Waste/Side Stream Testing Guidelines at Pfizer

Jeffrey B. Sperry, Ph.D.
2017 Process Safety Forum
28-Sep-2017
Outline

– Where we manufacture and generate waste
– How different sites handle waste (side) streams
– How we test waste streams
– Questions
Sites Involved in Tech Transfer

La Jolla, CA (CRD) 
Coming soon!

Kalamazoo, MI (PGS)

Groton, CT (CRD)

Little Island, Ireland (PGS)
Ringaskiddy, Ireland (PGS)

Sandwich, England (CRD)
Capabilities

- Groton, CT (Kilo-Lab)
  - 100-200L glass-lined carbon steel reactors
  - 75L Hastelloy reactor
  - 30L Hastelloy hydrogenation vessel
  - 5-20L glass vessels

- Sandwich, UK (Pilot Plant)
  - 250-2500L glass-lined carbon steel reactors
  - 250L Hastelloy (cryo)
  - 250L Hastelloy hydrogenation vessel
Waste Storage Guidelines

• Groton, CT (Kilo-Lab)
  – *All waste* from chemical production is drummed and sent out for incineration
  – Waste is drummed in 55 gallon poly-lined carbon steel containers
  – Significant Process Safety testing required to ensure safe storage and transport

• Sandwich, UK (Pilot Plant)
  – *Almost* all waste from chemical production is sent to one of two underground storage tanks (Organic and Aqueous)
  – All aqueous waste must be pH 5-9
  – All organic waste must be neutralized (no active species present)
  – Processes must be developed to neutralize waste
  – Waste sent for incineration off site
HOW DO WE TEST WASTE (SIDE) STREAMS?
Thermal Screening Unit (TSu)

TSu Design:

- 8 mL high pressure test cells
- Sufficient volume for truly representative samples, and the study of liquids, solids and reaction mixtures
- Temperature from ambient to 400 °C
- SS, Hastelloy, glass, titanium, carbon steel
Thermal Screening Unit (TSu)

Applications
• Thermal stability of feeds, intermediates and products
• Test liquids and solids
• Evaluate long term exposure at elevated temperatures (isothermal test)
• Evaluate safe operating and storage temperatures, as well as the consequences of a runaway reaction

Key Data
• Onset temperature of exotherm
• Rate of temperature rise
• Rate of pressure rise
• Maximum temperature and pressure
• Time from exotherm initiation to maximum rate
• Margin of Safety for TSu thermal onset is 75 °C
Waste (Side) Stream Testing

- Waste (side) streams generated in Groton KL are tested when any of the following criteria are met:
  - Process uses or generates gas
  - Process uses a reducing agent
  - Process uses an oxidizing agent
  - Process uses materials with HEFGs
  - Process uses carbonate or bicarbonate

- All waste streams generated in Sandwich Pilot Plant that are destined for drums are tested
Waste (Side) Stream Testing – TSu Methods

- Ramp TSu from 30 °C to 200 °C using either glass or Hastelloy test cell after considering MOC
- If the residual pressure at the end of the test is ≤ +0.2 bar then okay to drum
  - Recommendation made: “acceptable to store and ship under ambient conditions in sealed containers for up to one year”.
  - If positive residual pressure observed at the end of the test, then stream "fails ramp test“ and must undergo isothermal test
- Isothermal TSU at 60 °C for 24 hours is needed using a glass or Hastelloy test cell after considering MOC
- If the residual pressure at the end of the test is ≤ +0.2 bar then okay to drum
  - Recommendation made: “acceptable to store and ship under ambient conditions in sealed containers for up to one year”
  - If positive residual pressure observed recommendation will be made: "not suitable for ambient drum storage as tested"
Example: A side steam that passes the ramp test

Pressure change = -0.07 bara @ 30.1°C
Waste (Side) Stream Testing

- Example: A side steam that passes the ramp test

![Graph showing temperature and pressure changes over time with a note: Pressure change = -0.07 bara @ 30.1°C]
Waste (Side) Stream Testing

• Example: A side steam that **fails** the ramp test
Waste (Side) Stream Testing

- Example: A side steam that **fails** the ramp test

![Graph showing temperature and pressure changes over time](image)

- Pressure change = 0.43 bara @ 30°C
• Example: A side steam that **fails** the ramp test
• Example: A side steam that passes the isothermal test

Pressure change = 0.02 bara @ 30°C
Waste (Side) Stream Testing

• Example: A side steam that passes the isothermal test

![Graph showing pressure change and temperatures over time with a note: Pressure change = 0.02 bara @ 30°C]
Thermal Screening Unit (TSu)

- Example: A side steam that **fails** the isothermal test

Pressure change = 0.48 bara @ 30°C
Thermal Screening Unit (TSu)

• Example: A side steam that **fails** the isothermal test

\[
\text{Oven Temperature (°C)}
\]

\[
\text{Sample Temperature (°C)}
\]

\[
\text{Pressure (bara)}
\]

\[
\begin{align*}
70 &\quad 60 &\quad 50 &\quad 40 &\quad 30 &\quad 20 &\quad 0 \\
2.5 &\quad 2 &\quad 1.5 &\quad 1 &\quad 0.5 &\quad 0 \\
0 &\quad 200 &\quad 400 &\quad 600 &\quad 800 &\quad 1000 &\quad 1200 &\quad 1400 &\quad 1600 \\
\end{align*}
\]

\[
\text{Pressure change = 0.48 bara @ 30°C}
\]
Thermal Screening Unit (TSu)

- Bulging drums

+0 bar
Thermal Screening Unit (TSu)

- Bulging drums

+1 bar
Thermal Screening Unit (TSu)

- Bulging drums

+2 bar
Thermal Screening Unit (TSu)

- Bulging drums

+3 bar
Thermal Screening Unit (TSu)

• Bulging drums

+4 bar
Thermal Screening Unit (TSu)

- Bulging drums

+5 bar
Conclusions

• Groton, CT Kilo lab uses 5 criteria for testing drummed waste/side streams:
  • Process uses or generates gas
  • Process uses a reducing agent
  • Process uses an oxidizing agent
  • Process uses materials with HEFGs
  • Process uses carbonate or bicarbonate

• Sandwich Pilot Plant sends almost all waste to one of two storage tanks
  – All waste to be sent out is tested. All side streams held in sealed drums is tested

• TSu used to study pressure generation
  – Test 1: Ramp test from 30 °C to 200 °C, if it fails then
  – Test 2: Isothermal test at 60 °C for 24 hours
  – If pressure change at ambient temperature is \( \geq 0.2 \) bar then contents are not safe to store as tested
QUESTIONS?