



Comparison of Individual and Group TCA Analysis in Natural Cork Stoppers

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Introduction

Assessing the distribution of 2,4,6-Trichloroanisole (TCA) in batches of natural cork stoppers remains a critical challenge for the cork and wine industry. The conventional method for quantifying TCA, outlined in ISO 20752 [1], involves gas chromatographic analysis of a cork soak. As this approach is time-consuming, costly, and destructive, TCA distribution is often estimated using pooled cork samples - so-called cork group soaks. However, the reliability of this procedure is questionable.

This study addresses the issue by comparing TCA distribution results obtained from cork group soaks with those from individual cork analysis across seven batches. Due to the high number of measurements required (approximately 3,500 cork stoppers), a method emerging as the new gold standard for TCA analysis was employed: the Vocus Cork Analyzer (VCA). This system uses chemical ionization – time-of-flight mass spectrometry to assess TCA in individual corks in just two seconds [2,3]. The technique is non-destructive, allowing cork stoppers to be sold after analysis by the VCA.

Conclusion

This study demonstrated that analyzing TCA in individual cork stoppers provides a significantly more reliable assessment of TCA distribution in natural cork batches compared to group soak analysis by ISO 20752. The latter can often yield misleading indications of TCA presence in cork batches, potentially leading to inappropriate batch rejection or acceptance.

For instance, we showed that two batches with a similar percentage of corks containing releasable TCA concentrations above 1.5 ng/L could produce conflicting results when assessed by group soaks. In such cases, the ISO method may suggest the presence of cork groups above the threshold in one batch but not in the other, despite similar actual distributions.

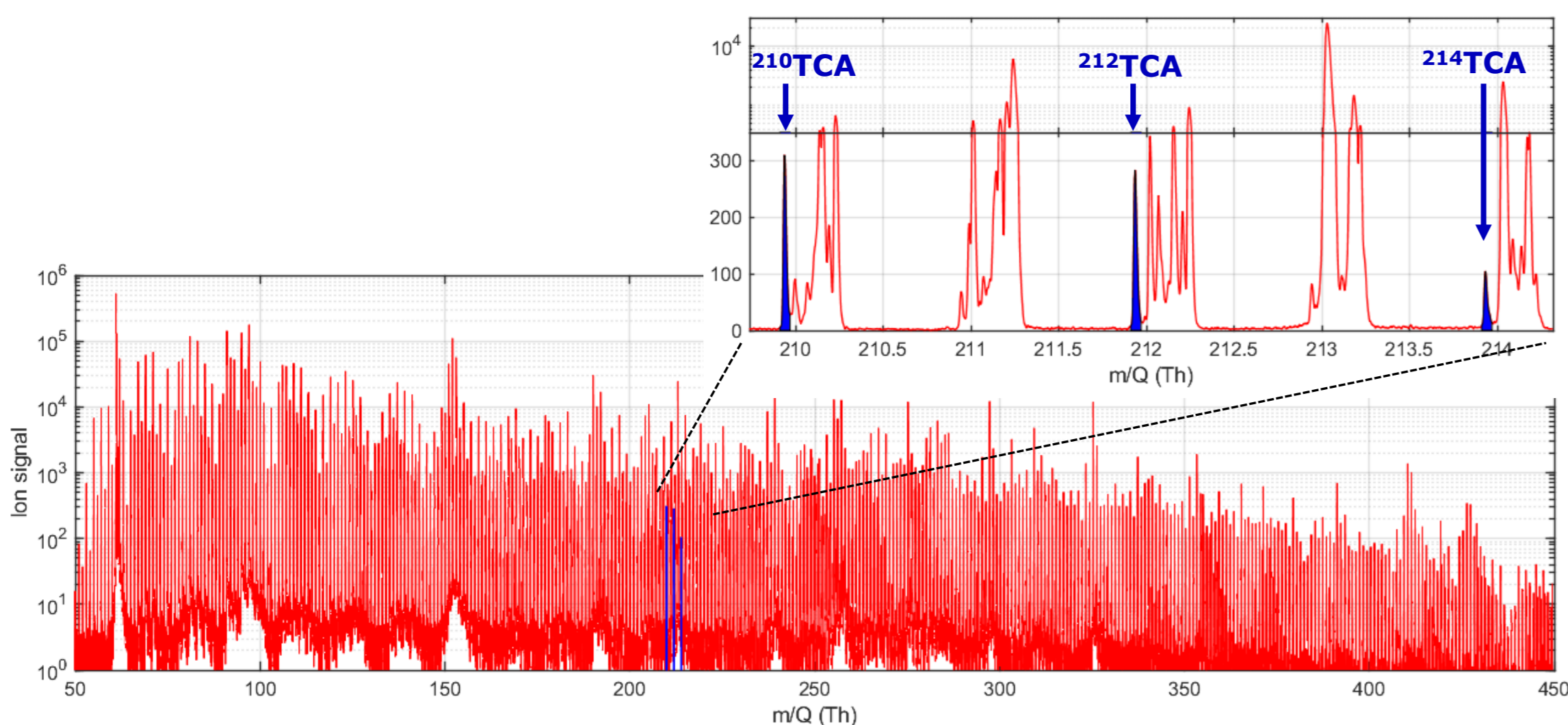
We confirm that the Vocus Cork Analyzer enables rapid, non-destructive TCA measurement - just two seconds per cork - thus allowing for accurate and efficient assessment of TCA distribution in entire batches within minutes.

Technology

Vocus Cork Analyzer

Chemical ionization coupled to high resolution mass spectrometry allows non-destructive detection of 2,4,6-Trichloroanisole (and all other haloanisoles and halophenoles) in natural cork stoppers with limits of detection below the sensory threshold. Cork stoppers are heated without causing any damage or structural changes and the thermally released TCA is measured in real-time.

Below: A heated cork mass spectrum. Measured TCA isotopes are in blue. TCA is a tiny fraction of total VOC emission from the cork. High-resolution MS spectrum acquired in 2 s.



VCA beyond ISO 20727



Vocus Cork Analyzer is now established in major natural cork stopper companies as the election industrial solution for individual TCA screening of TCA-free natural cork stoppers.

Right: Performance comparison of VCA and ISO 20752 for TCA analysis of individual natural cork stoppers.

COMPARISON OF METHODS	Standard Method HS-SPME-GCMS ISO 20752:2023	VOCUS CORK ANALYZER
Type	Destructive	Non-destructive
Analysis time	45 min	2 s
Multitarget	Not simultaneous	Simultaneous
Mass resolution	1	> 10,000 Th/Th
Isotopic ratio	No	Yes
Exact Mass	No	Yes (~ 1ppm accuracy)
Sample introduction	Preconcentration, 30 min	Direct, real-time
TCA LOD (ng/L)	0.50	0.05
TCA LOQ (ng/L)	0.80	0.15
Ext. Uncertainty (k=2; 0.0 – 1.0 ng/L)	96% (cork)	52% (cork)
TCA False positives/negatives	Yes	No
Throughput per year	Up to 10k tests	10 M corks

Vocus Cork Analyzer (VCA) Compared to Cork Group Soaks

CORK GROUP SOAKS

TCA analysis with ISO method of 30 groups of 50 corks each in seven cork sets (A, B,..., F)

CORK SET	A	B	C	D	E	F
<0.5 ng/L	24	28	29	29	29	29
0.5-1.0 ng/L	4	1	1	1	0	0
1.0-1.5 ng/L	2	0	0	0	0	1
>1.5 ng/L	0	1	0	0	1	0
Total (n. groups)	30	30	30	30	30	30

VOCUS CORK ANALYZER: Individual TCA analysis of about 500 corks each in the same seven cork sets (A, B,..., F).

CORK SET A			
	Counts	%	Max TCA
<0.5 ng/L*	418	90.1 %	
0.5-1 ng/L*	34	7.3 %	
1-1.5 ng/L*	4	0.9 %	
>1.5 ng/L*	8	1.7 %	96.6 ng/L*
Total	464	100 %	96.6 ng/L*
CORK SET D			
	Counts	%	Max TCA
<0.5 ng/L*	465	93.9 %	
0.5-1 ng/L*	21	4.2 %	
1-1.5 ng/L*	5	1.0 %	
>1.5 ng/L*	4	0.8 %	23.7 ng/L*
Total	495	100 %	23.7 ng/L*

CORK SET B			
	Counts	%	Max TCA
<0.5 ng/L*	479	94.1 %	
0.5-1 ng/L*	18	3.5 %	
1-1.5 ng/L*	4	0.8 %	
>1.5 ng/L*	8	1.6 %	12.2 ng/L*
Total	509	100 %	12.2 ng/L*
CORK SET E			
	Counts	%	Max TCA
<0.5 ng/L*	464	91.9 %	
0.5-1 ng/L*	16	3.2 %	
1-1.5 ng/L*	9	1.8 %	
>1.5 ng/L*	16	3.2 %	16.1 ng/L*
Total	505	100 %	16.1 ng/L*

CORK SET C			
	Counts	%	Max TCA
<0.5 ng/L*	492	98.4 %	
0.5-1 ng/L*	4	0.8 %	
1-1.5 ng/L*	1	0.2 %	
>1.5 ng/L*	3	0.6 %	13.4 ng/L*
Total	500	100 %	13.4 ng/L*
CORK SET F			
	Counts	%	Max TCA
<0.5 ng/L*	490	98.8 %	
0.5-1 ng/L*	3	0.6 %	
1-1.5 ng/L*	0	0.0 %	
>1.5 ng/L*	3	0.6 %	4.8 ng/L*
Total	496	100	4.8 ng/L*

References

- [1] ISO 20752, Cork stoppers - Determination of releasable 2,4,6-trichloroanisole (TCA)
- [2] Cappellin et al.; Analytical Chemistry, 2020
- [3] Hutterli et al.; ACS Food Sci. Technol., 2024



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