Closed Loop Development Tests of an Evaporating Experiment
for the
International Space Station Fluid Science Laboratory

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Abstract

CIMEX-1 (Convection and Interfacial Mass Exchange) is a microgravity research project foreseen to be carried out onboard the Columbus Module of the International Space Station. The project is sponsored by the European Space Agency and EADS Astrium is the prime contractor for developing the experiment. The main scientific purpose of the mission is studying mass transfer processes through interfaces and their coupling with the surface tension driven instabilities that affect mass and energy transfer. The experiment takes place in a cell, in which the liquid interface allows evaporation to take place through a flow of inert gas. The system is equipped with a liquid and gas closed loop in order to avoid the limitations caused by the use of consumables. The assignments which were carried out were building, testing and analyzing a breadboard setup which is meant to verify the closed loop and the components operability, as it has the same functional characteristics of the flight model. Due to the decision of changing the evaporating fluid from Ethanol to HFE-7100, the system behavior and operability had to be tested. A test campaign took place in June 2011 to collect experimental data and to verify the operability of the CIMEX-1 foreseen parameters range with the new fluid. Moreover, the test campaign aimed to assess the properties of new essential components like the liquid pump and the anti-wetting micro groove.
This work was carried out in the TO-52 department of Astrium Space Transportation in Friedrichshafen, Germany.

Symbols

Symbol	Description	Unit
$\grave{C}$	Concentration	%
Cp	Specific heat (const. pressure)	kJ/(kg K)
H	Specific enthalpy	kJ/mol
M	Molecular Weight	kg/kmol
$\dot{m}$	Mass flow rate	mg/min
$\dot{V}$	Volumetric flow rate	ml/min
y	Mole fraction	/
ρ	Density	kg/ m3
η	Efficiency	/

Introduction

Evaporation and heat transfer processes are common in many aspects of technology, science, nature and medicine. Industrial applications like heat pipes and cooling devices are dependent on the heat transfer and evaporation processes. Nowadays in the industry, there is a lack of fundamental knowledge which does not permit the optimization of the existing processes. Their models are, in fact, too dependent on empirical correlations with a consequentially limited range of applicability. Therefore, in 2002 many European research institutes, universities and companies started the development of CIMEX-1, a project foreseen to take place in the International Space Station (ISS) and sponsored by the European Space Agency (ESA). CIMEX-1 is meant to study mass transfer processes through interfaces and their coupling with the surface tension driven instabilities that affect mass and energy transfer. The undertaken investigation is about both the macro convection (which occurs when heat or mass fluxes are imposed along an interface) and micro convection, (which is about fluxes across an interface). The project encountered an important setback after the Safety Review 1, when Ethanol, the foreseen operative fluid, was not considered safe. A new liquid was therefore needed and after the assessment of alternative fluids, HFE-7100 was selected. Since the CIMEX components were designed or chosen for Ethanol, their functionality and operability had to be tested with the new liquid. Moreover, their behavior also had to be verified in the closed loop arrangement, since all of the equipment has to work together in CIMEX-1. If the system functionality of CIMEX-1 with the HFE-7100 was discovered to be limited, with a reduced range of the foreseen parameters, then finding a new solution would be necessary for the program to continue.

Objectives

The main assignments of this project were building, testing and performing an analysis of the CIMEX Closed Loop Breadboard. In the space industry breadboards (BB) are ground tests which simulate the real experiment to be sent to orbit. Their purpose is to reduce the design risk and expand the knowledge of the system for a better understanding of its behavior.

In the particular case of the Closed Loop BB, the aim is to verify the closed loop performances of CIMEX using the HFE-7100 fluid. This new liquid is meant to substitute the previous operative fluid which was Ethanol, as requested after the safety review 0/1. The target of this BB is to test the foreseen parameters, previously established for Ethanol, to verify system compatibility and/or to eventually find new operative ranges in order to guarantee the system stability (this condition is essential to perform the scientific measurements).

CIMEX-1 Description

Overview

The scientific experiment will be executed inside the Fluid Cell Assembly which is composed by the followings elements:

- Reservoir for the experiment liquid with the capability to allow a refill on-ground;
- Reservoir for nitrogen gas, which will be reused during the entire mission;
- The experiment cell: It is the core of CIMEX-1, providing the experimental volume within which the evaporation phenomenon to be studied takes place. The experimental liquid is placed in a chamber which is separated from the gas flow by a stainless steel thin foil. The metallic foil has a square opening that represents the interface between the liquid and the gas flow. A gas channel allows the gas to flow over the liquid surface which is kept steady by constant injection of experimental fluid. See configuration in Fig.1;
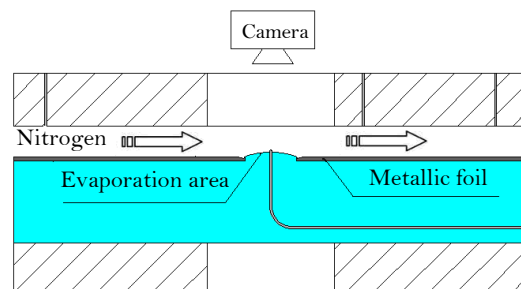

Figure 1: Experiment cell configuration

- The condenser separator subsystem (CSS). It allows the CIMEX-1 experiment to be executed in a closed loop mode without the limitation caused by the use of consumables. The main objective of the CSS is to decrease the vapor quality and to separate the vapor phase to the gas phase. The vapor from the evaporation cell enters the condenser, kept at a temperature of -10℃, and condensates due to the low temperature. A capillary channel film separator collects the liquid which is extracted from the bottom. The condensed liquid in the film separator meniscus is retracted by the liquid pump and transported back to the experiment cell. The amount of liquid in the CSS is visible thanks to a window, which show the meniscus position of the separator. See concept in Fig.2.
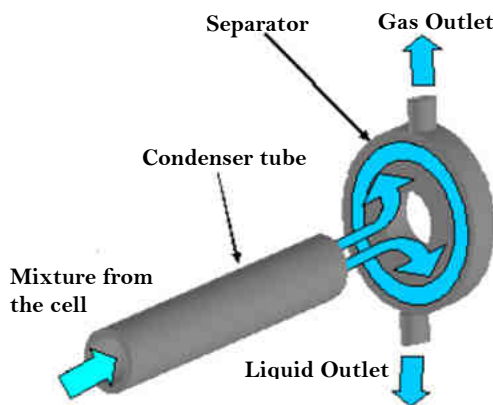

Figure 2: Condenser-Separator concept

- The liquid loop: it provides the initial filling of both the evaporation cell and the CSS from the liquid reservoir, as well as feeding the liquid chamber while the evaporation phenomenon is taking place. Moreover, it maintains the meniscus level of the CSS separator between two limits in order to assure the correct condensation, separation and extraction processes. This subsystem plays a key role in the whole BB steady state status;
- The gas loop: it guarantees the Nitrogen flow over the liquid surface of the evaporation cell chamber and it also permits adjustments of the flow rate according to the current experiment parameters. It provides the mixture gas flow from the evaporation cell to the CSS subsystem. Different pressures settings are possible thanks to a pressure control loop integrated in the gas loop subsystem. The gas

retraction pump and the pressure controller are the main components of this control loop.

Main components description

Gas circulation pump
It provides the gas flow passing over the experimental cell liquid and going from the evaporation cell to the CSS subsystem.

Gas retraction pump
It allows, together with the pressure control loop, changing the system pressure.

Liquid pump
It provides the liquid flow from the CSS to the evaporation cell in order to feed the liquid chamber when the evaporation phenomenon is taking place. The pump is also used to fill the cell during the system initialization, when the liquid chamber is still empty. The speed control is possible thanks to an external analog signal and the direction of the flow is reversible. This last feature helps the operator to better maintain constant the liquid level of the square opening.

Metallic foil with micro groove
The metallic foil, seen in Fig.3, separates the liquid chamber from the Nitrogen flow and provides a square opening (10x10mm) through which the liquid evaporates. In order to permit a correct evaporation, the liquid interface must be flat. The requirements specify a surface flatness of ±0,1mm. For this reason the metallic foil has to behave ideally as a non-wetting system.

Figure 3: Metallic foil with micro groove

Various solutions, like covering the metallic foil with special coatings, were tested in the past, but the

results were not sufficient. A new anti-wetting concept was therefore introduced which consists of creating a groove all around the edge of the square opening. The performances of the new anti wetting system was demonstrated with a dedicated stand alone test

Gas concentration sensor

The gas concentration sensor (GCC) is meant to measure the HFE-7100 vapor concentration at the entrance of the experiment cell.

Mass flow meter/controller

The mass flow controller (MFC) controls the gas mixture flow rate entering the experimental cell, while the mass flow meter (MFM) measures the mass flow rate exiting the cell. These devices use a measuring principle based on the heat conductivity of fluids to determine the mass flow. Thanks to the gas concentration sensor (GCS), the mass flow meter and the mass flow controller it is possible to establish the evaporation rate taking place in the evaporation cell.

Stand alone metallic foil tests

This test was performed in order to check the behavior of the anti wetting groove. According to the requirements, the new metallic foil should allow the liquid not to spill out from the square opening. To prove this, a thermocouple was placed in the middle of the metallic foil window and its tip was placed 1 mm above the metal foil level. The liquid interface height was therefore measured by making a comparison with the thermocouple tip.

The final results of this test are very important for the whole project for the following reasons:

- The liquid interface flatness has to fulfill the requirement.

- The liquid interface stability has to be guaranteed by assuring that the liquid does not spill out during the measurements. Moreover, the liquid has to be kept inside the window even when the cell is eventually shaken.

Test results

The metallic foil behavior was assessed by looking at the pictures taken from the camera which is placed above the cell (see Figure 1). Moreover, the liquid level was checked by using the thermocouple placed in the middle of the square opening. The liquid creates in fact a small circle all around the thermocouple, which permits to estimate the height of the liquid by making a comparison with the probe tip. The highest achieved height was estimated as 1mm.

Figure 4: Liquid interface height achieved with the metallic foil with micro groove

A comparison with the behavior of the metallic foil without any type of anti wetting system permits a better assessment of the micro groove performances (see Figure 5).
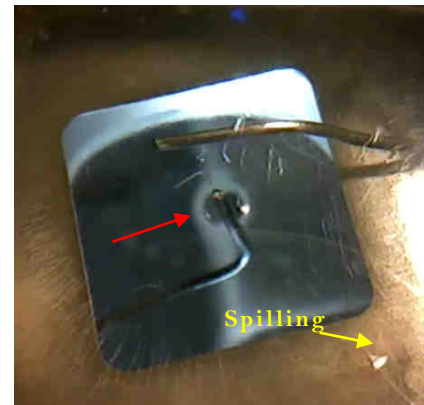

Figure 5: Liquid interface height without any kind of anti wetting system. As soon as more liquid is injected the spilling occurs.

From Figure 5, it is possible to see that as soon as the liquid interface height reaches the metallic foil level, a further injection of liquid causes the spilling. On the contrary, the metallic foil with the groove is able to keep the liquid inside the square window even when the liquid interface is well above the foil level (see Figure 4).

The anti-wetting groove tests were successfully completed. The good performances with the new metallic foil can be summarized as follows:

- In the middle of the evaporating area, a maximum height of about 1mm was achieved. Additionally, it has to be mentioned that the requirement for surface flatness is +/-0,1mm. This means that the anti-wetting groove clearly covers/exceeds the required performance;

- Stable behavior within the entire requirement of ranges of gas flow and pressure. Even with induced vibrations (e.g. shaking of the sample cell manually), the liquid was kept inside the evaporating area;

Closed loop behavior evaluation

System stability

The system stability is an essential condition that has to be verified for each single test, both during ground campaigns and during flight operations. Each measurement needs stable conditions for all of the experimental parameters such as temperatures, pressures, flow rates and gas concentration.

The main causes that affected the system stability were the following:

- *Back pressure waves caused by the gas circulation pump.* The pump induced back pressure waves on the cell interface, with a consequent loss of liquid surface flatness. The problem was solved by finding the pump speed that minimizes the wave intensity. In all of the cases, the waves were almost totally reduced;

- *Back pressure waves caused by the gas retraction pump.* This pump induced strong back pressure waves on the cell interface. Furthermore these fluctuations caused the spilling of the liquid with the consequent loss of the system stability. The problem was solved by excluding (thanks to two valves) the pressure control loop, once the desired pressure was reached;

- *Presence of leaks.* Even if much care was taken during the integration of the BB, leaks were still present in the loop and it was not completely possible to avoid them. The leaks were a cause of system instabilities because of the rising in pressure, the presence of bubbles in the cell and in the liquid loop. These issues were solved during the test campaign and all the tests were performed with stable conditions.

Boiling curve

The operability of CIMEX-1 is largely influenced by the physical properties of the HFE-7100. In fact, it is not possible to guarantee the interface flatness when the liquid starts boiling. This means that it is not possible to apply any indiscriminate combination of pressure and temperature to the system, pretending the surface to be flat.

Tests were performed in order to find the points of the curve. Table 1 shows the obtained results.

By making a comparison with values found in literature, it was observed that the boiling occurs, for the same pressure values, at lower temperatures than the pure HFE case. This behavior is probably due to the Nitrogen dissolved in the liquid. In fact, when the HFE is bubbling, the liquid also degasses. This leads to different values than the ones obtained from the literature.

Pressure [mbar]	Temperature [$°C$]
1	53.3
0.8	48
0.6	41
0.4	31.5

Table 1: Experimental points of the boiling curve

Measured raw data conversion and calculations

The raw values from the MFM, the MFC and the GCS had to be converted, since the instruments were calibrated for other operative conditions, more precisely:

- The GCS was calibrated for volumetric gas concentrations. Since the requirements impose

limitations on the mass concentration, conversion from volumetric to mass concentration was necessary;

- The MFM and the MFC were calibrated for another mixture of 95% of Nitrogen and 5% of Ethanol vapor.

Gas concentration conversion

The mixture of HFE-7100 vapor and Nitrogen was considered a mixture of ideal gases, moreover Amagat model was chosen for the calculation. The partial volume that the component "i" of mixture occupies, according to the Amagat model, could be calculated as follows:

$$V_i = \frac{n_i \cdot \overline{R} \cdot T}{p} \qquad \text{(Eq. 1)}$$

From the mass concentration definition it is possible to obtain the following relation:

$$C_{Mass} = \frac{\dot{m}_{HFE}}{\dot{m}_{HFE} + \dot{m}_{N_2}} = \frac{1}{1 + \frac{\dot{m}_{N_2}}{\dot{m}_{HFE}}} \qquad \text{(Eq. 2)}$$

The mass of the Nitrogen and the HFE-7100 can be calculated as following:

$$\dot{m}_{N_2} = \dot{n}_{N_2} \cdot M_{N_2} = \dot{n} \cdot y_{N_2} \cdot M_{N_2} \qquad \text{(Eq. 3)}$$

$$\dot{m}_{HFE} = \dot{n}_{HFE} \cdot M_{HFE} = \dot{n} \cdot y_{HFE} \cdot M_{HFE} \qquad \text{(Eq. 4)}$$

Considering that the HFE-7100 volumetric concentration, read from the sensor, is equal to the vapor mole fraction, then the Nitrogen mole fraction would be:

$$\Rightarrow \quad y_{N_2} = 1 - C_{vol} \qquad \text{(Eq. 5)}$$

Finally, the mass concentration could be calculated as follow:

$$C_{Mass} = \frac{1}{1 + \frac{M_{N_2}}{M_{HFE}} \cdot \frac{1 - C_{Vol}}{C_{Vol}}} \qquad \text{(Eq. 6)}$$

According to the Amagat model, the mass concentration of the mixture is function only of the volumetric concentration and of the molecular weights of the Nitrogen and the HFE-7100.

In order to rely on this conversion method, an experimental validation test was performed. The results of the test showed a maximum difference between the experimental and theoretical values of only 5%. It is therefore possible to say that the theoretical method approximates the real values very well. The Amagat and the Ideal gas assumptions are correct.

Calibration of the MFM and MFC

The results were first converted to the fictitious case of pure Nitrogen passing through the meters. This transformation was done by using a program from the supplier, named FLUIDAT [2], which provided a conversion factor of 1,048.

A conversion method, suggested from the supplier [1], was used for the calculations. The proposed formula in the manual for a gas mixture is:

$$\frac{1}{C_{mix}} = \frac{V_{N_2}}{C_{N_2}} + \frac{V_{HFE}}{C_{HFE}} \qquad \text{(Eq. 7)}$$

Where C_{mix} is the conversion factor for the whole mixture, C_i is the conversion factor for the "i" component of the mixture and V_i is the volumetric fraction of the "i" component in the mixture. V_{HFE} is simply the volumetric concentration read by the GCS, namely C_{vol}, and $V_{N2}=1-V_{HFE}$, therefore $V_{N2}=1-C_{vol}$.

According to [1], the conversion factor for each component of the mixture has to be calculated as:

$$C_{(1 \rightarrow 2)} = \frac{c_{p(1)} \cdot \rho_{(1)}}{c_{p(2)} \cdot \rho_{(2)}} \qquad \text{(Eq. 8)}$$

Where (1) is the gas to be calibrated and (2) is the gas to be measured. C_p is the specific heat at constant pressure and ρ is the gas density.

For $C_{N2 \rightarrow HFE}$ calculation, $C_{p(N2)}$ was obtained from the ideal gas table A-21 of reference [3] and $\rho_{(N2)}$ was calculated using the ideal gas state equation. $C_{p(HFE)}$ was calculated using the HFE-7100 vapor table and the definition of specific heat:

$$c_p = \left. \frac{\partial h}{\partial T} \right|_p \qquad \text{(Eq. 9)}$$

$C_{N2 \rightarrow N2}$ is one since the conversion is between two identical gases.

From the obtained results, it was discovered that the influence of the temperature and pressure on the conversion factor is negligible compared to the

influence of concentration. The conversion factor variations are only 1,5% throughout the temperature range, and only 1,4% throughout the pressure range. The conversion factor was therefore correlated only with the volumetric concentration, being sure that the final error will be negligeble. Figure 6 shows the final graph conversion factor-volumetric concentration, obtained with the above calculations.
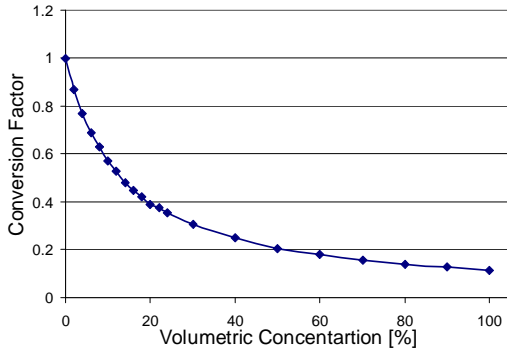

Figure 6: Conversion Factor for the MFM/MFC

Calculation of the gas concentration after the cell

The concentration value at the cell outlet is important because it allows a better assessment of the performances of the CSS. A mass balance was applied to the evaporation in order to relate the measured values of the MFM, MFC and the GCS to the unknown concentration. Considering aslo that the quantity of Nitrogen that enters the cell is the same as at the exit, the following equation was reached:

$$C_{massOUT} = 1 - \frac{\dot{m}_{MIXTUREIN} \cdot (1 - C_{massIN})}{\dot{m}_{MIXTUREOUT}} \qquad \text{(Eq. 10)}$$

Where $\dot{m}_{MIXTUREIN}$ is the value read by the MFC and $\dot{m}_{MIXTUREOUT}$ is the value read by the MFM.

Analysis of the test data

The aim of this section is to present the numerical results of the closed loop BB test campaign.

Cell behavior

The numerical results of the cell are the evaporation rate as a function of the main parameters: the cell

temperature, the gas loop pressure and the mixture flow rate.

In Figure 7, it is possible to see as the volumetric flow rate increases, the evaporating liquid flow rate also increases. Moreover, the lower the pressure, the higher the evaporation rate.
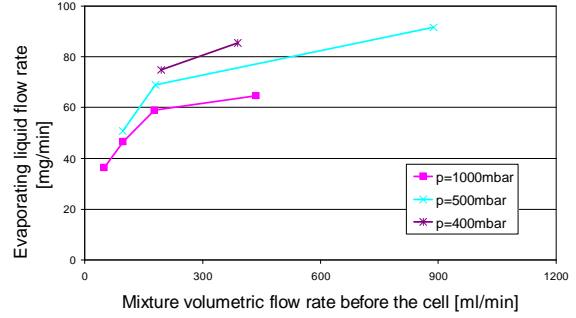

Figure 7: Evaporating fluid flow rate vs. mixture volumetric flow rate @ T=25°C
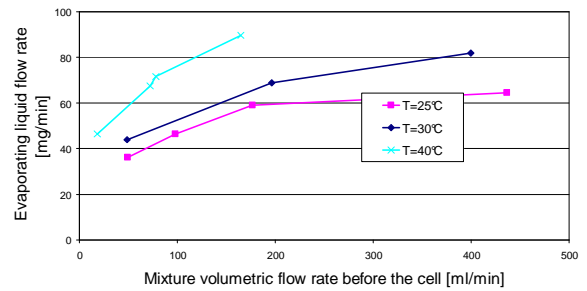

Figure 8 shows that, the higher the temperature, the higher the evaporation rate, and the higher the volumetric flow rate, the higher the evaporation rate.
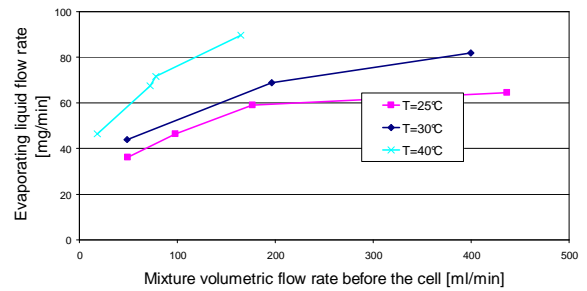

Figure 8: Evaporating fluid flow rate vs. mixture volumetric flow rate @ p=1000 mbar

CSS behavior

The CSS behavior was assessed using the efficiency defined as:

$$\eta = \frac{C_{massIN} - C_{massOUT}}{C_{massIN}} \qquad \text{(Eq. 11)}$$

This parameter was considered because it is independent of the flow rate. The efficiency is in fact

the ratio between the mass concentration reduced by the CSS and the mass concentration that enters in the condenser.

Figure 9 shows the results obtained. The information contained in the graph can be summarized as following:

- The higher the cell temperature, the lower the cell efficiency. This could be explained because of the higher evaporation rate at higher temperatures. With a higher evaporation rate, the cell efficiency decreases.

- The higher the volumetric flow rate, the lower the efficiency. This could be explained because at high volumetric flow rates, the evaporation rate increases and consequently the CSS efficiency decreases.
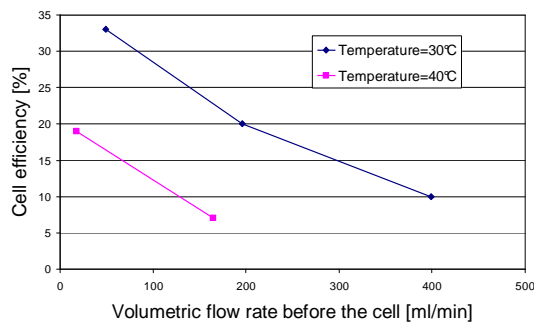

Figure 9: CSS efficiency vs. cell temperature @ 1000 mbar

Moreover, during the tests, it was discovered that the mass concentration after the CSS exceeds the 50% value for about half of the performed tests. This is very important for CIMEX-1, since the requirements state that the concentration should not exceed this value. A requirements redefinition or a new CSS design is therefore necessary.

Comparison with the Ethanol case
In 2008, VERHAERT space [4] tested the CSS with Ethanol. The results obtained with Ethanol were then compared to the ones obtained during the test campaign with HFE-7100. Figure 10 summarizes the result that VERHAERT obtained with Ethanol.
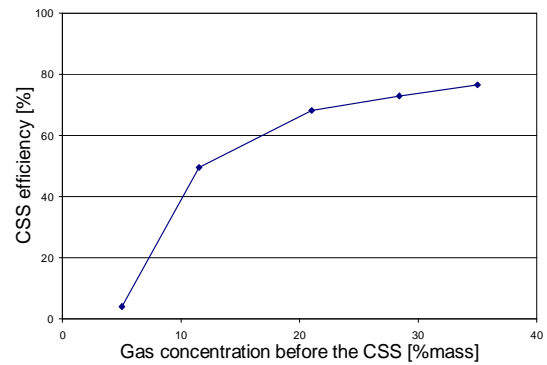

Figure 10: CSS efficiency vs. gas concentration entering the CSS, Ethanol case [5]

In Figure 11, it is possible to appreciate the CSS behavior with HFE-7100.
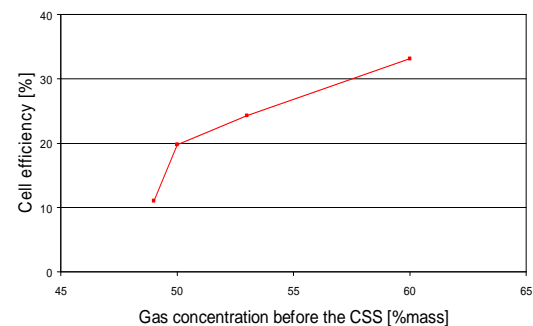

Figure 11: CSS efficiency vs. gas concentration entering the CSS, HFE-7100 case. Values obtained during the test campaign.

As it is possible to see, the CSS behavior is the same: the efficiency increases as the inlet gas concentration increases. However, the cell efficiency using HFE-7100 mixture is much less than the case with the Ethanol mixture.

A possible reason comes from the conversion factor used to obtain the mass concentration from the volumetric concentration. In fact, this transformation is affected by the molar weight ratio between the Nitrogen and the vapor, according to Eq.6. The graph in Figure 12 shows the different relationships between the mass and the volumetric concentration for the Ethanol case and the HFE case. For the same value of volumetric concentration, the corresponding mass concentration would be less in the case of Ethanol because its molar mass is only 46,07g/mol, while the HFE-7100 is 250g/mol. Therefore, the CSS would have more mass to condense in the case with HFE-7100 and the efficiency would consequently decrease.
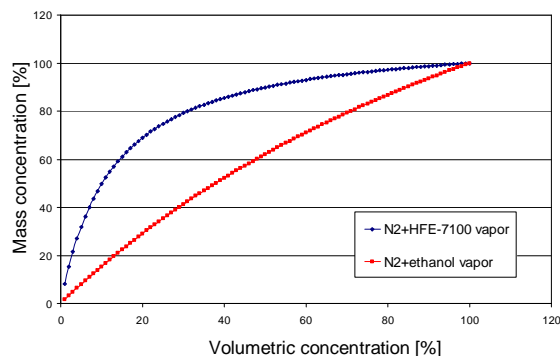
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Figure 12: Relationship between the mass and volumetric concentration. HFE-7100 vs. Ethanol.

Comments

The obtained results make physical sense but they cannot be considered valid for scientific use, since some instruments were not precise enough for scientific purposes. However, they are suitable for engineering purposes like redesigning components or proposing new requirements. In fact, one of the most important remarks of the test campaign was discovering that the mass concentration requirement was not satisfied. Therefore, a solution had to be found in order for the project to continue. Since much effort has been made for designing, building and testing the components, it was decided to change the requirements instead of performing a new design (in particular for the CSS). The next sections present a requirement redefinition which will be proposed and agreed with the European Space Agency and the teams of scientists.

System evaluation

The surface flatness and the meniscus stability were achieved by manual hand control of the injection rate. At lower pressures and high evaporation rates, the manual control becomes more challenging but it is still achievable. The system stability can be considered achieved.

The system evaporation across the loop was controlled. It was demonstrated that it is possible to recover the system to the initial operational conditions without opening the loop after chaotic conditions (e.g. boiling of HFE-7100). Moreover, if the spilling of liquid over the micro-groove was

enforced, the system recovery was demonstrated to be feasible within 1-3mins as well as a restart of the flatness control. The operability of the system and the system stability were achieved during the closed loop test campaign. From this point of view, the BB tests were successful.

Requirements redefinition

As a result of the previous findings, the following new requirements are proposed for CIMEX-1 in order to keep the already existing design and, when possible, components

The following requirements ID are intended for reference [6].

- Req. 3267:

"The CSS shall deliver at outlet a gas phase composed of at least 50% of gas. 95% is considered as a target".

Proposed values: "Up to 20% of Nitrogen (in Mass), target 95% or up to 60% of Nitrogen (in volume) target 95%".

- Req. 3205:

"The temperature setting range shall be 10℃ up to 60℃ ".

Proposed values: "10℃ to 50℃".

The hardware is designed to support a temperature up to 60℃. The upper limit of the temperature range is due to the bubbling/degassing of the HFE-7100 at 1bar, which is the maximum experiment pressure (see Table 1).

- Req. 3193:

"The gas flow rate shall be measurable in a range from 10 to max 1000 ml/min".

Proposed values: "10-1000 [ml/min]".

The Bronkhorst devices will be calibrated to pure gas (Nitrogen case), since the real volumetric flow is dependent on the gas concentration. This means post-processing of the volumetric flow will have to be considered by the scientific team during results evaluation.

- Req. 3197:

"The set pressure shall be adjustable in a pressure range from 400 mbar to 1000 mbar".

Proposed values: "Variable from 500 to 1000 mbar according to the boiling curve".

Pressure ranges are fine, with the remark that it is not possible to have all combinations with the temperatures (see Table 1).

Conclusions

This project demonstrated, for the first time, that CIMEX-1 is able to work in a complete closed loop with the new selected HFE-7100 liquid. This is an important step, since it is mandatory for the mission development to be continued. The system worked fine with challenging conditions (i.e. high cell temperatures and low pressures), and manual controlling was possible for all of the tests. The Bronkhorst meter/controller worked fine during all of the test campaign. Since the instruments were calibrated for another mixture, the measured values were converted using the indications provided by the supplier. It was discovered that this conversion was mostly affected by the mixture quality. Therefore, a conversion would be necessary even in the case that if the meters were calibrated for a mixture of and Nitrogen and HFE-7100 with a certain, but fixed concentration. The conversion formula was affected by an error of 5%. A more precise calculation method should be found in the future for better results. Moreover, for the same reason, the conversion factor variations with the mixture temperature and pressure should be taken into account.

The conversion from volumetric to mass concentration was successfully performed and the theoretical assumptions were validated by performing experimental tests. This conversion also explained why the system behavior with HFE-7100 is different than with Ethanol (i.e. because of the bigger molecular weight). However, a dedicated sensor calibration for mass concentrations is needed for better results.

The metallic foil with the anti-wetting groove worked effectively, demonstrating that is possible to keep the liquid level above the foil surface and to reach the liquid interface stability.

A proposal for new requirements was given in order to maintain wider parameter ranges (regarding pressures and flow rates). Another choice could be a re-design of some components (e.g. the CSS) and the results of this BB could be used for the first design iteration. The science teams, as well as ESA, have to verify which of these options is more suitable for the scientific purposes.

It is possible to conclude that CIMEX-1 Closed Loop BB was a success.

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