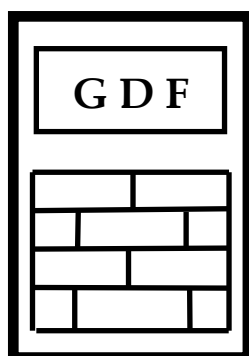


GDF DATA BANKS BULLETIN



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High Resolution Mixing Calorimetry (HRMC) redivivus.

3. Calibration

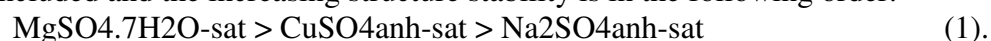
MOTTO: *Material science without calorimetry is blind.*

Previous results presented on HRMC experiments are obtained at room temperature and the same sensitivity of measuring ac-bridge [1, 2]. Parameters heat flow ($w(t)$), h_p , E and E_1 defined on thermograms are expressed in Volt, but taking into account that have different physical meanings. The aim of the present note is to establish the SI units of each such quantity by calibration. The special calorimetric cell with a heating element is used for this purpose and the power supply generating heat pulses controlled in power, $Ph(W)$ and time, t_p (Figure 5 and 6, [1]). The same power supply was used for heat capacity measurements [1].

All calibration experiments presented in this note were performed at the same sensitivity of the ac-bridge as in previous HRMC measurements.

Figures 1-3 shows series of thermograms (exothermals) obtained for three different Ph values. It can observe the different shapes as depending on both t_p and Ph . Figures 4-9 show the dependences of the main parameters defined initially on typical HRMC thermogram [1] as function of E in Joule as resulted from the calibration straight line in Figure 5 gathering values obtained in all calibration experiments. For each series of data the best fit functions are established by linear or non-linear regressions. These fits can be compared with the similar parameters resulted in other kinds of experiments [1, 2]. It is important to note that all these series of dependences are convergent to the same value for $E(J) = 0$.

It is also important to consider again the previous results obtained for heat capacity on 1 mL specimens (heat pulse experiments at the same Ph and $t_p=120$ s [1]) and mixing energy E_m (J) reported to mass of specimens obtained by mixing experiments on aqueous solutions by using absolute ethanol as structure developer [2]. Parameter h_p as expressed in $^{\circ}C$ is obtained by considering water as standard; h_p is also calibrated in unit of heat flow (J/s) according to the general purely dissipative law: $TS-TR = Rh*w$ ($Rh = 36.07$ $^{\circ}C*s/J$ = thermal resistance between sample and reference cells). Table 1 presents these data and Figure 10 shows the dependences of E_m/m (J/g) on the specific heat, $C_p(J/(g*^{\circ}C))$. It results different phylogenies defined by linear dependences gathering the same nature of structures, namely glycerol solutions with more stable structures than solutions of ionic salts, but increasing with glycerol content. In the series of ionic salts $Na_2CO_3anh-sat$ and water are not included and the increasing structure stability is in the following order:



Conclusions:

- (i) Calibration of HRMC quantities is necessary in view to make the right differences between nature and amplitude of revealed processes associated to mixing experiments.
- (ii) Mixing experiments on the same samples under test but by using different developers allow to evidence more accurately the nature and amplitude of their composite structure by establishing higher phylogenies.
- (iii) Heat capacity and specific heat appears important structural parameters obtained in accurate and rapid manner by HRMC technique revealing also the composite structure of tested samples completing structural data obtained by mixing experiments and other analytical techniques.
- (iv) Calorimetry, especially HRMC, must be promoted in material science research and technology as providing in highly efficient manner (accurate, rapid, easy to operate and low cost) structural information not available from much more sophisticated, expensive and difficult to operate analytical techniques.

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- [2] G. Dragan, HRMC redivivus. 2. Structure developing of aqueous solutions by mixing experiments, Databanks Bull., 19(2) 2015.

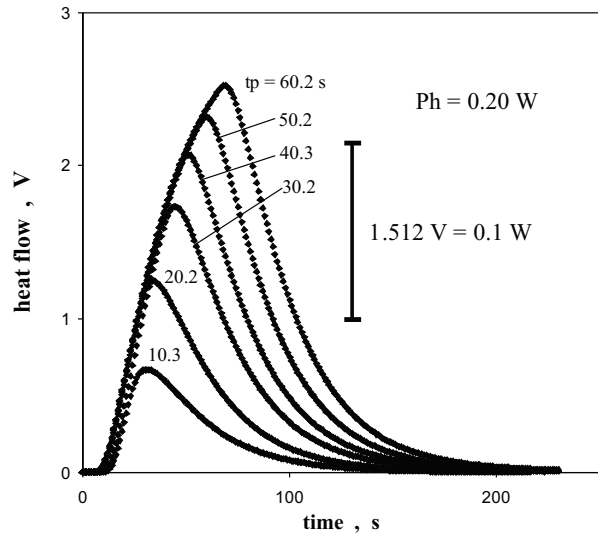


Figure 1.

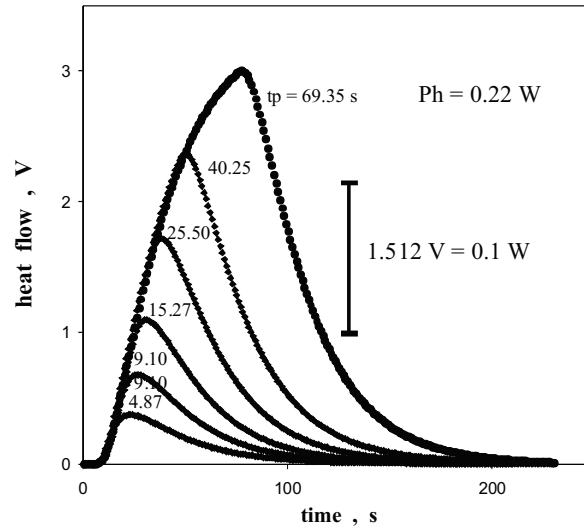


Figure 2.

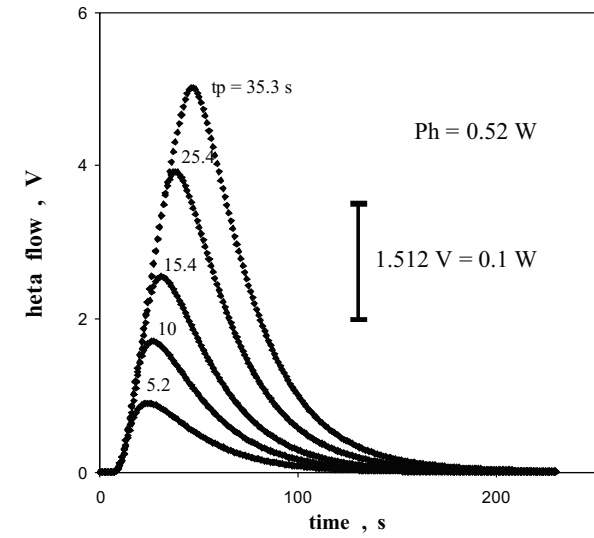


Figure 3.

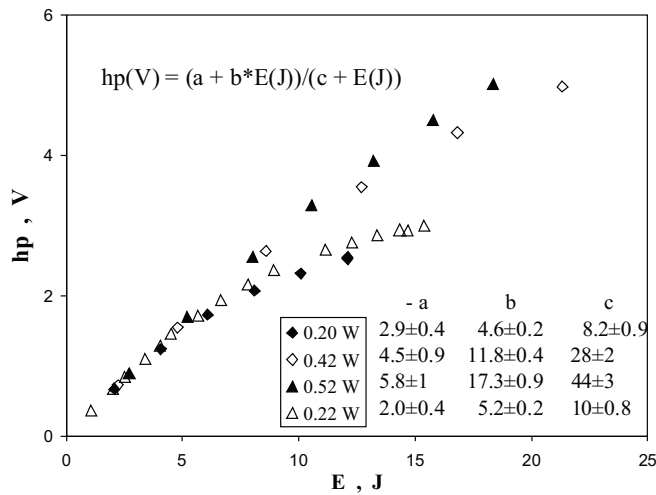


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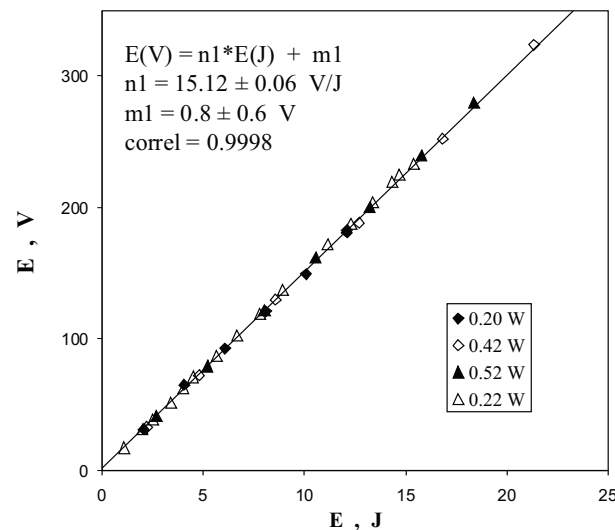


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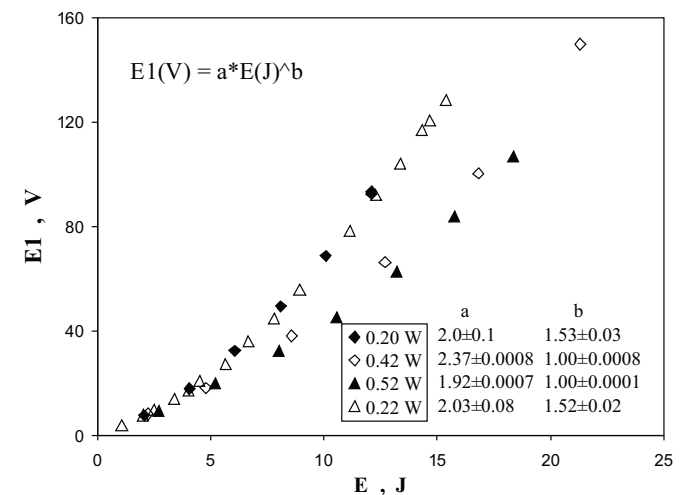


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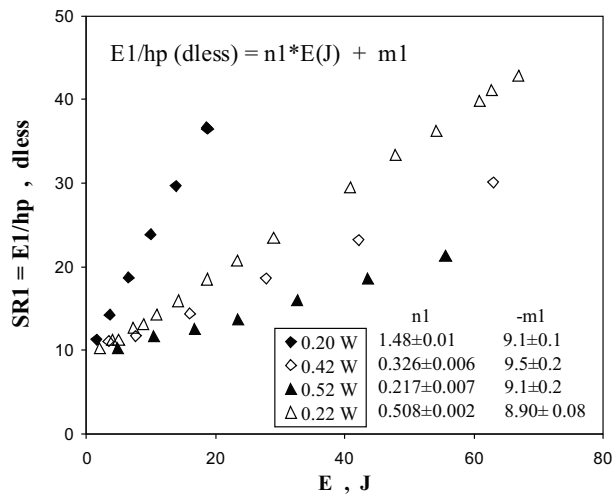


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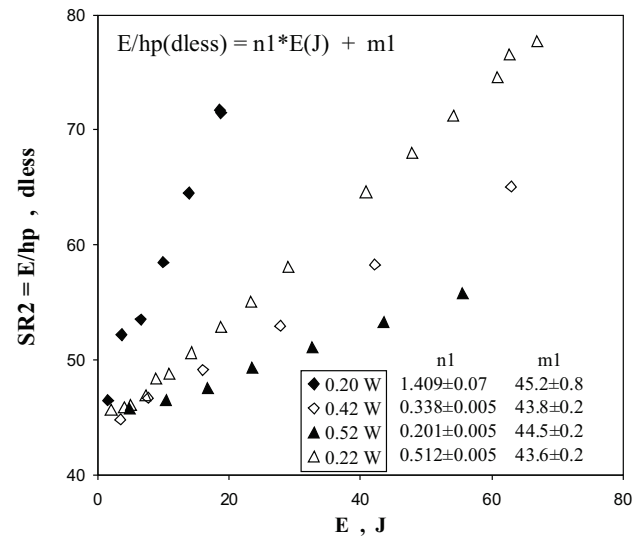


Figure 8.

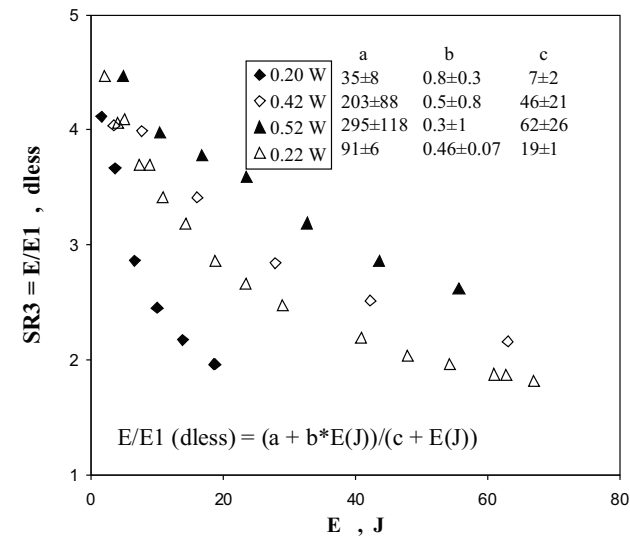


Figure 9.

Table 1. Comparative results obtained by heat pulse and mixing experiments after calibration of temperature and energy.

| | m , g/mL | heat pulse experiment, 1 mL, tp=120 s | | | mixing exp.* |
|-----------------|----------|---------------------------------------|-----------|---------------|--------------|
| | | hp ,oC | E/m , J/g | Cp , J/(g*oC) | Em/m , J/g |
| water | 0.9995 | 3.68 | 15.45 | 4.18 | 35.41 |
| CuSO4anh-sat | 1.1722 | 3.94 | 13.68 | 3.47 | 35.83 |
| Na2CO3anh-sat | 1.1208 | 3.67 | 13.69 | 3.73 | 22.13 |
| Na2SO4anh-sat | 1.0889 | 3.77 | 13.93 | 3.69 | 71.36 |
| MgSO4.7H2O-sat | 1.2667 | 3.54 | 11.83 | 3.34 | 14.15 |
| Glycerol 40%vol | 1.1222 | 3.68 | 13.86 | 3.77 | 4.20 |
| Glycerol 50%vol | 1.1515 | 3.20 | 11.73 | 3.67 | 2.62 |
| Glycerol 60%vol | 1.1889 | 2.99 | 10.64 | 3.56 | -1.31 |

all experiments at room temperature (23 ± 2 °C); * 0.5 mL soln. + 0.1 mL EtOH

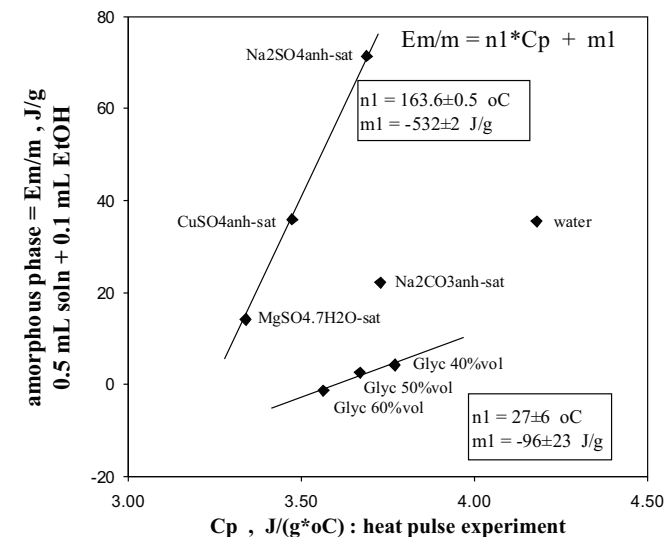


Figure 10.

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| 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 19 th SRH National Congress, 21-22 September 2004, Bucharest, Romania) | F |
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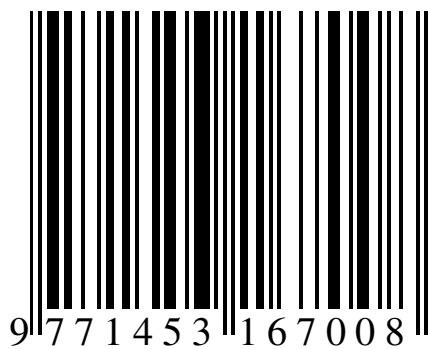
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