

# Demystifying Cork Quality Assurance



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ETS Laboratories

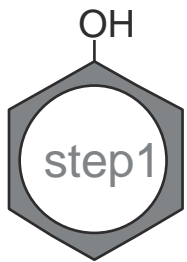
# Demystifying Cork Quality Assurance

1. What is Releasable TCA (RTCA)
2. RTCA from Group Soaks versus TCA in Bottled Wine
3. Group Cork Soaks Applied

# Haloanisoles

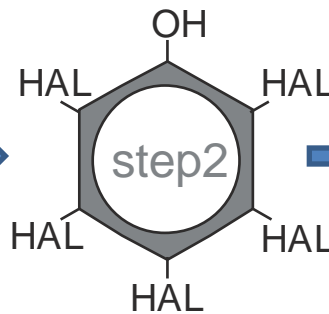
- ETS Laboratory's primary analysis for the cork industry
- Volatile off-aromas with low sensory threshold
- Multiple compounds with numerous sources

phenol



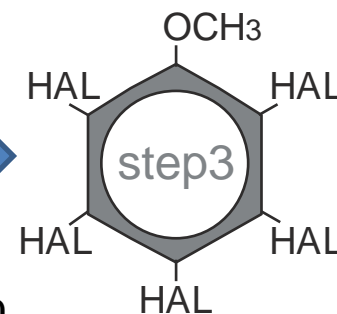
Halogenation

halophenol



Methylation

haloanisole



# Cork Taint

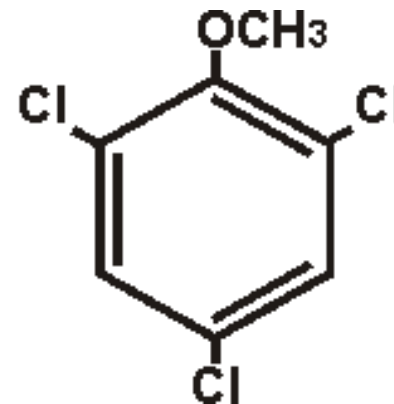
## Wine Related Haloanisoles

- Pentachloroanisole
- Tetrachloroanisole
- Trichloroanisole
- Tribromoanisole



## Cork Related Haloanisoles

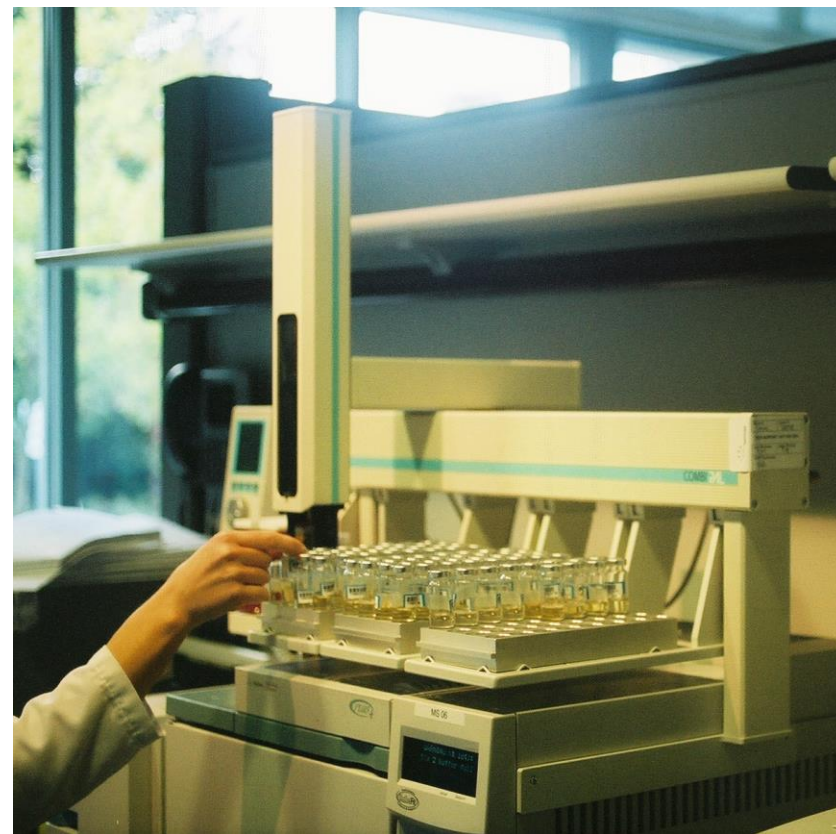
2,4,6-Trichloroanisole (TCA)



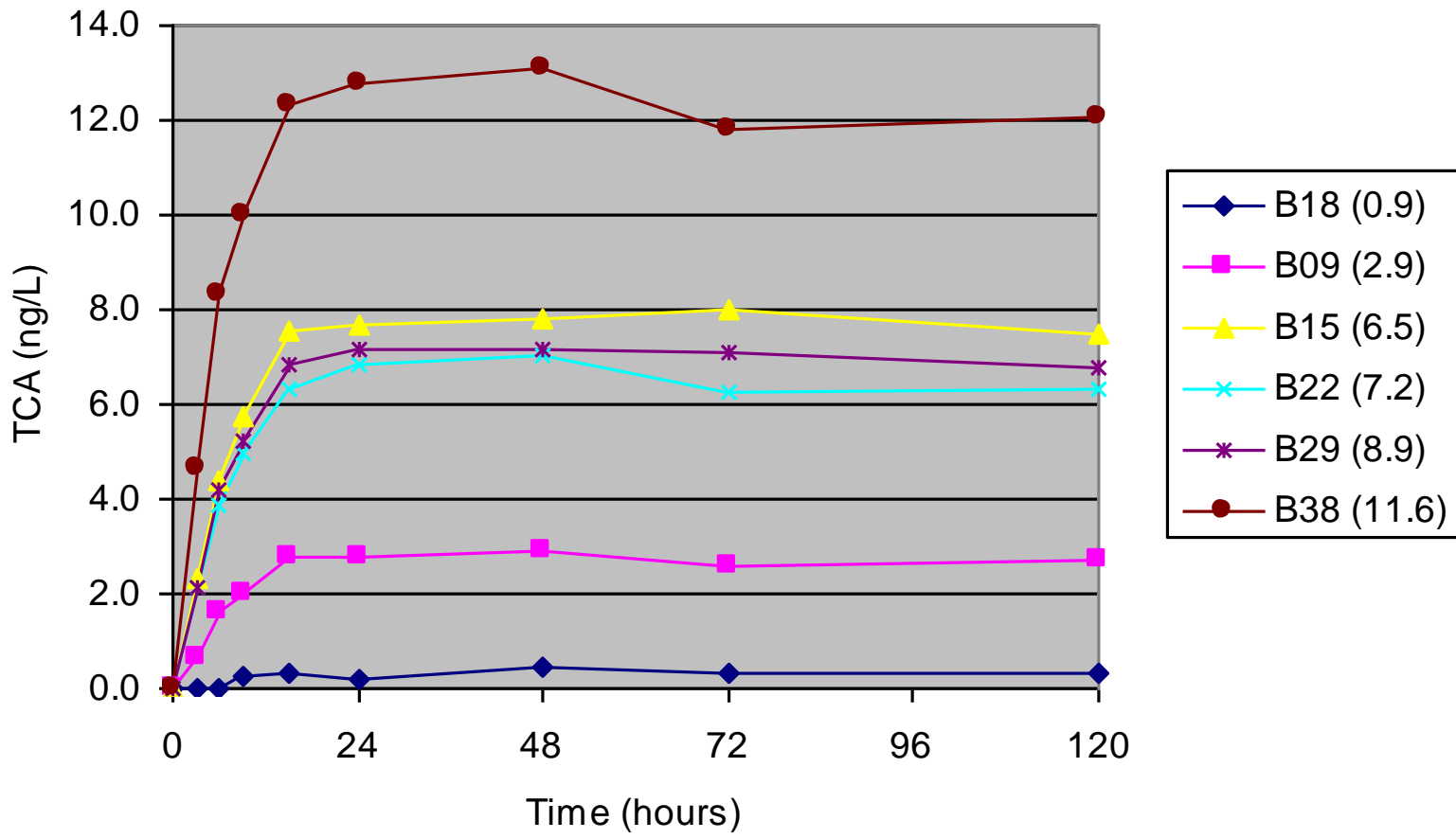
# Detecting TCA

## Gas Chromatography/ Mass Spectrometry – with SPME fiber absorption

- Quick and accurate
- Detects TCA below 1ppt
- Compares favorably to sensory detection levels
- We perform over 35,000 analyses per year for the cork industry.



# Kinetics of cork soaks: equilibrium

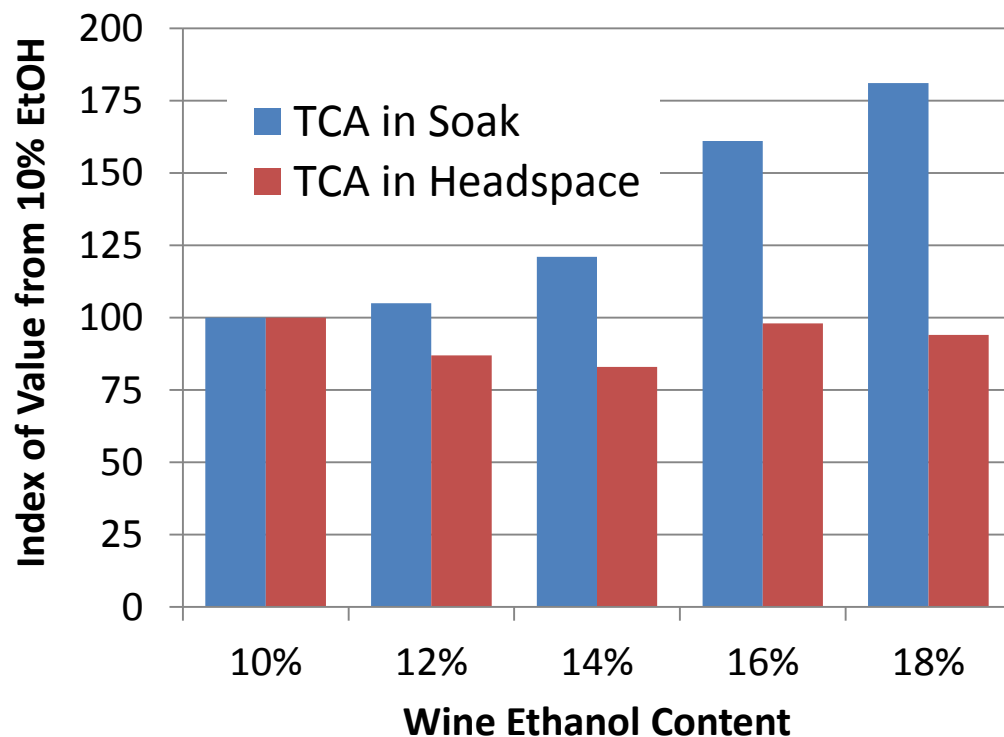


# Ethanol Concentration of Soak Wine

Higher ethanol wines produce higher TCA concentrations in soaks

... but TCA becomes harder to detect either by analysis or sensory.

**Preferred ethanol concentration between 10-12%**



# Releasable TCA

For a cork, or group of corks, the concentration of TCA reached at equilibrium in a wine soak.  
Expressed in ng/L (or ppt).

*Hervé E., Price S., Weber P. and Burns G. - ASEV 1999*



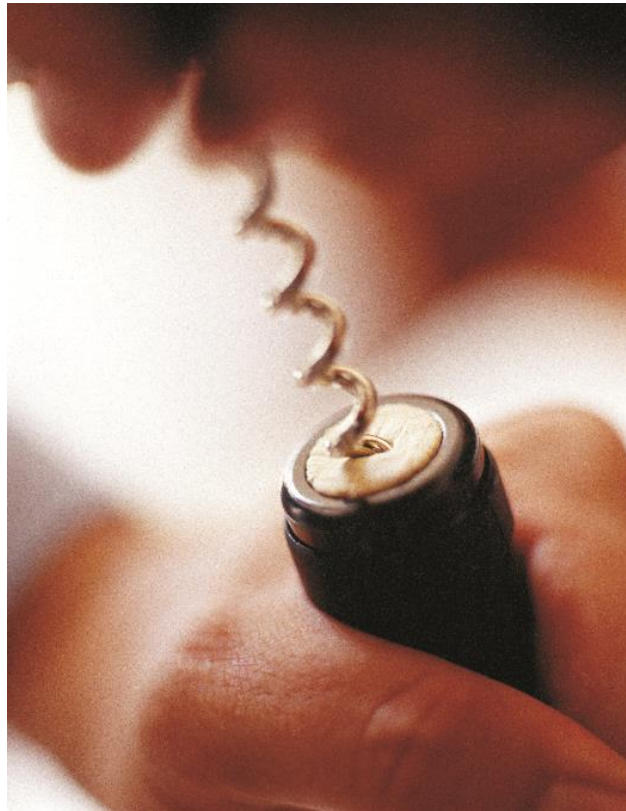
# Group Cork Soaks

## Preferred Sample Preparation

- Sample sizes of 50 and 100 corks give similar results
- Soak media is table wine at 10-12% ethanol
- Soak duration of 24 to 48 hours



## How does RTCA Relate to TCA in Bottled Wine?



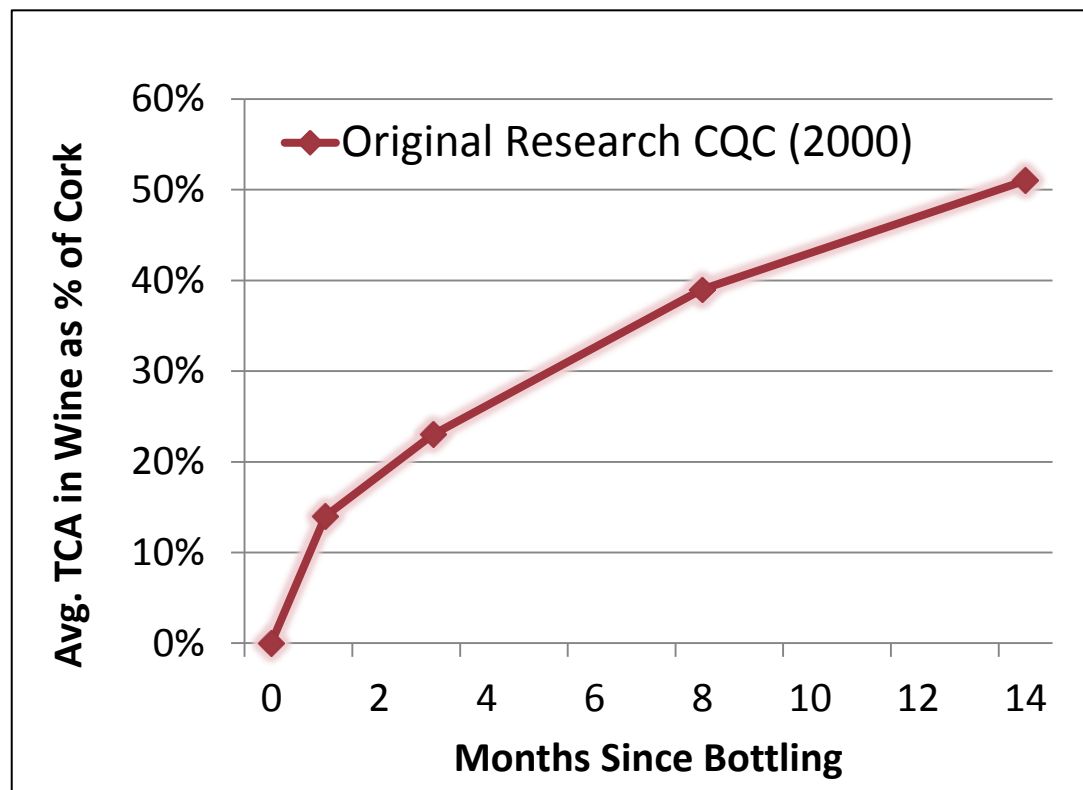
## How does RTCA Relate to TCA in Bottled Wine?

### “Transfer Rate”:

Original ETS research (2000) showed that bottle TCA was on average...

~40% of RTCA at 9 months

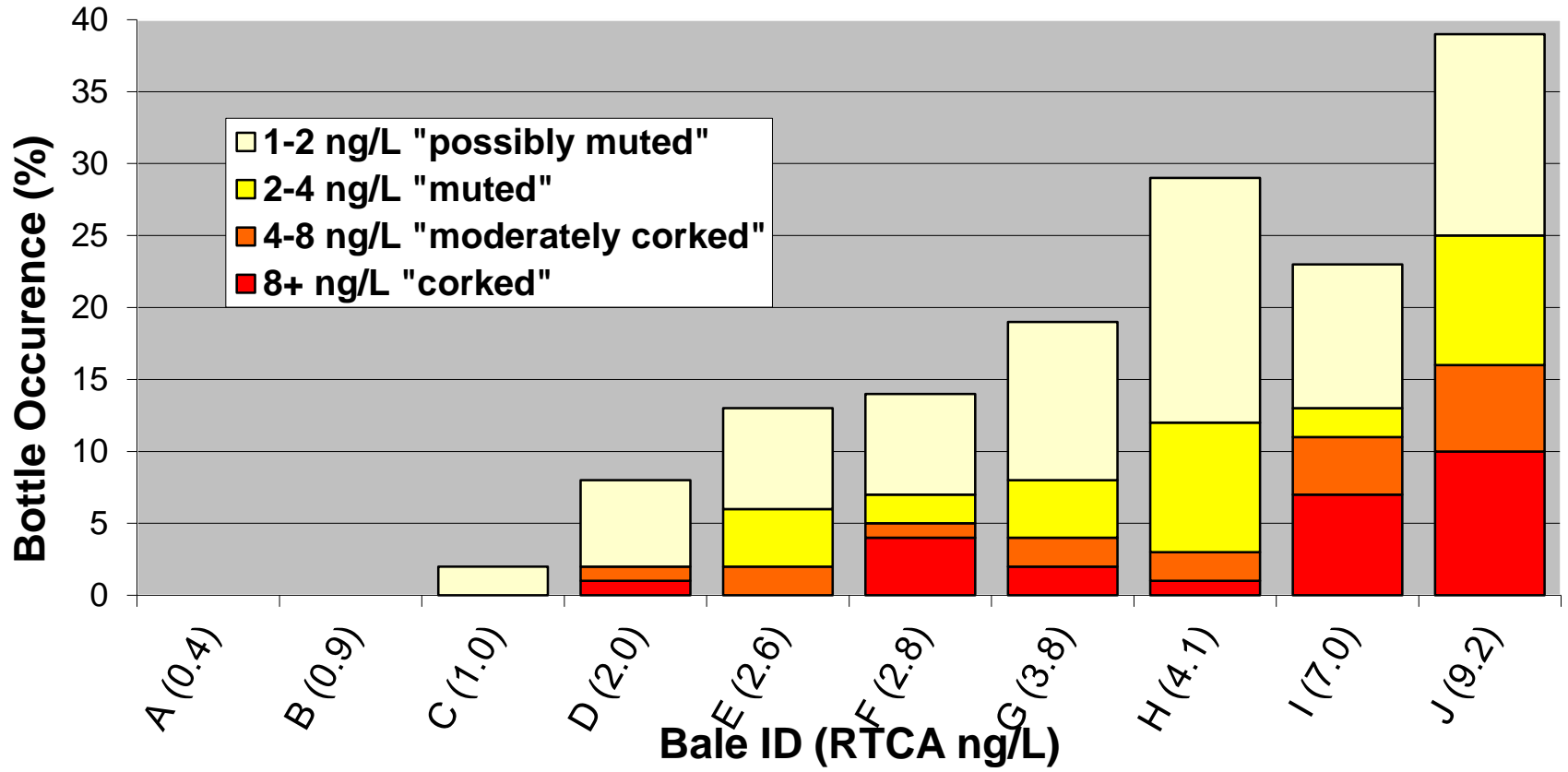
~50% of RTCA at 14 months



## How does RTCA Relate to TCA in Bottled Wine?

- 2004- 2006: ETS Study funded by the American Vineyard Foundation
- Population and Bottling Studies were done using 10 cork bales with a wide range of RTCA levels
- Ultimate goal was to determine the performance of cork soaks as a quality control tool

# Review of AVF Data for TCA in Bottled Wine

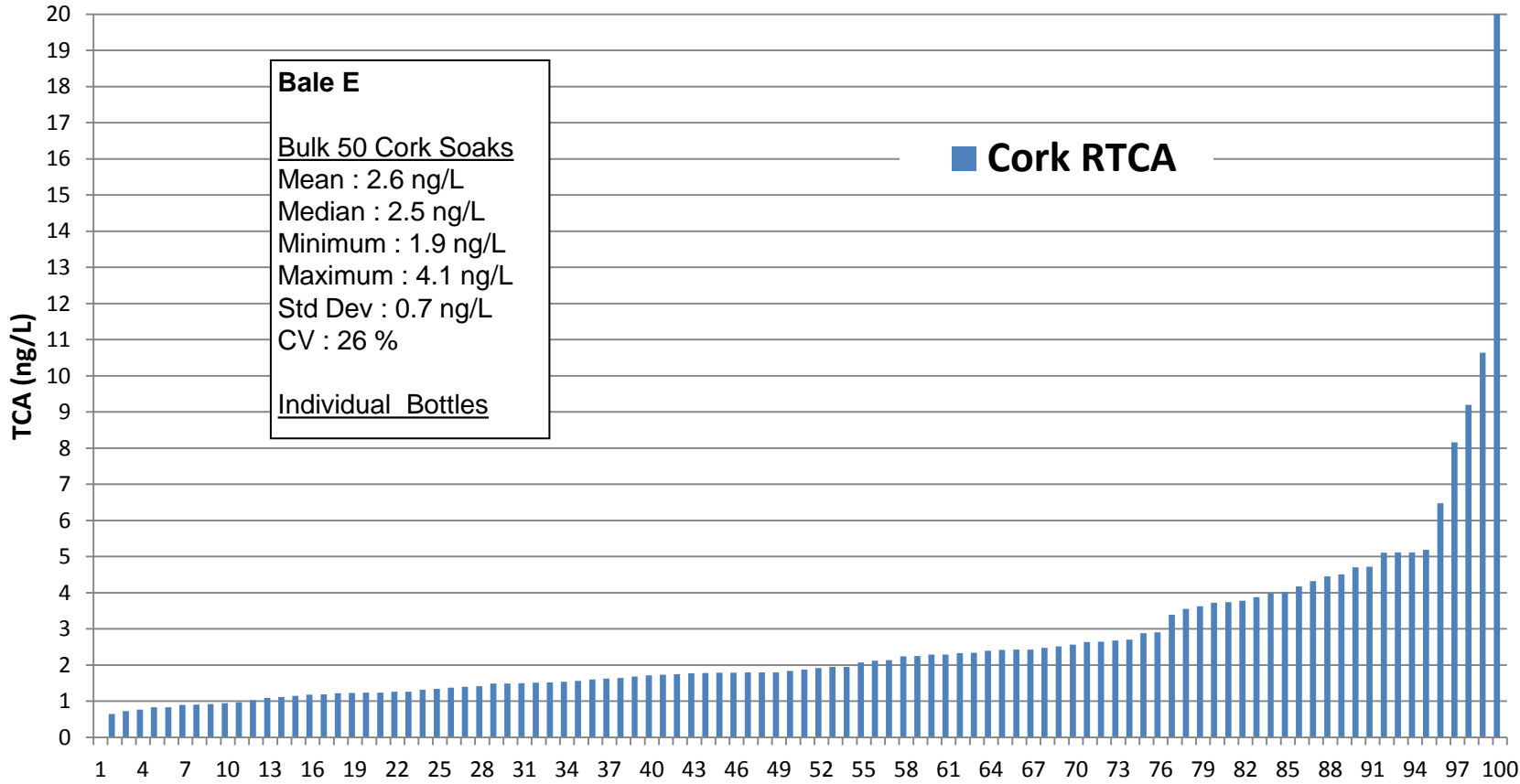


## How does RTCA Relate to TCA in Bottled Wine?

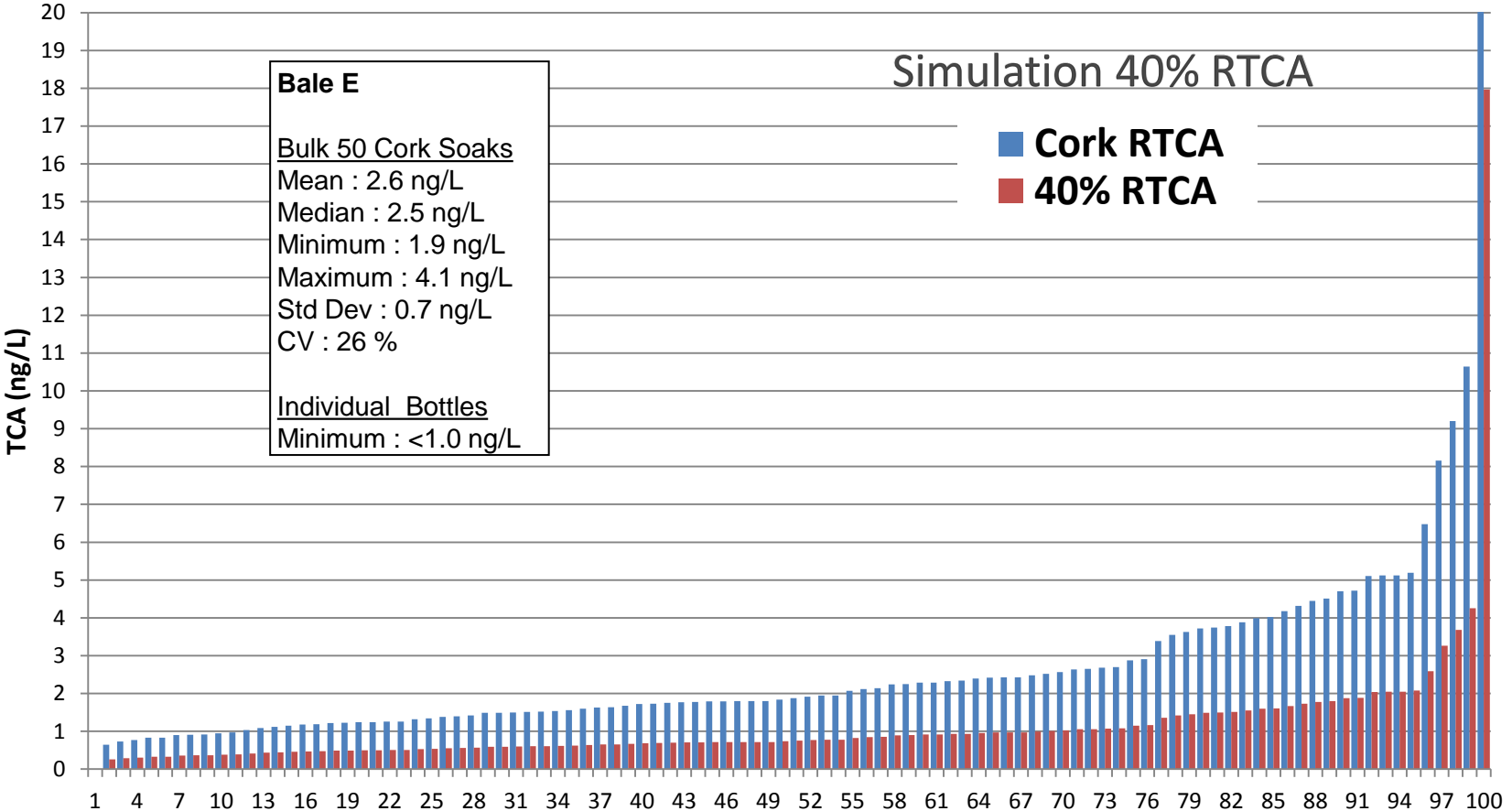
Also... when comparing 1,000 individual cork's RTCA data to bottle TCA results after 9 months and 20 months:

- Transfer rates appeared lower than 40-50% results from the initial study, especially for corks with low to moderate RTCA
- In fact, a “threshold” effect was apparent: RTCA needed to be at least ~ 4 ppt for TCA to reach 0.5 ppt in bottled wine.

# How does RTCA Relate to TCA in Bottled Wine?

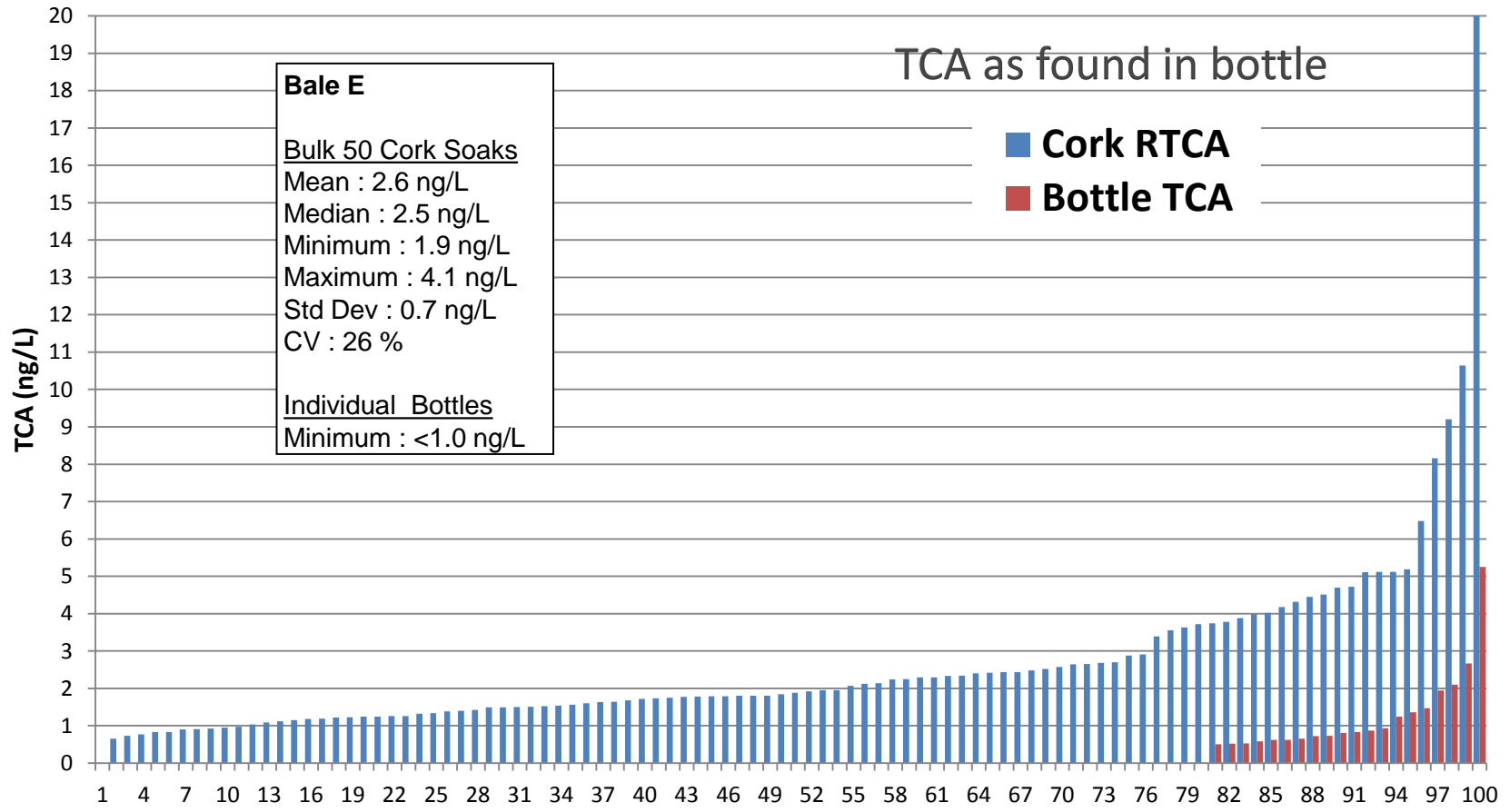


# How does RTCA Relate to TCA in Bottled Wine?





# How does RTCA Relate to TCA in Bottled Wine?



# Group Cork Soaks Applied

- The CQC uses group soaks for its attribute sampling program
- Based on ISO-2859-1 Inspection Standards
- The CQC has a reporting limit for TCA of 1 ppt and its conformity flag level is 1.5 ppt.

# Group Cork Soaks Applied

- CQC Members are required to subject all incoming corks to releasable TCA screening in accordance with approved guidelines
- Sampling is based on individual Cork Lots – defined as a single shipment from the same producer, cork type, visual grade, treatment and arriving on the same shipment
- Corks rejected during inspection are removed from inventory and are returned to manufacturer
- Adherence is subjected to an annual audit

# Group Cork Soaks Applied

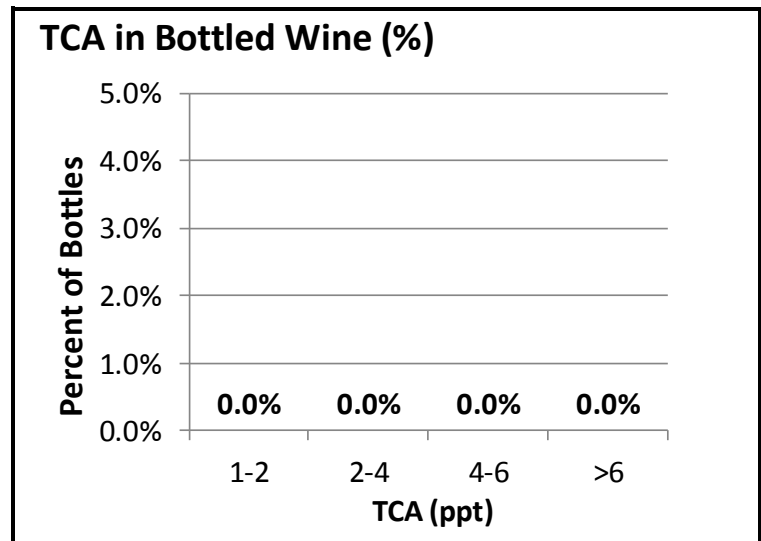
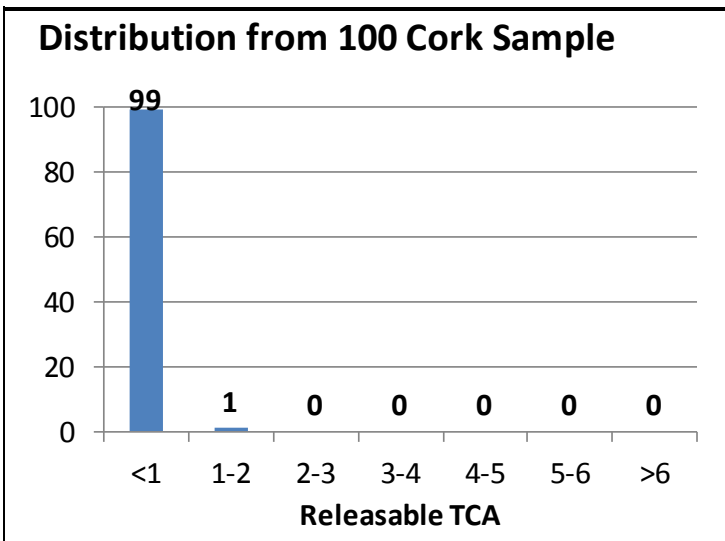
**BALE AVF-A**

Inspection Results **Pass**

Example From AVF Study

**50-Cork Group Soaks**

Replicate	1	2	3	4	5	6	7	8	9	10	Mean	StDev	Flags
rTCA	<0.5	<0.5	0.63	<0.5	0.92	<0.5	0.49	<0.5	<0.5	<0.5	<b>0.44</b>	<b>0.22</b>	<b>0</b>



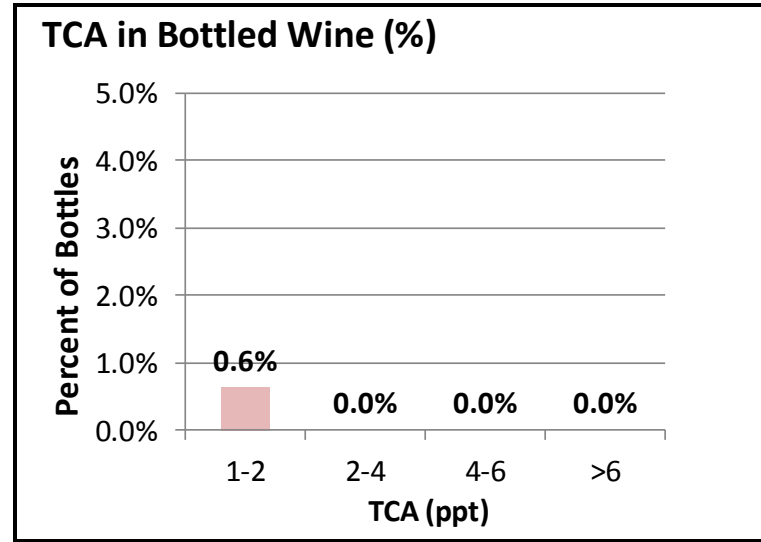
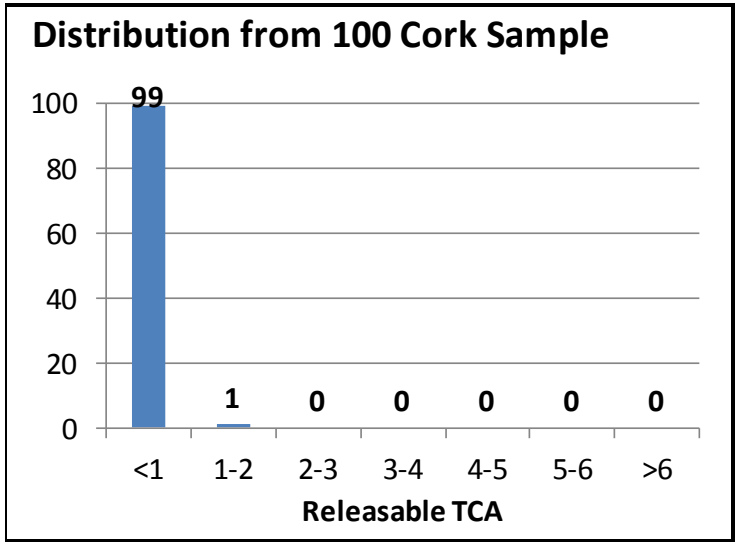
# Group Cork Soaks Applied

**BALE AVF-B**

Inspection Results **Expanded Resample**

**50-Cork Group Soaks**

Replicate	1	2	3	4	5	6	7	8	9	10	Mean	StDev	Flags
rTCA	1.14	0.70	0.89	0.53	<0.5	<b>2.82</b>	0.57	0.61	0.75	0.56	<b>0.90</b>	<b>0.71</b>	<b>1</b>



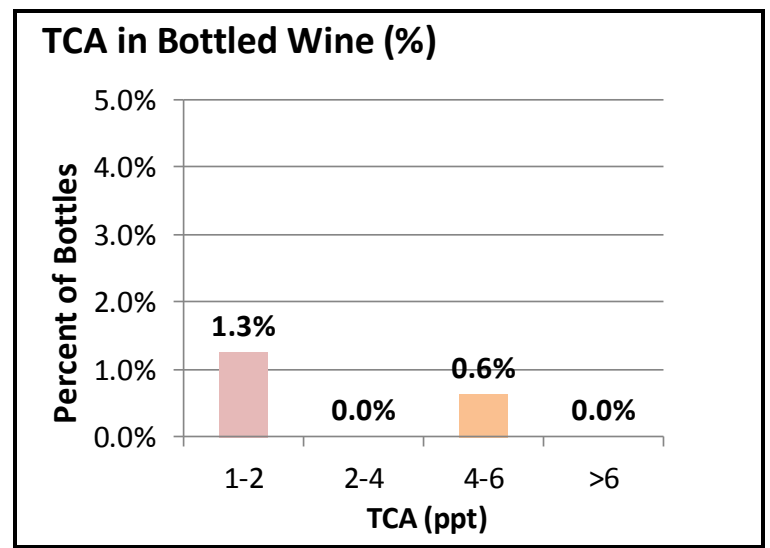
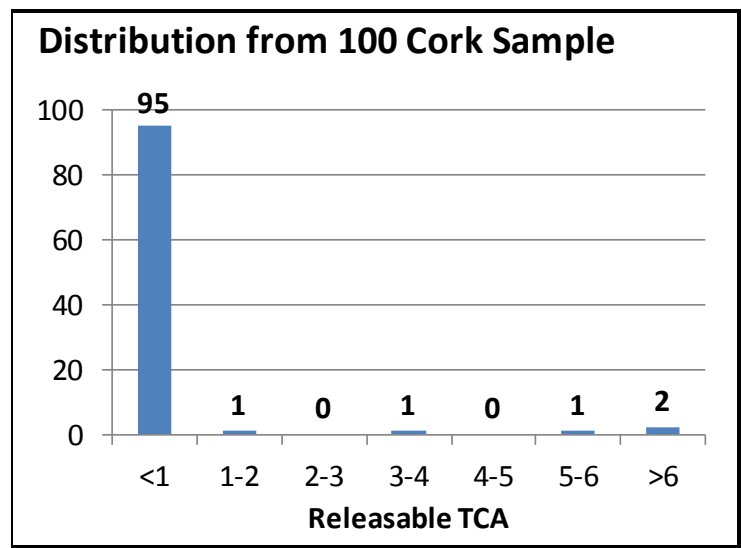
# Group Cork Soaks Applied

**BALE AVF-C**

Inspection Results **Expanded Resample**

**50-Cork Group Soaks**

Replicate	1	2	3	4	5	6	7	8	9	10	Mean	StDev	Flags
rTCA	<0.5	<0.5	<b>4.24</b>	<0.5	<0.5	<0.5	0.81	0.72	<0.5	<b>1.95</b>	<b>1.02</b>	<b>1.23</b>	<b>2</b>



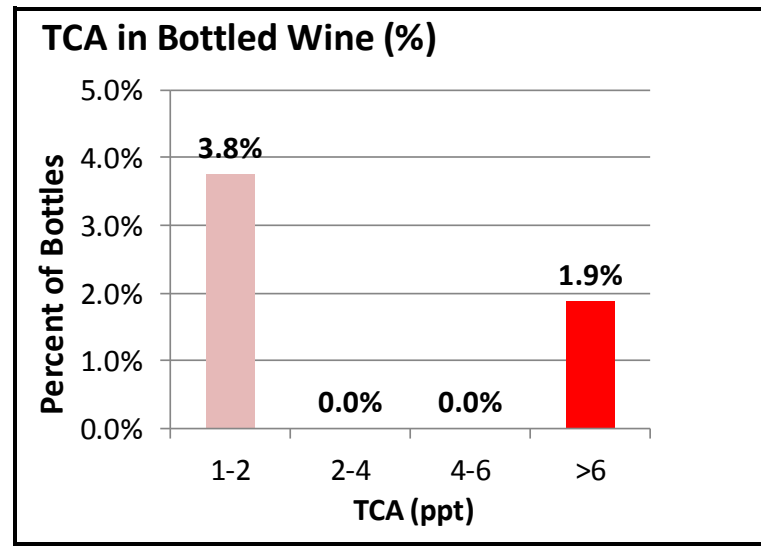
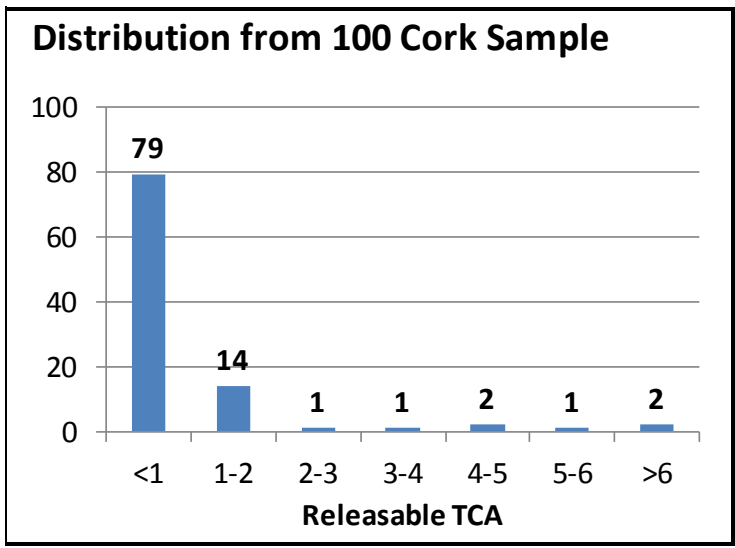
# Group Cork Soaks Applied

**BALE AVF-D**

Inspection Results **FAIL**

50-Cork Group Soaks

Replicate	1	2	3	4	5	6	7	8	9	10	Mean	StDev	Flags
rTCA	1.36	1.30	<b>1.75</b>	<b>1.70</b>	<b>1.86</b>	1.21	<b>5.20</b>	1.29	<b>2.14</b>	<b>1.69</b>	<b>1.95</b>	<b>1.18</b>	<b>6</b>



# Group Cork Soaks Applied

Minimum CQC Sampling  
and Acceptance Criteria for:

**Revised Protocol AQL 2.5**  
Approved 02/26/2013  
Effective Date 1/17/2014

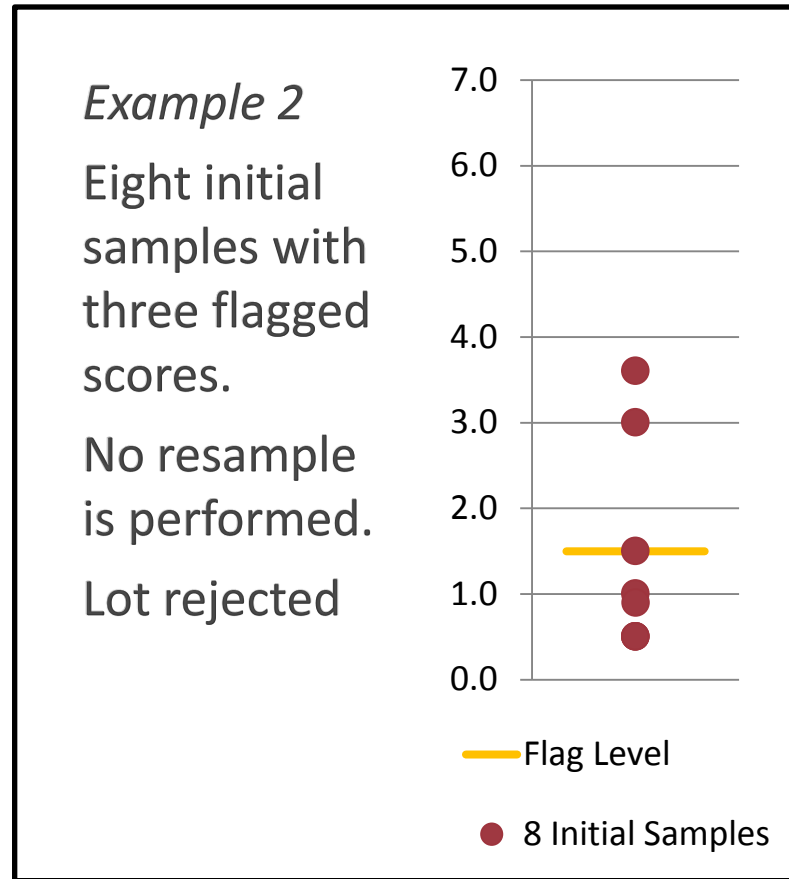
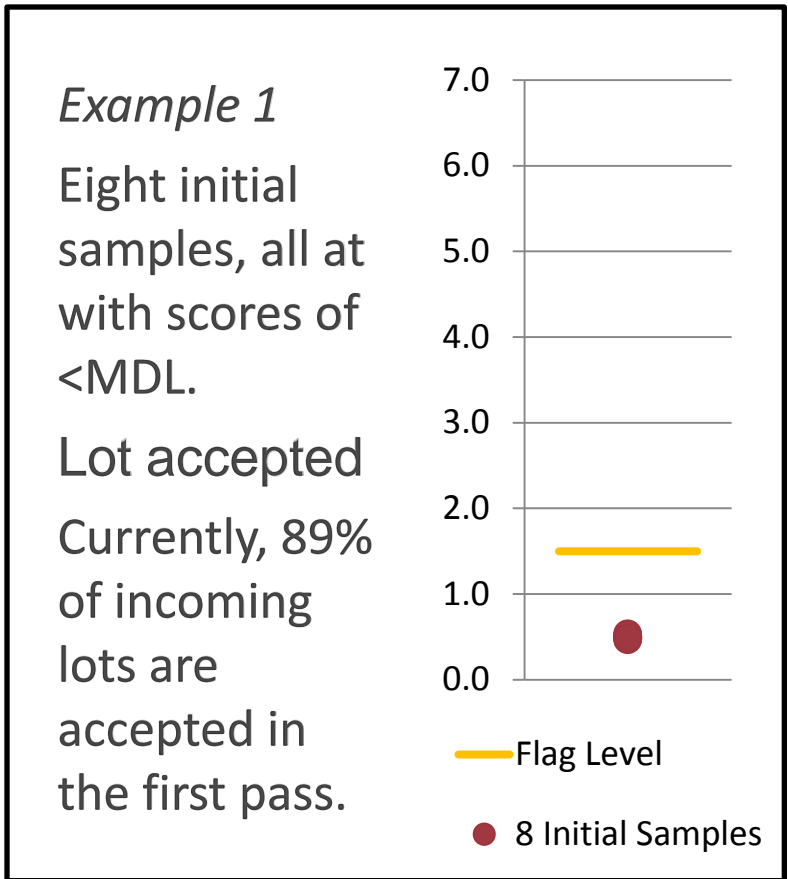
## NATURAL CORK LOTS

### Tests based on number of Bales

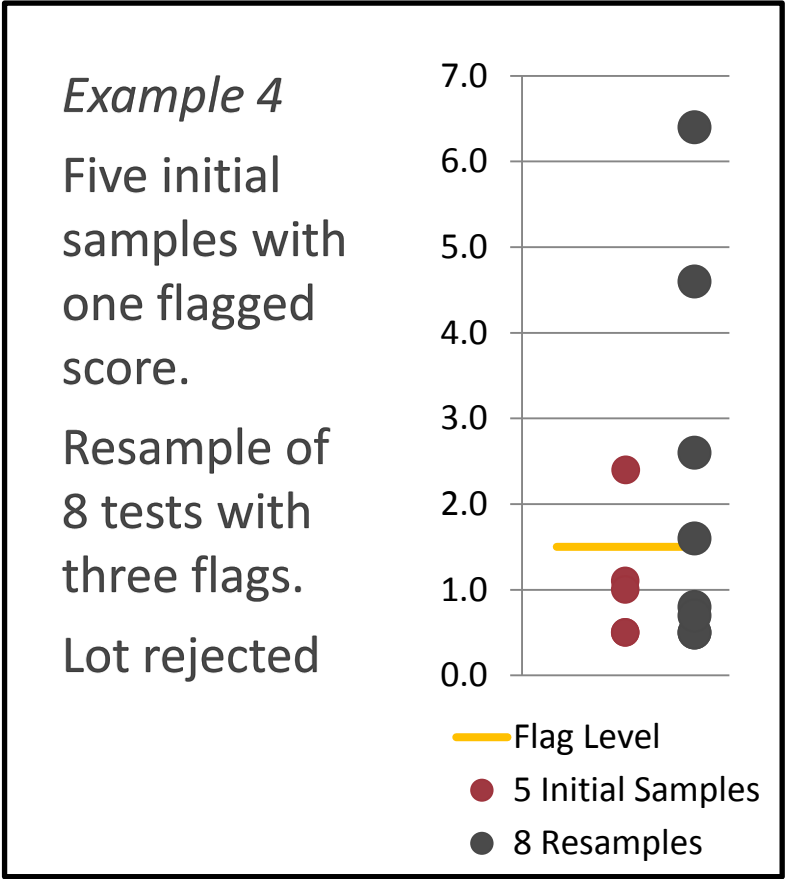
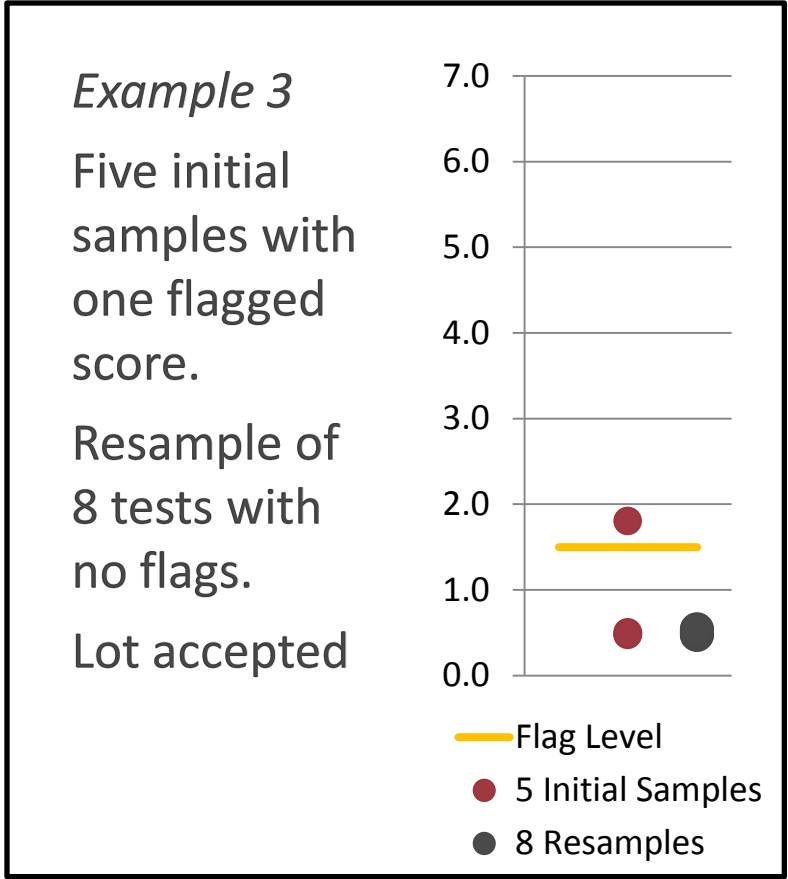
NUMBER OF BALES		2-8	9-15	16-25	26-50	51-90	91-150
<b>SPME TESTING</b>	<b>REQUIRED</b>						
<b>Initial Bale Samples</b>	<i>for all</i>	2	3	5	8	13	20
Pass / Fail	<i>natural corks</i>	0/1	0/1	0/1	0/1	1/2	1/2
<b>Supplemental</b>	<b>Conditional</b>						
<b>Sampling (48hr Soak)</b>	<i>based on</i>	3	5	8	13	20	32
Accept / Reject	<i>Initial Results</i>	0/1	0/1	0/1	1/2	1/2	2/3



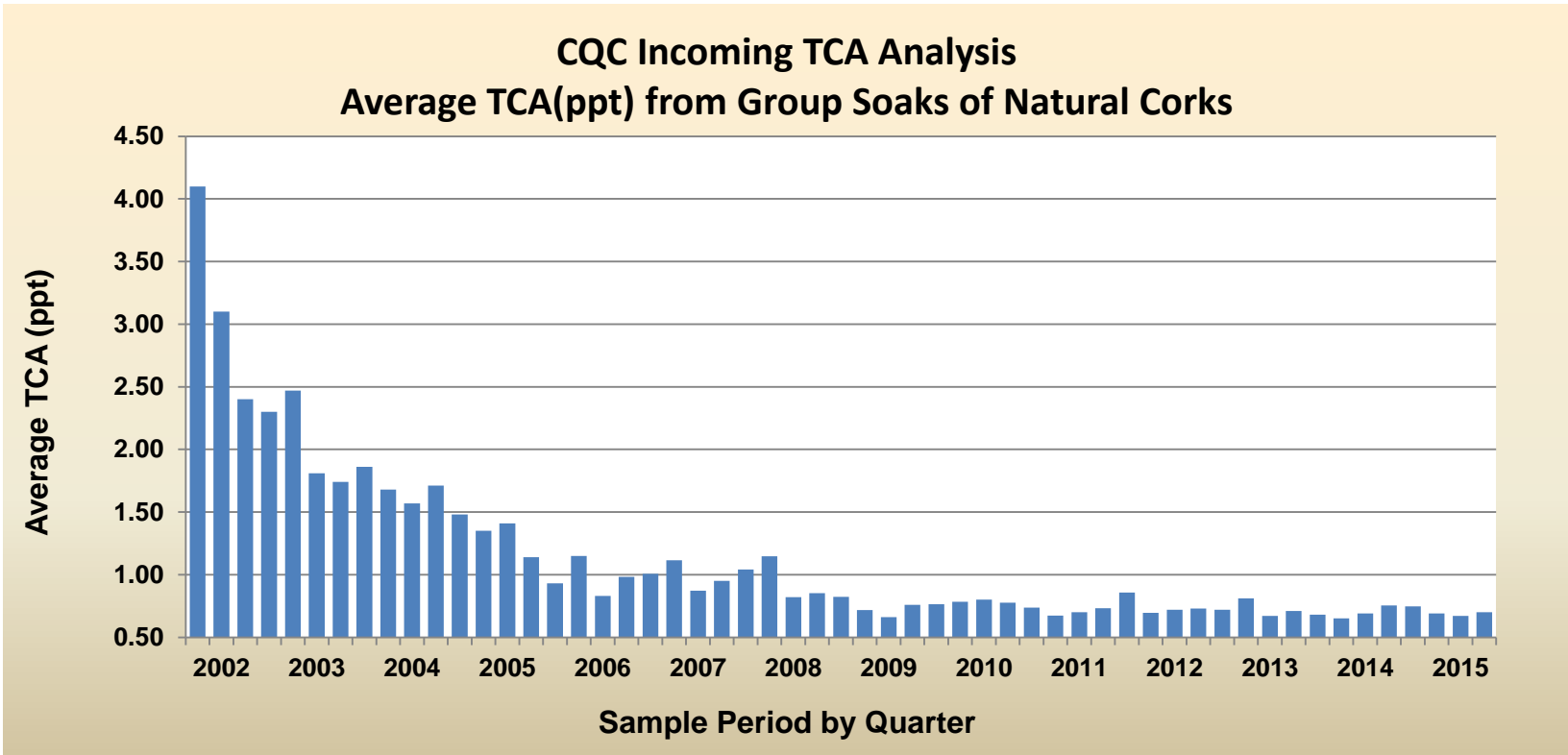
# Group Cork Soaks Applied



# Group Cork Soaks Applied



# Group Cork Soaks Applied



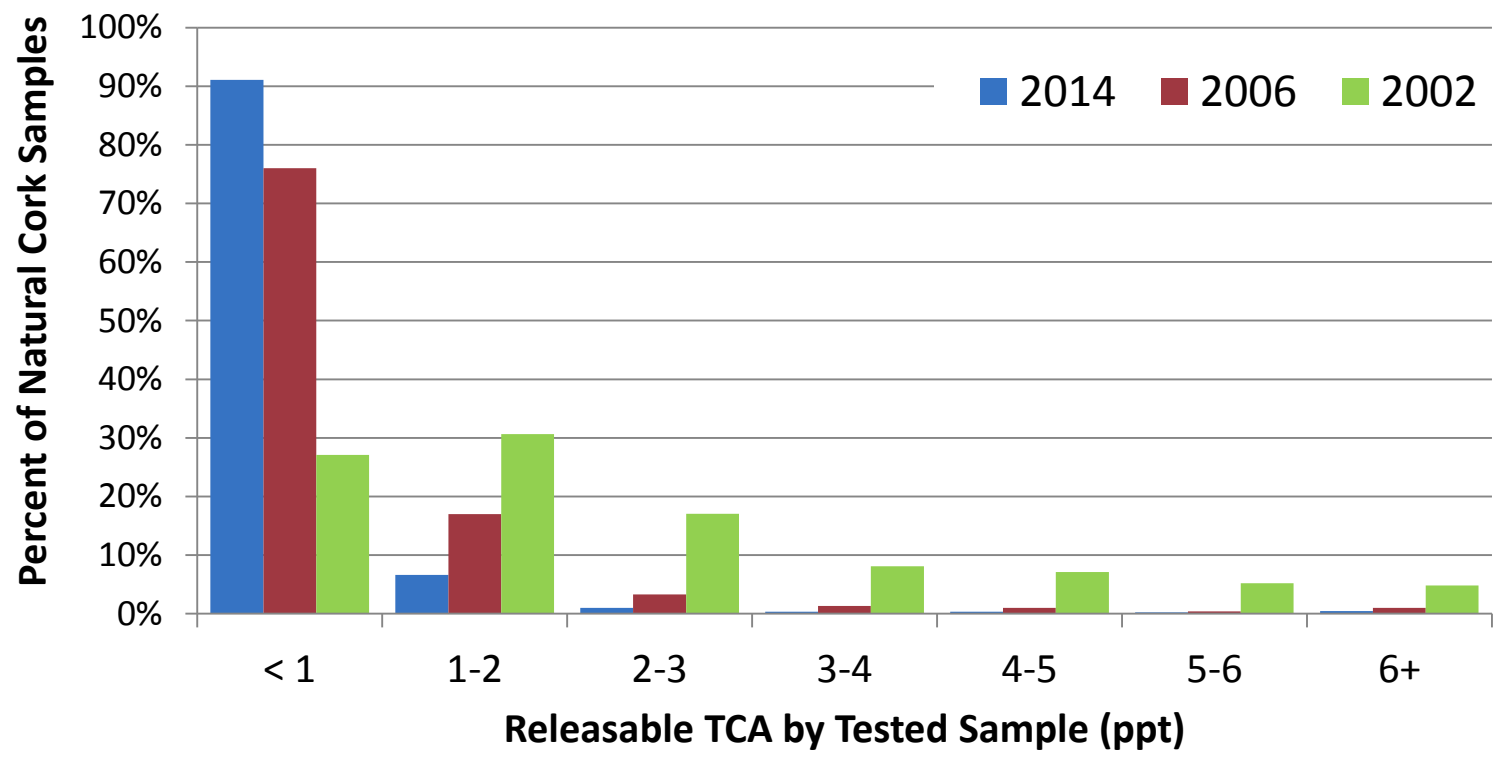
N = 200,000+

Not Corrected for Rejected Corks



# Group Cork Soaks Applied

Comparison of CQC Population Curves Over Time  
All Incoming Natural Corks - Not Corrected for Rejected Corks



## Group Cork Soaks Applied

The CQC recently updated population statistics by selecting seven cork bales from accepted inventory.

### Cork Population Profiles

<b>TCA Range</b>	<b>CQC 2015 700 Corks</b>	<b>AVF (A&amp;B) 200 Corks</b>
<1.0	98.0%	97.0%
1-2	0.7%	2.0%
2-3	0.3%	0.5%
3-4	0.4%	0.0%
4-5	0.3%	0.0%
5-6	0.0%	0.0%
>6	0.3%	0.5%
<b>Total</b>	<b>100%</b>	<b>100%</b>

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Thanks to the CQC for providing their data and helping compile this today's presentation.

