Landbrugsministeriet
Statens Planteavlsforsøg

Moisture determination in cereals, analytical details, oven drying

Part I. Handling of intact kernels and loading of oven
Part II. Disintegration, loss of moisture, sampling of meal, disharmony near predrying

Vandbestemmelse i korn, analytiske detaljer vedrørende ovntørring

Part I. Belastning af tørreskab, håndtering af hel kerne og rensning
Part II. Formaling, vandtab, udtagning af melprøver, disharmoni i omegnen af fortørring

Chresten Sørensen
Research Centre for Agriculture
Department of Plant Nutrition and Psysiology
Research Centre Foulum
DK-8830 Tjele

Tidsskrift for Planteavls Specialserie

Beretning nr. S 2222 - 1992

Landbrugsministeriet
Statens Planteavlsforsøg

Moisture determination in cereals, analytical details, oven drying

Part I. Handling of intact kernels and loading of oven
Part II. Disintegration, loss of moisture, sampling of meal, disharmony near predrying

Vandbestemmelse i korn, analytiske detaljer vedrørende ovntørring

Part I. Belastning af tørreskab, håndtering af hel kerne og rensning
Part II. Formaling, vandtab, udtagning af melprøver, disharmoni i omegnen af fortørring

Chresten Sørensen
Research Centre for Agriculture
Department of Plant Nutrition and Psysiology
Research Centre Foulum
DK-8830 Tjele

Tidsskrift for Planteavls Specialserie

Beretning nr. S 2222 - 1992

Contents Part I

Moisture determination in cereals, analytical details in oven drying. I. Handling of intact kernels and loading of oven

Vandbestemmelse i korn, analytiske detaljer vedrørende ovntørring. I. Belastning af tørreskab, håndtering af hel kerne og rensning

Chresten Sørensen

Summary

Results from investigations on steps in handling kernel samples for determination of moisture are reported. The loss of moisture while mixing and dividing kernel samples with less than 17 per cent of moisture was relatively small. The importance of even distribution of the sample at predrying was demonstrated. Predrying at 130°C for 7 minutes or at 35°C and up to 65°C for 16 hours affected the loss of moisture significantly. The total moisture content was not significantly different for 130, 65 or 55°C at the predrying mentioned. For ovens of the same type the time for warming up differed and the effect of drying varied from one oven to another by shortly 0.1 per cent moisture. There was only a tendency that different placement of samples in the oven affected the results. Loading of the oven from 110 to 460 cm^3 per 1 g meal did not affect the results but the time for reaching the 130°C after incertion of samples varied from 95 to 40 minutes. Few samples from predried material dried together with more samples from not predried material and vice versa did not influence the results. Barley, wheat, and oat were wetted reaching from 14 to 36 per cent moisture. Samples from this material with more than 17 per cent moisture lost from 0.15 to 0.30 per cent of moisture by cleaning, with the highest figure for oat, for which also the standard deviation on moisture determination was highest. The loss of moisture at predrying formed a linear relationship with the total moisture content.

Key words: Cereals, moisture analysis, predrying, cleaning.

Resumé

Der er meddelt resultater fra undersøgelser vedrørende behandling af kerneprøver til bestemmelse af vand. For kornprøver med mindre end 17 procent vand var vandtabet under blanding og neddeling af prøven relativt beskedent. Betydningen af jævn fordeling af prøven under fortørring er påvist. Fortørring ved 130°C i 7 minutter eller ved 35°C og op til 65°C i 16 timer influerede væsentligt på vandtabet. Det totale vandindhold var ikke signifikant forskelligt for de nævnte fortørringstemperaturer 130, 65 og 55°C. Opvarmningstiden for forskellige tørreskabe af samme type varierede, og tørringseffekten fra eet tørreskab til et andet afveg med knap 0,1 procent vand. Der var kun en tendens til, at forskellig placering af prøverne i tørreskabet influerede på resultatet. Belastning af tørreskabet fra 110 til 460 cm^3 pr. 1 g mel influerede ikke på resultaterne, men tiden for at opnå de 130°C efter indsætning af prøver varierede fra 95 til 40 minutter. Resultaterne var upåvirket af, om få fortørrede prøver blev tørret sammen med flere ikke fortørrede eller omvendt. Byg, hvede og havre blev fugtet til 14-36 procent vand, for prøver med mere end 17 procent vand var vandtabet under rensning 0,15-0,30 procentenheder, med de største tab for havre, for hvilke også standardafvigelsen for vandbestemmelsen var størst. Tabet af vand ved fortørring var en lineær funktion af det totale vandindhold.

Nøglerd: Korn, vandanalyse, fortørrring, rensning.

Introduction

The moisture content in cereal seeds is an important characteristic from different points of view. Among others it informs whether the material is suitable for harvest, storage, certain industrial items, it informs about the nutritional value and it influences on the price. Therefore the actuality of determination of moisture in cereals arises in many occasions. Difficulties in performing samples from a batch of cereals may be the primary cause of discrepancies among analytical results from the same batch. Among others Hummel-Gumælius (6) reports a standard deviation of 0.37 per cent protein for sampling from car-loads compared to 0.07 per cent for the laboratory side. But it is important that the description for moisture determination is so detailed that equal results can be obtained in different laboratories when analysing homogenous samples of the same material with the same method. Several modern methods for moisture determination have appeared, Karmas (7) and Wilson (14), but the oven drying method is still used to a great extent in analytical laboratories, and it is used as reference method for calibration of the modern apparatuses. The present investigations have been made concerning details in the various steps of the analytical procedure related to sample preparation in the laboratory, as well as cleaning and air oven drying for moisture determination. Results from investigations concerning grinding, optimizing sampling and the limit of predrying will be published in a separate paper (11).

Materials and methods

The cereal samples of wheat, barley, rye, and oat have preferably been collected from a commercial firm, where materials delivered from the field mainly have been in a reasonable good condition, and in the experimental work they have been worked on as received.

The samples have preferably been stored at 15°C, exceptionally at 5°C. Williams and Sigurdson (13) showed that a great loss of moisture might occur even when ground wheat was stored in untaped metal cans. In the present experiments intact kernels were stored in 0.08 mm polyethylene bags, which after filling in the kernels were straightened and bent to one side. After that the bag was folded and bound tightly by a string. After filling water into a bag and bound in this way and placed on the laboratory bench for 3 weeks a loss in weight of only 0.09 per mille per day was observed. Principally the ground materials were not stored. Temperature and humidity of the room were tested occasionally only, but may have been 20-25°C and 40-70 per cent relative humidity.

Larger samples (some kilos) of intact kernels were mixed for 2 hours in a rectangular box with quadratic cross section and turning on a diagonal axis. Small samples of 500 g or less were mixed crisscross repeated four times. Laboratory samples of 500-1000 g were subdivided by a conical divider or a multiple slot divider.

A winnowing machine was used for treatment of some samples. The kernel samples were fanned (4.5 m per s) free of chaff, and the machine had an upper sieve with 10 mm round holes and a bottom sieve with 1.5 times 20 mm openings.

Samples of 50 g for predrying were taken with a flat bottom shovel. Samples of about 25 g kernels for each grinding were taken with a flat bottom metal shovel, 2.5 cm wide and with 2.5 cm high rectangular sides and 6.2 cm long. The shovel contacted the sample support during sampling.

For disintegration of kernels the IKA-Analysenmühle A10 (Janke & Kunkel G. m.b.h. & Co. K. G.) was used. The volume of the grinding chamber is about 150 cm^3, two knives are placed at the bottom of the chamber and have a nominal speed of 20,000 rpm. The lower container and the lid are connected by a packing in process of grinding and so the connection is practically dust tight but unfortunately not absolutely air tight. This makes a problem in process of grinding because the vapour pressure of the moisture in the meal is greater than of that in the kernels, as quoted by Anderson (4) from Swanson and Anderson (10). The grinding chamber made from stainless steel is even and easy to clean, and air is not drawn through it in process of grinding. The meal was emptied on an aluminium paper foil to make a cone, and when twice 25 g of kernels were ground, the first portion was covered with aluminium paper foil or an upside down percelain bovl until the next 25 g were ground and emptied on the top of the first portion.

Samples of about 5 g meal for drying were taken by a shovel of the shape as mentioned for sampling of kernels, but 2 cm wide and with 2 cm high sides only. The shovel contacted the sample support during sampling.

In order to ascertain the particle distribution as smaller than 0.5 mm, 0.5 mm - 1.0 mm, and larger than 1 mm measured by quadratic holes, several samples of the ground material were sifted by means of a JEL-Prüfsiebmaschinen. The sieves were supplied with some 1 cm^3 rubber cubes during operation. The materials were ground so that more than 50 per cent of the particles by weight was smaller than 0.5 mm.

Containers for predrying of 50 g intact kernels consisted of thin metal dishes, 10 times 16 cm having 1 cm high sides and a weight of about 5 g each. Containers for drying meal consisted of cylindrical glasses, principally 5 cm wide and 3 cm high. The glasses were supplied with ground glass lids to fit inside and were placed almost vertical during drying and closed the containers at the end of the drying period. The glasses had a weight of about 50 g each. The material, about 226 mg of meal per 1 cm^2, was evenly distributed. After drying the samples were cooled in a desiccator with freshly dried silica-gel as desiccant for 45 minutes before weighing to the fourth decimal on a laboratory balance.

For drying of the samples three air ovens of the cylindrical Heræus type were applied. No. 1, volume 27.5 liter, 550 Watt, No. 2 and No. 3, volume 78.5 liter, 990 Watt and all with temperatureregulation of $\pm$ 1°C.

The principal temperature has been 130°C. This temperature may not be critical. Nelson and Hulett (8) did not find formation of gas or moisture in quantity from decomposition when cereals were heated up to 184°C for 4 hours.

The ovens had natural ventilation with a hole in the top of the cabinet. The temperature has been raised to 129-130°C before incertion of samples and the time of drying has been counted from the time after incertion of samples at which the temperature again had reached 129-130°C. The oven thermometer was incerted from the top of the oven and reached 4.5 cm inside. With loaded oven the temperature from 43 readings during 390 minutes varied from 129 to 130°C.

Experimental

Characteristic of ovens

The effect of the ovens mentioned were compared in respect of warming up time and drying efficiency. The ovens No. 1, No. 2, and No. 3 were heated to 130°C and 5 g samples of wheat meal with about 15 per cent of moisture were quickly incerted. The oven volume was 220 cm^3 per 1 g of meal. The temperatures of the ovens were then read at different times until 129°C was reached.

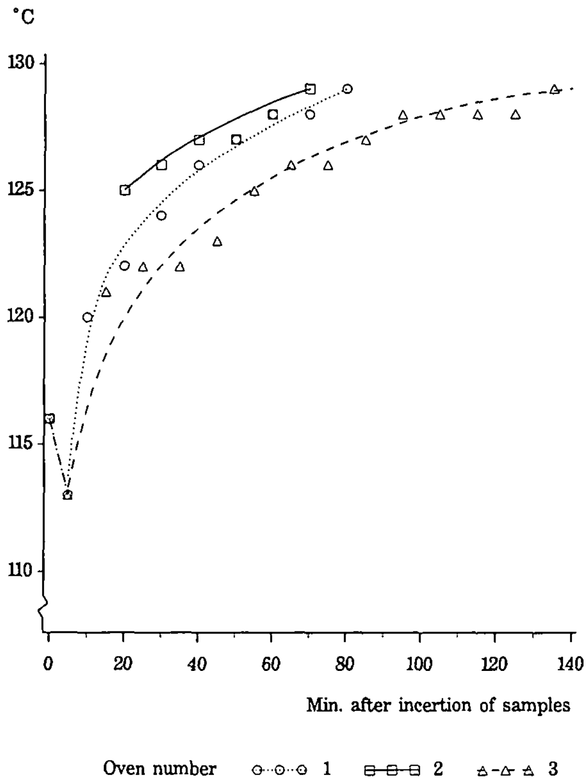

Fig. 1. Time for different ovens to recapture the temperature after incertion of the samples at 130°C. Oven volume was 220 cm^3 per 1 g of meal.

From Fig. 1 it can be seen that for oven No. 1, No. 2, and No. 3 it took 80, 70, and 135 minutes respectively to reach 129°C. Oven No. 3 has been used only when predrying overnight has been carried out. After incertion of the samples the time to recapture the temperature seems rather long, but by experience it is rather common when different drying ovens have been observed. Meanwhile EEC (3) and DA/ISO (2) prescribe that the temperature (130°C) should be reached within 45 minutes after incertion of the samples.

In the present experiment the samples were partly placed on the upper shelf and partly on the lower shelf. Three samples in each position were controlled. The rest of samples which an oven could hold in order to obtain the projected loading were incerted to fill up. The samples were dried for 2 hours after the temperature was recaptured.

In relation to an analysis of variance where ovens were considered as random and placement as fixed effect factor significant differences among the ovens were demonstrated according to the 99 per cent probability level. Samples dried in oven No. 1 differed from those dried in No. 2 and No. 3 by 0.09 per cent of moisture. The difference between upper and lower placement was 0.04 per cent of moisture. The over all standard deviation was 0.03 per cent moisture.

Oven No. 1 and No. 2 were compared under the same circumstances as mentioned for the three ovens. The ovens and placement as well were considered as fixed effect factors. The analysis of variance revealed that the difference between the two ovens was significant according to the 99 per cent level, and the difference between placements was significant according to the 95 per cent level of probability. For oven No. 2 the percentage of moisture was found to be 0.09 per cent higher than that found for oven No. 1 which is in agreement with the previous experiment. From the upper placement the percentage of moisture was found to be 0.06 per cent higher than those from the lower placement, but this test comprised 12 figures only. According to results from different experiments, 52 individuals, including placement showed that no significant difference between placement up or down could be proved according to the 95 per cent level of probability. The over all standard deviation was 0.03 per cent of moisture. When the ovens No. 1 and No. 2 were used in the same experiment, the results from oven No. 1 have been corrected in the following by adding 0.09 per cent of moisture.

Loading of the ovens

In the work with moisture determination various loadings of the oven will often occur. Three experiments were carried out with 5 g samples and the following loadings: - 110, 220, and 460 cm^3 oven volume per 1 g of wheat meal with 15 per cent of moisture. The 18 samples were placed partly on the upper shelf and partly on the lower shelf. The analyses of variance proved that there were no significant differences in moisture for loading or for placement according to the 95 per cent level of probability. When no effect was found for loading, the reason probably was, that after incertion of the samples it took 95 minutes to recapture the 130°C when the loading was 110 cm^3 oven volume per 1 g of meal, whereas with the loading of 460 cm^3 per 1 g of meal it took 40 minutes only. The over all standard deviation was 0.04 per cent of moisture.

Handling of the laboratory sample

When a sample reach the laboratory it has to be mixed and reduced. The conical divider or the multiple slot divider is often used for dividing. The multiple slot divider was used in the present experiment. Equal amounts of barley with 19.6 per cent and with 14.1 per cent of moisture were mixed by hand very shortly and simply in a pail. In this way an extremely heterogenous lot of barley kernels was performed. After that 10 samples of about 500 g each were taken. Each of these samples was divided in 2 of c. 250 g each by the multiple slot divider. From each of these, 2 samples of 50 g each were taken and 25 g hereof taken for grinding and 5 g of meal for drying. An analysis of variance revealed that there was no significant difference neither between the 500 g samples nor between the 250 g samples. A standard deviation of 0.14 per cent of moisture was calculated. This is surprisingly high compared to that found in other experiments, but it must be taken into consideration, that it includes the error by sampling from the 250 g samples, which were not mixed individually before the 50 g samples were taken out. Perhaps it may be concluded, that the multiple slot divider delivers two almost equal samples, but not necessarily two homogenous samples. The results emphasize the importance of mixing heterogenous samples received at the laboratory.

In order to obtain information about change in moisture content by dividing samples, 5 kg of barley with 14.1 per cent of moisture was spread on a disk and supplied with 380 g of distilled water by an atomizer. The kernels were then mixed for 1½ hour in the mixing box and kept at 5°C for two days and at 20°C for one day, finally they were mixed in the mixing box for 30 minutes. After that 4 samples of about 1 kg each were taken out. Sample A was left undivided and the samples B, C, and D were divided one, two or four times respectively by the multiple slot divider. After the first dividing of C and after the first, second and third dividing of D, the two halves were accumulated and divided again. After the final dividing four times 50 g were taken from A, and from the 500 g subsamples from B, C, and D, two 50 g samples were taken from each. The 50 g samples were predried at 65°C overnight. 25 g of each were ground for 60 seconds and 5 g were taken for the final drying. For samples undivided, divided once, divided two or four times the percentages of moisture were found to be 20.57, 20.59, 20.62, and 20.65, respectively. The over all standard deviation was $s = 0.03$. According to analysis of variance the difference in moisture content was significant between undivided and divided four times only, according to the 95 per cent level.

Predrying and equilibration

When kernels are wet they are inconvenient for grinding. An ordinary mill cannot function, evaporation may be incontrollable, and such material has to be predried before grinding.

From samples received in a laboratory duplicate samples must be taken for predrying. In the present investigations samples of 50 g kernel have been predried on a 10 times 16 cm metal dish, which on an average corresponds to c. 300 mg kernels per 1 cm^2 on the container.

Results from the following experiment illustrates the importance of even distribution on the dish. Samples of different weight of intact kernels were taken into drying glasses of equal size and form. Thereby various heights of the layer of kernels were obtained. The samples were dried at 130°C in different times, and the results from 2 hours drying are set out in Fig. 2.

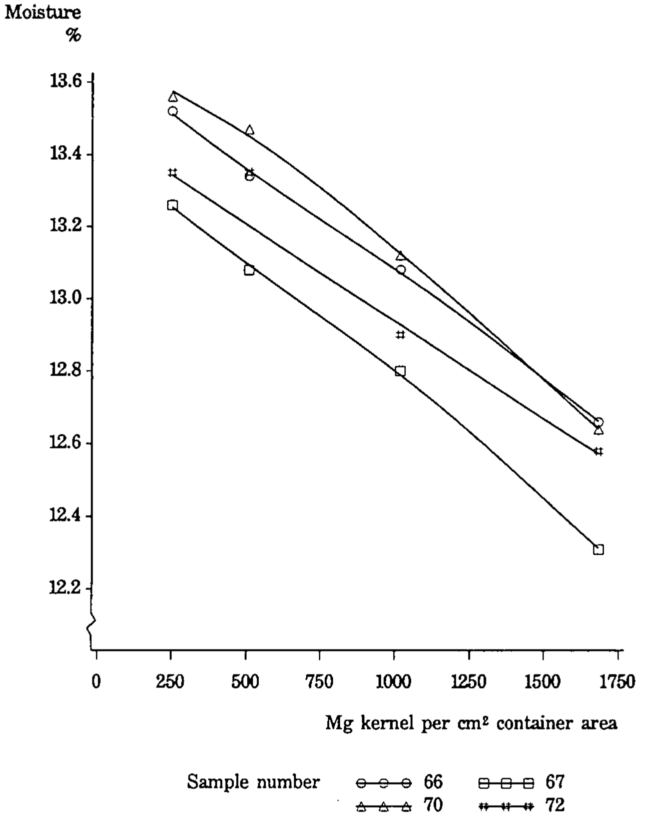

Fig. 2. Percentage of moisture at increasing height of kernels in the drying containers. Drying at 130°C for 2 hours. For comparison the results for No. 66 barley, No. 70 rye, and No. 72 wheat have been deducted 2 per cent, 1 per cent, and 0.5 per cent of moisture respectively, sample No. 67 is barley.

Ten replicates were performed and the standard deviations for the different sizes of sample varied from 0.05 to 0.10 per cent of moisture. From these results it may be concluded that uneven distribution of the kernels on the drying dish may result in a heterogenous sample. The difference in drying effect as a result of height of layer of kernels ceaces when time of drying increases. After drying of wheat kernels for 140 hours, there was no difference.

It will be of interest to know the effect of different temperatures for predrying. For this purpose two well mixed lots of barley with 23 and 16 per cent of moisture, and two lots of oat with 19 and 15 per cent of moisture were chosen. From each lot 3 samples of 50 g for each of 5 different temperatures were taken. At an oven volume of 130 cm^3 per 1 g kernel they were predried as reported in Table 1 and equilibrated for 2 hours. From each predried sample two samples of 25 g were ground for sixty seconds and emptied in a cone from which two samples of 5 g meal were taken for the final drying at 130°C for two hours. 120 samples in all.

From the results concerning predrying set out in Table 1 it can be seen, that the amount of moisture removed decreases considerably with decreasing temperature.

According to analysis of variance the effect of variety and of temperature was highly significant, and the interaction temperature x variety was significant according to the 99 per cent level.

Table 1. Predrying of various varieties at different temperatures, percentage of moisture at predrying.

Duration of drying		7 min.	16 hours			
Drying temperature, °C		130	65	55	45	35
Barley	45	18.03	17.42	16.10	14.39	12.03
-	34	10.92	9.84	8.61	6.89	5.21
Oat	51	15.71	14.43	13.31	11.54	9.51
-	43	12.06	10.86	9.69	8.04	6.28

This interaction effect is caused by a slightly higher temperature effect on samples with the largest moisture content. The standard deviation was s = 0.13. The results of the final drying showed an s = 0.066. The results for total moisture content with a standard deviation of s =

0.058 are presented in Table 2. From these results it can be seen that the decrease in moisture content in relation to decrease in the predrying temperature is moderate for the samples with the lowest moisture content (No. 34 and No. 43). The average of moisture for each temperature have an LSD value of 0.09 according to the 95 per cent level, which means that the differences among the temperatures 130, 65, and 55°C are not significant. The difference in moisture between 55 and 45°C is just 0.09 per cent and the difference from 45 to 35°C is 0.06 and highly different from the three first mentioned temperatures.

Table 2. Percentage of total moisture content in various varieties at different temperatures for predrying.

Duration of predrying		7 min.	16 hours			
Drying temperature, °C		130	65	55	45	35
Barley	45	22.77	22.85	22.66	22.60	22.49
-	34	15.98	15.95	15.94	15.84	15.85
Oat	51	18.87	18.88	18.91	18.72	18.75
-	43	15.45	15.50	15.44	15.44	15.27

When the respective figures for predrying are subtracted from those for total moisture content the moisture left in the kernels after predrying calculated on the original sample - residual moisture - is found. From these figures it could be seen that for oat No. 51 and 130°C the residual moisture content was 3.16 per cent, but the corresponding figure for barley No. 45 and 35°C was 10.46. The high temperature for predrying leaves much less residual moisture than the low temperature, which is caused partly by the temperature and partly by the longer time taken to reach the higher temperature in the oven.

The lower standard deviation for total moisture than for predrying and for final drying as found in this experiment is in agreement with the results in Table 5 and the related comments.

After predrying kernel samples were left open on the desk in order to equilibrate with the laboratory air to some extent before weighing and sampling for grinding. Predried samples of barley, wheat, and oat with a residual moisture content of from 3.3 per cent to 9.7 per cent showed an increase in weight of about 0.2 per cent per hour for the first two hours of equilibration, and an increase of about 0.15 per cent per hour for the third and fourth hour calculated on the weight of the original undried sample. It means an increase of about 0.003 per cent of moisture per minute. According to these results, the samples equilibrated for a couple of hours seem to be sufficient moisture stable for further handling, but to some extent the moisture stability may depend on the laboratory climate.

Dry and moist cereals

When moist and more dry samples are dried simultaneously perhaps some irregularity in moisture percentage might occur. In order to obtain information hereof the following experiments were carried out. Barley with about 15 per cent of moisture was used. Some of the samples were ground direct without predrying before incertion in the oven and for other samples the kernels were predried over night at 65°C loosing about 9 per cent of moisture before grinding for 60 seconds. In the drying process for 2 hours at 130°C some wet samples were dried together with a few predried and vice versa. 220 cm^3 oven volume per 1 g of meal. The results are set out in Table 3. When the percentage of moisture at predrying is designated m_1 and the percentage of moisture in the meal at the final drying is designated m_2 the total moisture content is calculated as

$$m_1 + m_2 - \frac{m_1 \cdot m_2}{100} = \text{per cent moisture}$$

Table 3. Percentage of moisture in barley when many wet samples are dried together with few predried and vice versa. The samples placed in the upper respectively lower part of the oven.

	Expt. I		Expt. II		Averages	
	Drying performance					
	direct	predried	direct	predried	direct	predried
	Number of samples:					
upper	12	3	3	12		
lower	8	2	2	8		
	% moisture:					
upper	14.95	15.11	14.90	15.13	14.94	15.12
lower	14.88	15.08	14.96	15.14	14.90	15.13
averages	14.93	15.09	14.93	15.13		

According to the results in Table 3, the simultaneous drying of more and less dry samples has not influenced on the percentage of moisture acertained in these experiments.

Cleaning of kernel samples

When kernel samples are impure it may be necessary to clean these by a winnowing machine. For kernels with a relatively high moisture content, moisture may be lost in this procedure. The following experiment was carried out in order to investigate this, and the samples employed were almost clean.

It was necessary to wet samples in order to obtain some variation in the moisture content. The wetting procedure has been carried out in different ways. Oxley et al. (9) wetted various types of cereals by adding the calculated amount of distilled water in about 1 per cent excess. Then the samples were kept in airtight containers at room temperature for 24 hours and thereafter dried back to the desired moisture content. Among other reasons for using this procedure they mention the evidence that recent wetted kernels have an excess of unbound water in the outer layers, and they expect by the drying back to remove this water and to bring the kernels to nearly normal conditions. Brekke (5) sprayed water on corn up to 15-16 per cent moisture. After a resting period of 9-43 hours water was added up to 21 per cent of moisture and after resting for 2½ hour furthermore 3 per cent of water was added. Williams and Sigurdson (13) wetted 250 g samples of wheat and barley by demineralized water and stored the samples in sealed cans at about 22°C for seven days and then at 5°C for two weeks. A pilot experiment had shown that the kernels attained a stabilized moisture level after 48 hours. Olesen (12) refer to moistening with about 2 per cent of moisture at a time and a rest period of one day allowing the moisture to penetrate.

The following experiment was carried out. From two well mixed quantities of each of barley, wheat, and oat samples of 1 kg were filled into plastic bags at 15°C, supplied with demineralized water so the kernels were covered. After that the samples were kept for 0, 1, 7, 20, 120 or 360 minutes at 5°C. The 0-samples were not wetted. After the moistening time the surplus of water was discarded and the individual sample was emptied on a tray (38 times 39 cm) supplied with some layers of blotting paper. In order to remove most of the adherent water, the samples were massaged intensively at 20°C for two minutes by hand with a water abosrbing paper folded several times which was changed often. Thereafter the kernels were transferred to a plastic pail (h. 24 cm, d. 26 cm) supplied by a dry drying up cloth and the pail closed, the pail was turned (rolled) for 1.5 minutes. Then the kernels were transferred to platic bags, which were tightly closed and placed at 5°C for 48 hours. During this time the bags were massaged and turned several times.

Thereafter the 1 kg samples, apparently dry on the surface were transferred to 15°C. By the procedure described samples with moisture content varying from about 14 to 36 per cent were prepared. Each 1 kg sample was divided by a conical divider in two, each transferred to a platic bag, tightly closed, and placed at 15°C. The one of the 500 g samples remained in the room and the other one was cleaned by the winnowing machine described under materials. The time from opening the sample bag and emptying the sample on the winnowing machine until the kernels were transferred to a plastic bag again and tightly closed amounted to 2.5 minutes on average, s = 0.7 minutes.

From each of the 500 g samples of kernels two samples of 50 g each were taken out and predried overnight at 45°C. After predrying the samples were airconditioned for 2 hours. Of the not moistened samples only oat was predried. Each of the 144 fifty g samples was ground with the Janke and Kunkel mill for 60 seconds and 5 g of each sample was taken for the final drying for 2 hours at 130°C.

Table 4. Percentage of moisture in barley, wheat, and oat at moistening and cleaning.

Moiste-	Barley				Wheat				Oat			
ning	No. 33		No. 49		No. 32		No. 47		No. 51		No. 54	
min.	nc[1)]	c	nc	c	nc	c	nc	c	nc	c	nc	c
0	15.07	15.03	15.20	15.17	15.50	15.51	14.18	14.18	18.96	18.65	17.49	17.35
1	20.29	20.12	22.34	22.19	20.97	20.78	20.19	20.12	26.59	26.16	24.35	24.18
7	22.09	21.88	23.40	23.29	21.11	20.90	21.37	21.19	26.98	26.62	27.39	26.98
20	23.11	22.98	25.42	25.24	22.38	22.15	22.34	22.33	29.81	29.51	30.13	29.89
120	26.82	26.75	29.09	29.10	25.90	25.78	26.29	26.12	32.37	32.27	33.77	33.56
360	30.31	30.15	31.78	31.50	29.80	29.61	30.19	29.86	35.12	34.63	36.40	35.90

[1)]nc = not cleaned, c = cleaned

From the results in Table 4 it can be seen that both moistening and cleaning influence the percentage of moisture. For the moistened samples an analysis of variance showed the effect of all the factors: - time of moistening, variety, and cleaning to be significant at the 99 per cent level, and so were the 2-way interaction effects. The LSD values for cleaning, time of moistening, and variety were 0.11, 0.84, and 0.92 respectively. The standard deviation was calculated to be s = 0.07. For the samples with less than 17 percentage of moisture the cleaning procedure did not influence on the moisture content.

Concerning the results for total moisture an analysis of variance was carried out with the numeric differences between duplicates from cleaned and from uncleaned samples as fixed factor and the varieties and time of moistening as random factors. According to this analysis and the 95 per cent level there was no significant effect of cleaning or time of moistening on difference between duplicates. Only for the varieties the variance was significant, and the LSD value calculated to 0.063 pointed oat No. 54 out to differ from wheat and barley. This means that the analytical error especially for oat No. 54 is a little larger than it is for wheat and barley. This might be related to a relatively higher degree of heterogenity of the ground material from oat with the higher shell content. On average the numeric differences between duplicates for total moisture content for wheat, barley, and oat were 0.039, 0.065, and 0.117, respectively.

The standard deviations for moistened kernels from the different varieties are set out in Table 5. From these results it can be seen that standard deviations for total moisture have been higher for oat than those for barley and wheat.

Table 5. Standard deviations for moisture content in moistened kernels (uncleaned and cleaned) at predrying, final drying and for total moisture content.

Variety		Uncleaned			Cleaned		
		pre-drying	final drying	total moisture	pre-drying	final drying	total moisture
Barley	33	0.20	0.25	0.06	0.10	0.10	0.06
-	49	0.11	0.09	0.06	0.19	0.16	0.05
Wheat	32	0.07	0.06	0.02	0.10	0.11	0.03
-	47	0.10	0.10	0.03	0.11	0.10	0.05
Oat	51	0.20	0.15	0.08	0.06	0.11	0.09
-	54	0.23	0.13	0.11	0.12	0.19	0.12

Furthermore from the results in Table 5 it can be seen that the standard deviations for predrying and for final drying are higher than those for total moisture. The reason for this could be that, if the one of the duplicate samples showed a relatively high moisture percentage at predrying it would show a relatively low percentage of moisture at the final drying and vice versa.

A regression analysis was carried out, where the results for percentage of moisture for the duplicates a and b at predrying are called ap and bp, and the results for the same duplicates at the final drying are called af and bf. When ap minus bp is named x, and af minus bf is named y, the following equation arrives:

$$y = -0.818x + 0.0102$$

With $s_{y/x} = 0.11$ per cent of moisture, $s_b = 0.07$, and $s_a = 0.01$ per cent of moisture. The correlation coefficient was calculated, $r = -0.83$, and was significant according to the 99 per cent level, thus the coefficient of determination was, $r^2 = 0.69$. When the coefficient of determination is not higher, the reason among others possibly is uneven distribution on the sheeds at predrying and that moisture preferably may be removed from the kernels at the surface of the sample.

From the figures in Table 4 it can be seen that by moistening it has been possible to perform kernels with a great variation in moisture content. For the samples with more than 17 per cent of moisture the loss in weight at predrying ranged from 10.7 per cent up to 30.1 per cent increasing with time of moistening. At the final drying the loss in weight ranged from 7.6 per cent up to 11.0 per cent only. From these figures it is seen that from wett kernels most of the moisture is lost at predrying at 45°C overnight forming a linear proportion of the total moisture. Regression lines for the area in question for percentage of moisture at predrying, y, on percentage of total moisture, x, were: -

barley, $y = 1.084x - 10.74$, $r^2 = 0.99$

wheat, $y = 1.126x - 11.44$, $r^2 = 0.99$

oat, $y = 1.165x - 12.27$, $r^2 = 0.99$

For all the varieties the r was significant according to the 99.9 per cent level. If more time had elapsed from moistening until analysis, perhaps the moisture of the kernels would have been nearer in equilibrium and a diminutive of the added water would have been bound. On the other hand the added water may preferably be present as free water which is easily removed.

Results for loss of moisture in process of cleaning can be seen from the figures in Table 6. These results do not include the samples with less than 17 per cent of moisture, because practically no moisture was lost from these according to the figures in Table 4.

Table 6. Decrease in moisture content during cleaning of barley, wheat, and oat with more than 17 per cent of moisture.

Variety		decrease % moisture	standard deviation	probability level
Barley	33	0.15	0.024	99
-	49	0.14	0.047	95
Wheat	32	0.19	0.016	99
-	47	0.15	0.055	95
Oat	51	0.33	0.056	99
-	54	0.27	0.051	99

A calculation was carried out for correlation of loss of moisture in process of cleaning on total moisture percentage.

This showed a correlation coefficient, r = 0.46 which was significant according to the 99 per cent level. The regression coefficient was 0.010. Here it should be borne in mind that the figures for decrease in moisture in process of cleaning include the analytical error from cleaned and uncleaned. As a result thereof the standard deviation on decrease in moisture during cleaning will be relatively large, for barley No. 40 the coefficient of variation amounts to 34 per cent.

An analysis of variance with time of moistening as random and cleaning as fixed factor was carried out for each variety. The effects of both factors were significant, as expected. Significant interaction effects were found for wheat No. 47 and for barley No. 49 only, according to the 99 and 95 percent level respectively.

According to the results in Table 6, the losses of moisture from kernels during the cleaning process have been about 0.15 per cent by weight for barley and wheat, and about 0.3 per cent for oat from kernels with more than 17 per cent of moisture. The higher loss from oat is probably because oat has a relatively high shell percentage and a much lower weight per liter than barley and wheat. As a result of that the oat kenels may be held in the air stream in the winnowing machine for a longer time than wheat and barley and thus dries more during this step.

Even if the moisture should not have been in exact equilibrium throughout the single seed or the duration of cleaning has not been reasonably precise the results will give a reasonable idea about loss in weight during cleaning of relatively wet kernels.

Discussion and conclusion

When results from different ovens of the same type differed by about 0.1 per cent of moisture it may point to the importance of testing the ovens. A change of loading of the oven from 460 to 110 cm^3 per 1 g of meal resulted in equal results for percentage of moisture, though the time for the oven to reach 130°C increased from 40 to 95 minutes after incertion of the samples. The extra time for reaching the planned temperature may possibly be related to the longer time it takes to remove the greater amount of free water. When this process ceases the temperature increases to the planned level. The loss of moisture by mixing and by reduction of the laboratory sample was up to 0.1 per cent for samples with 20 per cent moisture. The relatively small loss may be a result of the protection which the shell of the intact seed gives. Dividing by the multiple slot divider resulted in two equal samples, but they may be heterogenous. By placing a heterogenous sample on the divider, it may happen that the moisture content in the kernels at one end of the divider is higher than at the other. The importance of a thin layer and even distribution of the sample for predrying was clearly demonstrated though the time for predrying was two hours.

Results were independent of drying of a few samples with high moisture content together with more samples with a low moisture content or vice versa. The reason for that may possibly be that the drying intensity of the most dry samples is small until the difference in moisture content to a certain extent has been reduced.

The predrying for different time and temperature resulted in lower results for moisture content at predrying and for total moisture content for the lower temperatures. Among other facts the samples with the large amount of moisture removed at predrying may have gained a little moisture at grinding and the samples with the small amount of moisture removed at predrying may have lost moisture in process of grinding. This would be in agreement with other results in these investigations (11). With predrying at 35°C perhaps some metabolism may have been going on (Allerup, 1). Also the varieties behave differently and the nutritional history of these may have influenced. A duration of two hours for equilibration of the predried sample is acceptable, when work is going on reasonably fast. However if half an hour passes from weighing of the predried sample until grinding, the total moisture percentage may differ with up to 0.1 per cent.

There only was a tendency that the moisture content for samples on the upper shelves in the oven was found to be higher than for those on the lower shelf. It must be good laboratory technique to place replicates on different shelves.

From samples of barley and wheat with 14-15 per cent of moisture there was no loss of moisture in process of cleaning. From wetted kernels of barley, wheat, and oat with a moisture content ranging from 17 to 36 per cent the loss in process of cleaning was 0.15 per cent for barley and wheat and 0.30 per cent of moisture for oats. These losses of moisture were significantly dependent on the extent to which the kernels were moistened, but with an r value of only 0.46.

The reason for that may be variation in time of cleaning for the particular sample and that variation in moisture on the kernel surface is relatively small, also when the moisture content is higher than 17 per cent. The higher loss from oat may be a result of the lower weight per liter of this variety compared to the others. Though the shell content is higher for oat it remains in the wind stream on the winnowing machine for a longer time. Standard deviations for predrying and for the final drying as well are higher than for total moisture content. Some of the explanation was that for duplicate samples the one with the lowest moisture percentage at predrying had the highest moisture percentage at the final drying. The r was - 0.83. When the standard deviation for determination of moisture in oat was higher than those for barley and wheat it mainly may be a result of the high shell content for oat which results in a higher content of flakes at grinding and thereby a heterogenous meal for sampling. The standard deviation on moisture determination was independent of wetting and cleaning. As a whole for the experiments the standard deviation was about 0.06 per cent moisture.

Acknowledgement

Thanks are due to Danish State Seed Testing Station for the loan of the winnowing machine and to DLG, Danish Co-operative Farm Supply for various types of kernel samples.

References

1. *Allerup, S.* 1958. Effect of temperature on uptake of water in seeds. Physiologia Plantarum 11, 99-105.
2. *Anonymous.* 1980. Dansk Standard DS/ISO, 712. Korn og kornprodukter. Bestemmelse af vandindhold.
3. *Anonymous.* 1971. De Europæiske Fællesskabers Tidende 20.12.71. No. 1, 279/7. Kommissionens andet direktiv af 18. november 1971. Bestemmelse af vandindhold, 859-61.
4. *Anderson, J. E.* 1936. Some facts concerning vacuum - oven moisture determination. Cereal Chem. 13, 437-52.
5. *Brekke, O. L.* 1966. Corn dry-milling: A comparison of several procedures for tempering low-moisture corn. Cereal Chem. 43, 303-12.
6. *Hummel-Gumælius, T.* 1980. Olika provtagningsmetoder vid spannmålsleverans. Nordisk Jordbrugsforskning 62(2), 270.
7. *Karmas, E.* 1980. Techniques for measurement of moisture content of foods. Food Technol. 34(4), 52-59.
8. *Nelson, O. A. & Hulett, G. A.* 1920. The moisture content of cereals. J. Ind. Eng. Chem. 12, 40-45.
9. *Oxley, T. A.; Pixton, S.W. & Howe, R. W.* 1960. Determination of moisture content in cereals. J. Sci. Food Agric. 11, 18-25.
10. *Swanson, C. O. & Anderson, J. E.* 1935. Atmospheric factors in milling. The Northwestern Miller 182(2), 198.
11. *Sørensen, C.* 1992. Moisture determination in cereals, analytical details in oven drying. II. Disintegration, loss of moisture, sampling of meal, disharmony near predrying.
12. *Olesen, T.* 1979. Udvikling af analysemetoder og processer til fremstilling af cerealprodukter med hovedvægt på anvendelse i bryggerierne II, bilag 5. Erhvervsforskerprojekt. Bioteknologisk afdeling, Carlsberg Forskningscenter. Afdelingen for teknisk biokemi, Danmarks Tekniske Højskole.
13. *Williams, P. C. & Sigurdson, J. T.* 1978. Implications of moisture loss in grains incurred during sample preparation. Cereal Chem. 55(2), 214-29.
14. *Wilson, M. A.* 1986. Comparison of alternate approaches to measurement of moisture in grains and oilseeds. Cereal Foods World 31(6), 406-08.

Contents Part II

Moisture determination in cereals, analytical details in oven drying. II. Disintegration, loss of moisture, sampling of meal, disharmony near predrying

Vandbestemmelse i korn, analytiske detaljer vedrørende ovntørring. II. Formaling, vandtab, udtagning af melprøver, disharmoni i omegnen af fortørring

Chresten Sørensen

Summary

According to the investigations carried out it was found that cooling of the mill and reduction of air stream through it corresponded to reduction of loss of about 0.5 and 0.2 per cent of moisture, respectively. Different time of grinding resulted in a variation from 30 to 80 per cent of particles smaller than 0.5 mm, a difference of 1 per cent of moisture, and an increase in moisture lost in process of grinding on 0.15 per cent. Only at sufficient grinding, the post drying had the expected value. Gentle mixing of meal or forced mixing involved a loss of moisture of 0.02 and about 1 per cent, respectively. Sampling of meal direct from the mill or from emptied meal gave equal results for percentage of moisture and of standard deviation. Two samples for predrying and one sample of each for final drying involved a lower standard deviation than one sample for predrying and two from this for the final drying. Disharmony at the border between no predrying and when predrying in the analytical procedure is included could be overcome by including predrying as routine. Correction for loss at grinding may be appropriate. The loss of moisture at grinding without predrying of a sample with 17 per cent of moisture amounted to shortly 0.3 per cent of moisture.

Key words: Grinding, loss of moisture, sampling of meal, disharmony in the area of predrying.

Resumé

I henhold til resultaterne fra de gennemførte undersøgelser bevirkede køling af mølle og reduktion af luftstrøm gennem denne en reduktion i tab af vand på henholdsvis 0.5 og 0.2 procent. Forskellig formalingstid resulterede i en variation i partikelstørrelse fra 30-80 procent mindre end 0.5 mm, en forskel på 1 procent vand og en stigning i vandtab ved formalingsprocessen på 0.15 procent. Kun hvis formalingen var tilstrækkelig havde kontroltørring den forventede værdi. Svag og voldsom blanding af mel medførte et vandtab på henholdsvis 0.02 og ca. 1 procent. Udtagning af melprøve til tørring direkte fra møllen eller fra udtømt mel gav ens resultater for vandprocent og for standardafvigelse. Udtagning af to prøver til fortørring og een fra hver af disse til formaling og sluttørring medførte en lavere standardafvigelse, end når der anvendtes een prøve til fortørring, og der udtages to af denne til sluttørring. Disharmoni i omegnen af ingen fortørring og når fortørring gennemførtes i analyseproceduren kunne afhjælpes ved at gennemføre fortørring for alle prøver, uafhængigt af vandindholdet. Korrektion for formalingstab kan være hensigtsmæssig. Vandtabet under formaling af en prøve med 17 procent vand beløb sig til knap 0.3 procent.

Nøgleord: Formaling, tab af vand, prøveudtagning af mel, disharmoni i omegnen af fortørring.

Introduction

The grinding of kernels for moisture determination have called for attention, because change in moisture may occur. Various mills work in different ways, thereby that both the disintegration as well as the easiness of cleaning and the exposure of the samples to the surrounding air may vary. Wilson (11) discusses different methods for moisture determination and put attention to loss in process of grinding. Williams and Sigurdson (10) found great losses varying with different mills and increasing with moisture percentage. When wheat with 13.5-18.0 per cent moisture was ground, Anderson (1) found on average 0.11 per cent loss in one series and 0.19 per cent loss in another, ranging from 0 to 0.38 per cent of moisture. A Burr mill adjusted to grind as fine as possible was used, but no determination of particle size was made. Oxley et al. (8) observed that results obtained by various oven methods were influenced to a great extent by the type of grain used. When two samples gave equal results after drying by one oven technique, the results differed when another oven technique was used. Maybe that some of the discrepancies was related to the size of the particles, which was so that after grinding of wheat with 12 per cent moisture only 14 per cent of the particles, by weight, was smaller than 0.6 mm.

Karmas (5) discusses various techniques for moisture determination and underline the value of the direct methods for calibration of the indirect methods. The recommended drying temperature differ from one method to another. Nelson and Hulett (7) studied the formation of gases formed by decomposition of organic materials when using high vacuum technique and found only moderate decomposition of cereal products until 180°C for four hours. At higher temperatures the gas evolution accelerated.

Materials and methods

The handling of kernel samples and the oven technique have been investigated and results about that have been published in part I (9) where materials and methods have been described. These have also been used in the present investigations.

Results

The grinding procedure

The Janke and Kunkel mill was chosen because of the relatively tight chamber for the process of grinding, water cooling, simplicity and easy cleaning, and it is easy to exchange the knives when these become dull. In order to investigate its applicability some experiments were carried out. The grinding procedure is mentioned under methods. 25 g were ground each time. When kernels have been ground, the grinding chamber was rinsed by a brush, and special attention was paid to free the rim for dust in order to keep the grinding chamber tight. Meal for drying was sampled immediately after grinding. The time of grinding may influence on the size of particles and thereby on the distance for diffusion of the moisture inside the particles.

Mills

From Williams and Sigurdsons (10) results it could be seen that different mills may cause different loss of moisture in process of grinding. In the present investigations only few experiments were carried out concerning this topic. The Janke and Kunkel and the Christ mill were involved. From a lot of barley 30 samples of 50 g each were predried at 45°C overnight and equilibrated for 2 hours reaching 8 per cent of moisture. From each sample 25 g were taken for grinding (20 samples on the Janke and Kunkel mill and 10 on the Christ mill). Of the not predried material, samples of 25 g were taken direct for grinding. The Janke and Kunkel mill was tap water cooled as usual, but the Christ mill was not. Results are set out in Table 1.

Table 1. Percentage of moisture in barley caused by use of different mills for grinding.

	Janke and Kunkel mill cooled		Christ mill not cooled	
	not predried	predried	not predried	predried
n	20	20	10	10
$\bar{x}$	15.63	15.75	14.86	15.91
s	0.06	0.05	0.08	0.07

For the Christ mill the percentage of moisture in the non predried material is about 0.8 per cent moisture lower than the corresponding figure for the Janke and Kunkel mill. This is probably partly a result of transport of a lot of air through the Christ mill in process of grinding, and thereby moisture has been removed from the not predried samples and partly as a result of missing cooling of the Christ mill. For the predried material a relatively high percentage of moisture is found for the Christ mill, which probably to some extent is a result of absorbtion of moisture from the passing air during grinding.

The differences in moisture percentages between results for the Janke and Kunkel and the Christ mill, and between the results for predried and not predried were significant according to the 99 per cent level. Another similar experiment showed results agreeable to those in Table 1. The Christ mill did not become extreamly warm at this experiment because cleaning of the mill and preparation of the ground sample for drying here took some time after each grinding.

Effect of cooling the mill

Cooling of the mill may influence on temperature of the meal and therewith on the evaporation and moisture percentage measured. Some samples of barley were ground consecutive and as soon as the mill was stopped a thermometer was incerted in the meal which was pressed against it and the temperature read. From the results set out in Table 2 it can be seen, that when the mill is cooled, the temperature of the meal is almost constant about 30°C. Without cooling the temperature after grinding of eight samples has increased to almost 50°C. Room temperature was about 26°C.

Table 2. Meal temperature, °C, at consecutive grinding of samples on the Janke and Kunkel mill. With or without cooling. Different moisture content.

Grinding	20% moisture		15.5% moisture	
No.	cooling	no cooling	cooling	no cooling
1	32.6	37.3	31.7	39.2
2	31.6	42.2	32.3	41.7
3	32.2	43.3	32.1	43.7
4	30.7	44.6	31.5	45.8
5	31.3	45.5	31.2	47.1
6		46.2		47.5
7		48.1		49.9
8		48.9		49.8

At this high temperature reached in process of grinding it may be expected that moisture will evaporate from the meal during grinding and when the mill is opened. This is confirmed by the results from another series of consecutive grindings of barley.

Two different Janke and Kunkel mills were involved in the experiment. From the results set out in Table 3 it can be seen, that results from the two mills are identical and constant to grinding number, when the mills are cooled. Without cooling the percentages of moisture are lower and decrease with grinding number. These results are in accordance with those in Table 1 and Table 2, and are in agreement with Flohils (4) results.

Table 3. Influence of using different Janke and Kunkel mills and of cooling on the percentage of moisture measured in barley.

Grinding	Mill		
No.	I	II	
	cooling	cooling	no cooling
1	16.40	16.38	16.02
2	16.44	16.34	16.03
3	16.38	16.40	16.16
4	16.26	16.35	15.95
5	16.37	16.34	15.92
6	16.33	16.38	15.84
7	16.32	16.37	15.68
8	16.41	16.45	15.80
9	16.41	16.34	-
10	16.38	16.40	-

Time of grinding

Investigations of the influence of time of grinding on moisture content measured were carried out in the following way.

From each of A and B, barley, and C, wheat, seven samples of 50 g each were performed. The B, and C samples were predried at 45°C overnight. From each of the 50 g samples 2 (No. I and No. II) times 25 g were taken for grinding and from each of these 2 times 5 g were taken for drying. The samples incerted in the oven at 130°C and dried for 2 hours after the oven temperature again had reached 130°C. The 25 g samples No. I and No. II were observed in order to obtain information about whatever a separation of the kernels occurred when the first sample of 25 g of kernel was taken from the 50 g sample. Grinding was carried out from zero upto 120 seconds. Results are set out in Table 4.

When the 5 g samples for drying had been taken from the 25 g meal sample, the residue was shifted on a 1 mm and a 0.5 mm sieve, the different groups of particles were weighed, and the particle distribution calculated. In Table 4 only the figures for percentage of particles smaller than 0.5 mm or larger than 1.0 mm are set out. From the figures concerning moisture it can be seen that the increase in moisture for A from whole kernels to 10 seconds of grinding is appreciable compared to B which was predried. For C the corresponding increase amounts to about 2 per cent of moisture, which may be explained thereby that the awnless seeds of wheat are easier to disintegrate than those of barley. These findings correspond well to the relatively high percentage of particles smaller than 0.5 mm for C at 10 seconds of grinding. For the grinding time of 40 seconds and onwards there is no significant increase in moisture content for any of the samples according to the 99 per cent level.

Table 4. Influence of time of grinding on moisture determined and on particle distribution.

Grinding	Percentage of moisture			Particle size, % by weight					
sec.	barley	barley	wheat	≤0.5 mm			>1.0 mm		
	A	B	C	A	B	C	A	B	C
0	12.38	17.33	11.92	-	-	-	100	100	100
10	14.12	18.96	14.05	11	19	30	78	61	46
20	14.61	19.39	14.21	22	34	52	57	41	24
40	14.93	19.71	14.46	41	61	75	29	16	7
60	14.99	19.66	14.45	55	77	87	15	6	2
80	14.99	19.77	14.45	65	86	93	9	2	2
120	15.06	19.87	14.37	83	96	97	2	2	2

For the predried samples the amount of particles smaller than 0.5 mm increases appreciably until about 40 seconds compared to the non predried barley A, which increases most until 120 seconds. It is obvious that wheat C has a relatively small content of particles larger than 1 mm from 10 seconds onwards.

According to an analysis of variance there were no significant difference in moisture content in samples No. I and No. II, so the 50 g kernel samples did not become separated by sampling of the 25 g for grinding.

After a week the disintegration experiment was repeated concerning barley B and wheat C, but included the grinding times 40, 60, 80, and 120 seconds only. It was clearly seen and an analysis of variance confirmed that the results of this experiment were all in close accordance with those in Table 4. The statistical treatment of the results further revealed that the standard deviation for duplicates of the drying samples decreased from 0.06 per cent of moisture for the non ground and the 10 seconds ground samples to 0.01 and 0.02 for the 40 and 60 seconds ground samples.

For some samples of barley ground for 60 seconds and repeated during six months, the percentage of particles smaller than 0.5 mm varied from 55 to 69 for barley with 15 per cent of moisture and from 76 to 86 for predried barley with 8.5 per cent of moisture. These variations will not practically influence on the moisture determination. The loss of material at the process of sifting, which routinely was 4 minutes was about 0.6 per cent with a standard deviation of 0.4 per cent by weight. This loss will not interfere with the moisture determination.

Loss at grinding

Important is the loss in process of grinding. Some samples of barley with different moisture content were ground for different times. For each time of grinding the samples of 25 g each were ground consecutively and the meal from each sample transferred to a weighing glass immediately after grinding and weighed. From the results set out in Table 5 it can be seen, that the loss in weight increases with time of grinding, it means with finess of the meal. The loss is smaller, when the kernels have been predried to a moisture content of 6.8 per cent.

Table 5. Loss in weight in process of grinding, various moisture content and different time of grinding, barley.

% moisture	15			6.8
Grinding, seconds	10	20	60	60
Number of samples	6	6	10	9
Loss, % by weight	0.16	0.27	0.32	0.05
s	0.07	0.06	0.15	0.04

Concerning the magnitude of losses, the results agree with Anderson's (1) results, but are far below the figures reported by Williams and Sigurdson (10) using different mills. The losses could be a combination of loss of material, evaporation, and absorption of moisture. If it was a question about loss of dust it would have been seen when the samples were ground and transferred cousecutively, but such was not observed. Therefore the results are not in disagreement with the supposition that the loss mainly is related to evaporation. The smaller loss from the 6.8 per cent moisture samples agree with that. For this the actual loss amounts to 12 mg per 25 g sample.

In order to obtain information on loss from the meal from the time at which it is emptied off the mill and until it has been transferred to the drying glass some experiments were carried out. The meal was emptied on an aluminium paper foil and immediately covered with an upside down porcelain bovel or with aluminium paper foil, or it was left open to the air for 0, 1, or 2 minutes. Analysis of variance confirmed that no significant amount of moisture was lost or gained in up to 2 minutes, when predried barley with 6.8 per cent of moisture or barley with 15.7 per cent moisture were used as experimental materials. However when barley meal with 15.7 per cent of moisture were left for 10 minutes about 0.2 per cent of moisture, by weight evaporated.

Mixing of meal

It is good laboratory praxis and necessary to mix disintegrated material before sampling for chemical analysis. When the purpose is determination of moisture, the material may loose moisture by evaporation or absorb moisture from the sourounding air. Three experiments, I-III, were carried out using different lots of barley with about 15 per cent of moisture. Samples of three times 50 g kernels were taken per treatment. For grinding were taken 2 times 25 g of which the second from the mill were emptied on top of the first to make a cone, which was mixed in different ways. A no mixing, B the meal was mixed by transferring from the cone to make another cone using a flat 6 cm wide shovel, three transferrings. C was treated like B but six transferrings, and D the meal was mixed criss-cross six times on a square. Two times 5 g meal from each cone were then taken and dried for two hours at 130°C.

Table 6. Percentage of moisture in barley at various intensity of mixing of the meal. Standard deviation, s, based on 15 replicates each.

	Mixing			
	none, A	3 times, B	6 times, C	criss-cross, D
Expt I	14.93	14.79	14.59	13.99
- II	15.60	15.61	15.55	14.60
- III	15.62	15.61	15.61	14.59
s	0.07	0.03	0.03	0.04

From the results set out in Table 6 it can be seen that the criss-cross mixing has caused a loss in moisture of about 1 per cent. This great loss may not be a result of the manner of mixing only, but also as a result of the longer time the criss-cross lasts. For experiment I there are measurable decreases in moisture for three and six times mixing, which is related to a more vigorous transferring for this experiment. The values for standard deviation clearly decrease when the samples are mixed, even when the mixing has been very gentle. Three months elapsed between the experiments I and II, and two months between II and III.

Sampling of meal

It is well known that the concentration of protein varies tremendously within the single seed, for barley Michael et al. (6) found 8-14 per cent protein in the endosperm but 30 per cent of protein in the aleuron layers. The variation in moisture content may be smaller and so the relative heterogenity. Some investigations were carried out to reveal the variation in moisture determined when the meal (5 g) for drying was taken directly from the mill without any extra mixing and thereby fasten the sampling in order to minimize change in moisture content. This procedure was compared to sampling of meal from the material emptied from the mill.

From about 1 kg of barley kernels 8 samples of 25 g each were taken and ground for 60 seconds. From each of the emptied meal samples 2 x 5 g were taken for drying. Another 8 samples were ground in the same way, but the 2 x 5 g meal for drying were taken direct from the mill with a teaspoon of a size and form so it could reach and almost fit the bottom of the grinding chamber.

Furthermore 8 samples of 50 g each were taken for predrying at 45°C overnight, after that it hold 9.5 per cent of moisture at the time of grinding. From each of these were taken 2 samples of 25 g for grinding. From the one of these, 2 samples of 5 g of emptied meal (one from each side of the cone) were taken for drying, and from the other one, 2 samples of 5 g meal were taken with the teaspoon direct from the mill (one from each side of the knives). Another identical experiment was carried out with oat. The whole investigation comprised 128 drying samples.

From the results set out in Table 7 it can be seen (as expected) that predried materials give the higher percentage of moisture. The percentages of moisture are almost equal whether the meal samples for drying are taken from the emptied meal or direct from the mill. The standard deviations are lowest for the predried material, which have a higher degree of disintegration. The standard deviations for oat tend to be a little higher than those for barley, which may be related to the more heterogenous oat meal. The standard deviations for the whole material were 0.04 for emptied meal and 0.05 for samples taken direct from the mill. These standard deviations were not significant different according to the 95 per cent level. According to these results it seems to be justifiable to take out the meal samples direct from the mill.

Table 7. Percentage of moisture when the 2 x 5 g meal for drying are taken from the emptied meal or direct from the mill.

	Barley				Oats			
	not predried		predried		not predried		predried	
	2 x 5 g meal for drying taken							
	from emptied meal	direct from mill	from emptied meal	direct from mill	from emptied meal	direct from mill	from emptied meal	direct from mill
$\bar{x}$	19.80	19.80	20.05	20.07	14.98	14.96	15.12	15.08
s	0.06	0.06	0.02	0.02	0.06	0.08	0.03	0.04
	2 x 5 g meal taken							
	from emptied meal		direct from mill		from emptied meal		direct from mill	
s	0.04		0.04		0.05		0.06	

Optimizing sampling

From statistical calculations concerning 60 results for predrying and 120 results for total moisture content an example of the effect of method of sampling on the standard error for the final result could be calculated. The results are set out in Table 8.

Table 8. Standard error on the final result influenced by the method of sampling, when predrying is performed.

Number of samples at predrying	Number of 5 g samples for final drying per each predried sample: 1	2	3
1	0.089	0.079	0.075
2	0.063	0.056	0.053
3	0.051	0.046	0.044

As an example it can be seen that when two samples are taken for predrying and one sample from each of these is taken for grinding, and one sample from each ground sample is taken for final drying a more precise final result is obtained (0.063) - than when one sample is taken for predrying and two samples hereof are taken for grinding and final drying (0.079). It also can be seen from the figures, that it gives more effect to increase the number of samples for predrying than increasing the number for the final drying.

Post drying and grinding intensity

When intact kernels are dried at 130°C, the loss in weight will go on for a long time. 40 samples of wheat were dried at 130°C for different periods up to 140 hours. In the period from 2 to 4 hours the loss in weight was 0.40 per cent per hour but for the period from 72 hours until 140 hours the loss in weight was on average 0.003 per cent per hour. From ground material the moisture evaporate faster. 25 predried samples and 25 not predried samples of barley, all with original about 15 per cent of moisture, were ground for 60 seconds, dried for 2 hours at 130°C and thereafter dried (post dried) for half an hour. No significant loss in weight could be acertained at the post drying. It should be borne in mind that the 30 minutes were counted from the time when the oven again had reached 130°C.

On the other hand, when post drying is carried out for a longer time a loss in weight may still occur. 24 samples of wheat meal were post dried for 2 hours and a significant loss in weight of 0.09 per cent was found. In Table 4 results showing influence of grinding intensity on particle distribution were reported. In Table 9 results from the following experiment concerning grinding intensity and post drying are set out.

Table 9. The influence of grinding intensity on moisture determined in predried and not predried barley, and on loss at postdrying for half an hour.

Grinding seconds	Percentage of moisture					
	before post drying		after post drying		lost at post drying	
	predried	not predried	predried	not predried	predried	not predried
0	4.87	12.03	5.22	12.51	0.35	0.48
10	6.27	14.44	6.41	14.56	0.14	0.12
20	6.54	14.74	6.64	14.79	0.10	0.05
40	6.70	14.93	6.78	14.92	0.08	-0.01
60	6.78	15.01	6.82	15.00	0.04	-0.01
80	6.82	15.01	6.86	14.99	0.04	-0.02

From a lot of barley 6 samples of 50 g each were predried for 16 hours at 65°C and mixed into one sample again. Another lot of barley was not predired. From each of the two lots 6 x 2 samples of 25 g each were taken for grinding with the Janke and Kunkel mill for six different periods, as noted in Table 9. From each ground sample, two samples were taken for drying at 130°C for two hours, 48 samples in all.

After drying and weighing the samples were again incerted in the oven and postdried for half an hour and weighed. The percentage of moisture for 60 seconds and for 80 seconds are practically equal for predried and for not predried material, and no change in moisture percentage appears by postdrying for half an hour. Furthermore at final drying (before post drying) the standard deviation calculated on the four replicates only was 0.03 per cent moisture for 60 and 80 second grinding time for predried and not predried material. For 10 seconds grinding time the standard deviations were 0.12 and 0.20 per cent moisture for predrying and non predrying, respectively.

The figures for moisture lost at post drying show that drying for two hours is not sufficient when the material is not ground or ground for 10 seconds. For 20 seconds and 40 seconds of grinding the loss at post drying is not disquieting but the figures for percentage of moisture before and after post drying as well, compared to those for 60 and 80 seconds show that grinding for 20 or 40 seconds is not sufficient for reaching the relatively precise percentage of moisture found for 60 and 80 seconds grinding time. The results emphasize the importance of the intensity of grinding, and that a control drying for half an hour does not give sufficient information if the kernels are not adequately ground.

On the disharmony in the neighbourhood of predrying

Practically it is necessary to predry a wet sample in order to carry out the disintegration. The border between wet and not wet in this sense may necessarily be conventional. In the present investigation 17 per cent moisture has been looked at as the limit for not predrying, which is in agreement with the EEC procedure (2). However if a sample without predrying shows 17.05 per cent moisture, it has to be predried. When that is done it may show a percentage of say 17.35 per cent. It means that no samples will show moisture percentages just above 17 per cent. This situation may not be in agreement with nature. It may not seem surprising that a predried sample for which the final drying is carried out as for the not predried one shows a higher percentage of moisture, because the former has been dried for some extra time (the time for predrying). Therefore it is of interest to narrow the gap between the two results.

Some samples of barley and oat with moisture content ranging from 18 to 20 per cent were broadened on the disk and left for drying at room temperature. When the moisture content reached about 17.5 to 16.5 per cent subsamples were taken partly for predrying at 45°C for 16 hours before grinding and others were ground directly for moisture determination. This system was not successfull partly because the predried samples obtained the extra drying and showed a moisture percentage of about 0.20 per cent higher than the not predried, partly because the method of "room" drying require great space. The difference between predried and not predried was larger for oat than for barley. This agree with the conclusion of Oxley et al. (8) that different types of cereals may behave differently against a particular method.

In Table 10 results from the following experiment concerning variation in time for the final drying of predried samples are set out. From a homogenous lot of barley with 15.5 per cent of moisture, samples of 100 g were taken and spread out on a plastic foil and supplied with the calculated amount of water from an atomizer in order to reach the wanted moisture percentage. Each sample of 100 g kernels was transferred to a 250 cm^3 milk bottle, closed by a rubber cork. The weight was controlled and missing water was supplied. The bottles were shaken in an end over end shaking apparatus for six hours. 18 bottles of barley were prepared in this way.

Six samples were moistened to c. 16.3, six to 16.9, and six to 17.4 per cent of moisture. After that the kernels from each group of six samples with a particular moisture percentage were collected in a plastic bag, closed, and placed at 15°C for 120 hours. After this the water will have penetrated the kernels according to Butchers and Stenverts results (3).

From each bag 10 samples of 50 g each were taken for predrying at 45°C for 16 hours. From each of the predried samples 2 samples of 25 g were taken for grinding and 5 g from each were dried. The samples were dried for different time after the oven had reached 130°C. From the same bag 4 samples of 25 g were ground and 5 g taken for drying at 130°C for 2 hours without predrying. This procedure were carried out for each of the three moisture levels. 72 meal samples in all for drying.

Table 10. Influence of time for final drying compared to drying without predrying in the neighbourhood of 17% moisture. Percentage of moisture in barley.

Not	Time for final drying, hours				
predried	½	1	1½	2	4
16.33	16.22	16.36	16.46	16.51	16.62
16.88	16.94	16.92	17.04	17.08	17.17
17.35	17.33	17.45	17.59	17.59	17.69

An analysis of variance revealed the effect of time for final drying to be highly significant according to the 99 per cent level, and an LSD value of 0.07. The interaction moisture x time of final drying was highly significant. The standard deviation was, s = 0.05. From these results it could be supposed that a final drying for half an hour or one hour of predried samples would give more harmony in the neighbourhood of 17 per cent moisture.

Meanwhile it should be borne in mind, that the predrying for the results in Table 10 has been carried out at 45°C for 16 hours. According to earlier results (9) in these investigations the predrying for 7 minutes at 130°C gives results about 0.10 per cent moisture higher than when 45°C for 16 hours are performed. It may then be concluded that the procedure used for producing the results in Table 10 cannot be accepted for bringing harmony in the neighbourhood of 17 per cent of moisture.

In the experiments reported in Table 9 it was found that barley with 15 per cent of moisture did not loose in weight at post drying for half an hour, whether the barley was predried or not. This means, as far as half an hour post drying at 130°C is accepted as satisfactory control, that as an example barley with 15 per cent of moisture can be dried at 130°C for 2 hours. Therefore, when moisture including predrying at 130°C for 7 minutes appears with a higher result than moisture determined without predrying some moisture must have escaped with the non predrying technique.

Furthermore when predrying has been carried out at a relatively low temperature it gives a lower result for percentage of moisture, than when predrying has been carried out for 7 minutes at 130°C or for instance at 65°C for 16 hours. Such findings point to the idea that moisture may escape during grinding. There cannot be any uncontrolled loss from or absorption to the predried sample from weighing until the grinding process starts immediately thereafter.

After grinding no change in moisture was going on when the meal was laying on the aluminium foil for a couple of minutes or when the meal was mixed gently. It did not make any difference in moisture whether the meal sample for drying was taken direct from the mill or the meal was emptied before the sample for drying was taken. In the grinding chamber there will be about 100 cm^3 free space but even when the temperature was 30°C, the amounts of vapor in here would not make any significant difference to the moisture content measured. Hereafter the reason for loss in moisture content might be evaporation from the sample in the mill and then the vapor escapes through the connection between the container of the mill and the lid on it. Though this connection is practically dust tight, vapor may escape.

At which step moisture may be absorbed by the meal when the kernels are predried extremely is not clear. However, when the moisture content was 6.8 per cent, only 0.05 per cent by weight was lost (Table 5) but when the kernels in other experiments were dried down to about 3 per cent of moisture, the percentage of moisture increased in process of grinding. The reason for this seems to be absorption of moisture during grinding.

Investigations concerning loss and gain in process of grinding were carried out assuming predrying at 130°C for 7 minutes and final drying and drying without predrying for 2 hours at 130°C as standard. The materials were samples of barley and oat, with majority for barley, partly originating from pilot experiments. The results of the 67 tests presented in Fig. 1 are results of four replicates each, 268 moisture determinations in all. The investigation could be described in this way. Of samples with 15-20 per cent of moisture some samples, A were predried followed by grinding and final drying. Other samples, B (from same lot) were ground and dried without predrying. The difference percent moisture for A minus percent moisture for B is designated as loss of moisture during grinding without predrying.

From another lot of kernels with 15-17 per cent of moisture some samples, C were predried for 7 minutes at 130°C followed by grinding and final drying at 130°C for 2 hours. The results obtained are accepted as the correct moisture percentages. Other samples, D (from same lot) were predried down to range from 5 to 12 per cent of moisture. These, D samples were ground and finally dried at 130°C for 2 hours. The differences in moisture percentages, C minus D may only be considered as loss or gain of moisture during grinding of the D samples.

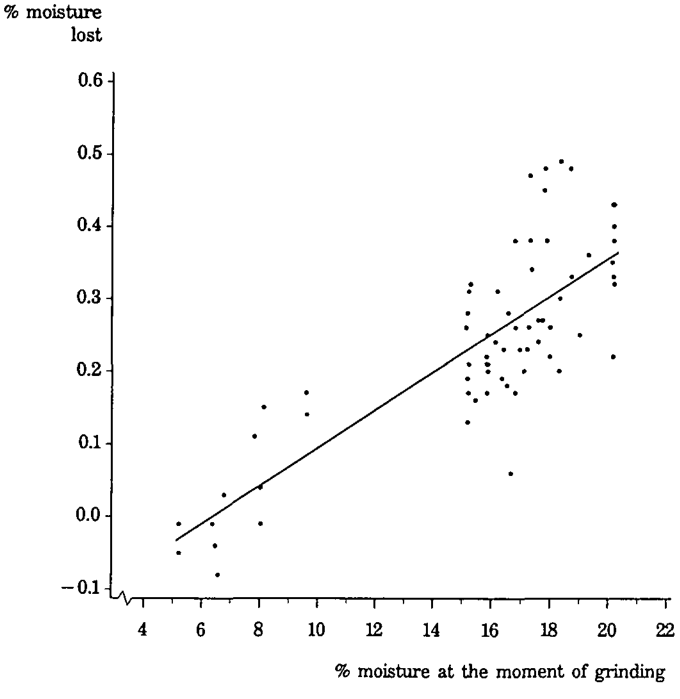

Fig. 1. Loss of moisture in process of grinding barley samples with different moisture content.

The equation of regression worked out was $y = 0.0261\,x - 0.1665$, where y means lost per cent of moisture, and x means the percentage of moisture in the kernels at the time when grinding is carried out. For the equation the $S_{y/x}$ and S_a were 0.080 and 0.038 percentage of moisture respectively, and S_b = 0.002. The correlation coefficient was r = 0.81, which was highly significant according to the 99 per cent level.

Considering Butchers and Stenverts (3) and Oxley et al.'s (8) results a great variation might be expected, when different types and varieties are included in the investigation. On the other hand variation in cereal samples for moisture determinations is just the situation in the professional skill of analytical work.

Within the area in question the equation seems to work quite well. For 6, 15 and 17 per cent of moisture the loss will be calculated to -0.01, 0.23, and 0.28 per cent of moisture, and these correspond quite well with the loss at grinding according to the figures in Table 5.

Discussion and conclusion

The task of this investigations has been to obtain a qualified idea on errors in the various steps of the procedure for determination of moisture from the analytical sample of kernels and forward. The intact kernels are more moisture stabile than the meal and therefore it is important to make sure that the analytical sample and the predried sample are carefully mixed, each of them. In process of grinding the important points are cooling of the mill, avoiding air stream through the mill, and grinding for sufficient time in order to reduce the size of particles satisfactorily. Although some flakes are there, when the Janke and Kunkel mill (a coffee mill) is used, they seem to play a small role, perhaps the flakes are scratched and the moisture may evaporate easy during drying. The flakes seem to make a small percentage of the sample. When samples are taken direct from the mill evaporation caused by mixing is avoided. Although there is no draft of air through the mill in process of grinding, loss of moisture occurres in this process and maybe also, when the mill is opened. The loss of moisture from a sample just below a border fixed for predrying may be important, and seems unavoidable, why all samples should be predried. Though samples are predried not always the same degree of dryness is reached and therefore a correction for loss of moisture in relation to the moisture content in the sample at the time of grinding should be considered.

References

1. *Anderson, J. L.* 1936. Some facts concerning vacuum-oven moisture determination. Cereal Chem. 13, 437-52.

2. *Anonymous.* 1971. De Europæiske Fællesskabers Tidende 20.12.71 No. 1, 279/7 Kommissionens andet direktiv af 18. November 1971. Bestemmelse af vandindhold 859-61.

3. *Butcher, J. & Stenvert, N. L.* 1973. Conditioning studies on australian wheat. III The role of the rate of water penetration into the wheat grain. J. Sci. Fd. Agric. 24, 1077-84.

4. *Flohil, J. T.* 1931. Protein and moisture determinations in wheat with special reference to conditions covering preparation of samples for analysis. Cereal Chem. 8, 306-15.

5. *Karmas, E.* 1980. Techniques for measurement of moisture content in foods. Food Technol. 34(4), 52-59.

6. *Michael, G.; Blume, B. & Faust, H.* 1961. Die Eisweisqualität von Körnern verschiedener Getreidearten in Abhängigkeit von Stickstoffversorgung und entwiklungszustand. Z. Pflanzenernähr. Düng. Bodenk. 92(137), 106-16.

7. *Nelson, O. A. & Hulett, G. A.* 1920. The moisture content of cereals. J. Ind. and Eng. Chem. 12, 40-45.

8. *Oxley, T. A. ; Pixton, S. W. & Howe, R. W.* 1960. Determination of moisture content in cereals. I Interaction of type of cereal and oven method. J. Sci. Food Agric. 11, 18-25.

9. *Sørensen, C.* 1992. Moisture determination in cereals, analytical details in oven drying. I. Handling of intact kernels and loading of oven.

10. *Williams, P. C. & Sigurdson, J. T.* 1978. Implications of moisture loss in grains incurred during sample preparation. Cereal Chem. 55(2) 214-29.

11. *Wilson, M. A.* 1986. Comparison of alternate approaches to measurement of moisture in grains and oil seeds. Cereal Foods World 31(6), 406-08.

Afdelinger under Statens Planteavlsforsøg

Direktionen

Direktionssekretariatet, Skovbrynet 18, 2800 Lyngby	45 93 09 99
Afdeling for Biometri og Informatik, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00

Landbrugscentret

Centerledelse, Fagligt Sekretariat, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00
Afdeling for Grovfoder og Kartofler, Forskningscenter Foulum, Postbox 21, 8830 Tjele	89 99 19 00
Afdeling for Industriplanter og Frøavl, Ledreborg Allé 100, 4000 Roskilde	42 36 18 11
Afdeling for Sortsafprøvning, Teglværksvej 10, Tystofte, 4230 Skælskør	53 59 61 41
Afdeling for Kulturteknik, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00
Afdeling for Jordbiologi og -kemi, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00
Afdeling for Planteernæring og -fysiologi, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00
Afdeling for Jordbrugsmeteorologi, Forskningscenter Foulum, Postbox 23, 8830 Tjele	89 99 19 00
Afdeling for Arealdata og Kortlægning, Enghavevej 2, 7100 Vejle	75 83 23 44
Askov Forsøgsstation, Vejenvej 55, Askov, 6600 Vejen	75 36 02 77
Borris Forsøgsstation, Vestergade 46, 6900 Skjern	97 36 62 33
Jyndevad Forsøgsstation, Flensborgvej 22, 6360 Tinglev	74 64 83 16
Rønhave Forsøgsstation, Hestehave 20, 6400 Sønderborg	74 42 38 97
Silstrup Forsøgsstation, Oddesundvej 65, 7700 Thisted	97 92 15 88
Tylstrup Forsøgsstation, Forsøgsvej 30, 9382 Tylstrup	98 26 13 99
Ødum Forsøgsstation, Amdrupvej 22, 8370 Hadsten	86 98 92 44
Laboratoriet for Biavl, Lyngby, Skovbrynet 18, 2800 Lyngby	45 93 09 99
Laboratoriet for Biavl, Roskilde, Ledreborg Allé 100, 4000 Roskilde	42 36 18 11

Havebrugscentret

Centerledelse, Fagligt Sekretariat, Kirstinebjergvej 10, 5792 Årslev	65 99 17 66
Afdeling for Grønsager, Kirstinebjergvej 6, 5792 Årslev	65 99 17 66
Afdeling for Blomsterdyrkning, Kirstinebjergvej 10, 5792 Årslev	65 99 17 66
Afdeling for Frugt og Bær, Kirstinebjergvej 12, 5792 Årslev	65 99 17 66
Afdeling for Planteskoleplanter, Kirstinebjergvej 10, 5792 Årslev	65 99 17 66
Laboratoriet for Forædling og Formering, Kirstinebjergvej 10, 5792 Årslev	65 99 17 66
Laboratoriet for Gartneriteknik, Kirstinebjergvej 10, 5792 Årslev	65 99 17 66
Laboratoriet for Levnedsmiddelforskning, Kirstinebjergvej 12, 5792 Årslev	65 99 17 66

Planteværnscentret

Centerledelse, Fagligt Sekretariat, Lottenborgvej 2, 2800 Lyngby	45 87 25 10
Afdeling for Plantepatologi, Lottenborgvej 2, 2800 Lyngby	45 87 25 10
Afdeling for Jordbrugszoologi, Lottenborgvej 2, 2800 Lyngby	45 87 25 10
Afdeling for Ukrudtsbekæmpelse, Flakkebjerg, 4200 Slagelse	53 58 63 00
Afdeling for Pesticidanalyser og Økotoksikologi, Flakkebjerg, 4200 Slagelse	53 58 63 00
Bioteknologigruppen, Lottenborgvej 2, 2800 Lyngby	45 87 25 10

Centrallaboratoriet

Centrallaboratoriet, Forskningscenter Foulum, Postbox 22, 8830 Tjele	89 99 19 00

Frederiksberg Bogtrykkeri a·s 92 1432