Technical

Quality, Morphology and Storability of Canola and Rapeseed Harvested after Overwintering in Northern Alberta

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Samples of canola and rapeseed harvested after overwintering in Northern Alberta were compared to samples of fall-harvested seed from the same area. Spring-harvested seed had a weathered appearance due to changes in the epidermis and received lower grades than the unweathered fall-harvested seed. Spring-harvested seed had more oil and protein than fall-harvested seed, but also had higher free fatty acid content and conductivity and lower viability. Spring-harvested seed did not store as well as fall-harvested seed, having larger increases in free fatty acids, conductivity and storage fungi and larger decreases in viability when stored hermetically at 10% or 12.5% moisture.

A snowfall in October, 1984 covered a large amount of canola in Northern Alberta while it was still in the field. Some of this seed was harvested the following spring. Processors and grain handlers expressed some concern regarding the quality of spring-harvested seed, especially as its external appearance was very different from seed harvested in the fall.

This report summarizes the morphology, quality and storability of spring-harvested canola from Northern Alberta compared with canola from the same growing area harvested the previous fall.

MATERIALS AND METHODS

Samples. Samples of fall-harvested canola were obtained from the Grain Research Laboratory's 1984 New Crop Survey (1). Samples of spring-harvested seed were obtained through the Canadian Grain Commission's Inspection Division. Twenty-two samples each of spring- and fall-harvested seed were collected. The samples were considered individually for quality testing and morphology studies. For storage studies, composite samples of fall- and spring-harvested seed were equilibrated to 8.5%, 10.0% and 12.5% moisture by addition of water, followed by gentle mixing in a closed container overnight. The spring-harvested seeds were observed to absorb the added moisture much more rapidly than the fall-harvested seeds. After equilibration, the samples were divided into subsamples and each subsample was stored hermetically for a different length of time before testing.

Analysis. Canadian Grain Commission inspectors graded the samples according to Canadian Grain Grading Regulations (2). Seed analysis for species determination was performed by an Accredited Seed Analyst (Agriculture Canada) based on microscopic examination for morphological characteristics. Individual seeds were photographed with a Wild M400 Photomacroskop and a Wild MPS 55 Photoautomat.

Oil contents were determined by nuclear resonance spectroscopy (3) using an accurately analyzed (4) composite from the 1984 New Crop Survey as a standard. Protein contents were determined by the Kjeldahl method (5). Chlorophyll was determined by spectrophotometry (6) using pure chlorophyll A as a reference standard. Free fatty acids were determined by titration (7) of oil extracted from ground seed by petroleum ether (16 hr on a Goldfisch extraction apparatus). Crude fiber contents were determined by titrimetry (8). Conductivity, a measure of ions passing into solution from the seed, was determined using a Radiometer CDM 83 conductivity meter with a CDC 114 electrode (9). Fatty acid compositions were determined by gas chromatography (10).

Germination tests were according to the Canadian Methods and Procedures of Seed Testing (11), except the seeds were first surface sterilized and 100 instead of 200 seeds per sample were used. Four petri plates of 25 seeds each on a Whatman #3 (9 cm) filter paper were moistened with 5 ml of sterile distilled water. A daily regimen of 15 C for 8 hr in the dark, followed by 25 C for 16 hr in the light, was maintained for 10 days. The number of normal, abnormal (defective) and dead seeds were determined after 4 and 10 days. Normal seedlings showed good root and shoot development while abnormal seedlings lacked either good root or shoot development or both. Dead seeds showed no visible signs of germination.

Seeds were prepared for estimation of fungal levels by surface sterilizing for one minute in a 0.3% sodium hypochlorite solution, air drying under a laminar flow hood, and plating 100 seeds onto both potato dextrose agar (PDA) and filter paper moistened by 5 ml of 7.5% sodium chloride solution (SFP). Levels and types of field fungi and bacteria levels were determined from the seeds plated on PDA, while storage fungi types and levels were determined from seeds plated onto SFP.

RESULTS AND DISCUSSION

Seed analysis. Brassica campestris varieties predominated in both the spring- and fall-harvested samples. *B. napus* varieties were found in quantity in four of 22 samples of fall-harvested seeds, and in two of 22 samples of spring-harvested seed. Immaturity or damage prevented complete characterization of samples of mixed species. This was particularly true for one sample which was severely weathered. Although *B. campestris* varieties were the predominant varieties grown in the Northern Alberta growing area (12), it is interesting that the number of *B. napus* samples was similar for both the spring- and fall-harvested sample groups. A larger number of the later-maturing *B. napus* samples might have been expected in the spring-harvested samples.

The majority of the *B. campestris* samples were partially yellow in color, indicating the presence of Tobin or

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Candle varieties. Three of the spring-harvested and the mixed samples of fall-harvested seed contained only dark-seeded *B. campestris* and must have been unlicensed varieties, as all currently licensed varieties are mixtures of yellow and dark seeds.

Foreign material, ranging from 0 to 3.7%, was found in both spring- and fall-harvested samples. Stinkweed (*Thlaspi arvense* L.), lamb's quarters (*Chenopodium album* L.) and soil fragments were the most common foreign materials. Ball mustard (*Neslia paniculata* L.), cleavers *(Galium spurium),* hemp nettle (*Galeopsis tetrahit* L.) and lady's thumb (*Polygonum persicaria* L.) also were found. There was little difference in foreign material between the spring- and fall-harvested samples, except that lamb's quarters and soil were more frequently present in the spring harvested samples.

Grading. The fall-harvested seed was graded mainly No. 1 Canada (20 samples). The two samples graded No. 2 Canada contained mostly *B. napus* seeds and were downgraded on the basis of more than 2% distinctly green seeds. This grading distribution was as expected, considering the weather during the preceding growing period.

The general degree of maturity was sufficient to qualify most samples of spring-harvested rapeseed as No. 1 Canada. However, due to the discoloration associated with overwintering, it became necessary to exercise judgment in making grade evaluations. The overall external color, in conjunction with the appearance of the crushed seed, was used to determine the factor "not of good natural color." Color is defined under Section 9.4.3 of the Grain Grading Guide (2) as "The general degree of maturity and the amount and degree of discoloration, such as from weathering, 'including' such factors as light rime or redness associated with growing conditions which affect the general appearance but which are not extremely detrimental to quality."

Only two of the spring-harvested samples were graded No. 1 Canada. The other 20 samples were graded No. 2 Canada, mainly on the basis of seed damage (green, shrunken) and "poor color." If external discoloration were disregarded, 9 further samples would have been graded No. 1 Canada.

Visual appearance and seed morphology. The spring-harvested samples were characterized by a distinctly "weathered" appearance. Overall, the samples appeared dull and grey as well as "rough," due to a variable seed size ranging from large, bloated seeds to small or shrunken seeds. Many seeds appeared immature. On close examination, the samples consisted of a mixture of normal appearing seeds and the distinctly weathered seeds.

The dull, grey, "weathered" appearance was due to the presence of a large proportion of seeds with blistered seed coats. The outermost layers, the cuticle and outer epidermis, appeared to have separated and lifted from the underlying seed coat (Fig. 1), forming a thin, loose membrane over the seed. This might have been caused by alternate wetting and drying, coupled with freezing and thawing. Mucilage, which has been shown to be present in the epidermis of canola (13), could contribute to the process by swelling when wetted.

In the majority of weathered seeds, the entire seed surface was slightly but evenly blistered. Less frequently, the epidermis was blistered over large areas or in small circular patches (Fig. 2). The blistered layer exhibited the same surface reticulations and granulations as the underlying layers of the seed coat. Occasionally, blistered portions were very loose and peeled free of the seed coat. Beneath the blistered or peeled epidermis, the underlying seed coat appeared normal.

Less frequently, the samples contained seeds with an

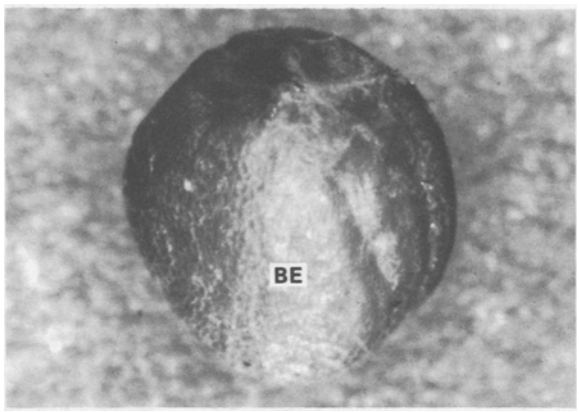

FIG. 1. Spring-harvested *B. campestris* with blistered epidermis (BE).

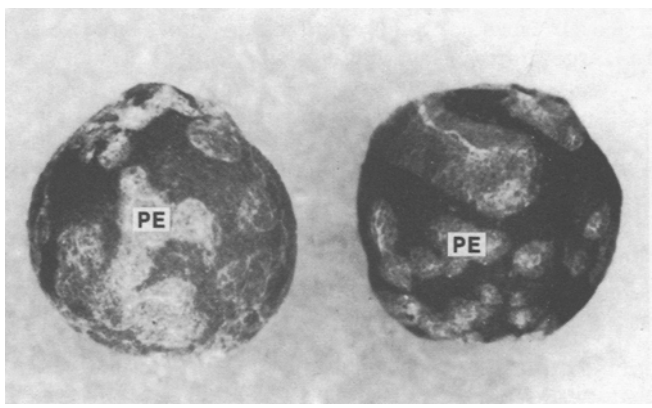

FIG. 2. Spring-harvested *B. campestris* with epidermis blistered in patches (PE).

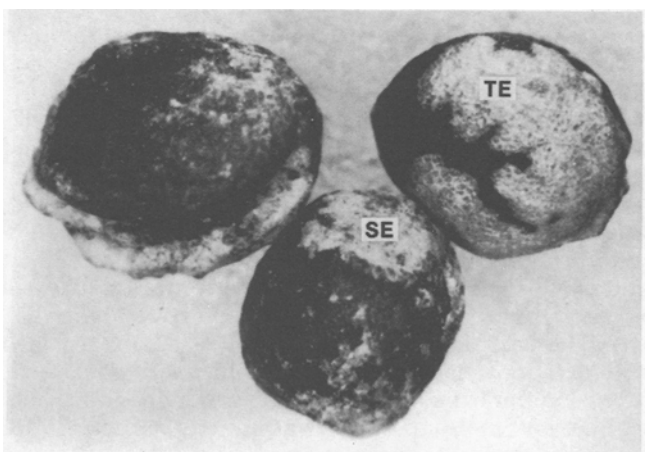

FIG. 3. Spring-harvested *B. campestris* with patches of thickened epidermis (TE) and superficial encrustations (SE).

apparently thickened epidermis or with superficial encrustations (Fig. 3). Seeds with a thickened epidermis were found particularly in association with immature seeds. Thickened areas appeared white to creamy in color. Their epidermal surface was more granular than blistered seeds. Scrapings of the surface treated with Melzer's solution contained scattered blue-black stained bodies, possibly starch granules as found previously in the epidermal layers of frost-damaged canola (14).

Superficial encrustations were creamy to brownish in color and sugary to finely matted in appearance. Scrapings treated with Melzer's solution did not contain stained bodies. The encrusted material may have been, in part, dried mucilage. In *B. campestris* varieties, up to 18% of the seeds of Tobin and 38% of the seeds of Candle may extrude mucilage when immersed in water (13). In such seeds, mucilage in the outer integument was observed to swell sufficiently when wetted to burst through the outer walls of the epidermis, forming a gelatinous film over the surface of the seeds. Mucilage containing seeds were found to be present in the fall-threshed *B. campestris* samples (Fig. 4).

Thick encrustations occasionally were found around the hilum, and in a number of cases the funicle was still attached. Such deposits could have originated from the pod. Pod membranes were found adhering to some seeds. Fungal mycelium, soil and other debris also were detected. Some seeds, which were encrusted with soil and mycelium, were soft and rotted.

Fall-harvested seeds were predominantly sound, clean, bright and reasonably mature. Small quantities of damage, such as green seed coats, cracked and loose seed coats, decorticated or broken seeds, shrunken or shriveled seeds and swollen ruptured seeds, were noted. Seeds with blistered or encrusted seed coats were very rare.

Quality measurements. Spring-harvested canola had more oil and protein, more free fatty acids, a higher conductivity and more unsaturated fatty acids than fall-harvested seed (Table 1). There was no difference between spring-harvested and fall-harvested seed for chlorophyll, fiber or seed size. One sample of spring-harvested *B. campestris* seed contained 25.4% erucic acid. This sample was obviously an unlicensed high erucic acid rapeseed variety. Varieties of *B. campestris* with high levels of erucic acid have not been licensed in Canada since 1971, but a very few farmers in the Northern Alberta growing area have persisted in growing these older varieties. Excluding this sample, there was no difference in the level of erucic acid between the spring- and fall-harvested samples.

The difference in grade distributions between the two sets of samples and presence of *B. napus* and *B. campestris* varieties made it difficult to draw conclusions from the overall data means. For example, the difference in free fatty acid levels was typical for the difference expected between No. 1 Canada and No. 2 Canada grades (15). Also, *B. napus* varieties have been found to have more oil and protein and different fatty acid compositions than *B. campestris* varieties (16). The effect of weathering on quality was assessed by comparing quality factors. Samples of spring-harvested *B. campestris* seeds which were graded No. 1 Canada, or would have been graded No. 1 Canada except for weathering, were compared with fall-harvested *B. campestris* seed graded No. 1 Canada (Table 2) and with spring-harvested samples graded No. 1 Canada (Table 3). The spring-harvested samples were ranked according to their degree of weathering, and Spearman's rank order correlation was computed between the weathering ranks and the quality factors (Table 4).

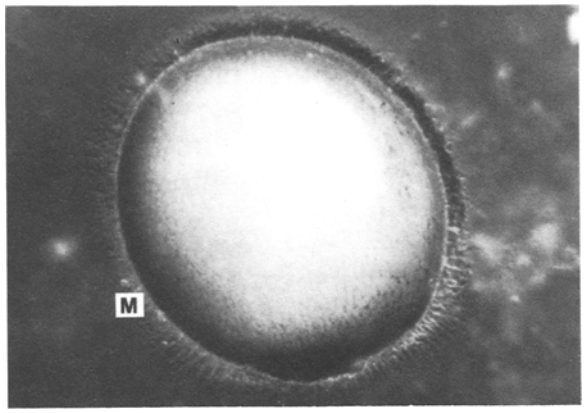

FIG. 4. Fall-harvested *B. campestris* in water with fringe of mucilage (M) extruding from epidermal cells.

Spring-harvested seed had more oil than fall-harvested seed of the same species. It is possible that the spring-harvested seeds had ripened more fully and deposited more oil than the fall-harvested seeds. In a previous study (17), frost-damaged *B. napus* seeds were observed to be immature and to contain less oil than non-damaged seeds. Spring-harvested seeds may have been planted late and may have, to a great extent, escaped the drought and heat which caused premature ripening and lower oil contents in much of the 1984 Western Canadian canola crop. A tendency toward more unsaturated fatty acids in the spring-harvested seeds also suggested that these samples had grown under cooler, wetter conditions than the fall-harvested seeds (18). Oil content was noted to decrease with increasing weathering. This suggested that although the spring-harvested seeds contained, on the average, more oil than the fall-harvested seeds, the damage within the spring-harvested seeds was related to maturity.

The higher conductivity and free fatty acid levels in the spring-harvested seeds resulted from the changes in the seed epidermis described above. Breaks in the epidermis would allow ions to escape easily, increase the likelihood of fungal invasion and allow moisture to enter the seed, possibly stimulating lipolysis. These results are similar to results reported previously for frost-damaged seeds (17).

Spring-harvested seeds were noted to be significantly more variable in size and fiber content than fall-harvested seeds (Table 5). This may be an important quality concern in crushing since, for a single roll spacing, more small seeds would remain intact, decreasing the oil extraction rate. The variability in fiber probably was linked to the variability in seed size.

The fall-harvested seed showed good germination, averaging 91% with 20 of the 22 samples having better than 80% normal germination (Table 1). The spring-harvested samples had much lower rates of normal germination, with a mean of 26% and maximum of 68%, only 8% higher than the lowest germination from the fall-

TABLE 1

Quality Tests for Spring and Fall Harvested Rapeseed

	1000 seed wt (g)	Oil content[a] (%)	Protein content[b] (%)	Crude fiber (%)	Chlorophyll (ppm)	Free fatty acids[c] (%)	Conductivity (μS)	Fatty acid composition			Germination			No. of samples
								C22:1[c] (%)	C18:3 (%)	Iodine[c] value	Normal (%)	Abnormal (%)	Dead (%)	
Spring-harvested														
Samples: Mean	2.229	42.8	36.4	11.9	7	0.7	241	1.5	2.6	121	26	26	27	22
Maximum	3.112	45.3	40.4	16.9	14	1.8	388	25.4	13.7	125	68	44	77	—
Minimum	1.794	39.8	34.6	7.7	2	0.3	79		9.5	111	9	1	2	—
Fall-harvested														
Samples: Mean	2.272	41.4	35.5	11.7	6	0.3	118	1.9	11.3	117	91	6	3	22
Maximum	3.32	46.0	40.5	16.0	25	1.4	151	7.5	13.7	124	100	20	22	—
Minimum	1.99	39.5	30.5	8.5	2	0.1	56	0.3	8.8	110	62	0	0	—

[a]5% Moisture basis.

[b]Oil-free, 8.5% moisture basis.

[c]A Wilk-Shapiro test of normality suggested that the population was not normally distributed about the mean.

TABLE 2

Comparison of *B. campestris* Samples[a]

	1000 seed wt (g)	Oil content[b] (%)	Protein content[c] (%)	Crude fiber (%)	Chlorophyll (ppm)	Free fatty acids[d] (%)	Conductivity (μS)	Fatty acid composition			Germination			No. of samples
								C22:1[d] (%)	C18:3 (%)	Iodine[d] value	Normal (%)	Abnormal (%)	Dead (%)	
Spring mean (median)	2.128	42.27	36.67	12.16	3.8	(0.4)	215.2	(1.25)	12.51	122.5	36.2	25.9	38.1	11
Fall mean (median)	2.112	40.97	35.52	11.20	2.4	(0.2)	127.2	(1.45)	11.66	118.5	91.1	5.7	3.3	18
Difference	0.016	1.30	1.15	0.96	1.4	(0.2)	88.0	(0.20)	0.85	4.00	−54.90	20.20	34.80	—
"t" value	0.267	2.495	1.336	0.876	−0.690		3.143		1.737		8.261	−5.811	−4.97	—
Sig. level	0.794	0.019	0.193	0.398	0.496		0.012		0.094		0.0001	0.001	0.001	—
Spring variance	0.032	2.79	3.80	10.54	4.5		7690		1.51		412	111	475	—
Fall variance	0.004	1.18	5.18	2.70	8.8		263		1.54		53	17	25	—
F-value	6.952	2.358	1.363	3.910	3.688		29.220		1.020		7.782	6.426	19.2	—
Sig. level	0.002	0.122	0.650	0.015	0.051		0.0002		0.978		0.002	0.002	0.002	—
Mann-Whitney statistic						165.5		99.5		129.5				
Critical value						143.0		132.0		132.0				
Sig. level						0.01		.05		.05				

[a]Fall-harvested samples graded No. 1 Canada and spring-harvested samples graded No. 1 Canada or would have been graded No. 1 Canada except for weathering.

[b]8.5% Moisture basis.

[c]Oil-free, 8.5% moisture basis.

[d]A Wilk-Shapiro test of normality suggested that the population was not normally distributed about the mean.

TABLE 3

Effect of Weathering on the Quality of Spring-Harvested *B. campestris* Seed

	1000 seed wt (g)	Oil content[a] (%)	Protein content[b] (%)	Crude fiber (%)	Chlorophyll (ppm)	Free fatty acids[c] (%)	Conductivity (μS)	Fatty acid composition			Germination			No. of samples
								C22:1[c] (%)	C18:3 (%)	Iodine[c] value	Normal (%)	Abnormal (%)	Dead (%)	
Mean *B. campestris* No. 1 Canada	1.900	41.07	36.67	15.46	4	0.5	118	15.1	11.0	115	63	19	19	2
Mean *B. campestris* No. 1 Canada except for weathering	2.185	42.5	37.0	12.0	4	0.5	242	1.1	12.9	122	30	26	44	9
Difference	−0.285	−1.5	−0.4	3.4	0	0	−124	14.0	−2.0	−8	33	−7	−25	—

[a]8.5% Moisture basis.
[b]Oil-free, 8.5% moisture basis.
[c]A Wilk-Shapiro test of normality suggested that the population was not normally distributed about the mean.

TABLE 4

Rank Order Correlations (Spearman's Rho) for Quality on Spring-Harvested Rapeseed vs. Severity of Weathering

Quality test	Spearman's rho All samples	Spearman's rho *B. campestris* No. 1[a]
Oil content	−0.343[b]	−0.608[c]
Protein content	0.495[d]	0.699[d]
Conductivity	0.449[d]	0.522[c]
Free fatty acids	0.421[c]	0.378
Chlorophyll	0.301	−0.121
Crude fiber	0.005	0.195
F.A. composition		
C22:1	−0.108	−0.42
C18:3	−0.144	0.03
Iodine value	−0.140	−0.072
1000 seed weight	0.124	−0.188
Germination		
Normal	−0.361[c]	0.777[e]
Abnormal	0.109	0.766[e]
Dead	0.398[c]	0.287

[a]*B. campestris* samples which graded No. 1 Canada or would have graded No. 1 Canada except for weathering.
[b]$p < 0.1$
[c]$p < .05$
[d]$p < .025$
[e]$p < .01$

harvested seed (graded No. 2 Canada). Abnormal germinations and particularly dead seeds were prevalent in the spring-harvested samples.

To further confirm the effect of weathering on quality, samples were ranked in order of weather damage. Significant rank-order correlations (Table 4) were found for oil content, protein content, conductivity, free fatty acids, chlorophyll and germination. Oil content decreased with increasing weather damage, while protein content, conductivity and free fatty acids increased. Germination was observed to decrease with increasing weather damage for the overall samples. Within the sub-grouping of *B. campestris* samples which would have been graded No. 1 Canada without weather damage, however, both normal and abnormal germination increased with increasing damage. Possibly with these last samples, the overall damage was insufficient to kill the seeds and the weather damage allowed easy penetration of moisture to start the germination process.

Storage. The change in the seed surface character with weathering, along with the increased conductivity and decreased viability of spring-harvested seeds, suggested that the spring-harvested seed would not store as well as fall-harvested seed.

Quality changes during storage. Some variability in results was noted, possibly due to insufficient mixing of the samples after equilibration of moisture content. The mixing problem was suggested by the presence of lumps of adhered seeds in some of the samples even though the samples were shaken gently during equilibration.

Both free fatty acids and conductivity increased more rapidly in the spring-harvested samples than in the fall-harvested samples (Figs. 5 and 6). Free fatty acids tended to increase throughout the period of the study, with the

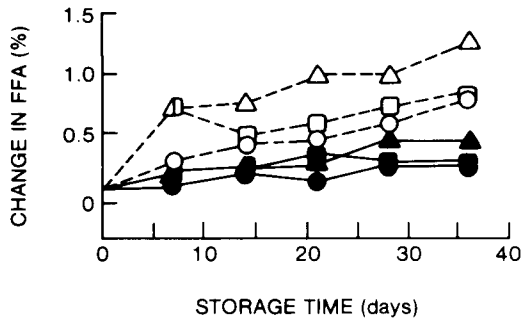

FIG. 5. Storage of spring- and fall-harvested canola. Changes in free fatty acid levels. Fall-harvested: ●, 8.5% moisture; ■, 10% moisture; ▲, 12.5% moisture. Spring-harvested: ○, 8.5% moisture; □, 10% moisture; △, 12.5% moisture.

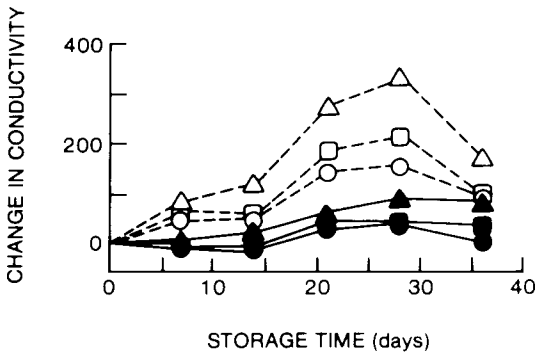

FIG. 6. Storage of spring- and fall-harvested canola. Changes in conductivities. Fall-harvested: ●, 8.5% moisture; ■, 10% moisture; ▲, 12.5% moisture. Spring-harvested: ○, 8.5% moisture; □, 10% moisture; △, 12.5% moisture.

increase being larger for samples stored at higher moisture contents. Conductivities tended to increase for the first four weeks of the study, following the same general pattern as free fatty acids with respect to moisture content. Conductivities decreased between the fourth and fifth weeks.

Germination of fall-harvested samples, at 8.5% and 10% moisture (Fig. 7), remained unchanged after five weeks storage. At the 12.5% moisture level, there was no change up to the third week, but by the end of the experiment the number of normal seedlings had declined by 26% and the number of abnormal seedlings had increased by 22%.

The spring-harvested seed did not maintain its initial level of viability as well as did the similarly treated fall-harvested samples. There was a decrease in normal seedlings for all moisture levels, the decrease being particularly significant for samples stored at 10% and 12.5% moisture. The incidence of abnormal seedlings increased over the full period of storage for samples at 8.5% and 10% moisture. Abnormal seedlings increased for the first three weeks for samples at 12.5% moisture, but decreased during the fourth and fifth week; the decrease was accompanied by a drastic increase in dead seeds. Dead seeds increased throughout the study for samples stored at 10% and 12.5% moisture but remained essentially constant for samples stored at 8.5% moisture.

Although spring-harvested seed was observed to have decreased storability compared to fall-harvested seed, seed storage did not become a major problem in 1984/85 due to the general short supply. In general, the grading assessment of the weathered seed (i.e., downgrading to No. 2 Canada in most cases) appeared to be reasonable because the weathered seed had higher levels of free fatty acids and poorer storability than unweathered seed.

Mycoflora growth during storage. Mycoflora found on the seeds after surface sterilizing were categorized into two groups, the field fungi and the storage fungi. *Alternaria* was by far the most dominant genus, comprising 80% of the field fungi found in the spring-harvested samples and 95% of those found in the fall samples. *Cladosporium* was found only on the spring-harvested samples, where it comprised about 15% of the field fungi. Of the remaining genera, *Chaetomium* was the most common, being found mainly on the spring-harvested samples. Of the four Aspergilli found, the *A. glaucus* group was the most common, followed by *A. candidus*, *A. nidulans* and *A. versicolor*.

The higher levels of field fungi in the spring-harvested samples indicated that these samples had experienced wetter field conditions than the fall-harvested crop. The presence of *Cladosporium* only in the spring-harvested samples lent support to this analysis, as this genus has been shown to be associated mainly with mature grain harvested in wet conditions (19,20).

While in both the spring and fall samples the levels of field fungi decreased over the 5-week storage period, the spring-harvested seed had a more precipitous decline in the levels of field fungi over the first three weeks than did the fall-harvested material (Fig. 8). Although a decrease in the levels of field fungi over time is a feature of stored seed (21), the decrease tends to be more rapid when seed is stored at higher moisture and temperature levels (22–24). The decrease in field fungi levels suggests that field fungi likely had negligible effects on the changes in the level of free fatty acids, conductivity and germination of the stored samples.

The effect of storage fungi was difficult to assess. The second most common storage fungi found on the samples, *A. candidus*, has been shown to be associated with increases in free fatty acids (25,26). Free fatty acid levels and seed viability did not parallel the fluctuations in storage fungi, but conductivity levels showed a similar peak after 3 to 4 weeks storage followed by a decrease after 5 weeks.

The overall level of storage fungi did increase during the 5 weeks of storage with a total of 49 counts of storage fungi in the spring-harvested samples and 25 counts in the fall-harvested samples. The initial levels of seed infection by storage fungi were 2% and 1% respectively, after which there was no steady increase but rather weekly fluctuations, with the final levels after five weeks not being appreciably different from the initial levels. In another study (24), Sinha and Mills found *Penicillium* species present on 100% of rapeseed stored at 12.5% moisture and 25 C for 30 days. The difference between their results and the results in this study may be due, largely, to the practice of surface sterilizing the seeds in the present study, a procedure not used by Mills and Sinha. In the present study, only those fungi which have penetrated the seed would survive the surface sterilization to develop later, while spores and mycelium on the

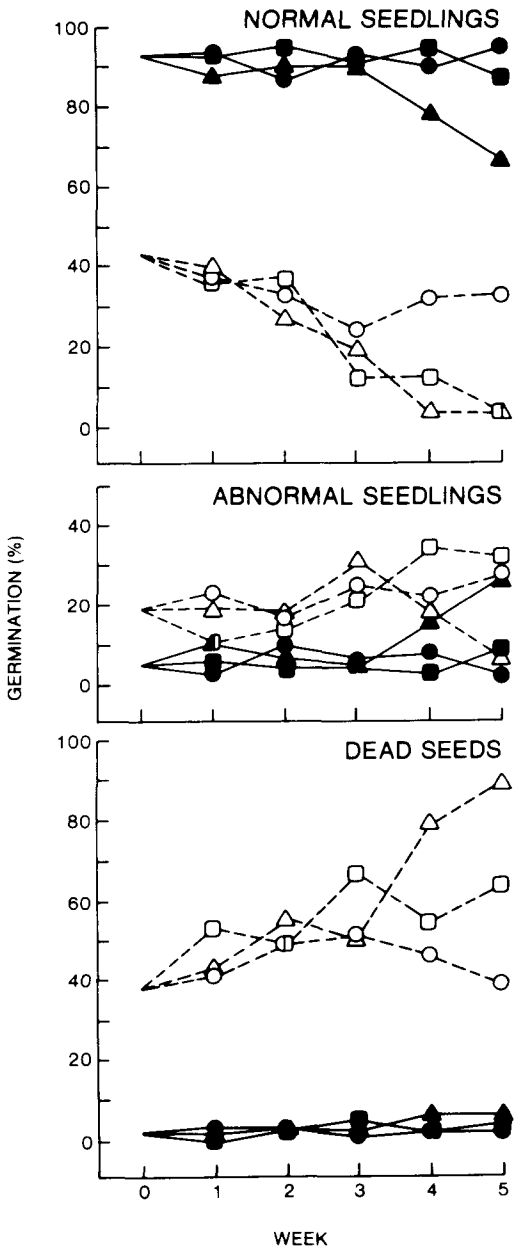

FIG. 7. Storage of spring- and fall-harvested canola. Effect on germination. Fall-harvested: ●, 8.5% moisture; ■, 10% moisture; ▲, 12.5% moisture. Spring-harvested: ○, 8.5% moisture; □, 10% moisture; △, 12.5% moisture.

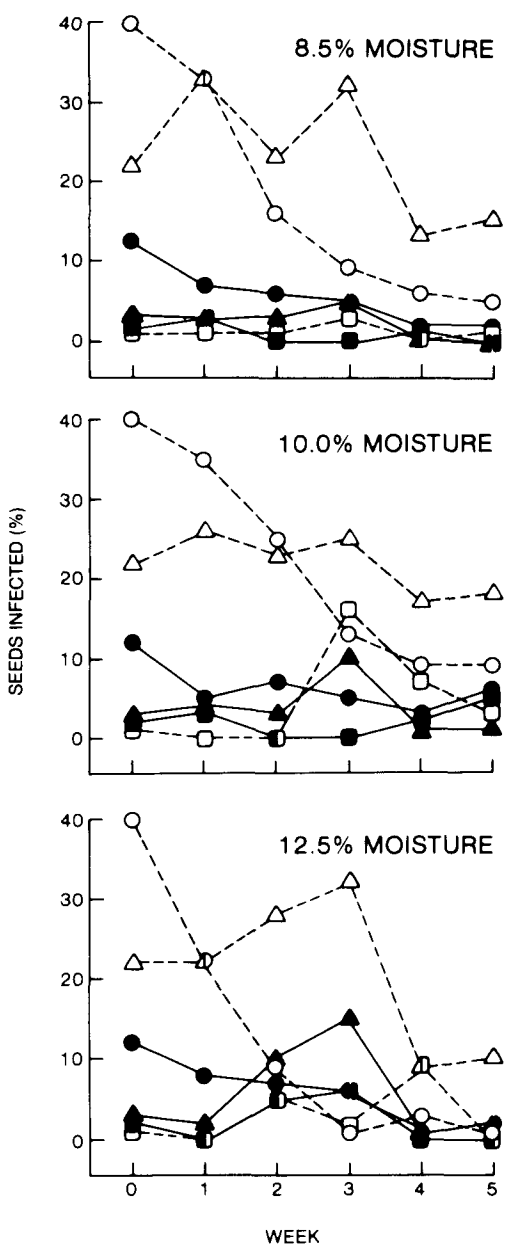

FIG. 8. Storage of spring- and fall-harvested canola. Fungal and bacterial levels. Fall-harvested: ■, storage molds; ●, field molds; ▲, bacteria. Spring-harvested: □, storage molds; ○, field molds; △, bacteria.

seed surface were eliminated. A build-up of CO_2 and other gases, accompanied by a decrease in O_2 inside the sealed containers, also may have inhibited fungi development in this study. While Mills and Sinha used a similar ratio of sample size to jar volume, they opened their jars on the average of every 18 days. In this study, up to 35 days passed before the jars were opened for sampling.

Bacteria levels generally decreased over the length of the study, but showed weekly fluctuations. As bacteria require a higher moisture content to multiply than was present in the storage experiment, the levels recorded may only reflect maintenance of initial levels, at least until later in the experiment when they began to die out. The initial levels of 22% in the spring-harvested samples, and 3% in the fall-harvested samples, suggest both wetter conditions and seed coat damage in the spring-harvested samples. Bacteria are not able to penetrate undamaged mature seed coats, but irregularities and breaks in the surface may provide protection from surface sterilizing techniques.

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REFERENCES

1. *Western Canadian Oilseeds 1984,* edited by C.J. Dempster, Canadian Grain Commission Grain Research Laboratory Crop Bulletin No. 156, Winnipeg, 1985.
2. Official Grain Grading Guide, 1985 Edition, Office of the Chief Grain Inspector, Inspection Division, Canadian Grain Commission, Winnipeg, 1985.
3. Robertson, J.A., and W.A. Morrison III, *J. Amer. Oil Chem. Soc. 56:*961 (1979).
4. *Official and Tentative Methods of the American Oil Chemists' Society,* Sunflowerseed, Oil content, Method Ai 3-75.
5. Davidson, L.D., in *Analytical Chemistry of Rapeseed and Its Products. A Symposium,* Canola Council of Canada, Winnipeg, 1980.
6. Daun, J.K., *J. Amer. Oil Chem. Soc. 53:*767 (1976).
7. Ke, P.J., and A.D. Woywoda, *Anal. Chem. Acta 99:*387 (1977).
8. Stringham, G.R., D.I. McGregor and S. Pawlowski, in *Proceedings of the 4th International Rapeseed Congress,* Geissen, DGF, Münster, 1974.
9. Mills, J.T., and W.K. Kim, *Can. J. Plant Sci. 57:*375 (1977).
10. Daun, J.K., P.B. Mazur and C.J. Marek, *J. Amer. Oil Chem. Soc. 60:*961 (1983).
11. *Methods and Procedures of Seed Testing.* Food Production and Inspection Branch, Agriculture Canada, Queen's Printer, Ottawa, 1965.
12. *Prairie Grain Varietal Survey,* edited by J.O. Wright, Canadian Cooperative Wheat Producers, Regina, 1984.
13. *Amended Description of Variety* Brassica campestris *Tobin,* Food Production and Inspection Branch, Agriculture Canada, Ottawa, 1981.
14. Van Caeseele, L., A.W. McGregor and J.T. Mills, *Amer. J. Bot. 72:*728 (1985).
15. Daun, J.K., and L.D. Burch, *J. Amer. Oil Chem. Soc. 61:*1117 (1984).
16. Downey, R.K., in *High and Low Erucic Acid Rapeseed Oils Production Usage and Toxicological Evaluation,* edited by Kramer, J.K.G., F.D. Sauer and W.J. Pigden, Academic Press, Toronto, 1983).
17. Daun, J.K., K.M. Clear and J.T. Mills, *J. Amer. Oil Chem. Soc. 62:*715 (1985).
18. Stefansson, B.R., in *Proceedings of the International Conference on the Science, Technology and Marketing of Rapeseed and Rapeseed Products,* Rapeseed Association of Canada, Vancouver, 1970.
19. Baker, G.H., E.N. Greer, J.J.C. Hinton, C.R. Jones and D.J. Stevens, *Cereal Chem. 35:*260 (1958).
20. Martens, J.W., W.L. Seaman and T.G. Atkinson, *Diseases of Field Crops in Canada, An Illustrated Compendium,* Canadian Phytopathological Society, Guelph, 1984, p. 44.
21. Christensen, C.M., and D.B. Sauer, *Storage of Cereal Grains and Their Products,* American Association of Cereal Chemists Inc., St. Paul, MN, 1982.
22. Robertson, J.A., G.W. Chapman, R.L. Wilson Jr. and R.B. Russell, *J. Amer. Oil Chem. Soc. 61:*768 (1984).
23. Lutey, R.W., and C.M. Christensen, *Phytopath. 53:*713 (1963).
24. Machacek, J.E., E. Robertson, H.A.H. Wallace and N.A. Phillips, *Can. J. Plant Sci. 41:*288 (1961).
25. McGee, D.C., and C.M. Christensen, *Phytopath. 60:*1775 (1970).
26. Bottomley, R.A., C.M. Christensen and W.F. Geddes, *Cereal Chem. 29:*53 (1952).
27. Mills, J.T., and R.N. Sinha, *Phytopath. 70:*541 (1980).

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The Determination of Fatty Acid Primary Amides by Capillary Gas Chromatography

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A method has been developed for the determination of 12 primary amides of long-chain fatty acids by capillary column gas chromatography. The method uses no derivatization or sample preparation other than extraction of the sample. A variety of commercial amide-containing materials have been analyzed successfully.

The amides, along with other soluble materials, are first separated from the host using refluxing 2-propanol containing an internal standard. The fatty acid amides are then identified and measured by gas chromatography over a programmed temperature range of 200 to 260 C.

The chromatograms obtained show sharp peaks, unique retention times and acceptable reproducibility for quantitation. *Cis-trans* isomers of several of the fatty acid amides were tested and found to be resolved under the conditions employed.

Fatty acid amides are used as lubricating additives in several types of applications in plastic food packaging. Fatty acid amides are often added to polyolefin and vinyl resins in order to modify their physical properties. The amides migrate to the surface of the plastic article, where they function as lubricants and static-charge reducers. The type of amide affects the rate of "bloom," or migration to the surface of the plastic article. Both the type and amount of amide affect the lubricating properties imparted to the product. Knowedge of the fatty acid amides content in the package permits the correlation of the type and concentration of various commercial amide preparations with changes in product performance. Knowledge of the amides in the product permits the assessment of product/package interactions. The interested reader is referred to McKenna (1).

The analytical method chosen had to cope with several complications:

(i) The amides are mixed commercially with the resin, but migrate to the surface as a result of their dissimilarity to the matrix and become inhomogeneously distributed.
(ii) The amide molecules contain polar and non-polar groups.
(iii) There exist *cis-trans* isomers of the unsaturated fatty acid amides.
(iv) Commercial materials consist of mixtures of fatty acid amides rather than a single species.