Initial Qualification of Stainless Steel Canisters for the Measurement of Trace (ppb) Levels of VOCs in Air

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INTRODUCTION

Background

- Newly purchased stainless steel canisters cannot be assumed to be fit for use for trace-level air toxics monitoring due to the potential for:
  - Manufacturing defects
  - Incomplete interior surface preparation and/or coating (fused-silica lining)
  - Residues from manufacturing process (e.g. cutting oils)
  - Residual artifacts from quality control checks
- Canister qualification is required!

Compounds for canister qualification

Compound	Purpose/comment
benzene	surrogate/recovery standard
chloromethane	surrogate/recovery standard
bromoform	assesses stability in FSL canisters
carbon tetrachloride	assesses stability in SUMMA canisters
benzyl chloride	difficult compound to recover from canisters
naphthalene	low volatility compound difficult to recover from canisters
1,4-diethyl benzene	C_{10} can more easily “slide”/”nestle” into nooks and crannies
1,2,4-trichlorobenzene	difficult compound to recover from canisters

TECHNICAL APPROACH

Canister Cleaning

- Heat in isothermal oven to 71°C (160°F)
- 10 cycles:
  - evacuation to > 23” Hg
  - pressurization with humidified hydrocarbon free zero air (HCF) to 20 psia
- Final evacuation to < 50 mTorr

Experimental Design

Canisters must be leak-tight and be shown not contribute to measurement bias

Qualification – Leak Check and Zero Challenge

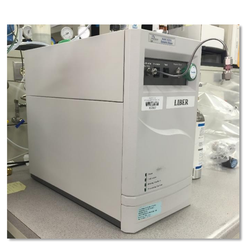
- Pressurize 14 canisters to ~ 16 psia with humidified HCF immediately following cleaning
- Measure baseline canister pressure
- Screen 2 canisters for target analytes
- Store cans at ambient laboratory conditions
- Analyze after 7 days for 15 NATTS PT VOCs
- Goal is for all target analytes < MDL (range 0.0037 [EDB] – 0.049 ppbv [acrolein])

NATTS PT VOCs

acrolein	1,2-dibromoethane	1,3-dichloropropene - trans
benzene	1,2-dichloroethane	1,1,2,2-tetrachloroethane
1,3-butadiene	dichloromethane	tetrachloroethylene
carbon tetrachloride	1,2-dichloropropane	trichloroethylene
chloroform	1,3-dichloropropene - cis	vinyl chloride

Qualification – Known Standard Challenge

- Pressurize 14 canisters to ~16 psia with 0.5 ppbv standard (69-component VOC mix) diluted with humidified HCF
- Store cans at ambient laboratory conditions
- Analyze after 7 days
- Goal is for recovery of all NATTS PT VOCs to be 70-130%



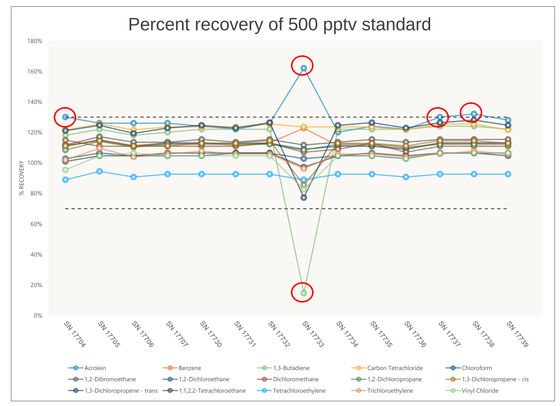
RESULTS

Qualification – Leak Check and Zero

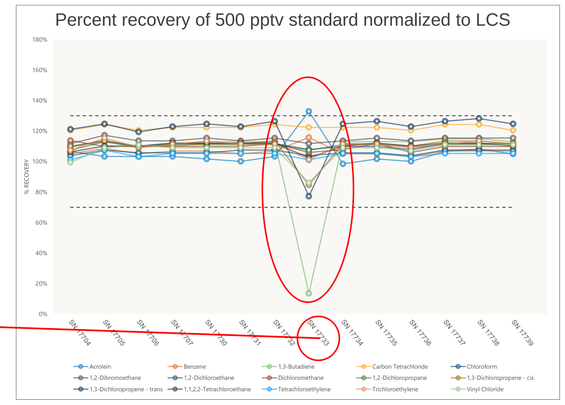
- All 14 canisters leak tight (< 0.1 psi change per day)
- 13 Canisters showed all 15 compounds as non-detect (ND)
- 1 canister (SN 17733) showed hits for 3 analytes:
  - acrolein (0.05 ppbv)
  - 1,3-butadiene (0.10 ppbv)
  - vinyl chloride (0.03 ppbv)

Qualification – Known Standard Challenge

- Recovery for all analytes 70-130% in all canisters except:
  - 4 canisters for acrolein (> 130%)
  - 1 canister for 1,3-butadiene (< 70%)
- Acrolein average recovery 129%

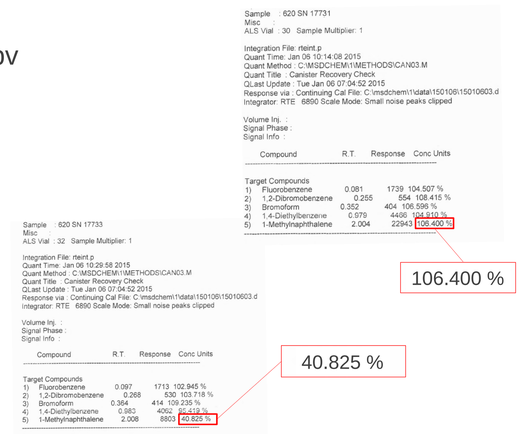


- Laboratory control sample (LCS) indicated high bias for acrolein
  - recovery +/- 12% except acrolein (+22%)
- All results normalized to laboratory LCS One canister shows poor recovery for two compounds and clearly stands out from the others
  - SN 17733



Investigating SN 17733

- 3 contaminants in HCF zero check: 0.03 – 0.10 ppbv
- High acrolein recovery (162%)
- Suppressed response (compared to 13 other canisters) for:
  - 1,3-butadiene (15% recovery)
  - cis-1,3-dichloropropene (86% recovery)
  - trans-1,3-dichloropropene (77% recovery)
  - 1,1,2,2-tetrachloroethane (97% recovery)
  - vinyl chloride (83% recovery)
- Investigate SN 17733:
  - Manufacturer supplies QC check analysis data:
  - Poor methylnaphthalene recovery (40.8%)



CONCLUSIONS

- New canisters must be qualified before initial use for in trace-level air toxics work
- Qualify your cans and contact the canister manufacturer if the cans are not fit for use!