

The Luginin thermochemical laboratory of the Moscow state university

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Abstract - The article has been dedicated to centenary of the thermochemical laboratory of the Moscow State University.

INTRODUCTION

The thermochemical laboratory was founded by V.F. Luginin in 1891 after his return to Russia from abroad. Having collected comprehensive precision devices, Luginin created the unique possibilities for study of thermochemical properties. The list of the Luginin papers (ref.1) shows that the diverse thermochemical quantities have been measured in the laboratory: heats of solution and vaporization, reaction heats, heat capacity etc. At that time the main attention was focussed on a determination of heats of combustion in oxygen.

The laboratory experience was summarized by Luginin in the book "Description of Various Methods of Determination of Combustion Heats of Organic Compounds" which before long, was published also in German (ref. 2). That book was an excellent guide on experimental procedures used in thermochemistry. The tables available in the book represent the heats of combustion and formation of many organic compounds.

The results obtained in the laboratory made it possible to establish some simplest thermochemical relationships. So, in the short course on thermochemistry, Luginin wrote about a constancy of $-CH_2$ -increment in the heats of combustion of alkanes, alkenes, 1-alcohols, alkyl halides, etc. The increments for substitution of hydrogen with bromine, iodine, carbonyl- and amino- groups were also determined (ref. 3).

The first Luginin's assistants - A.N.Shchukarev and P.V.Zubov continued the thermochemical measurements in the laboratory for a long period (ref. 4). Zubov alone measured combustion heats of more than 120 organic compounds: hydrocarbons, alcohols, ketones, ethers, cyclic compounds, etc.

Further on, a development of the thermal laboratory was closely related to the names of N.A. Umov, I.A. Kablukov and V.V. Sventoslavsky. N.A. Umov was at the head of the laboratory for 4 years up to 1915. After the death of N.A. Umov, I.A. Kablukov became a head of the laboratory. His first thermochemical determinations were carried out together with Luginin: they measured the heat of addition of bromine to unsaturated organic compounds (ref. 5). Later on, he studied heat capacity of alloys, measured combustion heats of halogenorganic compounds. I.A. Kablukov wrote manual "Thermochemistry" and its two editions appeared in 1931 and 1934 (ref.5).

The activity of V.V. Sventoslavsky in the laboratory lasted for 8 years (in 1918, V.V. Sventoslavsky, Pole in nationality, was repatriated to

Poland). Here he carried out a main part of calorimetric measurements for his Doctor's thesis "Thermochemical Investigations of Diazo Compounds". The results of these investigations were published as a book in 1917 (ref. 6).

In 1914, Sventoslavsky and Popov proposed a benzoic acid as a bomb calorimetric standard (Ref.7). This proposal was officially made by V.V. Sventoslavsky at the 1st International Chemistry Conference (IUPAC) (Ref. 8) and accepted at the 2nd International Conference (IUPAC) in 1921 (ref. 9). This work became an important landmark in development of the thermochemistry of organic compounds. Although at the present time no principal problems arise at electrical calibration of bomb calorimeters, the standard benzoic acid is used for calibration more often. Such a method has certain advantages over the electrical one, due to the fact that calibration and sample runs are performed under similar conditions, and this decreases a level of the unexcluded systematic error. At the same time an accuracy of determination of energy of combustion of benzoic acid is sufficiently high.

The work made together with V.V. Sventoslavsky predetermined the main direction of scientific activity of M.M. Popov whose whole life was devoted to the thermochemical laboratory. For years he was a deputy of Kablukov and in 1934 became a head of the laboratory. The first years after the revolution and civil war were difficult for the Moscow University. Popov not only maintained the Luginin laboratory but made great efforts for its provision with precise equipment.

A sphere of his scientific interests was extremely wide. Remaining within the framework of calorimetry as main experimental method, he accomplished various work in the thermochemical laboratory. Popov determined heats of combustion in oxygen and heats of solution of many substances, measured latent strain energy in the alloys, accomplished some work for the thermotechnical institute, Kashirskaya power station and the others. A series of the Popov's works was devoted to study of the mixed crystals. Popov and his collaborators determined the heat capacities and heats of formation of many mixed crystals (e.g. formed with halides of alkali metals) and also a rate of their decomposition and crystalline structure (ref. 10,11).

After the Second World war Popov was actively engaged in studying of the thermodynamic properties of actinides. The heats of formation and heat capacities of oxides and halides of uranium, plutonium (ref. 12-14), mean energy of tritium decay (ref. 15) etc. were determined under his supervision. The calorimeters for determination of a content of α - and β -radioactive substances were also developed.

M.M. Popov was a talented experimenter. He developed, together with P.K. Shirokich, the first moving-bomb calorimeter in which the agitation of the bomb contents was accomplished by rocking of the bomb (ref. 16). The calorimeter was employed for measurement of combustion heats of chloro- and bromorganic compounds. Now the calorimeters with moving (usually - rotating) bombs are widely used when it is necessary to accelerate the processes in the bomb (e.g. reactions of reduction or oxidation).

M.M. Popov and his co-workers developed calorimeters for measurement of heat capacity at high (ref. 17) and low (ref. 18,19) temperatures, calorimeters for determination of heats of solution (ref. 11), aneroid calorimeter and some other instruments. Popov's monograph "Thermometry and Calorimetry" (two editions) (ref. 20) played a significant role in thermochemical education.

A vivid page in the laboratory history was a period when S.M. Skuratov was a head of the laboratory (1956 to 1968). The man of tireless energy, talented organizer of the scientific work he put a lot of effort into providing the laboratory with a new-type equipment.

The removal to the new building on the Lenin hills (1953) was accompanied by complete re-equipment of the laboratory, replacement of the obsolete instruments, changing in technique. After placing in the new building, a new period started in the laboratory life. The tendencies formed in the middle of the fifties developed undergoing the required modification further on, when G.L. Galchenko was responsible for the Laboratory (1968-1988).

The main results obtained in the thermochemical laboratory during that period in basic topics have been reviewed below.

TERMOCHEMISTRY OF INORGANIC COMPOUNDS

The main purpose of the thermochemical study of inorganic compounds started at fifties was a precise determination of $\Delta_f H^\circ$ values of the most important compounds, so called "key" values. The non-stoichiometric solid phases, solutions and other objects were also studied. During these investigations the use was made of different calorimetric methods: determination of heats of combustion of the substances in oxygen in the calorimetric bomb, determination of heats of reaction at high temperatures with preliminary heating of the substances, determination of heats of solution in water and heats of reactions with acids, alkalies etc.

The heats of reaction at high temperatures

The enthalpy of formation of boron oxide has been determined by three independent methods (ref.21). For these measurements bomb calorimetry was applied, the essential feature of experiments was preliminary heating of boron in the special microfurnace up to 400° to 550°C at oxidation and chlorination and up to 1000° to 1300°C at nitration. The most important task was a precise measurement of energy introduced with electric heater; for this purpose, for measurements at temperatures from 1000° to 1300°C an integrating device was developed which was calibrated calorimetrically (ref. 22). Later on, using the same method of heating of reaction components in the bomb G.L. Galchenko with collaborators determined the enthalpy of decomposition of decaborane, and by the method of high-temperature chlorination, the enthalpies of formation of boron carbide, boron phosphide, and also of chlorides of zirconium, hafnium, and tantalum.

In the middle of the seventies the instrument was produced for introduction of a stable electric power to the heater; the instrument made it possible to increase considerably the precision of the energy measurement (ref. 23,24). For the last years this method was employed for determination of enthalpies of formation of SnO , SnO_2 , NbCl_5 , FeCl_3 , NiCl_2 , VCl_2 , CoCl_2 , V_2O_5 etc.

Combustion of inorganic substances in oxygen

In many cases the determination of enthalpies of combustion and formation of the inorganic compounds was accomplished using the traditional bomb calorimetry, without preliminary heating of the substances. Thus the thermochemical investigation of oxide and carbide phases of transition metals: zirconium, hafnium, niobium and tantalum was completed. As a preliminary stage of this study the key values - enthalpies of formation of higher oxides of zirconium, hafnium, niobium and tantalum have been determined (ref. 25).

The thermochemistry of solutions

The measurements of the heats of solution and heats of reaction were resumed in the laboratory in the end of fifties in order to use these data for the determination of the key $\Delta_f H^\circ$ values of crystalline (and sometimes gaseous) inorganic compounds. Afterwards these measurements prog-

ressed into fundamental research of the electrolyte solutions (ref. 26). Up to the present, the enthalpies of formation of about 20 ions (Rb^+ , Cs^+ , Mg^{+2} , Ba^{+2} , Sr^{+2} , Nd^{+3} , Gd^{+3} , Dy^{+3} , Er^{+3} , Lu^{+3} and the others) were determined. Using the $\Delta_f H^\circ$ values of ions together with the enthalpies of reaction, solution, and dilution measured in the laboratory the enthalpies of formation of many halides and oxides (MgCl_2 , MgI_2 , MgO , CaCl_2 , NdCl_3 , GdCl_3 , DyCl_3 , Nd_2O_3 and the others) have been obtained.

In the seventies, the start was made for the determination of enthalpies of solution of electrolytes (halides, perchlorates, chlorates of alkali and alkaline earth metals, lanthanide chlorides) in the heavy water, nonaqueous and mixed solvents: dimethylsulphoxide, formamide, dimethylformamide, propylenecarbonate, sulpholane and also water-organic mixtures (ref. 27,28). In these solvents, a dependence of enthalpy of solution on composition has been studied in ca. 50 systems.

The progress of solution thermochemistry called for a development of new calorimeters. For measurement of enthalpies of reactions accompanied by release of gaseous products, a hermetically sealed adiabatic calorimeter was produced (ref. 29). Later on, the hermetically sealed rocking calorimeters were made (ref. 30,31), and also the calorimeter with a magnetic stirrer (ref. 32).

THERMOCHEMISTRY OF ORGANIC COMPOUNDS

Heats of combustion of hydrocarbons and related compounds

The measurements of combustion heats were resumed in the laboratory in the fifties. At that time S.M. Skuratov designed a new self-sealing stainless steel calorimetric bomb; a new model of high-sensitive mercury thermometer was also developed (ref. 33,34). The new equipment made it possible to increase sufficiently the precision of the measurement and to study very small energetic effects. Later on, this technique was modified, e.g. the resistance thermometers and recording potentiometers were used.

In the middle of the fifties the energy of combustion of lactams, aliphatic aminoacids, cyclic formals, branched alkanes has been determined. Afterwards, priority was given to the investigation of cyclic compounds. M.P. Kozina et al determined the standard enthalpies of combustion and formation of ca. 100 cyclic compounds; this amount included monocyclic hydrocarbons and their derivatives, bicyclic and polycyclic hydrocarbons, aromatic compounds, oxygen- and nitrogen-containing cycles etc. The determination of $\Delta_c H^\circ$ and $\Delta_f H^\circ$ values of great number of organic compounds has been described in (ref. 35).

Polymerization reactions

To study reactions of polymerization, S.M. Skuratov et al. designed in 1950 a double calorimeter which made it possible to study also a kinetics of reaction (ref. 36). The polymerization of lactams was thoroughly studied by this method. It was shown, for example, that enthalpy of this reaction increases from α -pyrrolidone to η -capryllactam and then decreases gradually to ω -lauriclactam (ref. 37).

Since 1974, an investigation of thermodynamics and kinetics of synthesis of thermostable polymers was started. E.g. the reactions of polycyclotrimerization of cyanic ethers, copolymerization of cyanic and isocyanic ethers with carborane-containing epoxy oligomers and others were studied (ref. 38,39).

Halogenorganic compounds

Two methods of thermochemical study were used in the end of fifties: measurement of the enthalpy of reaction of gaseous compounds with alkali

metals in a static bomb (ref. 40) and measurement of the enthalpy of combustion in the rotating-bomb calorimeters (ref. 41,42). Later on, the bomb combustion in oxygen became the major method used in the laboratory. This method was successfully employed for investigation of such compounds as gaseous and volatile freons (e.g. $\text{CF}_2\text{ClCF}_2\text{Cl}$, $\text{CF}_2\text{ClCFCl}_2$), substances containing fluorine, chlorine and bromine (CF_3CHClBr , CF_2BrCFCl) perfluorinated compounds, etc. (ref. 43). A method of combustion of liquefied gases in the calorimetric bomb has been proposed that provided clean and complete combustion of the substances which do not burn completely in the gaseous state (ref. 44).

Boronorganic compounds

The systematic determinations of enthalpies of combustion of boronorganic compounds were started by G.L. Galchenko et al. in the sixties and continued until the last years. The enthalpies of combustion of boronalkyls, oxygen- and nitrogen-containing compounds, and others have been measured. An essential part of this study was a determination of enthalpies of combustion and formation of carboranes and their derivatives. The enthalpies of combustion of boronorganic compounds were determined in the static bomb calorimeters. It is important to mention that after combustion of boronorganic compounds a sooty residue containing unburned boron is often revealed. Therefore, especial attention was given to analysis of combustion products and to refining on the combustion procedure. In doing so, high degree of combustion, close to 100% could be obtained (ref. 45,46).

Enzymatic reactions

In 1976, G.L. Galchenko and M.V. Rekharsky started a thermochemical investigation of enzymatic reactions. The red-ox, hydrolytic, and other reactions proceeding under action of enzymes have been studied. For enthalpy measurements LKB-2107 and LKB-2277 microcalorimeters were used. An important value in the thermochemistry of enzymatic red-ox reactions - enthalpy of reduction of nicotinamide-adeninedinucleotide, was determined by different methods with the use of soluble and immobilized enzymes (ref. 47). Since 1982 the main efforts have been focussed on a study of hydrolytic enzymatic reactions, e.g. reactions of hydrolysis of esters, amides, peptides and so on (ref. 48).

Thermodynamics of vaporization

In 1970 R.M. Varushchenko et al. developed an original ebulliometer for a determination of a saturated vapor pressure (ref. 49). In 1977, a precise calorimeter was produced for determination of enthalpies of vaporization of organic substances (ref.50). Using these apparatuses Varushchenko et al. studied a dependence of the saturated vapor pressure on a temperature within a range from 2 to 102 kPa for ca. 90 organic substances, and measured the enthalpies of vaporization of some organic substances by calorimetric method. The critical parameters and so called "similarity criteria", used for estimation of thermal properties in a wide range of temperatures also could be obtained (ref. 51).

HEAT CAPACITY DETERMINATION

High-temperature heat capacity

In the late forties, M.M. Popov and G.L. Galchenko constructed a calorimeter for heat capacity measurement of powdery substances over the range 100 to 700°C (ref. 17). In this calorimeter, a method of continuous introduction of energy (scanning method, shortly before proposed by Sykes for metal samples) was realized. This method was successfully employed for heat capacity measurement of substances with a low heat conductivity: several salts (ref. 17) and also oxides and halogenides of uranium (ref.14,52). At the same time S.M. Skuratov and S.A. Lapushkin constructed a copper-block drop calorimeter with isothermal jacket for enthalpy measurement up to 900°C (ref. 53).

Low-temperature heat capacity

In the early fifties, the heat capacity measurements at low temperatures were resumed, firstly over the range 60 to 300 K (ref. 19). Some years later, the calorimeters were produced which allowed to start the measurements of heat capacity at hydrogen (ca. 12 K) or helium temperatures (ref. 54,55).

The obtained entropy values together with the reaction enthalpies were employed for calculation of the Gibbs energy. So, in the early sixties, the heat capacities of unsubstituted lactams and some polymers were measured and lactam's ability for polymerization reaction was studied (ref. 56). Further the $\Delta_f G^\circ$ values of some derivatives of bicyclo-(2, 2, 1)-heptane (ref. 57), fluorochloroderivatives of ethane and propane (ref. 58,59), fluorinated amines, cyclic perfluorocarbons (ref. 60) etc. were obtained. The "key" thermodynamic functions of some inorganic compounds, e.g. SnO_2 , Ta_2O_5 were also determined.

The study of transition phenomena have been an important part of the low-temperature calorimetric. The solid-phase transitions in organic compounds were usually interpreted in terms of the "order-disorder" concept. The onset of disorder was associated with diverse motion types, viz. either motion of the molecule as a whole (rotation in lattice sites, which is typical of plastic crystals) (ref. 57) or the motion of specific groups in the molecule (internal rotation) (ref. 59). The i.r. and Raman spectra of solid phases in conjunction with entropies of transition made it possible to disclose the nature of phenomena (ref. 59,61). G-transitions of plastic crystals were also often detected. A rare phenomenon - the gradual development of conformer transition was detected in the solid phase of 1,1,2,2-tetrafluoro-1,2-dibromoethane (ref. 59).

CONCLUSION

It is evident from the brief survey given above that the scientific activity in the laboratory was usually based on the precise calorimetric measurements. Therefore, a special attention was given to the technique and procedure of experiments. This was reflected in the numerous publications, a part of which has been cited above and also in the Popov's monograph "Thermometry and Calorimetry" (ref. 20), book "Thermochemistry" (ref. 29) etc.

Being a part of chemistry department, the laboratory was deeply engaged in thermochemical education from the very beginning. A systematic education of students was started by V.F. Luginin. Later on, up to the present, a special course of thermochemistry was given for students and post-graduates of the chemistry department. The basis for teaching was formed with the monographs mentioned above (ref. 2,20), textbooks (ref. 3,5,29), and numerous booklets of exercises.

Since 1988 L.N. Sidorov is responsible for the laboratory; after his appointment a new tendency appeared - an investigation of molecular equilibria by a method of high-temperature mass spectrometry. A development of this tendency is a subject of other lectures.

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