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Research Report

Final Report

Long-Term Storage of Canola

For:

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Long-Term Storage of Canola

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1. Abstract

Testing to determine the effect of seed moisture content (MC) and storage temperature on seed oil quality of four different canola samples was undertaken. Samples of green, clean, standard, and high oil content canola seed were conditioned to seven different MC and stored at five different temperatures for approximately four months. The temperature within all 140 test samples was monitored for an increase that might indicate spoilage. Two months after testing began the samples were tested for Acid Value (AV), Free Fatty Acid (FFA), Peroxide Value (PV), and P-Anisidine Value (ANV). These values were compared with tests done on the initial base samples to determine if oil quality had changed.

After analysing the lab results, and physically inspecting the samples, it was apparent there had been spoilage in the form of mold growth in several samples. However, there was no indication of spoilage from the temperature traces within the samples. Oil quality tests indicated that oxidation and oil quality deterioration processes had begun but had not necessarily exceeded accepted tolerance limits.

Additional tests to establish the effect of canola compression under its own in weight in tall bins was also performed. A compression test stand was developed to simulate the compression load of up to a 30.48 m (100 ft) column of grain. Samples of standard and high oil content canola were monitored under load, and measurements of compressed displacement were recorded. Samples were inspected for signs of oil exudation, and after compression testing, were sent for germination testing. None of the tested samples showed any negative effect due to the compression.

2. Introduction

Canola is a major cash crop in Saskatchewan and has steadily increased in acreage seeded each year. Producers are now growing canola with much higher oil content compared to 10 years ago. The current recommendations for safe storage of canola are based on previous varieties, which had much lower oil content than those commonly grown today. As a result, some storage issues that have arisen have been blamed on changes to canola seed from the increased oil content.

Additionally, bin size has increased in conjunction with higher oil content seeds causing concerns regarding the effect of compaction and compression due to the weight of canola in taller bins.

This report outlines tests conducted by PAMI to try to establish seed condition criteria and environmental conditions that could be recommended as safe for long-term storage of canola. It also looks at the possible impact of large storage bins on high oil content canola.

3. Methods

This project consisted of three testing activities. The primary activity was conducting small-scale canola storage trials to investigate the effects of temperature and grain MC on stored canola. The second tests were made to evaluate the compressive effect on seed stored in tall bins, looking primarily at the effect on the structural integrity of canola seeds. The third test was aimed at a larger-scale, longer-term storage trial, but the inconclusive results from the small-scale trial suggested that this was not practical at this time.

Additional testing investigated the instrumentation, test procedures, and assumptions involved in the small-scale testing. These were conducted at PAMI's expense in order to check the validity of the results and to provide direction for future testing.

3.1 Small-scale Storage Testing

To evaluate the effect of temperature and MC during storage, four base samples of canola were obtained. These included standard clean canola, high green count canola, high oil content canola, and high dockage canola. From these base samples, individual sample containers were prepared for testing in seven temperature and five MC conditions.

Upright freezers were converted into environmental chambers to provide five different temperature test conditions (**Figure 1**). The freezers were placed in a heated work area where they would not be disturbed during the six month test. Plastic shelving was removed from the doors to provide additional interior space for the 28 containers of canola undergoing tests in each unit. To accommodate instrumentation cables, a hole was drilled in the freezer doors and a rubber hose was installed as a protective grommet, (**Figure 2**).

Ranco Electronic temperature controls regulated each freezer's power supply using feedback from the temperature within the chamber to maintain the chamber's target temperature.

Figure 1. Five environmental chambers with thermocouples installed.

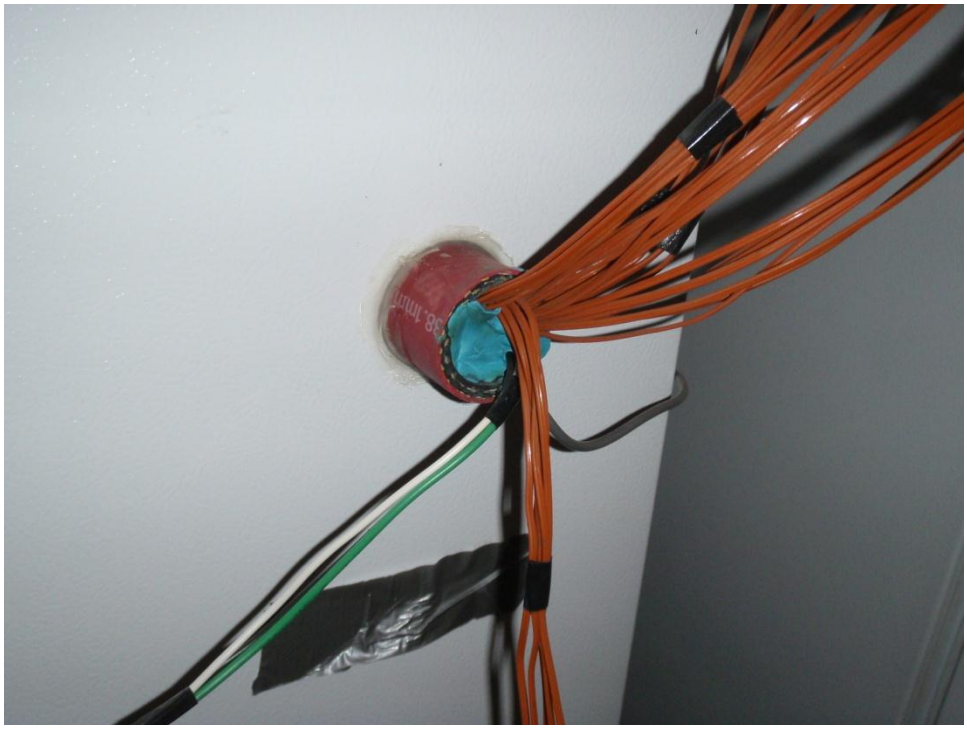

Figure 2. Rubber hose used as grommet for wire routing through door.

Samples were placed in 9 L (2 gallon) pails with lids. A hole was drilled in each pail for installation of a thermocouple. The thermocouple was installed to measure temperature as close to the center of the pail as reasonably possible. In the colder chambers, duct tape was used to seal the pails so that outside moisture would not get into the test sample.

Circulation fans were installed (**Figure 3**) to help maintain consistent temperature throughout the chambers. All thermocouple wires and fan power cords were routed through the holes in the doors. In the two warmest chambers (25°C (77°F) and 35°C (95°F)), a 400 W heater was installed to maintain the target temperature and a Proportional–Integral–Derivative (PID) controller was used to regulate both the freezer and heater operation.

Figure 3. Fan locations within each chamber.

A DataTaker Series 80 logged temperature in the 140 test samples (**Figure 4**). The DataTaker also recorded air temperature data from 25 thermocouples located within the environmental chambers, and three thermocouples were used to monitor ambient temperature in the testing room. The temperature was logged every 30 seconds for approximately the first month and every five minutes thereafter.

Figure 4. An internal view of environmental chamber during testing.

3.1.1 Sample Preparation

One hundred and forty sample containers were prepared from four different base canola samples. Each base sample was divided into subsamples for testing at seven MC and five temperatures resulting in 35 treatments for each base sample (35 treatments x four base canola samples = 140 samples total). **Figure A1** in **Appendix I** shows the test sample matrix.

Three of the base samples, including high oil content, high green count, and high dockage were obtained from the locations (origin) shown in **Table 1**. The high dockage sample was divided in two and the dockage was removed from one half to create the fourth clean base sample. Before the test samples were prepared, a small 150 g (0.15 kg) subset of each base sample was sent to POS Pilot Plant in Saskatoon, Saskatchewan, for AV, FFA, PV, ANV, moisture and volatiles, and oil content testing. A dockage and distinctly green seed count was then conducted on each sample by Bunge at Dixon, Saskatchewan.

Obtaining the three base samples proved more difficult than originally envisioned. First, it was late in the crop year and many producers had sold their canola. Secondly, oil content is not a commonly available specification in the industry. Farmers have no test method, elevators do not normally test for it, and although crushers do test it, they do not know the results until after the specific batch of canola has been mixed with the rest of the canola in their system. Without the benefit of a test for a specific bin of canola, the best indicator for potential oil content seems to be the typical oil content for a particular

variety. However, variety is not necessarily a guarantee of high oil content. Dependent on moisture and growing conditions, oil content can vary. As it turned out, the highest oil content canola that was obtained was 47.4%.

Table 1. Origin of base sample.

	High Oil Content Canola	High Green Count Canola	Clean Canola	High Dockage Canola
Amount (bu)	10	10	10	10
Origin	Bob Bartkewich G.D. Station Main North Battleford, SK S9A 2X5	Mark Gabriel Box 124 Englefeld, SK S0K 1N0	Alfabee Farms Box 503 Watson, SK S0K 4V0	
Variety	Cargill Victory 1037 INC	Invigor 5020	Dekalb 72-55 RR	
Crop Year	2009	2009	2009	
Oil Content	47.4%	45.9%	45.3%	45.8%
Moisture Content	7.74%	11.5%	8.71%	9.00%
Acid Value (mg KOH/g)	0.48	1.75	0.92	1.23
Free Fatty Acid	0.24%	0.88%	0.46%	0.62%
p-Anisidine Value	0.11	0.89	0.2	0.27
Peroxide Value (meq/kg)	0.2	1.8	0.2	0.35
Dockage	2.2%	1.6%	1.0%	3.3%
Distinctly Green Count	0.0%	30.3%	0.2%	1.0%
Approximate Harvest Date	September 9,2009, to September 11, 2009, and October 21, 2009, to October 23, 2009	Early December 2009	November 7, 2009	
Harvest Conditions	Plenty of snow and rain in between, early canola was approximately 8% MC and late canola was approximately 15%. All canola was mixed and aerated.			

To standardize testing, all canola samples were dried to less than 7% MC using PAMI's Natural Air Drying (NAD) research equipment. The NAD equipment consists of six vertical, 3.35 m (11 ft) high, 0.45 m (18 in) diameter cylindrical bins supported by load cells. Each bin is equipped with a fan that blows air into the bottom of the bin. Four sample ports are located along the height of each bin to allow sampling at the various levels.

Each base sample was mixed in a large rotary mixer for 20 minutes, and then randomly divided into seven separate batches of approximately 54.5 L (12 gallons) each. The mass and moisture content of each batch was measured, then the mass of water required to bring the moisture content up to the desired level was calculated. As each batch was mixing in a smaller rotary mixer, an appropriate amount of water was misted onto the seed with a hand sprayer. Each batch was then mixed for one half hour before being separated into five separate 9 L (2 gallon) test pails. The equation for calculating the water to be added to the samples is as follows.

$$\boldsymbol{Water\ to\ add\ (g)} = \left[\frac{(\boldsymbol{m_1 * mc_2})(\boldsymbol{1 - mc_1})}{(\boldsymbol{1 - mc_2})} - \boldsymbol{mc_1 * m_1}\right] \qquad [1]$$

where: m_1= initial mass (g)
mc_1=initial moisture content (decimal, wet basis)
mc_2=target moisture content (decimal, wet basis)

After moisture conditioning, the test samples were inserted into the temperature-controlled chambers with the time and date noted for each batch. All batches were placed in their respective environmental chambers between September 14, 2010, and September 29, 2010. The sample locations within a chamber are shown on the pail map (**Figure A2**, **Appendix I**). The sample location was chosen randomly once and that same pattern was used for all five temperatures trials.

Once MC had reached equilibrium, the MC was measured on each test sample approximately one week after the samples were put in the chambers. After two months, all 140 samples were again removed, physically inspected, and sampled for AV, FFA, PV, and ANV testing.

3.2 Structural Integrity (Compression) Testing

PAMI designed and fabricated a test stand that enabled weight to be added incrementally on top of a sample of canola seeds to simulate the force applied by the vertical column of canola seed stored in a tall bin.

The test stand consisted of a 13.3 cm (5.25 in) diameter tubular steel container with a removable bottom, a plunger assembly, and a support frame (**Figure 5**). The removable bottom (**Figure 6**) facilitated placement of a filter paper disc at the base of the sample. This allowed visual assessment of possible oil exudation. The plunger assembly employed a central mast that accommodated the addition of 11.33 kg (25 lb) weights.

Figure 5. Canola compression test stand.

Figure 6. Compression testing container with removable bottom.

For each test, a filter paper disc was placed at the bottom of the tube, which was then partially filled with moisture-conditioned canola seed. Another filter paper disc was placed on top of the canola in the column to check for oil seep at the plunger surface. The plunger assembly was inserted into the canola-filled tube. A “zero compression” measurement between the container edge and plunger surface was recorded once the plunger assembly was installed. Each time weight was added to the plunger assembly, the initial compression was measured and documented.

Based on dimensions of the tube and plunger, the weight of a vertical column of canola seed was calculated. Weight was alternately added in 22.67 kg (50 lb) and 34 kg (75 lb) increments to simulate the addition of approximately 3 vertical m (10 ft) of grain at a time starting from approximately 6 m (20 ft) and increasing to approximately 30 m (100 ft) (total targeted weight was about 290 kg (640 lb)). **Table 2** indicates the correlation between weight and grain column height. **Table 3** denotes the number of weights (with the plunger assembly) required to achieve the grain column height targets.

Table 2. Correlating weight of specified heights of grain column.

Target height of grain (ft)	Grain Weight (lb)
20	126.0
30	189.1
40	252.1
50	315.1
60	378.1
70	441.2
80	504.2
90	567.2
100	630.2

Table 3. Number of weights (including 13.1 lb plunger assembly) correlated to grain height.

# of 25 lb weights	total weight (lb)	Height of grain (ft)
5	138.1	21.9
7	188.1	29.8
10	263.1	41.7
12	313.1	49.7
15	388.1	61.6
17	438.1	69.5
20	513.1	81.4
22	563.1	89.3
25	638.1	101.2

After 25 weights had been added, they were left in place for a minimum of three days and the displacement was again measured. For some samples, displacement was measured again as the weights were removed to determine the permanent seed compression. After all weight was removed, the canola samples were inspected for structural damage and/or oil exudation.

Four separate samples were tested – Standard canola at low and mid MC, and high oil content canola at mid and high MC. The samples were conditioned to target MC of 5%,

8%, and 11%. The standard canola came out at 5% and 8.4% MC, and high oil-content canola at 7.3% and 10.5% MC.

Germination tests were conducted on each sample, prior to and again after compression testing, as an additional metric for evaluating structural integrity.

3.3 Larger Volume Storage Testing

Initially, it was planned to duplicate the intent of the small-scale storage testing using larger volumes of seed over a longer period of time. This testing would have used the PAMI NAD test facility which consists of a set of instrumented bins with precise aeration control and data acquisition for measuring temperature and moisture.

The intention was to conduct trials using seed with the highest MC for "safe storage" as determined by the small-scale testing. However, due to the inconclusive and conflicting nature of the small-scale testing results and questionable procedure, it was decided to not proceed with this testing.

3.4 Additional Testing

After completing the small-scale storage testing, several concerns arose. The data obtained conflicted with some assumptions that were the basis for the test procedure. It was believed that heat produced when the seed spoiled within the samples would be an early and reliable indication of grain spoilage. However, the temperatures recorded during the tests did not show any heat generation, yet many of the samples were believed to have spoiled. This will be discussed in greater detail in **Section 4**.

Several possible explanations for the lack of a measurable heat rise were considered and included:

- The thermal cooling capacity of the environmental chambers (freezers) was much greater than the sample's ability to produce heat; since the cooling coils were in direct contact with the sample containers, they effectively cooled the sample as fast as the canola could generate heat.
- The size of the sample was too small to either achieve a needed mass critical for generating heat or to provide sufficient thermal mass to insulate, contain, and sustain the heat generated by the sample.
- The data collection equipment failed to register the heat generated or failed to detect heat change.
- Heat was not generated by the samples.
- Spoilage that normally takes place in bins was different than what occurred in the samples.

PAMI undertook additional testing to address some of these possibilities. Three test cells of increasing size were manufactured from 3/4 in. plywood. The small test cell replicated the volume of the containers used in the small-scale storage testing (approximately .25 bu, approximately 8.1 L), the medium had approximately 1 bu (35.2 L) capacity, and the large was approximately 5 bu (176.2 L). Each box was insulated with rigid styrofoam insulation (**Figure 7**), which was equivalent to approximately 3 m (10 ft) of canola and effectively represented the conditions canola would be at in the center of a full 6 m x 6 m x 6 m (20 ft x 20 ft x 20 ft) bin.

Figure 7. Insulated test cells.

The boxes were instrumented using the same data-taker and thermocouples used previously, as well as another redundant, autonomously operated temperature measuring system. Instrumenting the cells in this fashion enabled comparison of the data from the two systems, which would confirm if the equipment had malfunctioned during the first tests.

Thermocouples were placed in the cells near the center, and along one side, as well as at several external locations for measuring the ambient temperature in the room where the test cells were located (**Figure 8**).

Figure 8. Multiple thermocouples in the test cells.

Canola seed was conditioned to 11.8% MC using a rotary mixer and the same procedure as used for the small-scale storage trials. Approximately 12% MC was selected, as previous testing suggested this would be a MC at which spoilage would occur, regardless of temperature. Each test cell was filled nearly full with canola before installing the lid and starting the temperature monitoring.

The temperature in and around the test cells was data logged for nearly six weeks, before samples were sent for oil quality analysis. A second test, under similar conditions, was conducted without the lids installed. Temperatures were monitored for approximately a month, but no oil analysis was performed at the end of the test.

4. Results

Test results from the various tests conducted are discussed in detail below.

4.1 Small-Scale Storage Testing

The samples for the small-scale storage trials were moisture conditioned to the desired MC by adding water according to the equation shown in **Section 3.1.1**. The equation worked well within reasonable tolerance, except for one condition. The MC of all seven high oil content batches was approximately one percentage point higher than anticipated when using the calculation. Considering this, the decision was made to discard the highest moisture content sample (12%) and prepare a new sample at the low end of the range (7%). The actual moisture contents for the rest of the test samples were also slightly higher than anticipated, but were within a suitable tolerance range. **Table A1** (**Appendix I**) denotes the sample moisture contents measured approximately one week after sample preparation and placement in the chambers.

The small-scale storage sample containers were stored for approximately four months in the environmental chambers at the various target temperatures. When inspected after two months of testing, spoilage in the form of mold and spores was observed on several samples, especially in the higher 25°C (77°F) and 35°C (95°F) environments (**Figure 9**). When these samples were disturbed, clumps were evident and a musty, “spoiled” odour was present.

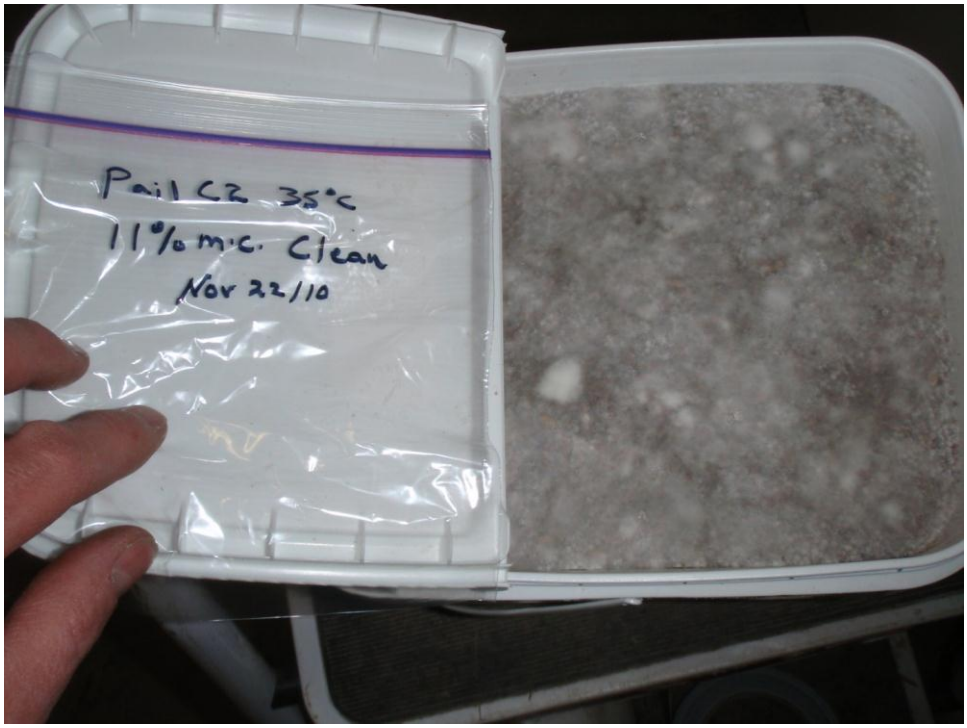

Figure 9. Pail 35°C 11% MC clean sample shown after two months in the chamber.

However, despite the odor and visible spoilage, the temperature traces for these samples did not register any distinguishable heating. **Figure 10** displays the temperature data for a visibly spoiled sample. Obviously there are extreme data points in this temperature graph that are suspected to be false (which is discussed in **Sections 3.4 and 5.3**), but the trend is a relatively stable temperature. The lack of a registered heating trend in these samples, combined with the "noisy" data, caused concern that heat generated by the samples was not being recorded by the data collection devices.

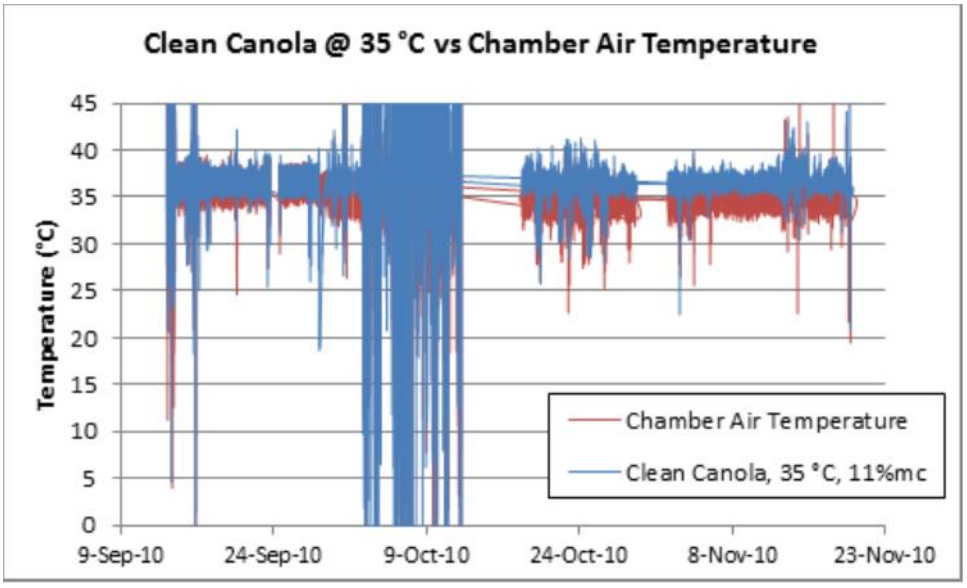

Figure 10. Visibly spoiled sample temperature trace.

Further analysis of the temperature data revealed that sample temperatures followed in step with the ambient environmental chamber temperature (**Figure 10**). This data suggests that it is unlikely that temperature rise went undetected in the samples, but rather that there was no rise in sample temperature.

Notable in **Figure 11** is that when the chamber temperature went down (around time stamp 00:30) the sample's temperature dropped substantially, but when the chamber temperature went up (around time stamp 0:00), sample temperature was not affected. The unaffected sample temperature may have been due to the fact that the sample containers were supported by the freezer's cooling coils, which are incorporated into the shelves. Possibly, the 9 L (2 gallon) sample size was too small, and not insulated well enough, to allow heat produced within the sample to overcome the cooling effect of the environmental chamber and register a temperature fluctuation. If the samples had been larger, or better insulated, the temperature within the canola samples may have risen enough to be detected by the thermocouples.

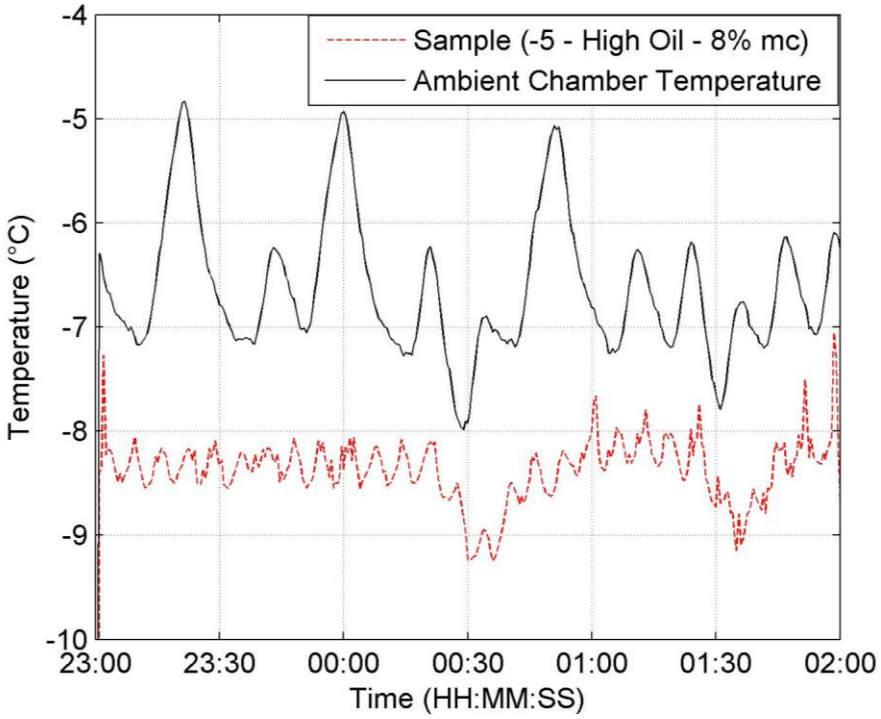

Figure 11. Ambient chamber temperature (E2) versus the temperature of a test sample (A3) over time.

With visibly spoiled samples, that lacked a sample temperature increase to provide early indication of spoilage activity, oil quality analysis was employed to verify canola condition.

POS Pilot Plant in Saskatoon conducted three tests on each sample (AV/FFA, p-AnV, PV). The original four base samples were tested at the beginning of the project and then

approximately two months after testing began. Samples were taken from all 140 test samples for the same lab analysis.

Initially the oil quality results were evaluated for positive percentage change relative to the base samples. **Table A1** through **Table A5** (**Appendix 1**) list the changes in the oil quality values of each sample, as a percentage of the base sample value. In these tables, an increase greater than 30% was assigned the maximum threshold before loss of oil quality, and samples highlighted in red are deemed to have spoiled.

Table A2 and **Table A4** are basically the same because AV and FFA are closely related, and calculated from the same measurement (AV = FFA x 1.99). As expected, the higher temperature environments showed a larger change in AV. Very few samples in the -5°C (23°F) and 5°C (41°F) chambers exceeded the threshold. Ten test samples in the 15°C (59°F) chamber, sixteen in the 25°C (77°F) chamber, and all 28 test samples in the 35°C (95°F) chamber spoiled. **Table A2** also show that in every chamber the high oil content test samples had greater change in AV, and presumably spoilage.

Table A3 illustrates the change in the PV, again with changes greater than 30% highlighted in red. Approximately seven samples in each of the -5°C (23°F), 15°C (59°F), and 25°C (77°F) chambers had greater than 30% change, while the 5°C (41°F) chamber had none and the 35°C (95°F) chamber had 12. This is not a clear progression like the AV, as some samples experienced a decrease in PV, nor is there a clear difference between the high oil test samples and the others. This is a reasonable result.

PV is a measure of primary oxidation products and is an indicator of the initial stages of oxidation. As oxidation progresses to successive stages, PV will decrease. Inconsistent or decreased PV results may just be an indication of continued oxidation progression rather than a signal that oxidation is not occurring.

As PV is an indicator of early stages of oxidation, p-AnV is a reflection of the later stages. It is a measurement of secondary oxidation products. **Table A5** lists the changes in p-AnV for the test samples. Though not a clear progression with temperature increase, all but two of the samples in the high 35°C (95°F) temperature environments exceeded the p-AnV change threshold. Apart from some of the clean samples in the 15°C (59°F) and 25°C (77°F) environments, only the high oil content samples indicated a greater than 30% change in the lower temperature environments. The high oil content samples consistently showed the greatest increases in p-AnV throughout the temperature range.

Analyzing these test results on the basis of relative change provides a good indication of whether the processes of oil quality degradation are in progress, but PAMI has come to understand that there are some industry accepted, absolute tolerance thresholds

associated with these values as well. **Table A6** to **Table A8** (**Appendix 1**) contain the raw test results (same ones used to calculate the relative percentage of change tables).

The industry standard for FFA oil content is a 1% maximum. **Table A6** denotes the tested FFA values for all the samples with values greater than 1% highlighted in red. This chart shows a similar picture to the percentage of change table with only the samples in the higher temperature (25°C (77°F) and 35°C (95°F) environments exceeding the maximum. Interestingly, in the 35°C (95°F) chamber, the high oil content samples were not greater than the 1% maximum, but most of the other samples were. In the 25°C (77°F) environment, many of the green samples were “spoiled”.

Generally, a lower PV is considered desirable, but maximum acceptable levels range from 2 meg/kg to 5 meq/kg. Using the lower threshold value, **Table A7** lists the PV with the values greater than 2 meq/kg highlighted in red. In this table, very few samples exceeded this lower limit, and of those, all were below the upper limit of 5 meq/kg. Only Green samples were beyond the threshold, and interestingly, none of the high oil-content samples were.

Maximum p-AnV is not as clearly defined. Again, lower values are desired from an oil quality perspective. Ranges from 0.5 up to 6 are cited as acceptable limits. **Table A8** provides the p-AnV results, with values greater than 0.5 highlighted in red to illustrate a threshold at the lower limit. Under all temperature conditions, most of the Green samples exceed this threshold. But, the initial sample p-AnV before storage tests commenced was above this threshold. Only two of the high oil content samples surpassed the threshold, but not in the highest temperature chamber. It should be noted that none of the samples p-AnV was higher than approximately 2, which is still substantially below the upper limit of the range.

Totox is a value used in the food oil industry to measure total oxidation. It is a combination of PV and p-AnV, calculated as follows: 2PV + p-AnV. Lower Totox values are desirable, but a quality threshold of four is used for soybean oil and believed to be applicable to canola oil. In **Table A9** the Totox values of the tested samples are listed. Only the Green samples had Totox values greater than four, and mostly at lower temperatures, but again, the Totox of the initial sample was beyond this limit.

4.2 Structural Integrity (Compression) Testing

Several types of data were collected from these tests to identify specific indicators that might indicate the effect on seed structural integrity. Visual observation of seed damage and seed shape/deformation, oil exudation, displacement under compression, and seed germination were all used. Treatment effects with respect to MC and oil content were investigated.

4.2.1 Observations

After testing, the samples did not appear to have incurred excessive damage or deformation. The layer of seed immediately below the plunger looked to be slightly deformed, having moderately flattened sides and cube-like shapes (**Figure 12**). This phenomena is thought to have been an “edge effect”, the only seeds affected were those that were in direct contact with the plunger surface. Some seeds in this same area were ruptured (**Figure 13**), but there were only a few and this was also attributed to the edge effect, as no other ruptured seeds were observed throughout the sample.

Figure 12. Deformed seeds at the top of the column against the plunger.

Figure 13. Ruptured seeds on the plunger.

Oil seepage was not apparent in any of the tests. The filter papers at both the top and bottom of the samples showed no evidence of oil absorption (**Figure 14**), although some indentations from seeds and the odd ruptured seed were observed (**Figure 15**).

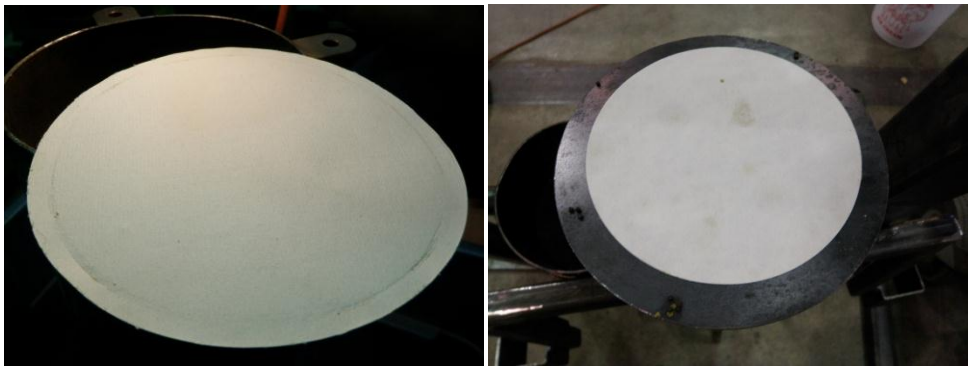

Figure 14. Filter paper discs with no evidence of oil.

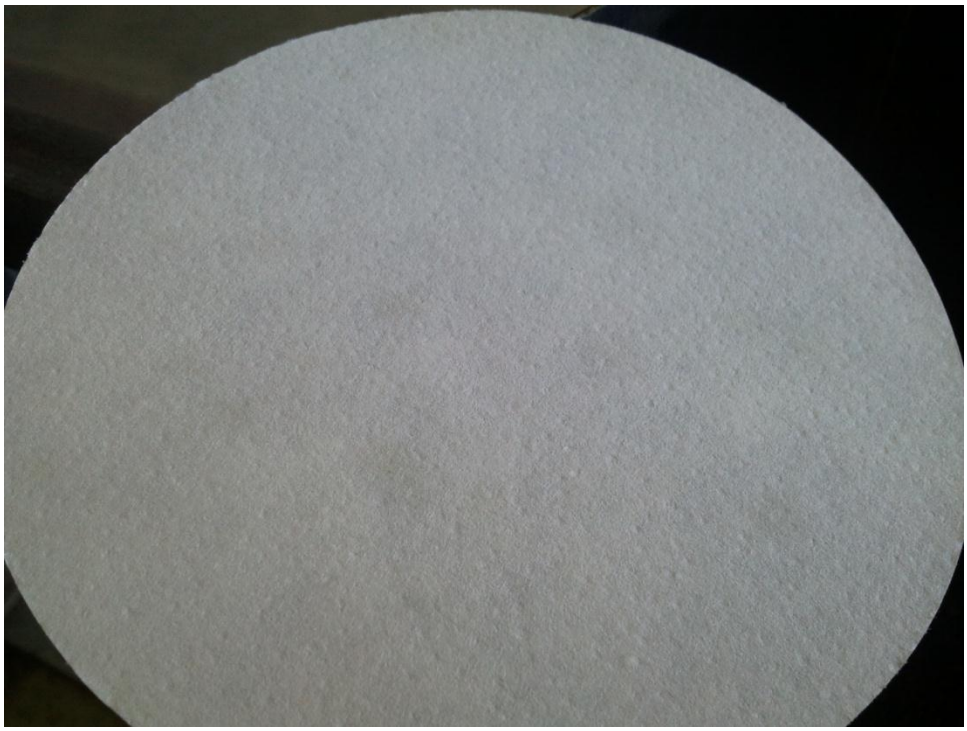

Figure 15. Seed indents evident on filter paper disc.

4.2.2 Compression Displacement

Displacement due to compression was measured from the edge of the sample container to the top of the plunger during loading (**Figure 16**). This measurement was taken immediately after weight was added and again three to five days later the weight still applied.

Figure 16. Compression displacement measurement method.

Table 4 charts the displacement of the standard canola sample at 5% and 8.4% MC as well as the high oil-content sample at 7.3% MC. Measurements for the high oil content sample at 10.5% MC were recorded but unfortunately were misplaced after testing was completed.

Table 4. Grain compression in mm as weight increases.

		Standard Canola		High Oil Canola
Height of Grain (ft)	**# of weights**	**~5%**	**8.4%**	**7.3%**
0	**0**	0	0	0
21.9	**5**	4	5	6
29.8	**7**	5	7	7
41.7	**10**	7	10	9
49.7	**12**	8	12	10
61.6	**15**	9	14	13
69.5	**17**	10	16	14
81.4	**20**	11	19	15
89.3	**22**	12	21	16
101.2	**25**	13	22	18

A graph of the results in **Table 4** shows that compaction was relatively linear up to the max weight/height target (**Figure 17**). It also suggests that the MC may be a factor in the

amount of displacement that takes place as the canola is compressed. As MC increases it appears that displacement increases with increased loading.

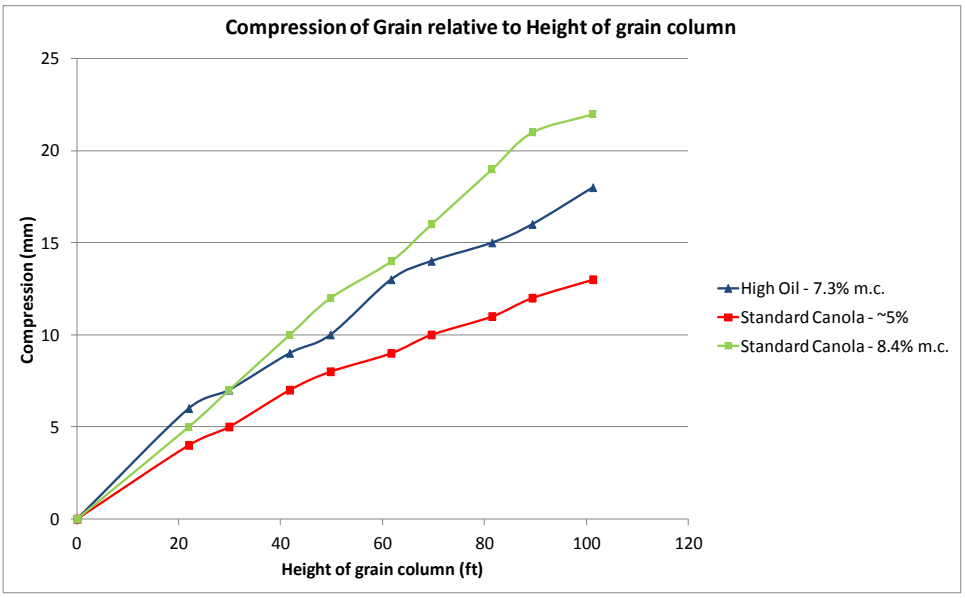

Figure 17. Canola compression at simulated grain column heights.

It is difficult to determine from this data whether higher oil content has an effect on compressibility. The difference between the standard and high oil content samples at approximately 8% MC is minimal, and as it is suspected that MC **does** affect compression, the increased displacement may be attributable to the higher MC of the standard sample. With only one repetition, that difference may not be significant.

However, displacement under sustained compression for several days shows a more pronounced difference. **Figure 18** illustrates a far greater difference in total displacement between the standard and high oil content samples at approximately 8% MC. The magnitude of compression was much greater compared to the initial displacement measure at the time of loading as both sample's displacement nearly doubled over time. This result is for very limited testing so no conclusions should be drawn. It should be considered that additional seed and varietal characteristic differences between the standard and high oil content samples, including seed size and shape, seed coat durability, seed age, and previous handling are not accounted for with this testing.

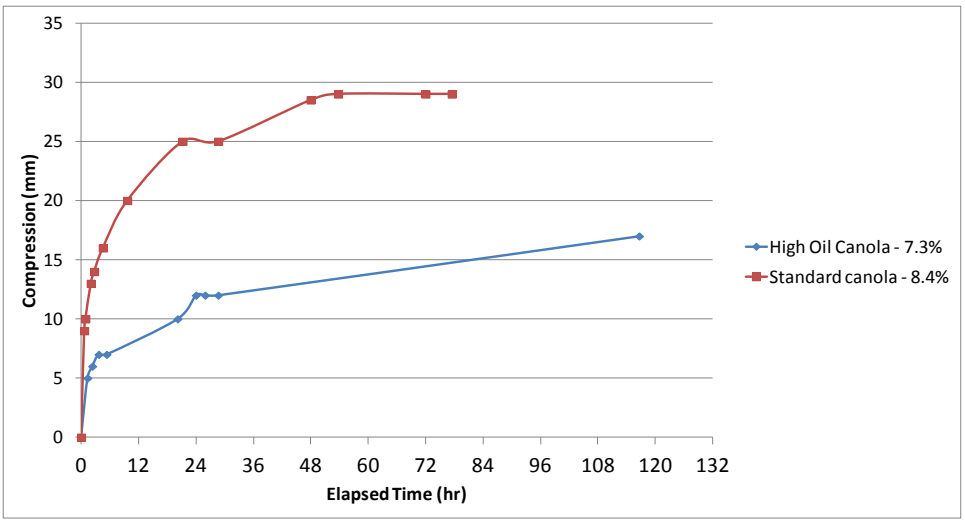

Figure 18. Sustained load compression displacement over time.

Measurement of the displacement as the compressive loading was removed showed that the canola "rebounded" very little. **Figure 19** indicates that any settling or seed deformation was relatively permanent, and that the seeds were not compressed like springs waiting to rebound once the load was removed.

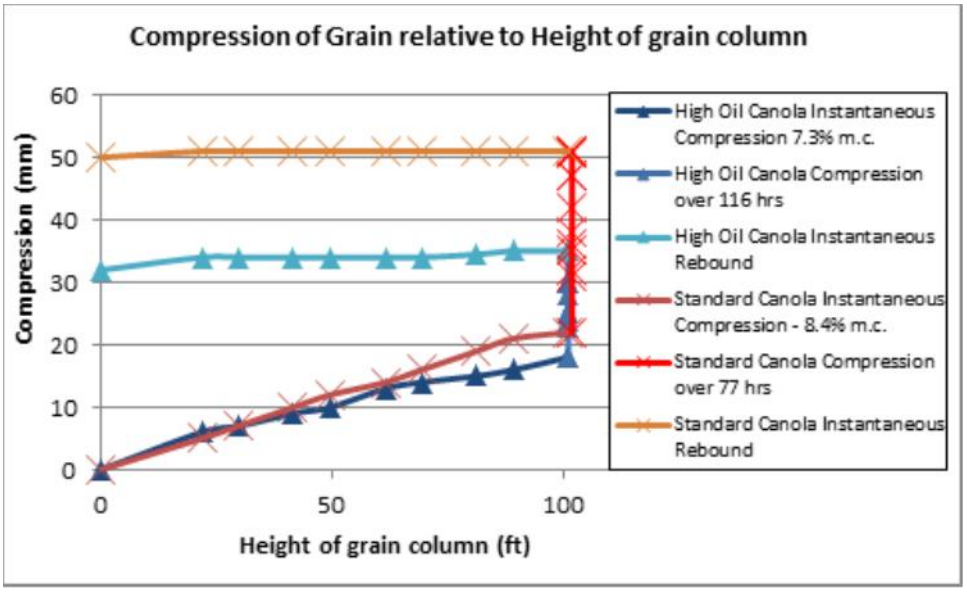

Figure 19. Measured rebound displacement with compression load removed.

4.2.3 Germination Results

Seed from each of the four samples (before and after testing) was sent to Discovery Seed Labs in Saskatoon, Saskatchewan, for germination analysis. **Table 5** displays the germination results and the relative change of each sample after compression testing.

Table 5. Compression testing germination results.

Sample		Germ (%)	Germ (%) Change	Abnormal (%)	Dead (%)
Standard Canola, 5.3% MC	Initial	72	-4	7	21
	Compressed	68		9	23
Standard Canola, 8.4% MC	Initial	72	4	8	20
	Compressed	76		5	19
High Oil Canola, 7.3% MC	Initial	91	-1	2	7
	Compressed	90		2	8
High Oil Canola, 10.5% MC	Initial	85	0	3	12
	Compressed	85		0	15

The standard canola samples showed both a positive and negative change in germination results after compression testing while the high oil content samples showed virtually no change. Interestingly though, the higher MC samples indicated greater compression displacements, but this did not result in lower germination.

Of note is that although the compression testing did not substantially affect the germination results, the samples did not have the high initial germination counts that might be expected. This may suggest that previous handling, seed age, or another factor may have already affected germination and/or seed structural integrity.

4.3 Additional Testing

The tests conducted with the three (small, medium, large) storage cells yielded multiple data streams.

4.3.1 Temperature Data

The logged temperatures in this testing provided information about both the temperature trend in the cells as well as the functional operation of the instrumentation and data collection equipment.

In general, the internal temperatures of the test cells followed the external ambient temperature changes. For example, in **Figure 20**, which maps the small cell temperatures, the diurnal oscillations of the ambient temperature are closely followed by the internal edge temperatures while the temperatures at the centre of the cell follow the long-term trend.

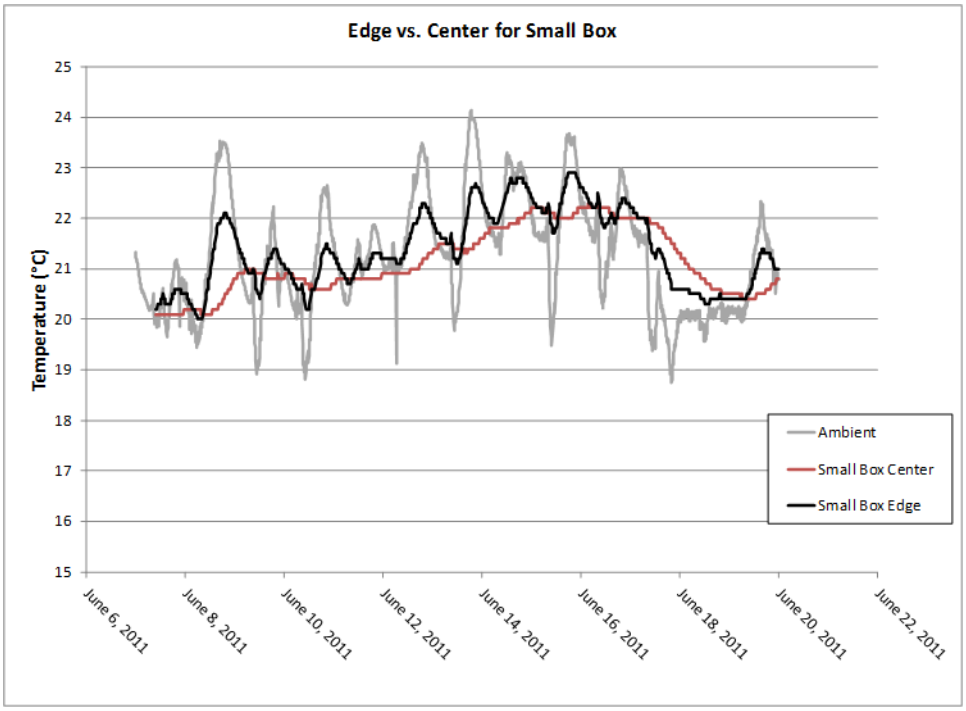

Figure 20. Small test cell edge and center temperature.

The trend of internal temperatures following those of the ambient temperature was consistent in all three test cells. The smaller cell reacted quicker and more abruptly, and the larger cells reacted slower with reduced amplitude, producing a much smoother temperature curve (**Figure 21**). All the test boxes were lined with equal thickness of insulation; therefore, theoretically the temperature at the edge of the boxes should respond similarly. However, the thermal mass of the canola seed itself appears to affect the temperature response. This lends support to the concept that a minimum size or critical mass may be required to accurately replicate real-world storage scenarios.

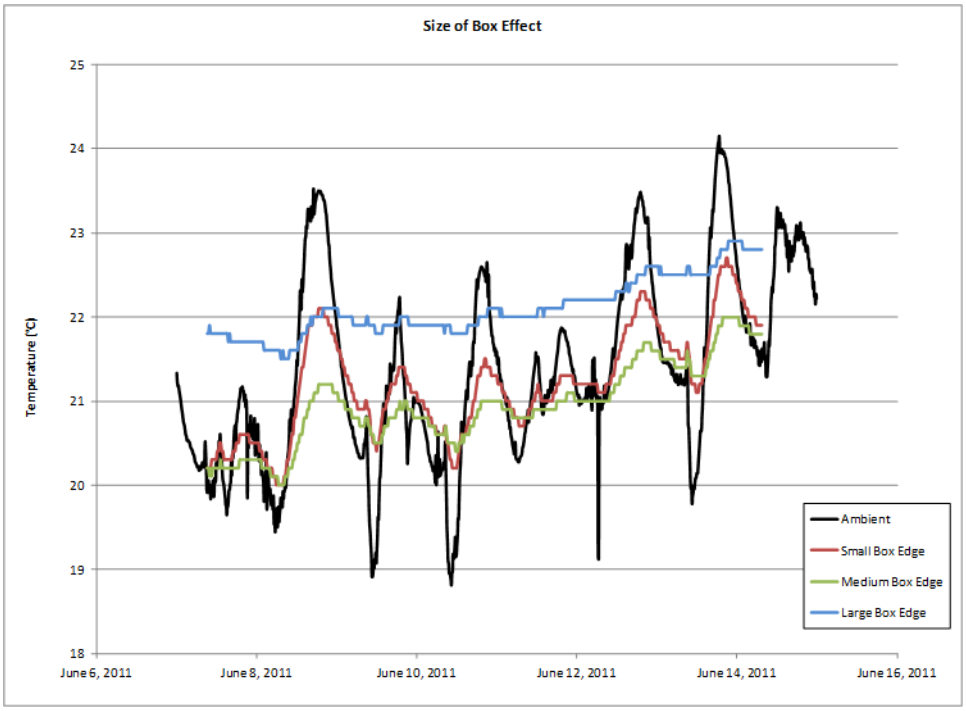

Figure 21. Temperature response for all three test cell sizes.

Most importantly, the temperature data did not reveal a temperature rise consistent with the conventional expectations for “heating” or spoiling canola. Despite what should have been ideal conditions for spoilage (approximately 12% MC, approximately 24°C (75.2°F), “heating” was not evident in any of the test cells regardless of size. Over the six week period, the temperature rose a few degrees but it directly correlated with a rise in ambient temperature. The fact that heating was not registered suggests that although consideration was given to sample size (critical mass and thermal insulation), these small-scale tests may not have properly duplicated real-world scenarios.

Testing with the lids removed to allow an aerobic environment produced identical results.

The temperature data also served to validate the data collection equipment used. In the previous small-scale storage tests, there had been concern regarding the accuracy of the system and potential issues with “noise” in the data. Through careful experimental design, including redundant sensors and multiple terminal connection configurations, specific thermocouple inputs were able to be compared. These comparisons revealed that despite manufacturer claims, unacceptable data “noise” was present with certain channels and terminal configurations. **Figure 22** illustrates the discrepancy and noise between multiple sensors measuring the same data. However, other channels and configurations provided acceptable accurate data.

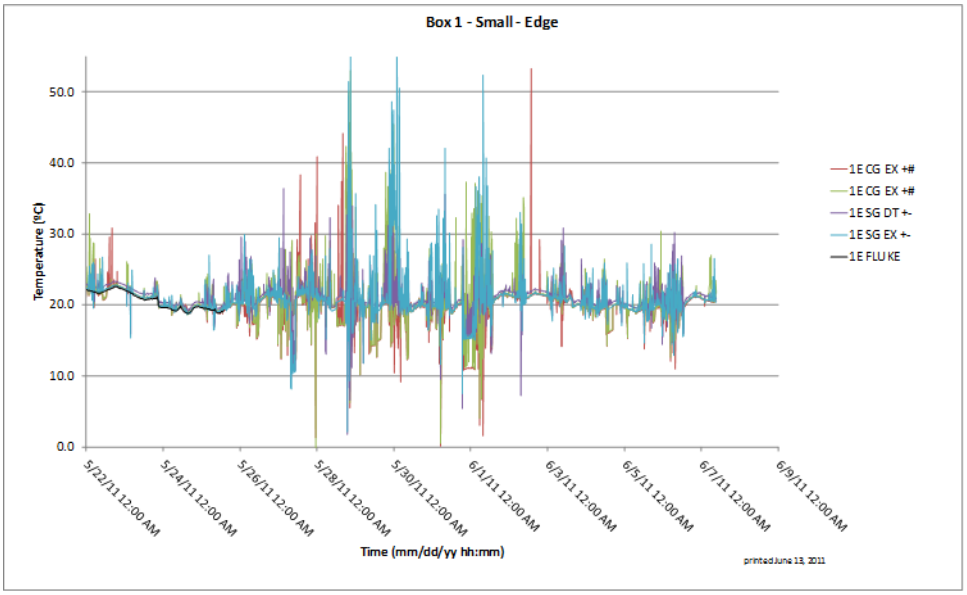

Figure 22. Variability of temperature measurement with redundant sensors.

Considering these results, it was concluded that although the data collection equipment had some problems, it was still functional. The instrumentation and data collection cannot be faulted for failure to register/record heating in the small-scale storage tests. It is most likely that heating did not occur.

4.3.2 Oil Quality Analysis

After approximately six weeks of storage in the three insulated test cells, samples were sent for the same oil quality analyses used in the small-scale storage testing. **Table 6** contains these results. Notably, none of the samples exceed the maximum thresholds discussed in **Section 4.1**. At these sizes, sample size does not appear to have had an impact on oil quality. From an industry oil quality perspective, these samples have not spoiled.

Table 6. Oil quality test results.

	Prior to Test		After Test				
	Initial	MC Conditioned	Small Cell	Medium Cell	Large Cell	Threshold Limit	Unit
Free Fatty Acids	0.23	0.28	0.29	0.29	0.32	1%	%
Acid Value	0.46	0.56	0.58	0.58	0.64	2	mg KOH/g
p-Anisidine Value	0.31	0.38	0.22	0.21	0.18	0.5	
Peroxide Value	0.70	0.20	0.30	0.40	0.30	2	meq/kg
TOTOX Value	1.71	0.78	0.82	1.01	0.78	4	

Because quality analysis was performed on samples before and after MC conditioning, the effect moisture addition has on seed oil quality is also evident from this table. FFA, AV, and p-AnV increased marginally after moisture conditioning, but the PV incurred a sizeable decrease.

Comparing the post-storage sample values with the moisture conditioned sample values it is evident that initial oxidation processes were occurring (indicated by the increased PV measurements). This is similar to the small-scale storage tests where oil quality degradation was underway but not to an extent that would jeopardize market acceptance.

4.3.3 Visual Assessment

At the completion of the tests, as the samples were collected for oil quality analysis, visual observations of the canola in each test cell were made. It was noted that the canola in the small cell exhibited no visual indications of mold or spoilage (**Figure 23**), but the canola in the medium and large cells had visible growth on the surface (**Figure 24** and **Figure 25**).

Figure 23. Small test cell.

Figure 24. Growth in medium test cell.

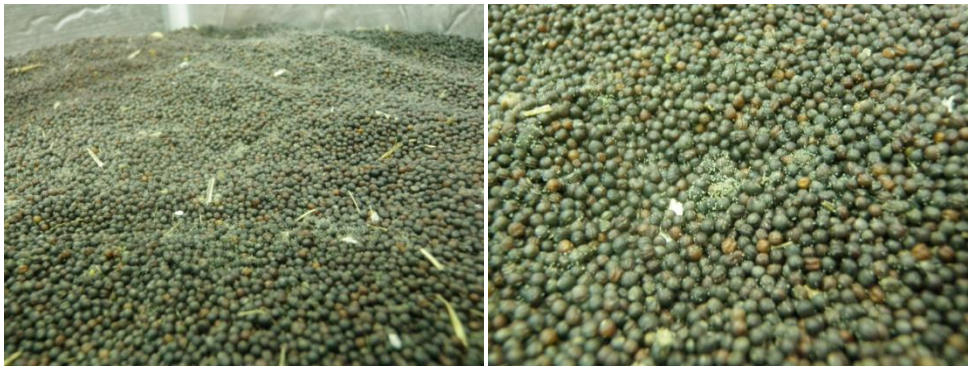

Figure 25. Growth in large test cell.

Test personnel reported that the two large test cells had a spoiled grain smell when the lid was removed.

It should be considered that “grain spoilage” may need to be better defined. It may be that the traditional definitions including mold/spore growth and fermented smell may not correlate directly with oil quality degradation.

5. Conclusions and Recommendations

5.1 Small-Scale Storage Testing

Based on visual observations of mold growth during the small-scale storage tests, it appeared that canola would spoil under warmer storage conditions. However, temperature monitoring of these samples did not indicate a coincident increase in temperature with this spoilage.

Depending on how the oil quality test results are analysed, these samples suggest that oxidation processes may have been active, but oil quality did not deteriorate beyond industry acceptable limits. Whether oxidation was progressing at a rate that would exceed defined limits under longer storage terms is not known.

The lack of firm trends makes it difficult to draw definite conclusions or storage recommendations. The AV data indicates high oil content canola appears to begin oxidation processes at lower storage temperatures and MC, but still within acceptable maximums.

Updating canola storage temperature and MC guidelines based on this data would be preliminary. The data is variable and does not support clear trends based on seed oil content and dockage on MC. Also, without temperature rise as a spoilage indicator, there is concern about the test procedures' and parameters' ability to replicate real world conditions.

5.2 Structural Integrity (Compression) Testing

The results of the structural integrity testing indicate that compression forces experienced by canola seed in tall bins (up to approximately 100 ft tall) are not detrimental to canola. The data collected suggests that compression is not extensive enough to corrupt the structure of canola seeds to a degree that would negatively affect germination.

Some deformation of seed shape may be experienced, especially with increased MC, but seed coat rupture or oil exudation do not appear to be common. Higher seed MC seems to correlate with increased compression displacement (compaction), but decreased germination was not evident.

Effect of seed oil content on structural integrity was not conclusive, but no indications that higher oil content is related to increased seed damage (germination, rupture, and oil exudation) exist Storage of high oil content canola in large, tall bins does not appear to

be a risk to seed integrity, based on these tests. Different MC, temperatures, seed varieties, etc., may produce different results.

5.3 Additional Testing

The additional tests performed validated the data collection equipment used in the small-scale storage testing and determined that although some channel/terminal configurations yielded “noisy” data, the data was not corrupted so as to disguise or conceal a heating trend. Despite some erroneous and extreme datum points recorded with these faulty configurations, the basic temperature trend was still recognizable. The corrupt data may have relegated the micro-scale detail of the temperature traces ineffective, but the macro-scale trend was still accurate. Also, many of the sensor channels were confirmed to have been functioning properly. This information confirms that heating in the samples of the small-scale storage testing did not go undetected.

These additional tests also provided information to suggest that the size of grain samples used for testing is important. As shown by the medium and large test cell internal temperatures, the larger test samples appeared to moderate the effect from external temperature by effectively insulating the core of the sample. However, even the center temperature of the small sample reacted more modestly relative to the edge-of-box temperature. As evident when comparing the edge temperatures to centre temperatures for all three test cells, the edge temperature more closely followed ambient temperature movements. Even the smallest sample size insulated the central core of the sample against external temperature influence delaying and moderating temperature response.

It is interesting to note that although all three test cells had equal insulation lining the box, the temperature response at the edge of the three cells was not equal. Again, the larger sample temperature was less reactive to the ambient temperature influence. This suggests there may be a critical mass required to replicate an in-bin scenario. It also indicates that substitution of commercial insulation for an insulating layer of canola may not have been representative. Despite an equivalent calculated thermal insulation value, the commercial Styrofoam insulation did not appear to be as effective as a mass of canola.

The primary intent of this additional testing was to create test conditions under which canola would heat and spoil to determine if the temperature rise could be measured. Unfortunately, despite providing conditions that should induce heating, no heating trend was recorded. Canola seed with a grain moisture of approximately 12% and storage temperatures of about 24°C (75.2°F) could be expected to heat and spoil, but this was not realized. Oil quality testing also determined oil quality to be within acceptable limits. It may be concluded from these tests that other unaccounted for factors such as seed

age, previous handling, storage conditions, or chemical or biological activity, have not been replicated in these small-scale tests.

5.4 Further Testing

This testing has shown that canola may **begin** spoilage at MCs as low as 7% moisture and at temperatures as cool as -5^{o}C (23°F). Since the current recommendation is 8% MC, this indicates that more research is required to understand and develop new safe storage recommendations for canola to define specific “safe storage” conditions.

Additionally, testing suggested that initial deterioration of canola oil quality may not be signalled by temperature increase. This is basically the only indication for canola spoilage used on farm. If temperature cannot be relied upon as a quality test, it will have major ramifications for producers.

Alternatively, it could be the test parameters were not properly devised to represent reality. The effect of critical sample size and proper sample insulation are not yet fully understood. In either case, this bears further investigation.

Spoilage was not indicated by temperature measurements from each thermocouple channel. This is thought to be due to the small sample size and lack of insulation around each test sample. Further testing should be done to determine whether larger, better insulated test samples would show a rise in temperature before the heat produced during spoilage is lost to the environment inside the chamber.

Because this is such a broad issue, there are many variables to consider including: time, temperature, MC, oil content, AV, FFA, PV, ANV, sample size, head space within the sample pails, sample preparation and handling before testing begins, seed age, and gas sample analysis. In order to simplify the problem, each of these variable needs to be looked at one at a time to establish its role in the degradation of oil quality during long term canola storage.

For example, the sole effect of time on oil quality should be investigated. Oil quality may simply decline with time, even in the best possible conditions. This could be studied by keeping one sample in a thin layer at room temperature and one sample outside for 12 months to 24 months and periodically testing for oil quality. This would show the quality degradation that happens exclusively due to time passage.

To isolate the temperature variable, a base sample could be prepared to a high moisture content and stored in multiple sample sizes with various levels of insulation. The sample temperatures could be monitored while they are kept in one of the environmental

chambers. This would determine the proper sample configuration so that any heat generated could be detected by a thermocouple within the sample.

The testing already completed has provided good insight into the degradation of oil quality during storage but it has also exposed more questions. This is a complicated issue that is very important to producers and large scale buyers of canola in Saskatchewan. PAMI is eager to look deeper into this matter to develop new guidelines to ensure Saskatchewan stays at the forefront of canola development.

Since temperature seems to be the most practical means for early spoilage detection, it is important that further testing be conducted. Future testing should include fewer, larger test samples. Each sample must be well insulated to allow heat produced during spoilage to raise the core temperature of the sample without being adversely affected by the surrounding environment. If this assumption is correct, this may provide results that would indicate the length of time before spoilage occurs at different moistures and temperatures. Testing should also be conducted to further define if heating occurs early on with spoilage or only once spoilage is advanced. Some slow spoilage, such as mold growth, may not involve much heat generation. While heating can occur from fermentation, oxidation of oils, germination of grain, insect contamination, or bacterial action (aerobic or anaerobic), it may not be measureable during the early stages of spoilage. Other possibilities for early spoilage detection such as gas sampling within the grain may possibly work but have not been investigated.

6. Bibliography

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Shahidi, F. (2005). Lipid oxidation: measurement methods. In F. Shahidi (Ed.), *Bailey's Industrial Oil and Fat Products, Volume 1* (Sixth ed., Vol. One, pp. 357-369). John Wiley and Sons, Inc.

Other Information Sources:

Lisa Campbell, Canola Council of Canada, personal communication, January 6, 2012

Nick Boguski, POS Pilot Plant, personal communication, January 6, 2012

7. Other

7.1 Personnel Costs

The following table shows personnel costs for this report period.

Postdoctoral Fellows	**Hours**	**Amount**
Hill, L.	6.5	$ 962.00
Leduc, P.	8.0	1,173.50
Total	**14.5**	**$2,135.50**
Technical Assistants	**Hours**	**Amount**
Bendel, J.	2.0	$ 200.00
Doepker, S.	2.0	164.00
Fleischhacker, J.	26.0	1,196.00
Gerspacher, J.	3.5	157.50
Gregg, N.	91.5	11,156.00
Haeusler, M.	2.0	160.00
Kimmen, M.	6.0	271.00
Lepage, D.	0.5	22.50
Lung, B.	2.0	240.00
McDonald, J.	52.0	5,297.00
Perlett, D.	14.0	1,568.00
Rhodes, P.	78.0	8,580.00
Total	**279.5**	**$29,012.00**

7.2 Expense Statement

The following table shows salaries and benefits and funds contributed.

	Original Total Budget	Approved Total Budget	Total Actual
Salaries and Benefits			
Postdoctoral Fellows	$ 7,378.00	$ 2,975.00	$ 2,975.00
Technical Assistants	52,075.00	68,068.00	68,068.00
Material and Supplies	17,900.00	7,570.00	7,570.00
Field Work	1,390.00	130.00	130.00
Total Funds ADF	**$ 78,743.00**	**$ 78,743.00**	**$ 78,743.00**
Funds Contributed			
Applicant - Cash	$ 10,282.00	$ 24,385.86	$ 14,889.28
Sask Canola Development Commission	41,000.00	41,000.00	41,000.00
Other			
Total Funds Contributed	**$ 51,282.00**	**$ 65,385.86**	**$ 55,889.28**
Total Funds	**$130,025.00**	**$144,128.86**	**$134,632.28**

Appendix I

Results and Analysis

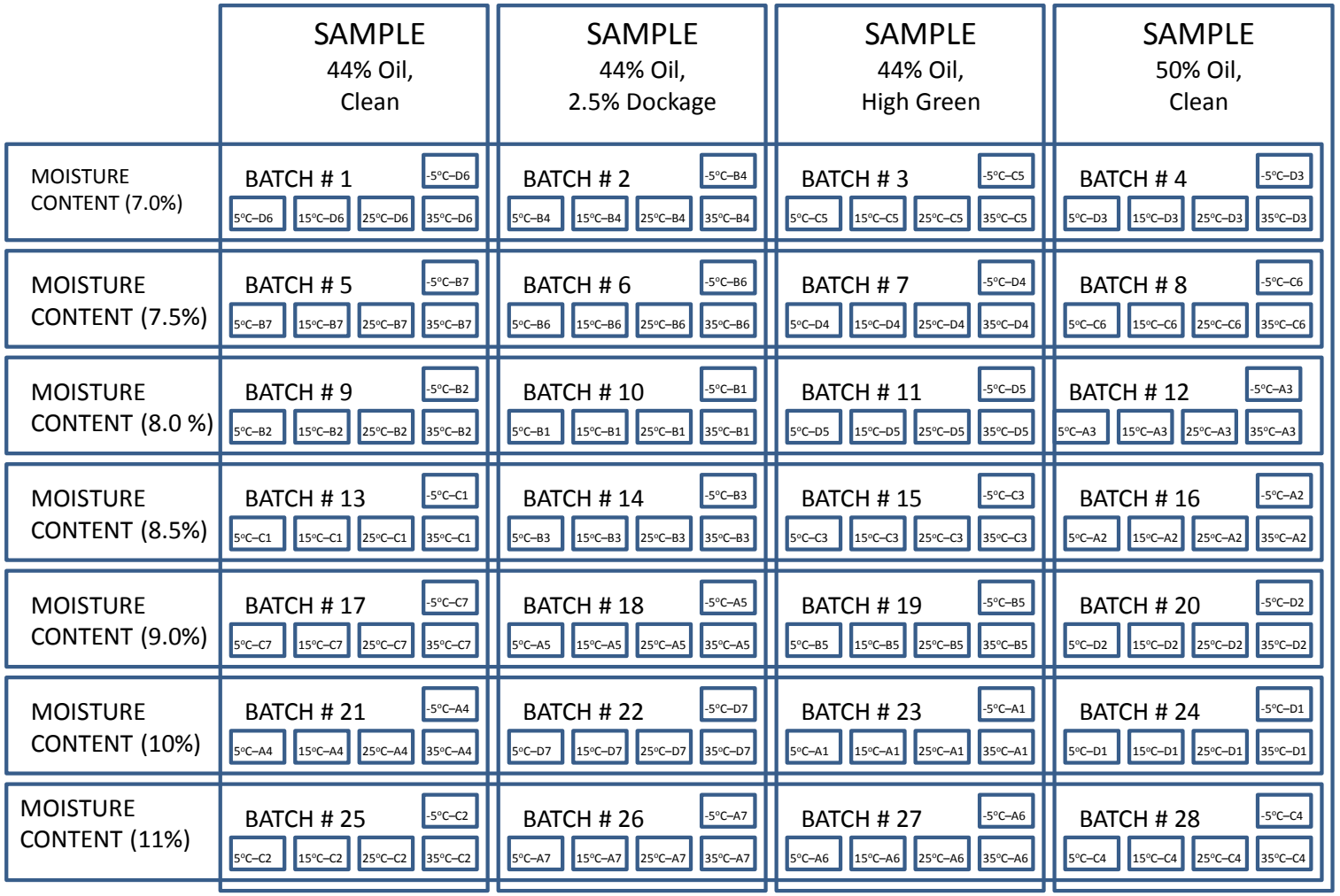

Figure A1. Pail map.

A3909-T1 PAIL MAP

BY LOCATION

LOCATION	SAMPLE	MOISTURE (%)
A1	Green	10
A2	Oil	8.5
A3	Oil	8
A4	Clean	10
A5	Dockage	9
A6	Green	11
A7	Dockage	11
B1	Dockage	8
B2	Clean	8
B3	Dockage	8.5
B4	Dockage	7
B5	Green	9
B6	Dockage	7.5
B7	Clean	7.5
C1	Clean	8.5
C2	Clean	11
C3	Green	8.5
C4	Oil	11
C5	Green	7
C6	Oil	7.5
C7	Clean	9
D1	Oil	10
D2	Oil	9
D3	Oil	7
D4	Green	7.5
D5	Green	8
D6	Clean	7
D7	Dockage	10

BY SAMPLE

SAMPLE	MOISTURE (%)	LOCATION
Clean	7	D6
Clean	7.5	B7
Clean	8	B2
Clean	8.5	C1
Clean	9	C7
Clean	10	A4
Clean	11	C2
Dockage	7	B4
Dockage	7.5	B6
Dockage	8	B1
Dockage	8.5	B3
Dockage	9	A5
Dockage	10	D7
Dockage	11	A7
Green	7	C5
Green	7.5	D4
Green	8	D5
Green	8.5	C3
Green	9	B5
Green	10	A1
Green	11	A6
Oil	7	D3
Oil	7.5	C6
Oil	8	A3
Oil	8.5	A2
Oil	9	D2
Oil	10	D1
Oil	11	C4

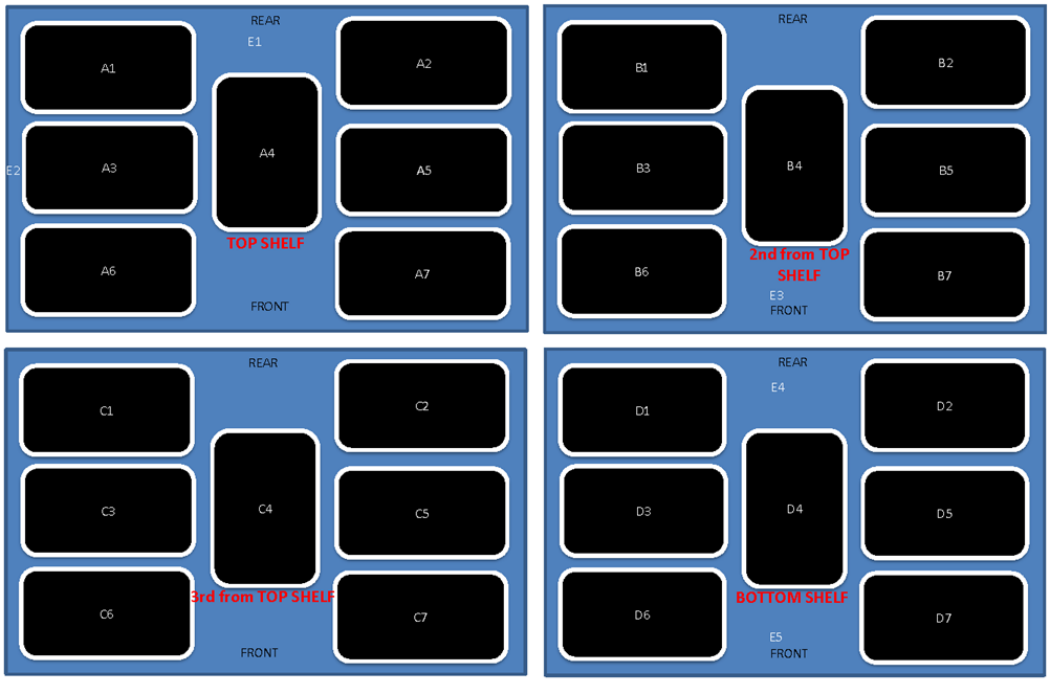

Figure A2. Sample locations with a chamber.

Table A1. Sample moisture contents.

Temperature (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
7	7.1	6.8	7.1	6.9	7.0	7.1	6.8	7.1	6.9	7.0	7.1	6.8	7.1	6.9	7.0	7.1	6.8	7.1	6.9	7.0	7.1	6.8	7.1	6.9	7.0
7.5	7.6	7.7	7.7	8.3	7.8	7.6	7.7	7.7	8.3	7.8	7.6	7.7	7.7	8.3	7.8	7.6	7.7	7.7	8.3	7.8	7.6	7.7	7.7	8.3	7.8
8	8.4	8.4	8.2	8.9	8.5	8.4	8.4	8.2	8.9	8.5	8.4	8.4	8.2	8.9	8.5	8.4	8.4	8.2	8.9	8.5	8.4	8.4	8.2	8.9	8.5
8.5	8.7	8.9	8.8	9.3	8.9	8.7	8.9	8.8	9.3	8.9	8.7	8.9	8.8	9.3	8.9	8.7	8.9	8.8	9.3	8.9	8.7	8.9	8.8	9.3	8.9
9	9.3	9.4	9.1	9.8	9.4	9.3	9.4	9.1	9.8	9.4	9.3	9.4	9.1	9.8	9.4	9.3	9.4	9.1	9.8	9.4	9.3	9.4	9.1	9.8	9.4
10	10.2	10.3	10.1	10.3	10.2	10.2	10.3	10.1	10.3	10.2	10.2	10.3	10.1	10.3	10.2	10.2	10.3	10.1	10.3	10.2	10.2	10.3	10.1	10.3	10.2
11	11.2	11.2	11.1	11.1	11.1	11.2	11.2	11.1	11.1	11.1	11.2	11.2	11.1	11.1	11.1	11.2	11.2	11.1	11.1	11.1	11.2	11.2	11.1	11.1	11.1
Average	8.9	8.9	8.8	9.2	9.0	8.9	8.9	8.8	9.2	9.0	8.9	8.9	8.8	9.2	9.0	8.9	8.9	8.8	9.2	9.0	8.9	8.9	8.8	9.2	9.0

Table A2. Percent change in acid value (AV) test results.

Temperature (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
7	-35%	-27%	-15%	46%	-8%	25%	-20%	-16%	17%	1%	-2%	-30%	-1%	144%	28%	9%	-18%	1%	444%	109%	101%	37%	32%	110%	70%
7.5	-24%	-28%	0%	4%	-12%	-22%	-28%	-17%	8%	-15%	9%	-15%	-1%	17%	2%	7%	-5%	2%	88%	23%	123%	94%	56%	169%	110%
8	-17%	-18%	-15%	-4%	-14%	-13%	-35%	-3%	29%	-6%	-9%	-3%	-3%	88%	18%	21%	18%	11%	79%	32%	147%	70%	86%	210%	128%
8.5	-24%	-15%	-73%	8%	-26%	-24%	-20%	-13%	4%	-13%	0%	-7%	-7%	54%	10%	60%	29%	23%	104%	54%	147%	86%	70%	160%	116%
9	-4%	-13%	-16%	8%	-6%	-13%	-8%	5%	8%	-2%	47%	-8%	1%	54%	23%	60%	83%	22%	177%	85%	92%	94%	84%	302%	143%
10	-13%	-25%	41%	4%	2%	-13%	-10%	-13%	88%	13%	36%	44%	5%	63%	37%	92%	62%	41%	165%	90%	114%	202%	84%	215%	154%
11	-17%	0%	-10%	21%	-2%	10%	-3%	-2%	104%	27%	62%	21%	9%	127%	55%	92%	57%	46%	181%	94%	365%	170%	107%	406%	262%
Average	-19%	-18%	-13%	13%	-9%	-7%	-18%	-8%	37%	1%	20%	0%	0%	78%	25%	49%	32%	21%	177%	70%	156%	108%	74%	225%	141%

Table A3. Percent change in peroxide value (PV) test results.

%Change in Peroxide Value																									
Temperature (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
7	-5%	29%	-26%	30%	7%	25%	-17%	-28%	20%	0%	460%	-34%	-10%	150%	141%	70%	14%	-17%	220%	72%	-10%	-14%	50%	250%	69%
7.5	-45%	-51%	9%	-15%	-26%	-50%	-14%	-22%	-50%	-34%	10%	-37%	14%	-10%	-6%	35%	-9%	-4%	40%	15%	15%	-51%	-59%	95%	0%
8	250%	129%	-10%	-35%	83%	-45%	-69%	-15%	-50%	-45%	90%	-69%	-23%	0%	0%	-5%	-20%	-23%	35%	-3%	375%	157%	-71%	175%	159%
8.5	-30%	129%	-14%	-5%	20%	-25%	-69%	-34%	-35%	-41%	10%	-31%	-22%	75%	8%	5%	-40%	-64%	-50%	-37%	75%	14%	69%	-50%	27%
9	195%	14%	-17%	-25%	42%	5%	-69%	-11%	-50%	-31%	65%	-26%	-14%	10%	9%	25%	-51%	-32%	-50%	-27%	500%	-43%	-61%	-50%	87%
10	100%	-46%	58%	150%	66%	-20%	-37%	-29%	-5%	-23%	110%	-34%	-6%	0%	18%	125%	-34%	-39%	-40%	3%	-50%	-40%	-67%	100%	-14%
11	-5%	-14%	-14%	0%	-8%	-30%	-40%	21%	0%	-12%	20%	-43%	-14%	-40%	-19%	75%	-20%	-51%	-25%	-5%	-50%	143%	-39%	140%	48%
Average	66%	27%	-2%	14%	26%	-20%	-45%	-17%	-24%	-27%	109%	-39%	-11%	26%	21%	47%	-23%	-33%	19%	2%	122%	24%	-25%	94%	54%

Table A4. Percent change in free fatty acid (FFA) test results.

%Change in FFA																									
Temperature (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
7	-35%	-27%	-15%	46%	-8%	26%	-21%	-16%	17%	1%	-2%	-31%	-1%	146%	28%	9%	-18%	1%	446%	109%	102%	37%	32%	113%	71%
7.5	-24%	-29%	0%	4%	-12%	-22%	-29%	-17%	8%	-15%	9%	-15%	-1%	17%	2%	7%	-5%	2%	88%	23%	124%	94%	56%	171%	111%
8	-17%	-18%	-15%	-4%	-14%	-13%	-35%	-3%	29%	-6%	-9%	-3%	-3%	88%	18%	22%	18%	11%	79%	33%	148%	69%	86%	213%	129%
8.5	-24%	-15%	-16%	8%	-12%	-24%	-21%	-13%	4%	-13%	0%	-6%	-7%	54%	10%	61%	29%	23%	104%	54%	148%	85%	69%	163%	116%
9	-4%	-13%	-16%	8%	-6%	-13%	-8%	5%	8%	-2%	48%	-8%	1%	54%	24%	61%	82%	22%	179%	86%	93%	94%	84%	304%	144%
10	-13%	-26%	41%	4%	2%	-13%	-10%	-13%	88%	13%	37%	44%	5%	63%	37%	93%	61%	41%	167%	91%	115%	202%	84%	217%	154%
11	-17%	0%	-10%	21%	-2%	11%	-3%	-2%	104%	27%	63%	21%	9%	129%	56%	93%	56%	45%	183%	95%	367%	169%	107%	408%	263%
Average	-19%	-18%	-4%	13%	-7%	-7%	-18%	-8%	37%	1%	21%	0%	0%	79%	25%	49%	32%	21%	178%	70%	157%	107%	74%	227%	141%

Table A5. Percent change in p-anisidine value (p-AnV) test results.

%Change in -p Anisidine																									
Temperature (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
7	25%	0%	17%	218%	65%	30%	7%	3%	73%	28%	235%	-19%	40%	427%	171%	-5%	-15%	2%	764%	187%	110%	37%	2%	145%	74%
7.5	-10%	-22%	38%	73%	20%	-20%	-7%	-1%	-9%	-9%	-10%	-22%	52%	82%	25%	10%	22%	-19%	82%	24%	125%	89%	29%	127%	93%
8	5%	4%	8%	82%	25%	5%	-41%	11%	0%	-6%	35%	22%	21%	200%	70%	-5%	-11%	-13%	109%	20%	140%	96%	71%	191%	124%
8.5	-15%	4%	13%	136%	35%	-15%	-7%	-1%	91%	17%	15%	-22%	4%	109%	27%	50%	11%	-2%	127%	47%	205%	133%	40%	245%	156%
9	25%	15%	-6%	91%	31%	-30%	-26%		118%	21%	100%	22%	3%	82%	52%	10%	30%	-12%	64%	23%	100%	107%	66%	200%	118%
10	10%	-30%	61%	164%	51%	15%	-26%	2%	91%	21%	50%	33%	47%	27%	39%	60%	-4%	0%	45%	25%	85%	193%	37%	118%	108%
11	-45%	-4%	6%	36%	-2%	-20%	-19%	1%	118%	20%	20%	4%	9%	45%	20%	20%	70%	-1%	45%	34%	150%	89%	30%	136%	101%
Average	-1%	-5%	20%	114%	32%	-5%	-17%	3%	69%	13%	64%	3%	25%	139%	58%	20%	15%	-7%	177%	51%	131%	106%	39%	166%	111%

Table A6. Free fatty acid (FFA) test results.

November FFA Values - Cells Highlighted if >1%																									
Temp. (°C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
Initial	0.46	0.62	0.88	0.24		0.46	0.62	0.88	0.24		0.46	0.62	0.88	0.24		0.46	0.62	0.88	0.24		0.46	0.62	0.88	0.24	
7	0.30	0.45	0.75	0.35	0.46	0.58	0.49	0.74	0.28	0.52	0.45	0.43	0.87	0.59	0.59	0.50	0.51	0.89	1.31	0.80	0.93	0.85	1.16	0.51	0.86
7.5	0.35	0.44	0.88	0.25	0.48	0.36	0.44	0.73	0.26	0.45	0.50	0.53	0.87	0.28	0.55	0.49	0.59	0.90	0.45	0.61	1.03	1.20	1.37	0.65	1.06
8	0.38	0.51	0.75	0.23	0.47	0.40	0.40	0.85	0.31	0.49	0.42	0.60	0.85	0.45	0.58	0.56	0.73	0.98	0.43	0.68	1.14	1.05	1.64	0.75	1.15
8.5	0.35	0.53	0.74	0.26	0.47	0.35	0.49	0.77	0.25	0.47	0.46	0.58	0.82	0.37	0.56	0.74	0.80	1.08	0.49	0.78	1.14	1.15	1.49	0.63	1.10
9	0.44	0.54	0.74	0.26	0.50	0.40	0.57	0.92	0.26	0.54	0.68	0.57	0.89	0.37	0.63	0.74	1.13	1.07	0.67	0.90	0.89	1.20	1.62	0.97	1.17
10	0.40	0.46	1.24	0.25	0.59	0.40	0.56	0.77	0.45	0.55	0.63	0.89	0.92	0.39	0.71	0.89	1.00	1.24	0.64	0.94	0.99	1.87	1.62	0.76	1.31
11	0.38	0.62	0.79	0.29	0.52	0.51	0.60	0.86	0.49	0.62	0.75	0.75	0.96	0.55	0.75	0.89	0.97	1.28	0.68	0.96	2.15	1.67	1.82	1.22	1.72
Average	0.37	0.51	0.84	0.27	0.50	0.43	0.51	0.81	0.33	0.52	0.56	0.62	0.88	0.43	0.62	0.69	0.82	1.06	0.67	0.81	1.18	1.28	1.53	0.78	1.20

Table A7. Peroxide value (PV) test results.

November Peroxide Values - Cells Highlighted if > 2																									
Temp. (˚C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
Initial	0.20	0.35	1.80	0.20		0.20	0.35	1.80	0.20		0.20	0.35	1.80	0.20		0.20	0.35	1.80	0.20		0.20	0.35	1.80	0.20	
7	0.19	0.45	1.33	0.26	0.56	0.25	0.29	1.30	0.24	0.52	1.12	0.23	1.62	0.50	0.87	0.34	0.40	1.50	0.64	0.72	0.18	0.30	2.70	0.70	0.97
7.5	0.11	0.17	1.96	0.17	0.60	0.10	0.30	1.41	0.10	0.48	0.22	0.22	2.06	0.18	0.67	0.27	0.32	1.72	0.28	0.65	0.23	0.17	0.73	0.39	0.38
8	0.70	0.80	1.62	0.13	0.81	0.11	0.11	1.53	0.10	0.46	0.38	0.11	1.39	0.20	0.52	0.19	0.28	1.38	0.27	0.53	0.95	0.90	0.53	0.55	0.73
8.5	0.14	0.80	1.55	0.19	0.67	0.15	0.11	1.18	0.13	0.39	0.22	0.24	1.40	0.35	0.55	0.21	0.21	0.64	0.10	0.29	0.35	0.40	3.04	0.10	0.97
9	0.59	0.40	1.50	0.15	0.66	0.21	0.11	1.60	0.10	0.51	0.33	0.26	1.55	0.22	0.59	0.25	0.17	1.22	0.10	0.44	1.20	0.20	0.70	0.10	0.55
10	0.40	0.19	2.84	0.50	0.98	0.16	0.22	1.28	0.19	0.46	0.42	0.23	1.70	0.20	0.64	0.45	0.23	1.09	0.12	0.47	0.10	0.21	0.60	0.40	0.33
11	0.19	0.30	1.55	0.20	0.56	0.14	0.21	2.17	0.20	0.68	0.24	0.20	1.55	0.12	0.53	0.35	0.28	0.89	0.15	0.42	0.10	0.85	1.10	0.48	0.63
Average	0.33	0.44	1.76	0.23	0.69	0.16	0.19	1.50	0.15	0.50	0.42	0.21	1.61	0.25	0.62	0.29	0.27	1.21	0.24	0.50	0.44	0.43	1.34	0.39	0.65

Table A8. P-anisidine value (p-AnV) test results.

November -p Anisidine Values - Cells Highlighted if > 0.5																									
Temp. (˚C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
Initial	0.20	0.27	0.89	0.11		0.20	0.27	0.89	0.11		0.20	0.27	0.89	0.11		0.20	0.27	0.89	0.11		0.20	0.27	0.89	0.11	
7	0.25	0.27	1.04	0.35	0.48	0.26	0.29	0.92	0.19	0.42	0.67	0.22	1.25	0.58	0.68	0.19	0.23	0.91	0.95	0.57	0.42	0.37	0.91	0.27	0.49
7.5	0.18	0.21	1.23	0.19	0.45	0.16	0.25	0.88	0.10	0.35	0.18	0.21	1.35	0.20	0.49	0.22	0.33	0.72	0.20	0.37	0.45	0.51	1.15	0.25	0.59
8	0.21	0.28	0.96	0.20	0.41	0.21	0.16	0.99	0.11	0.37	0.27	0.33	1.08	0.33	0.50	0.19	0.24	0.77	0.23	0.36	0.48	0.53	1.52	0.32	0.71
8.5	0.17	0.28	1.01	0.26	0.43	0.17	0.25	0.88	0.21	0.38	0.23	0.21	0.93	0.23	0.40	0.30	0.30	0.87	0.25	0.43	0.61	0.63	1.25	0.38	0.72
9	0.25	0.31	0.84	0.21	0.40	0.14	0.20		0.24	0.19	0.40	0.33	0.92	0.20	0.46	0.22	0.35	0.78	0.18	0.38	0.40	0.56	1.48	0.33	0.69
10	0.22	0.19	1.43	0.29	0.53	0.23	0.20	0.91	0.21	0.39	0.30	0.36	1.31	0.14	0.53	0.32	0.26	0.89	0.16	0.41	0.37	0.79	1.22	0.24	0.66
11	0.11	0.26	0.94	0.15	0.37	0.16	0.22	0.90	0.24	0.38	0.24	0.28	0.97	0.16	0.41	0.24	0.46	0.88	0.16	0.44	0.50	0.51	1.16	0.26	0.61
Average	0.20	0.26	1.06	0.24	0.44	0.19	0.22	0.91	0.19	0.35	0.33	0.28	1.12	0.26	0.50	0.24	0.31	0.83	0.30	0.42	0.46	0.56	1.24	0.29	0.64

Table A9. TOTOX value test results.

NOVEMBER TOTOX = (2*PV + p-AnV) - Cells Highlighted if > 4																									
Temp. (˚C)	-5					5					15					25					35				
Target Moisture Content (%)	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average	CLEAN	DOCKAGE	GREEN	OIL	Average
Initial	0.60	0.97	4.49	0.51		0.60	0.97	4.49	0.51		0.60	0.97	4.49	0.51		0.60	0.97	4.49	0.51		0.60	0.97	4.49	0.51	
7	0.63	1.17	3.70	0.87	1.59	0.76	0.87	3.52	0.67	1.46	2.91	0.68	4.49	1.58	2.42	0.87	1.03	3.91	2.23	2.01	0.78	0.97	6.31	1.67	2.43
7.5	0.40	0.55	5.15	0.53	1.66	0.36	0.85	3.70	0.30	1.30	0.62	0.65	5.47	0.56	1.83	0.76	0.97	4.16	0.76	1.66	0.91	0.85	2.61	1.03	1.35
8	1.61	1.88	4.20	0.46	2.04	0.43	0.38	4.05	0.31	1.29	1.03	0.55	3.86	0.73	1.54	0.57	0.80	3.53	0.77	1.42	2.38	2.33	2.58	1.42	2.18
8.5	0.45	1.88	4.11	0.64	1.77	0.47	0.47	3.24	0.47	1.16	0.67	0.69	3.73	0.93	1.51	0.72	0.72	2.15	0.45	1.01	1.31	1.43	7.33	0.58	2.66
9	1.43	1.11	3.84	0.51	1.72	0.56	0.42	3.20	0.44	1.20	1.06	0.85	4.02	0.64	1.64	0.72	0.69	3.22	0.38	1.25	2.80	0.96	2.88	0.53	1.79
10	1.02	0.57	7.11	1.29	2.50	0.55	0.64	3.47	0.59	1.31	1.14	0.82	4.71	0.54	1.80	1.22	0.72	3.07	0.40	1.35	0.57	1.21	2.42	1.04	1.31
11	0.49	0.86	4.04	0.55	1.49	0.44	0.64	5.24	0.64	1.74	0.72	0.68	4.07	0.40	1.47	0.94	1.02	2.66	0.46	1.27	0.70	2.21	3.36	1.22	1.87
Average	0.86	1.15	4.59	0.69	1.82	0.51	0.61	3.90	0.49	1.35	1.16	0.70	4.34	0.77	1.74	0.83	0.85	3.24	0.78	1.43	1.35	1.42	3.93	1.07	1.94