Webinar on “test gas generators”

CEA Grenoble

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Outline

1. Introduction
2. System based on entrainment of saturated gas
3. System based on syringe into vacuum vessel
4. Conclusion

1- Introduction

- **Gas generators are necessary**
  - For component not available in gas bottle tank (i.e. condensable gas as naphthalene, methanol…)
  - For expensive components (furfural…)
  - For on line gas analyzers calibration
- **CEA needs**
  - Study naphthalene and benzene kinetic decomposition in a simulated syngas
  - Study kinetics of gas release during torrefaction process
- **CEA has developed two kinds of gas generators**

1. System based on entrainment of saturated gas
2. System based on syringe into vacuum vessel

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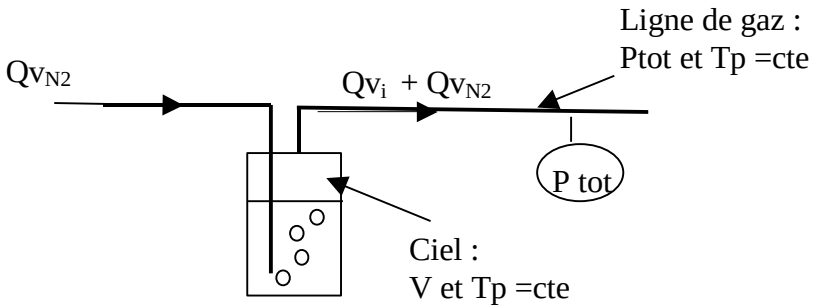
2-System based on entrainment of saturated gas

- **Naphthalene and benzene kinetic decomposition study**
  - high temperature tar craker PEGASE facility
  - with a simulated syngas from bottle gas (H_2, CO, CH_4, H_2O, C_2H_2, C_2H_4, C_2H_6..)
- **For PEGASE facility we need a gas generator**
  - Resistant to pressure > 1 bar
  - Able to give very high concentrations of benzene or naphthalene (**dilution about 100**)
  - To give continuous stable tar flow rate for several hours

Entrainment of the saturated vapor pressure by a carrier gas (nitrogen)

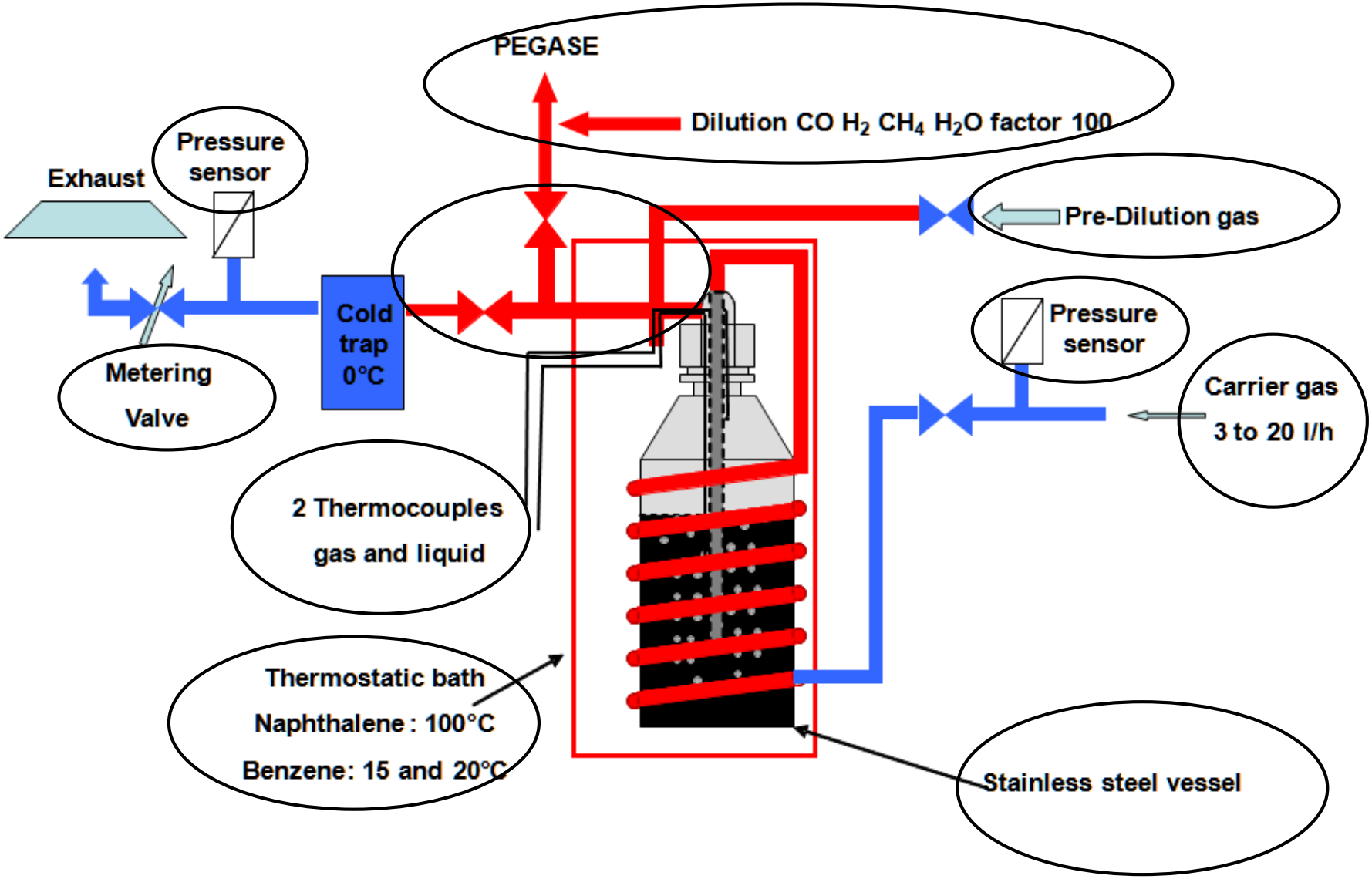


Volume concentration = $Qv_i / (Qv_i + Qv_{N2}) = Psat_i / Ptot$

Temperature **Total pressure**

Development of a gas generator for "high" pressure and high concentration

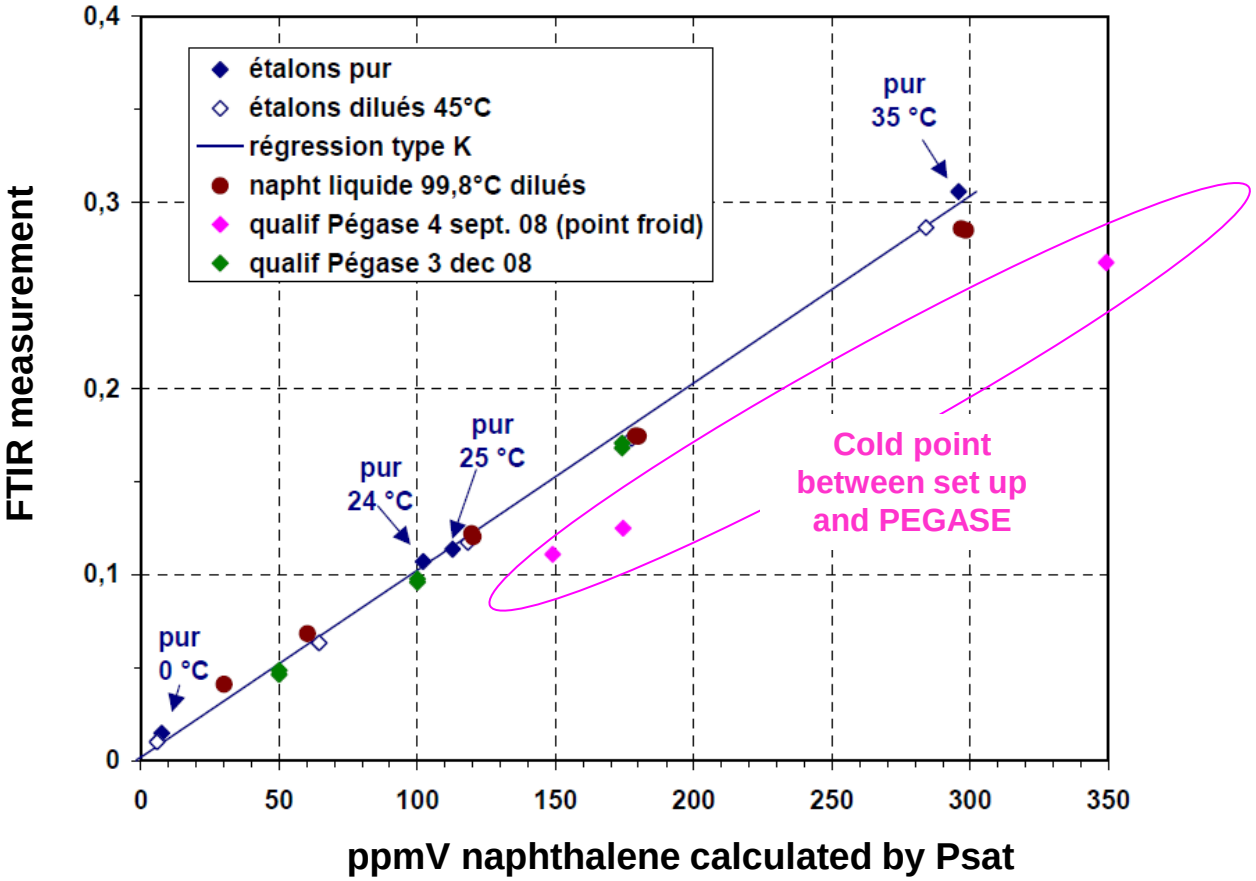
2-System based on entrainment of saturated gas



2-System based on entrainment of saturated gas

Naphthalene

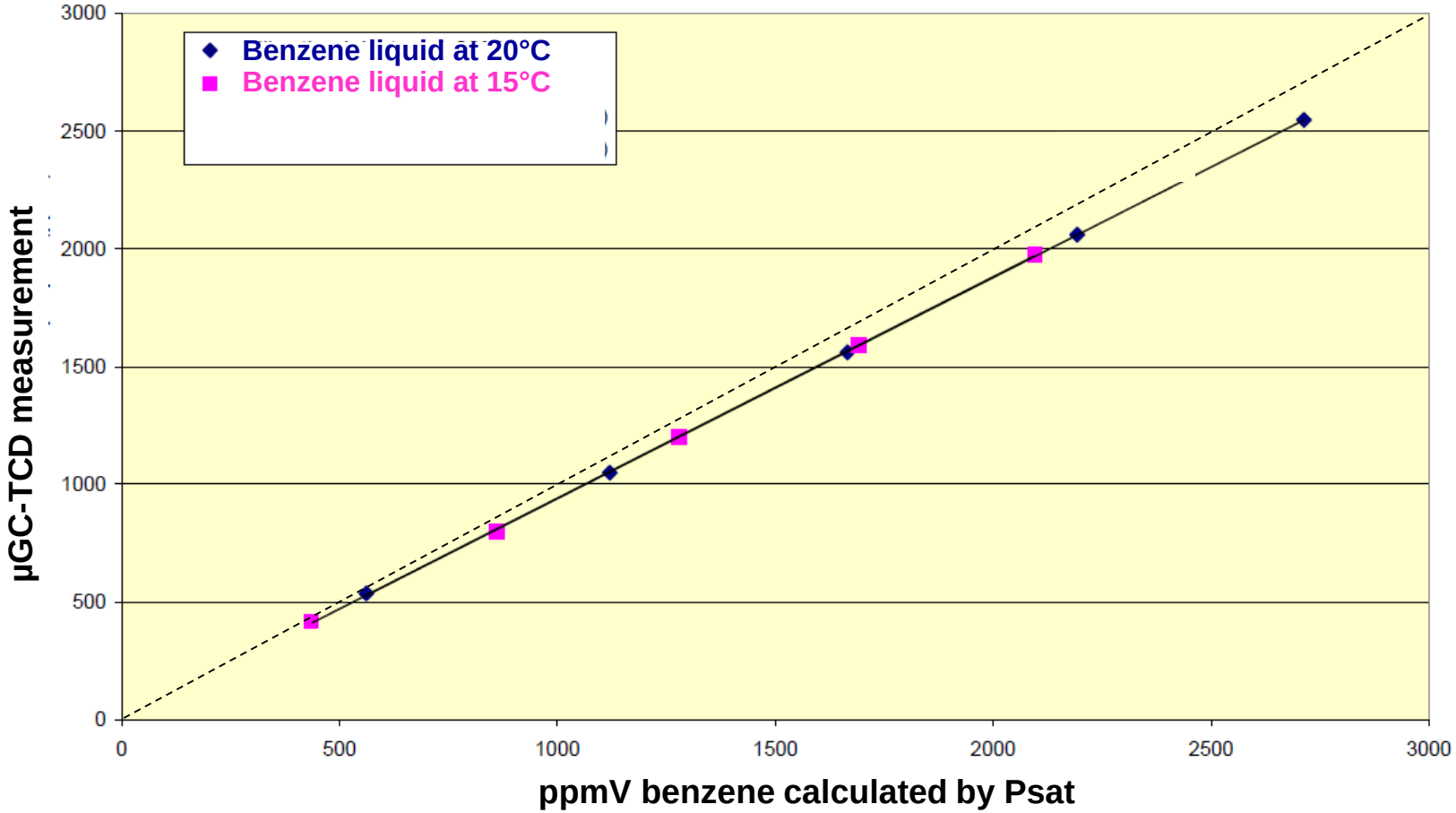
- Set up qualification
  - **Without dilution (19000 ppmV at 1.3 bar or 25000 ppmV at 1 bar)**
    - By weight of condensate
  - **With dilution in PEGASE facility (cold) 0 - 300 ppmV**
    - By FTIR (calibrated with another set up based on saturated gas above naphthalene solid)



2-System based on entrainment of saturated gas

Benzene

- Set up qualification
  - **With dilution in Pegase (cold) 500 - 3000 ppmV**
    - By µGC-TCD with calibration bottle tank at 3000 ppmV



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3- System based on syringe into vacuum vessel

- **For kinetics study of gas release during torrefaction process**
- **During torrefaction we need to follow on line by FTIR some condensable species as:**

	$T_{boiling}$ (°C)	Range of calibration (ppmV)
$C_2H_4O_2$ Acetic acid	118	100 to 3300
CH_2O_2 Formic acid	101	40 to 1000
H_2O Steam	100	0 to 8000
CH_3OH Methanol	65	100 to 1100
C_2H_4O Acetaldehyde	20	60 to 600
CH_2O Formaldehyde*	-19	100 to 2200

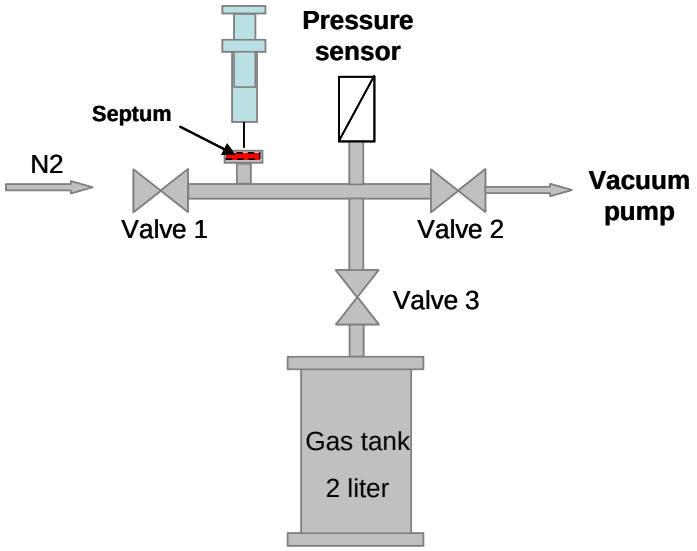
** 37% CH_2O + 53% H_2O + 10% CH_3OH*

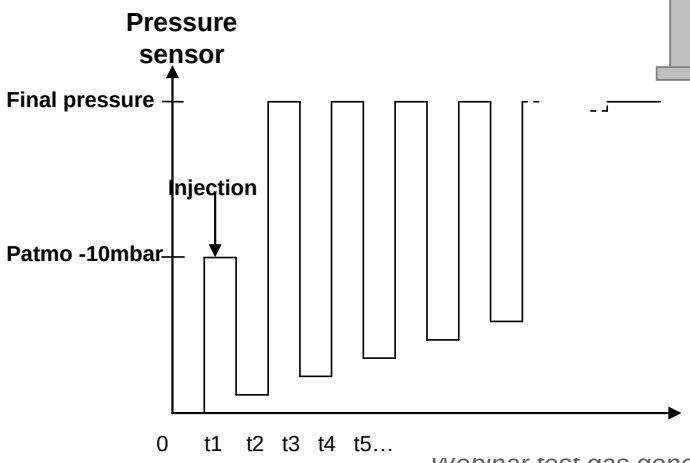
Development of a gas generator for these species to calibrate FTIR

3- System based on syringe into vacuum vessel

- **Need a gas generator**
  - For pure component (gas not polluted by solvent)
  - With about 6 liters of calibrated gas

Filling the Gas Tank





1_ t=0 : V2, V3 open, vacuum is done

2_ t1 : close V2, V3 and open V1 until pressure is slightly below atmospheric pressure.

3_ injection of the liquid by the syringe

4_ t2 : open V3 and then close

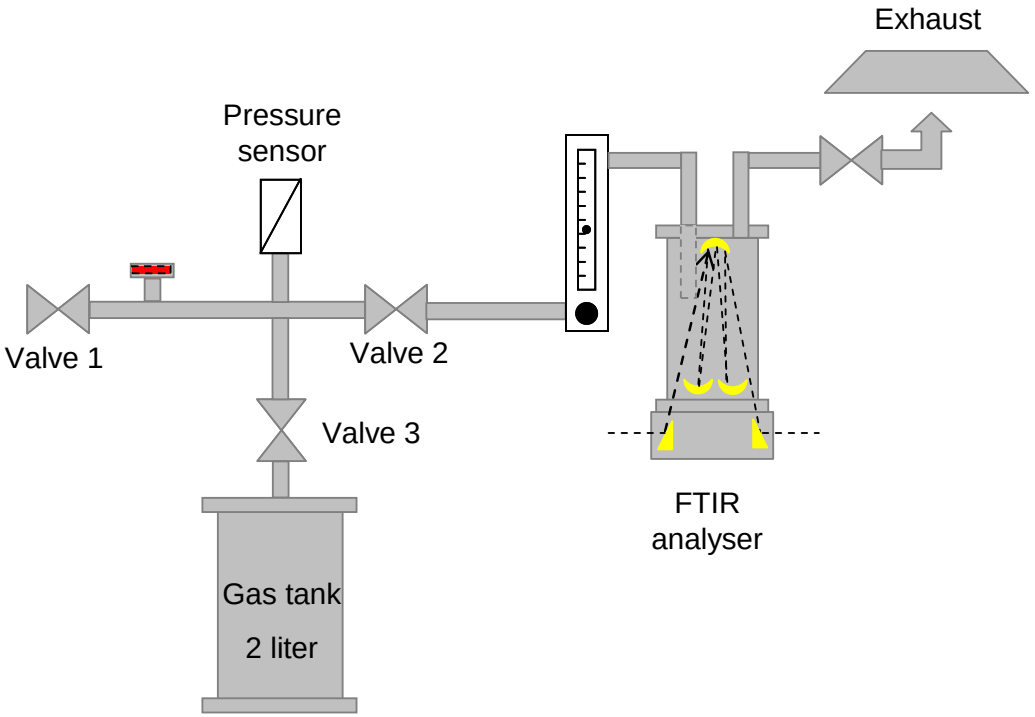
5_ t3 : open V1 for N2 injection to the final pressure and then close V1

6_ t4 : open V3 and then close

And repeat steps 5 and 6… until you reach the final pressure

3- System based on syringe into vacuum vessel

FTIR calibration



- **Syringe + vacuum system validated by comparing with:**
  - Other gas generator (bubbling) for steam, acetaldhehyde, methanol
  - FTIR integrated values with cold trap residue + GC-MS
- **Problem with formaldehyde (adsorption on the surface of the vessel in stainless steel)**

4- Conclusions

CEA has developed two kinds of gas generators

1. System based on saturated gas working at pressure (1.3 bar) for high concentration (naphthalene and benzene)
2. System based on syringe into vacuum vessel for condensable components as acetic and formic acid or acetaldehyde or formaldehyde

Thanks to these systems scientific studies could be performed

- naphthalene and benzene kinetic decomposition study in the high temperature tar craker PEGASE facility
  - **presented in 17th EBCE Hambourg by S. Valin & Al**
- kinetics study of gas release during torrefaction process (T. Nocquet thesis)
  - **presented in 20th EBCE Milan and in 21th EBCE Copenhagen**

Thank you for your attention

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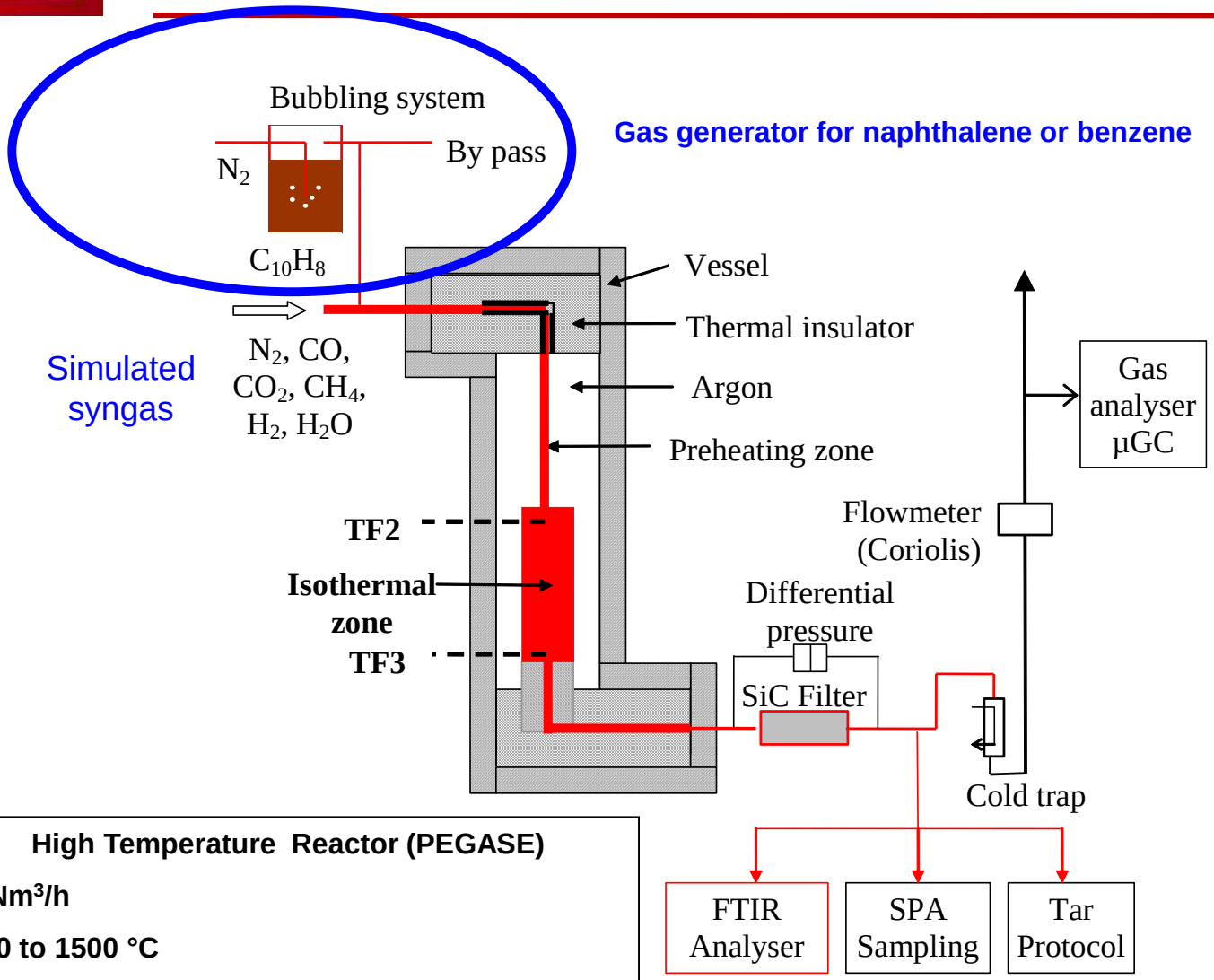
Summary

1. System based on saturated gas working at pressure (1.3 bar) for high concentration (naphthalene and benzene)
   1. **Build for simulating a real gas under repeatable conditions**
   - **System chosen because it allows high concentration, high pressure and long duration stable test reached in few minutes**
   - **Checked for accuracy by on line measurements (FTIR or µGC-TCD)**
   - **Pure component are chosen because Solvent are not present in the real syngas**
   - **Can be used with any other component that is preferably liquid**
     - 100°C is a maximum temperature to be comfortable with cold point
     - Naphthalene is the worst case for cold point
     - Check compatibility between the material of the vessel and the compound to be analyzed
2. System based on syringe into vacuum vessel for condensable components
   1. **Build for calibration on line analyzer (FTIR)**
   - **System chosen because a small quantity of gas is necessary (~ 6 liters), reproducible, accurate (syringe)**
   - **Checked for accuracy by other gas generator (example saturated gas)**
   - **Pure component are chosen because:**
     - Solvent disturbs FTIR signal
     - Mixture of components are possible (as formaldehyde + H_2O + methanol)
   - **Can be used with any other component that is preferably liquid**
     - Check the saturation vapour pressure
     - Check compatibility between the material of the vessel and the compound to be analyzed

PEGASE facility



High Temperature Reactor (PEGASE)

- **2 Nm³/h**
- **900 to 1500 °C**
- **1 to 4 bars**
- **Working alone with gas bottle or linked to high temperature fluidized bed (LFHT)**