



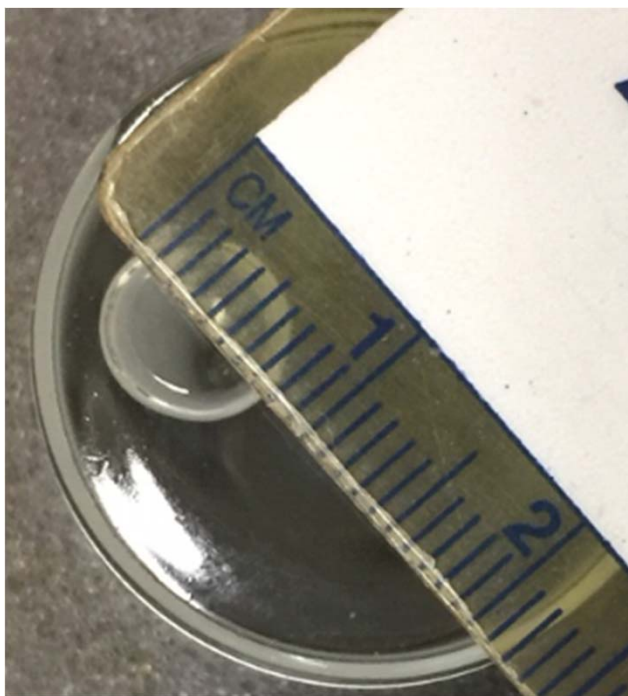
Bubble Trouble Solution



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Trouble from One Bubble



History Lesson



March 1990

Labcert Bulletin published by the Office of Drinking Water

Note: By the time the sample arrives at the lab, a small **bubble** may have developed. As long as this is **no larger than the size of a pea** the sample may be considered valid.

Courtesy of Jerry Parr

Method 5030B - 1996



6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 ... Samples should be stored in capped vials, with minimum headspace, at 4°C or less in an area free of solvent fumes. The size of any **bubble** caused by degassing upon cooling the sample should not exceed 5 - **6 mm**.



The Solution



← water dome



10 in-lb

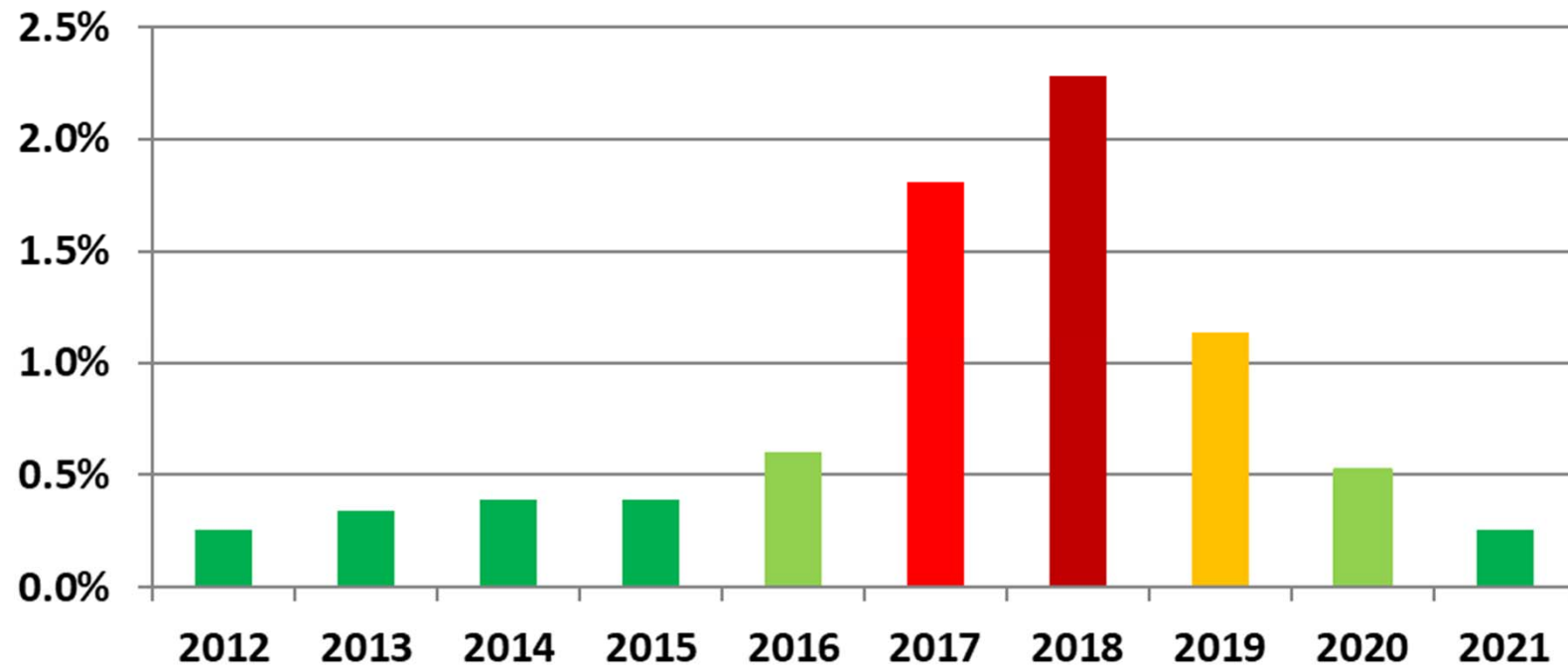


← Bowed septum

Recent History - Year



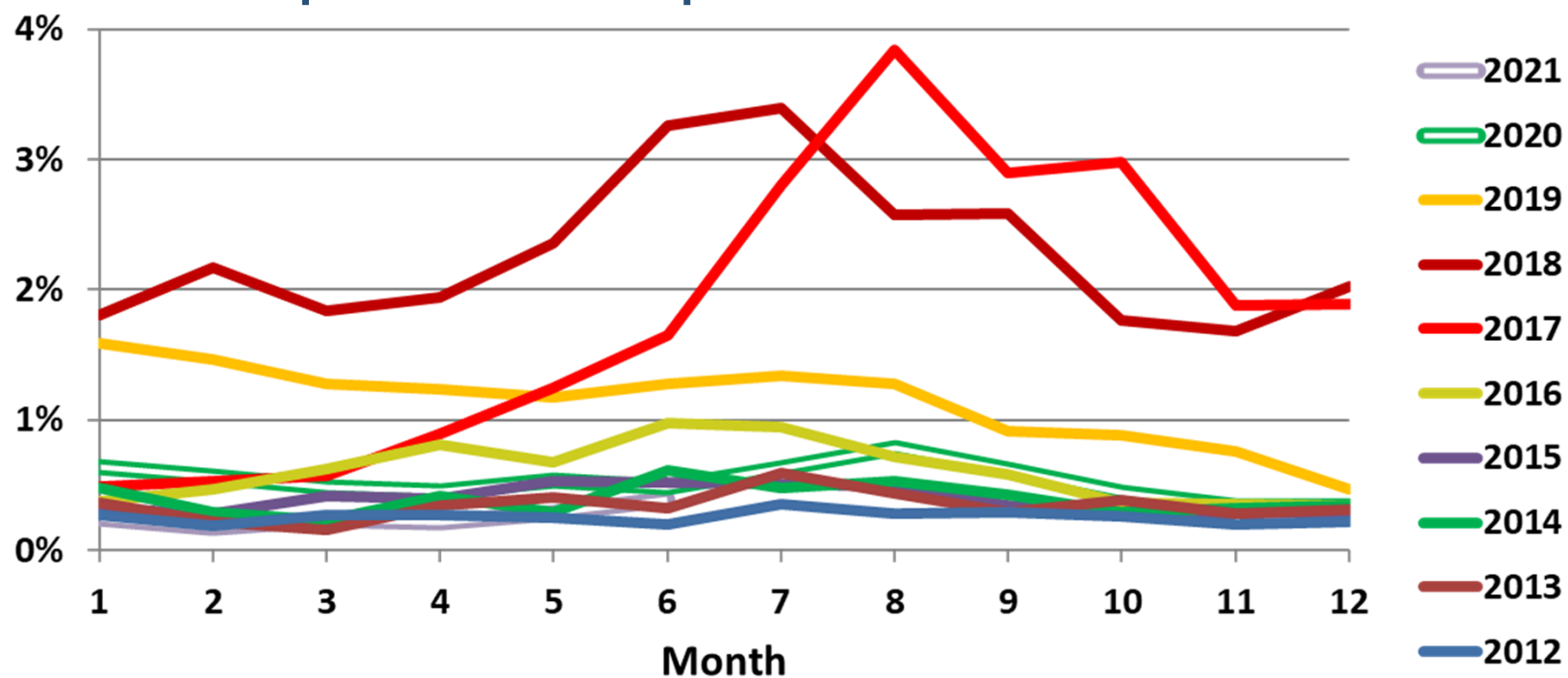
%VOC water samples with headspace non-conformance memos



Recent History - Month



%VOC water samples with headspace non-conformance memos



Factors Studied



Sample	Collection Technique
Carbonate concentration	Entrained air bubbles due to vigorous pouring
Dissolved gas concentration	Water dome height above the top of bottle
<u>Water temperature at time of sampling</u>	<u>Under tightened cap</u>
	Over tightened cap

Factors Studied



Transport and Storage

Temperature drop to 0-6°C

Cap loosening due to vibration or temperature changes

Air pressure changes due to air shipment or elevation changes between site and laboratory

Factors Studied



Container	
Temperature of bottle at time of use	<u>Stiffness (durometer) of septum</u>
Long term storage temperature of bottles	HCl preservative source
Diameter of septum, cap and top of glass bottle	Thickness of Teflon face on septum
Thread match between cap and bottle	Press fit vs bonded septum
Inside surface of cap that contacts the septum	Shape of septum & position in cap
Glass bottle sealing surface (rough, round, flat)	Septum position after tightening

VOC Vials Studied Extensively



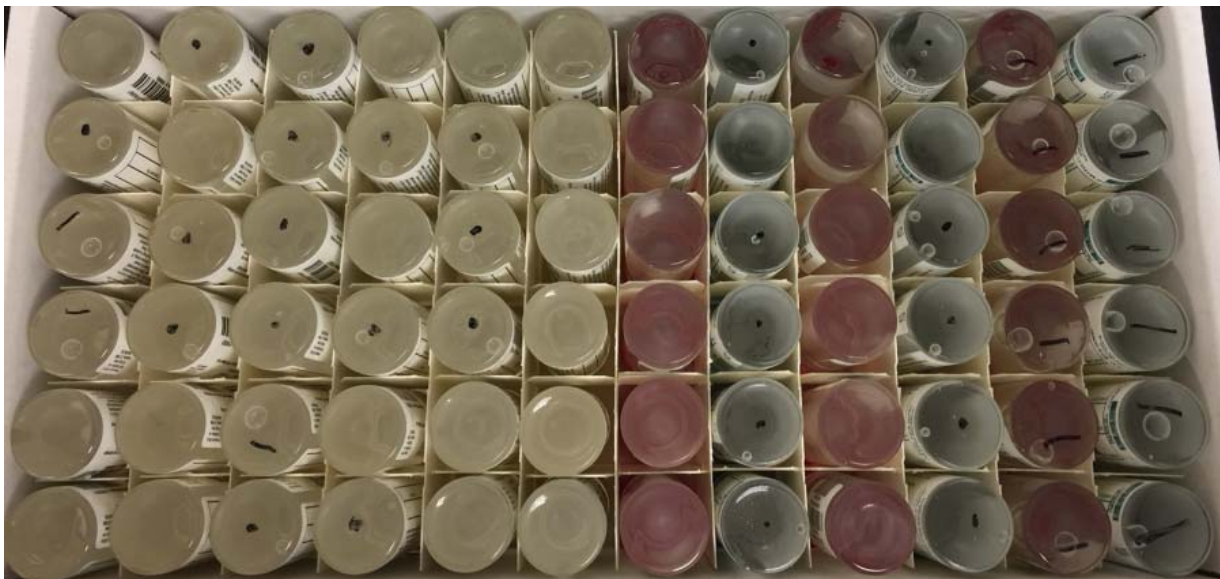
3+ Years

5 suppliers

57 Experiments

2584 Bubbles

5526 Vials



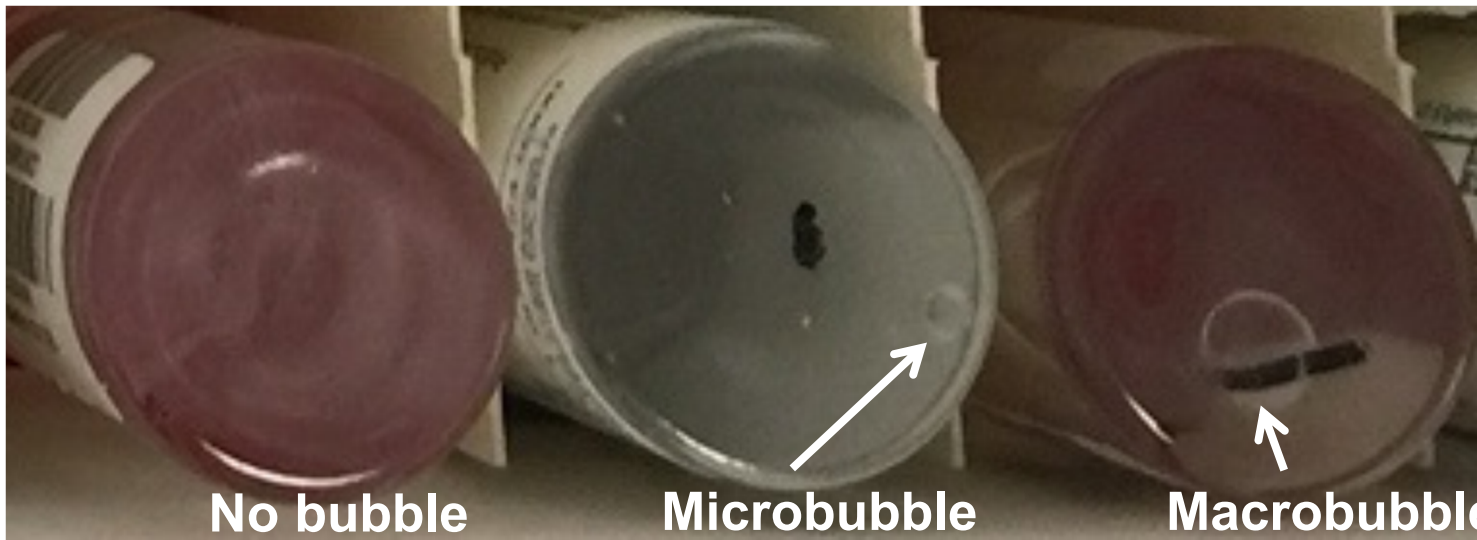
Headspace Classification



No bubble

Microbubble (<6 mm)

Macrobubble (>6 mm)



Cap Tightness Classification



Level 0 – septum is bowed down

Level 1 – septum is flat

Level 2 – slightly bowed upward, (6-8 in-lb)

Level 3 – bowed upward, extends 1-2 mm above the cap (10-12 in-lb)

Level 4 –bowed upward, extends > 3 mm above the top of the cap



After Nearly a Year of Lab Work



Investigate all previously listed potential factors

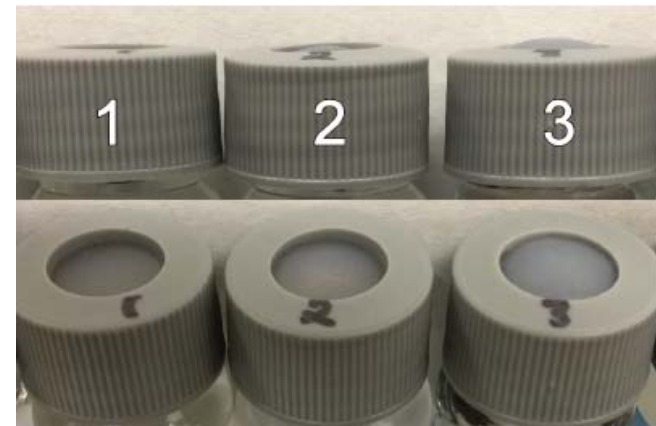
Major client with two sites with recurring bubble trouble

Field Test

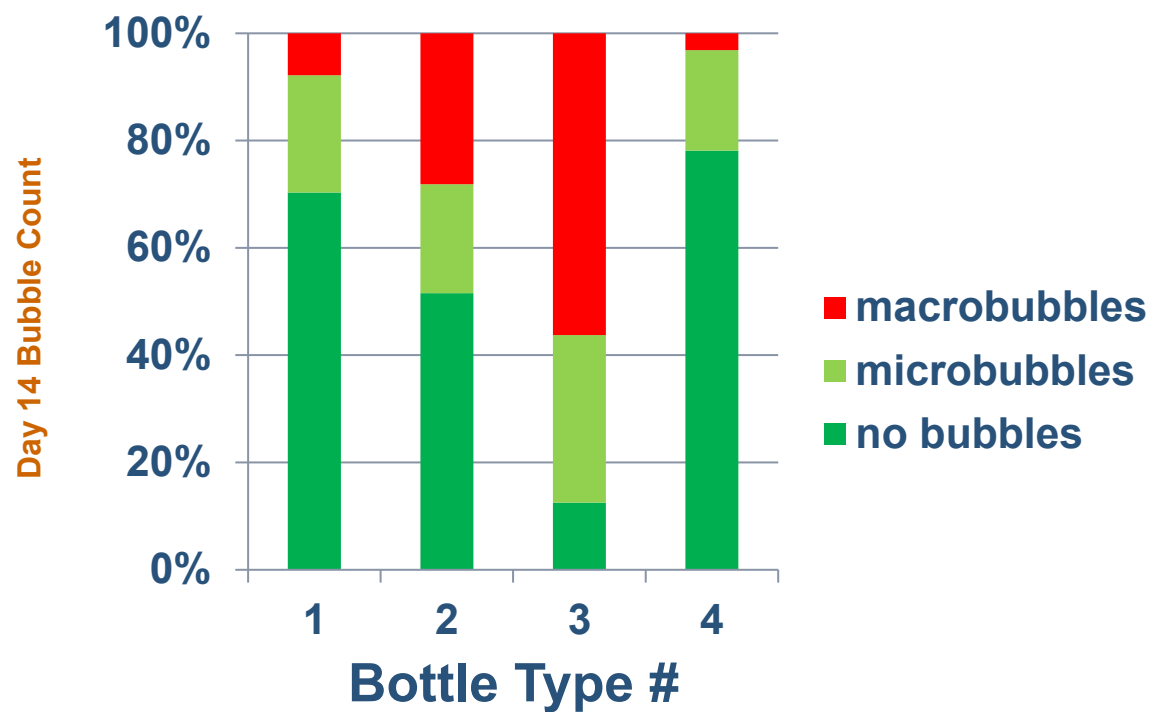


Experimental Factors

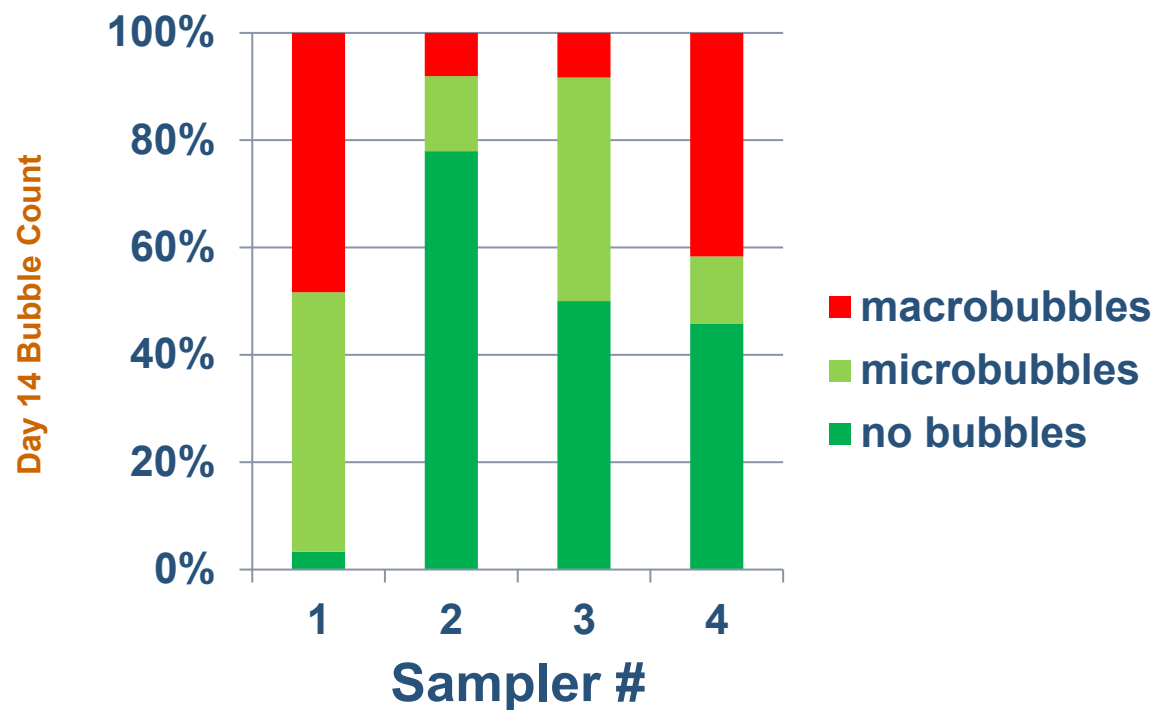
- Site (2)
- Well (10)
- Sampler (4)
- Bottle type (4)
- Cap tightness (3 levels, 1, 2 & 3)



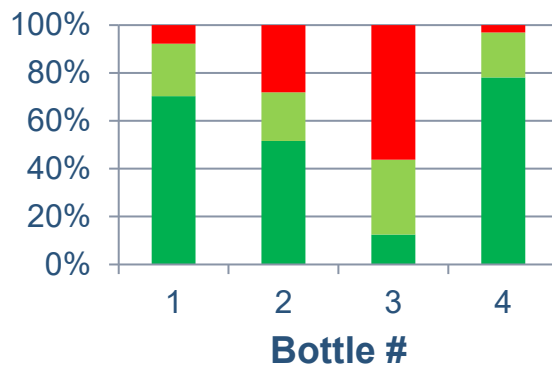
Bubbles by Bottle Type



Bubbles by Field Sampler

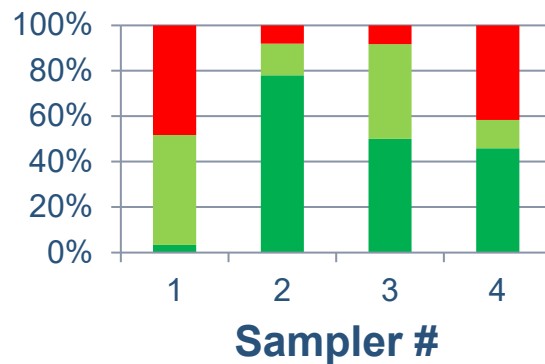


Interpretation



Bottle #4 was best
Bottle #1 was close

- **Key Difference**
 - **Septum**
not too soft, not too stiff
+/- 12% specification



Sampler #2 was best
Sampler #3 was close

- **Key Difference**
 - **Cap tightness (Level 3)**

Best Combination



Bottle #4

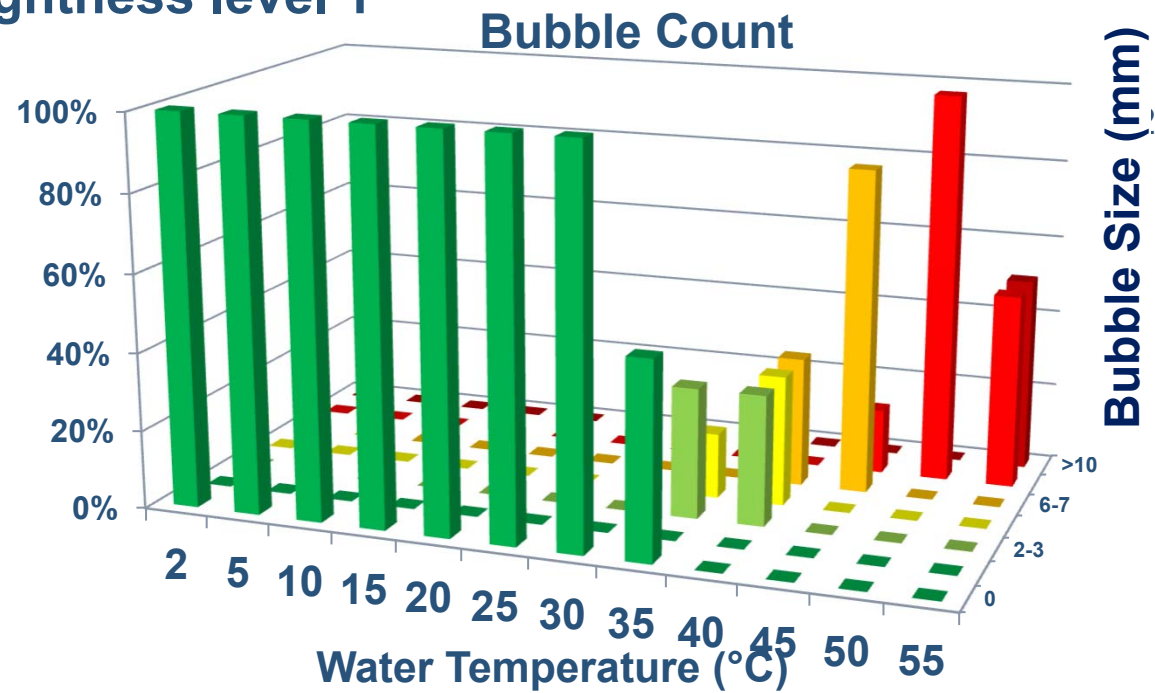
Sampler #2 (level 3 tightness)

**No Bubbles
100% of vials
Over 10 wells
And 2 sites**

Sample Temperature



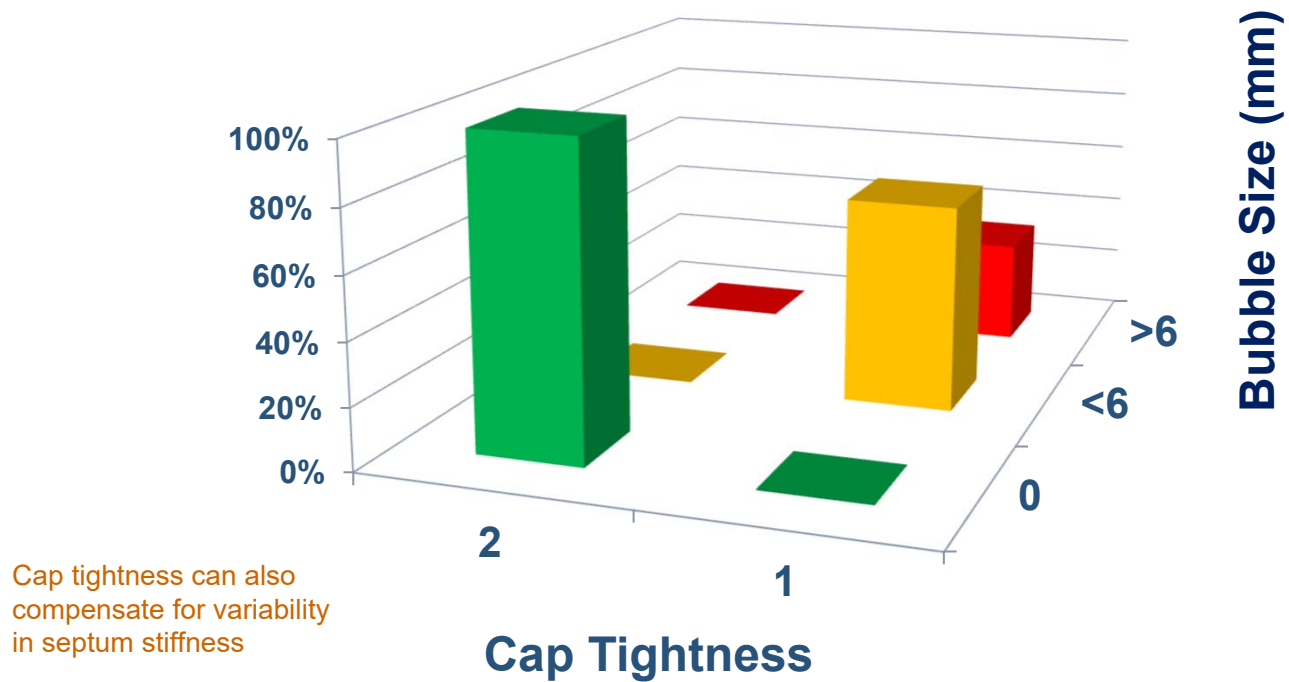
Bottle #4 – tightness level 1



How to Compensate for Warm Samples



Bottles 1 & 4, 35°C



What is Happening?



Hypothesis

Sample water contracts on cooling

Pulling air past the septum seal to form a bubble

Larger the temperature drop or the less tight cap

The more air pulled in

Hypothesis Test



Measure VOC increase due to air bubble

Cool vials in jar with ethyl ether in air

40 uL EE, 4 L air

Low torque vial caps 4 in-lb

High torque vial caps 12 in-lb



Hypothesis Test – Bubble Count

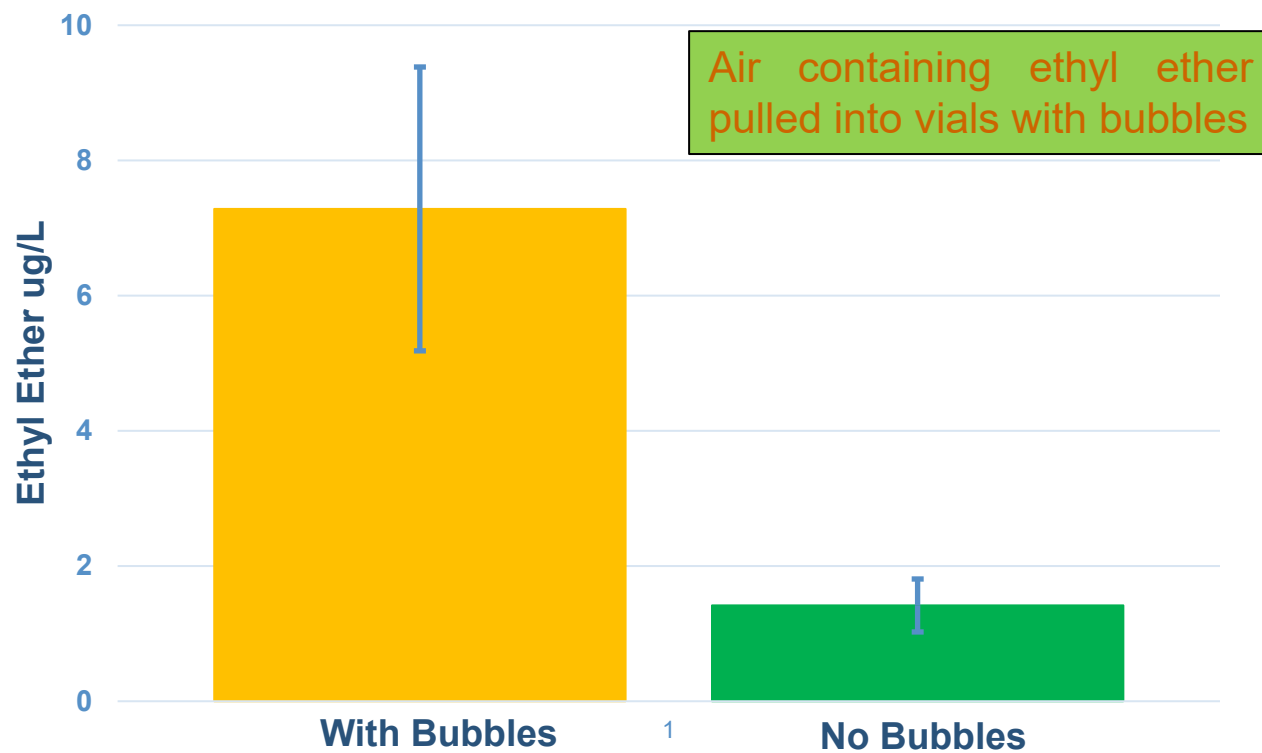


Low torque vials – all had 6-8 mm bubbles

High torque vials – no bubbles



Hypothesis Test - Results



Conclusions



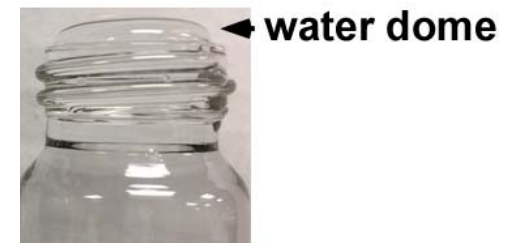
Use VOC vial with good septum and cap

Cut resistant gloves / disposable gloves

Water dome 2-4 mm

Cap Tightness

- at least Level 2
- better Level 3
- ~10 in-lb





Bubbling Questions?

Contact Information



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