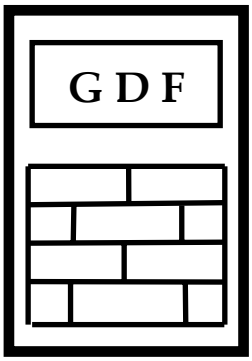
GDF DATA BANKS BULLETIN

Adiabatic Calorimetry



VOL. 12 , No. 3

SYDNEY March 2008

AUSTRALIA

Adiabatic Calorimeter

In fact represents a series of adiabatic calorimeters which can be adapted to any kind of processes triggered by mixing of initial components reacting exothermally (releasing heat) as: polymerization, curing, crystallization, hydration of anhydrous salts (in cement, plaster), combustion, fermentation, etc.

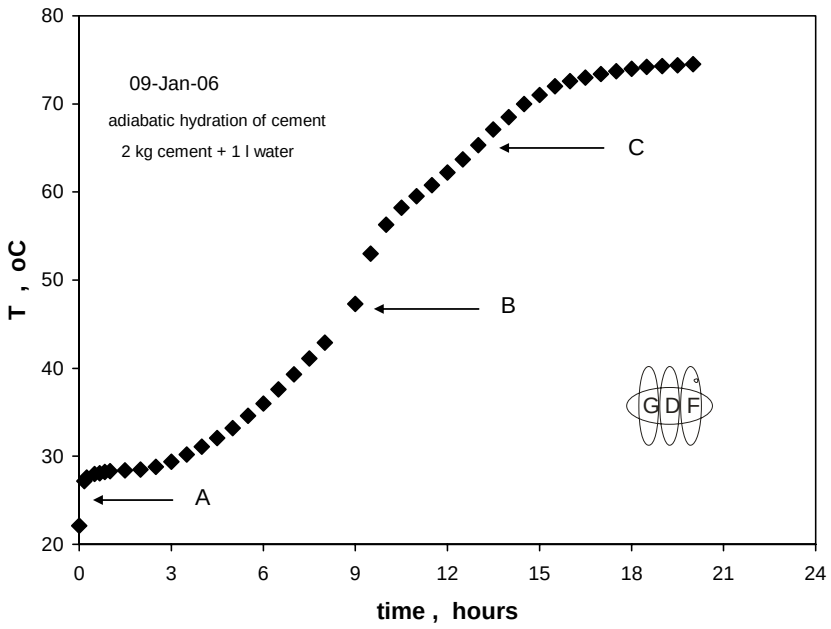
Adiabatic calorimeters and original procedures allow establishing the nature and the extent of participation of each component to the evidenced reaction process defining the properties of the end product. The following three main operations are performed:

1. efficient calibration of temperature, specific heat of components, heat capacity of reaction mass and released heat;
2. correct and efficient measurements of important quantities during process development ;
3. efficient processing of measured quantities in view to identify, optimize and scaling of evidenced process to different volumes taking into account that almost all processes dramatically change from laboratory size at industrial size.

Adiabatic calorimeter can be adapted to any exothermal process at its specific features by modifying and/or addition of accessories and measuring instruments for specific quantities, namely:

(i) volume and shape of sample holder;
(ii) temperature sensitivity and range;
(iii) hydrodynamic regime (mixing conditions of initial components and during reaction);
(iv) controlled atmosphere;
(v) calorimetric measurements in isothermal conditions.

Adiabatic calorimeter with 2 l sample holder for hydration of concrete mixtures in the temperature range of room temperature and + 110 ^{0} C (optional under room temperature) is shown in the picture below. Electronic blocks are available for all applications (calibration block not shown). Calorimetric test consists mainly in three distinct stages: (a) preparation of components; (b) start of adiabatic function, measurement of specimen temperature and mixing of components; (c) retrieval of temperature variation during process development according to original procedures. The given graphic was obtained for hydration process of commercial grade cement. Different stages of hydration are revealed characterized by temperature and time of reaction depending on the specimen volume, nature and proportion of reacting components.



APPLICATIONS: identification and/or optimization of reacting formula for a wide range of processes in chemical industry, pharmaceutical industry, food and beverage industry, building materials, electronics, mining industry, etc.

ADIABATIC CALORIMETER

Summary description of the demo prototype

Sydney – AUSTRALIA

February 2008

Content:

www.gdfdatabanks.ro dragan_gdf@yahoo.com

1. Main objectives of adiabatic calorimeter

Calorimetry represents a wide series of measurements based on heat transfer and/or temperature variation between a tested specimen placed in a specimen holder (SH) and an enclosure temperature of which is well controlled. Both surfaces are considered as equipotential, i.e. temperature has the same value in any point on each one.
In adiabatic calorimetry (AC) the SH temperature is determined by the processes triggered by different means in that specimen. SH and the outer adiabatic shield (AS) are considered as equipotential surfaces. AS temperature is maintained equal with the SH temperature in view as between the two equipotential surfaces the heat flow is always zero. Because the temperature control of an equipotential surface can be controlled with high accuracy by heating, but with poor accuracy by cooling, it results that adiabatic function (AF) can be done for exothermal processes (which release heat).
For instance, if the tested specimen does not show any transformation in a temperature range tested, we can give to it a well controlled amount (pulse) of heat and it results in AF condition a stepwise rise of SH temperature defining the heat capacity of the specimen = the heat amount necessary to rise the specimen temperature by 1 K. Heat capacity and specific heat (the heat amount necessary to rise temperature of 1 kg of specimen by 1 K) are quantities related with the structure of the specimen.
In general, transforming processes in tested specimens are triggered by mixing of at least two components and these are not constituted by only one process (elementary or unimodal processes), but a succession (cascade) of unimodal processes.
AC allows to evidence and to identify in efficient manner (simple and accurate) that cascade for a wide category of practical cases.

2. Important features of AC

The following features must be emphasized for users just before they begin work with AC:

2.1. AC is slow measurement extended on hours even months depending on the temperature accuracy and specimen mass.
2.2. AC requires additional measurements concerning the composition of the tested specimens (mass, volume, heat, etc.).
2.3. AC requires calibrations with high accuracy, especially for temperature. AF must have a better accuracy than temperature measurement of tested specimen. The actual AC has AF defined with an accuracy of approximately 0.01 ^{0}C, so that specimen temperature can be read with an accuracy of 0.1 ^{0}C. However, thermometer must

be calibrated periodically by using a standard thermometer (SPRT = standard Pt resistance thermometer with a measuring uncertainty under 0.01 ^{0}C) with the probe immersed in an inert material (water for instance) placed in the SH. Calibration on the all temperature range specific to the studied processes, will evidence both the accuracy of AF and temperature measurement.

2.4. The creation of data banks on related specimens tested in the same standard experimental conditions (SEC) is necessary for a good retrieval of obtained results with deep structural and practical significances. For details see literature on topoenergetic working principles (www.gdfdatabanks.ro and on Google search).

2.5. Among these related specimens must be inserted so called standard or reference materials with well known AC behaviour and structure. For instance, water, γ-alumina, sapphire, etc. are classical standard materials in AC practice. However, AC users for specific transforming processes must create their own standard materials representing each class of materials studied.

3. Description of AC device

AC device for routine measurements mainly consists in two basic parts:

3.1 - the basic disposition or calorimetric vessel.
Figure 1 schematically shows an axial section in AC vessel. All AC vessels have cylindrical symmetry allowing the best AF. SH in actual AC consists in two identical truncated cone vessels one is fixed to basic disposition having all sensors and heating elements and the other one can be moved out for cleaning and replacement. In view to avoid their blocking their contact surfaces must be covered with inert grease.
3.2 – the electronic block (Figure 2) consists in AF block (T-control) and thermometer for SH.

Additionally there are the following accessories:

- connecting cables (main, T-control, T-recording at a data logger);
- accessories specific to tested specimens (conditioning tools; mixing tools, scales, etc., not included);
- retrieval accessories (data logger, PC, memory sticks, etc., not included);
- calibration accessories (SPRT, calibration block for heat capacity measurements, etc., not included);

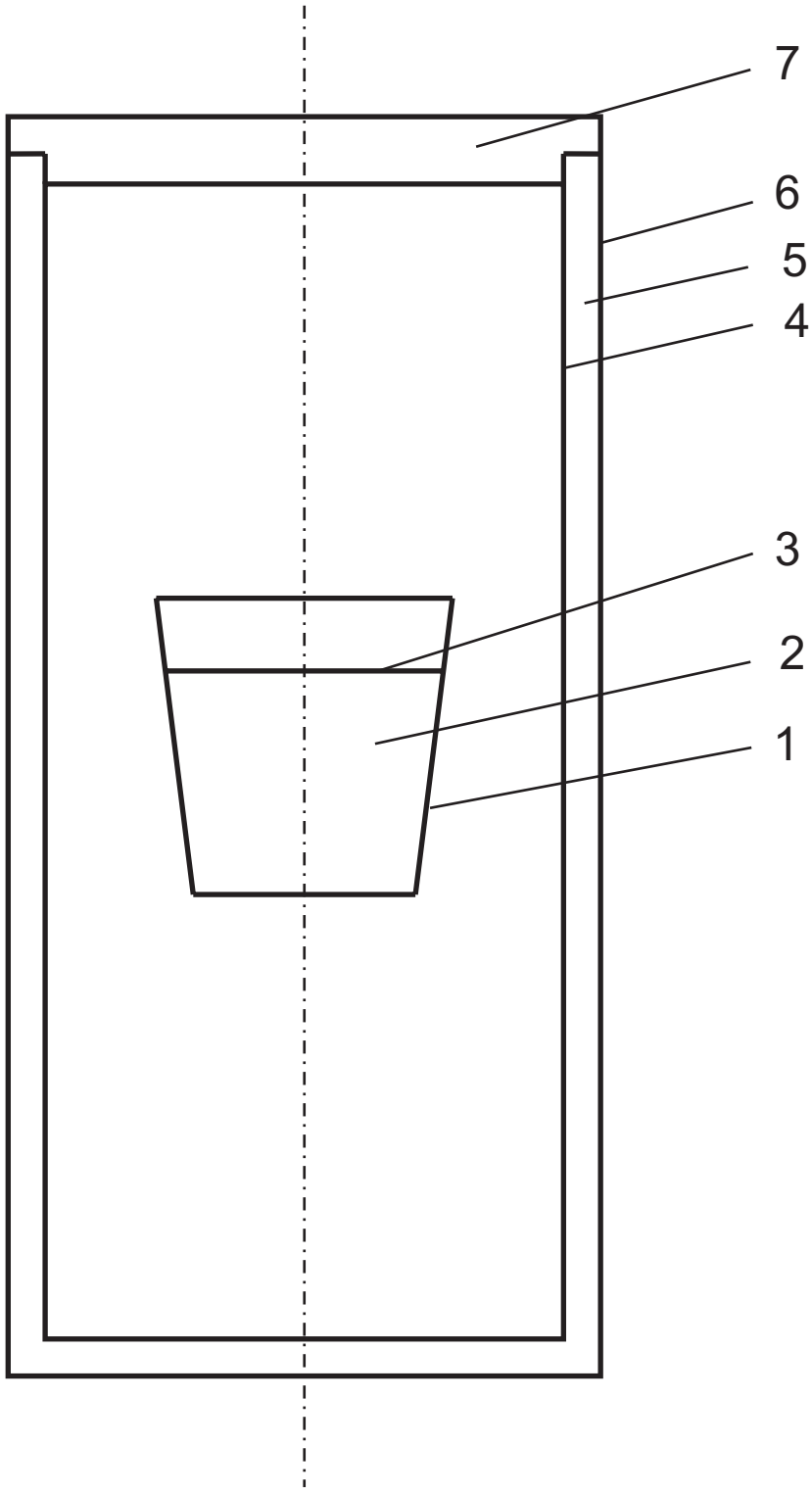


Figure 1. Axial section of the AC basic disposition.

1 - sample holder (SH);
2 - tested specimen;
3 - level defining the maximum filling degree;
4 - adiabatic shield (AS);
5 - thermal insulation;
6 - outer shield;
7 - lid.

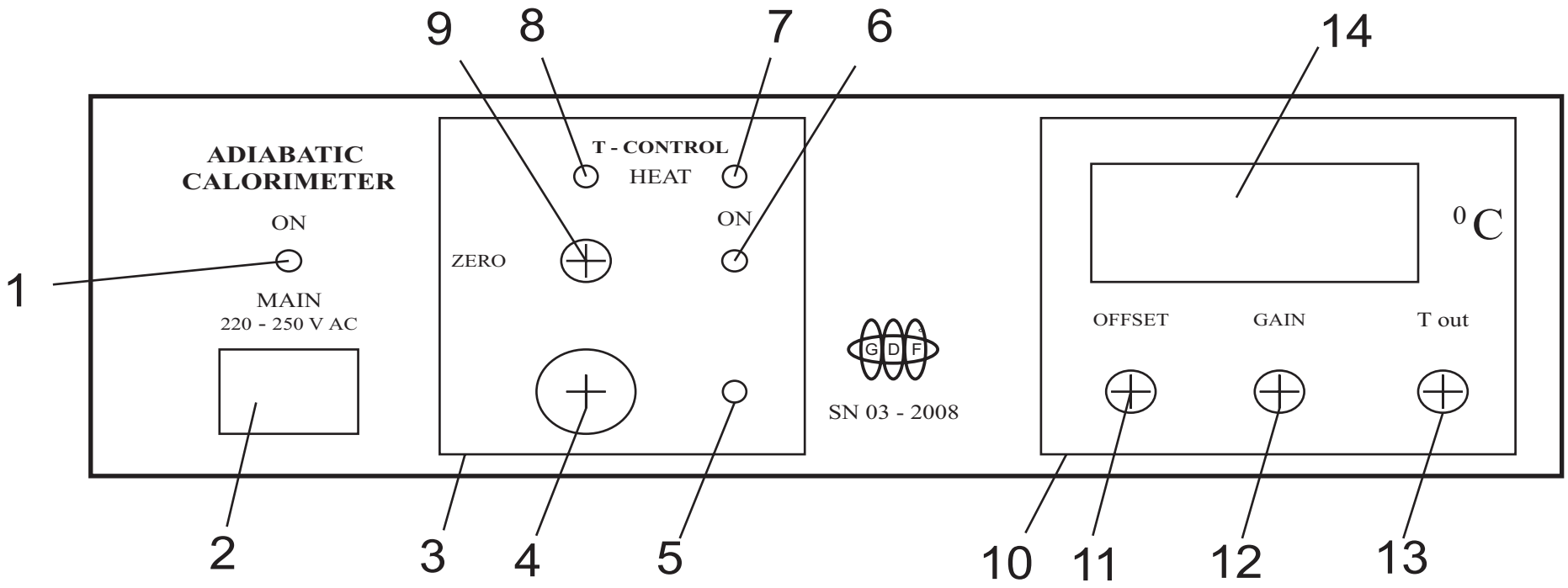


Figure 2. Schematic draw of front panel of the electronic block for adiabatic measurements

1 - main switch;
2 - main connector;
3 - panel of T-control for adiabatic function;
4 - connector for T-control;
5 - LED indicating main plugged and continuity of adiabatic heater;
6 - coupling switch for adiabatic heater;
7 - LED indicating coupling of adiabatic heating;
8 - LED indicating temperature difference between sample holder and adiabatic shield;
9 - potentiometer adjusting the electronic difference between temperature of sample holder and adiabatic shield;
10 - panel of thermometer for sample holder;
11, 12 - OFFSET and GAIN adjustments ONLY for thermometer calibration;
13 - connector for external reading/recording of temperature.

IMPORTANT: Thermometer is calibrated according to the display readings (14) which can be different from an external instrument (data logger for instance).
Make all connections and finally plug in to the main.

Details for measurements in specific applications and for calibrations will be given in the operating manual and need a special training of the users.

4. General features in obtaining and retrieval of AC experimental results

In general all mixing experiments can be reduced at 2 components. For instance for hydration of concrete mixtures these are the solid mixture and aqueous solution. The two components must be conditioned before mixing just in the AC sample holder at an initial temperature, Tin, the same for all related series of tested specimens. This condition belongs to SEC necessary in retrieval and comparison of series of related specimens.
It is important to mention the main features of SEC ensuring high repeatability of the experimental results:

- same Tin;
- same volume of the tested specimens (volume normalization);
- same operator;
- same AC device and accessories tools;
- same laboratory;

The measurement uncertainties can be established on standard materials.
According to the basic topoenergetic principle, the volume normalized specimens showing a transformation process consists in an inert component, Cin, which is not involved in the process and a transforming component, Ctr gathering all kinetic units responsible for this process (Figure 3).

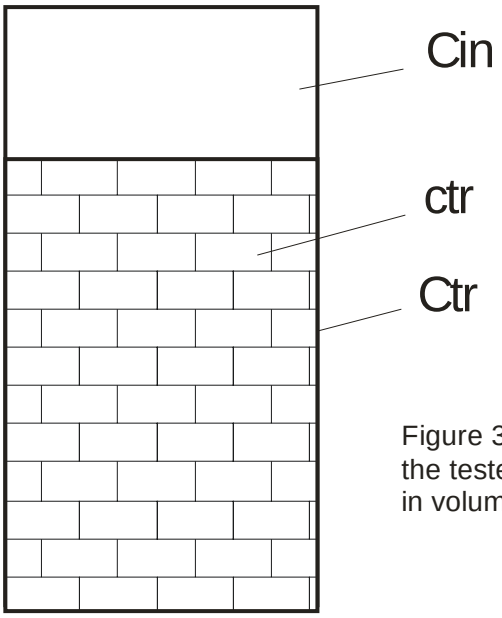


Figure 3. Schematic draw of the tested specimen normalized in volume.

Huge experimental results obtained in a wide category of measuring instruments have revealed that not all amounts of the two initial components are involved in transformation processes evidenced by these instruments.
To be more specific, this means that for hydration of cement with water (no other additives) either no all cement and/or no all water are involved in hydration process revealed by AC.

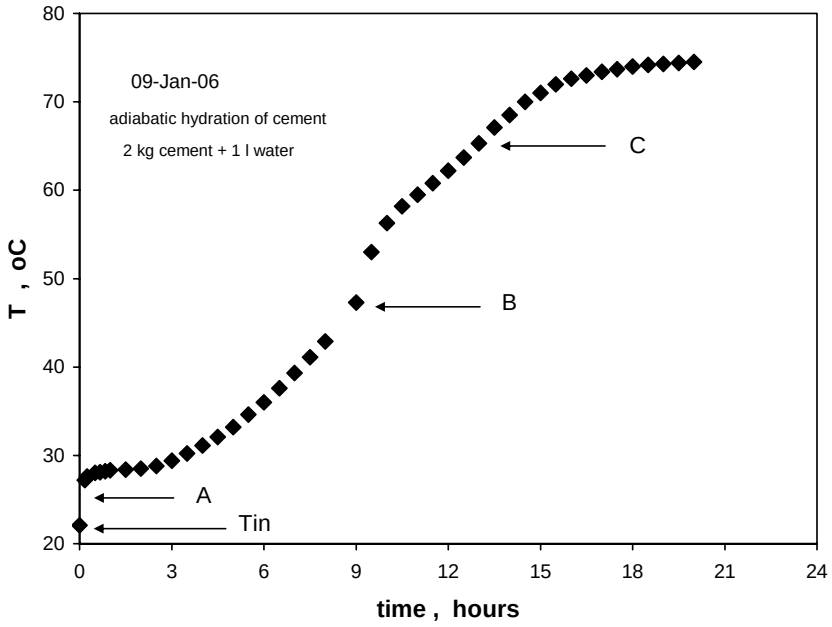


Figure 4.

Figure 4 shows the hydration of commercial grade of cement with tap water initially conditioned at room temperature of 22 ^{0}C. After manual mixing of the 2 components in AC an immediate temperature rise occurs (process A) followed by other processes (B, C, etc.) in the cascade. The temperature-time spectra of these elementary processes are characterized by specific Cin, Ctr and ctr established in the framework of data banks created.

In the same manner, the data banks created for the same specimens, but using different size of SH, will allow to establish the size at which each elementary process and/or overall process change their nature and scaling up the results for higher size impossible to be reproduced at laboratory scale.

About the author:

First name	Gheorghe
Last name	Dragan
Born	1 September 1945, Ploiesti, Prahova (Romania)
Studies	Faculty of Physics, University of Bucharest, Romania (1963-1968) Ph.D.in Physics, University of Bucharest, Romania (1980)
experience	● Head of material testing laboratory, ICECHIM, Polymer Department, Bucharest (1969-1979); ● Initiator and leader of the research project on new forms and sources of energy; ICECHIM, Center of Physical Chemistry (1979-1988); ● Head of laboratory of analytical devices and measuring instruments, AMCO-SA, Bucharest (1988-1993); ● Technical manager of GDF-DATA BANKS, Bucharest (1993-present); ● Expert metrologist, Romanian Bureau of Legal Metrology, Bucharest, Romania (1997-2000).
publications	● 90 scientific papers ● 70 scientific communications ● 17 patents ● 5 books
Addresses:	Str. Abrud 25, Bucharest 011315, Romania c/o 19 Weaver Place Minchinbury, NSW 2770, Australia (0415-674-742, 02-9625-9906) dragan_gdf@yahoo.com

Previous issues of GDF DATABANKS BULLETIN

Year	VOL	NO	Content (titles)	$*)
1997	1	1	Editorial: Databanks – the compulsory language. LOGKOW – a Databank of evaluated octanol-water partition coefficients (James Sangster). Solubility behavior introducing topoenergetic working principles. Comments on 1-octanol-water partition of several n-alkane related series.	F
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
1998	2	4	VAPORSAT-01: Databanks of thermally driven VLE. The first 100 simple molecules	AFI
1999	3	1	Editorial: New trends in material science: nanostructures (Dan Donescu) DIFFUTOR: Databanks of diffusion kinetics. VAPORSAT: Databanks of vapor-liquid separation kinetics.	F
1999	3	2	Discussions on Applied Metrology	AFI
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

continued

Year	VOL	NO	Content (titles)	$*)
2002	6	1	MOSATOR-01: Topoenergetic databanks for one component molten salts; thermally driven viscosity and electrical conductance.	AFI
2002	6	2	Editorial: HuPoTest - Operator calibration or temporal scale psychic test. MOSATOR: topoenergetic databanks of one component molten salts; thermally driven viscosity and electrical conductance.	F
2002	6	3	Editorial: Quo vadis Earth experiment? ISOCALT® : Report on metrological tests	F
2003	7	1	Editorial: Time – an instrument of the selfish thinking. 1st NOTE: Homoeopathy: upon some efficient physical tests revealing structural modifications of water and aqueous solutions. I. Mixing experiments.	F
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
2005	9	3	Universal representation of Cancer Diseases. 1. First sight on NSW-2003 report. Universal representation of Cancer Diseases. 2. UK cancer registrations on 1999-2002. Vital Potential can estimate our predisposition for cancer diseases.	F
2006	10	1	NTC – thermistors -1	AFI
2007	11	1	HuPoTest - 40 years of continuous research Basic rules for preventing and vanishing cancer diseases Climate change = change of mentality Hot nuclear fusion – a project of actual mentality	F
2007	11	2	MT – Introduction to Mental Technology HuPoTest – general procedure, assignments of results, specimen of complete test , order and obtain your complete HuPoTest report	F

Year	VOL	NO	Content (titles)	$*)
2007	11	3	TRESISTOR© - data banks of materials with thermally driven electric and magnetic properties TRESISTOR© - NTC -1 - data bank of NTC thermitors	AFI
2008	12	1	Australian population: life, death and cancer	F
2008	12	2	Pattern of Cancer Diseases	F

*) F=free, AFI=ask for invoice.

GDF DATABANKS BULLETIN, VOL. 12, NO.3, 2008
Please feel free to distribute in integral form this issue.
All correspondence at: dragan_gdf@yahoo.com

www.gdfdatabanks.ro

This Bulletin is registered at:

- Biblioteca Nationala a Romaniei, Bucharest and
- National Library of Australia, Canberra

ISSN 1453-1674