Quality and Trading Standards for the Australian Oil Industry

Rod Mailer
NSW Department of Primary Industries
Wagga Wagga Research Institute
Pine Gully Road, Wagga Wagga, NSW 2650 Australia
rod.mailer@agric.nsw.gov.au

Abstract

Uniformity in testing procedures within the Australian oil industry is essential. Comparison between laboratories

Key words: oil — testing — analysis — quality — standards

INTRODUCTION

The methods used for oil analysis vary widely between laboratories and industries. Several organisations produce official and recommended methods of analysis, specifically for oil. These include: American Oil Chemists’ Society (AOCS), Federation of Oils, Seeds and Fats Association (FOSFA), International Olive Oil Council (IOOC) and the International Standards Organisation (ISO).

The application of these methods and the results they generate may be used in different situations with different levels of reliance on the methods accuracy. For a plant breeding program, perhaps the most important factor is to be able to select the lines in relative order of highest to lowest oil content. However, in trade, the slightest variation may amount to very large differences in monetary value.

The Australian oilseed industry is relatively small on world standards. The Australian Oilseed Federation has developed standards for the industry in Australia which aim to ensures that laboratories use similar methods to produce similar results. To further enhance this collaboration, the AOF developed Test Chek which is a laboratory proficiency program. Samples are sent from a central site to all of the participating laboratories each month. The samples are analysed for oil content, admixture, free fatty acids, fatty acid profiles and test weights by each laboratory. The results are statistically analysed and provided back to the individual participants. This program has been highly successful, particularly for oil content where variation between laboratories has been reduced from several percent to generally less than 1%.

Despite the gains generated by the AOF Test Chek program, there is some concern that some methods may be inaccurate. Oil content for example may be underestimated by the current method compared to other international standards. This is important where the results are determining the monetary value of the crop. Currently in Australia canola seed attracts a bonus of 1.5% for each 1% above 42% oil and therefore the oil content is critical for trade. The AOF have now initiated a study to compare oil extraction methods and to identify the method most useful in assessing the value of the crop.

Other methods are also critical for the oil industry to ensure the true value of the commodity. Chlorophyll for example has been responsible for quality reduction in canola. The AOF have recently initiated a project to help select a meaningful test designed to identify the level of chlorophyll at which it becomes a problem for refiners to remove in processing. This will replace the AOF methods which were based on AOCS methods.

Near Infra Red spectrophotometers have found their way into many applications in determining sample quality. These instruments rely totally on the calibration data entered into the software which has been generated by laboratory chemical analysis. The method used to generate that calibration set will determine the results which the instrument will provide. However, this type of application is commonly used in trade situations as well as in rapid
assessment of plant material in breeding programs. It has proven to be reliable in the hands of competent operators to satisfy both requirements of accuracy.

The methods utilised in sample analysis should be based on the application of the commodity. The oil, chlorophyll or other tests should tell the industry about the crop value. Methods must be reasonably easy to carry out by a well established laboratory and be economical. The method must also provide a result in an acceptable timeframe and the results must be reproducible.

**MATERIALS AND METHODS**

**Oil content**

Comparison of oil concentration was carried out using the methods of the American Oil Chemists’ Society (AOCS), the Australian Oilseeds Federation (AOF), Federation of Oils, Seeds and Fats Association (FOSFA) and International Standards Organisation (ISO). All methods rely on a similar method of extraction using a butt tube or Goldfische extractor (see diagram) and solvent such as hexane or petroleum spirit. Seed from five samples provided from the AOF Test Chek program were analysed.

ISO 659 is equivalent to FOSFA and AOCS AM 2-93. All three methods require the sample to be ground on three consecutive occasions between three oil extractions. Grinding is carried out using a mill in the first instance. Subsequent grinds may be carried out with Swedish Tube or ball mill. To facilitate rapid solvent extraction, an apparatus such as the Foss Soxtec extractor may be used.

AOF 4-1.25 is equivalent to AOCS Af 3-54. This is also the method identified by AOCS to be used in the Laboratory proficiency program. This method requires two grinds and two solvent extractions.

WWAI method is carried out using a single extraction after grinding. The sample is ground in a water cooled Foss mill using a short series of grinds and mixing of the sample. The sample is then extracted overnight with Goldfische extractors.

**Protein**

The AOF 4-33 method is based on AOCS Baa 4e-93, combustion method. Alternatively, a Kjeldahl digest method is described in AOF 4-34 equivalent to AOCS Ba 4d-90.

**Glucosinolates**

AOF 4-1.22 is a glucosinolate hydrolysis method developed at the Wagga Wagga Agricultural Institute based on the Canadian Grain Commission’s HPLC test. The HPLC method determines individual intact glucosinolates whereas the hydrolysis method measures the total concentration of glucosinolates as glucose. The glucose method has been used to establish a calibration for NIR.

**Chlorophyll**

Four methods are described by AOCS for the measurement of chlorophyll: chlorophyll in seed, AOCS Ak2-92 and chlorophyll in oil, AOCS Ch4-91, Cc13d-55, and Cc13i-96. All use different factors in the calculation designed to account for breakdown of chlorophyll to by-products such as pheophytins. Therefore all give different results. AOF have agreed to use Cc13d-55 for oil to get uniformity amongst the industry. However, this still results in differences between seed chlorophyll tests and oil.
RESULTS

Oil content
Duplicate results obtained from five samples analysed by three different methods are illustrated in Table 1. The results from the WWAI method using one grind is similar to the AOF method. The average difference between the two grind method of the AOF (AOCS Af 3-54) and the single grind used by WWAI was only 0.11\% and well within the accepted standards set by the Test Chek program. The results are also within close agreement with the results of the combined laboratories of the Test Chek program indicating the method is reproducible between laboratories.

The ISO three grind method was consistently higher than the other two methods by 0.7\% on average. The difference varied for each sample indicating that some samples may be easier to extract than others.

Table 1. Comparison of three methods of extraction of oil from canola on five samples previously analysed in the AOF Test Chek program.

<table>
<thead>
<tr>
<th>Sample</th>
<th>AOF Test Check Program</th>
<th>WWAI</th>
<th>AOF</th>
<th>ISO-659</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td></td>
<td>42.95</td>
<td>43.35</td>
<td>43.75</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>42.92</td>
<td>43.15</td>
<td>43.96</td>
</tr>
<tr>
<td>#4</td>
<td></td>
<td>38.69</td>
<td>38.22</td>
<td>39.13</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>38.49</td>
<td>38.67</td>
<td>39.13</td>
</tr>
<tr>
<td>#5</td>
<td></td>
<td>46.29</td>
<td>46.60</td>
<td>47.18</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>46.36</td>
<td>46.54</td>
<td>47.04</td>
</tr>
<tr>
<td>#7</td>
<td></td>
<td>39.92</td>
<td>38.81</td>
<td>40.14</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>39.71</td>
<td>39.54</td>
<td>40.35</td>
</tr>
<tr>
<td>#8</td>
<td></td>
<td>37.12</td>
<td>36.86</td>
<td>37.99</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>37.50</td>
<td>37.08</td>
<td>38.10</td>
</tr>
</tbody>
</table>

Protein
No comparisons have been made for protein analysis methods although several studies previously have shown that Kjeldahl produces consistently lower nitrogen levels than the Dumas or combustion method (Wiles and Kissling 1998, Williams et al. 1998).

Chlorophyll
The traditional method for determining chlorophyll at bulk handling receival sites has been to crush 100 seeds and look for the number of distinctly green ones. This method is flawed and can produce erroneous results due to the variability in the intensity of greenness. A more accurate approach has been to extract the oil and measure the chlorophyll content by spectrophotometry. The WWAI are currently using methods Ak2-92 for seed and Cc13d-55 for oil but using the same factor for both, that is the factor used for Ak2-92, \( f = 13 \).
**Glucosinolates**

Glucosinolates have been measured by glucose hydrolysis and compared to analysis by NIR (Fig. 1). Results of the two methods agreed within 4 µmoles for 89% of the 253 samples tested. The average difference of the other 25 samples was 7 µmoles. Previous comparisons with the glucose method and the HPLC method have shown good comparisons (Mailer and Vonarx, 1989).

*Fig. 1. Comparison of glucosinolate analysis by NIR and glucose oxidase analysis.*

**DISCUSSION**

Despite the availability of internationally recognised standards for many of the tests for oil content and oil components, there is often more than one method and the methods often give different results. This is particularly important where the value of the commodity depends on the results of the test such as for oil content.

The need to establish uniformity in the industry is obvious to ensure that trade, particularly with export, is presenting data on a similar basis. This can influence the returns to growers and can also influence the comparison of the Australian crop with trading competitors.

The Australian Oilseed Federation, through the Standards Committee, has done a lot to establish methods for use in the Australian industry. This has ensured that in most cases laboratories are using similar standards. However, there remain some differences, particularly with oil content, and these differences need to be overcome. Other tests which the AOF are working to standardise within Australia include chlorophyll in canola seed and protein quality in canola meal. These projects are currently in process.

Proficiency programs, between laboratories, ensure that operators are competent to carry out the methods and that results are similar. There are several such test programs including the AOCS proficiency program, IOOC laboratory evaluation and the AOF Test Chek program. All laboratories engaged in oil analysis are encouraged to take part in one or more of these programs.

Participation in the AOF Standards Committee by chemists is also encouraged to allow the interchange of ideas on improvements to the standards and associated projects.

**ACKNOWLEDGEMENTS**

Data for this presentation were obtained by Rebecca Stooke and Amanda McFadden of the Wagga Wagga Agricultural Institute. Samples were obtained from NSW Department of Primary Industry canola trials.

**REFERENCES**

McFadden, R., R.J. Mailer and P.A. Parker, 2004: Quality of Australian Canola, Volume 11. ISSN 1322-9397.


