1 Experiment 3a: Moisture and protein analyses on mixed wheat sub-sub samples

Sample	Cultivar/Origin	Device	Sub	Rep	Moisture	Protein	Sample	Cultivar/Origin	Device	Sub	Rep	Moisture	Protein
1	Argentina	Australia	a	1	12.5	12.4	2	Argentina	Australia	a	1	12.0	11.3
1	Argentina	Australia	a	2	12.5		2	Argentina	Australia	a	2	11.9	
1	Argentina	Australia	b	1	12.5	12.6	2	Argentina	Australia	b	1	11.9	11.4
1	Argentina	Australia	b	2	12.5		2	Argentina	Australia	b	2	12.0	
1	Argentina	Australia	c	1	12.5	12.5	2	Argentina	Australia	c	1	11.9	11.1
1	Argentina	Australia	c	2	12.5		2	Argentina	Australia	c	2	11.8	
1	Argentina	Germany	a	1	12.4	12.7	2	Argentina	Germany	a	1	11.9	11.5
1	Argentina	Germany	a	2	12.4		2	Argentina	Germany	a	2	11.9	
1	Argentina	Germany	b	1	12.4	12.3	2	Argentina	Germany	b	1	11.9	11.4
1	Argentina	Germany	b	2	12.4		2	Argentina	Germany	b	2	11.9	
1	Argentina	Germany	c	1	12.3	12.6	2	Argentina	Germany	c	1	11.8	11.5
1	Argentina	Germany	c	2	12.3		2	Argentina	Germany	c	2	11.8	
1	Argentina	Canada	a	1	12.7	12.4	2	Argentina	Canada	a	1	11.9	11.4
1	Argentina	Canada	a	2	12.6		2	Argentina	Canada	a	2	11.9	
1	Argentina	Canada	b	1	12.6	12.6	2	Argentina	Canada	b	1	11.9	11.3
1	Argentina	Canada	b	2	12.6		2	Argentina	Canada	b	2	11.8	
1	Argentina	Canada	c	1	12.6	12.3	2	Argentina	Canada	c	1	11.9	11.4
1	Argentina	Canada	c	2	12.6		2	Argentina	Canada	c	2	11.9	
1	Argentina	SA 1	a	1	12.7	12.4	2	Argentina	SA 1	a	1	11.9	11.2
1	Argentina	SA 1	a	2	12.6		2	Argentina	SA 1	a	2	11.9	
1	Argentina	SA 1	b	1	12.6	12.4	2	Argentina	SA 1	b	1	12.0	11.2
1	Argentina	SA 1	b	2	12.5		2	Argentina	SA 1	b	2	11.9	
1	Argentina	SA 1	c	1	12.5	12.4	2	Argentina	SA 1	c	1	11.9	11.4
1	Argentina	SA 1	c	2	12.5		2	Argentina	SA 1	c	2	11.8	
1	Argentina	France	a	1	12.5	12.4	2	Argentina	France	a	1	11.9	11.5
1	Argentina	France	a	2	12.5		2	Argentina	France	a	2	11.9	
1	Argentina	France	b	1	12.5	12.6	2	Argentina	France	b	1	12.0	11.4
1	Argentina	France	b	2	12.4		2	Argentina	France	b	2	11.9	
1	Argentina	France	c	1	12.5	12.5	2	Argentina	France	c	1	12.0	11.5
1	Argentina	France	c	2	12.5		2	Argentina	France	c	2	11.9	
1	Argentina	USA	a	1	12.3	12.4	2	Argentina	USA	a	1	12.1	11.5
1	Argentina	USA	a	2	12.3		2	Argentina	USA	a	2	12.0	
1	Argentina	USA	b	1	12.3	12.4	2	Argentina	USA	b	1	12.0	11.5
1	Argentina	USA	b	2	12.3		2	Argentina	USA	b	2	12.0	
1	Argentina	USA	c	1	12.3	12.4	2	Argentina	USA	c	1	12.0	11.4
1	Argentina	USA	c	2	12.3		2	Argentina	USA	c	2	12.0	
1	Argentina	UK	a	1	12.5	12.5	2	Argentina	UK	a	1	11.9	11.4
1	Argentina	UK	a	2	12.4		2	Argentina	UK	a	2	11.9	
1	Argentina	UK	b	1	12.4	12.5	2	Argentina	UK	b	1	12.0	11.3
1	Argentina	UK	b	2	12.4		2	Argentina	UK	b	2	11.9	

Table 7.3.1.1 continued

1	Argentina	UK	c	1	12.4	12.6	2	Argentina	UK	c	1	11.9	11.4
1	Argentina	UK	c	2	12.4		2	Argentina	UK	c	2	11.9	
1	Argentina	SA 2	a	1	12.5	12.4	2	Argentina	SA 2	a	1	12.0	11.5
1	Argentina	SA 2	a	2	12.5		2	Argentina	SA 2	a	2	12.0	
1	Argentina	SA 2	b	1	12.6	12.3	2	Argentina	SA 2	b	1	12.0	11.6
1	Argentina	SA 2	b	2	12.5		2	Argentina	SA 2	b	2	12.0	
1	Argentina	SA 2	c	1	12.6	12.3	2	Argentina	SA 2	c	1	11.8	11.5
1	Argentina	SA 2	c	2	12.6		2	Argentina	SA 2	c	2	11.9	
3	USA Soft	Australia	a	1	12.4	10.4	4	USA DNS	Australia	a	1	12.8	12.8
3	USA Soft	Australia	a	2	12.5		4	USA DNS	Australia	a	2	12.8	
3	USA Soft	Australia	b	1	12.4	10.5	4	USA DNS	Australia	b	1	12.8	12.7
3	USA Soft	Australia	b	2	12.4		4	USA DNS	Australia	b	2	12.7	
3	USA Soft	Australia	c	1	12.4	10.4	4	USA DNS	Australia	c	1	12.8	12.4
3	USA Soft	Australia	c	2	12.3		4	USA DNS	Australia	c	2	12.8	
3	USA Soft	Germany	a	1	12.4	11.0	4	USA DNS	Germany	a	1	12.7	12.4
3	USA Soft	Germany	a	2	12.4		4	USA DNS	Germany	a	2	12.7	
3	USA Soft	Germany	b	1	12.4	10.8	4	USA DNS	Germany	b	1	12.9	12.3
3	USA Soft	Germany	b	2	12.4		4	USA DNS	Germany	b	2	12.8	
3	USA Soft	Germany	c	1	12.5	10.8	4	USA DNS	Germany	c	1	12.8	12.3
3	USA Soft	Germany	c	2	12.5		4	USA DNS	Germany	c	2	12.8	
3	USA Soft	Canada	a	1	12.5	10.9	4	USA DNS	Canada	a	1	12.9	12.6
3	USA Soft	Canada	a	2	12.4		4	USA DNS	Canada	a	2	12.8	
3	USA Soft	Canada	b	1	12.4	10.9	4	USA DNS	Canada	b	1	12.8	12.4
3	USA Soft	Canada	b	2	12.4		4	USA DNS	Canada	b	2	12.8	
3	USA Soft	Canada	c	1	12.4	11.0	4	USA DNS	Canada	c	1	12.8	12.4
3	USA Soft	Canada	c	2	12.4		4	USA DNS	Canada	c	2	12.9	
3	USA Soft	SA 1	a	1	12.5	11.2	4	USA DNS	SA 1	a	1	12.8	12.6
3	USA Soft	SA 1	a	2	12.5		4	USA DNS	SA 1	a	2	12.8	
3	USA Soft	SA 1	b	1	12.5	11.1	4	USA DNS	SA 1	b	1	12.9	12.6
3	USA Soft	SA 1	b	2	12.5		4	USA DNS	SA 1	b	2	12.9	
3	USA Soft	SA 1	c	1	12.4	10.9	4	USA DNS	SA 1	c	1	12.9	12.5
3	USA Soft	SA 1	c	2	12.4		4	USA DNS	SA 1	c	2	12.8	
3	USA Soft	France	a	1	12.4	10.8	4	USA DNS	France	a	1	12.8	12.3
3	USA Soft	France	a	2	12.4		4	USA DNS	France	a	2	12.8	
3	USA Soft	France	b	1	12.5	10.9	4	USA DNS	France	b	1	12.8	12.3
3	USA Soft	France	b	2	12.3		4	USA DNS	France	b	2	12.8	
3	USA Soft	France	c	1	12.4	11.0	4	USA DNS	France	c	1	12.8	12.2
3	USA Soft	France	c	2	12.4		4	USA DNS	France	c	2	12.6	
3	USA Soft	USA	a	1	12.5	11.2	4	USA DNS	USA	a	1	12.9	13.1
3	USA Soft	USA	a	2	12.4		4	USA DNS	USA	a	2	12.9	
3	USA Soft	USA	b	1	12.5	11.2	4	USA DNS	USA	b	1	12.8	13.0

Table 7.3.1.1 continued

3	USA Soft	USA	b	2	12.4		4	USA DNS	USA	b	2	12.6	
3	USA Soft	USA	c	1	12.4	11.1	4	USA DNS	USA	c	1	12.9	12.9
3	USA Soft	USA	c	2	12.4		4	USA DNS	USA	c	2	12.8	
3	USA Soft	UK	a	1	12.5	11.1	4	USA DNS	UK	a	1	12.9	12.8
3	USA Soft	UK	a	2	12.4		4	USA DNS	UK	a	2	12.8	
3	USA Soft	UK	b	1	12.4	11.1	4	USA DNS	UK	b	1	12.8	12.8
3	USA Soft	UK	b	2	12.4		4	USA DNS	UK	b	2	12.8	
3	USA Soft	UK	c	1	12.4	11.1	4	USA DNS	UK	c	1	12.9	12.8
3	USA Soft	UK	c	2	12.4		4	USA DNS	UK	c	2	12.9	
3	USA Soft	SA 2	a	1	12.5	10.7	4	USA DNS	SA 2	a	1	12.9	12.5
3	USA Soft	SA 2	a	2	12.4		4	USA DNS	SA 2	a	2	12.9	
3	USA Soft	SA 2	b	1	12.4	10.9	4	USA DNS	SA 2	b	1	12.9	12.5
3	USA Soft	SA 2	b	2	12.4		4	USA DNS	SA 2	b	2	12.8	
3	USA Soft	SA 2	c	1	12.5	11.1	4	USA DNS	SA 2	c	1	12.7	12.3
3	USA Soft	SA 2	c	2	12.4		4	USA DNS	SA 2	c	2	12.7	
5	Australia	Australia	a	1	11.4	13.5	7	N. Cape	Australia	a	1	11.9	10.7
5	Australia	Australia	a	2	11.3		7	N. Cape	Australia	a	2	11.9	
5	Australia	Australia	b	1	11.5	13.5	7	N. Cape	Australia	b	1	11.9	10.7
5	Australia	Australia	b	2	11.4		7	N. Cape	Australia	b	2	11.9	
5	Australia	Australia	c	1	11.4	13.6	7	N. Cape	Australia	c	1	11.7	10.6
5	Australia	Australia	c	2	11.4		7	N. Cape	Australia	c	2	11.7	
5	Australia	Germany	a	1	11.4	13.7	7	N. Cape	Germany	a	1	11.8	10.8
5	Australia	Germany	a	2	11.4		7	N. Cape	Germany	a	2	11.8	
5	Australia	Germany	b	1	11.4	13.4	7	N. Cape	Germany	b	1	11.8	10.9
5	Australia	Germany	b	2	11.4		7	N. Cape	Germany	b	2	11.8	
5	Australia	Germany	c	1	11.4	13.4	7	N. Cape	Germany	c	1	11.9	10.8
5	Australia	Germany	c	2	11.4		7	N. Cape	Germany	c	2	11.9	
5	Australia	Canada	a	1	11.5	13.5	7	N. Cape	Canada	a	1	11.9	10.9
5	Australia	Canada	a	2	11.4		7	N. Cape	Canada	a	2	11.9	
5	Australia	Canada	b	1	11.4	13.5	7	N. Cape	Canada	b	1	11.9	11.1
5	Australia	Canada	b	2	11.4		7	N. Cape	Canada	b	2	11.9	
5	Australia	Canada	c	1	11.4	13.5	7	N. Cape	Canada	c	1	11.9	10.9
5	Australia	Canada	c	2	11.4		7	N. Cape	Canada	c	2	11.9	
5	Australia	SA 1	a	1	11.4	13.5	7	N. Cape	SA 1	a	1	11.7	10.8
5	Australia	SA 1	a	2	11.4		7	N. Cape	SA 1	a	2	11.7	
5	Australia	SA 1	b	1	11.4	13.5	7	N. Cape	SA 1	b	1	11.9	10.9
5	Australia	SA 1	b	2	11.4		7	N. Cape	SA 1	b	2	11.9	
5	Australia	SA 1	c	1	11.5	13.6	7	N. Cape	SA 1	c	1	11.8	10.9
5	Australia	SA 1	c	2	11.4		7	N. Cape	SA 1	c	2	11.8	
5	Australia	France	a	1	11.2	13.6	7	N. Cape	France	a	1	11.9	10.7
5	Australia	France	a	2	11.3		7	N. Cape	France	a	2	11.9	

Table 7.3.1.1 continued

5	Australia	France	b	1	11.4	13.5	7	N. Cape	France	b	1	11.9	10.6
5	Australia	France	b	2	11.4		7	N. Cape	France	b	2	11.9	
5	Australia	France	c	1	11.5	13.4	7	N. Cape	France	c	1	11.9	10.7
5	Australia	France	c	2	11.4		7	N. Cape	France	c	2	11.9	
5	Australia	USA	a	1	11.4	13.5	7	N. Cape	USA	a	1	11.7	11.0
5	Australia	USA	a	2	11.4		7	N. Cape	USA	a	2	11.7	
5	Australia	USA	b	1	11.4	13.5	7	N. Cape	USA	b	1	11.8	11.1
5	Australia	USA	b	2	11.4		7	N. Cape	USA	b	2	11.8	
5	Australia	USA	c	1	11.5	13.4	7	N. Cape	USA	c	1	11.9	10.7
5	Australia	USA	c	2	11.4		7	N. Cape	USA	c	2	11.9	
5	Australia	UK	a	1	11.4	13.5	7	N. Cape	UK	a	1	11.9	10.6
5	Australia	UK	a	2	11.4		7	N. Cape	UK	a	2	11.9	
5	Australia	UK	b	1	11.5	13.4	7	N. Cape	UK	b	1	11.8	10.6
5	Australia	UK	b	2	11.4		7	N. Cape	UK	b	2	11.8	
5	Australia	UK	c	1	11.4	13.7	7	N. Cape	UK	c	1	11.8	10.6
5	Australia	UK	c	2	11.4		7	N. Cape	UK	c	2	11.8	
5	Australia	SA 2	a	1	11.4	13.4	7	N. Cape	SA 2	a	1	11.9	11.0
5	Australia	SA 2	a	2	11.6		7	N. Cape	SA 2	a	2	11.9	
5	Australia	SA 2	b	1	11.4	13.7	7	N. Cape	SA 2	b	1	11.8	11.0
5	Australia	SA 2	b	2	11.3		7	N. Cape	SA 2	b	2	11.8	
5	Australia	SA 2	c	1	11.4	13.6	7	N. Cape	SA 2	c	1	11.9	11.0
5	Australia	SA 2	c	2	11.4		7	N. Cape	SA 2	c	2	11.9	
8	N. Cape	Australia	a	1	12.4	10.5	10	Germany	Australia	a	1	13.0	11.0
8	N. Cape	Australia	a	2	12.4		10	Germany	Australia	a	2	13.0	
8	N. Cape	Australia	b	1	12.4	10.5	10	Germany	Australia	b	1	12.9	11.0
8	N. Cape	Australia	b	2	12.4		10	Germany	Australia	b	2	12.9	
8	N. Cape	Australia	c	1	12.4	10.6	10	Germany	Australia	c	1	13.1	11.1
8	N. Cape	Australia	c	2	12.4		10	Germany	Australia	c	2	13.1	
8	N. Cape	Germany	a	1	12.6	10.4	10	Germany	Germany	a	1	13.1	10.9
8	N. Cape	Germany	a	2	12.6		10	Germany	Germany	a	2	13.1	
8	N. Cape	Germany	b	1	12.6	10.5	10	Germany	Germany	b	1	13.0	10.8
8	N. Cape	Germany	b	2	12.6		10	Germany	Germany	b	2	13.0	
8	N. Cape	Germany	c	1	12.6	10.4	10	Germany	Germany	c	1	13.1	10.9
8	N. Cape	Germany	c	2	12.6		10	Germany	Germany	c	2	13.1	
8	N. Cape	Canada	a	1	12.5	10.5	10	Germany	Canada	a	1	12.9	11.0
8	N. Cape	Canada	a	2	12.5		10	Germany	Canada	a	2	12.9	
8	N. Cape	Canada	b	1	12.5	10.7	10	Germany	Canada	b	1	12.9	11.0
8	N. Cape	Canada	b	2	12.5		10	Germany	Canada	b	2	12.9	
8	N. Cape	Canada	c	1	12.4	10.6	10	Germany	Canada	c	1	13.0	11.0
8	N. Cape	Canada	c	2	12.4		10	Germany	Canada	c	2	13.0	
8	N. Cape	SA 1	a	1	12.4	10.5	10	Germany	SA 1	a	1	13.0	10.9

Table 7.3.1.1 continued

8	N. Cape	SA 1	a	2	12.4		10	Germany	SA 1	a	2	13.0	
8	N. Cape	SA 1	b	1	12.7	10.5	10	Germany	SA 1	b	1	12.9	11.1
8	N. Cape	SA 1	b	2	12.7		10	Germany	SA 1	b	2	12.9	
8	N. Cape	SA 1	c	1	12.6	10.5	10	Germany	SA 1	c	1	12.9	10.8
8	N. Cape	SA 1	c	2	12.6		10	Germany	SA 1	c	2	12.9	
8	N. Cape	France	a	1	12.5	10.6	10	Germany	France	a	1	12.9	11.1
8	N. Cape	France	a	2	12.5		10	Germany	France	a	2	12.9	
8	N. Cape	France	b	1	12.5	10.4	10	Germany	France	b	1	13.0	10.8
8	N. Cape	France	b	2	12.5		10	Germany	France	b	2	13.0	
8	N. Cape	France	c	1	12.5	10.5	10	Germany	France	c	1	13.0	10.9
8	N. Cape	France	c	2	12.5		10	Germany	France	c	2	13.0	
8	N. Cape	USA	a	1	12.6	10.5	10	Germany	USA	a	1	13.1	11.1
8	N. Cape	USA	a	2	12.6		10	Germany	USA	a	2	13.1	
8	N. Cape	USA	b	1	12.7	10.2	10	Germany	USA	b	1	13.1	10.9
8	N. Cape	USA	b	2	12.7		10	Germany	USA	b	2	13.1	
8	N. Cape	USA	c	1	12.5	10.3	10	Germany	USA	c	1	13.1	10.9
8	N. Cape	USA	c	2	12.5		10	Germany	USA	c	2	13.1	
8	N. Cape	UK	a	1	12.6	10.4	10	Germany	UK	a	1	12.9	11.0
8	N. Cape	UK	a	2	12.6		10	Germany	UK	a	2	12.9	
8	N. Cape	UK	b	1	12.4	10.2	10	Germany	UK	b	1	13.0	11.0
8	N. Cape	UK	b	2	12.4		10	Germany	UK	b	2	13.0	
8	N. Cape	UK	c	1	12.5	10.3	10	Germany	UK	c	1	12.8	11.0
8	N. Cape	UK	c	2	12.5		10	Germany	UK	c	2	12.8	
8	N. Cape	SA 2	a	1	12.4	10.5	10	Germany	SA 2	a	1	13.1	11.0
8	N. Cape	SA 2	a	2	12.4		10	Germany	SA 2	a	2	13.1	
8	N. Cape	SA 2	b	1	12.6	10.4	10	Germany	SA 2	b	1	13.1	11.0
8	N. Cape	SA 2	b	2	12.6		10	Germany	SA 2	b	2	13.1	
8	N. Cape	SA 2	c	1	12.5	10.4	10	Germany	SA 2	c	1	12.9	11.0
8	N. Cape	SA 2	c	2	12.5		10	Germany	SA 2	c	2	12.9	
11	W. Cape	Australia	a	1	12.2	11.5	12	SST 88	Australia	a	1	11.7	11.8
11	W. Cape	Australia	a	2	12.3		12	SST 88	Australia	a	2	11.7	
11	W. Cape	Australia	b	1	12.3	11.5	12	SST 88	Australia	b	1	11.8	11.7
11	W. Cape	Australia	b	2	12.2		12	SST 88	Australia	b	2	11.7	
11	W. Cape	Australia	c	1	12.2	11.5	12	SST 88	Australia	c	1	11.8	11.8
11	W. Cape	Australia	c	2	12.3		12	SST 88	Australia	c	2	11.8	
11	W. Cape	Germany	a	1	12.3	11.6	12	SST 88	Germany	a	1	11.8	11.7
11	W. Cape	Germany	a	2	12.3		12	SST 88	Germany	a	2	11.8	
11	W. Cape	Germany	b	1	12.4	11.6	12	SST 88	Germany	b	1	11.8	11.8
11	W. Cape	Germany	b	2	12.5		12	SST 88	Germany	b	2	11.8	
11	W. Cape	Germany	c	1	12.5	11.5	12	SST 88	Germany	c	1	11.8	11.7
11	W. Cape	Germany	c	2	12.5		12	SST 88	Germany	c	2	11.8	

Table 7.3.1.1 continued

11	W. Cape	Canada	a	1	12.3	11.3	12	SST 88	Canada	a	1	11.9	11.7
11	W. Cape	Canada	a	2	12.2		12	SST 88	Canada	a	2	11.9	
11	W. Cape	Canada	b	1	12.3	11.4	12	SST 88	Canada	b	1	11.9	11.5
11	W. Cape	Canada	b	2	12.2		12	SST 88	Canada	b	2	11.9	
11	W. Cape	Canada	c	1	12.1	11.5	12	SST 88	Canada	c	1	12.0	11.6
11	W. Cape	Canada	c	2	12.2		12	SST 88	Canada	c	2	12.0	
11	W. Cape	SA 1	a	1	12.2	11.3	12	SST 88	SA 1	a	1	12.0	11.6
11	W. Cape	SA 1	a	2	12.2		12	SST 88	SA 1	a	2	12.0	
11	W. Cape	SA 1	b	1	12.5	11.4	12	SST 88	SA 1	b	1	12.0	11.8
11	W. Cape	SA 1	b	2	12.3		12	SST 88	SA 1	b	2	12.0	
11	W. Cape	SA 1	c	1	12.2	11.5	12	SST 88	SA 1	c	1	12.0	11.9
11	W. Cape	SA 1	c	2	12.2		12	SST 88	SA 1	c	2	12.0	
11	W. Cape	France	a	1	12.3	11.5	12	SST 88	France	a	1	11.8	12.0
11	W. Cape	France	a	2	12.3		12	SST 88	France	a	2	11.7	
11	W. Cape	France	b	1	12.3	11.6	12	SST 88	France	b	1	11.8	11.9
11	W. Cape	France	b	2	12.3		12	SST 88	France	b	2	11.8	
11	W. Cape	France	c	1	12.3	11.7	12	SST 88	France	c	1	11.7	12.0
11	W. Cape	France	c	2	12.3		12	SST 88	France	c	2	11.3	
11	W. Cape	USA	a	1	12.3	11.6	12	SST 88	USA	a	1	11.9	12.0
11	W. Cape	USA	a	2	12.3		12	SST 88	USA	a	2	11.9	
11	W. Cape	USA	b	1	12.3	11.7	12	SST 88	USA	b	1	11.8	11.9
11	W. Cape	USA	b	2	12.3		12	SST 88	USA	b	2	11.8	
11	W. Cape	USA	c	1	12.3	11.5	12	SST 88	USA	c	1	11.8	11.9
11	W. Cape	USA	c	2	12.2		12	SST 88	USA	c	2	11.8	
11	W. Cape	UK	a	1	12.3	11.6	12	SST 88	UK	a	1	11.8	11.9
11	W. Cape	UK	a	2	12.3		12	SST 88	UK	a	2	11.8	
11	W. Cape	UK	b	1	12.3	11.6	12	SST 88	UK	b	1	11.8	12.0
11	W. Cape	UK	b	2	12.3		12	SST 88	UK	b	2	11.8	
11	W. Cape	UK	c	1	12.3	11.5	12	SST 88	UK	c	1	11.8	11.9
11	W. Cape	UK	c	2	12.3		12	SST 88	UK	c	2	11.7	
11	W. Cape	SA 2	a	1	12.4	11.7	12	SST 88	SA 2	a	1	12	11.6
11	W. Cape	SA 2	a	2	12.4		12	SST 88	SA 2	a	2	11.9	
11	W. Cape	SA 2	b	1	12.5	11.6	12	SST 88	SA 2	b	1	11.9	11.8
11	W. Cape	SA 2	b	2	12.4		12	SST 88	SA 2	b	2	11.9	
11	W. Cape	SA 2	c	1	12.3	11.5	12	SST 88	SA 2	c	1	11.9	11.7
11	W. Cape	SA 2	c	2	12.3		12	SST 88	SA 2	c	2	11.9	

Sub = Sub-sub sample; Rep = Repetition; DNS = Dark Northern Spring; N. Cape = Northern Cape; W. Cape = Western Cape

Table 7.3.2 Experiment 3b: determination of variation in hectolitre mass (HLM) within and between HLM devices using South African cultivars

Sample	Sub-	Rep	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
14	a	1	81.97	79.10	78.79	78.79	77.24	77.12	78.80	78.81
	a	2	80.68	79.50	78.78	78.89	77.11	77.41	78.80	78.81
	b	1	81.28	79.30	78.88	78.89	77.01	77.30	78.80	78.94
	b	2	81.29	79.30	78.88	78.79	77.26	77.30	78.80	78.94
	c	1	81.89	79.50	79.14	78.79	77.34	77.16	78.80	78.94
	c	2	80.91	79.50	79.25	78.79	77.18	77.17	78.80	78.94
15	a	1	81.77	79.30	78.79	79.09	76.89	76.89	78.40	78.55
	a	2	80.68	79.30	78.82	78.89	76.69	76.71	78.40	78.68
	b	1	81.28	79.10	78.69	78.69	76.98	77.17	78.40	78.68
	b	2	81.29	79.30	78.55	78.89	76.93	77.16	78.40	78.55
	c	1	81.89	79.30	78.50	78.79	76.97	76.94	78.40	78.55
	c	2	80.91	79.10	78.62	78.89	77.06	76.88	78.00	78.68
16	a	1	82.42	80.50	79.78	80.49	78.30	78.73	80.00	79.97
	a	2	82.86	80.50	79.98	80.29	78.43	78.52	80.00	80.10
	b	1	81.80	80.50	79.78	80.79	77.96	78.26	80.00	79.71
	b	2	82.64	80.50	79.73	80.69	78.26	78.64	80.00	79.84
	c	1	84.10	80.70	80.12	80.39	78.33	78.47	80.00	79.84
	c	2	83.48	80.70	80.03	80.49	78.35	78.28	80.00	79.71
17	a	1	82.16	79.70	79.70	79.69	77.96	78.59	78.80	79.20
	a	2	82.71	80.00	79.72	79.89	77.98	78.25	79.20	79.20
	b	1	82.07	80.00	79.69	79.99	78.00	77.86	79.60	79.33
	b	2	82.32	80.10	79.36	79.99	78.06	78.19	79.20	79.46
	c	1	82.31	80.30	79.57	79.99	77.99	78.02	79.20	79.33
	c	2	82.36	80.30	79.53	79.99	77.80	78.05	79.60	79.33
18	a	1	85.79	83.60	83.21	83.70	81.46	82.00	83.20	83.07
	a	2	85.34	83.60	83.27	83.90	81.67	81.94	82.80	83.20
	b	1	86.42	83.80	83.50	84.00	81.87	81.79	83.20	83.33
	b	2	86.05	83.60	83.41	84.00	81.61	81.91	83.20	83.33
	c	1	87.05	83.80	83.43	83.80	81.60	81.83	82.80	82.69
	c	2	86.39	84.00	83.59	83.60	81.61	81.51	82.80	82.82

Table 7.3.2 continued

19	a	1	78.85	75.50	74.76	76.58	72.65	73.12	75.60	74.68
	a	2	78.86	75.50	74.49	76.68	72.08	73.16	75.20	74.68
	b	1	78.01	75.10	74.61	76.68	72.39	73.38	75.20	74.29
	b	2	78.55	75.50	74.40	76.68	72.24	73.33	75.20	74.42
	c	1	78.23	75.50	74.79	76.78	72.24	73.16	75.20	74.29
	c	2	78.44	75.10	74.45	76.68	72.53	73.34	74.80	74.29
20	a	1	78.92	77.00	76.55	77.88	74.54	74.45	76.80	76.36
	a	2	78.68	76.70	76.77	77.88	74.21	74.35	77.20	76.10
	b	1	79.37	76.70	76.29	77.78	74.20	74.32	77.20	76.10
	b	2	79.84	77.00	76.28	77.78	74.29	74.42	76.80	75.97
	c	1	79.36	77.10	76.37	77.48	74.28	74.32	76.80	76.36
	c	2	79.03	77.00	76.13	77.48	74.29	74.39	76.80	75.97
21	a	1	76.13	73.10	73.30	74.18	71.18	71.55	73.60	73.38
	a	2	76.25	73.00	73.49	74.28	71.24	71.36	74.00	73.25
	b	1	76.14	73.10	73.34	74.18	71.22	71.39	74.40	73.13
	b	2	76.64	73.10	73.40	74.28	71.12	71.32	74.80	73.38
	c	1	76.12	73.30	73.26	74.08	71.10	71.27	73.60	73.00
	c	2	76.75	73.10	73.31	73.98	71.05	71.32	73.60	72.87
24	a	1	83.88	81.80	80.91	81.79	79.49	79.47	81.20	80.75
	a	2	83.48	81.80	80.97	81.89	79.29	79.49	81.20	80.75
	b	1	83.52	81.40	81.05	81.89	79.34	79.46	80.60	80.75
	b	2	83.84	81.60	81.03	81.89	79.36	79.58	81.20	80.88
	c	1	83.61	81.60	81.10	81.79	79.38	79.48	80.80	80.75
	c	2	84.16	81.60	81.15	81.89	79.39	79.60	81.00	80.62
26	a	1	78.89	76.30	75.48	76.98	73.77	74.25	76.40	75.45
	a	2	79.04	76.20	75.38	77.18	73.43	74.29	76.00	75.45
	b	1	78.58	76.30	75.23	76.88	73.80	74.16	76.00	75.06
	b	2	78.08	76.30	75.53	76.68	73.60	73.79	76.00	75.19
	c	1	77.42	76.50	75.28	76.28	74.24	73.03	75.20	75.19
	c	2	77.72	76.50	75.47	76.28	73.97	72.70	75.60	75.06

Table 7.4.1 Experiment 4a: comparison of hectolitre mass (HLM) devices using a single work sample of mixed wheat

Sample	Sub-	Rep	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
1	a	1	84.26	81.60	81.31	81.39	79.40	79.76	81.40	81.14
	a	2	84.10	81.80	81.35	81.69	79.47	79.76	81.00	81.14
	b	1	84.26	81.60	80.93	81.19	79.27	79.36	81.20	80.75
	b	2	83.67	81.60	81.15	81.19	79.18	79.30	81.00	81.01
	c	1	83.58	81.80	81.36	81.39	79.55	79.46	81.80	81.01
	c	2	83.94	81.20	81.27	81.39	79.13	79.48	81.40	81.01
2	a	1	86.52	83.60	83.48	83.40	81.11	81.54	83.80	82.94
	a	2	86.80	83.40	83.11	83.50	81.43	81.48	83.60	83.07
	b	1	86.53	83.60	83.38	83.40	81.53	81.78	83.80	82.94
	b	2	87.08	84.00	83.27	83.40	81.52	81.71	83.60	82.82
	c	1	86.40	83.80	83.01	83.30	81.26	81.31	83.80	80.75
	c	2	86.78	83.60	83.03	83.30	81.51	81.43	83.60	80.75
3	a	1	84.07	81.80	81.02	80.69	78.99	79.49	80.80	80.75
	a	2	84.08	81.80	81.13	80.89	79.34	79.35	81.20	81.01
	b	1	84.42	82.00	81.41	81.09	79.50	79.83	81.20	81.01
	b	2	84.52	81.80	81.43	81.29	79.59	79.56	81.40	81.14
	c	1	84.64	82.20	81.24	81.19	79.48	79.73	81.20	81.01
	c	2	84.60	82.00	81.24	81.19	79.60	79.88	81.20	81.26
4	a	1	86.11	83.40	82.63	82.49	80.66	81.01	82.60	82.04
	a	2	86.19	83.20	82.66	82.49	80.85	81.03	82.20	82.30
	b	1	84.96	83.00	82.39	81.99	80.55	80.57	82.00	82.04
	b	2	85.52	83.00	82.58	82.29	80.75	80.62	82.40	82.30
	c	1	85.67	82.80	82.26	81.89	80.36	80.39	82.00	82.30
	c	2	85.33	82.80	82.29	81.89	80.43	80.06	82.00	82.04
5	a	1	86.25	83.60	82.96	82.79	80.98	81.14	83.00	82.69
	a	2	85.96	83.60	83.02	82.89	81.10	81.24	83.00	82.69
	b	1	85.71	83.40	82.75	82.69	80.83	81.06	83.00	82.43
	b	2	85.90	83.40	82.83	82.69	80.87	81.02	83.00	82.43
	c	1	86.23	83.40	83.06	82.89	81.20	81.44	82.80	82.94
	c	2	86.64	83.60	83.18	82.99	81.28	81.40	83.00	82.94

Table 7.4.1 continued

7	a	1	85.47	82.60	82.11	82.79	80.46	80.75	82.40	81.78
	a	2	84.80	83.00	82.06	82.69	80.55	80.98	82.60	82.17
	b	1	84.70	82.80	81.88	82.29	79.95	79.87	82.20	81.39
	b	2	85.21	82.20	81.79	82.29	80.21	80.13	82.00	81.52
	c	1	85.05	82.60	82.21	82.49	80.18	80.67	82.20	81.78
	c	2	84.83	82.80	82.34	82.59	80.25	80.43	82.40	81.78
8	a	1	84.09	81.40	80.82	80.89	78.94	79.20	81.20	81.14
	a	2	83.17	80.70	80.45	80.79	78.77	79.26	80.60	80.75
	b	1	84.45	81.60	81.11	81.19	79.16	79.34	81.40	80.49
	b	2	84.41	81.20	81.14	81.09	78.97	79.50	81.40	80.49
	c	1	83.98	81.20	80.66	80.89	78.79	79.04	81.20	82.43
	c	2	84.13	80.70	80.90	80.69	78.68	78.92	80.80	82.56
10	a	1	82.35	78.58	78.98	79.29	76.83	76.89	78.80	78.03
	a	2	81.53	78.50	78.76	79.29	76.94	76.73	78.80	78.29
	b	1	81.85	78.70	79.19	79.39	76.70	76.92	78.80	78.16
	b	2	82.37	79.30	78.93	79.49	77.19	76.99	79.40	78.42
	c	1	83.26	79.00	79.12	79.39	76.89	77.02	78.80	78.03
	c	2	81.47	78.70	78.73	79.19	76.83	76.54	79.40	78.29
11	a	1	86.21	84.20	83.74	83.40	82.04	81.98	83.60	83.33
	a	2	86.75	84.20	83.79	83.70	82.13	81.51	83.60	83.46
	b	1	85.98	83.80	83.14	82.69	81.35	80.84	82.80	82.69
	b	2	85.31	83.60	83.25	82.79	81.39	81.04	83.60	82.94
	c	1	84.86	83.00	82.21	82.09	80.50	80.13	82.40	82.04
	c	2	85.21	83.20	82.41	82.29	80.02	81.00	82.40	82.04
12	a	1	82.49	80.50	79.66	79.99	77.78	78.20	80.00	79.20
	a	2	82.26	80.50	79.81	80.29	78.05	78.38	80.20	79.59
	b	1	82.55	80.10	79.53	79.89	77.27	78.16	79.20	78.94
	b	2	82.59	80.50	79.94	80.09	77.47	78.18	80.40	79.46
	c	1	83.29	80.30	79.64	80.19	77.75	78.19	79.60	79.33
	c	2	82.39	80.30	79.90	80.39	77.87	78.14	79.80	79.46

Table 7.4.2 Experiment 4b: comparison of hectolitre mass (HLM) devices using a single work sample of South African cultivars

Sample	Sub-	Rep	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
14	a	1	81.36	79.30	78.96	78.79	77.33	77.24	78.80	78.68
	a	2	82.71	79.50	79.03	78.79	77.26	77.44	78.40	78.94
	b	1	81.67	79.70	78.83	78.79	77.35	77.34	78.80	78.94
	b	2	82.14	79.70	79.27	78.99	77.22	77.53	87.80	78.94
	c	1	82.49	79.70	79.38	78.89	77.39	77.60	79.20	79.07
	c	2	82.69	79.70	79.25	79.09	77.34	77.56	78.80	79.20
15	a	1	82.43	79.10	78.91	78.59	76.85	77.16	78.80	78.42
	a	2	82.30	79.30	78.78	78.69	77.00	77.22	78.80	78.42
	b	1	82.04	79.50	78.90	78.79	77.05	77.32	78.80	78.55
	b	2	82.10	79.30	78.84	78.89	77.33	77.24	79.20	78.81
	c	1	82.08	79.50	79.00	78.79	76.97	77.30	79.20	78.68
	c	2	82.20	79.30	78.99	78.79	77.24	77.30	79.20	78.81
16	a	1	82.26	80.70	80.22	80.49	78.40	78.29	80.00	79.84
	a	2	82.89	80.70	80.17	80.49	78.52	78.24	80.40	79.97
	b	1	83.00	81.20	80.37	80.49	78.58	78.71	80.40	80.23
	b	2	83.19	80.70	80.28	80.79	78.21	78.48	80.40	80.23
	c	1	83.19	81.00	80.11	80.69	78.44	78.65	80.00	80.10
	c	2	83.55	81.00	80.53	80.69	78.44	78.51	80.40	80.23
17	a	1	83.39	80.00	79.67	79.99	77.77	77.79	79.60	79.07
	a	2	82.52	80.10	79.99	79.69	77.52	78.28	79.20	79.33
	b	1	83.90	80.10	80.01	80.18	78.15	78.32	80.00	79.46
	b	2	83.22	80.30	80.26	80.29	78.12	78.20	79.60	79.59
	c	1	82.97	80.30	80.06	80.18	78.11	77.92	80.00	79.59
	c	2	82.92	80.50	79.89	80.49	78.20	78.06	80.00	79.71
18	a	1	86.19	83.80	83.30	83.80	81.34	81.69	83.20	82.82
	a	2	86.75	83.80	83.34	83.80	81.50	81.90	83.20	83.20
	b	1	86.75	83.40	83.13	83.80	81.56	81.86	83.60	82.69
	b	2	86.82	83.80	83.33	83.80	81.57	81.80	83.20	83.07
	c	1	86.88	84.00	83.44	83.70	81.69	81.84	83.20	82.82
	c	2	86.05	84.00	83.35	83.90	81.76	81.97	83.60	83.20

Table 7.4.2 continued

19	a	1	78.61	75.70	75.17	76.88	73.35	73.60	75.60	74.93
	a	2	78.27	75.70	74.89	76.88	73.48	73.44	75.60	74.80
	b	1	78.02	75.30	75.02	76.68	72.85	73.10	76.00	74.80
	b	2	77.89	75.10	75.01	76.68	73.30	73.03	76.00	74.80
	c	1	78.45	75.30	74.87	76.58	72.97	73.24	76.40	74.80
	c	2	78.32	75.50	74.83	76.58	72.93	73.26	75.60	74.68
20	a	1	80.79	76.50	76.47	77.78	74.45	74.25	77.20	76.23
	a	2	80.37	76.70	76.58	77.68	74.26	74.45	76.80	75.97
	b	1	80.80	76.70	76.60	77.58	74.45	74.66	77.60	76.36
	b	2	80.40	76.70	76.59	77.58	74.24	74.34	77.60	75.97
	c	1	80.00	76.70	76.51	77.88	74.43	74.46	77.60	76.36
	c	2	79.81	76.70	76.47	77.68	74.16	74.40	77.20	76.10
21	a	1	76.45	73.00	72.50	74.18	70.52	70.69	73.60	72.61
	a	2	76.48	73.00	72.37	74.08	70.47	70.48	73.60	74.29
	b	1	76.20	73.10	72.72	74.28	70.56	70.42	73.60	72.61
	b	2	75.76	73.00	72.50	74.08	70.44	70.60	73.20	72.48
	c	1	76.92	73.30	72.67	74.38	70.62	70.54	74.00	72.74
	c	2	76.27	73.10	72.48	74.28	70.48	70.49	74.00	72.48
24	a	1	83.97	81.60	81.00	81.99	79.06	79.48	81.20	80.75
	a	2	83.76	81.60	81.00	81.98	79.08	79.20	81.20	80.49
	b	1	83.71	81.60	81.18	82.09	79.32	79.51	81.20	80.88
	b	2	83.57	81.60	81.42	82.09	79.21	79.66	81.20	80.75
	c	1	84.34	81.70	81.36	81.99	79.18	79.28	81.20	80.75
	c	2	84.44	82.00	81.22	82.09	79.08	79.66	81.20	80.75
26	a	1	80.27	76.50	75.81	77.08	74.20	74.53	76.00	75.45
	a	2	80.08	76.50	75.89	77.59	74.29	74.51	76.40	75.71
	b	1	78.35	76.10	75.70	77.18	74.31	74.13	76.00	75.32
	b	2	78.54	76.10	75.66	77.48	74.02	74.27	76.00	75.58
	c	1	78.87	76.50	75.80	77.48	74.09	74.17	76.40	75.58
	c	2	78.66	76.50	76.09	77.48	74.26	74.32	76.40	75.84

Table 7.5 Experiment 5: comparison of ten respective South African hectolitre mass (HLM) devices

Sample	Rep	SA 1	SA 2	SA 3	SA 4	SA 5	SA 6	SA 7	SA 8	SA 9	SA 10
28	1	75.91	76.04	76.20	76.12	76.50	75.90	75.80	75.60	76.00	75.80
	2	75.79	75.80	76.48	76.28	76.30	76.60	75.80	75.70	76.30	76.31
29	1	79.11	79.48	79.56	79.74	79.50	79.60	79.00	79.30	79.40	79.35
	2	79.39	79.54	79.55	79.72	79.80	79.60	79.10	79.40	79.70	79.27
30	1	78.10	78.60	78.62	78.82	78.60	78.90	78.50	78.40	78.50	78.38
	2	78.19	78.70	78.80	78.98	78.60	79.00	78.60	78.40	78.70	78.35
31	1	77.63	78.04	78.15	78.42	78.20	78.50	77.70	77.80	78.10	77.71
	2	77.81	78.12	78.21	78.38	78.40	78.50	77.70	77.90	78.20	77.81
32	1	77.75	77.86	78.22	78.48	78.30	78.50	78.00	78.00	78.40	78.22
	2	78.08	78.44	78.33	78.52	78.20	78.50	78.00	78.10	78.40	78.21
33	1	77.34	77.52	77.48	77.78	77.90	77.70	77.50	77.40	77.40	77.20
	2	77.13	77.60	77.51	77.88	77.70	78.00	77.40	77.40	77.60	77.44
34	1	78.99	79.62	79.60	79.56	79.80	79.90	79.30	79.30	79.30	79.30
	2	79.31	79.72	79.67	79.72	79.50	80.00	79.30	79.30	79.30	79.38
35	1	76.99	77.08	77.30	77.62	77.60	77.40	76.90	77.00	77.40	76.98
	2	76.90	77.06	77.28	77.52	77.30	77.70	77.10	77.00	77.10	76.93
36	1	77.03	77.52	77.75	77.74	77.70	78.50	77.30	77.50	77.70	77.63
	2	77.01	77.48	77.68	77.82	77.70	78.20	77.20	77.60	77.60	77.57
37	1	73.60	74.18	74.12	74.24	74.40	74.50	73.40	74.00	74.10	73.91
	2	73.83	74.40	74.18	74.32	74.30	74.30	73.70	73.90	74.40	74.06

Table 7.6 Experiment 6: effect of impurities on hectolitre mass (HLM) determinations

Sample	Rep	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
Before impurities have been removed									
28	1	78.77	77.50	75.93	75.08	74.48	74.84	75.80	74.80
	2	77.63	77.30	76.11	75.68	74.05	73.96	76.60	75.58
29	1	83.67	81.20	80.20	80.59	78.28	78.36	80.80	79.97
	2	84.08	81.40	80.69	80.89	78.56	78.50	80.80	80.10
30	1	81.22	80.10	79.07	79.19	77.17	77.54	79.40	78.68
	2	81.03	79.50	79.29	79.39	77.44	77.28	79.40	78.94
31	1	82.13	79.10	78.67	77.98	76.36	76.56	77.80	77.65
	2	80.35	79.50	78.57	77.88	75.57	76.94	77.60	77.91
32	1	80.66	79.50	79.17	78.59	76.96	76.94	78.20	78.29
	2	82.26	80.00	79.44	78.79	77.41	77.44	79.20	78.68
33	1	81.05	79.10	78.51	78.39	76.09	76.61	78.00	77.65
	2	79.12	79.10	78.60	78.39	76.04	76.78	78.60	78.29
34	1	81.00	81.00	79.73	79.29	77.26	78.38	79.40	78.94
	2	81.31	80.70	79.82	79.69	77.41	77.93	80.20	78.94
35	1	79.31	79.00	78.23	77.48	75.89	76.14	77.80	77.13
	2	79.33	78.50	77.89	77.78	75.34	76.05	77.60	77.52
36	1	80.01	79.30	78.34	78.59	76.36	76.67	78.20	78.03
	2	80.22	79.10	78.32	78.79	76.65	76.85	77.80	78.29
37	1	76.30	75.10	74.67	74.88	72.84	72.67	74.80	73.90
	2	77.13	75.50	74.42	75.38	72.85	73.22	74.80	74.29
After impurities have been removed									
28	1	79.62	78.00	77.34	76.78	75.28	75.35	76.40	76.87
	2	79.14	78.00	77.35	77.08	75.23	75.74	76.80	77.00
29	1	84.45	81.60	81.02	81.49	79.14	79.29	81.00	80.75
	2	83.78	81.60	81.02	81.59	79.19	79.34	80.80	80.75
30	1	81.86	80.70	80.18	80.49	78.41	78.53	79.80	79.71
	2	82.64	80.50	80.21	80.49	78.12	78.46	80.00	79.84
31	1	81.48	80.00	79.36	78.89	77.06	77.37	78.60	78.94
	2	81.11	80.30	79.59	79.29	77.30	77.71	79.00	78.94
32	1	80.57	80.10	79.44	79.39	77.43	77.78	78.60	79.20
	2	81.19	80.10	79.56	79.29	77.74	77.65	79.20	79.20
33	1	80.30	78.70	79.16	79.29	77.24	77.56	78.80	78.81
	2	81.11	80.00	79.17	79.39	77.19	77.44	79.00	78.94
34	1	82.35	81.60	80.97	81.09	79.25	79.53	80.80	80.49
	2	83.87	81.60	81.05	81.09	79.11	79.49	80.60	80.88
35	1	81.11	79.50	78.73	78.89	76.77	77.38	78.60	78.29
	2	80.01	80.00	78.89	79.09	77.06	77.13	78.40	78.55
36	1	81.02	79.70	79.07	79.19	76.96	77.78	79.00	78.68
	2	81.50	79.70	79.39	79.29	77.32	77.72	78.80	78.94
37	1	76.72	76.00	74.76	75.88	73.34	73.64	75.20	74.68
	2	75.87	76.00	75.00	75.98	73.57	73.50	75.40	74.93

Table 7.7 Experiment 7: effect of operator on hectolitre mass (HLM) measurements

		Operator 1		Operator 2		Operator 3	
Sample	**Rep**	**SA 1**	**SA 2**	**SA 1**	**SA 2**	**SA 1**	**SA 2**
28	1	74.98	75.67	74.82	74.99	76.53	75.66
	2	75.29	75.88	75.20	75.10	76.02	75.22
	3	75.34	76.27	75.15	75.03	76.29	75.43
	4	75.59	76.06	74.91	75.20	76.51	76.26
	5	75.54	75.89	75.23	75.05	76.75	75.63
	6	75.34	75.62	75.39	75.29	75.79	75.73
	7	75.52	75.74	75.20	75.08	76.32	76.14
	8	75.92	75.95	75.00	74.97	75.48	75.59
	9	75.89	76.19	75.04	75.23	75.97	76.52
	10	75.68	75.93	75.18	75.34	76.05	75.96
29	1	79.20	79.79	79.21	79.38	79.74	80.00
	2	79.22	79.71	79.48	79.49	80.01	80.20
	3	79.44	79.85	79.42	79.60	80.54	79.76
	4	79.28	79.63	79.36	79.37	80.49	79.23
	5	79.15	79.77	79.36	79.25	79.84	79.94
	6	79.40	77.83	79.39	79.31	79.84	79.38
	7	79.47	79.72	79.42	79.42	79.08	79.69
	8	79.60	79.77	79.20	79.36	79.54	79.43
	9	79.51	79.36	79.22	79.38	79.39	80.04
	10	79.55	79.63	79.26	79.42	79.71	79.70
30	1	78.30	78.67	78.24	78.48	79.03	79.03
	2	78.22	78.81	77.87	78.20	78.51	79.02
	3	78.48	78.85	77.82	78.03	79.46	78.53
	4	78.40	78.68	77.96	77.89	78.61	78.69
	5	78.37	78.59	78.22	78.01	79.74	78.62
	6	78.51	78.71	78.62	78.59	78.71	78.55
	7	78.40	78.59	78.58	78.40	79.62	79.47
	8	78.42	78.68	78.50	78.31	78.44	78.82
	9	78.67	78.80	77.90	78.09	78.60	79.46
	10	78.61	78.81	77.82	78.21	79.13	79.05
31	1	79.69	78.02	77.40	77.27	78.47	78.39
	2	78.02	78.28	77.60	77.83	79.16	77.96
	3	77.83	78.27	77.58	77.82	77.71	78.15
	4	77.77	78.13	77.64	77.80	78.99	78.83
	5	77.91	78.21	77.42	77.59	78.73	77.59
	6	78.00	78.14	77.51	77.54	78.96	78.51
	7	77.98	78.35	77.62	77.65	78.77	78.14
	8	78.27	78.15	77.80	77.81	79.34	79.33
	9	78.11	78.16	77.53	77.42	79.53	77.97
	10	78.21	78.24	77.64	77.77	78.71	78.29

Table 7.7 continued

32	1	77.98	78.31	77.63	77.76	79.07	77.91
	2	77.81	78.39	77.59	77.62	78.54	78.10
	3	77.83	78.45	77.60	77.62	78.44	78.02
	4	77.89	78.34	78.00	77.78	78.59	78.52
	5	78.04	78.18	77.78	77.80	78.87	78.05
	6	77.95	78.35	77.87	77.90	78.99	78.25
	7	77.97	78.05	77.75	77.64	78.72	79.07
	8	78.00	78.11	77.67	77.78	77.93	78.69
	9	78.01	78.44	77.83	77.74	78.81	78.62
	10	78.24	78.36	77.82	77.85	78.55	78.31
33	1	77.05	77.81	77.54	77.41	78.14	77.44
	2	77.23	77.45	77.25	77.14	78.25	77.30
	3	77.32	77.57	77.27	77.22	78.34	78.41
	4	77.34	77.56	77.42	77.98	77.89	78.22
	5	77.34	77.46	77.42	77.20	77.73	77.52
	6	77.24	77.63	77.40	77.37	77.85	77.45
	7	77.46	77.54	77.40	77.40	78.37	77.57
	8	77.45	77.71	77.51	77.43	78.33	77.60
	9	77.57	77.70	77.35	77.27	77.26	78.00
	10	77.24	77.67	77.39	77.43	77.89	77.67
34	1	78.98	79.55	79.52	79.54	79.52	79.97
	2	79.03	79.56	78.98	79.29	79.80	80.14
	3	79.15	79.54	78.99	79.24	79.12	79.63
	4	79.32	79.59	79.11	79.21	80.39	78.91
	5	79.32	79.48	78.98	79.17	79.83	79.88
	6	79.09	79.63	79.20	79.21	80.26	79.32
	7	79.49	79.56	79.18	79.06	80.05	79.76
	8	79.58	79.48	79.14	79.24	80.80	80.10
	9	79.34	79.65	79.02	79.00	81.18	79.14
	10	79.56	79.64	79.17	79.02	80.33	79.95
35	1	76.95	77.39	77.26	77.25	78.19	76.77
	2	76.99	77.51	76.96	77.16	78.12	77.49
	3	77.21	77.47	76.96	77.15	78.16	77.80
	4	77.04	77.43	77.02	77.20	77.95	78.53
	5	77.15	77.40	77.00	77.20	77.74	77.35
	6	76.95	77.52	77.26	77.23	79.05	77.22
	7	77.17	77.45	77.20	77.19	78.96	77.64
	8	77.14	77.34	76.79	77.00	77.53	77.44
	9	77.27	77.51	77.23	77.39	77.65	77.91
	10	77.28	77.35	77.23	77.28	78.27	76.96

Table 7.7 continued

36	1	77.23	77.47	77.03	77.22	78.58	78.25
	2	77.36	77.54	76.68	77.20	77.10	77.65
	3	77.24	77.81	76.72	77.15	78.05	78.95
	4	77.36	77.67	77.18	77.22	78.40	77.32
	5	77.56	77.47	76.99	77.17	78.70	78.02
	6	77.53	77.58	77.20	77.27	77.45	77.64
	7	77.54	77.70	77.02	77.16	78.62	77.70
	8	77.62	77.74	77.27	77.23	77.79	77.53
	9	77.49	77.83	77.12	77.67	77.60	77.71
	10	77.39	77.79	77.18	77.53	78.67	77.79
37	1	73.75	74.45	73.80	73.66	75.19	74.26
	2	74.03	74.46	73.75	73.83	75.38	74.40
	3	74.19	74.37	73.76	73.80	74.63	73.86
	4	73.93	74.50	73.62	73.84	74.12	74.08
	5	74.26	74.50	73.57	73.80	74.40	74.04
	6	74.13	74.45	73.74	73.76	74.89	74.58
	7	74.13	74.50	73.31	73.82	73.96	74.40
	8	74.07	74.56	73.48	73.64	74.09	74.16
	9	74.29	74.55	73.78	73.90	75.09	73.90
	10	74.36	74.70	73.71	73.82	74.97	74.61

Table 7.8 Experiment 8: effect of wet and dry cycles on hectolitre mass (HLM) values

Sample	Moisture	Rep.	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
Original moisture content										
5	11.2	1	85.70	83.20	82.81	82.59	80.88	80.84	82.60	82.56
		2	84.97	83.20	82.83	82.59	80.71	81.10	82.20	82.43
7	12.1	1	82.43	81.00	80.28	80.79	78.49	78.48	80.20	79.71
		2	82.31	80.50	80.49	80.79	78.28	78.73	80.80	79.97
13	11.3	1	78.67	77.00	76.25	76.88	74.54	75.07	76.40	76.23
		2	78.72	77.10	76.76	76.88	74.59	75.05	76.80	76.36
17	11.8	1	82.00	80.30	79.77	80.29	77.82	78.06	80.60	79.46
		2	81.70	80.50	80.06	80.39	78.00	78.14	80.80	79.59
Original to 14% moisture content										
5	14.1	1	81.03	79.10	78.23	78.08	76.20	76.57	78.20	77.52
		2	80.42	79.50	78.56	78.79	76.40	76.85	78.80	78.55
7	14.7	1	76.31	75.10	75.19	76.18	73.26	73.30	75.40	74.42
		2	77.36	76.00	75.26	75.78	72.97	73.16	75.60	74.93
13	14.1	1	76.42	74.50	73.69	74.08	72.24	72.32	74.80	73.38
		2	76.72	75.00	74.12	74.48	72.53	72.06	74.20	74.16
17	13.8	1	78.71	77.50	77.03	76.98	75.01	75.07	77.40	76.10
		2	78.53	77.70	77.33	77.38	75.13	74.90	77.20	77.13
Original to 16% moisture content										
5	15.7	1	78.27	76.30	75.49	75.58	74.34	73.87	76.40	74.93
		2	79.49	77.00	76.46	76.88	74.27	74.13	76.80	76.74
7	16.1	1	74.95	73.70	73.03	73.07	70.90	70.70	73.60	71.83
		2	76.22	73.70	73.22	73.88	71.27	70.80	73.40	73.00
13	15.7	1	75.38	73.10	72.05	72.47	70.38	71.07	72.80	71.57
		2	75.81	73.50	72.53	73.07	70.59	70.90	73.60	72.61
17	15.3	1	77.59	75.30	75.04	74.78	72.71	73.25	75.60	74.03
		2	77.91	76.00	75.57	75.58	72.83	73.57	75.80	75.19
Original to 18% moisture content										
5	16.7	1	74.82	73.70	73.18	72.97	71.20	71.81	74.40	72.09
		2	76.76	75.10	74.59	74.68	71.53	71.85	75.20	74.42
7	17.7	1	74.88	73.30	71.87	72.47	70.00	70.89	72.80	71.19
		2	75.64	73.70	72.91	73.58	70.43	70.89	73.40	72.61
13	17.0	1	74.22	72.00	71.16	70.87	69.29	68.81	71.80	70.02
		2	73.61	72.00	71.56	71.87	69.39	69.01	72.00	71.57
17	16.7	1	75.60	73.30	72.76	72.47	71.25	70.54	73.20	71.83
		2	76.19	74.00	73.96	73.78	71.40	71.09	73.80	73.64
Original moisture content to 10% moisture content										
5	9.8	1	86.21	83.60	83.50	82.79	81.41	81.23	83.20	82.82
		2	85.61	83.40	82.94	82.59	80.94	81.13	82.80	82.56
7	10.6	1	82.98	81.00	80.40	80.79	78.38	78.58	80.60	80.23
		2	83.00	81.00	80.31	80.89	78.56	78.66	80.60	79.84
13	10.2	1	79.98	77.50	77.02	77.18	75.20	75.62	77.20	76.74
		2	79.61	77.50	76.70	77.28	74.91	75.29	77.40	76.74
17	10.4	1	83.84	81.00	80.30	80.49	78.45	78.50	80.60	79.97
		2	83.89	80.50	80.22	80.59	78.38	78.80	80.40	79.84

Table 7.8 continued

14% moisture content to 10% moisture content										
7	9.5	1	79.16	76.50	75.99	76.58	74.26	74.26	75.80	75.19
		2	79.44	76.30	76.07	76.68	73.98	74.17	76.00	75.32
13	9.9	1	77.95	75.70	74.92	75.28	73.00	73.53	75.40	74.55
		2	78.33	76.00	75.31	75.58	73.36	73.33	75.40	74.93
17	9.4	1	82.05	79.10	78.34	78.79	76.41	76.44	78.60	77.91
		2	81.29	79.00	78.66	78.99	76.72	76.48	78.60	78.16
16% moisture content to 10% moisture content										
7	10.8	1	77.11	75.30	74.66	75.28	72.84	73.17	75.20	74.03
		2	77.29	75.50	75.05	75.38	73.39	72.88	75.00	74.55
13	10.8	1	76.80	75.30	74.58	74.88	72.93	73.22	74.80	74.16
		2	76.76	75.30	74.59	75.18	73.09	73.10	75.00	74.68
17	10.6	1	80.82	78.00	77.25	77.58	75.39	75.61	77.60	76.61
		2	80.02	78.00	77.37	77.68	75.23	75.57	77.80	76.87
18% moisture content to 10% moisture content										
7	9.9	1	77.11	74.30	74.18	74.88	71.30	72.67	74.40	72.87
		2	76.02	75.00	74.44	75.58	71.54	72.70	74.80	73.90
13	9.8	1	75.81	74.00	73.18	73.88	71.26	71.57	73.60	72.48
		2	75.42	74.10	73.91	74.38	71.67	71.83	74.00	73.38
17	9.5	1	78.63	76.70	75.99	76.78	74.13	74.31	76.80	75.32
		2	78.71	77.00	76.26	77.08	74.52	74.41	76.60	75.84
10% moisture content to original moisture content										
5	11.2	1	83.57	82.00	81.33	81.19	79.26	79.44	81.00	80.88
		2	83.65	82.20	81.12	81.09	79.23	79.47	81.20	81.14
7	11.7	1	81.73	80.10	79.42	79.89	77.69	77.94	79.60	79.33
		2	81.30	80.70	79.56	79.99	77.36	77.59	79.20	79.20
13	11.0	1	78.72	77.50	76.72	76.88	75.04	74.84	76.60	76.36
		2	79.02	77.10	76.86	76.88	75.04	75.09	76.80	76.61
17	11.6	1	81.24	79.30	78.85	78.99	76.70	77.18	79.40	78.29
		2	81.09	79.30	78.97	79.29	77.06	77.21	79.00	78.81
10% moisture content to 14% moisture content										
7	14.5	1	76.60	75.10	74.60	74.88	72.80	72.95	74.80	74.03
		2	76.18	75.30	74.78	74.98	72.67	72.63	74.80	74.29
13	13.7	1	76.72	75.70	74.85	74.98	73.04	73.43	74.80	74.68
		2	76.60	75.70	75.05	75.08	73.11	73.28	75.00	74.93
17	13.6	1	79.26	77.50	77.16	76.88	75.39	75.29	76.80	76.48
		2	79.18	77.70	77.15	77.08	75.03	75.08	77.20	76.74
10% moisture content to 16% moisture content										
7	15.7	1	76.24	74.10	73.33	73.58	71.45	71.66	74.00	72.74
		2	76.15	74.00	73.95	74.18	71.58	71.93	74.00	73.38
13	15.2	1	76.38	74.70	74.09	73.68	72.06	72.40	74.40	74.03
		2	75.71	75.00	73.99	74.08	72.38	72.42	74.60	74.29
17	16.1	1	77.08	74.10	73.87	73.78	71.91	71.81	74.40	73.64
		2	76.46	74.10	74.56	74.38	72.12	71.98	74.80	74.29
10% moisture content to 18% moisture content										
7	16.9	1	74.29	72.70	71.40	71.57	69.91	69.20	72.40	70.67
		2	75.45	72.30	72.85	72.67	70.13	69.92	72.80	72.22
13	17.0	1	73.17	71.10	70.06	70.07	69.24	68.38	71.20	70.02
		2	73.92	71.30	71.19	71.07	68.93	69.08	71.40	70.54
17	17.3	1	74.11	71.70	71.26	71.37	69.52	70.69	72.20	70.93
		2	74.73	72.30	72.81	72.67	69.88	70.52	72.00	72.61

Appendix 8

Table 8.1 Experiment 1: repeatability within the respective hectolitre mass (HLM) devices

Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
81.53	76.32	81.18	76.98	77.38	77.63	78.20	77.86
82.07	76.65	81.23	77.18	76.95	77.53	78.00	77.73
81.64	76.14	81.59	77.48	76.91	77.60	78.20	77.22
81.54	76.84	81.44	77.48	77.42	77.71	77.60	77.22
81.81	76.87	81.48	77.28	77.68	77.65	78.20	77.73
81.94	76.44	81.64	77.68	77.13	78.06	77.80	77.35
81.55	76.81	81.26	77.58	76.98	77.77	78.40	77.48
81.17	76.77	81.30	77.48	77.78	77.92	78.40	77.48
81.75	76.68	81.68	77.18	77.33	77.97	78.00	77.48
81.54	76.50	81.66	77.48	77.48	77.92	77.60	77.73

Table 8.2 Experiment 2: effect of repeated analysis of the same maize sample on its hectolitre mass (HLM)

Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
Sample 3							
80.04	75.56	79.77	76.38	76.58	76.50	76.80	76.45
80.57	75.60	79.99	76.08	76.95	76.59	76.40	76.58
80.73	75.62	80.00	76.08	76.71	76.62	76.60	76.83
80.35	75.69	79.91	76.48	77.16	76.10	77.00	76.96
80.94	75.39	79.71	76.18	76.46	76.35	77.00	76.58
80.47	75.63	80.00	76.58	76.82	76.50	76.40	76.45
80.45	75.71	80.03	76.38	76.93	76.51	76.60	76.71
80.37	75.55	79.99	76.38	77.18	76.68	76.60	76.83
80.52	75.58	79.90	76.48	77.41	76.21	76.40	76.83
80.60	75.73	80.12	76.18	77.54	76.73	76.40	76.45
Sample 4							
81.81	76.60	81.41	77.38	77.93	77.15	78.60	77.48
82.16	76.51	81.40	77.68	77.45	77.22	78.40	77.86
82.11	77.10	81.62	77.68	78.02	77.59	77.40	77.61
81.45	76.46	81.57	77.68	77.98	77.91	78.00	77.22
81.43	76.81	81.30	77.78	77.89	77.78	78.00	76.96
81.75	76.24	81.49	77.58	78.07	77.51	77.80	77.48
81.82	75.80	81.56	77.48	77.53	77.61	77.80	77.48
81.77	76.98	81.50	77.38	77.74	77.71	78.40	77.09
81.56	76.57	81.65	77.78	78.14	77.39	78.00	77.73
82.16	76.92	81.45	77.28	77.69	77.31	78.60	77.61
Sample 5							
77.59	73.38	77.58	73.78	73.90	74.67	74.40	74.13
77.36	73.55	77.28	73.78	74.70	74.39	74.20	74.13
77.43	73.17	77.68	73.88	74.87	74.62	74.60	74.39
77.60	73.71	77.35	74.18	74.44	74.79	74.20	74.00
77.55	73.18	77.44	73.88	74.90	74.22	74.40	74.39
77.27	73.65	77.69	73.68	74.18	74.32	74.20	74.13
78.02	73.76	77.74	74.08	74.41	74.80	74.20	74.39
77.96	73.01	77.58	74.08	74.84	74.20	74.20	74.13
77.53	73.94	77.36	73.78	74.90	74.73	74.20	74.39
77.62	72.78	77.67	73.98	74.64	74.95	74.40	74.39

Table 8.3 Experiment 3: determination of variation in hectolitre mass (HLM) within and between HLM devices

Sample	Sub-	Rep.	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
1	a	1	81.43	76.03	81.26	76.98	76.66	77.04	76.80	76.45
	a	2	81.34	75.74	80.71	76.98	76.57	76.27	76.80	76.83
	b	1	81.42	76.10	81.13	77.28	76.89	77.23	77.20	76.96
	b	2	80.74	76.44	80.72	77.18	76.48	76.81	77.60	76.58
	c	1	82.08	75.85	80.84	77.08	77.07	76.62	77.20	76.71
	c	2	81.91	75.33	81.16	76.88	77.37	76.98	77.60	76.83
2	a	1	80.91	76.38	81.04	77.58	77.17	77.14	76.80	77.22
	a	2	81.17	76.34	81.35	77.28	77.36	77.40	77.20	77.35
	b	1	81.61	76.02	81.41	77.68	77.29	76.74	77.20	78.64
	b	2	81.75	76.22	81.46	77.58	77.46	77.10	77.20	77.35
	c	1	81.50	76.11	81.25	77.48	76.89	77.10	76.80	78.38
	c	2	82.09	76.16	81.26	77.08	76.79	76.97	77.20	77.35
3	a	1	80.27	75.19	80.23	75.98	77.06	76.16	76.00	76.32
	a	2	79.77	75.66	80.08	76.18	76.88	76.16	76.40	76.71
	b	1	80.41	75.44	79.91	76.18	77.11	76.76	76.40	76.96
	b	2	80.17	76.04	79.72	76.48	77.12	76.18	76.40	76.71
	c	1	80.23	75.98	79.68	76.28	76.79	76.48	76.00	76.45
	c	2	80.24	75.29	80.19	76.38	77.17	76.68	76.00	76.32
5	a	1	78.55	74.02	78.63	74.08	74.01	73.93	73.60	74.65
	a	2	78.34	73.80	78.72	74.18	74.90	73.82	73.60	74.65
	b	1	78.11	73.50	77.74	74.38	73.79	74.69	74.00	74.39
	b	2	78.01	73.55	77.64	74.28	74.45	74.41	74.00	74.26
	c	1	78.27	73.81	77.75	74.38	74.57	76.48	74.40	74.26
	c	2	78.12	73.19	77.55	74.68	74.71	74.60	74.00	74.39
6	a	1	81.37	76.45	81.16	77.68	77.74	76.61	77.60	77.48
	a	2	81.88	75.91	81.21	77.68	77.18	77.35	78.00	77.22
	b	1	81.81	76.49	81.33	77.38	77.63	77.79	76.80	77.09
	b	2	82.13	75.94	81.53	77.78	77.39	77.89	76.80	77.86
	c	1	81.69	76.60	81.61	77.98	77.40	77.58	77.20	77.86
	c	2	81.31	76.50	81.26	77.68	77.46	77.33	77.60	77.09

Table 8.3 continued

7	a	1	82.49	77.12	81.90	77.78	78.04	78.22	78.80	78.25
	a	2	81.79	77.48	82.37	77.98	78.60	77.63	78.40	78.38
	b	1	82.16	77.10	82.01	78.08	77.99	78.32	78.00	77.35
	b	2	82.64	77.05	82.02	77.98	78.09	77.97	77.60	78.12
	c	1	82.42	77.38	81.94	78.08	78.40	78.39	77.20	76.83
	c	2	82.23	77.52	81.72	77.88	78.12	77.38	77.20	78.25
8	a	1	82.78	77.81	82.37	78.69	79.54	78.88	78.40	78.64
	a	2	82.30	77.93	82.32	78.49	78.85	78.66	78.40	78.64
	b	1	82.48	77.64	82.03	78.08	79.26	78.80	78.80	79.02
	b	2	82.48	77.77	81.87	78.59	79.00	78.76	78.40	78.51
	c	1	82.46	78.04	82.23	78.89	78.39	78.66	78.40	78.38
	c	2	82.06	77.52	82.28	78.39	78.21	78.98	78.40	79.02
9	a	1	79.76	75.03	79.78	75.68	75.93	75.67	75.60	76.19
	a	2	79.56	75.51	80.26	75.58	75.87	75.05	75.20	76.32
	b	1	80.06	75.04	79.71	75.88	75.07	75.53	75.60	76.19
	b	2	80.00	75.11	80.04	75.98	75.24	75.93	76.00	76.19
	c	1	79.72	75.43	79.94	76.08	76.40	75.81	76.00	75.93
	c	2	79.98	74.97	80.04	75.88	76.06	75.52	76.00	76.06
10	a	1	75.49	71.57	75.01	72.27	72.50	71.61	72.00	71.94
	a	2	75.26	71.24	74.94	72.07	72.51	71.51	72.00	72.07
	b	1	75.90	71.42	74.79	71.77	70.43	71.57	72.00	72.07
	b	2	75.46	71.24	75.13	72.07	72.83	71.82	72.40	72.07
	c	1	75.62	70.74	75.13	71.67	72.88	72.28	72.00	71.69
	c	2	75.47	71.19	74.94	72.07	72.26	71.88	72.40	71.81
11	a	1	81.53	76.18	80.37	77.08	77.11	76.92	77.20	77.48
	a	2	80.93	75.78	80.23	76.88	77.33	76.96	76.40	76.96
	b	1	80.86	75.88	80.73	76.98	76.64	76.43	77.20	77.22
	b	2	81.94	75.90	80.92	77.18	76.81	76.15	76.80	77.22
	c	1	80.89	76.33	80.94	77.38	77.86	76.66	77.20	76.96
	c	2	80.56	76.00	80.84	77.28	77.37	76.98	77.20	76.96

Table 8.4 Experiment 4: comparison of hectolitre mass (HLM) devices using a single work sample of maize

Sample	Sub-	Rep.	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
1	a	1	81.28	75.73	80.89	76.68	77.46	76.98	77.60	76.32
	a	2	81.16	75.81	80.75	76.58	76.81	76.89	77.20	76.58
	b	1	81.65	76.30	80.42	77.18	76.65	77.18	76.80	76.83
	b	2	81.36	75.54	80.48	77.18	77.05	76.19	77.60	76.45
	c	1	80.92	76.20	80.16	76.98	76.46	76.66	77.20	76.45
	c	2	81.41	75.99	80.65	77.38	77.71	77.03	76.40	76.96
2	a	1	82.18	76.82	81.49	77.18	77.91	77.08	78.00	77.35
	a	2	81.77	77.02	81.64	77.48	77.87	78.17	76.40	77.48
	b	1	81.66	76.73	80.80	77.38	77.48	77.42	77.60	77.61
	b	2	81.67	76.39	81.55	77.38	77.78	77.06	78.40	77.99
	c	1	81.89	76.21	81.18	77.08	77.52	76.89	78.00	77.22
	c	2	81.77	76.28	80.98	77.88	78.65	77.70	77.60	77.73
3	a	1	80.07	75.81	80.11	76.18	76.60	76.36	76.40	76.19
	a	2	79.98	76.32	80.09	76.08	76.59	76.76	76.00	76.71
	b	1	80.13	75.96	79.45	75.18	76.08	76.68	76.80	76.71
	b	2	79.95	76.47	79.78	75.78	76.34	76.04	76.00	76.83
	c	1	79.80	75.29	79.74	75.88	76.72	76.29	76.40	76.45
	c	2	79.79	75.67	79.71	76.08	76.29	76.78	76.40	76.19
5	a	1	78.54	73.35	78.31	74.28	74.96	75.40	74.00	74.39
	a	2	78.90	73.67	77.98	74.48	74.68	74.99	74.80	74.65
	b	1	78.73	73.67	77.76	74.38	74.98	75.07	74.40	74.77
	b	2	78.44	73.28	78.23	74.48	74.77	74.47	74.78	74.39
	c	1	78.84	73.94	77.70	74.68	74.96	74.82	73.20	74.39
	c	2	78.55	73.31	77.58	74.18	75.02	74.65	74.00	74.77
6	a	1	81.78	76.52	81.17	77.08	77.14	76.98	77.60	76.96
	a	2	81.23	76.12	81.05	77.18	77.82	76.89	77.20	77.09
	b	1	82.23	76.41	81.30	77.28	77.89	77.18	77.20	77.48
	b	2	81.75	76.62	81.44	77.18	77.40	76.19	77.20	77.35
	c	1	81.85	76.62	81.44	77.58	77.94	76.66	78.00	77.73
	c	2	81.73	76.41	81.43	77.48	77.57	77.03	78.00	77.73

Table 8.4 continued

7	a	1	82.28	77.05	81.61	78.25	78.19	78.02	78.80	78.25
	a	2	82.04	77.22	81.91	78.25	77.87	78.19	78.00	78.25
	b	1	82.53	76.96	81.88	78.25	78.64	78.20	78.40	78.25
	b	2	82.36	77.52	81.99	78.25	78.71	78.07	78.80	78.25
	c	1	82.29	76.91	81.29	78.38	78.34	78.93	78.00	78.38
	c	2	81.99	77.23	81.72	78.25	77.92	78.11	78.00	78.25
8	a	1	82.64	77.67	82.34	78.69	79.60	79.20	78.00	79.28
	a	2	82.56	78.11	82.77	78.49	79.30	79.65	78.80	79.54
	b	1	82.48	77.73	82.40	78.69	78.93	79.06	78.40	79.02
	b	2	82.83	78.16	82.22	78.19	78.32	78.48	78.40	79.28
	c	1	82.94	78.00	82.46	78.49	79.02	78.81	77.60	79.28
	c	2	82.29	77.57	82.25	78.59	78.88	78.44	78.00	79.15
9	a	1	80.92	75.32	79.64	75.68	76.20	76.26	75.60	76.19
	a	2	81.41	76.21	79.68	75.88	76.53	76.44	75.60	75.80
	b	1	80.74	75.57	79.40	76.48	76.13	75.86	75.60	76.45
	b	2	80.96	75.23	79.84	75.68	76.28	76.30	76.00	76.32
	c	1	80.36	75.41	79.70	75.88	75.58	75.75	74.80	75.93
	c	2	80.56	74.81	80.05	75.78	76.40	76.11	75.20	76.32
10	a	1	75.34	71.56	74.64	71.47	71.99	71.63	72.00	72.07
	a	2	75.35	71.05	74.92	71.77	72.16	71.78	71.60	72.46
	b	1	75.58	71.33	74.96	72.17	72.38	72.24	72.00	71.94
	b	2	75.66	71.37	75.08	71.87	72.54	72.08	72.00	72.33
	c	1	75.79	71.75	74.85	71.87	72.66	71.97	71.60	71.81
	c	2	75.35	71.83	74.86	72.37	71.90	72.21	72.80	72.20
11	a	1	82.28	75.90	80.60	76.48	77.10	76.91	76.80	76.45
	a	2	82.04	76.09	80.44	76.78	77.55	76.91	76.80	77.09
	b	1	82.53	76.23	80.49	76.68	76.54	76.90	76.40	76.83
	b	2	82.36	76.10	80.36	76.48	76.98	76.59	77.20	77.09
	c	1	82.29	76.34	80.56	76.88	77.25	77.13	76.40	76.58
	c	2	81.99	76.71	80.73	77.18	77.10	76.55	76.40	77.22

Table 8.5 Experiment 5: effect of impurities on hectolitre mass (HLM) determinations of maize

Sample	Rep.	Australia	Canada	France	Germany	SA 1	SA 2	UK	USA
Before impurities have been removed									
1	1	81.28	78.50	80.89	76.68	77.46	76.98	77.60	76.32
	2	81.16	79.00	80.75	76.58	76.81	76.89	77.20	76.58
2	1	82.18	79.70	81.49	77.18	77.91	77.08	78.00	77.35
	2	81.77	80.00	81.64	77.48	77.87	78.17	76.40	77.48
3	1	80.07	78.70	80.11	76.18	76.60	75.40	76.40	76.19
	2	79.98	79.30	80.09	76.08	76.59	74.99	76.00	76.71
5	1	78.20	76.00	77.64	73.98	74.18	74.78	73.80	73.36
	2	78.22	76.50	78.07	74.68	74.60	73.92	75.20	74.13
6	1	81.78	79.50	81.17	77.08	77.14	78.02	77.60	76.96
	2	81.23	79.20	81.05	77.18	77.82	78.19	77.20	77.09
7	1	82.28	80.00	81.61	78.25	78.19	79.20	78.80	78.25
	2	82.04	80.10	81.91	78.25	77.87	79.65	78.00	78.25
8	1	82.64	80.70	82.34	78.69	79.60	79.20	78.00	79.28
	2	82.56	81.20	82.77	78.49	79.30	79.65	78.80	79.54
9	1	80.92	79.10	79.64	75.68	76.20	76.26	75.60	76.19
	2	81.41	79.00	79.68	75.88	76.53	76.44	75.60	75.80
10	1	75.34	74.30	74.64	71.47	71.99	71.63	72.00	72.07
	2	75.35	73.70	74.92	71.77	72.16	71.78	71.60	72.46
11	1	82.28	78.70	80.60	76.48	77.10	78.02	76.80	76.45
	2	82.04	79.00	80.44	76.78	77.55	78.19	76.80	77.09
After impurities have been removed									
1	1	81.40	79.00	81.04	76.38	76.83	76.90	77.40	77.35
	2	81.56	79.10	81.14	76.68	76.79	77.27	76.80	76.96
2	1	80.77	79.30	80.57	76.78	77.17	76.58	77.20	76.96
	2	80.96	79.00	80.83	76.98	76.94	77.20	77.20	76.83
3	1	80.77	79.70	80.72	76.38	77.90	77.16	77.00	77.09
	2	81.11	80.00	80.69	76.58	77.74	77.56	77.20	77.61
5	1	78.32	77.00	78.21	74.58	74.76	75.04	74.60	75.03
	2	78.28	77.10	78.33	74.68	75.48	75.12	74.80	75.42
6	1	81.87	79.30	81.65	77.48	77.50	77.17	78.20	77.22
	2	82.18	79.70	81.32	77.48	77.51	78.30	78.20	77.61
7	1	82.09	80.50	82.51	78.29	78.93	78.73	78.40	78.12
	2	82.89	80.70	82.05	78.08	78.85	78.92	78.20	78.51
8	1	82.93	81.60	82.57	78.89	79.88	80.02	79.40	79.54
	2	82.78	81.80	82.99	78.99	80.32	79.35	79.00	79.54
9	1	81.54	79.00	80.52	76.48	77.73	76.79	77.00	77.09
	2	81.25	79.30	80.77	76.58	77.44	77.36	77.40	77.61
10	1	75.37	74.10	74.92	71.77	72.61	72.29	72.00	72.20
	2	75.78	74.50	74.92	71.57	72.40	72.24	72.20	71.81
11	1	81.75	80.10	81.32	77.58	78.25	78.03	77.40	77.73
	2	81.44	80.10	81.58	77.48	77.74	77.66	78.80	77.86