The National Isotope and Trace Element (NITE) Profile for Australian Citrus

Enabling traceability differentiation of Australian oranges by Country of Origin and Australian Region

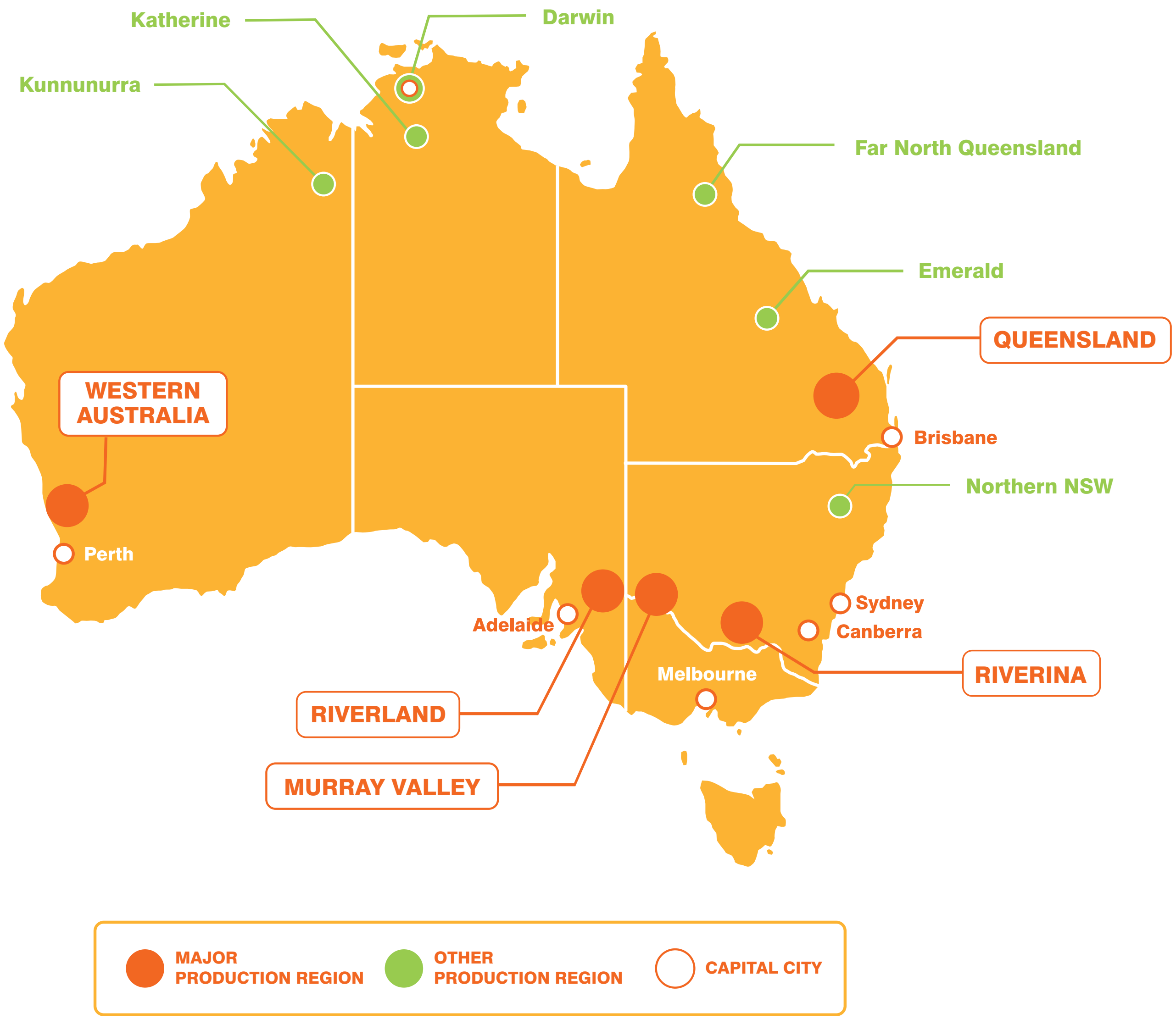
This pioneering project report outlines how a fundamental scientific traceability data set has been established for Australian citrus, creating the **National Isotope and Trace Element (NITE) Profile**

"This project has demonstrated the use of science for citrus traceability. Isotope and Trace Element testing has proved how Australian fruit can be differentiated from the fruit of other nations, when it's in market. What's more, we can also characterise fruit from our five Australian growing regions.

This gives us more certainty in the face of a food recall, or when protecting Australian brands from food fraud. We now have a repository of information for proving the origin of Australian oranges and we need to keep getting samples to grow our database. We look forward to working with growers and citrus organisations around the world as we develop the National Isotope and Trace Element (NITE) Profile for Australia into the future."

NATHAN HANCOCK, CEO, CITRUS AUSTRALIA

The Value and Diversity of Australian Citrus Exports



The Australian citrus industry currently exports up to $540 million worth of premium citrus products to key overseas markets.

The increased global demand for high-quality food products has led to premium prices for Australian products in international markets. However, with market growth also comes the need for increased traceability and origin verification of products to protect brands and ensure continued consumer satisfaction.

Today, over 30,000 hectares of citrus are planted by Australian growers. Australia has five major citrus production areas. The major production regions are in the Riverina, Queensland, Murray Valley, Riverland, and Western Australia.

For citrus fruit sold under Australian labelling and packaging into domestic and international markets, there are several significant risks that could occur including:

- food fraud
- food substitution with inferior products
- biosecurity incidents
- food recalls
- mislabeling of origin or variety

The Challenges of Food Fraud

Australian citrus products undergo stringent food safety and biosecurity processes to ensure that safe, tasty, premium fruit is available to customers in export markets. Competitor countries also supply both premium and sub-premium fruit to these same export markets.

Potential issues in supply chains can include food substitution / food fraud, with inferior product being sold under Australian labelling or packaging. This poses a risk for food safety or biosecurity incidents and may incur unnecessary costs and reputational damage to the Australian citrus industry.

The Benefits of Traceability

The Australian Citrus industry is seeking to increase the traceability of its fruit products as they are distributed across multiple supply chains to customers in many countries.

Traceability solutions can protect Australian fruit by demonstrating the journey of food from farm to the consumer. Traceability benefits include:

- food substitution / food fraud protection
- accurate product selection in food recalls
- biosecurity incident responses
- providing verified scientific data to support claims or litigation
- brand protection for Australian growers
- consumer trust in the safety and quality of Australian products

Options for Traceability

Traceability solutions can operate in different ways including:

DIGITAL TRACEABILITY

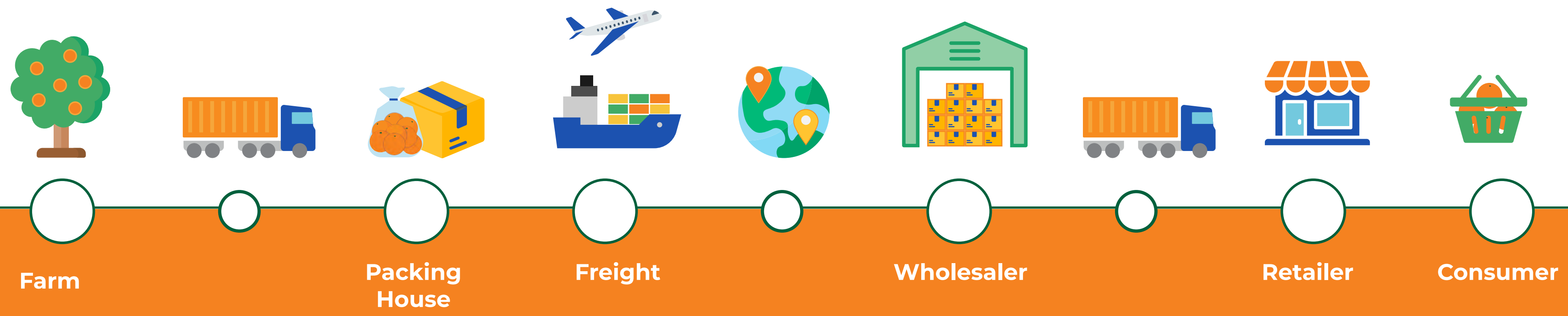
Everyday citrus traceability can be achieved through serialized unique labelling of fruit, with smartphone scanning of product labels by supply chain partners and consumers providing product authentication, and maintenance of data within a product cloud. A recent example is the Citrus Traceability Project with Mildura Fruit Company, with application of GS1-Digital Link enabled QR codes on fruit labels. See the Citrus Traceability Project final report

LABORATORY TRACEABILITY

Costs and time constraints make laboratory testing traceability most appropriate for product authentication during crises, claims or litigation. Laboratory testing provides a scientific method of tracing the source of fruits to their place of growth. This pioneering project report outlines how a fundamental scientific traceability data set has been established for Australian citrus, creating the National Isotope and Trace Element (NITE) Profile

MAPPING TRACEABILITY

The National Citrus Map has recently been developed to provide a visual data set of citrus orchards across Australia. Read the National Citrus Map final report here



What is Laboratory Traceability?

Origin verification using isotopes and trace element analysis has been undertaken in a variety of food commodities including wine, avocado, fruit juices, rice and those of animal-origin. Differences in isotope ratios and trace element contents are an effective means of tracing foods due to unique profiles caused by geology, environmental conditions, and agricultural practices. The main premise of laboratory origin verification testing is to find variables with sufficient discriminant ability for the countries, regions or farms of interest. This may be possible using one or two variables, or it may take many. Ideally, the aim is to discover which variables have the most influence and rank them to eliminate analyses that do not add value.

The National Isotope and Trace Element (NITE) Profile for Australian Citrus

For this project, Citrus Australia and Agriculture Victoria partnered to establish the National Isotope and Trace Element (NITE) Profile for Australian citrus through scientific testing. This study is the first of its kind to identify Australian oranges at a regional and international level. The key goal is to provide an internationally accepted traceability method that addresses 2 key questions:

1. **Can we determine if an orange is from Australia?**
2. **Can we differentiate Australian oranges into one of the 5 Australian growing regions?**

The project successfully answered 'yes' to both questions.

A testing program also indicated, with further data, a third question could be answered in future:

3. **Can we differentiate Australian oranges from separate orchards within a single production area?**

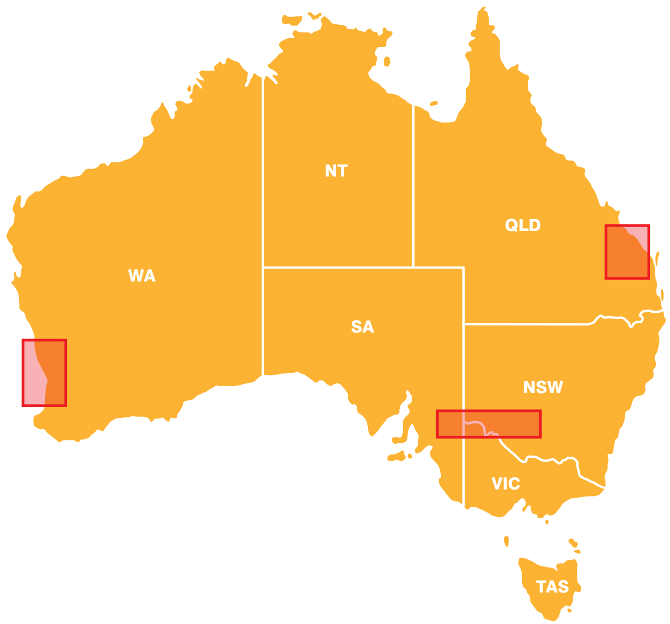
26 Australian samples were collected from 5 Australian growing regions: Western Australia, Queensland, and in close proximity to each other, New South Wales, Victoria and South Australia.

13 samples of oranges grown in 4 other nations were collected: New Zealand, China, USA, Egypt.

The project was conducted over two Phases. Dividing the project into Phases 1 and 2, provided the opportunity to sample across 2 seasons and to review and modify Phase 2 test parameters as required.

Isotope ratios and trace elements were measured, and the results statistically analysed, developing a set of parameters to define fruit at two levels:

1. Country-of-Origin
2. Australian Regions

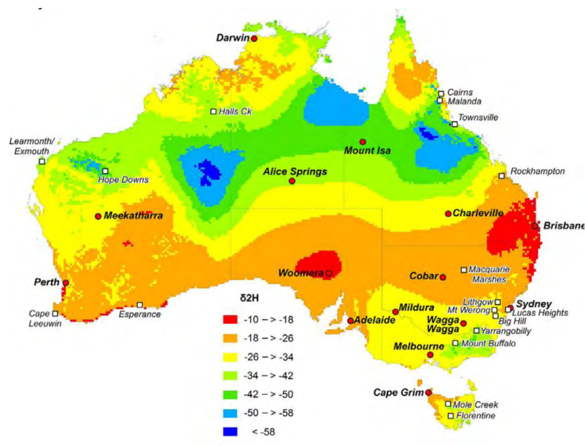


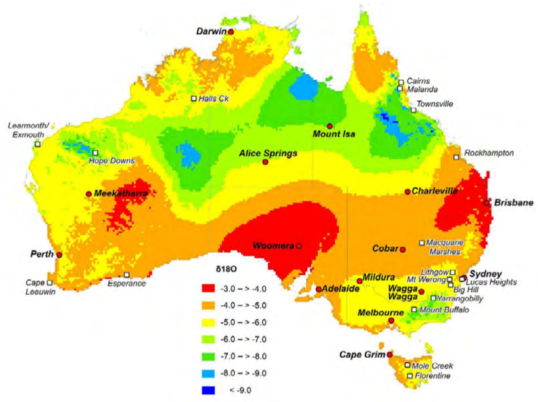
Isotope Ratios

What are isotopes? Elements may exist in slightly different forms depending on the number of neutrons in their atomic nucleus. These varied forms are called isotopes and have different atomic masses, changing the way they move naturally through the environment. Their ratios can be used as environmental tracers reflecting the source, history and origin of each element.

Isotope ratios in agricultural products such as fruit, differ depending on local geology, climatic and environmental conditions and agricultural practices, thereby providing a form of atomic-level identification specific to a particular product and region that cannot be tampered with, or altered.

Isotope	Association	Influenced by
Hydrogen ($\delta\,^{2}H$)	Geographical origin. Strongly latitude-dependent.	Associated with the local climate and environmental conditions such as precipitation, humidity, temperature, and altitude. Subsequently, $\delta\,^{2}H$ values of fruit are directly correlated to the water taken up from the soil.
Oxygen ($\delta\,^{18}O$)	Geographical origin. Strongly latitude-dependent.	Associated with the local climate and environmental conditions such as precipitation, humidity, temperature, and altitude. Subsequently, $\delta\,^{18}O$ values of fruit are directly correlated to the water taken up from the soil and the plant CO_2 respiration.
Carbon ($\delta\,^{13}C$)	Geographical origin	Carbon isotopes are primarily affected by CO_2 respiration from plants. $\delta\,^{13}C$ values can be used for geographical origin determination as a decreasing carbon isotope (^{13}C /^{12}C ratio) temperature gradient exists for plants from the equator to the poles.
Nitrogen ($\delta\,^{15}N$)	Farming practices	Highly indicative of soil nutrients and specific farming practices. While they are not a geographical indicator, they can provide information about fertilizer use and type. These agricultural practices may be highly reflective of a specific region or country.
Sulphur ($\delta\,^{34}S$)	Geographical origin, farming practices, industrial processes	Influenced by both environmental and human factors. Natural sources of sulphur are sea water, mineralised and volcanic rocks, bacterial processes and human inputs from fertilizers and industrial emissions.
Strontium ($^{87/86}Sr$)	Geographical origin	Closely related to the geological age of underlying rocks which contribute to the local growing soils. The ratio of $^{87/86}Sr$ in soils and plant products can be used to infer location, especially between older, weathered soils and more modern, younger volcanic soils. N.B. An Australian perspective: Traditionally, $^{87/86}Sr$ isotopes have been found to be a valuable tool in distinguishing the origin of foods from different countries. However, in this project, $^{87/86}Sr$ isotopes were found to be » similar between Australia and Chinese fruit » similar across the fruit of some Australian regions. Strontium isotopes therefore were not considered useful when discriminating Australian fruits and consequently testing for $^{87/86}Sr$ isotopes was not conducted during Phase 2 of the project.





Hydrogen and oxygen ($\delta^{2}H$ and $\delta^{18}O$) isotope gradients of Australian precipitation, demonstrating strong latitude dependence.

Source: https://apo.ansto.gov.au/dspace/bitstream/10238/10479/1/1-s2.0-S0048969718325666X-main.pdf

Trace Elements

Trace elements can also be used as an authentication and traceability tool. Around 20 to 30 key trace elements are known to be transferred from the soil into plants at a measurable level. Often the combination and concentration of specific trace elements are useful to determine subtle differences between plants, cultivars, and growing regions. These values vary depending on soil, geology, fertilisers, top dressings, and irrigation water, and may discriminate specific farming practices and geographical regions.

There are several useful trace elements found in fruit juice and pulp:

Trace Element	Association
Calcium (Ca)	Applied as a top dressing to soils in Australia to increase fruit rind quality and shelf life.
Caesium (Cs)	Derived from past atmospheric nuclear events and is enhanced in eroded soils. Lower levels may also be an indicator of topsoil redistribution and tilling, associated with increased land cultivation.
Rubidium (Rb)	Rubidium is enriched in recent crystal rocks, such as those found in New Zealand's tectonically active environments. Rubidium decays over time to strontium, therefore Rubidium (Rb) and strontium (Sr) concentrations have inversely related trends.
Strontium (Sr)	Strontium is more abundant in older weathered rocks, such as ancient, weathered rocks that form Australian soils.
Magnesium (Mg) Cobalt (Co) Barium (Ba) Lanthanum (La) Cerium (Ce) Zinc (Zn)	Typically found within specific soil types. Frequently found in mineral fertilisers and are likely to be specific to different cultivation practices from different countries. Lanthanum and cerium are naturally occurring rare earth elements (REE), occurring in some rock types, and distribution may be enhanced by processes such as mining.

Sample Protocol

A single sample was comprised of the juice and pulp extracted from 5-6 fruit (Navel Oranges) from a single farm (collection site).

Of high importance during the sample collection process was that water or carbon-based products (e.g., soil) were not introduced into the samples.

Oranges from a single farm were collected into one composite sample collecting as much pulp (at least 2Tbsp per jar) as possible and obtaining at least 200ml of combined juice and pulp, avoiding inclusion of any pith.

Samples were then thoroughly mixed to produce a homogenous sample and divided into four aliquots of approximately 50-70mls and separated in to 90ml- 95kPa compliant specimen jars, sealed and frozen.

One sample from each farm was shipped for laboratory testing with three replicate samples stored frozen by Citrus Australia as backup samples.

Shipping samples to the laboratory as frozen assisted with their preservation and consistency, as well as minimising biosecurity requirements during shipping.

Sample Numbers

Trace Element	Navel Orange Samples			Notes
	Phase 1	Phase 2	Total	
Australia	10	16	26	Domestic samples from 25 separate farms: 5x New South Wales, 5x South Australia, 5x Queensland, 5x Western Australia, 5x Victoria. 1 Australian fruit sample was also sourced outside of Australia.
New Zealand (NZ)	2	2	4	International samples were sourced within Australia, New Zealand, and China.
China (China)	2	2	4	
USA (USA)		4	4	
Egypt (Egypt)		1	1	
Total			**39**	

Testing Method

All samples were measured in New Zealand at the Stable Isotope Laboratory at GNS Science in Lower Hutt and at University of Otago.

ISOTOPE RATIOS

Isotope ratios were calculated against working in-house standards and calibrated and normalised against international reference materials.

Isotope	Method	Equipment
Carbon (δ ^{13}C)	EA-IRMS Bulk combustion (pulp) EA-IRMS Bulk combustion (juice)	» EuroVector EA3000, Italy » Isoprime 100, Elementar, England
Nitrogen (δ ^{15}N)	EA-IRMS Bulk combustion (pulp)	» EuroVector EA3000, Italy » Isoprime 100, Elementar, England
Oxygen (δ ^{18}O)	HTEA-IRMS Bulk combustion (pulp) CO^2 Gas exchange (juice)	» Isotope PYRO Cube, Elementar, Germany » Isoprime 100, Elementar, England » Isoprime 100 mass spectrometer using an Aquaprep device and CO2 equilibration procedure
Hydrogen (δ ^{2}H)	Direct injection Hokko bead H_2 gas exchange (juice)	Method 1. » EuroAS300 liquid autosampler onto a EuroVector Chrome HD elemental analyser » IsoPrime isotope ratio mass spectrometer in continuous-flow mode (EA-IRMS) Method 2. » Isoprime mass spectrometer using Hokko beads and an Aquaprep device
Strontium ($^{87/86}$Sr)	HR MC-ICP-MS	» Nu Plasma-High Resolution MultiCollector ICP-MS in wet plasma mode

TRACE ELEMENTS

Trace Elements	Method	Equipment
Ce, Cs, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sm, Tm, U, Yb, Co, V, Ni, Ba, Cd, Rb, Sb, Sn, Li, Mn, As, Se, Pb, Cu, Mo, B, Cr, Zn, Al, Na, K, Fe, Ca, P, Sr, Mg	Microwave digestion in HNO_3	Nu Plasma-High Resolution MultiCollector ICP-MS

Testing Phases

Testing was undertaken in two distinct phases; Phase 1 was conducted to determine a characteristic trace element and isotope ratio profile for Australian navel oranges and was limited to 3 Australian states. Phase 2 refined the results of Phase 1 and was expanded to include 5 Australian states and fruit from other countries.

TRACE ELEMENTS	ISOTOPE RATIOS
Ce, Cs, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sm, Tm, U, Yb, Co, V, Ni, Ba, Cd, Rb, Sb, Sn, Li, Mn, As, Se, Pb, Cu, Mo, B, Cr, Zn, Al, Na, K, Fe, Ca, P, Sr, Mg	**Juice** $\delta^{13}C$, $\delta^{18}O$, $\delta^{2}H$ **Pulp** $\delta^{13}C$, $\delta^{18}O$, $\delta^{15}N$, $^{87/86}Sr$

Phase 1 Analysis*

Fruit samples

- 9x Australia – VIC(1), NSW(5), SA(3)
- 1x Australia (Unknown origin, sourced in NZ)
- 2x New Zealand
- 2x China

19 most useful trace elements	3 most useful Isotope Ratios
B, Ca, Ce, Co, Cs, Cu, Fe, K, La, Mg, Mn, Na, Ni, P, Pb, Rb, Sn, Sr, Zn	**Juice** $\delta^{18}O$, $\delta^{2}H$ **Pulp** $\delta^{18}O$

Phase 2 Analysis*

Fruit samples

- 16x Australia –VIC(4), QLD(5), SA(2), WA(5)
- 2x New Zealand (excluding trace elements)
- 2x China
- 4x USA (excluding trace elements for 2 samples)
- 1x Egypt

Final Analysis Results

Country-of-Origin
Most useful combined trace elements and isotope ratios for discrimination of Australia from other countries

Australian Regions
Most useful combined trace elements and isotope ratios for discrimination of Australian regions from each other, with further testing recommended to build a baseline data set

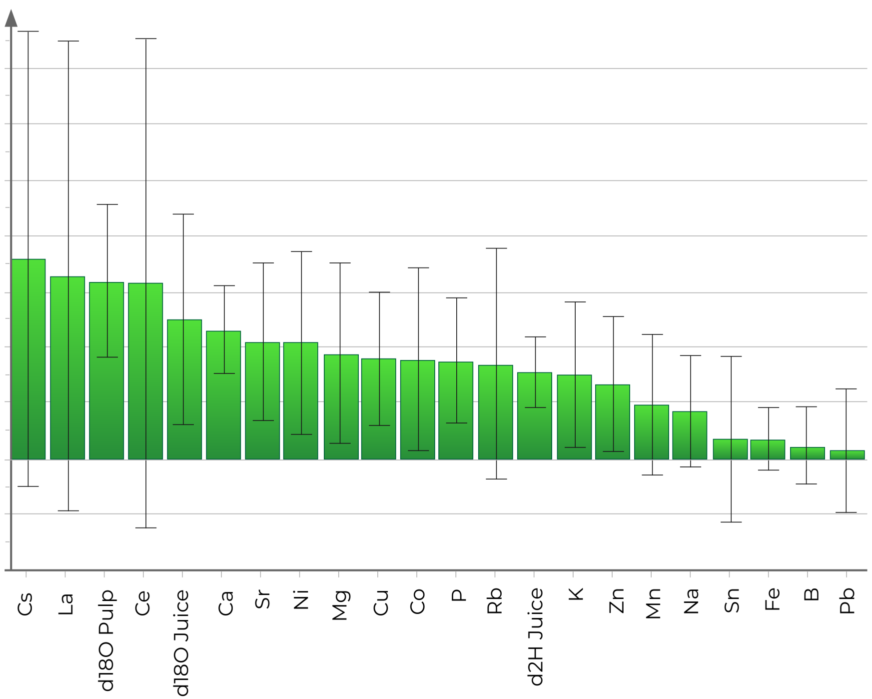
**Phase 1 and Phase 2 Analysis: Biplots and multivariate supervised statistical analysis undertaken to determine which variables were most useful for origin classification. Individual isotopes plotted against each other to determine correlations between isotopes (R2), and multivariate supervised classification undertaken via a partial least squares discriminant analysis (PLS-DA) model.*

Trace Elements and Isotope Ratios for Australia

The 19 trace elements and 3 isotope ratios below, pinpointed during Phase 1 and 2 testing, were found to be the best variables for Australia to classify different fruit origins by country and Australian regions.

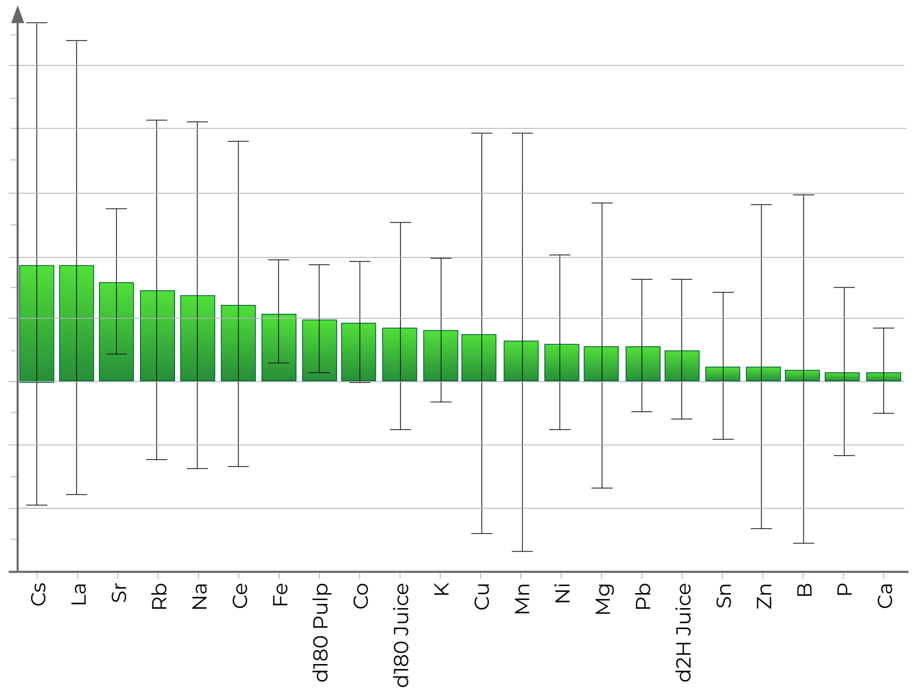
COUNTRY-OF-ORIGIN

Key trace elements and isotope ratios for characterising Australian oranges based on country differences from the other countries tested are shown in the diagram below, in order of priority.



AUSTRALIAN REGIONS

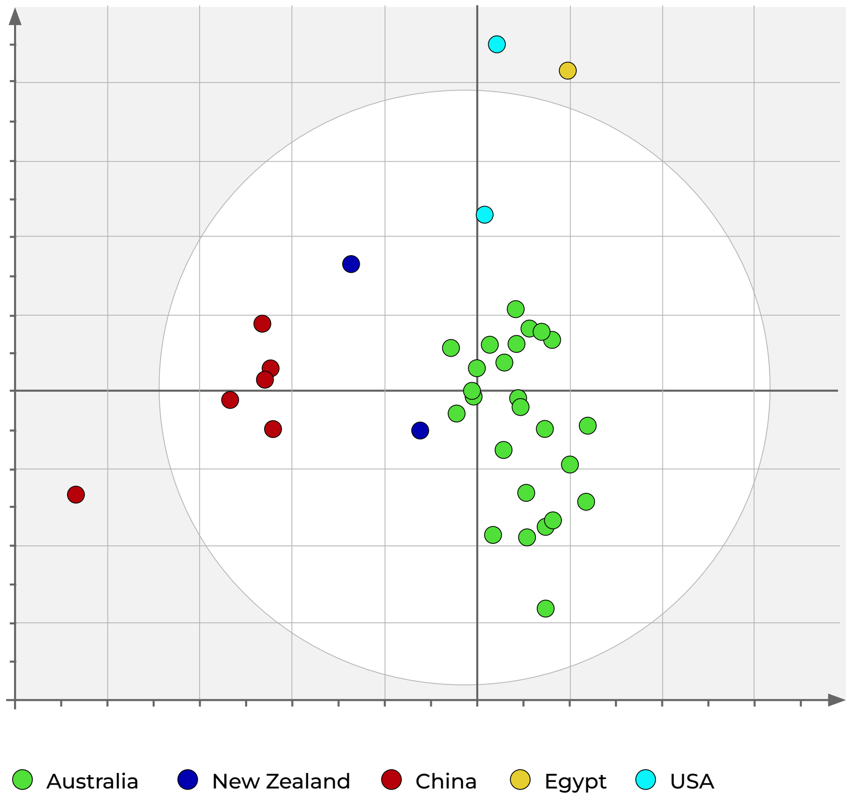
Key trace elements and isotope ratios for characterising Australian oranges based on regional differences are shown in the diagram below, in order of priority.



Australian Country-of-Origin Results

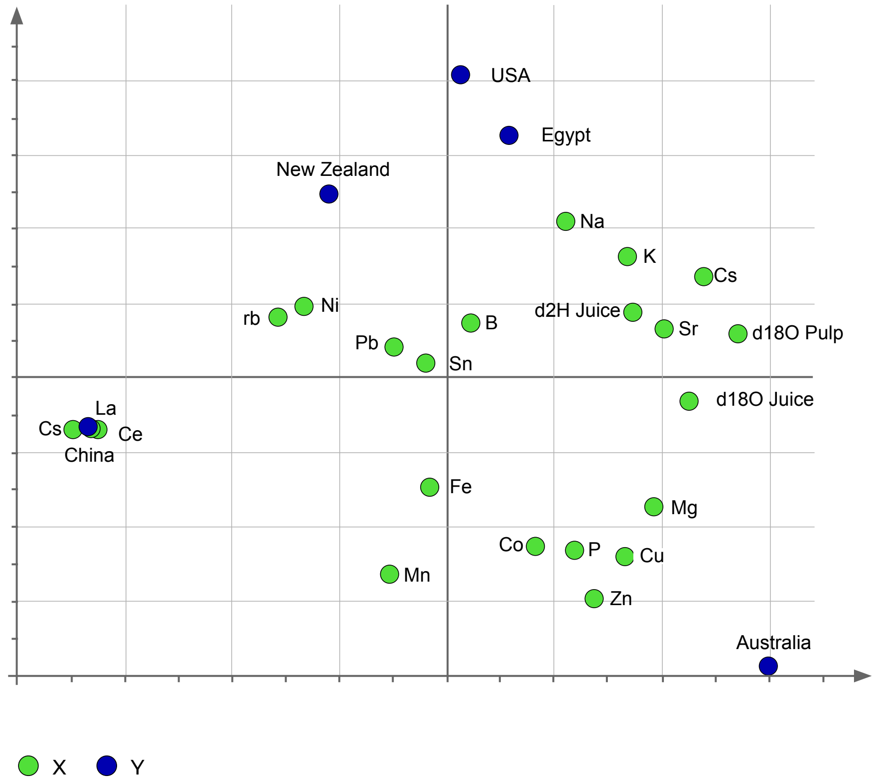
When navel oranges from Australia are compared to four other countries, there is:

- clear separation from other countries, although a USA sample and New Zealand sample plotted close to the Australian cluster.
- clear differentiation from Chinese and Egyptian citrus



Different countries correlate best with different trace elements and isotopes:

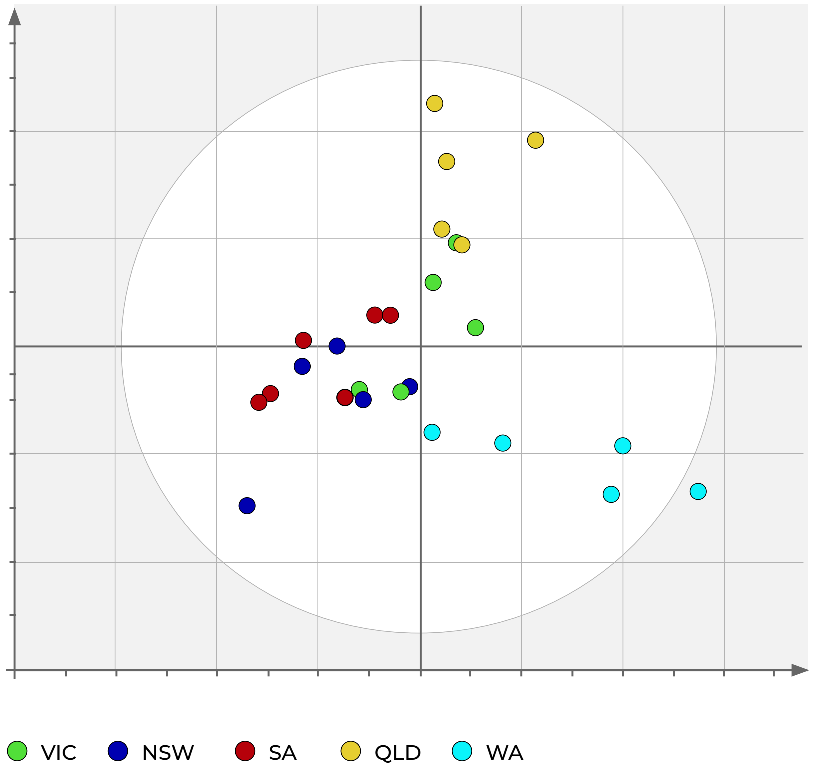
- Australian oranges correlate best with Zn, Cu, Mg, P and Co variables.



Australian Regions Results

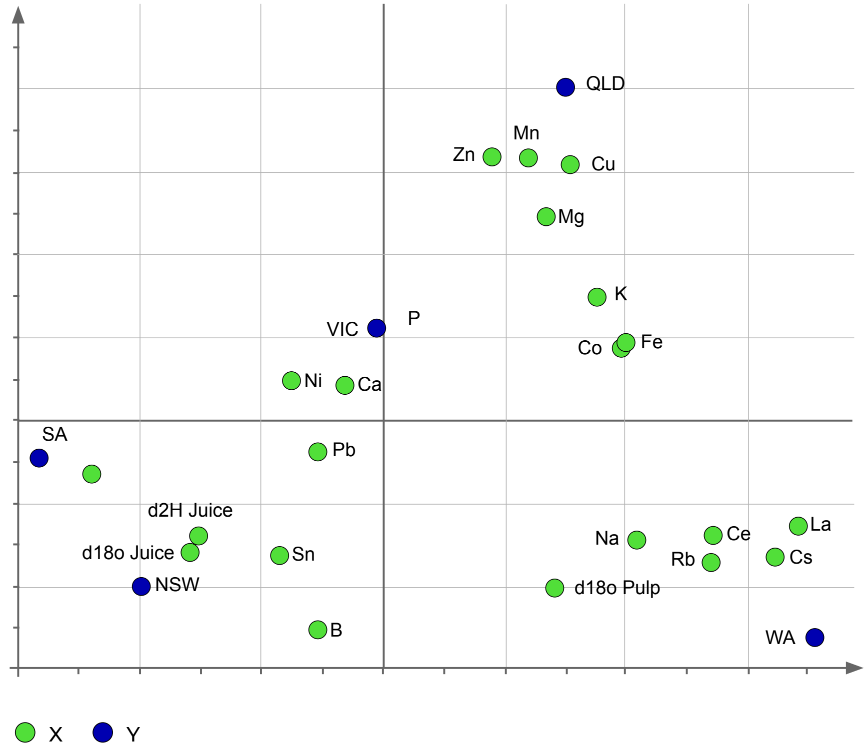
When navel oranges from the five Australian regions are compared there is:

- excellent separation of Queensland and Western Australia samples from the other three regions
- considerable overlap of the New South Wales, South Australia, and the Victoria samples, which is understandable given the close proximity of the farms tested (refer to the previous testing site map).



Different Australian regions correlate best with different trace elements and isotopes:

- Western Australia oranges correlate best with Rb, La, Ce, Cs, Na and δ^{18}Opulp
- Queensland oranges correlate best with Zn, Mn, Cu and Mg
- NSW and South Australia samples correlate best with Sr, δ^{18}Ojuice, δ^{2}Hjuice, Sn and B



Developing a Rapid Screening Method

The methods shown above combine multiple isotope ratio and trace element results to give an accurate result. However, this testing and result development takes considerable time, expertise, and expense.

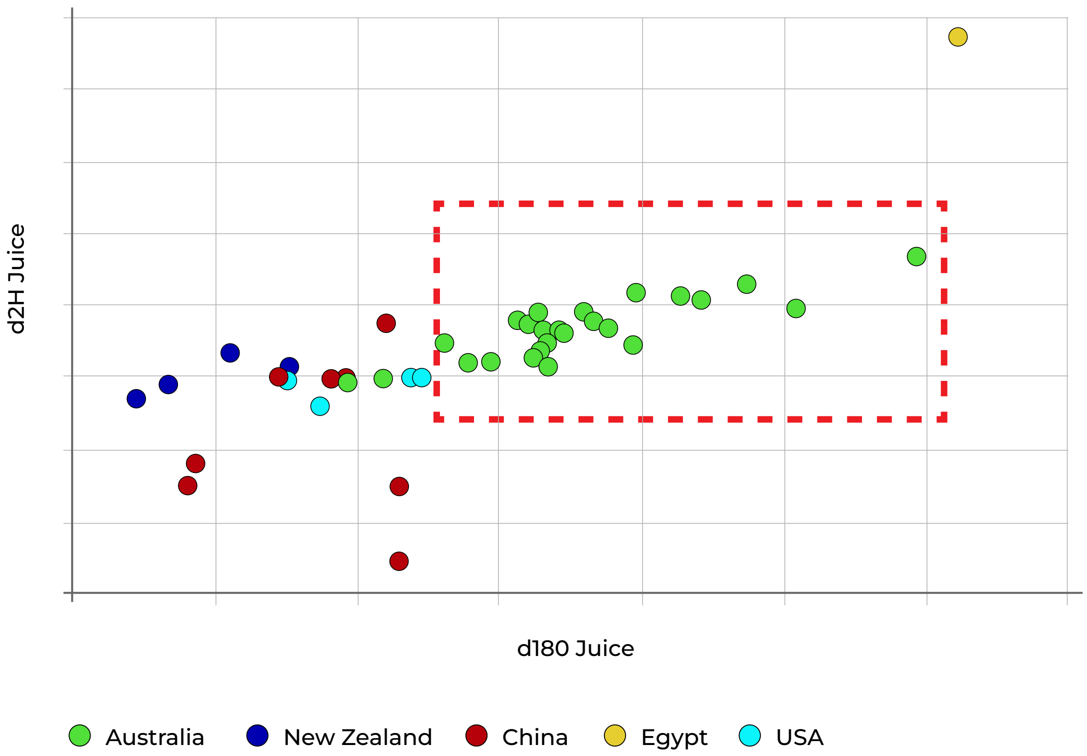
In a crisis, it is helpful to have an additional 'quick, cheap and easy' test to use as a rapid screening method, to evaluate "Are the oranges in question from Australia or not?"

Most of the Australian navel oranges tested have $\delta^{18}O$juice (Oxygen isotope ratios in juice) values which did not overlap with the four countries in this study. Therefore the $\delta^{18}O$juice value of oranges shows potential for use as an ultra-fast, cheap, and easy screening test. The $\delta^{18}O$juice value, when compared to the 'normal range' of this study and future data, could offer the most practical analyte to determine if citrus products are from Australia or not.

If further testing is warranted, then the full isotope and trace element profile testing could be undertaken subsequently.

This offers the potential opportunity of a two-tier testing system:

1. $\delta^{18}O$juice : Rapid, low cost, lower accuracy test
2. Full isotope and trace element profile: Slower, higher cost, higher accuracy test



Conclusions

The outcomes of this study have determined that Australian oranges have distinctive trace element and isotope ratio patterns when compared with the oranges originating from the other countries tested in this study and provides a strong basis for the use of a Country-of-Origin classification technique to protect Australian products in international markets.

Trace element and isotope ratio analyses were undertaken to characterise Australian, New Zealand, Chinese, Egyptian and USA navel oranges and to develop an orthogonal partial least squares discriminant analysis (OPLS-DA) model to best enable Australian Country-of-Origin and Australian Regions discrimination.

Summary table of the classification accuracy of three different OPLS-DA models for Australian Country-of-Origin and Australian Regions discrimination.

Model	Trace Elements only	Isotope Ratios only	Trace Elements and Isotope Ratios combined
Country-of-Origin	91.9 %	79.1 %	97.3 %
Australian Regions	76.9 %	69.2 %	92.3 %

The Australian Country-of-Origin discrimination rate for citrus fruit in this study was high at 97.3% when using a combination of trace elements and isotope ratios.

The high level of Australian Regions discrimination at 92.3% is encouraging for future Australian regional navel orange characterisation.

A key limitation of the Country-of-Origin OPLS-DA model is the limited number of samples from each country beyond Australia. Model accuracy and robustness will improve with further addition of samples to the database including samples from large and diverse countries. Consideration should also be given to investigating other factors that have the potential to produce variability in test results. These include spatial distribution, temporal variations, fruit cultivars, storage times and temperature, and agricultural practices such as fertilisers.

Overall, on a Country-of-Origin basis, the strength of the tight Australian Navel Orange clustering is very distinctive for this large group of Australian fruit, suggesting that clear patterns exist that are unique to Australian oranges. Furthermore, clear differences are also seen in Australian Regions citrus testing.

Recommendations

This preliminary testing program confirms the ability of trace element and isotope ratio patterns to characterise Australia's fruit in international markets. This report demonstrates the usefulness and viability of an OPLS-DA model of trace element and isotope ratios as a traceability tool to protect exported Australian-branded citrus fruit.

Recommendations, in order of priority, for future industry and market testing include:

Further testing to investigate markets at high-risk of citrus substitution (food fraud)

- Further testing should be undertaken on citrus fruit in export markets that receive Australian citrus fruit
- Build on sample numbers to meet robust market testing requirements
- Verify the conclusions in this report with further samples and develop a strong verification program for Australia
- Implement a primary (Country-of-Origin) and secondary (Australian Region) verification system
- Develop an ongoing sampling program of fruit from specific high-risk markets

Build a larger data set for Australia

- Promote traceability of Australian regional citrus fruit and further industry testing to continue to build the Australian citrus database
- Increase testing numbers to a minimum of 10-30 samples from each Australian region to improve regional understanding and ensure more statistically robust data
- Evaluate at least 2-3 years of data to verify any annual climatic effects

Develop a Two-Tier Testing Program

- $\delta^{18}O$juice: Rapid, low cost, lower accuracy test. To determine if fruit is from Australia or not in the early stage of a crisis.
- Full isotope and trace element profile: Slower, higher cost, higher accuracy test. For more detailed follow up.

Data sets for individual growers

- Identify specific growers that may be interested in verification testing and characterisation of their own fruit and offer a service to these growers to include their data within the wider Australian dataset

Build larger data sets for global citrus

- Determine the scope for participation in collaborative citrus traceability studies with global partners or international peak bodies
- Build on the number of other countries tested so that climatic variations as well as geographical gaps may be adequately examined

Further Testing to provide an appropriate confidence level

- Identify subtle or potentially unique differences around irrigation water/precipitation supply and soil characteristics which could refine regions

Evaluation

This project has provided a successful pioneering study to identify Australian oranges at a regional and international level. The project achieved its original aim of delivering an internationally accepted traceability method that addresses 2 key questions:

1. Can we determine if an orange is from Australia?
2. Can we differentiate Australian oranges into one of the 5 Australian growing regions?

The answer to both questions is ‘yes’.

In evaluating the project, the project partners felt this project had been useful and valuable and provided feedback on the following themes:

Further testing

- » As the first priority, Citrus Australia is highly interested in further testing to build a larger dataset and improve robustness of analyses
- » A larger, robust dataset is important to
  - support testing during crises e.g., food safety or food fraud crises regarding oranges in export markets
  - aid investigation of variety or country-of-origin labelling claims on fruit juice
  - demonstrate efficacy to citrus growers to promote future participation
  - support scientific publication of data
  - foster collaboration with other citrus or horticulture peak bodies nationally and internationally
  - develop standardised methodologies and databases
  - bring together fruit, water, and soil testing information to build predictive result maps for Australia and other countries
- » Citrus Australia also sees value in testing different types of Australian fruits/varieties and evaluating how that data compares
  - Navel, Valencia, and mandarins are high value products of key priority
- » Citrus Australia notes that government traceability funding is difficult to obtain nationally and would welcome support for further testing work

Marketing

- Citrus Australia is interested in developing testing opportunities for farmers to characterise their own fruit, thereby strengthening their own brands
- Large grower businesses may be particularly well placed to test their fruit and contribute to the database of national results

Evaluation

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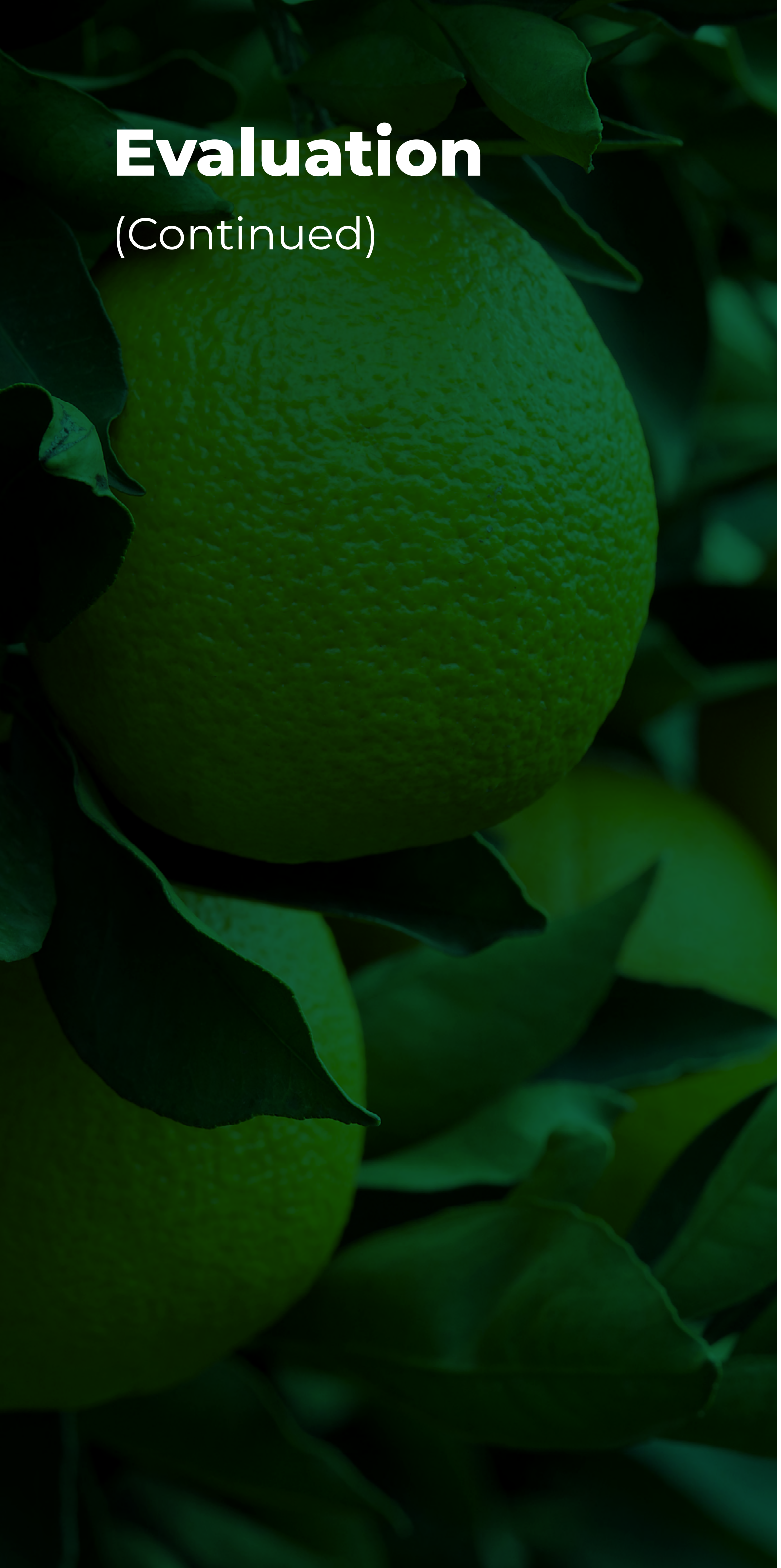

Scientific Publication

- Scientific publication of the full results of this study, together with future larger datasets, would be of great benefit to furthering knowledge in all horticulture sectors and scientific communities globally
- Scientific publication under peer review also supports validation of the methods of this study

National and International Collaboration

To protect the value of brands, and to build internationally robust datasets and standards, Citrus Australia sees potential collaboration opportunities with

- other citrus fruit producers such as the Southern Hemisphere Association of Fresh Fruit Exporters (SHAFFE)
- other Australian horticulture producers, where similar geology, climatic, environmental conditions and agricultural practices may result in similarities in testing procedures and result profiles
- other international laboratories in key export markets; the analyses and work undertaken in this project is fully reproduceable in appropriate competent laboratories as the methods used were standard methods

Uptake Challenges for Isotope Ratio and Trace Element Testing

Despite many studies that demonstrate the usefulness of elemental and isotopic verification systems, there has been very limited uptake of this technology to date by food industries. Challenges include:

- High cost of entry (acquiring a range of suitable samples, background information and building a database)
- Sophisticated data interpretation requirements and variable levels of certainty attainable (accessible to multi-variate and spatial data analysis tools)
- Bespoke technology requiring a high level of expertise for implementation

Evaluation

(Continued)

Uptake Challenges for Isotope Ratio and Trace Element Testing (contined)

- Lack of result and method transparency due to intellectual property arrangements of many laboratories, leading to lack of data ownership by customers, and lack of transferable information between different studies
- Low levels of awareness (acceptance by regulatory authorities and industry)
- Logistical challenges associated with sample procurement across and within multiple countries
- Slower verification time compared to everyday traceability methods such as digital traceability through labelling.

It is hoped that this report and the check list for laboratory testing (provided within the appendix) will provide useful information and guidance to industries seeking to undertake isotope ratio and trace element traceability programs.

Intellectual Property of Isotope Ratio and Trace Element Testing Results

Note that this report is a summarised high-level public report, with the full result details currently remaining the unpublished, confidential property of Citrus Australia.

For anyone wishing to undertake an isotope ratio and trace element project, consider if you wish to keep full study results confidential to your industry / share them with other laboratories / share them with other peak industry bodies globally / openly publish full results in scientific journals. It is very important to establish who will have ownership of the full results of your study: your industry, or the laboratory. It is also important to understand the methods used by the laboratory and if these can be shared openly with other laboratories, industry groups and the scientific community. You are advised to ask laboratories specifically about the intellectual property of the testing methods and project results. This has implications for how you can access, retain, transfer, share and build upon the data beyond the life of the project.

Project Partners

CITRUS AUSTRALIA

Citrus Australia is the peak industry body representing the nation's $850 Million citrus industry.

Citrus Australia, as the peak industry body, has an established program of successful projects across biosecurity, communications, market access development, R&D coordination, and public affairs. It is a not-for-profit company It manages funding through grants and levies in partnership with other organisations including Plant Health Australia and Horticulture Innovation.

Key Project Personnel:
Nathan Hancock, Mara Milner

AGRICULTURE VICTORIA

Agriculture Victoria is the state organisation working in partnership with farmers, industry, and communities to grow and secure agriculture in Victoria.

Agriculture Victoria has a strong track record of innovation and export development programs across many commodity sectors. Discover more about Agriculture Victoria's traceability program here.

Key Project Personnel:
Stacey Barlow, Caroline Barrett

GNS SCIENCE

GNS Science, Te Pū Ao, (Institute of Geological and Nuclear Sciences Limited) is a New Zealand Crown Research Institute and foremost geological and environmental research organisation and leading provider of Earth, geoscience and isotope research and consultancy services. GNS Science provides specialist scientific services via consultancy and analytical services in a diverse range of areas.

Key Project Personnel:
Karyne Rogers

Appendix 1:

Laboratory Traceability: A Check List for Trace Element and Isotope Ratios Testing

Questions for laboratories during the quotation process

Question	Answer
Laboratory	
Provide the address of your laboratory / laboratories. If samples will be 'received' and 'tested' in different laboratories, provide addresses of all of them.	
Are all of the above laboratories NATA accredited (or have other relevant accreditations)?	
Provide Laboratory Submission Form(s)	
Provide a copy of any import permits for samples submitted from all states of Australia (or your country)	
Can your laboratory accept the following sample types from all states of Australia (or your country)?	
• Whole fruit?	
• Pulp?	
• Fresh juice?	
• Pasteurised juice?	
• Irrigation water?	
• Soil?	
• Any other sample type?	
Customs regulations for samples	
Provide customs forms for all relevant sample types	
Sample Handling	
Provide a **sampling protocol** that can be issued to each sample collector to assist with sampling consistency	
Provide sample pot, storage, packaging and transit instructions	
If you will supply any sampling or packaging materials, provide details and costs	
Provide details of method of sample transit from all states of Australia to your laboratory / laboratories e.g. mail, courier etc	
If you will provide transit of sample materials (e.g. arrangement of couriers), provide costings for transit of sample materials	
Testing Plan & Pricing Quotation	
Provide a **test plan**	
Provide a **pricing quotation** for isotope and/or trace element testing.	
Include any costings relating to forwarding of samples between laboratories.	
Turnaround Times	
List your total Turnaround Times for results (including any time for forwarding samples between laboratories)	
Final Results	
Outline which testing methods will be used	
Provide example sheets of final results, including imagery to illustrate method analysis, graphs / plots and confidence intervals for horticultural testing	
Results Ownership and Transferability	
Will you provide fully transparent method, units, numerical and graphical results?	
Will these results be provided in sufficiently transparent detail that they could be replicated by other laboratories?	
Do you have any intellectual property arrangements that prevent fully transparent sharing of the methods and results?	
Who will own the fully transparent results at the end of the testing process?	
If the relationship between our businesses ends, is the information generated by this testing process fully transferable to other laboratories as a data set?	
Scientific Advice and Results Interpretation	
Provide name and contact details of scientist who will provide ongoing project advice and results interpretation	
If the cost of advice and interpretation is not included within the testing costs, provide costings	
Examples of Previous Studies	
Provide examples of previous studies, including final results, imagery to illustrate method analysis, graphs / plots and confidence intervals	
Provide examples of published results, preferably horticulture studies in peer-reviewed journals	
Results Publication	
Confirm that you are in agreement with open publication of the study results	

Appendix 1:
(Continued)

Information to provide to laboratories during the quotation process

Provide a background to your industry
List the reasons why you wish to undertake laboratory traceability
Provide a basic map of your growing regions
Provide basic statistics about the number of orchards per region
Provide basic statistics about production volume per region
Describe the variety of fruit you wish to test
Outline your harvest season timings

Test Plan and Pricing Quotation Outline

Request the laboratory to provide the minimum testing protocol that will answer these questions in a crisis event:

1. Is this product from my country, yes or no?
2. If yes, which growing region of my country is this product from?
3. If required, which farm in my country is this product from?

	Cost per sample	Growing region 1	Growing region 2 etc	Competitor Country 1	Competitor Country 2 etc	Total
Number of Samples						
Isotope Ratio: Sample preparation						
Isotope Ratio: Isotope 1						
Isotope Ratio: Isotope 2 etc						
Trace Element: Sample preparation						
Trace Element: Element 1						
Trace Element: Element 2 etc						
Sampling Materials e.g. pots, chiller pads, polystyrene boxes etc						
Transit Costs e.g. initial transit to laboratory, or transit between laboratories						
Scientific Interpretation and Reporting Costs						
GRAND TOTAL						

Sampling Protocol

1. See example earlier in the document
2. Include instructions on
    a. equipment e.g., juicing method
    b. storage of the samples in transit
    c. documentation: sample submission forms, import permit and quarantine requirements
    d. Transport containers, packing materials and transit method