Changes in composition during the maturation of Western Schley pecans grown in Australia were investigated. Pecans of different maturity levels were collected at monthly intervals between March and June in 1999 and 2000 and analyzed for the concentrations of moisture, total lipid, sucrose, raffinose, protein, and the minerals aluminum, boron, calcium, copper, iron, potassium, magnesium, manganese, sodium, phosphorus, sulfur, and zinc. Moisture, total lipid, and calcium contents changed significantly \((p < 0.05)\) with harvest time and maturity, whereas the other components did not. Western Schley pecans grown in Australia should be harvested after the shuck has opened and it is either green or brown in color to maximize total lipid content and quality. This occurred after May 11 in 1999 and after May 17 in 2000.

**KEYWORDS:** Pecan; Western Schley; composition; maturity; harvest time; Australia

**INTRODUCTION**

The moisture content of pecans is used as a chemical index of maturity. However, it may fluctuate substantially with sequences of wet and dry weather and therefore cannot be relied upon as a guide to harvest pecans \((1)\).

The maturity of pecans varies depending on whether the nuts are harvested early or late \((2)\). Nuts from early harvests are considered to be mature after the shuck opens and the shell turns brown. At this stage the shuck is still green \((3–5)\). Nuts from the late harvest are left on the tree until their shucks naturally dry out and turn brown \((2)\). For the best quality (maximum oil content), nuts should be harvested as soon as possible after the shuck opens \((6)\). As shuck characteristics are an important index in determining maturity and harvest time, these aspects were used in this study to classify pecans into different maturity groups.

Although some information is available \((7–10)\) on the chemical composition of Western Schley pecans grown in Australia, none of this relates to changes in composition with time of harvest and maturity. This study looks at current practices in determining harvest time, which are based on either timing or subjective assessment of the shuck, and comparing these with a number of chemical indices in order to determine a reliable index for ensuring optimum quality at harvest. Data from this study will also add more information on pecan compositional profiles including moisture, total lipid, sucrose, raffinose, protein, and the minerals aluminum, boron, calcium, copper, iron, potassium, magnesium, manganese, sodium, phosphorus, sulfur, and zinc.

The aim of this study was to investigate compositional changes during the maturation of Australia-grown Western Schley pecans over two consecutive years \((1999 \text{ and } 2000)\).

**MATERIALS AND METHODS**

**Maturity Indices.** Time of harvest and maturity groups based on shuck description were used as maturity indices. For time of harvest, pecans were harvested at monthly intervals between March and June in both 1999 and 2000. For maturity groups, three categories based on shuck characteristics were delineated as follows: green-closed shuck [pecan shucks were green and closed (Figure 1)]; green-open shuck [pecan shucks were green and open (Figure 2)]; brown-open shuck [pecan shucks were brown and widely open (Figure 3)].


For each harvest, 500 nuts of each available maturity group were collected from random positions across three trees in one row. The
three trees per row were considered as one field block, and three field blocks were considered as the blocking factor in the statistical analysis.

**Sample Preparation.** At each harvest, nuts were collected from the trees by hand and stored in a cold room (0–4 °C) before being delivered to the laboratory for processing and analysis. After the shucks had been removed, samples (10–30 g) of nut-in-shell (NIS) and kernel (2 g) were randomly selected for initial moisture analyses. The remaining nuts were dried at 30 °C in a dehumidified dryer to a kernel moisture content of 3–4 g/100 g. The dried nuts were cracked and, after separation, the kernels were sealed under vacuum (–65 kPa) in lacquered, metal cans and kept at 0–4 °C until analyzed.

**Reagents.** All reagents, except acetonitrile (HPLC grade), were of AR grade and were purchased from Sigma-Aldrich, unless otherwise stated.

**Chemical Analysis.** For each treatment, all chemical analyses were conducted in duplicate on identical subsamples, unless otherwise stated.

**Moisture Content.** The moisture content of the kernel and NIS was determined using AOAC International method 40.1.04 (11) with the exception that the vacuum oven was operated at 75 °C and –70 kPa.

**Total Lipid Content.** The total lipid content was determined using a Soxhlet apparatus as described in AOAC International method 40.1.05 (11).

**Sugar Analysis.** Sugar content was determined using a modified method of Wills et al. (12) on 5 g of dried and defatted pecan residue. Ethanol/water (85% v/v) was used instead of methanol/water (85% v/v), and an amine bonded phase Waters high-performance carbohydrate column (250 × 4.6 mm, 4 µm particle size) was used instead of a Bondapak/carbohydrate column. Additionally, acetonitrile/water (83% v/v) was used as the mobile phase instead of the 80% concentration. The 1999 harvested samples were assessed using a Waters HPLC with a differential refractive index detector, and a Shimadzu HPLC (Shimadzu Class VP 5.03 with a differential refractive index detector) was used for the samples harvested in 2000. The operational conditions for these two HPLC systems were the same. Standard solutions (1 g/100 g) of glucose, fructose, and sucrose were prepared to allow comparison of retention times and quantification. A raffinose standard solution (1 g/100 g) was also prepared for comparison of the samples harvested in the 2000 season.

**Protein Content.** The protein content of ground pecans (not defatted) was determined using the Kjeldahl method as described in AOAC International method 40.1.06 (11). The protein conversion factor was 5.30.

**Mineral Analysis.** The mineral content of pecans was prepared according to the modified nitric acid/perchloric acid digestion method of Baker and Smith (13). The whole pecan kernel (not defatted) was frozen in liquid nitrogen and ground in a IKA model M20 grinder. The samples (prepared in triplicate) were dried at 65 °C, and 300 mg was digested with a mixture of nitric and perchloric acids [15 mL; 5:1 (v/v)], by slowly heating on a hot-plate from 100 to 210 °C. Upon reaching 210 °C, the solution was allowed to boil for a further 30 min and then allowed to cool to room temperature. The remaining mineral solution was diluted to 25 mL with triple-deionized water. The digests were measured for elemental composition on a Spectro model P&M inductively coupled plasma–atomic emission spectrophotometer (ICP-AES). The Australian Soil and Plant Analysis Council (ASPAC) standards were run as quality control check samples in each batch, and all digests were blank corrected. Results are presented on a dry weight basis.

**Statistical Analysis.** Data conformed to a randomized block design and were analyzed by analysis of variance (ANOVA). For all variables for which significant F values (p < 0.05) were found, comparisons of means were conducted using Fisher’s least significant difference (LSD) procedure. As the harvest dates and maturity group distributions for both years were different, no attempt was made in the experimental design to statistically compare data across the two years.
RESULTS AND DISCUSSION

\textbf{Maturity Indices.} Three maturity groups based on shuck characteristics were used to classify pecans at each harvest to obtain samples with varying compositions. However, not all maturity groups were available at all harvests. In 1999, only the green-closed shuck samples were obtained at the first harvest, whereas the green-closed and green-open shuck samples were obtained at the second harvest and the green-open and brown-open shuck samples were obtained at the third harvest. Only the brown-open shuck samples were available at the final harvest.

In 2000, the green-closed and green-open shuck samples were obtained at the first harvest, the green-closed, green-open, and brown-open shuck samples were obtained at the second and third harvests, and only the brown-open shuck samples were obtained at the final harvest.

More shuck description treatments were obtained from the experiment in 2000 compared to the 1999 trial. This could have been due to the alternate bearing habit of pecans (14-17). The year 2000 was a low production year during which fewer pecan fruits were produced on the trees than in the year 1999, which was a high production year. Alternatively, this difference may have just been due to natural variation. The differences in the number of treatments of the two years mainly occurred during the middle harvesting dates, when the pecans had started developing their physical, chemical, and sensory properties.

\textbf{Moisture Content.} 1999 Season. The NIS and kernel moisture content decreased significantly \((p < 0.05)\) as the harvest date was delayed and shuck description changed (Table 1). The green-closed shuck sample harvested on March 16 had a significantly higher \((p < 0.05)\) NIS and kernel moisture content than all of the other samples, whereas the brown-open shuck sample harvested on June 15 had a significantly lower \((p < 0.05)\) NIS and kernel moisture content than all other samples except the brown-open shuck sample harvested on May 11.

2000 Season. The NIS and kernel moisture content decreased significantly \((p < 0.05)\) with time of harvest and change in shuck description (Table 2), showing the effect of harvest date and maturity on the moisture content. Irrespective of harvest date, the NIS and kernel moisture content of the green-closed shuck samples was higher than that of the green-open shuck samples, and that of the green-open shuck samples was higher than that of the brown-open shuck samples, indicating the effect of maturity on the moisture content.

These results agree with those of Heaton et al. (1), Herrera (5), Love and Young (18), Heaton and Beuchat (19), Herrera et al. (20), and Silva et al. (21, 22), who reported that the moisture content of pecans decreased as the harvest date was delayed. It has been reported that moisture content cannot be relied upon as a guide to harvest time due to its possible fluctuation with sequences of wet and dry weather (1). However, the results from this study show that the moisture content of NIS and kernel decreased significantly \((p < 0.05)\) with harvest time and maturity, suggesting that moisture content may be a possible index of maturity. However, to confirm this proposal it would be necessary to correlate changes in moisture content with quality evaluation by sensory analysis.

Early-harvested pecans (green-open shuck samples harvested on April 13, 1999, and April 19, 2000) would need to be dried to lower the kernel moisture to 4.5 g/100 g to achieve good quality (4, 23, 24) and avoid quality deterioration caused by microorganisms (25). However, pecans harvested later (some-time after April 19) when the shuck had already turned brown and opened would require less drying to reach a stable moisture content. Pecans harvested after June 15, 1999, and May 17, 2000, would not require drying at all. Harvesting later in the season when the nuts are more mature could reduce processing.
Compositional Changes of Pecans during Maturation


costs considerably by reducing or eliminating the drying process. However, harvesting pecans too late in the season may result in rot development, mold growth, sprouting, rancidity, bitterness (4, 26), and increased exposure time to pests, insects, and adverse weather conditions (27). Moreover, darkening of the kernels is increasingly evident (18, 28, 29).

Total Lipid Content. 1999 Season. The total lipid content increased significantly \( (p < 0.05) \) with delay in harvest time and change in shuck description. However, there was no significant change \( (p > 0.05) \) in lipid content from May 11 to June 15, suggesting that lipid synthesis was complete on May 11 (Table 1). The highest total lipid content was obtained from pecans harvested between May 11 and June 15. Pecans with the highest oil content typically rate highest in flavor and quality (29, 30), and thus the pecans harvested after May 11 would be considered the best quality pecans for this season.

2000 Season. The total lipid content increased significantly \( (p < 0.05) \) with time of harvest and change in shuck description, reaching a peak of 73.97 g/100 g for the brown-open shuck sample harvested on May 17, and did not change significantly \( (p > 0.05) \) thereafter (Table 2). The green-closed shuck samples had a significantly lower \( (p < 0.05) \) total lipid content than that of the green-open and brown-open shuck samples, consistent with the literature (31) in that stages of nut maturity affect lipid content.

Smith and Loustalot (4) and Rudolph et al. (32) reported findings consistent with this work, in that the level of lipid content was low in early-harvested pecans, increased until the mid-season harvest, and thereafter remained almost constant.

Overall, maturity and harvest date affected the total lipid content of pecans in both years, and total lipid content could be used as an objective index of maturity. For maximum total lipid content, pecans should be harvested after the shuck has turned brown and opened. In 1999 this was from May 11 onward and in 2000, from May 17 onward. Variations in total lipid content between pecans from the two seasons were noted, and this is likely due to the alternate bearing of pecan trees (33, 34).

Sugar Analysis. In this study, an attempt was made to identify glucose, fructose, and sucrose contents in pecan kernels. Sucrose was the only sugar found in detectable quantities, although the concentrations of fructose and glucose approached the detection limits \((<0.01 g/100 g)\) of the instrument but could not be confidently quantified. Raffinose was also detected in pecans from the 2000 season.

Sucrose Content. 1999 Season. The ANOVA for this component was not significant \( (p > 0.05) \), indicating that the sucrose content of pecans did not change significantly \( (p > 0.05) \) with time of harvest and change in shuck description (Table 1).

2000 Season. The sucrose content of pecans harvested on March 22 was significantly lower \( (p < 0.05) \) than that of all the later harvests irrespective of shuck description (Table 2). However, after the harvest on April 19 there was virtually no further change in the sucrose content.

Wood and McMeans (35) reported that the sucrose content of pecans increased as the kernel developed. This finding is consistent with the results from the 2000 experiment but not with those from the 1999 experiment.

Western Schley pecans from the last harvest in 1999 (0.917 g/100 g) and 2000 (0.82 g/100 g) had lower sucrose contents than reported in previous studies by Wansri (7) (2.48 g/100 g) in 1996 and by Wakingling (8) (1.99 g/100 g) in 1995–1997, showing that the sucrose concentration fluctuates with year and growing location. Wood and McMeans (35) reported that a decrease in total sugars was attributed to an increase in total lipids in developing pecans. The results from the current study suggest that the increase in total lipids may be due to conversion of carbohydrates other than sucrose to total lipids, as there were no obvious changes in sucrose content in the 1999 season and there was little increase in sucrose content with time of harvest and maturity in the 2000 season. However, conversion of sucrose to lipid is a function of the available sucrose, and to a degree it is used for respiration and fatty acid metabolism. Therefore, these factors can interact to affect the sucrose content at any given time.

Pruning and shaping of the pecan trees undertaken at Trawalla orchard (especially in the 2000 season) (36) in an attempt to achieve more fruiting wood and better production (37) may have contributed to the lower level of sucrose in pecan kernels. Carbohydrates are produced by leaves (16), so the lower number of leaves per tree as a result of pruning would result in lower levels of carbohydrates produced by such trees. Variations in the results may also be due to differences in horticultural practices (31, 38), season, and geographical locations (38).

Raffinose Content. Raffinose was also detected in pecan kernels harvested in 2000, which contradicts the results of Wood and McMeans (35), who stated that sucrose was the only sugar found in pecan kernels at maturity. This is the first reported study where pecans have been found to contain raffinose. However, raffinose along with sucrose were the only quantitatively important components of sugar in ripe almond (39). The sucrose content of almond increased while the raffinose content decreased with time of harvest (39). A similar relationship between sucrose and raffinose was found for pecans in this study.

The raffinose content (Table 2) was highest in the green-closed shuck sample harvested on March 22 and decreased as harvest date was delayed and shuck color changed from green to brown. However, no raffinose was found in pecans harvested in 1999.

Protein Content. 1999 Season. The ANOVA for this component was not significant \( (p > 0.05) \), indicating that the protein content of pecans did not change significantly \( (p > 0.05) \) with time of harvest and change in shuck description (Table 1).

2000 Season. Although there were some significant changes \( (p < 0.05) \) in the protein content with harvest date and shuck description (Table 2), overall there is no obvious trend from these data.

The protein content in mature pecan kernel has been reported at 7.8 g/100 g (40, 41) and 5.08 g/100 g (8). These values are generally lower than all of the values reported in this study (Tables 1 and 2). However, the protein contents from this study are comparable to the values found by Meredith (42) and Hammer and Hunter (43) of 10 and 9.5 g/100 g, respectively. Variations may be due to differences in cultivars, cultural practices, season, and geographical location.

Protein accumulation occurs when the cotyledon expands and is completed 3 weeks after shell lignification (40). The protein content of pecans harvested in 1999 did not change significantly \( (p > 0.05) \) with time of harvest or change in shuck color, suggesting that the first sampling date was performed later than 3 weeks after the shell hardening and, therefore, no further increase in the protein was detected. Even though there were some significant changes \( (p < 0.05) \) in the protein content of pecans harvested in 2000, it would again appear that all of the samples were selected after shell hardening.
Mineral Analysis. 1999 Season. No significant differences \((p > 0.05)\) were found in the concentrations of aluminum, boron, copper, iron, potassium, sodium, phosphorus, and sulfur with time of harvest and change in shuck description. However, significant variations \((p < 0.05)\) were found in the concentrations of calcium, magnesium, manganese, and zinc (Table 3).

The calcium content decreased significantly \((p < 0.05)\) with time of harvest and change in shuck description. The green-closed shuck sample harvested on March 16 had a significantly higher \((p < 0.05)\) calcium content than all of the other samples except for the green-closed shuck sample harvested on March 16.

In general, the concentrations of calcium, magnesium, manganese, and zinc changed in a similar manner. The green-closed shuck sample harvested on March 16 appears to have higher concentrations of these minerals than the other samples. This suggests that the translocation of these minerals from soil or fertilizers to the kernels occurred while the shuck was closed. However, after the shucks had opened, this process seemed to have ceased, thus showing no effect of either harvest date or maturity on the concentration of these minerals.

Of the 12 minerals, only aluminum, calcium, copper, iron, potassium, magnesium, manganese, and zinc changed in a similar manner. The green-closed shuck sample harvested on March 16 had a significantly higher \((p < 0.05)\) zinc content than all of the other samples except for the green-closed shuck sample harvested on March 16.

In 2000 Season. Of the 12 minerals, only aluminum, calcium, copper, iron, potassium, magnesium, manganese, and zinc showed significant differences \((p < 0.05)\) in concentration with time of harvest and change in shuck description (Table 4). Although there were some significant differences \((p < 0.05)\) in the concentrations of calcium, magnesium, manganese, and zinc, overall there is no obvious trend from the results. Thus, neither time of harvest nor change in shuck description influenced the concentrations of these minerals. However, there appears to be a trend that copper content increases as harvest date is delayed. In addition, the calcium content changed significantly \((p < 0.05)\) with time of harvest and change in shuck.

### Table 3. Mineral Content (Dry Weight Basis) of Western Schley Pecans Harvested on Four Different Harvest Dates in 1999

<table>
<thead>
<tr>
<th>harvest date</th>
<th>shuck description</th>
<th>Al(^b)</th>
<th>B(^b)</th>
<th>Ca(^b)</th>
<th>Cu(^b)</th>
<th>Fe(^b)</th>
<th>K(^b)</th>
<th>Mg(^b)</th>
<th>Mn(^b)</th>
<th>Na(^b)</th>
<th>P(^b)</th>
<th>S(^b)</th>
<th>Zn(^c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>March 16</td>
<td>green-closed</td>
<td>0.88</td>
<td>0.85</td>
<td>114c</td>
<td>0.497</td>
<td>3.07</td>
<td>514</td>
<td>164b</td>
<td>13.70c</td>
<td>4.10</td>
<td>351</td>
<td>131</td>
<td>4.17cd</td>
</tr>
<tr>
<td>April 13</td>
<td>green-closed</td>
<td>0.49</td>
<td>0.38</td>
<td>78b</td>
<td>0.447</td>
<td>2.94</td>
<td>492</td>
<td>147a</td>
<td>9.70b</td>
<td>4.17</td>
<td>340</td>
<td>137</td>
<td>4.20cd</td>
</tr>
<tr>
<td>April 13</td>
<td>green-open</td>
<td>0.73</td>
<td>0.57</td>
<td>73ab</td>
<td>0.433</td>
<td>2.57</td>
<td>421</td>
<td>139a</td>
<td>10.00b</td>
<td>5.90</td>
<td>326</td>
<td>118</td>
<td>2.77ab</td>
</tr>
<tr>
<td>May 11</td>
<td>green-open</td>
<td>3.91</td>
<td>1.91</td>
<td>73ab</td>
<td>0.500</td>
<td>2.84</td>
<td>497</td>
<td>142a</td>
<td>8.77b</td>
<td>6.73</td>
<td>325</td>
<td>127</td>
<td>1.97a</td>
</tr>
<tr>
<td>May 11</td>
<td>brown-open</td>
<td>2.77</td>
<td>0.59</td>
<td>77ab</td>
<td>0.493</td>
<td>3.12</td>
<td>514</td>
<td>142a</td>
<td>7.70a</td>
<td>4.93</td>
<td>321</td>
<td>130</td>
<td>3.20bc</td>
</tr>
<tr>
<td>June 15</td>
<td>brown-open</td>
<td>0.65</td>
<td>0.27</td>
<td>72a</td>
<td>0.583</td>
<td>2.84</td>
<td>508</td>
<td>141a</td>
<td>8.73ab</td>
<td>6.30</td>
<td>329</td>
<td>125ab</td>
<td>2.70ab</td>
</tr>
<tr>
<td>LSD</td>
<td></td>
<td>NS</td>
<td>NS</td>
<td>6.2</td>
<td>NS</td>
<td>NS</td>
<td>10.5</td>
<td>NS</td>
<td>1.567</td>
<td>NS</td>
<td>NS</td>
<td>NS</td>
<td>0.971</td>
</tr>
</tbody>
</table>

\(^a\) Means in columns followed by a common letter are not significantly different \((p > 0.05)\). \(^b\) ANOVA not significant \((p > 0.05)\). \(^c\) ANOVA significant \((p < 0.05)\). NS means not significantly different \((p > 0.05)\).
description, and it appears to decrease as harvest date is delayed and the shuck begins to open whether it is green or brown. Previous studies showed that potassium, magnesium, and phosphorus accumulate rapidly in kernels during filling and maturing, whereas calcium accumulates in very small quantities (43, 44). These findings somewhat contradict the results in this study as potassium and phosphorus content increased from early harvest to midharvest and then decreased for later harvest dates, whereas calcium and magnesium contents decreased with maturity. This indicates the effect of year, location, and cultural practices on the mineral content.

The mineral content of Western Schley pecans grown in Australia and the United States is shown in Table 5. For this study, within Australia, the mineral content varies between years and appears to be comparable to the levels reported by Wakeling (8). Whereas the concentrations of potassium and sodium decreased from 1999 to 2000, the concentrations of the other minerals increased or decreased with alternate years. This could be due to alternate bearing as previously mentioned. The U.S.-grown pecans (45) had much lower concentrations of potassium, magnesium, manganese, and, particularly, calcium but higher concentrations of copper, phosphorus, and zinc than Australia-grown pecans.

Calcium levels have been reported to relate to opalescence (internal cell rupture), which is associated with low calcium content (8), and the concentration of calcium is known to decrease with maturity (1, 43). It is therefore expected that immature pecans would have lower opalescence than mature pecans.

Some minerals in pecan kernels have been shown to have health benefits to humans if consumed regularly. Pecan kernels contain high concentrations of copper and magnesium, which have been associated with a reduced risk of coronary heart disease (CHD). The relatively high intake of copper with the consumption of nuts has been suggested (46) as a possible contributor to the reduced incidence of CHD in the California Seventh-Day Adventist study (47). Copper deficiency results in high plasma cholesterol and high blood pressure and adversely affects dilation of blood vessels (46, 48). Therefore, consumption of nuts, high in copper, may help protect against cardiovascular disease. On average, consumption of 100 g of nuts can add 1.3 mg of copper to daily intake (46). The pecans in this study contained 0.6–0.8 mg/100 g copper, lower than walnuts (1.34 mg/100 g), hazelnuts (1.23 mg/100 g), and almonds (1 mg/100 g) but higher than macadamias (0.43 mg/100 g) (49).

The intake of magnesium from nuts has been suggested (45, 47) to have a possible protective influence on CHD events (47). The recommended dietary allowance for adults is 300 mg of Mg/day (50). The pecans in this study contained 141–152 mg/100 g of magnesium, lower than the level found in almonds (270 mg/100 g) but higher than that found in macadamias (100 mg/100 g) (48). Thus, if an individual consumes 100 g of pecans daily, this would represent half of the recommended dietary allowance for magnesium and would help prevent CHD.

Conclusions. For optimum quality (high total lipid and low moisture), Western Schley pecans grown at the Trawalla orchard should be harvested after the shuck has opened and it is either green or brown in color. The highest lipid content pecans were obtained from the harvests after May 11, 1999, and from May 17, 2000, onward. Pecans harvested after these dates would have low moisture content and high oil content and, therefore, high quality.

Pecans harvested prior to April 13, 1999, or April 19, 2000, showed reduced quality likely due to high moisture content and low oil content. Moreover, these pecans were difficult to handle and costly to process because the shucks were still closed and difficult to remove in a commercial situation.

Moisture content, total lipid, and calcium contents may be useful as objective indices for maturity and harvest date of pecans, and it is recommended that further work on the maturation process of Australia-grown pecans should concentrate on changes in these components. It would be useful to accumulate data over several more seasons to obtain a reliable overall picture of the variability in these important components.

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