Russian Federation Ministry for Atomic Energy
Aleksandrov Research Institute of Technologies (NITI)

Phase diagrams for multicomponent systems containing corium and products of its interaction with NPP materials (CORPHAD)

Project 1950.2

WORK PLAN

Sosnovy Bor
2002

I. Summary Project Information

1. Project Title

Phase diagrams for multicomponent systems containing corium and products of its interaction with NPP materials

2. Project Manager

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3. Participating Institutions

3.1. Leading Institution

Short reference: **NITI**	
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3.2. Other Participating Institutions *None*

4. Foreign Collaborators/Partners

4.1. Collaborators

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If approved by the European Comission

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4.2. Partners None

5. Project Duration

36 months

6. Project Location and Equipment

<table>
<tr><th>Institution</th><th>Location, Facilities and Equipment</th></tr>
<tr><td>Aleksandrov Research Institute of Technologies (NITI)</td><td>Address: 188540, Sosnovy Bor, Leningrad Oblast, NITI.

Location: LSK «Radon» in Sosnovy Bor, Leningrad Region, Russia

Equipment and premises: Experimental facilities “RASPLAV-2” and “RASPLAV-3” for producing realistic molten corium and studying its interaction with NPP materials, LSK Building 12.

Data acquisition system. Devices and instruments for physico-chemical analysis (mass-spectrometer, X-ray diffractometers and spectrometers, chromatograph, etc.). LSK Building 12

Computers and office equipment, LSK Building 12, Building 11, offices 404, 405.

Location: ISC RAS, Makarov Emb. 2., Room 463, St. Petersburg.

Equipment and premises:

Equipment for synthesis, measuring solidus and liquidus temperatures in vacuum, inert atmosphere and air up to 2300°C, room 463. Devices and means for physico-chemical analysis, room 463. Computers and office equipment, room 463.

Location: SPbGETU, Professor Popov Str. 5, St. Petersburg.

Equipment and premises:

Experimental facility comprising 5 HF units for induction melting of oxide and metallic materials at different frequencies, room DS-2. Computers and office equipment, rooms DS-2 and 125A.</td></tr>
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II. Specific information

1. Introduction and Overview

The main project objective is to increase the PWR and BWR safety level in case of severe accidents entailing core degradation and melting. The specific subject of the proposed project is to experimentally determine phase diagrams for the multi-component corium systems and for products of its interaction with NPP materials.

One of the ways to safely manage the PWR or BWR severe accident is to localize the molten core inside the containment and to keep the ex-containment release of volatile and low-volatile fission products within safe levels.

Currently two severe accident management approaches are accepted for the operating and future PWR and BWR reactors.

Mostly for medium-capacity reactors: the in-vessel corium retention accompanied by the outside water cooling with boiling water.

Higher-capacity reactors are provided with an ex-vessel core catcher, where the molten core is stabilised and cooled.

The numeric modelling of interaction between the molten core and construction/structural materials of the reactor unit, concrete pit and core catcher requires adequate phase diagrams of corium and corium-based mixtures. The calculations of phase diagrams of multi-component systems use thermodynamic computer codes and corresponding databases, incorporating the experimental data.

At present, the experimental data on phase diagrams of realistic corium systems are still limited, which is explained by the following:

- Experimental studies with a high-temperature (up to 3300 K), chemically active, molten corium are very complicated, only a limited number of currently available experimental facilities can provide the required conditions.
- Newly developed PWR and BWR units employ advanced construction and structural materials, including sacrificial material used in severe accident management.
- New phenomena, specific for the in- and ex-vessel stages of a severe accident have been identified. They influence the molten pool structure and characteristics, e.g. the U and Zr extraction from the sub-oxidised corium melt by the molten steel, as it was observed on the "Rasplav-3" test facility during the test of the OECD/MASCA project. The phenomenon had been predicted by [Hofmann, 1976], [Gueneau, 1999] and [Parker, 1982].
- National norms and regulations of some countries impose certain restrictions on activities in which radioactive materials are handled.

Due to the above-mentioned reasons, the necessity for additional experimental data on phase diagrams is obvious, and their acquisition is the key objective of the CORPHAD Project.

The main experimental phase diagram studies of uranium-bearing systems will be performed on the NITI experimental facilities, where realistic reactor conditions can be provided. These experimental installations have been successfully operated during the last 14 years. In 1994 - 1997 NITI used them in the ISTC Project 064-94 "Zirconia-based core catcher"; from April 1999 till December 2000 they were employed in the ISTC Project 833-99 "Investigation of corium melt interaction with NPP reactor vessel steel". From 2001 until present time the experimental studies within OECD/MASCA project are carried out there.

The proposed work will be implemented with the participation of experts from the Research Institute of Silicate Chemistry of the Russian Academy of Sciences (ISC RAS) and St. Petersburg Electrotechnical University (SPbGETU).

The proposed project will use the expertise of highly-qualified specialists, who study severe accidents, interaction of high-temperature corium with different materials, physical chemistry and non-metallic material technologies; they have been authors and co-authors of many research papers and reference books, written on the subject of the proposed project. There are 3 professors and 13 doctors of sciences in the project team.

Most relevant publications:

1. Bechta S.V., Vitol S.A., Krushinov E.V., Granovsky V.S. et al. Water boiling on the corium melt surface under VVER severe accident conditions. // Nuclear Engineering and Design (NED). v.195 (2000) 45-56.
2. S.V. Bechta, S.A. Vitol, E.V. Krushinov et. al. Fission Product Release from Molten Pool: Ceramic Melt Tests // Proc. of SARJ meeting 1998, Nov. 4-6, 1998, Tokyo, Japan.
3. Souvorov S.A., Semin E.G., Gusarov V.V.. Phase diagrams and thermodynamics of solid oxidic melts // L.: Leningrad University, 1986, 140 p. (In Russian).

4. V.V. Gusarov. Polycrystalline oxide phase diagrams: measurements, thermodynamic optimization and applications // 9-th Int. Conf. High Temp. Mater. Chem. Abstracts Penn., U.S., 1997, P. 15.
5. S.V. Bechta, V.B. Khabensky, E.V. Krushinov et al. Corium Melt - Zirkonia Concrete Interaction: Oxide Melt Tests// Proc. OECD Workshop on Ex-Vessel Debris Coolability. Karlsruhe, Germany, 15-18 November 1999.
6. K. Froment, B. Duret, J.M. Seiler, S. Hellmann, M. Fischer, S. Bechta, D. Lopukh, A. Pechenkov and S. Vitol. Analysis of Ceramic Ablation by Oxidic Corium // Proc. OECD Workshop on Ex-Vessel Debris Coolability. Karlsruhe, Germany, 15-18 November 1999.
7. D. Lopukh, S. Bechta, A. Pechenkov et. al. New Experimental Results on the Interaction of Molten Corium with Core Catcher Material // Proc. Int. Conf. ICONE-8, April 2-6, 2000, Baltimore, MD, USA.
8. S.V. Bechta, V.B. Khabensky, S.A. Vitol et. al., Experimental Study of Oxidic Corium Interaction with Reactor Vessel Steel Samples // RASPLAV Seminar 2000, Munich, Germany, 14-15 November, 2000.
9. S.V. Bechta, V.B. Khabensky, S.A. Vitol et. al., Experimental Studies of Oxidic Molten Corium - Vessel Steel Interaction // Nuclear Engineering and Design 210 (2001) 193-224

2. Expected Results and Their Application

The project belongs to the category of applied studies. Its implementation will produce the relevant experimental data on phase diagrams of binary, ternary, quaternary and prototypic multi-component systems. The data will be used for refining numeric codes and optimising databases, including the data on suboxidized U – Zr – Fe – O systems with miscibility gaps. This will include:

- liquidus and solidus concentration curves;
- coordinates of characteristic points: eutectics, peritectics and others;
- component solubility limits in the solid phase;
- temperature-concentration regions of the miscibility gap;

Table shows the overall experimental matrix of CORPHAD tests, jointly developed by the project participants and collaborators The matrix was approved at the 1st meeting of the Project Steering Committee in St. Petersburg, May 29-30, 2002 (Minutes of the meeting are attached).

Experimental Matrix of CORPHAD # 1950.2 ISTC Project

Task	Composition	Temperature range, °C	Atmosphere	Measurement method	Priority level
1	UO_2- FeO [4),5)]	Up to 3000	Argon	IMCC VPA[1)] DTA[2)] HTM[3)] Galakhov microfurnace	High
	$UO_{2\pm X}$ - FeO_y [4),5)]	Up to 3000	Air		Middle-Low
	ZrO_2 – FeO [4)]	Up to 3000	Argon		High
	UO_2 – Cr_2O_3 [5)]	Higher than 2400	Argon		Middle
	SiO_2– Fe_2O_3	Up to 2000	Air/Oxygen		Middle
	UO_2– SiO_2	Up to 2500	Argon		Middle - Low
	(BaO,SrO) - UO_2 [4)]	Up to 3000	Argon		Low
2	Eutectic composition [4)5)] of UO_2– ZrO_2-FeO	Up to 2500	Argon	IMCC VPA, DTA, Galakhov microfurnace HTM	High
	Eutectic composition[5)] of UO_{2+x} – ZrO_2-FeO_y		Air		Middle
	U-O-Fe		Argon		High
	Zr-O-Fe		Argon		High
	U-O-Zr		Argon		High
	UO_2 – ZrO_2-SiO_2 (CaO)[7)]		Argon		Middle
3	Quaternary system Different composition in U-Zr-Fe-O system	Up to 2600	Argon	IMCC VPA, Galakhov microfurnace HTM	High
4	Corium oxidation kinetic test[6)]: C-32 (U-Zr-O)	Up to 2600	Argon replacement by air	IMCC	High
	Corium oxidation kinetic test: 90%C-32 + 10% Fe				
5	Eutectic composition measurement of a prototype complex corium mixture	Up to 2500	Argon	IMCC VPA, DTA, Galakhov microfurnace HTM	High

Notes:

1) Induction Melting in a Cold Crucible with Visual Polythermal Analysis
2) Differential Thermal Analysis
3) High Temperature Microscopy
4) Determination of the solubility limit
5) Determination of the eutectic composition and temperature
6) Decision concerning these tests will be taken upon completing the first three stages of the project
7) For tests with CaO the decision will be made using the ENTHALPY project results.

The data resulting from the project implementation will be used:

- to complement the data bank with missing experimental information on the phase diagrams of oxide and metal-oxide corium systems;
- to refine the numeric models, in particular in terms of modelling the miscibility gap and quasi-equilibrium states in thermal gradient conditions;
- to verify the numeric thermodynamic codes used for modelling phase diagrams of the multi-component systems, produced by the interaction of molten corium with construction and structural materials of the reactor, concrete pit and core catcher;
- to ensure and improve the safety of operating and future PWR and BWR reactors.

3. Meeting ISTC Goals and Objectives

During the three years of CORPHAD implementation about 40 researchers and experts will be employed, more than 25 of them were previously engaged in the projects related to the development of military technologies and equipment. In this respect the work fully concurs with ISTC tasks and objectives. The proposed project will:

- help specialists, previously engaged in the development of defence technologies, to reorient to non-military research;
- support applied studies targeted at solving society-relevant problems, in particular in the field of environmental protection, energy generation and nuclear safety;
- promote the integration of Russian scientists into the world scientific community and support the research potential of Russia;
- facilitate the transition to the market economy oriented towards demands of the society.

4. Scope of Activities

In accordance with experimental matrix presented in the table of section 2 the implementation of 5 tasks is foreseen.

All tasks are targeted at the implenetation of the overall project objective – getting experimental data on phase diagrams, they differ only by the number of components in the studied phase diagram systems, their succession is based on the principle “from simple to complex”

Task 1 To investigate binary oxide systems
Task 2 To investigate ternary systems
Task 3 To investigate the U-Zr-Fe-O system in the miscibility gap region
Task 4 To investigate the oxidation kinetics of a simple sub-oxidized system and a system with miscibility gap
Task 5 To determine the eutectic composition and temperature of the prototype multi-component system

Each task implementation is divided into stages comprising activities on test preparation and performance, experimental data analysis, pre-and post-test calculations and the analysis of the results.

Task 1

Task description and main milestones	Participating Institutions
To investigate binary oxidic systems in order to get the experimental data on phase diagrams necessary for the optimisation of databases for calculating multi-component systems and determining the solubility limit of iron, strontium and barium oxides in the solid uranium (zirconium) oxide to specify the subsolidus region of the corresponding binary diagrams (proposed by collaborators). Main stages: 1-1. Analysis of publications. 1-2. Preparation and performance of experiments, primary analysis of experimental data 1-3. Physico-chemical post-test analysis of samples 1-4. Pre- and posttest calculations using thermodynamic codes 1-5. Integrated analysis and summary of the experimental data	NITI

Description of deliverables	
1	Report on the studies of the binary oxidic systems
2	Progress report

Task 2

Task description and main milestones	Participating Institutions
To investigate ternary systems in order to get experimental data and specify the basic phase diagram of oxidic corium (U – Zr – Fe – O) and metal/oxide systems (U-Fe-O), (U-Zr-O), (Zr-Fe-O); and phase diagrams of corium mixed with components of concrete or sacrificial material UO_2 – ZrO_2 – SiO_2 (CaO). Main stages: 2-1. Analysis of publications. 2-2. Preparation and performance of experiments, primary analysis of experimental data 2-3. Physico-chemical post-test analysis of samples 2-4. Pre- and posttest calculations using thermodynamic codes 2-5. Integrated analysis and summary of the experimental data	NITI

Description of deliverables	
1	Report on the studies of ternary systems

Task 3

Task description and main milestones	Participating Institutions
To investigate the U-Zr-Fe-O system in the miscibility gap region in order to determine the temperature-concentration regions of the miscibility gap under low oxygen potential (high concentration of free zirconium) and determine the quasi-equilibrium compositions of oxidic and metallic liquids at different temperatures Main stages: 3-1. Analysis of publications. 3-2. Preparation and performance of experiments, primary analysis of experimental data 3-3. Physico-chemical post-test analysis of samples 3-4. Pre- and posttest calculations using thermodynamic codes 3-5. Integrated analysis and summary of the experimental data	NITI

Description of deliverables	
1	Report on the studies of the U-Zr-Fe-O system
2	The results of Task 3 and generalized results of Tasks 1 and 2 will be discussed in detail at the seminar with collaborators and experts.

Task 4

Task description and main milestones	Participating Institutions
To investigate the oxidation kinetics of a simple sub-oxidized system and a system with miscibility gap in order to estimate the oxidation kinetics of the sub-oxidized prototype melts of the U – Zr – O system and to identify key phenomena important for the analysis of the ex- and in-vessel corium behaviour; to develop proposals for the detailed studies Main stages: 4-1. Pretest calculations to determine the oxidant concentration above the melt surface. 4-2. Preparation and performance of experiments; primary analysis of results 4-3. Physico-chemical analysis of samples 4-4. Pre- and posttest calculations using thermodynamic codes 4-5. Integrated analysis and summary of the experimental data,	NITI

identification of key phenomena, development of proposals for their detailed study.	

Description of deliverables	
1	Progress report on the studies of prototype melt oxidation kinetics

Task 5

Task description and main milestones	Participating Institutions
To determine the eutectic composition and temperature of the prototype multi-component system in order to test numeric and experimental methods of this system forecasting. Main stages: 5-1. Development of the pretest numeric forecast of the multi-component system. 5-2. Preparation and performance of the test; measurement of the eutectic temperature. 5-3. Physico-chemical posttest analysis and determination of the eutectic composition. 5-4. Pre- and post-test calculations using thermodynamic codes. 5-5. Integrated analysis and summary of the experimental data. 5-6. Comparison of the calculated and experimental data, evaluation of the forecast accuracy.	NITI

Description of deliverables	
1	Report on the studies of multi-component systems
2	Minutes of the meeting with collaborators

Note: In accordance with the decision taken at the 1st meeting the collaborators will participate at stage #4 (Pre- and posttest calculations using thermodynamic codes) and #5 (Integrated analysis and summary of the experimental data) in the implementation of all stages.

The compositions will be specified and decisions about the test on the suboxidized melt oxidation kinetics (Task 4) (or its adequate replacement) taken after completing the previous tasks and the analysis of available data, including those produced in the currently implemented MASCA, ENTHALPY and COLOSS projects.

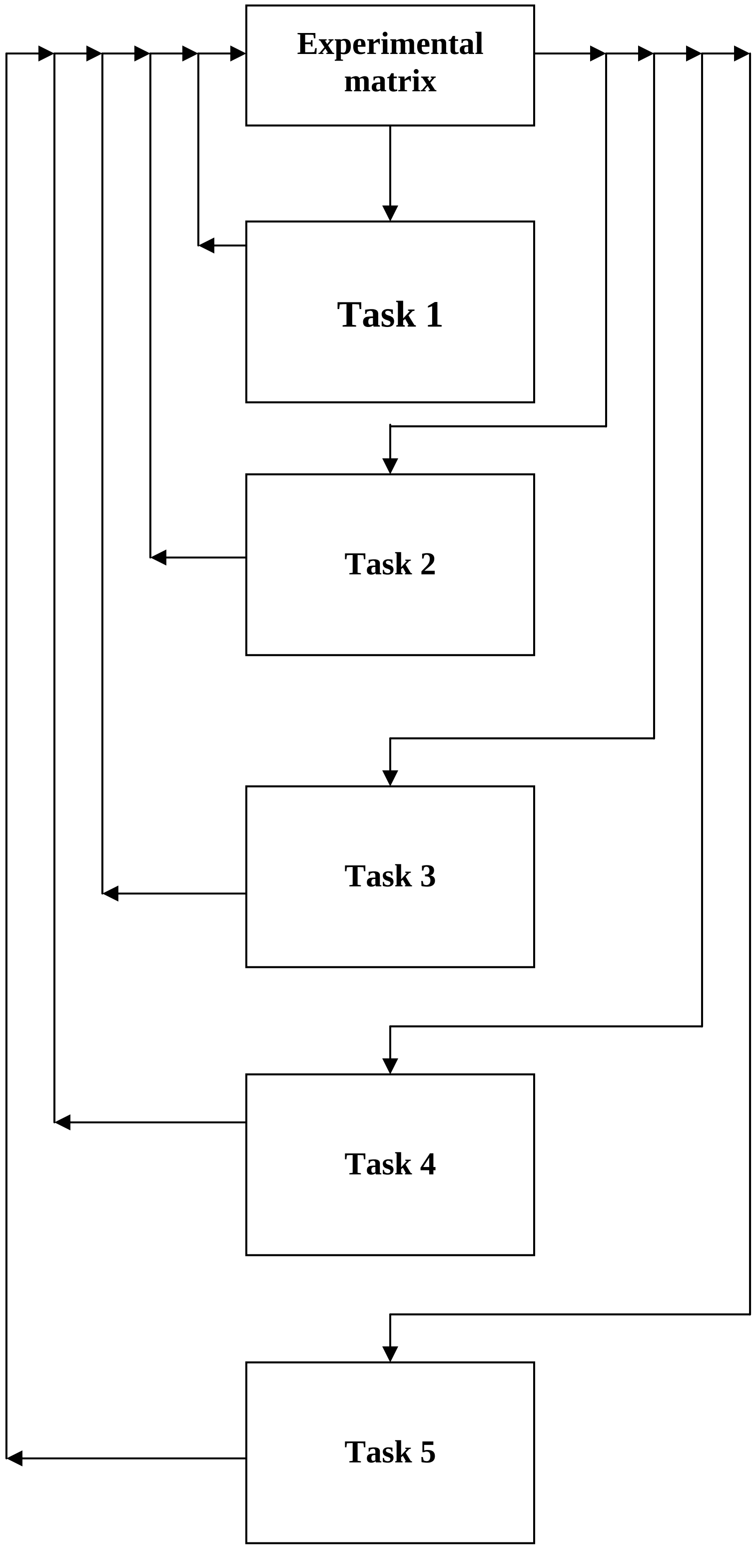
Each experiment will be preceded and followed by the pre- and posttest calculations using codes MULTICOM and GEMINI.

The total number of tests (experimental points in phase diagrams) will be ~ 50.

Each quarter is completed by writing the progress report. The completion of each task and each year of work is formalized by the analytical report on the period. The whole work is presented in the Final Report, which is finalised after the review and comments on its draft by the collaborators and ISTC.

The diagram of the task interrelation is shown in the figure below. The experimental matrix will be updataed after the completion of each test series and analysis of the results.

Correlation of Tasks 1-5



5. Role of Foreign Collaborators/Partners

The following activities performed jointly with foreign collaborators are foreseen in the framework of the proposed project:

- Joint development and update of the experimental matrix.
- Discussion and decision about the concentration regions for phase diagram studies, experimental methodologies and methods for the physico-chemistry analyses, the experimental matrix updates.
- Joint pre- and posttest calculations based on the thermodynamic codes MULTICOM (project participants) and GEMINI (collaborators), discussion of the resulting calculations.
- Continuous information exchange in the course of the project implementation.
- Discussion of reports (quarterly, annual, final, and task reports).
- Information exchange about the international projects on related subjects (MASCA, COLOSS and others) to be used in the CORPHAD data analysis.
- PSC meetings to be held not less than once a year.
- Joint papers, publications and presentations at conferences.

6. Technical approach and Methodology

In CORPHAD studies the “Rasplav-2” and “Rasplav-3” experimental facilities will be used. They were built for experiments with prototype molten corium having temperatures up to 3300 K, for investigating melt interaction with structural materials and properties of interaction products. “Rasplav-2” is designed for tests with oxidized and suboxidized systems; “Rasplav-3” – for oxidized and suboxidized metal-oxide systems. To produce molten corium the methodology of induction melting in cold crucible (IMCC) is used. The method is quite suitable for the phase diagram studies, because the presence of solid phase (crust) between the melt and cold crucible prevents the crucible-melt mass transfer, ensures the melt retention and its high purity (close to that of initial components). The IMCC method ensures contact-free power input into the melt and volumetric power in it. Currently “Rasplav-2” and “Rasplav-3” can produce up to 8 and 2 kg high-temperature melt respectively, in the inert, air and steam atmosphere.

Some experimental studies will be performed at the facilities of the Institute of Silicate Chemistry of the Russian Academy of Sciences (ISC RAS) and St. Petersburg State Electrotechnical University (SPbGETU). The facilities include: Galakhov microfurnaces, high-temperature microscope, derivatographs, high-temperature differential thermoanalyzer. This equipment enables phase diagram studies up to the temperatures of 2800 K and getting reliable and accurate experimental data.

The following methods are going to be used for the phase diagram studies:

- Visual polythermal analysis (VPA)
- Visual polythermal analysis in the cold crucible (CCVPA)
- Differential thermal analysis (DTA+TGA)
- Galakhov microfurnace (GM)
- High-temperature microscopy (HTM)

These methods have proven their reliability in projects ISTC-64, ISTC-833, CIRMAT, CIT, ENTHALPY, OECD/MASCA, ECOSTAR.

The following methods are proposed for the physico-chemical analysis:

1. Elemental composition analysis
    - X-ray fluorescence (XRF)
    - Chemical analysis
    - Mass-spectrometry with inductively-coupled plasma (ICP MS)
    - Spark source mass-spectrometry (SS MS)
2. Analysis of phase composition
    - X-ray structure analysis (XRS)

- X-ray phase analysis

3. Metallo- and ceramography
   - Optical microscopy
   - Scanning electron microscopy and microprobe analysis (SEM/EDX)

These methods have been tested in international projects, such as ISTC-64, ISTC-833, CIT, ENTHALPY, OECD/MASCA.

Both “Rasplav-2” and “Rasplav-3” test facilities have the data acquisition systems for measuring, recording and primary processing of data on:

- electrical characteristics of the induction furnace;
- coolant temperatures and flow-rates;
- molten corium temperature;
- power distribution in the melt and **crucible components;
- video-recording and display of the molten pool surface behaviour.

CORPHAD 1950-01 Project Development Grant was used for the partial modernization of equipment, software and laboratory devices. The basic methodologies for the physico-chemical studies of uranium-bearing corium have been developed. During the project implementation the modernization will be continued, which will enable to increase the range of studied parameters.

The original thermodynamic numeric code MULTICOM is used to optimise the experimental procedures, to evaluate the concentration range of characteristic regions and specific phase diagram points and to check the accuracy of numeric forecasts. Code MULTICOM is used for calculating binary and multi-component systems with a possibility of modeling the miscibility gap. MULTICOM can also be used for forecasting the quasi-equilibrium states in thermal gradient systems. This code has been verified using the experimental data of OECD/MASCA and ECOSTAR projects.

6. Technical Schedule

	Quarter 1	Quarter 2	Quarter 3	Quarter 4	Quarter 5	Quarter 6	Quarter 7	Quarter 8	Quarter 9	Quarter 10	Quarter 11	Quarter 12	Person*days
Task 1				Report	Meeting with collaborators	Progress report and preparation of publication							
Person*days	**1150**	**1100**	**1100**	**1100**	**600**	**400**							***5450***
Task 2								Report	Meeting with collaborators				
Person*days					**500**	**600**	**1100**	**500**	**100**				***2800***
Task 3											Progress report Meeting with collaborators		
Person*days								**500**	**900**	**600**	**500**		***2500***
Task 4												Progress report. Presentation at the conference	
Person*days										**500**	**500**	**450**	***1450***
Task 5												Report Meeting with collaborators	
Person*days											**100**	**509**	***609***
TOTAL	**1150**	**1100**	**1100**	**1100**	**1100**	**1000**	**1100**	**1000**	**1000**	**1100**	**1100**	**959**	***12809***

8.2. Managerial responsibilities