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High Resolution Mixing Calorimetry (HRMC) redivivus.
1. General presentation and heat capacity measurements.

MOTTO: ***Material science without calorimetry is blind.***

HRMC was initiated in 1983 after a long experience in calorimetry with the view to evidence in an efficient manner the interaction between water and different solutes [1].Solubility was the key word in starting and developing the topoenergetic principles [2] and calorimetry introduced the polarity of transforming processes [3]. HRMC became rapidly a highly efficient instrument (rapid, sensitive, reproducible, easy to use, low cost) in revealing the composite structure of water and aqueous solutions, namely the co-existence of at least two main distinct phases in liquid phase: one ordered = the so called "crystalline phase" untouched by mixing with solute and an amorphous one participating to solution process. HRMC experiments were focused on structures of water submitted on a wide category of treatments by mixing with specific "structural developers" as ethanol, methanol, phenol, amines, etc. Furthermore, HRMC technique was used for evidence the kinetics of a large kind of processes triggered by mixing: cement hydration, catalysis, solubility of a large category of substances (for instance pharmaceuticals). Special experiments were carried out on cold fusion processes triggered and controlled both by caloric and electric quantities (results to be published). HRMC was used as scanning calorimetry and for heat capacity measurements as well [4].

The main purpose of present notes is to point out some qualitative features of HRMC technique by considering water and a series of aqueous solutions. HRMC and in general all analytical techniques are greatly improved by digital recording and retrieval of experimental results.

Figures 1-4 present basic parts of HRMC. Figure 5 presents the schematics of operating procedures. Figure 6 present the cross section in the calorimetric cell used for heat flow calibration and Figure 7 the cross section in the calorimetric cell assembly used for mixing experiments.

One of the most important structural quantities unreasonably neglected by researchers is heat capacity and specific heat. It is a direct measure of degrees of freedom of kinetic entities. One explanation could be the difficult experiments by classical adiabatic calorimetry, but modern differential scanning calorimetry (DSC) can do that in highly efficient manner. In fact, the real explanation is that calorimetry is not taken seriously and this is one of symptoms of actual science.

Some heat capacity results on water and aqueous solutions are presented. The experiments were carried out at room temperature ($23 \pm 2\ ^0$C), all on the same sensitivity and by controlled heat pulses with the same power (Ph = 2W) and different time periods, tp (H1, Figure 5). Figure 8 shows the subsequent endothermal thermograms: S – primary endothermal; E – endothermal of empty calorimetric cell; their difference (S-E) by considering common starting point (t=0). There is showed also (S-E) endothermal with corrected baseline (difficult to be distinguished on the graph). Figure 9 present the series of final corrected (S-E) for different tp values and Figure 10 gives the main parameters taken into consideration. Energy values E1 and E are obtained by summation of all heat flow values, w(t) measured from baseline. Figures 11-16 present the results on specimens of 1 mL. All thermograms were recorded with 16 bit resolution at 1 s sampling rate and digitally processed in Excel under Windows®. Table 1 gives the main results obtained on 1 mL specimens. Figures 17-22 show similar results for saturated solution CuSO4anh-sat on specimens of different volumes. In a future note details on structural significances of these results will be discussed.

Conclusion: After a long experience in material testing and especially by developing calorimetric techniques, HRMC is a highly efficient tool in evidencing kinetics and structural details of large categories of reacting systems. I use to encourage researchers involved in material science to adopt HRMC experiments in their projects: without calorimetry material science is blind.

References

[1] G.Dragan, Study of the interaction between ionic salts and water by HRMC, J. Thermal Anal., 31, 679-691, 941-954 (1986); 32, 293-300 (1987).

[2] G.Dragan, Solubility behavior introducing topoenergetic working principles, GDF Databanks Bull., 1(1), (1997).

[3] G.Dragan, Polarity of the transformation processes in thermal measuring systems, J. Thermal Anal.,23,173-183 (1982).

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Figure 1. Front view of the HRMC basic disposition

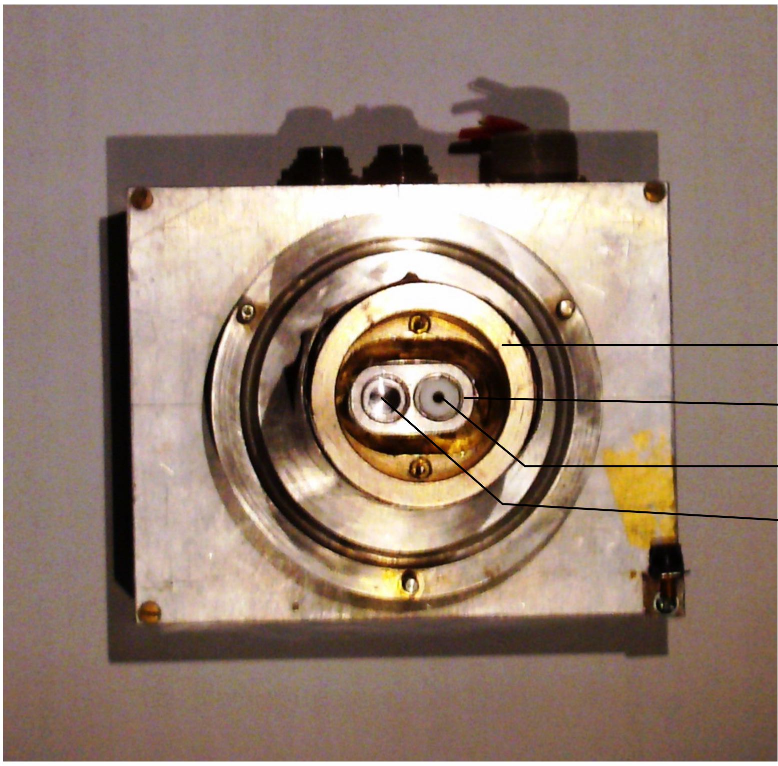


Figure 2. Top view of HRMC basic disposition (see operating schematic in Figure 5).

Outer temperature controlled block

Inner block with calorimetric cells

Reference calorimetric cell

Calorimetric cell for specimen under test

Figure 3. Basic electronic assembly.

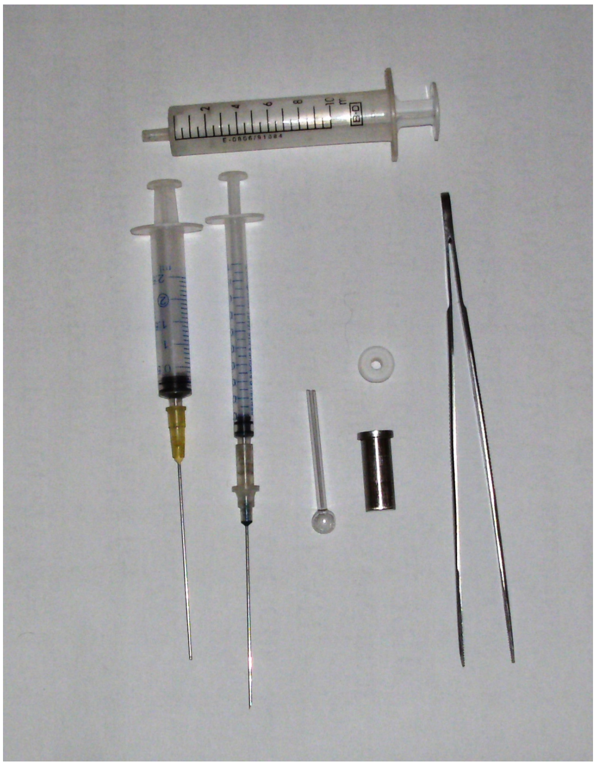


Figure 4. Calorimetric cell for test specimen (component 1), glass bubble for component 2 and most used tools (tweezers, syringes of 1 mL for liquid component 2, 2 mL for liquid component 1 and $\geq$ 5 mL for washing calorimetric cell.

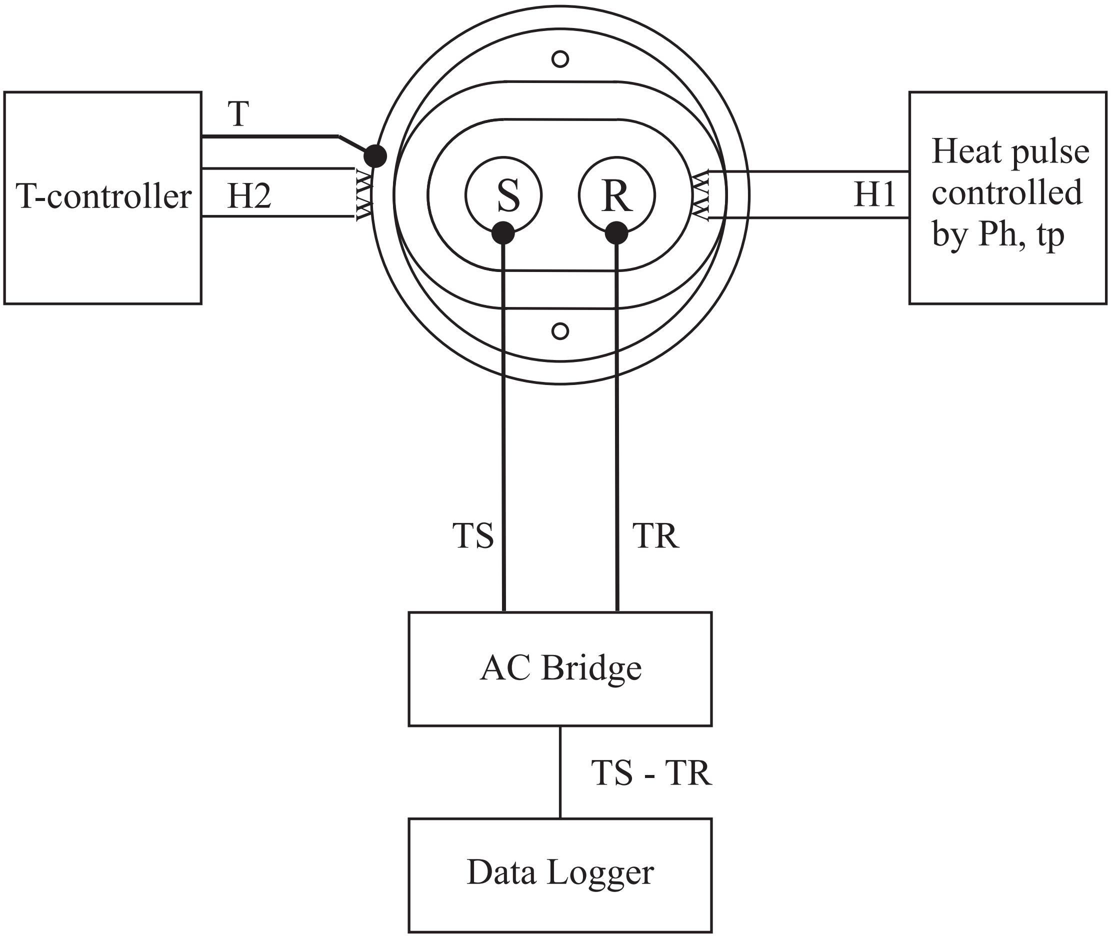


Figure 5. HRMC operating schematics.

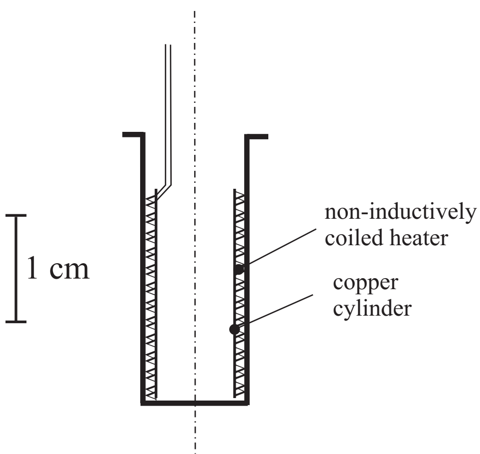


Figure 6. Cross section of calorimetric cell used only for calibration.

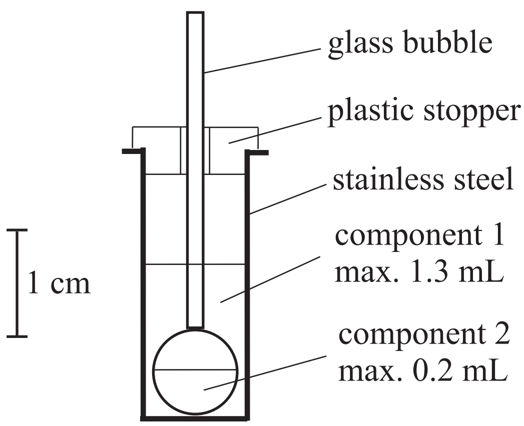


Figure 7. Cross section of calorimetric cell used for mixing studies.

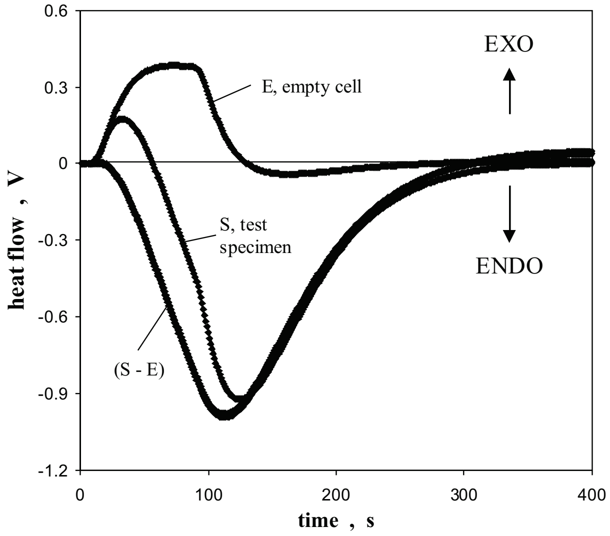


Figure 8.

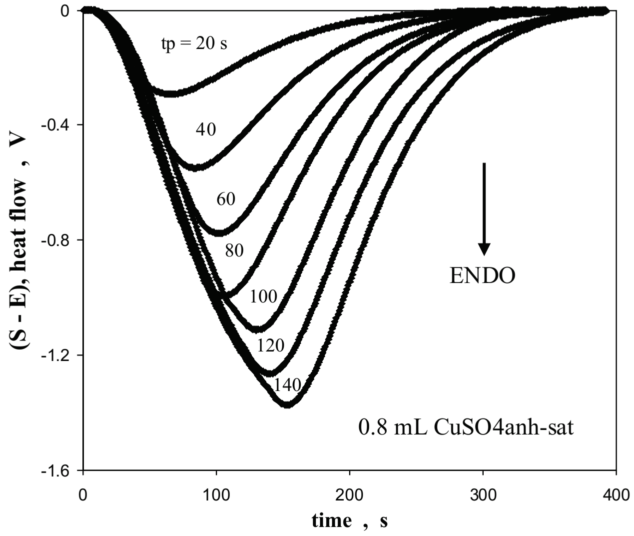


Figure 9.

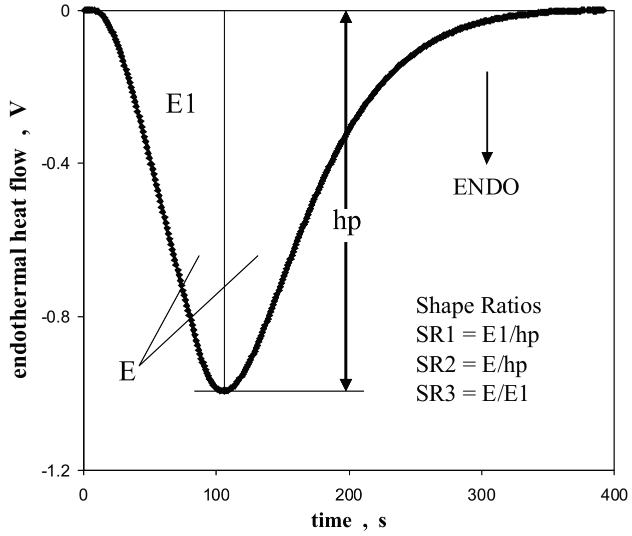


Figure 10.

Table 1. Relationships of main kinetic parameters associated to pulse experiments on aqueous specimens of 1.0 mL (Figures 11-16).

tested specimens	E = n1*tp + m1				E1 = a*tp^b				E/E1 = n1*tp + m1			
	n1	m1	u(n1)	u(m1)	a	b	u(a)	u(b)	n1	m1	u(n1)	u(m1)
water	1.959	1	0.1	10	0.118	1.424	0.04	0.08	-0.0118	3.64	0.002	0.2
CuSO4anh-sat	2.021	0.01	0.004	0.3	0.163	1.367	0.07	0.09	-0.0094	3.29	0.002	0.1
Na2CO3anh-sat	1.931	3	0.1	9	0.162	1.343	0.04	0.05	-0.0105	3.54	0.002	0.2
Na2SO4anh-sat	1.913	-0.4	0.006	0.5	0.148	1.374	0.06	0.08	-0.0101	3.37	0.0009	0.08
MgSO4.7H2O-sat	1.782	14	0.05	5	0.177	1.325	0.05	0.06	-0.0136	3.87	0.0002	0.02
Glycerol 40%vol	2.018	-4	0.2	13	0.136	1.389	0.05	0.07	-0.0093	3.43	0.004	0.3
Glycerol 50%vol	1.694	2	0.03	2	0.103	1.419	0.02	0.04	-0.0146	3.95	0.0009	0.08
Glycerol 60%vol	1.533	10	0.09	8	0.098	1.417	0.02	0.04	-0.0174	4.27	0.0007	0.06

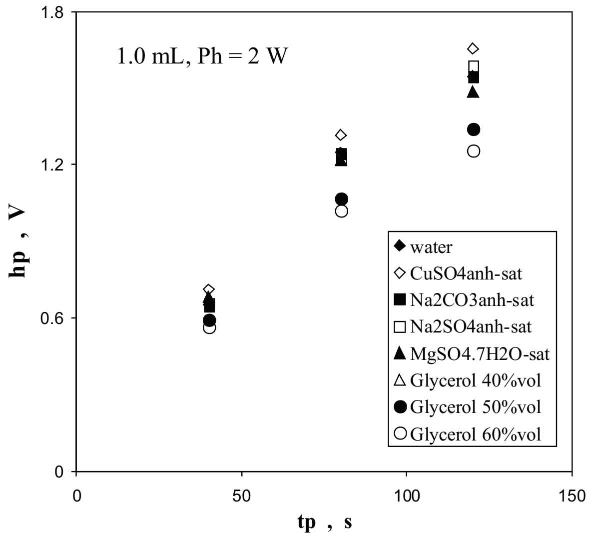


Figure 11.

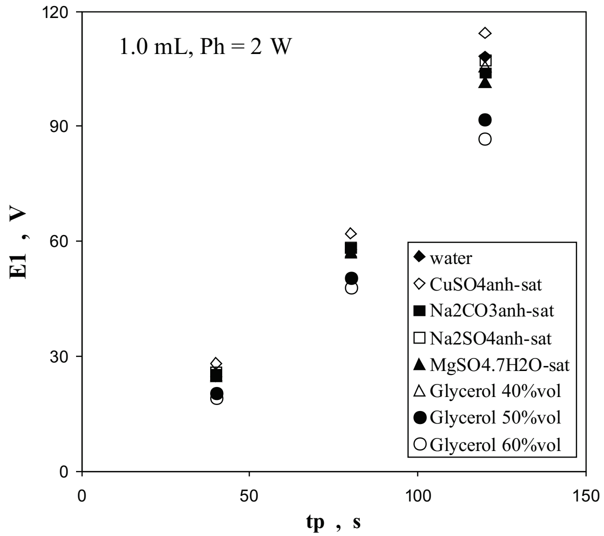
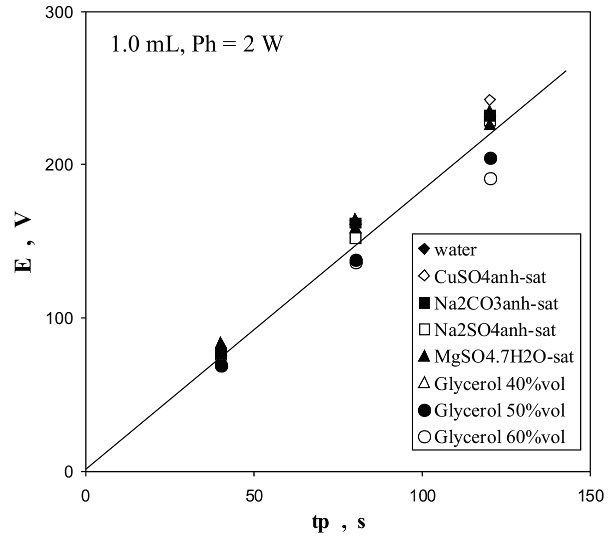


Figure 12.



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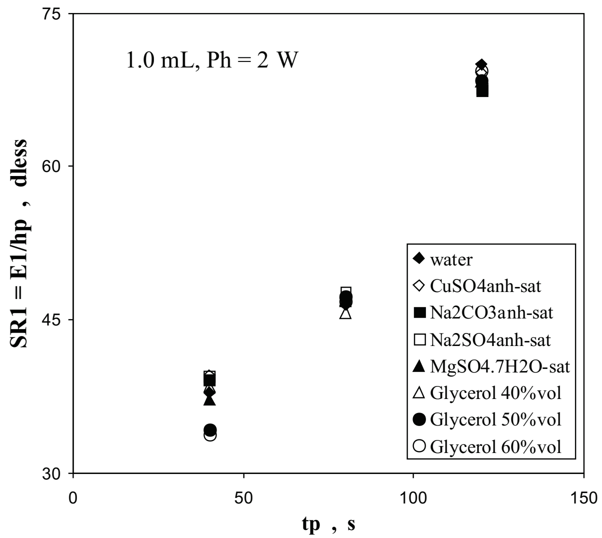

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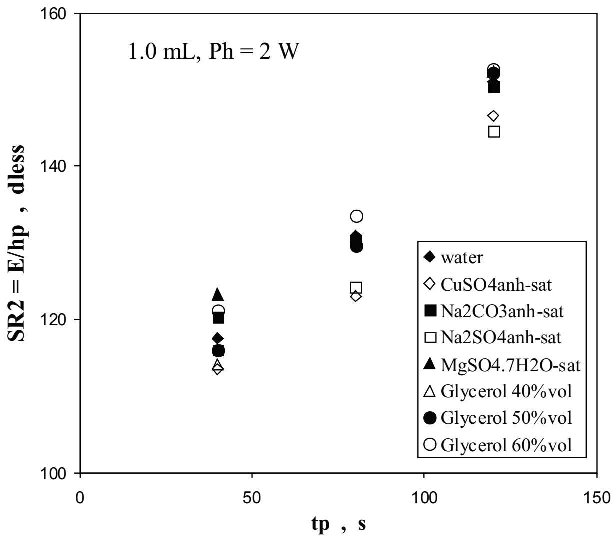

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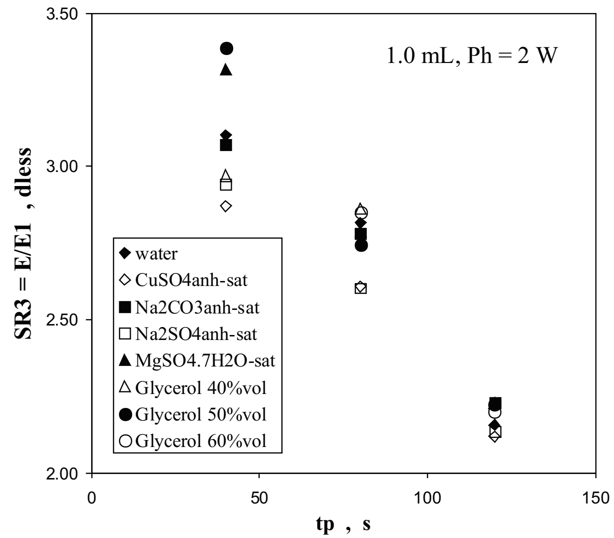


Figure 16.

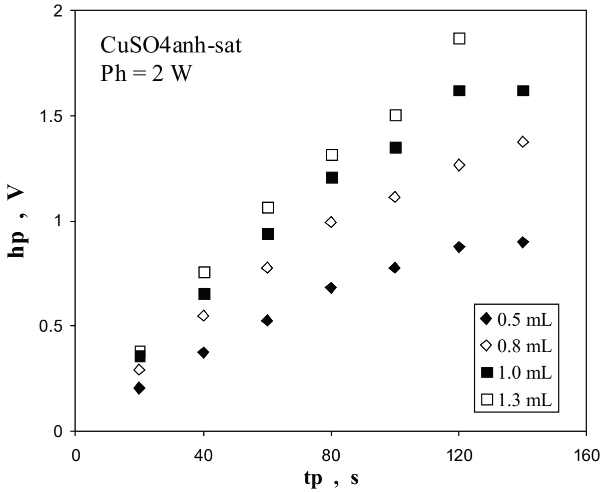
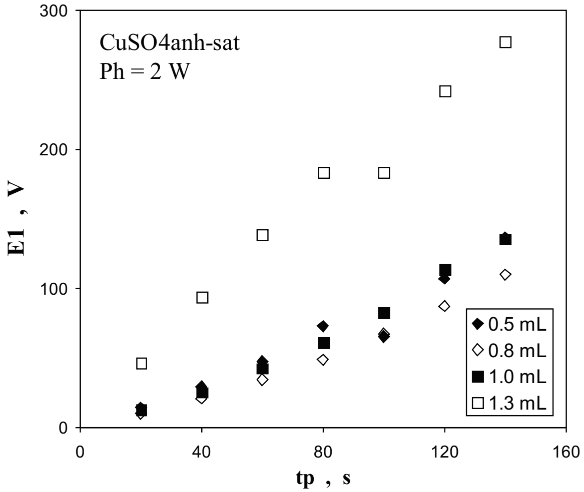


Figure 17.



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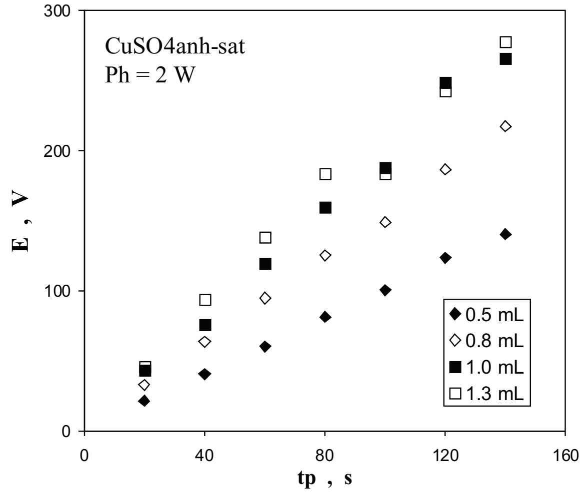

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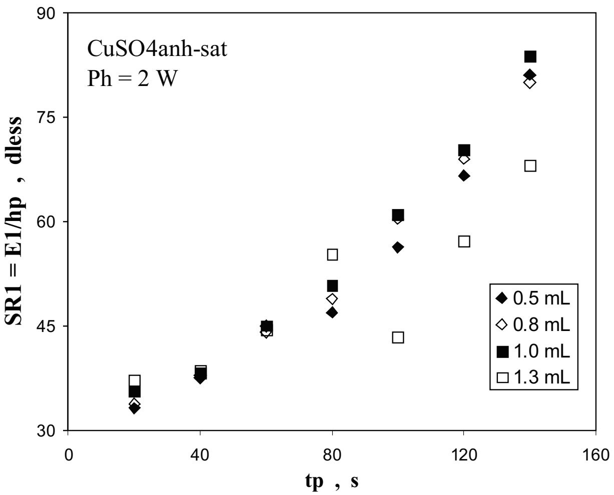

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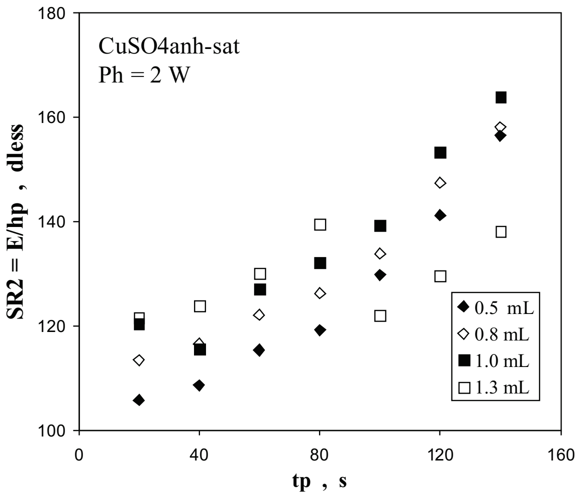

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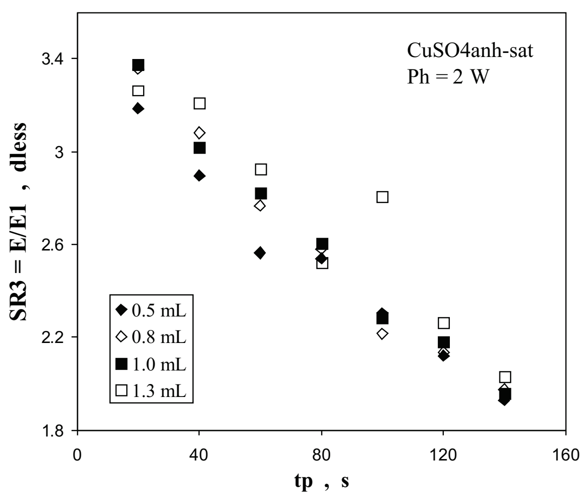

Figure 22.

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1997	1	2	Guide of good practice in metrology (Romanian)	AFI
1998	2	1	Editorial: socio-psychological implications in creation and utilization of a databank (Ioan-Bradu Iamandescu); Behavior in vapor-liquid equilibria (VLE): I. Structural aspects; Behavior in vapor-liquid equilibria: II. Several structures in databanks; Symposium on VDC-4 held on 30 October 1997 at Lubrifin-SA, Brasov (Romania).	F
1998	2	2	Practical course of metrology (Romanian)	AFI
1998	2	3	DIFFUTOR-01: Thermally driven diffusion in pure metals	AFI
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2000	4	1	Editorial: Laboratory accreditation and inter-laboratory comparisons (Virgil Badescu) Doctoral Theses – important data banks. GDF intends to open new series of experiments on thermo-physical properties. Some comments on uncertainty: global budget and DFT analysis. Events: The 9th International Metrology Congress, Bordeaux, France, 18-21 October 1999.	F
2000	4	2	Measurement and Calibration.	AFI
2001	5	1	Editorial: Metrology ensures moral and technological progress. Topoenergetic aspects of amorphous-crystalline coupling. I. Composite behavior of water and aqueous solutions (paper presented at nanotubes and Nanostructures 2001, LNF, Frascati, Rome Italy, 17-27 October 2001). Events: Nanotubes and nanostructures 2000.School and workshop, 24 September – 4 October 2000, Cagliari, Italy.	F
2001	5	2	Editorial: Viscosity – a symptomatic problem of actual metrology. Visco-Dens Calorimeter: general features on density and viscosity measurements. New vision on the calibration of thermometers: ISOCALT® MOSATOR: Topoenergetic databanks on molten salts properties driven by temperature and composition.	F

2002	6	1	MOSATOR-01: Topoenergetic databanks for one component molten salts; thermally driven viscosity and electrical conductance.	AFI
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2004	8	1	Metrological verification and calibration of thermometers using thermostats type ISOCALT® 21/70/2. Metrological verification and calibration of thermometers using thermostats type ISOCALT® 2.2R.	F
2004	8	2	Aspects of correct measurements of temperature. I. measurement of a fixed point according to ITS-90. Physics and Homoeopathy: some physical requirements for homoeopathic practice.(Plenary lecture at the 19th SRH National Congress, 21-22 September 2004, Bucharest, Romania)	F
2005	9	1	AWARD for ISOCALT® at the International Fair TIB-2004, October 2004, Bucharest. ISOCALT® 3/70/21 was awarded in a selection of 20 products by a commission of experts from the Polytechnic University of Bucharest. Upon some aspects of temperature measurements. (12th International Metrology Congress, 20-23 June 2005, Lyon, France)	F
2005	9	2	A new technique for temperature measurement and calibration. National Society of Measurements (NSM). Important warning for T-calibrator users: MSA has chose metrology well calibrators from Fluke (Hart Scientific).	F
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2014	18	2	Mental field-water interaction as evidenced by Isothermal Convection Flow Calorimetry (ICFC). II. Effect of convection flow power.	F
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2015	19	2	Gold versus money. 2. Rich, middle and poor countries.	F

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