

# Review of Results from 2002 Workshop and 2012 Industry Questionnaire

PAC LP

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# 2002 Workshop

- Workshop developed a list of recommended practices for D5800 Procedures A and B
- Areas of focus were:
  - Correct manometer use and fluid choice
  - Reduction of air flow around instrument
  - Proper vacuum pump operation and cleaning
  - Sample handling

# 2012 Industry Questionnaire

- Questionnaire was prepared to look at operational steps which may be impacting test performance

## 21 instruments covered by the questionnaire

<u>Instrument</u>	<u>#</u>	<u>Age</u>
ISL NCK1:	1	
ISL NCK2:	4	1997-1999
ISL NCK2 5G:	11	2009-2012
ISL V112-230:	1	
ISL PS4000:	2	
Tannas SVT1:	2	2002

# Feedback from Questionnaire

- Airflow around instruments
  - 3 of 10 labs run their instruments in a fume hood
  - None use an external fume apparatus
  - 4 labs have instruments with draft shields
- Matching units
  - 8 of 9 labs use matching lid/crucible pairs with all but one of the 8 having multiple matched pairs
  - 6 of the 8 matched pairs are used on the same instrument
  - Few labs (2 of 8) have replaced only a lid in a matched set
  - 2 of 7 labs have calibrated the vacuum for each matched set
- Lids and crucibles were primarily purchased with the instruments, but have been replaced due to wear or damage

# Feedback from Questionnaire

- Extraction tube information
  - Purchased with original equipment (3 of 8) or unknown (4 of 8)
  - Gasket replacement done annually (3 of 7), as needed (2 of 7), or rarely (2 of 7)
  - Tightening of extraction tube nut is done prior to each run (3 of 7), as needed (3 of 7) or annually (1 of 7)
- RTD calibration
  - Calibration checked annually (6 of 9) or every six months (2 of 9)
  - Multiple methods are used to confirm calibration
  - Most (6 of 7 labs) use calibration offsets and check thermocouple/RTD input
  - Thermocouple/RTD input checked annually (3 of 7), monthly (2 of 7), daily (1 of 7), or as needed (1 of 7)

# Feedback from Questionnaire

- Air hole nozzle
  - All labs clean the nozzle either daily or weekly
  - 6 of 9 labs do not check that the nozzle holes remain constant
- Vacuum pressure
  - All labs use a manometer to measure vapor pressure
  - Vapor pressure is checked daily (4 of 9), weekly (1 of 9), before and during run (1 of 9), as needed (2 of 9), before run (1 of 9), or annually (1 of 9)
  - Manometer fluid is replaced primarily on an annual basis (5 of 8)
- Air leaks
  - 8 of 10 labs routinely check for leaks by looking at the tubes, glassware, and seals. 1 of 10 labs follows D5800 28.20 (Procedure C)
  - Most labs use the reference standard to determine if there are air leaks

# Feedback from Questionnaire

- Air filter cartridge
  - 4 of 8 labs have instruments with air filter cartridges
  - Cleaned or replaced as needed (3 of 4) or every 6 months (1 of 4)
  - Cleaning process uses solvent and drying either in air or an oven
- Coalescing Filter
  - 4 of 8 labs have instruments with a coalescing filter
  - Filter is cleaned as needed (2 of 4), after every run (1 of 4), or biweekly (1 of 4)
  - 2 of 4 labs report seeing a change in the sample weight loss after the filter is changed, while all labs check the vacuum and verify reference oil performance after changing the filter
- Electronics warm-up
  - All labs allow electronics warm-up
  - 3 of 10 labs indicate that the instrument is always on

# Feedback from Questionnaire

- Pressure/Temperature Graph
  - 6 of 8 labs check the temperature and pressure graph for the instruments
    - 2 labs report pressure fails, 2 labs report temperature fails, and 2 labs reports fails for both pressure and temperature
  - 5 of 7 labs report rejecting results based on the graph, while 2 labs report taking corrective action
- Reference oils
  - 7 of 10 labs use various CEC reference oils while the other 3 use another reference oil (Tannas or ISL reference)
  - 4 of 8 labs use a QC sample, with those 4 labs using both the QC and reference oils
  - Labs without QC sample: Reference run daily (2 of 4 labs) or after every 10 runs (2 of 4 labs)
  - Labs with QC sample: QC run daily with reference being run as needed (4 of 4 labs)



# Feedback from Questionnaire

- Condensate bottle
  - 7 of 9 labs do not clean the condensate bottle before every run
  - Bottle is emptied at half inch level (2 of 7), daily (2 of 7), every four days (1 of 7) or when it reaches the fill line (1 of 7)
- Sample Weight
  - All labs measure the initial and final tare weights without the lid and to the nearest 0.01g
- Lid inspection
  - 6 of 9 labs inspect the lid for oil at the end of the test
  - If oil is present on the lid, 4 of 6 labs rerun the sample while 2 of 6 labs clean the lid, but retain the result

# Feedback from Questionnaire

- Cup Holder
  - All labs report using a supported cup holder at the start and the end of the test
- Vacuum disconnect
  - All labs report disconnecting the vacuum within 15 seconds from the end of the 60 minute heat cycling
- Water cooling
  - All labs report water cooling at the end of the test, with 8 of 9 reporting the time to be within a minute
  - Cooling is done by ice in water (3 of 9), cold water from tap (4 of 9), room temperature water (1 of 9) or water checked by a thermometer (1 of 9)
  - All labs are waiting at least 30 minutes before performing the EOT measurement with the cup being completely dry

# Summary

- 21 instruments were covered through the questionnaire, over half which were installed in 2009 or later
- A number of differences among the labs were found from the questionnaire results
  - Impact of draft shields
  - Impact of extraction tube
  - Impact of air hole nozzle
  - Impact of filters
  - Impact of different reference/QC oils
  - Impact of different water for cooling