To evaluate the use of the Isotope Ratio Mass Spectrometry (IRMS) as a tool to Authenticate South African wines

C Small
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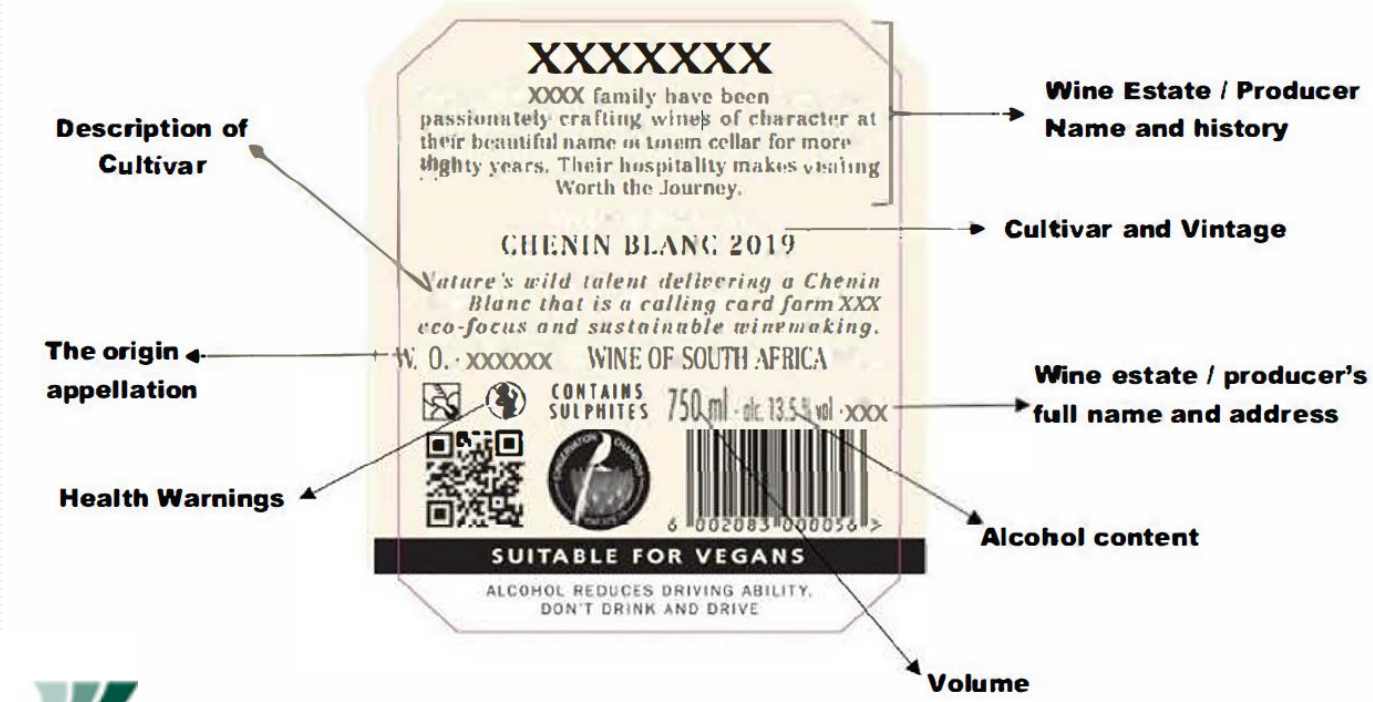
What is Wine Authentication?

- Process where various chemical markers can be evaluated and used to establish a fingerprint for identification.
- Can be described as process to certify that a wine belongs to a certain geographical region, as well as to verify the legitimacy of product as labelled.



Importance of Wine Authentication

- The EU views authentication as a cornerstone of its agricultural policy.
- According to the EU its defined as a system of controls and requirements within an analytical framework to:

  - Maintain consumer trust and product integrity
  - Support economic fairness
  - Protect geographical origin.

- Relevance to SA?
  - Regulatory framework and trade agreements are of immense importance, considering EU is the top destination for South African wine exports.

How do you Authenticate wine?

Step 1	Step 2	Step 3
Analyze unknown wine sample to obtain the $\delta^{13}C$	*Compared $\delta^{13}C$ to established reference values (databank)*	*If the chemical marker falls within reference ranges, the product is deemed "authentic and compliant to label claims"*

- Example of a key marker that contributes to the overall chemical fingerprint of South African wines = $\delta^{13}C$ values
- Laboratories with proven Analytical Methods, comparable to the OIV methods should be used for authentication tests.

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Cost Implication

Authentication analysis mostly done abroad. Very expensive!!!

A local laboratory was identified with suitable technology and its methods were optimized and verified.

Biogrip Node/ CAF University Stellenbosch

Benefit of local facility:

Analysis cost is reduced significantly & TAT is improved as samples can reach the laboratory faster.

Aim

- This study aims to evaluate the efficiency of stable $\delta^{13}C$ isotope ratio analysis by IRMS as a technique to authenticate South African wines, while accounting for natural variations caused by botanical origin, geographic region, and vintage.

Objectives

- To extract ethanol with alcohol grade >95% and analyze the stable $\delta^{13}C$ isotope ratio of the extracted ethanol using the EA IRMS.

- To establish reference ranges (Library) for $\delta^{13}C$ isotope ratios in South Africa for the purpose of geographical and botanical authentication of South African wines.

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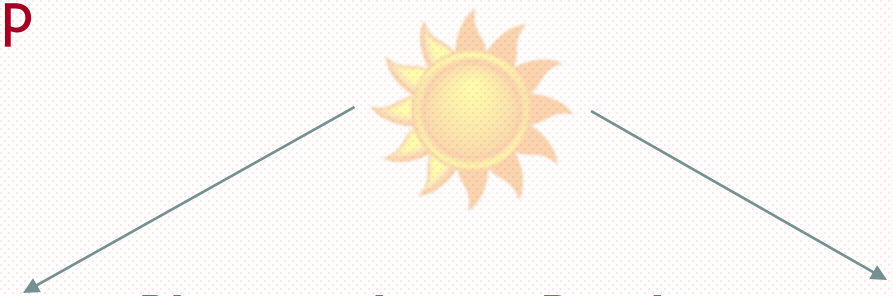
$\delta^{13}C$ Formation and Chemistry

- During Photosynthesis, carbon fixation (*inorganic CO2 converted to organic carbon*) can occur through:
  - C3 metabolism (Calvyn Cycle) OR C4 metabolism
  - The $\delta^{13}C$ produced through each metabolic process varies significantly making it easy to identify glucose plant origin.

C3 -group

Grape vine



Photosynthesis _ Resulting in unique $\delta^{13}C$
Glucose = Alcohol

C4-group

Sugar Cane

Corn

Sampling Plan

- *500 samples ; 3vintages; 13 wine districts ; 10 different cultivars*

2023

50 Micro vinified

50 Commercial

2024

100 Micro vinified

100 Commercial

2025

100 Micro vinified

100 Commercial

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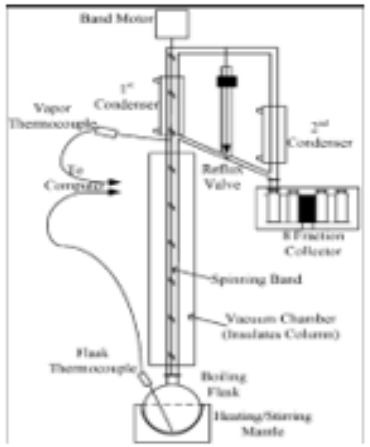
Method to be evaluated in this study

- *Fractional Distillation (Spinning band distillation)_ Sample preparation*
- *EA-IRMS (δ¹³C analysis)*

Micro vinification

Distillation



Harvesting
Grapes
10 different cultivars
13 Wine Districts

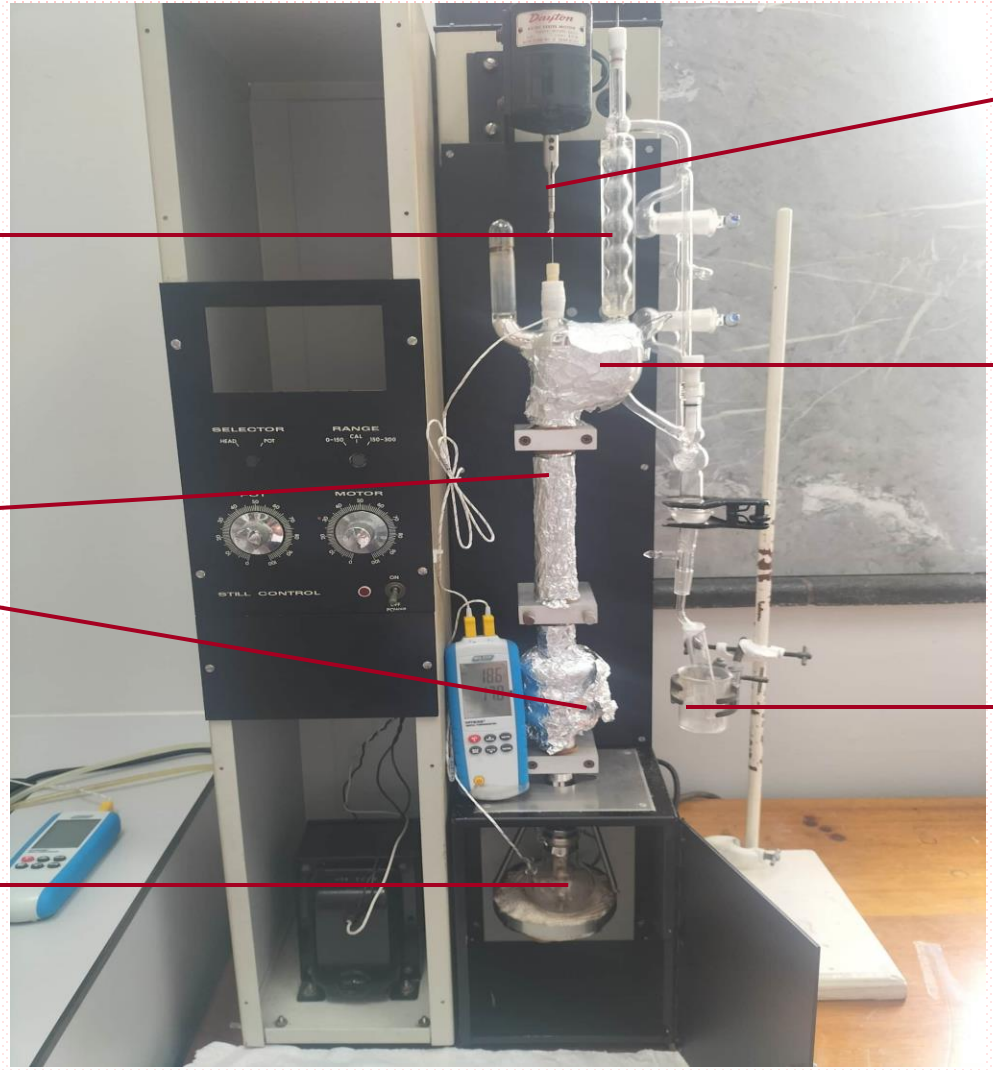
EA-IRMS
δ¹³C analysis

Methodology

Sample Preparation Technique: Spinning band Distillation

- Preferred OIV extraction method for ethanol from wine. Micro distillation i.e 60ml wine
- During fractional distillation, allowing separation of components. Ethanol vapor temperature is monitored and distillate collected at Ethanol's boiling point of 78.4°C ± 0.3°C
- The collected distillate alcohol grade >95%v/v in single distillation.

Spinning Band Distillation



Helical Teflon tube spinning

Condenser tube

Distillate vapour – Temp monitoring

Fractionating column

Ethanol distillate collection vial

60ml Wine sample on heating mantel

Methodology

Alcohol strength measurement

According to OIV methods - Primary methods:

- Pycnometry
- Electronic densimetry
- Hydrostatic balance

→ Not suitable for small sample size

Methodology

Alcohol Strength measurement

REFRACTOMETER (***Affordable***)

- Both suitable for small sample size.
- Data for samples analysed on both methods compared well

HPLC (***Expensive***)

According to OIV methods secondary method:

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Methodology

EA-IRMS Carbon stable isotope analysis $\delta^{13}C$

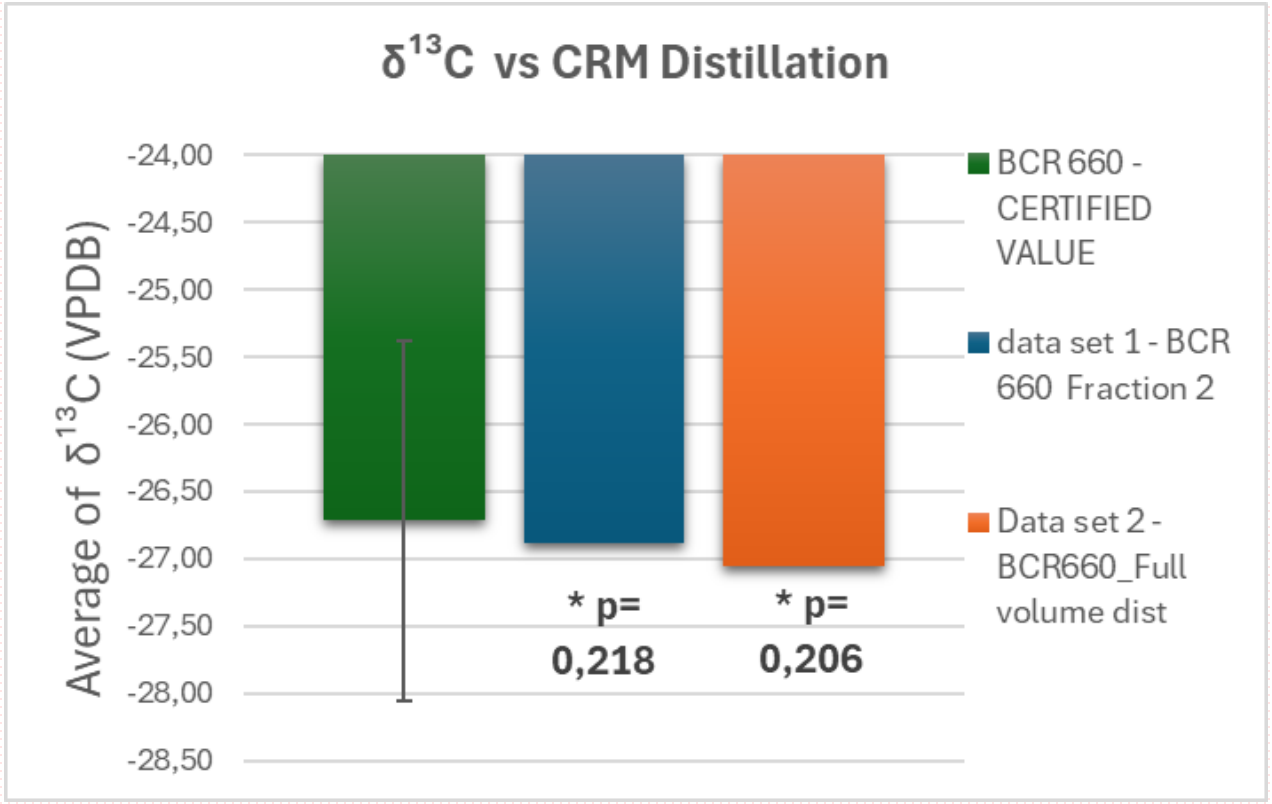
- 200µl distillate to be injected

Samples to be quantified against international calibration standards.

Method Optimization

- Distillation, and stable carbon isotope ratio analysis (δ^{13}C-IRMS)
  - Validation with International Certified Reference BCR660

P ≥ 0.05
No significant difference between BCR660 certified value standard and distilled & analysed BCR660 standard

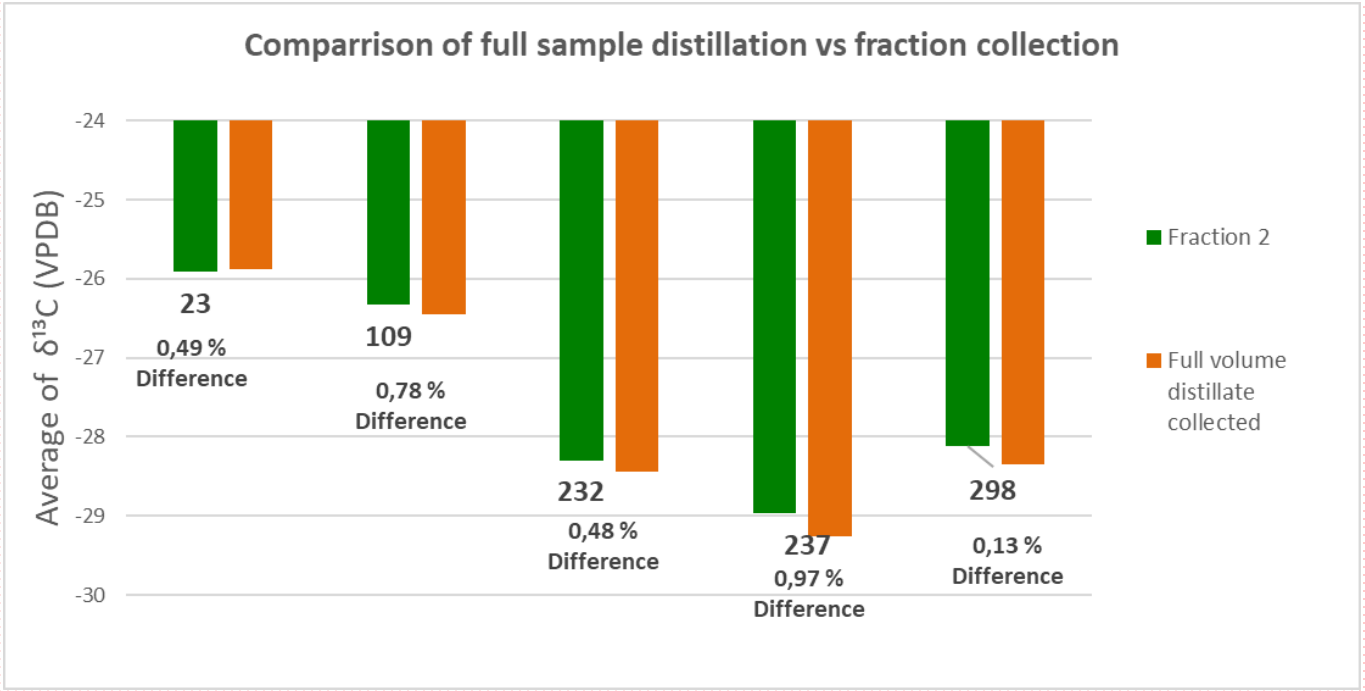


Method Optimization

- Ethanol Extraction via Spinning band distillation
  - To distill >98% total alcohol from sample = +- 2hours /sample
  - To distill representative fraction = +- 45 minutes

% Difference <1%

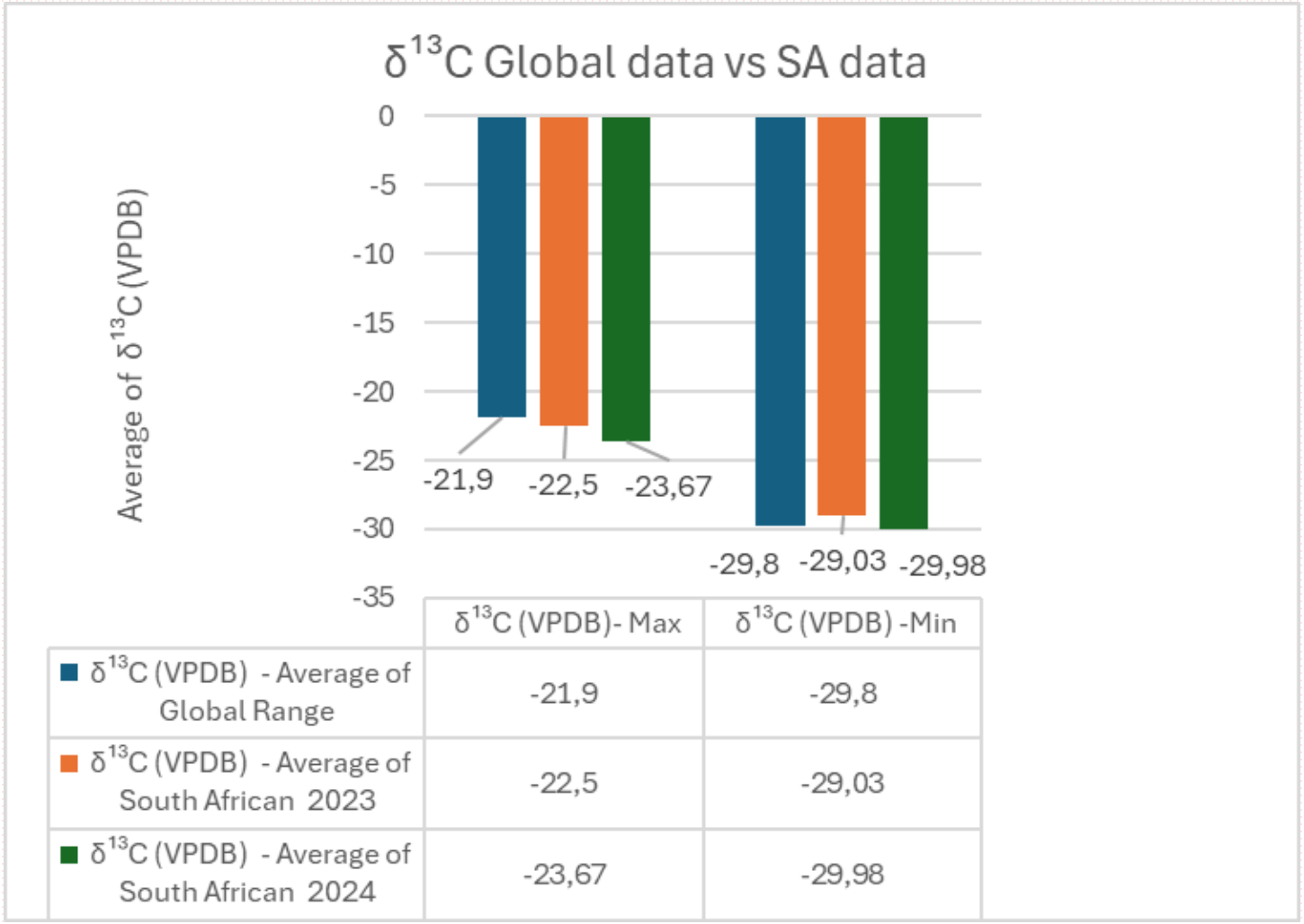
No significant difference in $\delta^{13}C$ between full volume distillate collected and fraction collection



Results & Discussion

- 2023 – 100 samples
- 2024 – 200 samples

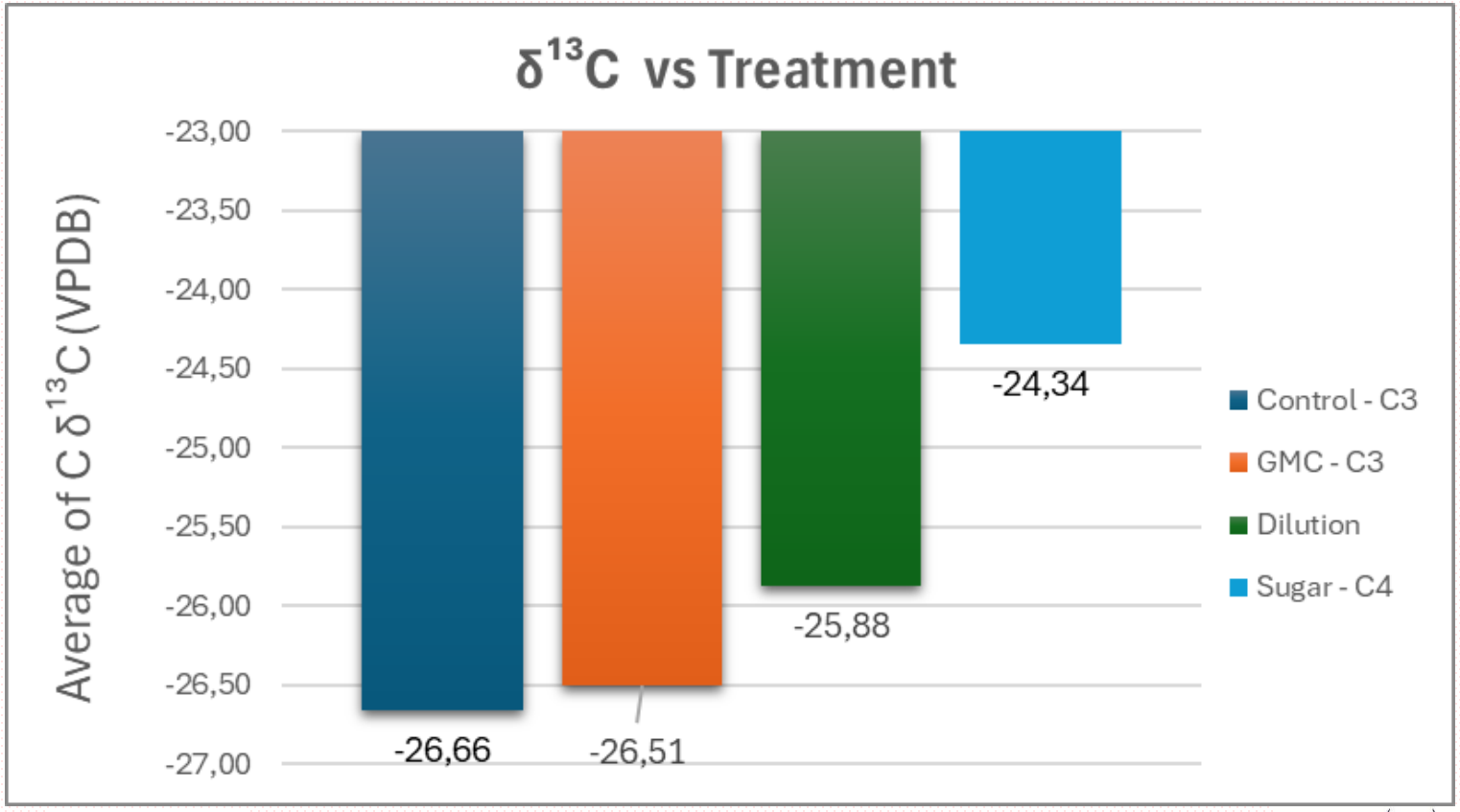
Both SA 2023 &2024 data fit within the normal global range



	δ13C (VPDB)- Max	δ13C (VPDB) -Min
δ13C (VPDB) - Average of Global Range	-21,9	-29,8
δ13C (VPDB) - Average of South African 2023	-22,5	-29,03
δ13C (VPDB) - Average of South African 2024	-23,67	-29,98

Results

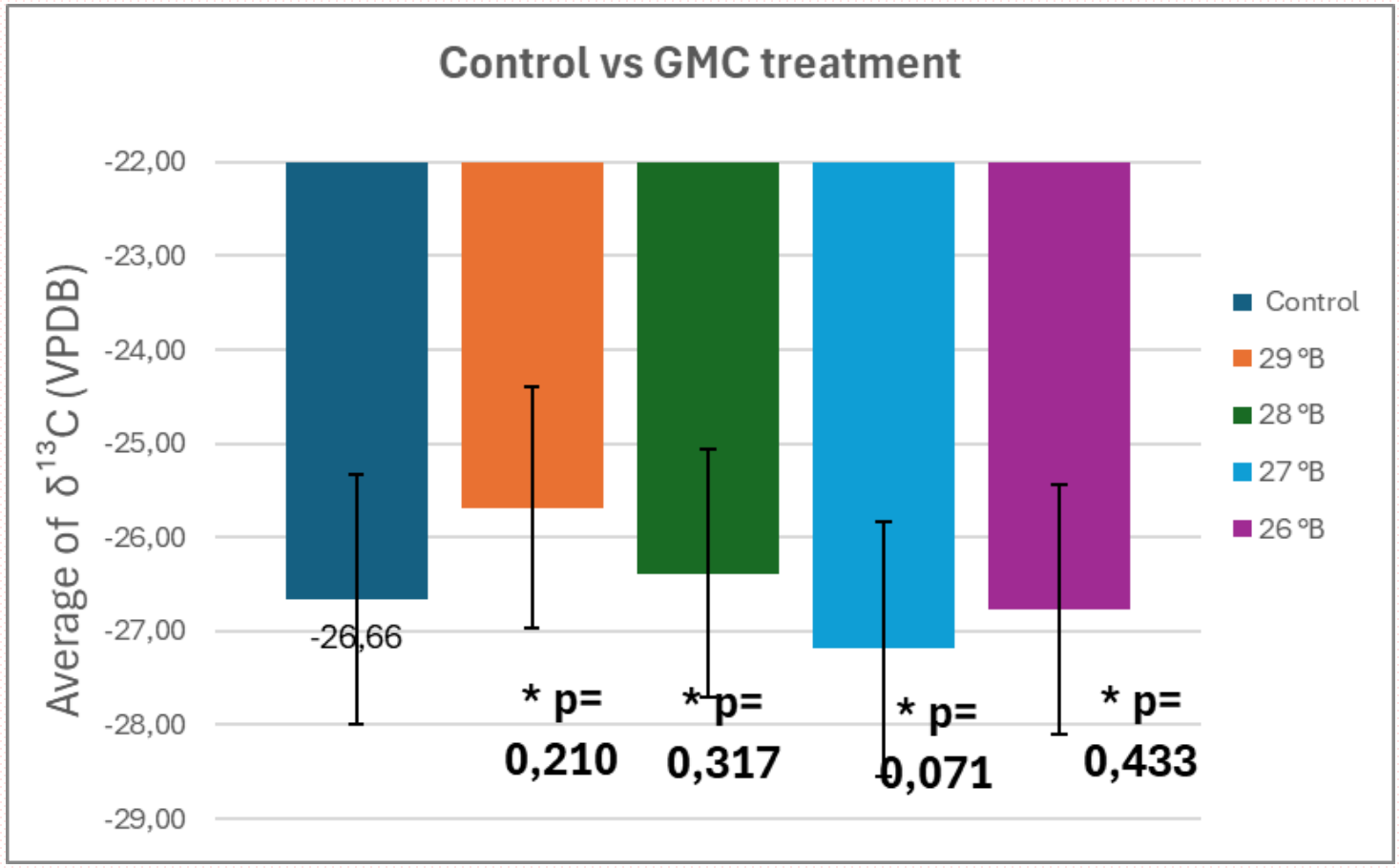
- Adulteration experiment_ Intentionally modified - 3 different treatments.



Results

- Variation of Grape Must concentration treatment

P > 0.05
No significant difference between Control and treated GMC

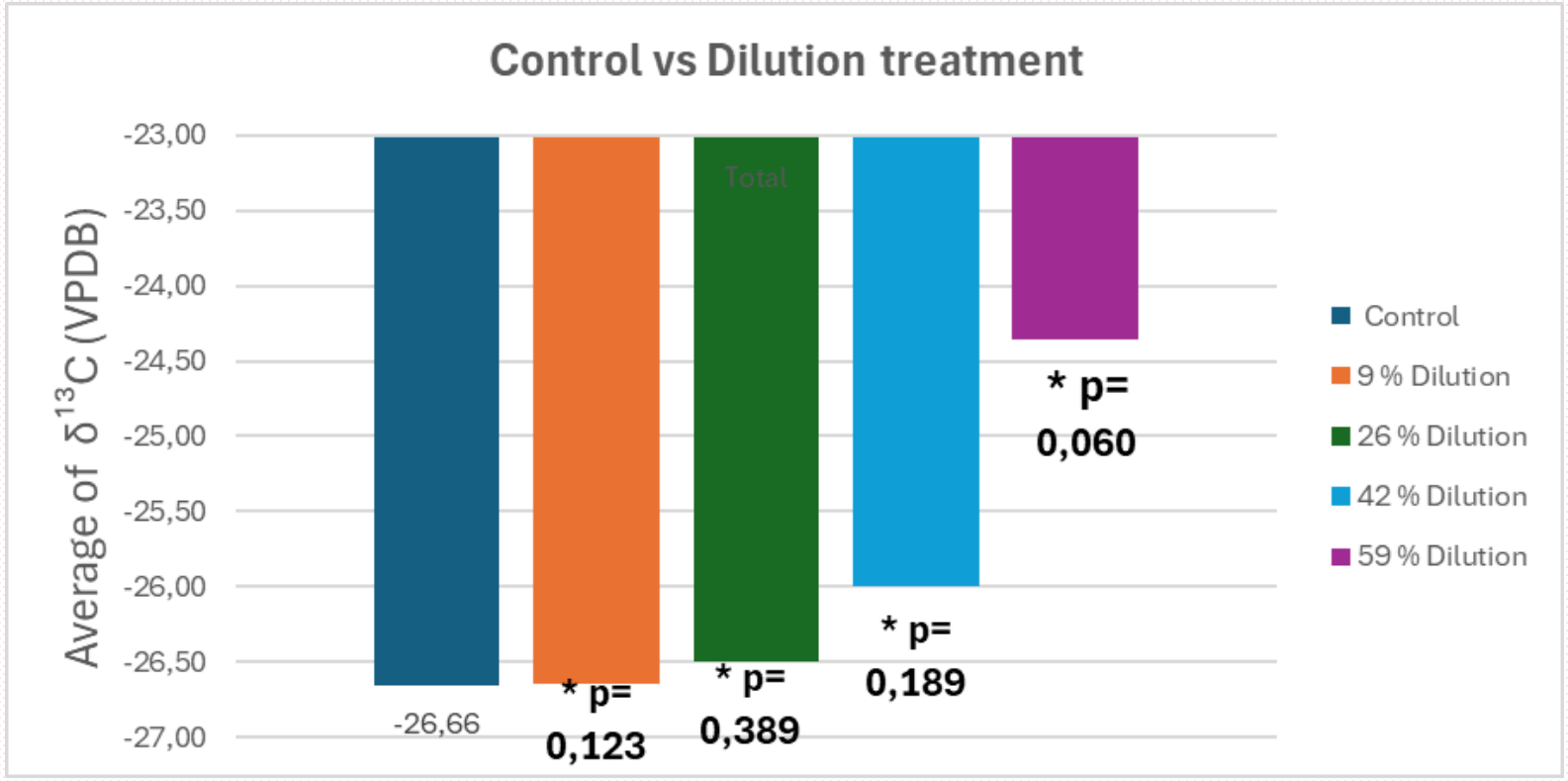


Results

- Variation of water dilution treatment

P > 0.05
No significant difference between diluted samples

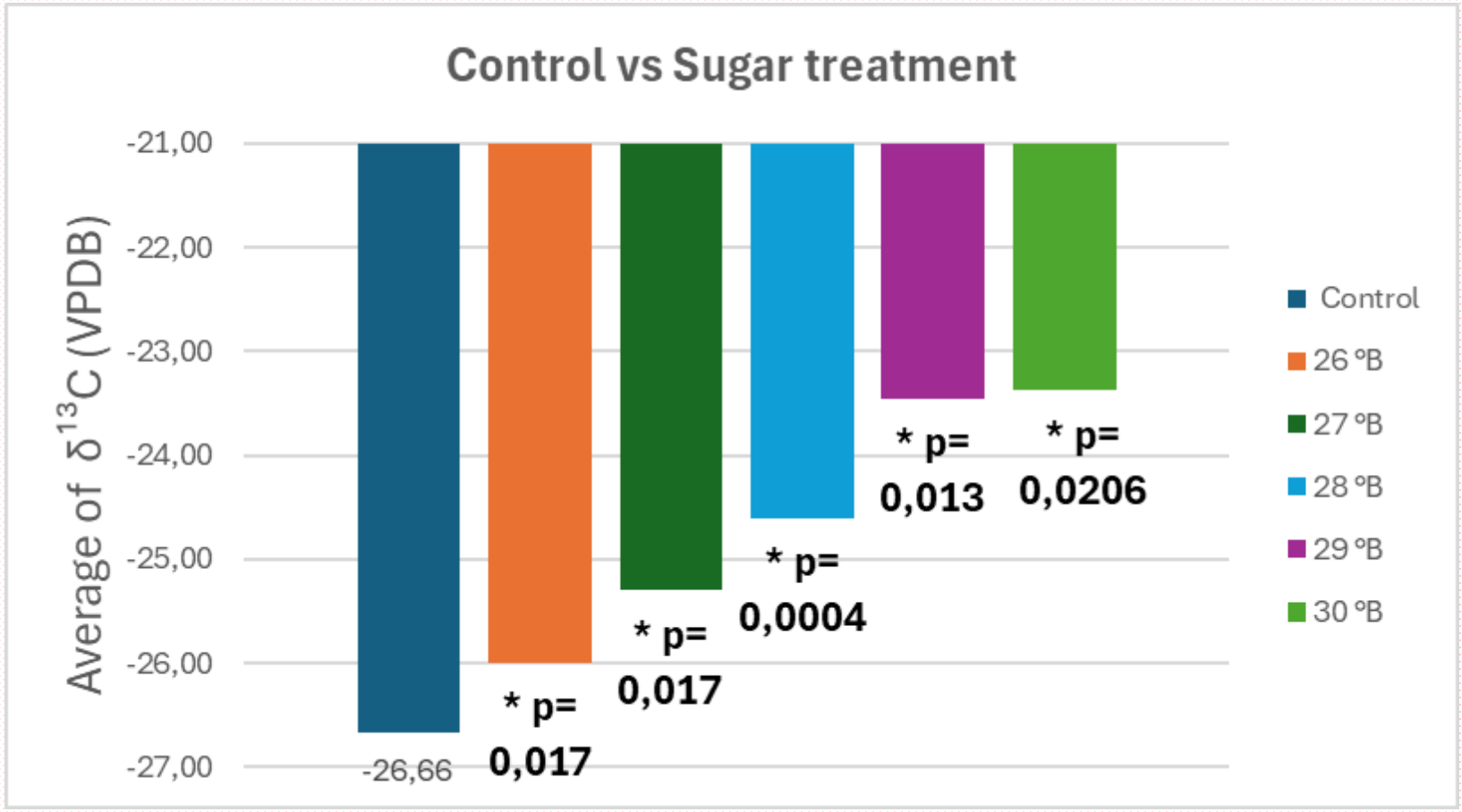
However, 59% dilution on border of significance



Results

- Variation of C4-plant - Sugar addition treatment

P < 0.05 Significant difference between control and modified samples



Conclusion

- 2025 Vintage - 200 samples data to be added to expand dataset for geographical origin evaluation.
- Both wine distillation and IRMS methods shows reproducible and accurate results providing confidence in the data.
- Method shows the ability to detect modified wines - specifically on sugar C4 additions. Limitations in showing variation in cultivars.
- A local laboratory is already established with a validated method for routine $\delta^{13}C$ analysis to be used in Wine authentication processes.

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