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Development of an Apparatus for Determining Surface Tension in Drops: Post-Flight Analysis of STS-108

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Abstract: This paper presents a description of the design and ensuing development of an automated liquid droplet generator and related utilization aboard the space shuttle, a) as a fluid positioning system for materials processing (attached droplet method), and b) as a means to measure surface oscillation of droplets under microgravity for determining their surface tension.

Keywords: drops, surface tension, microgravity.

1 Introduction

The formation process of most materials is characterized by a liquid-to-solid phase transformation. Hence control of liquid phase is essential to obtain materials of appropriate structures and properties.

Fluid behavior in phase-change problems is very complex especially when fluid-mechanical, physical–chemical and thermo-dynamical phenomena interact. Reduced gravity conditions can help in decreasing the complexity of the problem. For instance, under microgravity conditions natural convection, segregation and precipitation are reduced, whereas other phenomena (surface-tension-gradient-induced convection, etc) that are masked under Earth conditions become dominant.

In microgravity experiments, fluid positioning systems play a significant role. There are several systems with different pros and cons. These may be classified into three categories:

a) Totally confined systems (ampoules, etc.), where the container is completely liquid filled.

b) Partially confined systems (partially filled containers, liquid bridges, attached drops), where part of the liquid wets solid surfaces and anchors to them, and the free surface of the liquid is controlled by surface tension forces.

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c) Non-confined systems (free drops), where there is no contact with solid walls, and positioning occurs outside the walls, in the vicinity of a radiation field node (acoustic, electromagnetic, electrostatic, etc.).

When the container wall interference does not represent a problem for the experiment, the first option is the most appropriate strategy to better control the liquid. When any potential contact must be avoided, the third method is required. However, the interaction with the positioning field may cause movement in the liquid inner core.

In some circumstances, a non-radiative positioning is preferred, so as to avoid any potential contact with container walls. This is the case of liquid bridges or drops fixed to a solid disk, as used in the monocrystalline growth or purification of high-fusion-point materials.

The mechanical model of the attached drop technique is the object of this study conducted at the Microgravity Laboratory of Argentina (Lässig, 2004).

In such mechanical model, is thermal and diffusive phenomena are ignored. The negation of these two phenomena is based on characteristic times considerations. In general, the time associated to the mechanical processes (capillary response to oscillation) in fact is shorter than the time associated to the thermal (advance of the solidification front) or diffusive effects.

The main purpose of our team was to develop a device able to automatically generate drops that remain attached to a disk, in the Space Shuttle. This type of equipment is required for several processes involved in the analysis of materials under microgravity, like liquid bridge handling, determination of surface tension of liquid state metals by vibration frequency measurement (Egry, Lohöfer, Seyhan and Feuerbacher, 1998), etc.

The current study demanded four distinct steps. First, to ensure that formation of a liquid drop attached (adhered) to a disk is possible. Second, to develop a system that is simple and able to measure drop surface oscillation. Third, to test the system under a microgravity environment (KC-135). And forth, to carry out a space flight experiment to determine whether the vibration frequency of a drop adhered to a disk differs from Rayleigh's theory (1879).

2 Drop Attached (adhered) to a Disk

This apparatus has been designed to operate into a NASA GAS Canister (GAS-761), and to fly on board of the NASA Space Shuttle Endeavour.

The first goal of this experiment is the formation of a big drop that remains attached to a disk.

Based on Martinez's (1984) experience on Spacelab-1, Experiment ES-331, it is essential that the appropriate velocity range be established to enable the injected liquid to adhere to the disk.

Several studies have been performed on jets into surrounding fluids: Viilu (1962), Reynolds (1962), Mc Naughton and Sinclair (1966), Taylor (1967). However, all of these were under the influence of gravity and were analyzed from the stability point of view. The adherence of droplets to the injector in a microgravity environment was not considered.

Trinh and Depew (1995) have recently conducted experiments with several types of disks with the intention of determining the way different fluids would stick to the solid surface with different contact angles.

Robinsa and An-you Chai (1997) developed a device to form liquid drops in microgravity, but the appropriate injection velocity to get the drop stick to the disk was not mentioned in their studies.

If the jet speed is too intense, its inertia force will be greater than the surface tension force originated in the jet output perimeter and the drop will not adhere to the injector disk. A first theoretical approach is to assume that the jet inertial force is equal to or lower than the surface tension force at the injector output, thus:

$$\rho V^2 \phi^2 \frac{\pi}{4} \leq \sigma \phi \pi \quad (1)$$

Where:

V	Speed of liquid injection.
ϕ	Injector diameter.
ρ	Density of injected liquid.
σ_{ik}	Surface tension.

Using water, and expressing diameter in millimeters:

$$V \leq 0.5366\phi^{-0.5} \quad (2)$$

Figure 1 shows equation (1). The zone under the curve would be stable, i.e., an adherence zone. Experimental results indicate this is not the case, possibly due to the use of a stepper electric motor to inject the liquid, which induces pulses to the fluid when running.

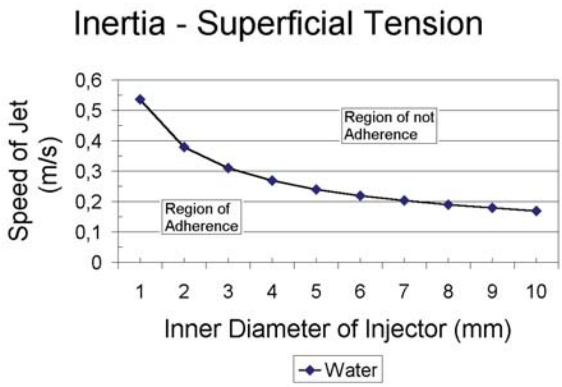


Figure 1: Theoretical Curve of liquid drop attached to the injector as a result of the equalization of inertial force with surface tension force.

2.1 Dimensional Analysis and Definition of the Experimental Setup

The first step is to perform a dimensional analysis of the problem. Applying the Buckingham's theorem (1914), the variables involved in the phenomenon are:

$$V = f(\phi, \rho, \sigma_{ik}) \tag{3}$$

Subindeces are: (i) injected fluid, (k) injector material.

The application of the theorem yields one dimensionless number, the Weber number, defined as:

$$We = \frac{Fluid\ Inertia}{Surface\ tension} = \frac{\rho V^2 \phi}{\sigma_{ik}} \tag{4}$$

Also, for practical reasons it is convenient to define the Reynolds number for the fluid at the injector output:

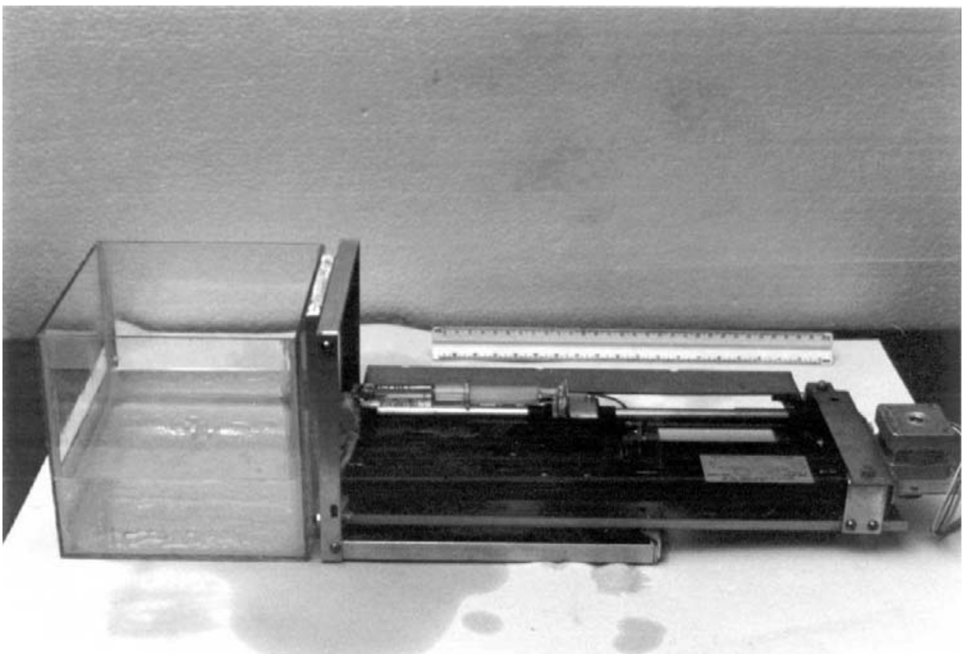
$$Re = \frac{Inertial\ forces}{Viscous\ forces} = \frac{\rho V \phi}{\mu} \tag{5}$$

where μ is the liquid dynamic viscosity.

In order to complete the experiments, a 3.3-liter neutral buoyancy tank was built, as shown in Picture A.

A brass injector disk and an aluminum injector disk were built.

The liquid was injected through the injector disk by means of a stepper-motor-driven piston sliding in a glass cylinder. The injected volume and the injection speed were controlled by an electronic circuit commanding the stepper motor pulses. The injected volume was estimated based on the frequency of pulses and the known kinematic chain of the movement mechanical transmission. Finally, the injection speed was determined using the injector diameter. For the experiment, a water-methylic alcohol mixture was used as the injection fluid, and Dow Corning 200 silicone oil as the receiving fluid. The densities of both fluids were equalized. Ten samples were taken under the same *Re* number and *We* number conditions, the measurements consisting in observing whether the injected liquid clung or failed to cling to the injector liquid.



Picture A: Neutral Buoyancy Tank.

2.2 Results from the Neutral Buoyancy Tank Test

Three different zones were observed (Figures 2 & 3).

i) for Reynolds numbers below 100 and Weber numbers below 0.068, the injected fluid anchored to the disk in all cases (Picture B: a, b & c);

ii) for Reynolds numbers above 273 and Weber numbers above 0.5, the injected fluid detached from the injector in all cases, forming free drops in the receiving fluid (Picture B: f);

iii) The third is an intermediate zone. There are a "p" percentage of observations where the fluid adhered to the disk and an "m" percentage of observations where the fluid failed to stick to the disk. For this reason this was called the "Transition" zone (Picture B: d & e).

$$\frac{p}{(m+p)}+\frac{m}{(m+p)}=1 \tag{6}$$

Remarkably, when the non-sticking drops do not withdraw enough due to the low level of inertia or to the high viscosity of the receiving fluid, the result would be a chain formed by the drops that have detached from the injector (Picture B: e & f).

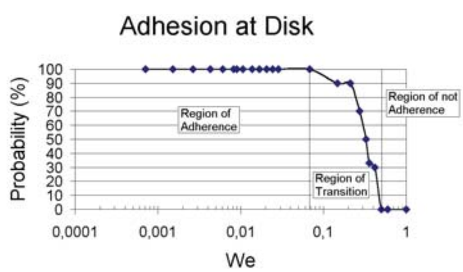


Figure 2a: Experimental results with the 2-mm-diameter brass injector at different Weber numbers.

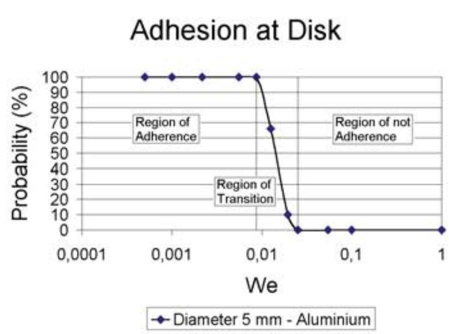


Figure 2b: Experimental results with the 5-mm-diameter aluminum injector at different Weber numbers.

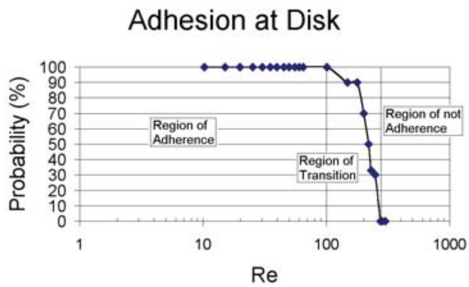


Figure 3a: Experimental results with the 2-mm-diameter brass injector at different Reynolds numbers.

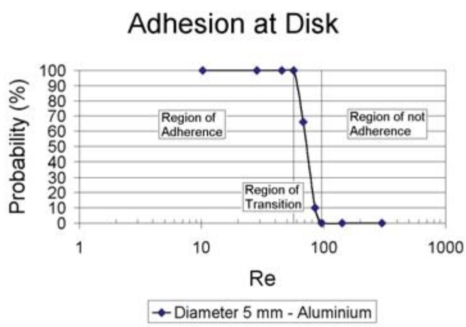
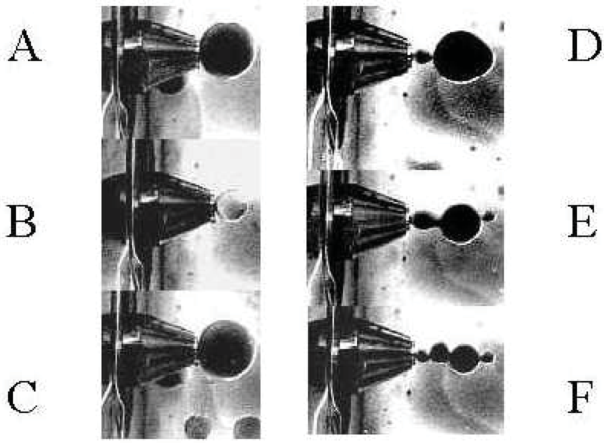


Figure 3b: Experimental results with the 5-mm-diameter aluminum injector at different Reynolds numbers.

2.3 Parabolic Flight Tests

The drop generator was obviously designed to meet the Adherence Zone parameters: Reynolds numbers below 100 and Weber numbers below 0.068. The drop generator basically consisted in a cylinder or sleeve serving as a piston chamber and a piston powered by a stepper motor with electronic control on start, stop and piston speed regulation. The piston forces the water contained in the cylinder out through an injector.

All the parts and pieces used to build the drop generator were made at the National University of Comahue (U.N.C.). The drop generator also has an IR sensor which measures drop vibration. Once the drop is generated, the sensor is moved towards the drop by means of a second stepper motor similar to that used to drive the piston.



Picture B: Adherence Zone (a) for Re=15 and We=0.001; (b) for Re=34.9 and We=0.008; (c) for Re=100.6 and We=0.068. Transition Zone (d) for Re=177.2 and We=0.211; (e) for Re=249.1 and We=0.417. No Adherence Zone (f) for Re=273 and We=0.502.

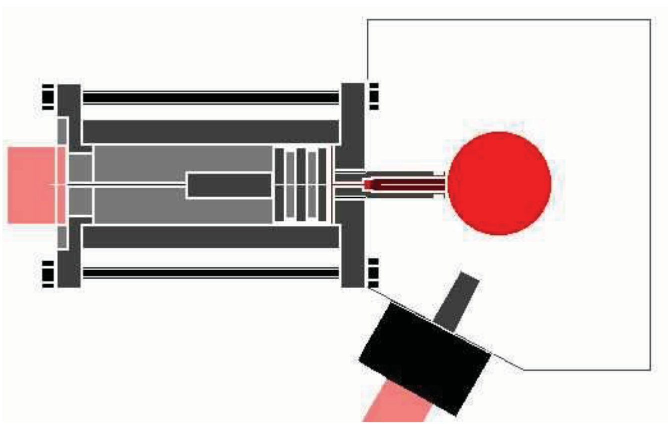
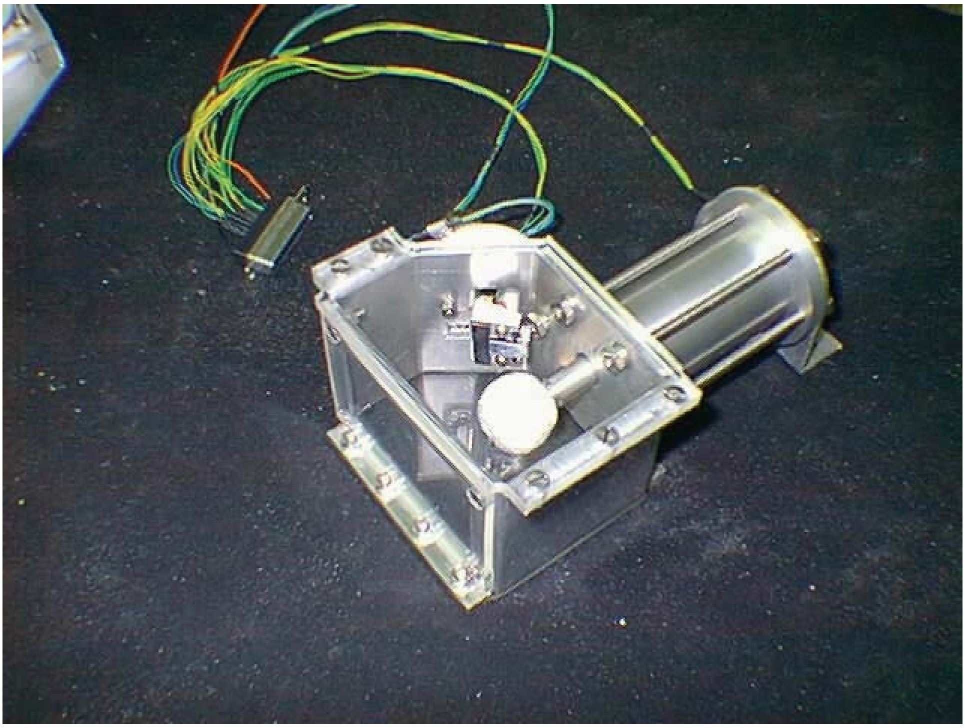


Figure 4: Drop Generator Schematic

With the apparatus ready for use, approval was obtained from the Johnson Space Center Reduced-Gravity Office to test the generator on the KC-135 aircraft before flying onboard NASA's Space Shuttle.



Picture C: Drop Generator.

The test mission on board KC-135 aircraft lasted two days, August 5th and 6th, 1997, with 40 parabolic flights carried out each day. As observed in Picture D, drops were repeatedly produced in a satisfactory way.

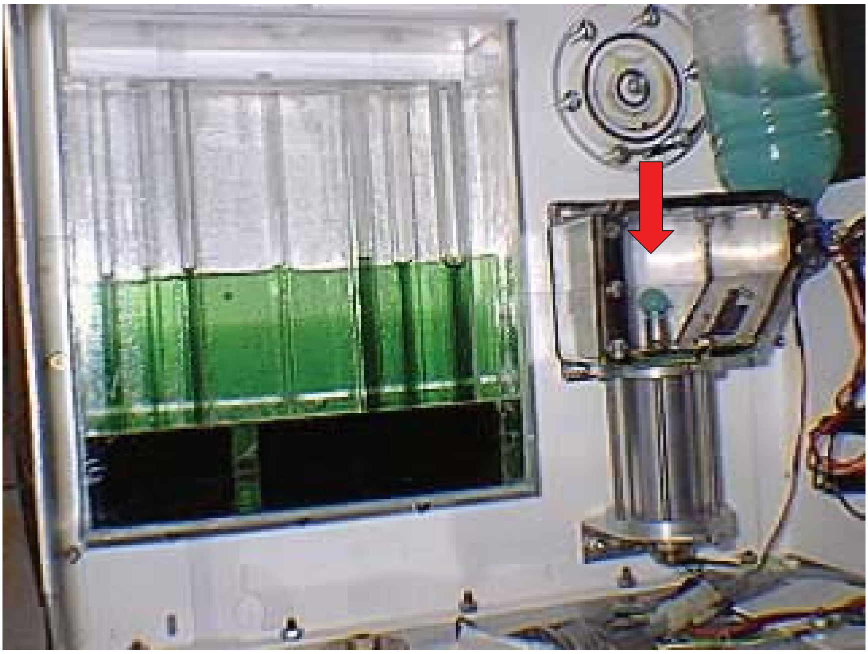
3 Measurement System

The system has been programmed to measure the vibration signal of water drops of three different diameters d1, d2 and d3 of 15, 20 and 25 mm respectively. The digitizer signal for different volumes allow to confirm the relationship between diameter and frequency, and to estimate the noise involved in the measurement process.

The Figure 5 shows the proposed scheme. The stepper motor M1 controls, through the amount of steps, the volume of injected water.

The vibration sensor, described below is fixed on an axis controlled by the stepper motor M2, which allows the sensor to approach the water drop.

The system is commanded by a microcontroller that taking the following actions:



Picture D: The Drop Generator on one of the Parabolic Flights.

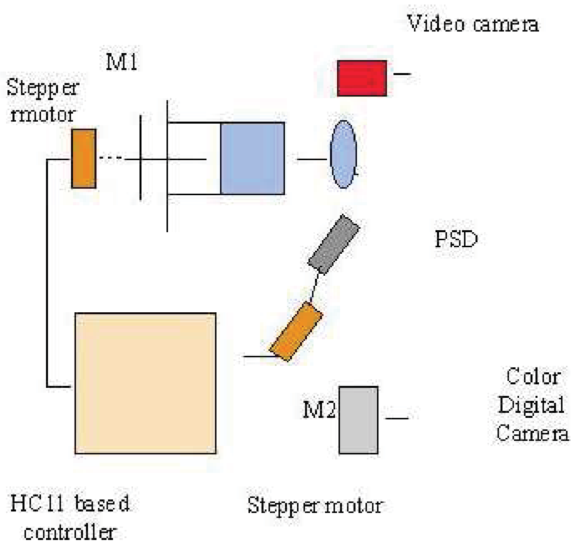


Figure 5: Measurement system.

- Generate the drop of size d1 by stepper motor M1.
- Stepper motor M2 is switched on and the optical sensor begins the approximation towards the drop. The approximation phase is carried out moving one step and measuring the reflection signal on the drop. The output signal from the sensor is digitized with a 10 bits A/ D converter.
- When the reflected signal reaches a programmable threshold, condition that is found between 3 to 5 mm from the drop's surface, the signal is stored into an eprom memory. The sampling rate is fixed at 100 S/sec. The vibration signal is recorded for 45 sec.
- At the same time, the drop is videorecorded with an 8mm videorecorder and also digital pictures are taken with a color digital camera.
- Once the recording process has finished, the optical sensor is restored to home position and the previous steps are repeated for drops of size d2 and d3.

3.1 Optical Sensor Description

So far, to measure the oscillation of the drop, video recordings have been used, as the works of Egry (1998), Rosner (1998), Flemings (1996).

We have added a second mensuration, and we have developed in this way another technique, consisting of measuring, on the surface of the drop, the superficial variation through the reflection of light IR.

The optical sensor used is a new micro displacement reflective sensor. It combines a position sensing diode and an IR led. It can detect up to 10 um movement. Figure 6 shows the optical sensor scheme. The I(A)/I(B) ratio is used to determine the displacement. As x increases I(B) increases and I(A) decreases and vice versa. Hence both signals I(A) and I(B) are used to determinate the light position.

An example of the sign of exit of the sensor one can observe the figure 7, where the oscillation of the surface of the drop is plotted as a function of time.

3.2 The Automation of the Experiment in the Gas Canister

3.2.1 General Aspects

An injector commanded by a stepper motor was employed to generate as explained before, drops of 15, 20 and 25 mm diameter and software controlled the size of the drop to be formed.

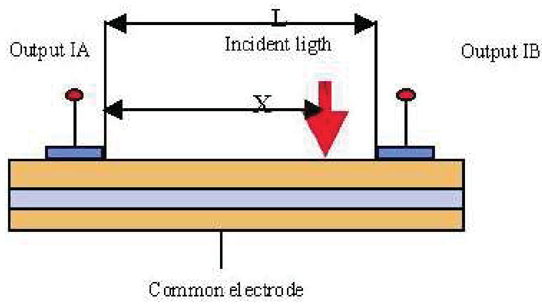


Figure 6: PSD sensor.

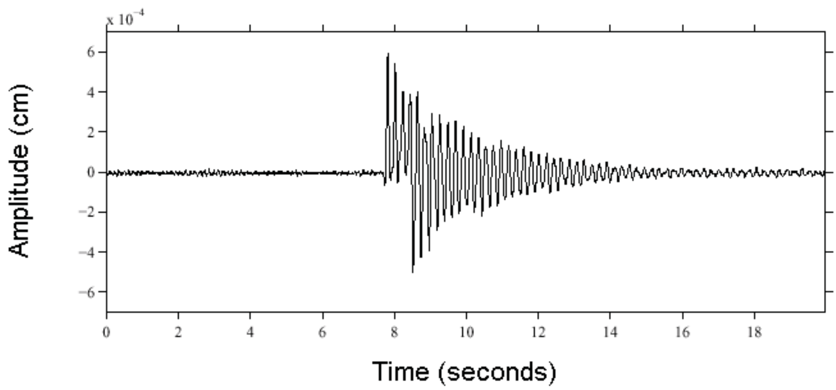


Figure 7: Oscillation of the surface of the drop in the time.

Owing to the need of control, data logging and registration of the indicated experiment, hardware and software were designed based on a previous analysis of the space environment to which the system would be exposed.

3.2.2 Description of Hardware

The premises taken into account in the design of the hardware are divided into two different aspects. The general one involved the volume, weight, consume and data logging requirements. The second aspect entails the experiment own requirements in flight, for which the following criteria were determined:

- Independent control of the experiments, except in the use of the image recording devices. The control module based on the MC68HC11 was designed for this purpose.

- RS232 communication through the Serial Communication Interface (SCI), which is an asynchronous full-duplex communication system, employed in the programming and recovery of the information to be processed once the MET had landed.

- Data logging and registration of the environmental parameters of the MET in non-volatile memory.

- Data logging and registration of the experiment results also in non-volatile memory.

- Realization of a diagnosis of the conditions for the beginning of the experiment and verification of its development.

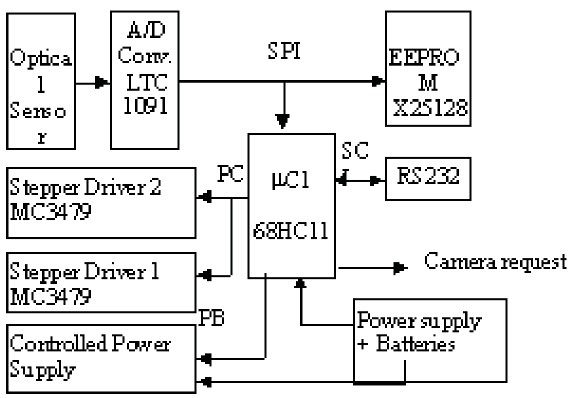


Figure 8: Block diagram of the experiment.

This hardware was designed in order to test the MET environment and to determine the initial conditions of the experiment and its control. In approximately 5 minutes periods (programmable by software) the measurement of the environmental parameters (temperature in 6 points of the MET and pressure in a single point by means of a MPX200 sensor, absolute pressure from 0 to 15 PSI) is possible. In both cases, controlled by software, temperature sensors and pressure sensors are switched on and, after the measurement is taken they are switched off in order to prevent self-heating of the mentioned sensors.

3.2.3 Module description

In time T2 (2.5 Hs being in orbit), the experiment begins with the request of light and video camera to the auxiliary services control module who turns on the lights and 2 seconds later activates the camera. Then, the first drop is built injecting a volume of liquid by means of the stepper motor M1. Once the 15-mm diameter drop is obtained, motor M2 is switched on to approach the micro sensor (ZAD-A01) optic-reflexive of high sensitivity PSD (Position Sensitive detector) to a distance between 3 and 5 mm. The analog output of the PSD is injected in an A/D 10 bits converter (LTC1091) and then, with a sampling rate of 100 samples/sec., at the serial SPI of the microcontroller. The information is stored in the EEPROM via the same port (SPI).

Once the formation of the drop is checked and the measurements are completely carried out, the micro sensor (PSD) returns to its original position automatically. The generation and measurement process of the resting drops, 20 and 25 mm diameter, is repeated in the same way. The estimated time of the experience is 5 minutes.

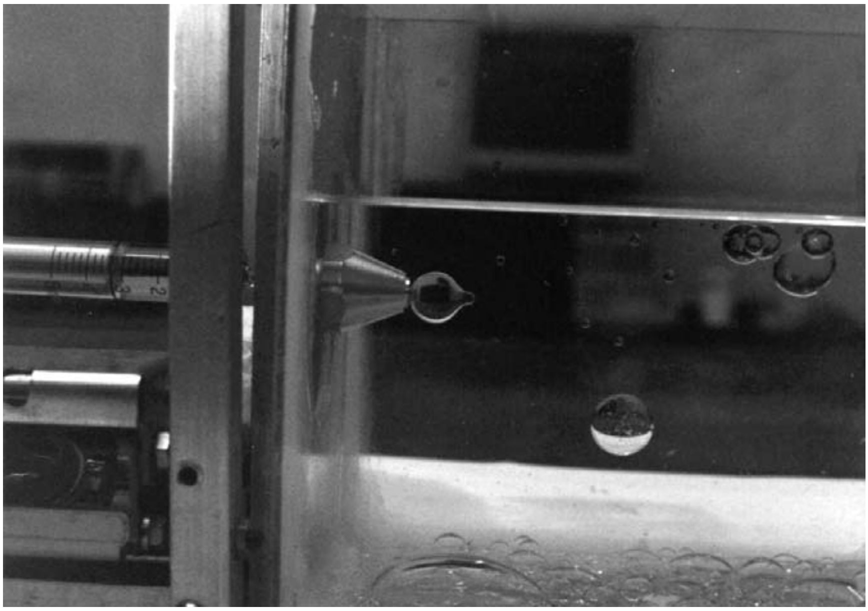
3.3 Description of the Excitation Method

The Neutral Buoyancy Tank tests demonstrated that as the liquid was injected, a significant excitation was created on the drop surface. This was caused by the stepper motor driven injection piston, which induced vibrations of the same pulse frequency as that of its stepper motor. It was therefore concluded that the drop could be excited from the injector itself, by setting the stepper motor at the desired pulse frequency.

4 Space Flight

The last week in September 2001, the equipment integration was completed at the Kennedy Space Center. Pictures F, G, H and I show images of the event and of the container ready to be installed in the shuttle orbiter bay.

The experiment was carried out aboard the Space Shuttle Endeavour, which took off on December 5th, 2001 on mission STS-108, and landed back on December 16th, 2001. The G-761 canister was located in the MACH-1 payload support structure. On the third flight day of the mission, the GAS Canister experiments were activated by astronaut Linda M. Godwin, and our equipment went on 2.5 hours later. The temperature recorded inside the experiment module at that moment was 10°C.



Picture E: A droplet impacting in the inner surface of a bigger drop attached to the injector. This causes excitation on the bigger drop surface.

5 Results

The experiment was smoothly run, and 10-to-25-mm size drops were successfully formed and remained attached to the injector disk without detaching. After frequencies had been measured, the software sent the instructions for the reabsorption of drops into the container cylinder.

Picture H and I show two sample drops of 12 and 25 mm respectively, formed during the experiment conducted under microgravity conditions.

Table I summarizes the frequencies of drop vibration as a function of drop size and the theoretical calculations of frequency based on Rayleigh equation.

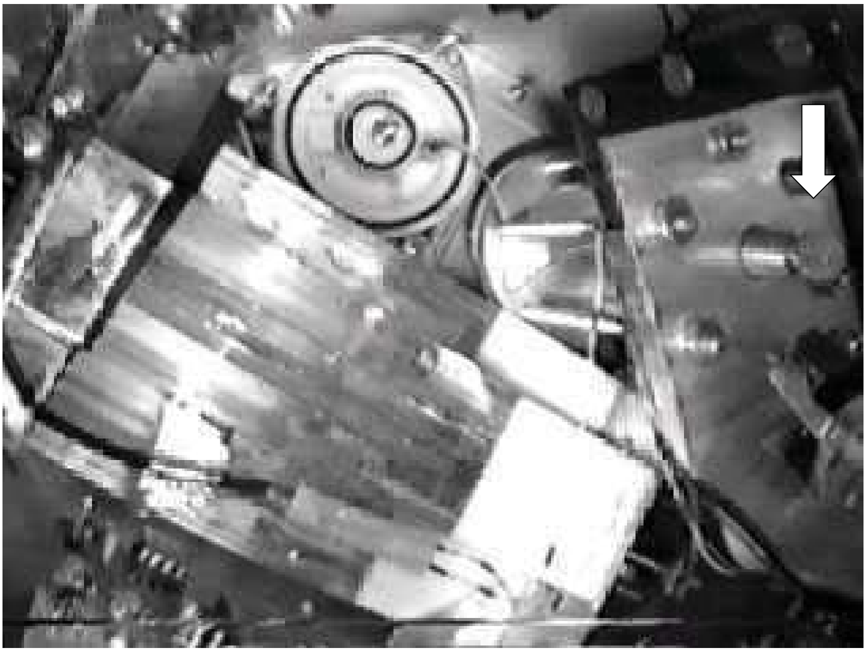
6 Analysis

The measured frequencies of drop vibration are fairly consistent with the results of the theoretical calculations performed with the Rayleigh equation. For the experiment, water was mixed with 30% acrylic polymer Glo Cot, which gave the fluid a milky white color to make it "readable" by the optical sensor.

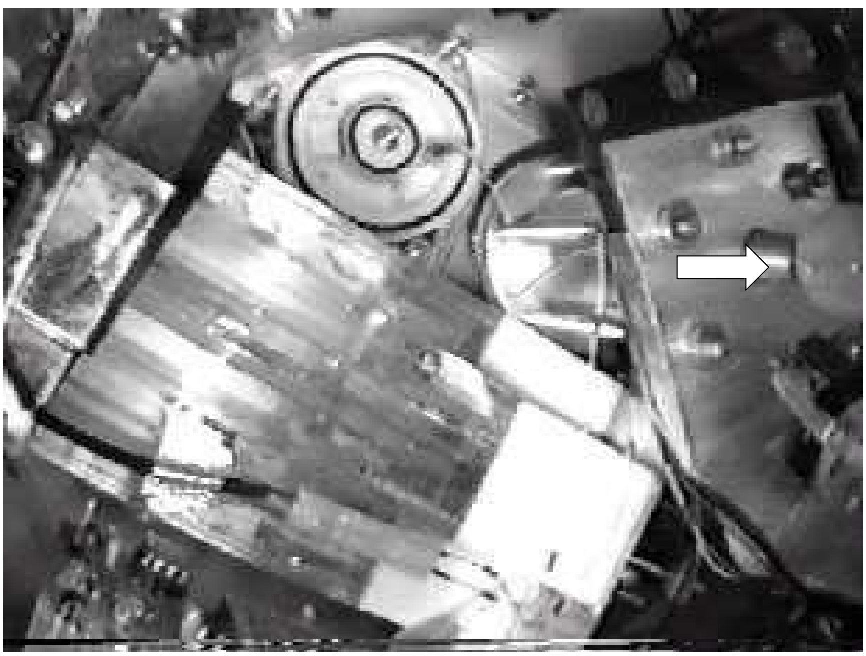
The vibration frequency values for drops attached to the injector disk under real microgravity conditions (aboard Space Shuttle) were quite similar to the theoretical

Picture F: Container (at the back) and P.A.D.E. experiment modules.

Picture G: Internal view of the P.A.D.E Fluming module.



Picture H: A 12-mm diameter water drop.



Picture I: A 25-mm diameter water drop.

Table 1: Surface Vibration Frequency of Drops, per diameter, measured in the Fluming module inside GAS canister G-761.

Drop Diam. (mm)	Rayleigh Frequency (Hz)	Measured Frequency (Hz)
13	5.07	5.5
15	4.09	4
19	2.87	2.5
25	1.90	2

values calculated applying Rayleigh's theory. This means the experimental apparatus successfully measured drop vibrations used to determine the liquid surface tension values.

As observed in Figure 10, the vibration frequency error between the value calculated for a free drop (Rayleigh's theory) and the value measured for the drop attached to the disk is below 12%.

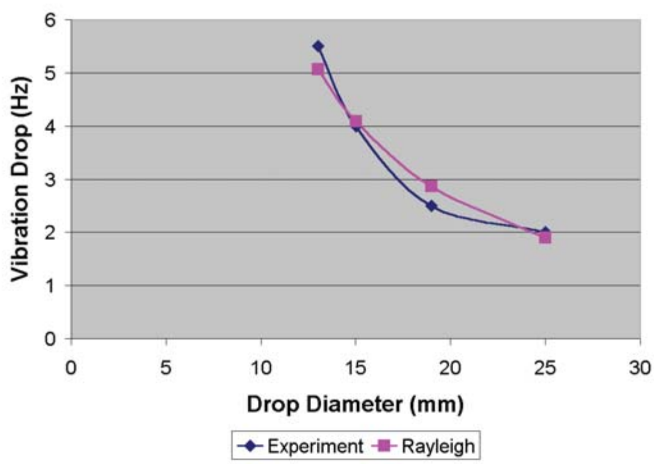


Figure 9: Vibration Frequency Graph developed from values in Table 1.

Now, the question is what realistic applications could be given to this hardware that has been developed and tested during flight. Potential uses are:

a) to measure real surface tension values for liquids under special conditions, like drops of liquid metals close to the freezing point, in liquids below or close to the critical point, etc.;

b) as a liquid positioning system to process materials (attached drop method);

c) as a research device to conduct microdrop injection experiments in the context of microcomponent soldering technique.

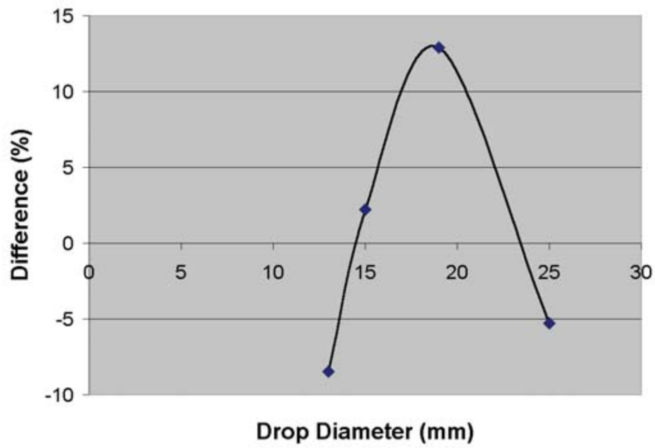


Figure 10: Vibration Frequency Error between Rayleigh theoretical values and measured values.

7 Conclusions

The effect of jetting a liquid in microgravity was quantified by two dimensionless numbers. Additionally, three regimes corresponding to the following conditions were identified: adherence to injector, adherence fluctuation (transition zone) and lack of adherence of the liquid to the injector.

An apparatus able to generate drops that remain attached to a disk was developed and tested in real space conditions. With a very limited budget, this versatile, flexible and efficient drop generator was designed and built considering the specific restrictions imposed by volume and weight specifications associated to the space environment.

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