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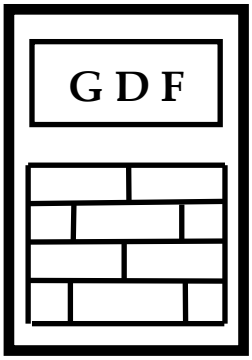

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DTA study of water freezing. I. Upon some aspects of repeatability.

Summary

Isothermal freezing experiments on water were performed in the latest 6 years by using differential thermal analysis (DTA) in view to establish Arhhenius and Universal relationships as they have been applied for a large variety of thermally driven processes. Values of the induction time for crystallization process showed large variations in the apparent repeatable conditions leading to improve progressively experimental conditions. Finally, I established that mental field created around the experimental place governs the water crystallization, so such experiments may be used to evaluate the risk of diseases in human communities.

Introduction

Previous works on water and aqueous solutions [1-6] have revealed their composite structure similar with crystalline polymers [7, 8]. More exactly, liquid water has a crystalline phase and an amorphous (inter-crystalline) phase responsible for solution processes [1, 5, 6] and other treatments applied on it [2-4]. By melting of completely frozen water or aqueous solutions the two phases were developed as two endotherms revealing their coupling [2].

The main goal of this series of experiments was to evidence the Arrhenius and Universal behaviour of crystallization process of water and some aqueous solutions in view to reveal their structural details in relation with applied treatments and composition according to topoenergetic principles [9]. Differential thermal analysis (DTA) was adopted as the simplest and most accurate measuring procedure by transferring the water specimen from room temperature to the freezing temperature. This steplike type experiments obey the basic topoenergetic principles and it was efficient for a large number of transforming processes [9].

In 2006 I resumed experiments on isothermal freezing of water, so a huge number of experiments have been performed in more and more accurate conditions in view to control some of the main driving potentials, namely: temperature, volume and sampling conditions of water specimens, thermal transfer function of DTA disposition, room temperature (RT) and relative humidity. Experiments revealed two distinct exothermal processes: one instantly occurred and associated to the amorphous phase generally denoted as inert component, Cin, and another one delayed at an induction time (ti) associated to the crystallization process as transforming component, Ctr. These two processes correspond to the general topoenergetic principles [7-9]. Unfortunately (or contrary fortunately), very strange behaviour relative to the other thermally driven transforming processes studied in the same manner was observed, namely

ti values show wide dispersion not obeying Arrhenius and Universal laws.
More exactly, crystallization process of water (called for simplicity as Ctr-process) is governed additionally by other potential(s) not identified and uncontrolled yet. Both processes (Cin and Ctr) have lambda shape specific to disorder-order transitions, more prominent Ctr process. Furthermore, by melting of completely frozen specimens (both Cin and Ctr occurred), the T1 and T2 endotherms appear as order-disorder processes [2], similar as in crystalline polymers [7, 8, 10].
Although these potentials driving the behaviour of Ctr process are not established completely yet, latest experiments clearly showed that the mental field created around the experimental place can block or allows Ctr process, so that these results could substantiate the experiments reported by Masaru Emoto [11], pharmacodynamic potency of homoeopathic remedies and water memory induced by mental activity. Experiments on mental activity and field created showed its coupling with a wide category of transforming processes [12]. HuPoTest [13] and some others techniques (to be published) are able to evaluate mental activity/field in view to establish an objective cause-effect correlation.

Experimental details

Figure 1 shows the cross section of the freezing thermostat and details for temperature (T) and DTA (dT) sensors. This thermostat is placed in a 20 L bucket with small polystyrene (PS) balls as thermal insulation (not shown). T sensor is LM335 giving freezing temperature in Kelvin read on a 4 ½ digital voltmeter. T was previously calibrated on site by using glass thermometers with liquid column with 0.01 ^{0}C marks.
DTA sensors consist in two pairs of diodes glued with Araldite® on polyester (PES) disk. A plate of Kanthal® with dimensions of 4x5x0.1 mm was glued on each pair of diodes (not shown). The water specimen was placed on one of these plates, the same for all DTA scans. The three DTA terminals were continued with copper wires (CuEm Ø 0.3 mm) fixed on a rigid plastic board with dimensions of 2x15x300 mm ending with a connector for electronic block. Differential signal is amplified by a fixed factor of 1000 with manually adjusted offset. The resulted signal is taken by a USB data logger with 12 bit resolution on the range of ± 2 V and sampling rate of 1 S/s. The plastic board with DTA sensors assembly is maintained all time in vertical position and PES disk with dT sensors in horizontal position.
Series of 10-20 DTA scans were performed in one working day. Majority of experiments, especially the latest ones considered in this report, were performed between 9 am – 3 pm when I was alone in all house. Important to mention that our house is separated by large yards from other houses in a very quiet place and suburb. At 7 am the freezing thermostat was prepared, so up to 9 am temperature was already equilibrated and keeps as constant over a period of at least 8 hours.

The main steps of each DTA scan are:

(i) cleaning of DTA sensors, especially to remove water traces;

(ii) placing of water specimen; I used initially a bamboo stick, but because no significant differences were observed and for a better control of specimen volume I used micro-syringes of 10 and 50 μL (L for litre).

(iii) The DTA assembly with the specimen is maintained at RT until the dT signal reached a constant value due by endotherm of vaporization (approx. 5 minutes).This signal gives the vaporization thermal flow (w vap) (Figures 2 and 4). The results considered here are obtained in RT = 26 ± 2 ^{0}C and Relative Humidity (RH) = 58 ± 3 %.

(iv) DTA assembly is transferred shortly in the plastic tube of freezing thermostat so the PES disk makes contact with the brass block. For a better and constant thermal transfer function several drops (approx. 0.5 mL) of paraffin oil is initially added.

After each primary DTA scan (dT) with water specimen, a blank DTA scan (dTblank) was performed without water specimen (both scans with the same offset value!), in view to remove the unbalanced heat capacity of DTA sensors and obtaining the secondary DTA scan as (dT – dTblank). By repeating several times dTblank it was possible to check the repeatability of experimental conditions.

DTA primary and secondary scans called as IN VIVO measuring systems [2, 7, 8, 10] give exotherms Cin and Ctr. By transferring the DTA assembly from freezing thermostat after both these processes occurred in an identical plastic tube at RT (here sensors assembly with frozen water specimen is maintained in air at a fixed position), it is possible to record the T1 and T2 melting endotherms. This IN VITRO measuring system was previously used in similar cases [2, 7, 8, 10].

Water samples: fresh tap water, boiled and cooled tap water, succussed boiled and cooled tap water (100 mL a in glass ampoule with glass stopper, 2 beats/s for 6 minutes), fresh rain water, concentrated solution of CuSO4.

All uncertainty values given in the present study are standard deviations for 68.3% confidence level, excepting RT and RH for which (±deviation) correspond to minimum and maximum values.

Results and discussion

Figures 2, 3 and 4 show typical IN VIVO primary, secondary and IN VITRO DTA scans, respectively, for the same 10 μL water specimen. The main quantities considered in this study are graphically shown.

Figure 5 shows the relationship between Cpsolid and Cpliq resulted from primary DTA scans for a series of water specimens where Cpo = unbalanced

DTA heat capacity is revealed. Figure 6 shows this relation from secondary DTA scans for which results Cpo=0.

Figure 7 shows this relation for a large series of water specimens resulted from primary DTA scans separated in thermograms with both Cin and Ctr exotherms and only Cin (ti > 3000 s).

During a large number of experiments, I observed that there are some important conditions in view to obtain close results for the same water sample and different specimens. In Tables 1-3 are given three successive series of results obtained in more and more accurate conditions.

Table 1 shows two series of results obtained when I was alone in house (all specimens having both exotherms) and when family arrived home the same sample plus other ones have had Ctr process inhibited for ti > 3000 s.

Table 2 and 3 show subsequent results obtained when I was alone in house and more mentally focused on the experiments. These experiments were performed in the period of four month HuPoTest study on my mental state reported recently [13].

It is important to point out the following observed facts:

1. Cin and Ctr processes have a lambda shape, more pronounced for Ctr. The Ctr shape flattens and the top of the peak becomes horizontal line by increasing the specimen volume, see the ratio A2/h2 = peak (area/height);
2. water traces from previous specimen can trigger Ctr process in the next specimen;
3. a specimen for which the first DTA scan had not Ctr process (ti > 3000 s), at the second scan shows Ctr process at small ti values (100-300 s);
4. ti(not succussed water) > ti(succussed water);
5. for the same tap water sample it results the following decreasing order of uncertainties: fresh > boiled-cooled > boiled-cooled-succussed;
6. IN VITRO DTA scans give an average value of $\alpha = h1/(h1+h2) = 0.83 \pm 0.06$ for splitting coefficient of melting process.
7. Specimens with only Cin exotherm, show only T1 endotherm/exotherm in the melting thermogram. This fact substantiates once again that crystalline and amorphous phases co-exist separately even in liquid state [2,7,8,14]. After Cin exotherm amorphous and crystalline phases progressively separate accumulating between them mechanical stresses [2] responsible for volume increase and T1 endotherm/exotherm.

Concluding remark

Due by their high efficiency, such experiments must be performed in other places in view to establish by inter-laboratory comparisons more accurately the cause-effect relationships on Ctr process and to correlate the results with morbidity (especially for cancer and diabetes) in different human communities [15].

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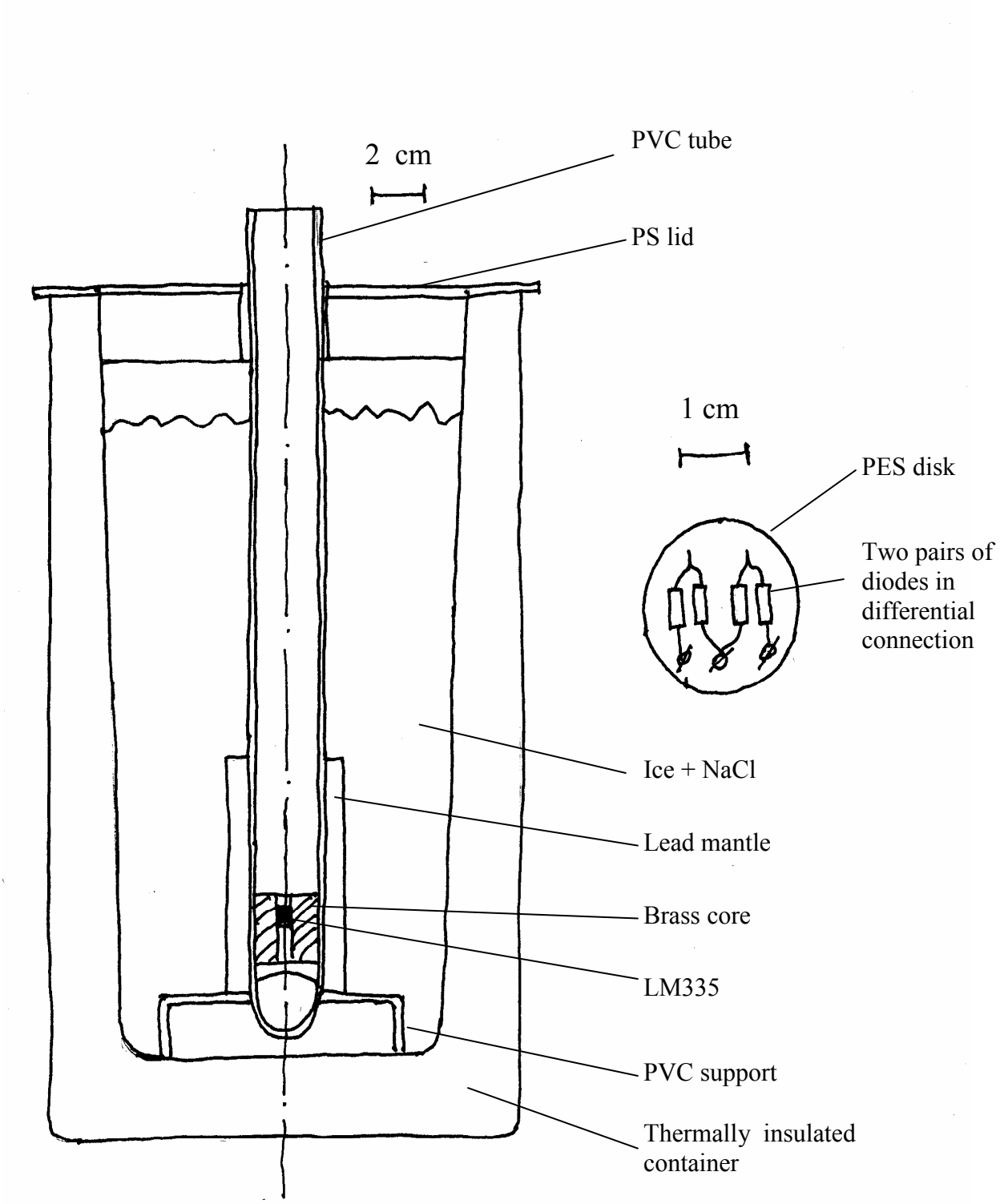

Figure 1. Cross section of DTA disposition with details for T and dT sensors.

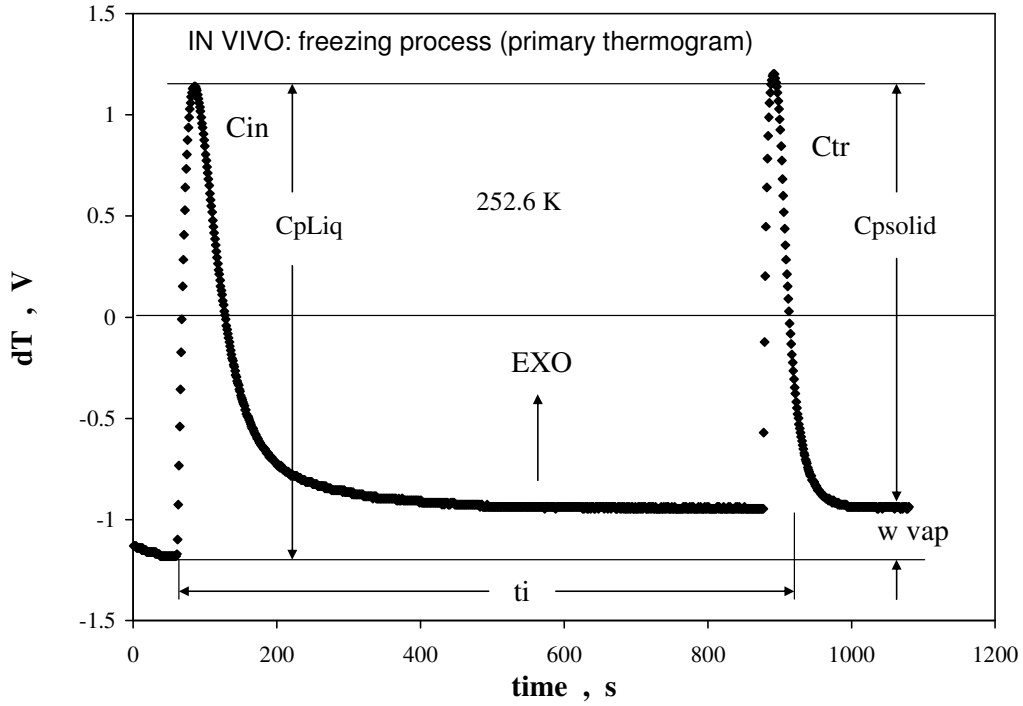

Figure 2.

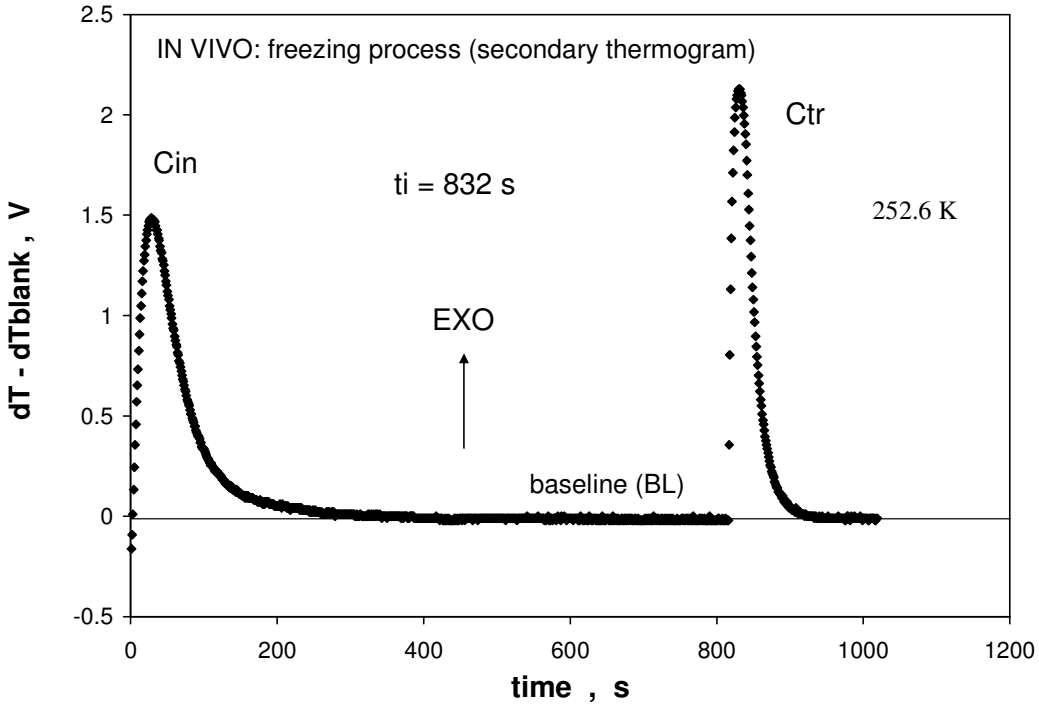

Figure 3.

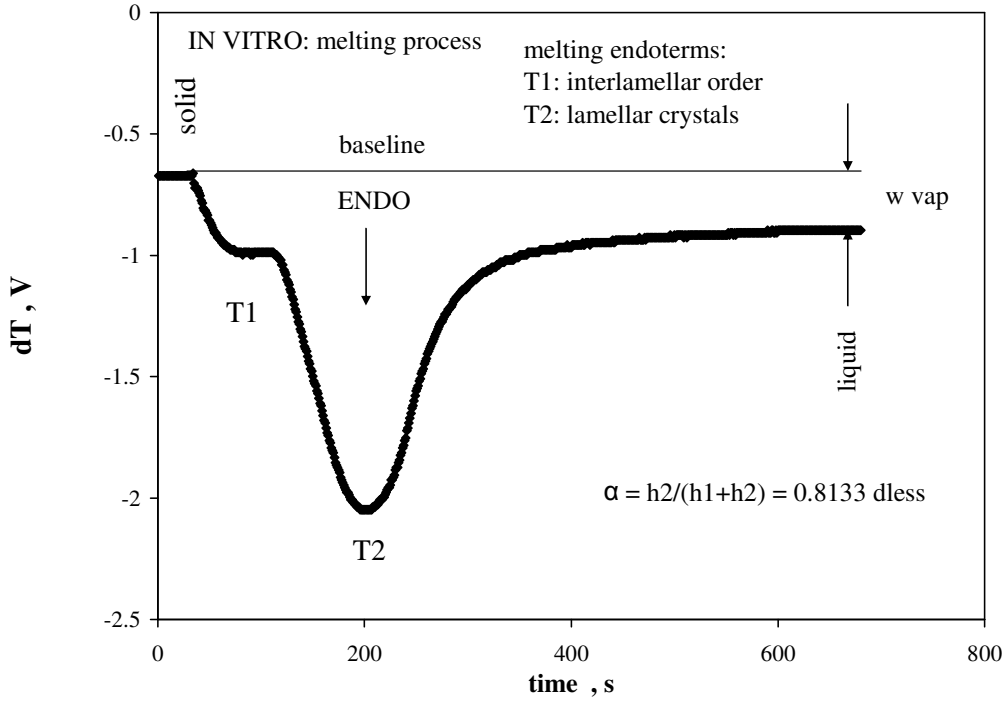

Figure 4.

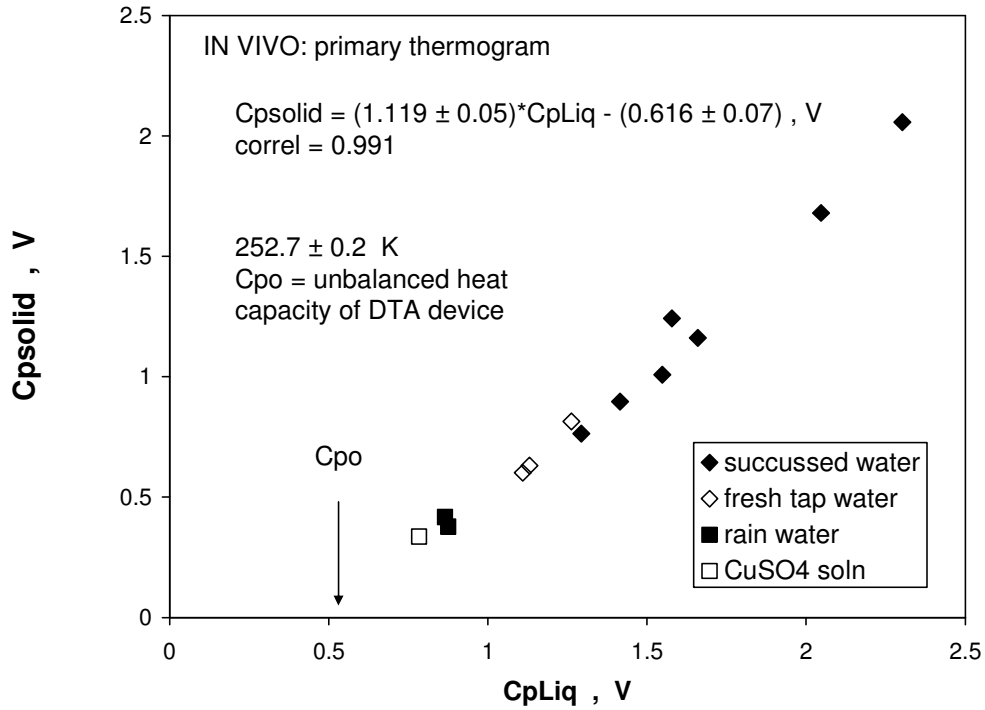

Figure 5.

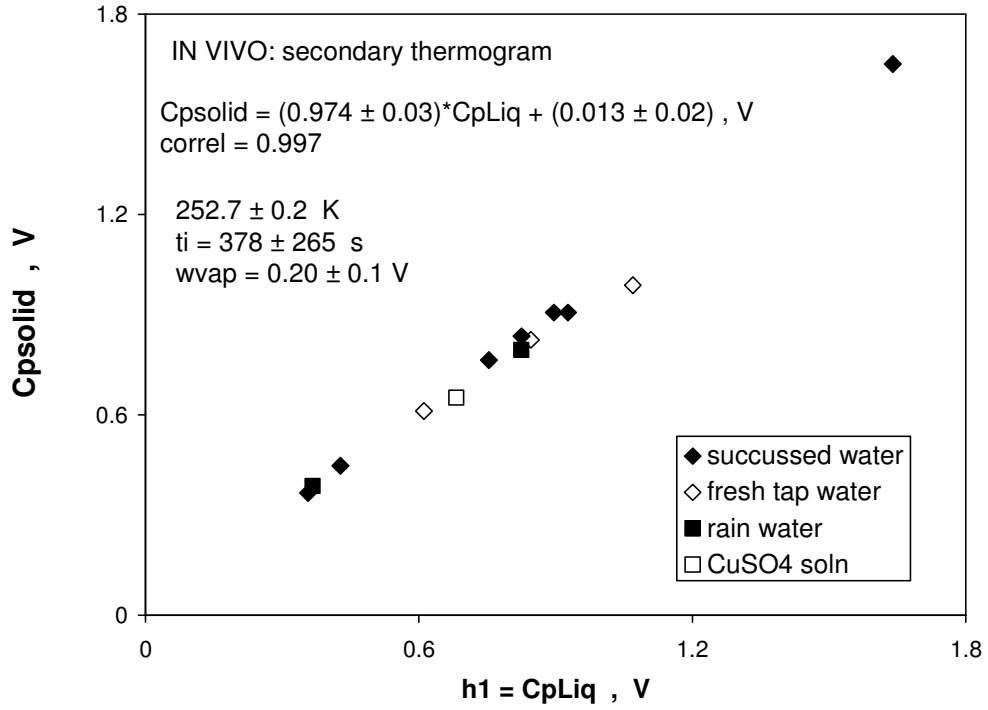

Figure 6.

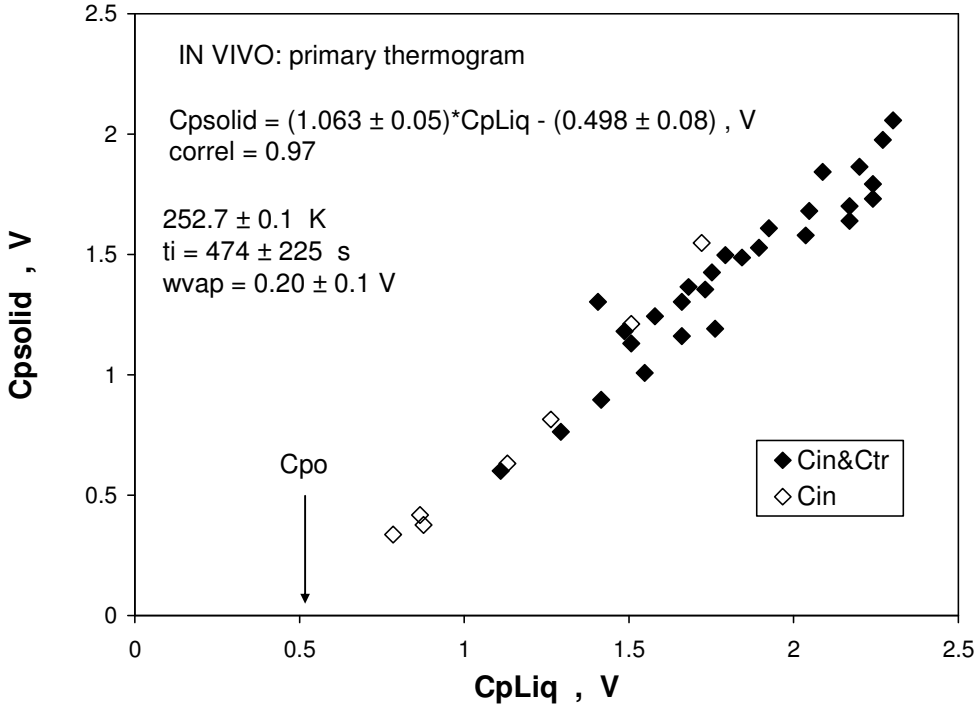

Figure 7.

Table 1

specimens	No. scans	ti , s	h2 , V	A2 , V*s	A2/h2 , s	w vap, V	T , K
10 µL boiled-cooled kept 24 hrs @ RT, succussed 2Hz/6 minutes	7	378 ± 265	2.11 ± 0.1	94 ± 5	45 ± 2	0.20 ± 0.1	252.7 ± 0.2
10 µL idem + different waters	12	ti > 3000 s					

Table 2

10 µL fresh tap water	18	343 ± 188	1.60 ± 0.2	88 ± 10	55 ± 3	0.186 ± 0.09	252.9 ± 0.4
30 µL fresh tap water multiple scans on a single specimen	11	221 ± 19	1.71 ± 0.2	215 ± 18	126 ± 4	0.38 ± 0.1	253.3 ± 0.4

Table 3

10 µL boiled-cooled kept 24 hrs @ RT	5	484 ± 119	2.26 ± 0.2	106 ± 13	47 ± 4	0.131 ± 0.05	254.6 ± 0.9
10 µL boiled-cooled kept 24 hrs @ RT, succussed 2Hz/6 minutes	5	205 ± 23	1.84 ± 0.2	88 ± 10	48 ± 3		254.8 ± 1

All results are obtained IN VIVO DTA scans; h2 = peak height and A2 = peak area for Ctr process.

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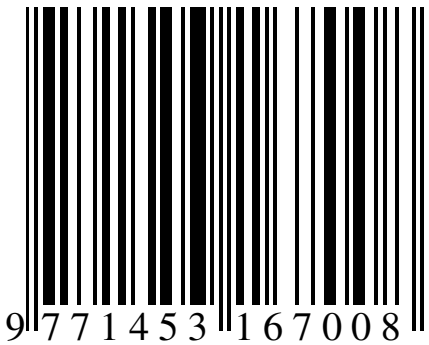

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