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► **To cite this version:**

Jean Colombani, J Bert. Holographic visualization of convection during thermotransport: application to microgravity experiments. *Measurement Science and Technology*, 1999, 10, pp.886-892. 10.1088/0957-0233/10/10/308 . hal-03526859

**HAL Id: hal-03526859**

**<https://hal.science/hal-03526859>**

Submitted on 26 Jan 2022

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# Holographic visualization of convection during thermotransport

## Application to microgravity experiments

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**Abstract.** The performance of two microgravity measurements of the Soret effect in molten salt mixtures have led us to analyze their insufficiencies essentially in terms of hydrodynamic diagnostic. As a consequence, and after earth preliminary measurements of the Soret effect on a particular class of vitrifying liquids by means of a holographic technique, we propose in collaboration with two other groups, a new microgravity experiment on molten salts mixtures. The scientific interest and feasibility of this new experiment is analyzed in detail. New solutions are given to overcome the experimental problems of such an experiment which should provide a decisive proof of non-convective experimental conditions in space for molten salts.

**Key words:** Soret, thermal diffusion, molten salt, microgravity, holography

### 1. Spatial relevance of mass thermotransport properties measurements in liquids

Among the classes of mass transport phenomena in liquids, thermal diffusion (Soret effect) is related to a particularly large number of scientifically and technologically pressing questions. This effect is characterized by the existence of a spontaneous matter flux in an inhomogeneously heated fluid mixture, this flux tending to separate the constituents [1]. The scientific interest of thermal diffusion lies in microscopic as well as macroscopic considerations. Firstly, it is a nonequilibrium cross property, which still needs a lot of work on phenomenological modeling, especially for multiconstituent systems [2]. Secondly, the molecular bases of the Soret effect are still vague and no statistical theory is able to show even a qualitative agreement with the experiments [3]. But recent interesting results of molecular dynamics numerical simulations permit to expect fruitful theory/experiment comparisons [4].

From a more industrial point of view, whenever multiconstituent liquids are submitted to long lasting temperature gradients, they are liable to experience thermal diffusive flows, modifying the distribution of the species concentration. Among the cases where this effect has been emphasized, we can mention the elaboration of high technology materials (solidification of semi-conductors [5]), the production or purification of light metals (electrolysis of aluminum, lithium, ...[6]), the optimization of petroleum extraction (by modeling of the hydrocarbon distribution in the reservoirs [7])

So the knowledge of quantitative values of the Soret coefficient (which is the parameter characteristic of the phenomenon) for a wide domain of materials, in various thermodynamic conditions and physical configurations is needed for the above mentioned basic and applied purposes. But unfortunately, reliable values of these coefficients have revealed themselves to be very difficult to gain. For example, agreement was found only recently about the values of the Soret coefficient of the well-studied toluene/n-hexane mixture measured by different techniques [8].

The experimental determination of Soret coefficients in binary mixtures, noted  $S_T$ , is obtained by measuring the steady concentration gradient produced by the imposition of a constant and uniform temperature gradient. This concentration gradient achieves a stationary state when thermal diffusion and interdiffusion (which tends to rehomogenize the mixture) exactly compensate each

other. However, the mechanical stability of such non-isothermal liquids is hard to guarantee. Indeed, the free convection critical temperature gradient may be easily exceeded and a convection pattern may attenuate or even completely change the concentration gradient [9]. In addition, even in a globally diffusive regime, small experimental defects in the cell unavoidably exist and are the source of transverse temperature gradients, which initiate localized convection loops that disturb the measurements [10]. Several attempts of compensating this gravity induced convection have been made, but they can be used only for a highly restricted number of materials [11]. Under these conditions, the only opportunity of getting disturbance free reference values of  $S_T$  are microgravity measurements. Facing this necessity of reduced gravity, experiments have been made in sounding rockets or in the space shuttle.

We will present here our experience of the problems and solutions for the space measurement of the Soret coefficient for a particular class of material, the molten salts.

## 2. Thermoelectric Soret measurements in molten salts in space

Numerous techniques are available for the measurement of the Soret coefficient in molten salts mixtures, and are a priori usable in space:

- direct measurements of the steady concentration gradient, in various configurations (shear cell [12], flow cell [13], ...), are widely used thanks to their simplicity. But their major drawback is their invasive character: taking samples from the liquid for concentration evaluation is impossible during the diffusion, so kinetic measurements are lacking. Therefore we are compelled to turn towards nondestructive techniques;

- classical optical methods are very attractive (beam deflection method [14], classical interferometry [15], ...). One of their limitations is their relatively low accuracy compared to electrical measurements and their absolute requirement of high optical quality surfaces (flatness and polishing);

- new subtle optical methods such as classical Rayleigh scattering [16], Thermal Diffusion Forced Rayleigh Scattering [17], fiber optics spectroscopy [18], ... are very recent at the present time and have provided too few results to be reliably validated. Furthermore, they often need several runs for one measurement, advanced data processing (filter, fits,...) and the use in the computations of delicate thermophysical properties, which makes remote real-time measurements difficult;

- finally, electrical methods (thermoelectric [19], conductimetric [20], ...) are from our point of view, the best candidates for spatial measurements, combining a high level of accuracy, a relatively handy procedure convenient for a spatial utilization, and the access to real-time measurements. These characteristics were indeed the main requirements for the choice of the technique.

So we have performed two thermoelectric Soret experiments in the eutectic mixture  $\text{AgI}_{0.72}/\text{KI}_{0.28}$  in the Spacelab during the D1 and D2 flights of the space shuttle (1985 and 1993) [21]. This molten salts system had been chosen for at least three reasons:

- firstly, the presence of the silver ion enables the convenient use of reversible silver electrodes (no interface electrical capacitance);

- secondly it exhibits a particularly low melting point ( $238^\circ\text{C}$ ) which was required for reduced energy consumption in the shuttle;

- thirdly it exhibits the properties of an ionic superconductor, in the solid state, which can be interesting in the field of solid-state batteries;

The main experimental problems that had to be solved were the following.

- the mixture being unavoidably solid during the storage at ambient temperature prior to the flight, the expansion due to the temperature increase and extra volume due to the solid-liquid transition had to be absorbed in order to avoid any free volume. The solution adopted for this requirement was the use of a porous ceramic crucible surrounding the liquid;

- the liquid and electrodes had to be totally electrically isolated in order to avoid any short circuit between the electrodes, through the frame;
- during the flights, all experiments were requested to provide a triple screen between the liquid and the Spacelab in order to insure the astronauts' safety. The sketch of the device is given figure 1. This device was integrated in the GHF furnace (Gradient Heating Facility) developed by CNES for several flights.

Figure 2 displays the typical evolution of the Soret induced thermopower during the D2 experiment. This experimental behavior can be fitted by well known laws in order to give access to the steady state value (at infinite time). The stationary concentration difference in the mixture,  $\Delta c_\infty$ , is then calculated from the difference between the initial  $\varepsilon_0$  and the final  $\varepsilon_\infty$  thermopowers. The Soret coefficient is then given by  $S_T = 1/(1-c) * \Delta c_\infty / \Delta T$ ,  $c$  being the initial concentration and  $\Delta T$  the applied temperature difference.

Although a few technical solutions might be improved for future flights, these first experiments can be considered as a good success from this standpoint. However several important questions remain from the scientific point of view, as we will explain below. The two main results were the following:

- the direction of the effect in the mixture was measured reliably for the first time:  $S_T$  is positive, i.e., conventionally, the silver ions migrate towards the cold end of the cell;
- the quantitative value of the Soret coefficient has been determined:  $S_T = 1.0 * 10^{-3} \text{ K}^{-1}$ .

But beside these successes, we have encountered the following limitations:

- the characteristic time of the Soret experiment,  $\tau$ , is directly linked to the interdiffusion coefficient  $D$  through the formula  $\tau = h^2 / (\pi^2 * D)$ ,  $h$  being the diffusion length. A computation of  $\tau$  from diffusion earth measurements had given a value of 1 hour. The actual measured value, 4 to 5 h, corresponds to a diffusion coefficient  $D$  which is then four times smaller than expected. This was the initial reason of making a new experiment in D2, with a much longer duration than the too short D1 experiment;

- as can be assessed from the D2 curve given figure 2, the thermoelectric power has deviated from the expected exponential behavior during several hours in the course of the experiment. The actual origin of this trouble is still unknown but it could be bound to thermal regulation of a cooling fluid in the Spacelab, as well as vacuum variations. The consequences on the experiment are unknown;

- the Soret coefficient has also been estimated from a chemical determination of the concentration distribution in the quenched mixture after return to earth. The discrepancy between the chemical and electrical values is about 40%; it has not been clearly explained yet.

These three limitations might have three different causes but what precisely handicaps the interpretation is that these measurements are practiced in the blind. The spatial environment should provide convection free diffusion but we had no possibility to check the actual absence of convection and the encountered troubles could originate in convective flows due to the residual gravity. Thus has arisen the necessity of performing a new experiment enabling to visualize the mass fluxes during the diffusion. In this way, the spatial relevance of this kind of experiment could be properly demonstrated.

### 3. Preliminary holographic Soret measurements on earth

We first made an exhaustive examination of the possible techniques which could provide, during the same experiment, a physical measurement (determination of the concentration gradient) and a mechanical observation (check of the absence or presence of convective flows). The most powerful method then fulfilling these requirements and permitting space measurements is holographic interferometry. Indeed, it gives the advantage of an interferometric technique, i.e., the recording of the refractive index evolution (due to temperature or concentration in our case) is visualized through interference fringes. But above all, it permits to work without any physical reference system: the latter is simply replaced by the 3D studied system recording at a chosen given time on a so-called

reference hologram. So the reference time can be changed during the course of the experiment. Furthermore, high optical quality is not absolutely required for most of the device components.

However, a preliminary phase of experimentation on earth was essential because the holointerferometric procedure was not established for Soret experiments. As specified above, thermal diffusion is most of the time disturbed on earth by gravity induced non-diffusive fluxes. So we had to use a trick to compensate buoyancy effects. The chosen one was inspired from chemical experiments but never used before in this case: an instability spontaneously develops in free convection if buoyancy forces due to the thermal gradient exceed the dissipative effects (viscosity and heat diffusion). So instead of playing on the former, we played on the latter, taking advantage from our long experience on vitrifying liquids [22], i.e., we enhance the viscosity in place of reducing the gravity. Indeed the liquids of this class exhibit high increase of their viscosity when temperature is decreased. We have finally chosen the (LiCl, H<sub>2</sub>O) mixture, remaining thus in the ionic liquid class. The main experimental doubts to be cleared up were the following [23]:

- temperature and concentration variations in time are both diagnosed in holointerferometry by the same fringes, implying comparable refraction index changes, so the problem of decoupling arises. The first possible solution was the use of two wavelengths holointerferometry, where the comparison of the two sets of fringe patterns, corresponding to each wavelength, permits to deconvolve the solutal and thermal contributions [24]. But this technology was not programmed for any future flight facility within the next years. So we had to discard this type of technical solution and find a physical one. Thereby, it has turned out that heat and mass transport exhibit characteristic times that have considerably different orders of magnitude. This is quantified by the Lewis number,  $Le = D/D_T$ ,  $D$  being the interdiffusion coefficient and  $D_T$  the thermal diffusivity.  $Le$  is of order of  $10^{-2}$  in liquids. According to this time scale difference, in our system, the temperature fringes appear in about half an hour, amounting to the establishment of the temperature gradient. We consider that the mixture remains homogeneous during this time, short compared with the diffusion time. After the taking of a new reference hologram, the newly appearing fringes can be attributed to the concentration evolution. The time duration of this process is about one week;

- another point to deal with was the unusual simultaneity of the diffusion measurement and the hydrodynamic diagnostic. A purely diffusive Soret experiment is shown in figure 3 in (LiCl, 9.7 H<sub>2</sub>O) at a mean temperature of  $-26.5^\circ\text{C}$  ( $\Delta T = 6.1^\circ\text{C}$ ). The good parallelism of the fringes indicates the existence of a mass flux in the direction of the long axis of the diffusion cell. The number of these non-disturbed fringes can then be linked to the evolution of the concentration difference between both ends of the cell, as displayed in figure 4. In contrast, the development of an instability due to exceeding the  $\Delta T$  threshold of thermal convection is shown in figure 5. The distortion of the fringes, corresponding here to isotherms, can readily be interpreted in terms of non-diffusive mass fluxes leading to the appearance of a convection loop. We must pay particular attention to the variety of the convection patterns, in order to be able to detect all kinds of disturbances. figure 6 shows, for instance, an oscillatory convective cell, which occurred as the thermosolutal threshold was exceeded (this periodic behavior is caused by the interplay between a destabilizing temperature gradient and a stabilizing concentration gradient).

In addition to these experimental clarifications, significant scientific results were inferred from this preliminary phase [25]:

- first, the reliable determination of the evolution of the Soret coefficient with temperature ( $-60$  to  $0^\circ\text{C}$ ) and concentration (0.5 to 9 mol/kg) of lithium chloride in aqueous solutions has enabled the test of several theories. Dealing with the interpretation of characteristic features of the thermal diffusion (inversion of the effect with temperature, existence of a minimum with concentration) those were missing experimental confrontation, since reliable experiments were lacking. Relevant physical processes playing significant roles in this effect have accordingly been demonstrated;

- second, in order to test the ability of our procedure to highlight convective disturbances, we have studied the hydrodynamic behavior of (LiCl, H<sub>2</sub>O) in our cell geometry (30mm x 10 mm x

10 mm) [23]. We have especially investigated the  $\Delta T > 0$  and  $\Delta c > 0$  domain of the stability diagram (heated from below and salt migrating downwards), where amazing convection patterns occur (see figure 6). Although many studies of such thermosolutal convection exist, very few have been undertaken in such a confined configuration, most of them having been made in the well known Rayleigh-Benard geometry [26].

Finally, this preliminary phase has yielded the confirmation that Soret experiments are extremely difficult to perform in liquids with standard viscosity on earth, convection arising too easily.

#### **4. Preparation of holographic measurements of Soret effect in molten salts in space**

According to its long experience in microgravity measurements on molten salts and in the holographic technique, our group has proposed to ESA a combined holographic interferometry and thermoelectric study of the thermodiffusion process appearing in a molten salts solution, in the International Space Station (ISS). This proposal is made in collaboration with the group from RWTH Aachen (J. Richter) and with a group specialized in hydrodynamics (D. Henry and H. BenHadid from the École Centrale de Lyon)

##### *4.1. Choice of the mixture*

Obviously, this choice is crucial for the study. In the class of the binary mixtures of molten salts with a common anion, we have pointed out the adequacy of  $\text{AgNO}_3/\text{KNO}_3$  for at least 6 leading reasons:

- simplicity. From a physical and chemical point of view, it is a simple mixture with well known transport properties, phase diagram, thermal expansion, electrical and thermal conductivity, thermoelectric power, viscosity...;
- melting point. This liquid exhibits a particularly low melting point (eutectic point  $130^\circ\text{C}$ ) among the liquids of this class, which guarantees both a low energy consumption of the heating facility in the ISS, and the possibility of making optical measurements without problems bound to high temperature, especially in the space environment;
- optical transparency. This fluid is completely transparent in the visible range, which is not the case for the mixture used during preceding flights,  $\text{AgI/KI}$ , which shows a large absorption band, variable with temperature, in the visible range. This permits an optimization of the holointerferometric diagnostic;
- comparison with preceding experiments. The choice of  $\text{AgNO}_3/\text{KNO}_3$  will permit a comparison with the  $\text{AgI/KI}$  measurements during the D1 and D2 flights. The discrepancy (or coincidence) of their Soret coefficients will highlight the part of the anion during the diffusion mechanism in these two very similar systems;
- silver anion enables the use of reversible silver electrodes for the thermoelectric measurements (no interface electrical capacitance);
- no particular hazard by ingestion, or vapor inhalation. This condition is important in the confined space laboratory environment. Corrosion reactions must be avoided by the use of silica or glass or corrosion resistant alloys (Hastelloy C-276).

##### *4.2. Facility*

ESA has ordered the design and making of a facility the aim of which is to support scientific microgravity research in the field of fluid physics: the Fluid Science Laboratory (FSL) [27]. It is a flexible, highly modular facility for the Columbus Orbital Facility (COF), which is the European module of the International Space Station. It really yields new and up-to date possibilities compared to older systems like HOLOP for instance [28, 29].

The FSL facility fulfills most of our experiment requirements except for the Digital Image Holography (DIH) technique [30], for which the reference state can be chosen among all the recorded states of the system. In addition, DIH produces high-contrast interferograms even if the experimental conditions are not ideal, compensating the influence of additional light sources and the effects of thermal deformation of the interferometer in the course of the experiment; this is particularly true in case of long duration experiments as we propose here (30 hours). The interference fringes produced by DIH can be interpreted in the same way as those from real-time holography. This particular technique is then required by our group and collaborators in order to refine the possibilities of classical holography which is permitted by the FSL. In addition to DIH, adaptive filter algorithms for interference patterns of diffusion measurements [31] will permit the confident detection of signal peaks represented by interference fringes avoiding misinterpretations of the experimenter and could possibly lead to automatic diffusion measurement if desired.

#### 4.3. *Experimental problems arising from space experiments and new solutions*

Let us analyze with a few details the main experimental problems occurring when a liquid diffusion experiment is performed in space: The first one comes from the absolute necessity of a total cell filling, the second one from the parasitic presence of bubbles on the cell walls during optical measurements, and the third one from convective instabilities.

4.3.1. *Total cell filling.* The 100% cell filling is necessary in order to avoid any free surface effect leading to Marangoni convection. This obligation must be examined with taking care of the volume increase of the salt when melting (about 5%) combined with the normal thermal expansion (1 to 5%, depending on the working temperature). Several possibilities exist to solve this kind of problem. As said above, we have already tested an absorption of the extra volume by means of a controlled porosity alumina cylinder surrounding the salt [21]. This solution cannot be considered as fully satisfactory because of an insufficient knowledge of the evolution of the wetting properties with sample compositions. In addition, this solution is inconsistent with an optical measurement. In the programmed future experiments, the expansion when melting will then be absorbed thanks to metal bellows situated at one end of the sample, with the condition that a directional melting should be programmed (from the bellows to the other end of the cell).

4.3.2. *Absence of bubbles on the optical walls.* These small bubbles which get stuck on the cell optical walls come from initially dissolved gas or simply from the saturated vapor pressure, the bubbles total volume being possibly increased by a cell filling defect. Preceding earth and space experiments performed by our collaborators have permitted to be aware of this problem [28]. In the case of aqueous solutions used at room temperature, a careful filling and closure of the cell at the same temperature permits to avoid the two problems (presence of bubbles and incomplete filling). As molten salts cannot be kept in the liquid state ( $T > 130^{\circ}\text{C}$ ) during the storage time prior to the flight, one has to find other types of solutions. We are now working on an earth Facility in which the bubbles stuck on the optical wall can be thrown away by an energetic stirring induced by a fan situated at one end of the cell. This stirring also has the advantage of permitting a good homogenization before the initiation of the thermal diffusion experiment. This new kind of apparatus which has been designed by CNES and COMAT Industry will be tested on earth in the near future then adapted to microgravity with the help of experiments performed during short microgravity sequences in the Airbus A300-Zero g from CNES-NOVESPAC during parabolic flights.

4.3.3. *Hydrodynamic stability.* This point can be treated on earth by means of numerical simulations applying confined thermosolutal convection to the microgravity environment. These computations will additionally constitute a major part of the calculations of the ideal size (aspect

ratio) of the experimental cell by finding the best compromise between mechanically stabilizing dimensions and optical constraints.

## 5. Conclusion

We have shown that the good knowledge of the transport parameters of liquids is of primordial importance both for basic research and from an industrial point of view. Among those parameters, the Soret coefficient, characteristic of the thermodiffusion process is one of the most difficult to obtain due to the quasi impossibility of obtaining the mechanical stability of non-isothermal liquids on earth except for a limited number of materials. A special type of liquid combined with the holographic technique has then permitted us to get non gravity disturbed earth thermal diffusion measurements as well as a hydrodynamic diagnostic. So, except for this highly restricted number of materials, there is a necessity of microgravity measurements, particularly in the case of molten salts, the physical characteristics of which leading them to be mechanically unstable. In addition, even with the use of special earth techniques, the reliability of such an approach also has to be tested. Two initial microgravity blind experiments performed during D1 and D2 flights have shown us the necessity of an hydrodynamic diagnostic and has led us to propose, in collaboration with two other groups a new thermal diffusion experiment in the ISS with an holographic visualization. This experiment is based on a new approach which combines in-situ measurement techniques with continuous optical control which is a guarantee of reliability. This will provide a decisive proof of non-convective experimental conditions in space for this kind of experiment.

## Aknowledgments

All the work on space experiments was supported by the French space agency (CNES). We are then indebted to CNES for its constant help, especially F. Gonzalez and D. Buso and COMAT Industry for helpful discussions leading to the new earth apparatus which could be the precursor of the future ISS diffusion experiment facility.

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## Figures captions

**Figure 1.** Thermodiffusion cell: (1) pure impervious alumina cylinder, (2) porous alumina cylinder (pore diameter 30  $\mu\text{m}$ ), (3) non-expanding metal tube, (3B) silver o'ring (impervious sealing), (4) alumina ring (electrical insulation), (4B) stainless steel ring, (5) and (5B) silver plugs (electrodes for the thermopotential measurements), (6) and (6B) stainless steel plugs tightened with screws, (7) and (8) plugs soldered to the stainless steel tube (9).

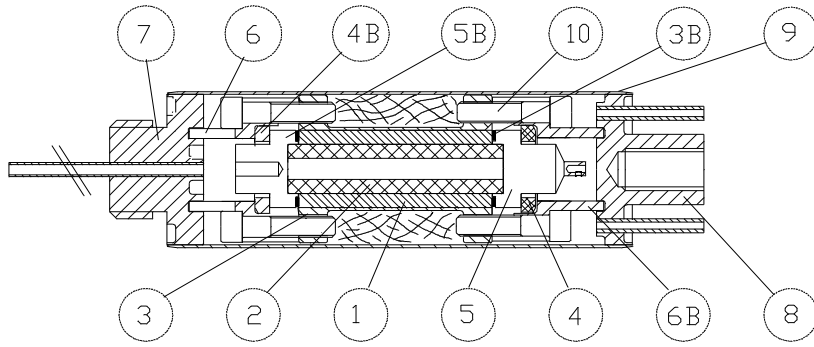
**Figure 2.** Variation of the thermoelectric power during D2 in the molten mixture AgI/KI inside a blind cell during a thermal diffusion experiment. Lacking of optical and residual gravity data, the disturbances appearing after 5 hours are not explained at the present time.

**Figure 3.** Concentration fringes evolution with time in the mixture (LiCl, 9.7H<sub>2</sub>O) during a thermodiffusion experiment.  $T_{\text{cold}}=-26.5^{\circ}\text{C}$ ,  $\Delta T=6.1^{\circ}\text{C}$  (from 0 to 202 hours).

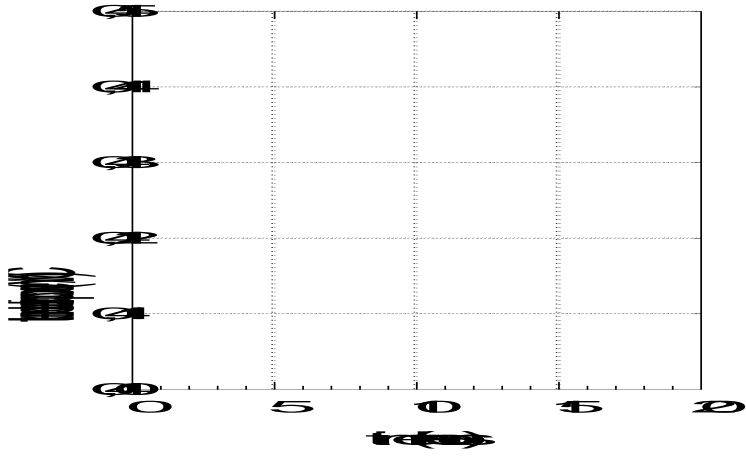
**Figure 4.** Evolution of the concentration difference (between top and bottom of the cell) with time for the experiment shown figure 3.

**Figure 5.** Isotherms obtained for the respective temperature differences of  $0^{\circ}\text{C}$  ;  $4.9^{\circ}\text{C}$  and  $10.8^{\circ}\text{C}$  along a cell heated from the bottom and containing the mixture (LiCl, 9.7H<sub>2</sub>O) with  $T_{\text{cold}}=-10^{\circ}\text{C}$ . (the last temperature difference value corresponds to steady convection)

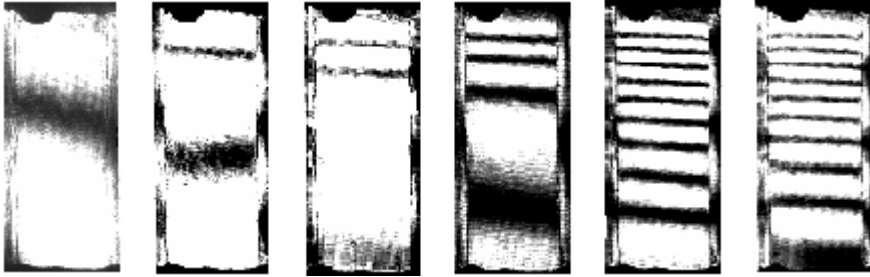
**Figure 6.** Isotherms showing an oscillatory convection during half a period. in the mixture (LiCl, 9.7H<sub>2</sub>O) for  $T_{\text{cold}}=-31.9^{\circ}\text{C}$ ,  $\Delta c=0.54$  weight % and  $\Delta T=29.1^{\circ}\text{C}$ . The period is 348s and the interferograms are equally time spaced.



**Figure 1.**



**Figure 2.**



**Figure 3.**

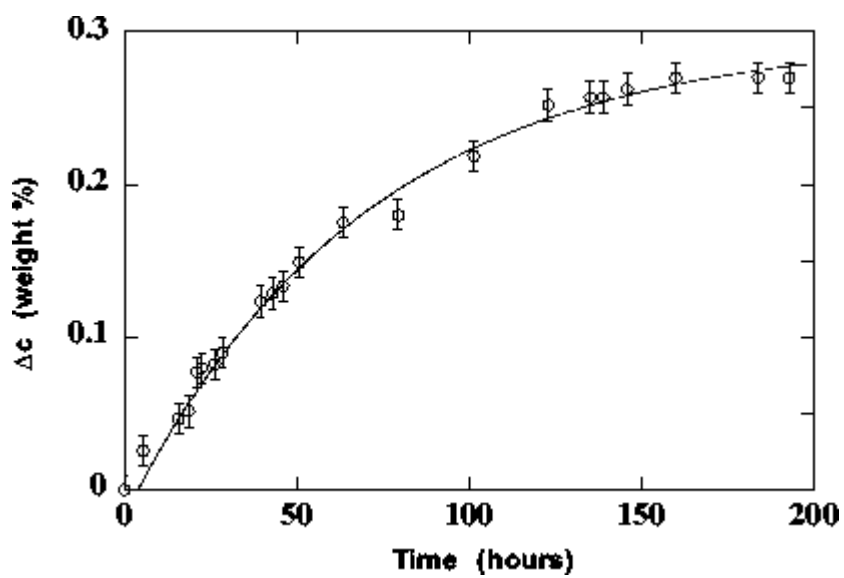
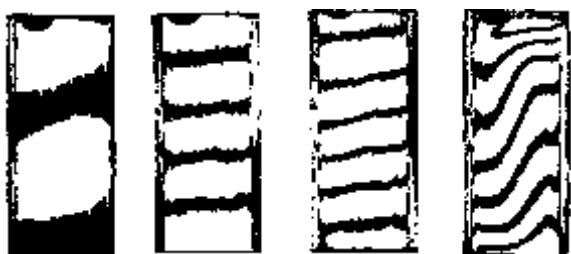
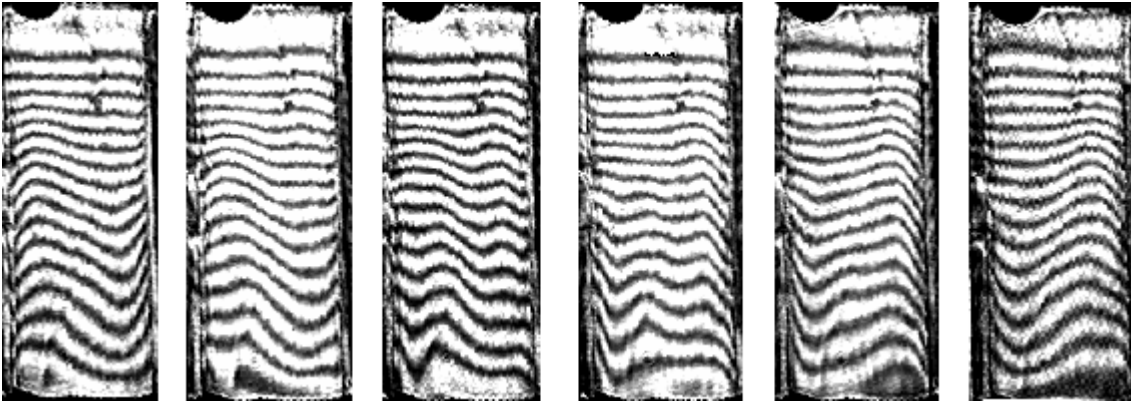


Figure 4.



**Figure 5.**



**Figure 6.**