# Canadian Canola Quality Parameters: Comparison of Results from Harvest and Export Surveys

## Douglas R. DeCLERCQ and James K. DAUN

Grain Research Laboratory Canadian Grain Commission 1404-303 Main Street Winnipeg, MB Canada R3C 3G8 www.grainscanada.gc.ca

### Abstract

Canola quality parameters (oil, protein, chlorophyll, glucosinolates, free fatty acids, erucic acid, linolenic acid, and iodine value) from the 1980 to 2004 Grain Research Laboratory harvest surveys and export monitoring programs of western Canadian canola were compared. In the fall of each year, surveys of newly harvested canola provide stakeholders with quality data on the new crop. Throughout the year vessel loadings are sampled and tested for the same parameters and provide information to exporters on the quality of the canola moving out of the country. Over the 25-year period examined harvest surveys were found to be a reliable predictor of the quality of exports for the ensuing shipping season. Production carry over in certain years and the inclusion of dockage in commercial export shipments were likely responsible for differences between harvest survey and export data for some of the parameters.

#### Introduction

The quality of Canadian canola depends on a variety registration system that encourages the development of cultivars with quality factors demanded by the end-user. Canola quality factors include levels of glucosinolates and erucic acid within the canola specification and acceptable levels of oil content, protein content free fatty acids and chlorophyll in the seed. Variety development is key in maintaining levels of glucosinolates, oil, protein and erucic acid while levels of free fatty acids and chlorophyll are highly dependent on growing conditions.

The ability to export canola of consistent quality is maintained through the grain quality assurance system in which canola shipped from a wide areas is bulked to make up shipments. During the gathering and transportation of canola from the farm to the port, the canola is subjected to grading that segregates the seed into parcels with similar quality according to specifications under the Canada Grain Act. One of the roles of the Canadian Grain Commission (CGC)'s Grain Research Laboratory is to provide technical and analytical information on the quality and composition of canola as it moves through the grain handling system. Some of this information is provided through annual harvest surveys (Canadian Grain Commission, 2005a) (DeClercq, 2004) and from ongoing export monitoring programs (Canadian Grain Commission, 2005b). Harvest survey data would expected to predict the quality of the subsequent export shipments. Factors such as carry over, blending, differences in cleaning and competition for seed between the domestic crushing industry and the export industry may result in differences in results for the two surveys. This study was undertaken to compare data generated by these two different monitoring programs in order to highlight differences and similarities in the two sets of data.

#### Methods

**Samples**: Data used in this study were derived from CGC harvest surveys for western Canadian canola and canola export monitoring programs conducted for the period 1980 to 2004. The analytical data presented are for top grade, No. 1 Canada Canola, samples although samples from other grades also were collected and tested. Oil, protein, and glucosinolate values are expressed on a constant 8.5% moisture basis in order to permit annual comparisons. This value is a little higher than the average loading moisture content for the period studied (7.7% with a range from 5.6% to 10.0%). Annual mean values from annual harvest surveys were weighted by production from each provincial crop district while annual mean values for exports were tonnage-weighted means of individual samples. The harvest survey means were based on about 1500 samples per year while annual export means were based on about 150 samples per year.

Canola harvest survey samples were collected from producers, crushing plants and grain handling offices across western Canada. The samples were cleaned to remove foreign matter prior to testing as these field samples can contain from 5% to 20% admixture in the form of dockage, debris and soil. Individual harvest survey samples were analyzed for oil, protein, chlorophyll and total glucosinolates using a NIRS 6500 scanning near-infrared spectrometer. Composite samples were prepared for each provincial crop district and were tested for free fatty acids and fatty acid composition analyses by the reference procedures. The ISO reference method was used to determine chlorophyll in samples with large amounts of frosted seed.

Throughout the crop year as canola moves to export position export vessel loading samples are obtained and forwarded to GRL for testing by the reference analytical methods. Export samples are tested on a *tel quel* dockage basis, which is typically about 2.0% for commercially cleaned Canola, No.1 Canada.

#### **Analytical Tests**

Analytical methodology has changed somewhat over the years, with NIR instrumentation used primarily in surveys since 1990. Whenever a change in methodology took place, archival samples were tested in order to ensure that there was no significant difference in results due to methodology. Where the newer method resulted in a significant change, as with the adoption of the Dumas nitrogen procedure in 1992 (Daun & DeClercq, 1994), the previous data has been adjusted to compensate. The reference testing procedures (DeClercq & Daun, 1998) used to test official export loading samples and to calibrate the NIR instruments were:

**Oil content** by nuclear magnetic resonance according to International Organization for Standardization methods (ISO 10565:1998, 1998) (ISO 5511:1992, 1998). NMR instruments were calibrated with canola seed samples extracted with petroleum ether according to an official extraction method (AOCS Official Method Am 2-93, 1997). Results were reported as percentage, calculated to an 8.5% moisture basis.

**Protein content** by the Dumas method (AOCS Official Method Ba 4e-93, 1997) using a LECO FP-428 Nitrogen Determinator. Results were reported as percentage crude protein measured as percent of nitrogen x 6.25, calculated to an 8.5% moisture basis. Prior to 1990, protein was determined by Kjeldahl method (American Association of Cereal Chemists, 2002).

**Chlorophyll content** by extraction and spectrophotometry (AOCS Official Method Ak 2-92, 1997). Results were reported as milligrams per kilogram, seed basis. No adjustment was made for moisture.

**Glucosinolate content** by glucose release (ISO AWI 9167-3:2002, 1992), or HPLC (ISO 9167-1:1992, 1992)or gas liquid chromatography (Daun & McGregor, 1981) Results were reported as total glucosinolates on a whole seed basis expressed as micromoles per gram; 8.5% moisture basis.

**Fatty acid composition** by gas chromatography (ISO 5508:1990, 1998) of the methyl esters prepared by base catalyzed transmethylation (ISO 5509:2000, 1998). A 15 m by 0.32 mm column with a 0.5 micrometer Supelcowax 10 coating was used from 1985 forward and a 2.5 m x 2 mm glass column of GP 3% SP-2310/2% SP 2300 on 100/120 Chromosorb W AW was used prior to the availability of capillary columns.

**Iodine value** calculated from the fatty acid composition, (AOCS Recommended Practice Cd 1c-85, 1997) Major and important minor fatty acids were included in the calculation. The actual units for iodine value (g I/100 g oil) are, by convention, not reported.

**Free fatty acid content** by titration of the oil extracted with petroleum ether(Ke & Woyewoda, 1978), and expressed as percent free fatty acids in the oil (as oleic acid). Canola oil can often be highly colored making it difficult to determine the endpoint of the titration using the solvents in the official method(ISO 660:1996, 1996). The ternary solvent mixture used gives a clear solution for better endpoints. The method was validated against the official method within the GRL.

#### Results and discussion

Quality parameters of Canadian oilseeds, as measured in the newly harvested crop, vary from year to year due to environmental influences (Daun & DeClercq, 2000). It is expected that these changes in quality will be reflected in the quality of exported canola as the newly harvested seed enters the export market. The actual timing of the change in export quality and the amount and rate of change depends on the time of harvest as well as on the amount of seed from previous years that has not been sold. Environmental effects coupled with variety differences mean that individual farm samples have a large range of values (Table 1). Collection and bulking together of individual samples through the handling system results in a reduced range of values for export samples.

Over the 25-year period studied, Canada exported No.1 Canada seed with an average over 42% oil (Figure 1A) and 21% protein (Figure 1B). Export samples contained an average of 0.4% lower oil content compared to harvest survey estimates. Commercially clean canola in Canada can contain up to 2.5% dockage that contains very little oil and the oil content difference between an "as loaded" export sample and a "laboratory cleaned " survey sample varied depending on the amount and composition of the dockage. In some years (1982, 1998, and 2004) the differences were in excess of 1%. On average, differences in protein content between export surveys and harvest survey estimates were negligible at 0.1%. Dockage fractions contain significant amounts of protein. Environmental factors such as heat, frost or drought caused large annual fluctuations in these two major seed constituents (Daun & DeClercq, 2000).

The levels of glucosinolates (Figure 1D), erucic acid (Figure 2A), linolenic acid (Figure 2B) and iodine value (Figure 2C) in export shipments were all accurately predicted by harvest survey estimates. Breeding efforts have reduced the levels of seed glucosinolates and erucic acid and these continue to be well below canola specifications. Changes in the linolenic acid and iodine value over the past few years have been a combination of environmental effects coupled with the shift in species from a mixture of *Brassica. napus* and *B. rapa* to nearly exclusively *B. napus* types which traditionally have lower iodine values and higher levels of saturated fatty acids. (Daun & DeClercq, 1998)

Chlorophyll and free fatty acids were significantly higher in export shipments than in harvest survey estimates. Free fatty acid levels are higher in export materials because of the presence of dockage in that material. Dockage consists, in part, of small shriveled and broken seeds that contain significant amounts of free fatty acids. Levels of FFA also may increase as seed is stored. As a result the final export mean 0.3% higher than initial survey estimates. Over the entire 25 years, Canada has exported No.1 Canada seed with 18 mg/Kg of chlorophyll, 5 mg/kg higher than the survey estimates. Over the last 5 years, Canada has exported No.1 Canada seed with 24 mg/kg of chlorophyll, 7 mg/Kg higher than the survey estimates. Small seed with very high levels of chlorophyll can be a significant component of the dockage, particularly in years where the crop has been affected by frost. A recent study, (Daun & Siemens, 2005) showed that the dockage component of exported material in the 2004 crop year contributed, on average, about 4 mg/kg to the total amount. Part of the trend to higher chlorophyll contents in recent years may partly be due to the increased proportion of *B. napus* canola in the crop. *B. napus* canola usually has much higher levels of chlorophyll than *B. rapa* canola. Weather patterns; particularly cool growing conditions that slow maturation or early frosts that stop maturation have cause higher levels of chlorophyll in canola.

The factor most closely associated with differences between results from export surveys and harvest surveys appears to be the level of dockage included in the export samples. This dockage has been included as a part of the testing in order to provide results on a *tel quel*basis. Since many processors of canola carry out only minimal cleaning of the seed, reporting on this basis probably gives a true estimate of the quality of the material purchased. It would not be practical or even possible to provide harvest survey data on the same *tel quel*basis. Harvest survey samples, received directly from producers, may contain a wide range of dockage material and it is necessary to remove this material for accurate analysis. It is probably better to understand the rationale behind the differences than to attempt to manipulate the samples in order to remove the differences.

#### Conclusions

The CGC harvest surveys have been a reliable predictor of the quality of exports for the ensuing shipping season. Production carry over and the inclusion of dockage in commercial export shipments caused differences for some of the measured parameters. Chlorophyll and free fatty acid values were higher in export samples while oil contents were lower.. Chlorophyll levels may be heavily influenced by weather patterns; particularly cool

growing conditions that slow maturation or early frosts that stop maturation. Small, frosted seed, which contain very high levels of chlorophyll, could be a significant component of the dockage in years affected by frost.

#### References

American Association of Cereal Chemists (2002) Crude Protein -- Improved Kjeldahl Method, Copper-Titanium Dioxide Catalyst Modification, *AACC Approved Methods*. AACC Method 46.16, American Association of Cereal Chemists, St. Paul, MN.

AOCS Official Method Ak 2-92 "Determination of Chlorophyll Content in Rapeseed/Canola (Colza) by Spectrometry." (1997) *Official Methods and Recommended Practices of the AOCS* (Firestone, D. E., ed.), AOCS Press, Champaign IL.

AOCS Official Method Am 2-93 "Determination of Oil Content in Oilseeds." (1997) *Official Methods and Recommended Practices of the AOCS* (Firestone, D. E., ed.), AOCS Press, Champaign IL.

AOCS Official Method Ba 4e-93 "Generic Combustion Method for Determination of Crude Protein." (1997) *Official Methods and Recommended Practices of the AOCS* (Firestone, D. E., ed.), AOCS Press, Champaign IL.

AOCS Recommended Practice Cd 1c-85 "Calculation of Iodine Value." (1997) *Official Methods and Recommended Practices of the AOCS* (Firestone, D. E., ed.), AOCS Press, Champaign IL.

Canadian Grain Commission (January 2005a) *Harvest Quality - Harvest Survey* [Web Page] Available at http://www.grainscanada.gc.ca/Quality/harvsur/hs-e.htm, Accessed Aug. 12, 2005a.

Canadian Grain Commission (April 2005b) *Harvest Quality - Export quality data and reports* [Web Page] Available at http://grainscanada.gc.ca/Quality/exports-e.htm, Accessed Aug. 12, 2005b.

Daun, J. K., & DeClercq, D. R. (1994) J. Amer. Oil Chem. Soc. 71, 1068-1072.

Daun, J. K., & DeClercq, D. R. (1998) Bull. GCIRC 15, 27-29.

Daun, J. K., & DeClercq, D. R. (2000) Bull. G.C.I.R.C. 17, 98-103.

Daun, J. K., and D. I. McGregor. "Glucosinolate Analysis of Rapeseed(Canola). Method of the Canadian Grain Commission Grain Research Laboratory." Winnipeg: Canadian Grain Commission, Grain Research Laboratory, 1981.

Daun, J. K., & Siemens, B. (2005) Bull. GCIRC .

DeClercq, D. R. "Quality of Western Canadian Canola 2004." Web page, 2004 [accessed 16 August 2005]. Available at <u>http://grainscanada.gc.ca/Quality/Canola/2004/canola-2004-e.pdf</u>.

DeClercq, D. R., & Daun, J. K. (1998) *Quality Monitoring of Canadian Oilseed Crops*. [Web Page] Available at http://www.cgc.ca/GRL/Oilseeds/Oilseeds-e.htm., Accessed 1998.

ISO 10565:1998. "Oilseeds -- Simultaneous Determination of Oil and Water Contents --Method Using Pulsed Nuclear Magnetic Resonance Spectrometry."Geneva: International Organization for Standardization, 1998. ISO 5508:1990. "Animal and Vegetable Fats and Oils -- Analysis by Gas Chromatography of Methyl Esters of Fatty Acids ." Geneva: International Organization for Standardization, 1998.

ISO 5509:2000. "Animal and Vegetable Fats and Oils -- Preparation of Methyl Esters of Fatty Acids ."Geneva: International Organization for Standardization, 1998.

ISO 5511:1992. "Oilseeds -- Determination of Oil Content -- Method Using Continuous-Wave Low-Resolution Nuclear Magnetic Resonance Spectrometry (Rapid Method) ."Geneva: International Organization for Standardization, 1998.

ISO 660:1996. "Animal and Vegetable Fats and Oils -- Determination of Acid Value and Acidity."Geneva: International Organization for Standardization, 1996.

ISO 9167-1:1992. "Rapeseed - Determination of Glucosinolates Content - Part 1: Method Using Gradient Elution High Performance Liquid Chromatography." Geneva: International Organization for Standardization, 1992.

ISO AWI 9167-3:2002. "Rapeseed -- Determination of Glucosinolates Content -- Part 3: Spectrometric Method for Total Glucosinolates by Glucose Release." Geneva: International Organization for Standardization, 1992.

Ke, P. J., & Woyewoda, A. D. (1978) Anal. Chim. Acta 99, 387-391.

		Harves	t Survey		Export Survey				
Year	Samples	Mean	Standard Deviation	Range	Samples	Mean	Standard Deviation	Range	
1992	2220	42.5	2.3	16.5	96	42.0	0.5	2.5	
1993	1971	43.4	2.2	13.5	147	42.9	0.7	4.5	
1994	2250	43.0	2.1	13.8	197	46.5	0.5	3.8	
1995	1524	42.4	2.6	17.0	144	42.8	1.3	6.9	
1996	1589	43.4	1.9	13.5	122	43.2	0.9	5.0	
1997	1871	42.5	2.4	15.3	138	42.6	0.6	5.1	
1998	1223	42.9	2.3	13.7	171	41.7	0.5	2.8	
1999	1154	43.4	1.9	13.2	147	42.5	0.5	4.2	
2000	1108	43.2	1.9	12.4	149	42.4	0.4	2.9	
2001	978	42.9	2.1	14.7	102	42.1	0.5	3.4	
2002	1010	42.8	2.0	13.5	92	41.8	0.4	2.2	
2003	2161	41.7	2.2	16.0	126	41.3	0.7	4.4	
2004	1252	43.5	2.1	13.6	110	41.8	0.9	5.4	

Table 1. Oil contents (%, 8.5% moisture basis) for harvest survey and export survey samples from 1992 to 2004 showing the range of results for individual samples.

Table 2. Comparison (paired T test) of average values for quality parameters from harvest and export surveys, 1980 to 2004.

Parameter	Mean from Survey		Difference	Paired T	Prob. T
r arameter	Harvest <sup>a</sup>	Export <sup>b</sup>			
Oil Content, %, 8.5% M.B.	42.4	42.0	0.3	3.66	0.001
Protein Content, %N x 6.25, 8.5% M.B.	21.2	21.3	-0.1	-1.01	0.322
Chlorophyll, mg/kg	13.0	18.1	-5.2	-7.00	0.000
Erucic Acid (% of total fatty acids)	0.50	0.54	-0.04	-3.14	0.004
Total Glucosinolates ( $\mu$ M/g, whole seed, 8.5% moisture basis	15.1	15.1	0.0	0.09	0.933
Linolenic Acid (% of total fatty acids)	10.3	10.5	-0.2	-2.13	0.044
Iodine Value	115.2	115.3	-0.1	-0.54	0.595
Free Fatty Acids (% in oil, as oleic)	0.28	0.55	-0.26	-12.25	0.000

<sup>a</sup> Cleaned sample <sup>b</sup> Cleaned to export specifications (up to 2% dockage)





Figure 1. Mean values from harvest and export surveys for A oil content, B crude protein content, C chlorophyll content and D glucosinolate content for Canadian canola, 1980 to 2004







Figure 2. Mean values from harvest and export surveys for A erucic acid, B linolenic acid, C iodine value and D free fatty acids for Canadian canola,