

C6K validation tasks to be outsourced

P. Gorbounov, 25/06/2015, PFC-Alternative(s) General Meeting

Global validation tasks/problems

- Initial coolant composition and water contamination effects
- Radiation effects
- Compatibility with materials
- Purification methods
- Monitoring (sampling, online) – *at CERN?*

- What if C6K fails to be validated (other Novec fluids?)

General remarks

- Validation program depends on the cooling scenario assumptions
 - (C6F14 study, PG) – stress on initial and online purification
 - (The community) – stress on the compatibility of the system materials with hydrolysis and radiolysis products
 - Accept big liquid water accidents or not?
- The requested actions should be not over-defined, some degree of freedom (element of R&D) should be left to contractors
 - Methods of composition analysis (spectro- , chromo- ...)
 - Test setup configuration
- Currently have established contacts with: **3M** (USA), 3M (Switzerland), **Dynalene** (USA, coolants, filters, corrosion study), ILK (Dresden)
- To be done: contacts with Swiss academic labs

Composition and water-related effects (I)

Water: the contaminant that will have detrimental effects both for low- and high-dose application (C6F14 study, cooling experts, my presentation of 18.06 “C6K and Water”)

- | | |
|-------|--|
| C6K | a) Standard (as provided) |
| | b) Purified in situ (activated carbon, silica gel, Drierite) at room T |
| | c) Purified and partially and fully saturated with humid air (e.g., 10 ppm and 20 ppm) |
| | d) Purified, “saturated” with liquid water (to achieve pH=1 in the aqueous phase) |
| Water | e) Aqueous phase after d) |
| Gas | f) Head space after d) |

Actions:

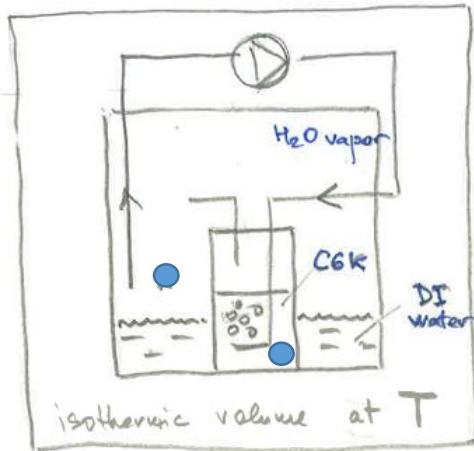
- Detailed spectrometric (IR, FTIR, UV-VIS, NMR etc) and/or chromatographic (GC, GC/MS, GC/??, HPLC etc) of a) and b), with identification of C6K and all contaminants
- Identification of all C6K hydrolysis products (PFPrA, HFC-227ea) + fluoride and HF in c)...f)

Composition and water-related effects (II)

Study of water solubility and methods of moisture monitoring

- a) Absolute water content (ppm) and Relative water saturation, as functions of time and T
- b) Effects of direct contact with liquid and solid water phase
- c) Test usability of capacitive humidity sensors/probes with C6F14 and C6K

Actions:



T = 30°C
20°C
10°C
0°C

● - RS capacitive sensors

$$\text{ppm(abs), RS} = f(t, T)$$



$$pH_{(aq)} = f(t, T = 20^\circ\text{C} < 0^\circ\text{C})$$

$$P_{(g)} = f(t, T)$$

Radiation effects

Actions:

- Detailed spectro- and/or chroma- composition analysis, with identification in all contaminants in irradiated samples and corresponding head space gas
- Comparison with the corresponding initial (non-irradiated) samples

Doses (γ): 0 Gy (in vials), 500 Gy (typical of low-dose apps), 5 kGy, 20-50 kGy

Samples: Purified C6K and standard (option: purified vapour-saturated)

Redundancy: x2

3M has already expressed an interest in performing this study at St. Paul (USA)

Material compatibility (I)

Accelerated “ageing” tests according to industrial standards, e.g. AST-MD-471-72 (like with C6F14). 1000 h at 80°C under pressure (or relaxed: 500 h at 50°C)

Material list (compiled by Dina)

- 1) Polyethylene high density (UHMWPE))
- 2) NBR (Nitrile Butadien Rubber)
- 3) Graphite
- 4) Stellite
- 5) Peek
- 6) gylon

Pipes (compiled by Dina)

- 7) brass
- 8) stainless steel 304 %306
- 9) titanium (Gr. 5!)
- 10) copper
- 11) steel
- 12) multilayer pipe (geberit or Alupex)
- 13) polyethylene

Extra materials (proposed by PG)

- High-purity, platinum-cured, non-plasticized silicone
- ???

Fluids

- C6K as provided (99.8%)
- C6K fully saturated with water vapour
- PFPrA aqueous solution at pH=1

Remark: not all plastics or elastomers are usable at <-40°C

Material compatibility (II)

Actions (tests after the exposure)

- SEM surface inspection for metals and hard plastics
- Volume and weight change
- Hardness (and elongation for elastomers)

ILK (Dresden) and any industrial certification lab (CETIM-CERMAT, SGS ...) can do this.

Purification

Coolant and the expansion tank head space!

Potential contractors: 3M, Dynalene, academic labs

Actions

- Study molecular sieves (MS) compatibility with C6K (at 25, -30, -50°C)
- Study ion-exchange resins (IER) performance and usability at low temperatures. IER can be an alternative to MS, are ~compatible with C6K (evidence by 3M!), can be efficient desiccants (cation types) and acid removal agents (anionic types)
- Validate common desiccants: Drierite, silica gel (recommended by 3M)
- Close-circuit venting of expansion tank with dry N₂ (through head space only, or through liquid + head space), with filtration (*membrane?*) – to remove H₂O, O₂, HF, COF₂. The solution has to prevent C6K vapor removal!