Special Review

NOVEL MULTIPLE METHODS AND RESULTS IN THERMAL ANALYSIS

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The development of the multiple thermal methods and the results obtained with them during the past three years are discussed.

More than 900 papers dealing with thermal analysis have been published in the past three years (1973–1975). This corresponds to a 150% increase as compared to the preceding three years. The literature sources on which this review is based, as well as numerical data are listed in Table 1. In view of the great number of papers, the author could not undertake to survey the complete material. The object of the present review is the discussion of instruments for multiple thermal techniques and of the results obtained with these techniques.

Development of instruments

The Hungarian representative of instruments for multiple techniques is the derivatograph manufactured by MOM, Budapest. F. Paulik and J. Paulik have lately further developed the method towards quasi-isothermal and quasi-isobaric measuring techniques [1]. Thereby the investigation of decomposition processes close to the equilibrium temperature and pressure (in the physicochemical sense) became feasible. Thermal results obtained with the novel procedure have been reported by the inventors in several papers [2–4].

Further development consists in combining the quasi method and gas titrimetry. In this manner, quasi-thermogravimetric curves (QTG) and quasi-thermogastitrimetric curves (QTGT) are simultaneously obtained [5].

Polish researchers [6] made use of the derivatograph supplied with a 1500° furnace for determining the Curie point of ferromagnetic substances. The magnetic field produced by the heating current establishes a magnetic relation with the ferromagnetic substance. At the Curie point, where the substance turns paramagnetic, a sharp apparent weight decrease is measured.

The efforts to combine the light microscope and the DTA method appear of interest. The DLI (depolarized light intensity) technique simultaneously records the DTA curve and measures changes in light intensity under dynamic conditions [7]. The authors claim particular importance of the method in the plastics

industry for studies of crystallization, rates of crystallization and melting points of polymers.

American researchers report about combinations of high-pressure TG and magnetic susceptibility measurements in the 1–68 atm pressure range. The method has been successfully applied in investigations of coordination compounds where a change occurs in the number of the odd electrons of the central metal atom [8].

There is an increasing demand for the analysis of volatiles evolved during thermal analysis. This task is solved in an up-to-date manner by combining thermal analysis and mass spectrometry. Various such combinations are known. American researchers report results obtained by combining TG-DTA-MS-computer equipment, particularly for geochemical applications [9].

DTA-MS-magnetic tape data storage [10] has been used in studies of inorganic compounds, coordination compounds, organic metal compounds.

The combination TG-DTG-DTA-MS [11] has been used to study the dehydratation of $MgCl_2.6H_2O$.

The decomposition of $UO_4.2NH_3.2HF$ has been investigated with the combination TG-DTA-MS to clarify the reaction mechanism [12].

Simultaneous DTA-EC (electrical conductivity) measurement has been reported [13].

DTA-EC-EGA (evolved gas analysis) has been used by Soviet authors for the analysis of 21 sulphide-containing compounds [14].

The essence of DTA-TG-ETA (emanation analysis) is that some radioactive isotope, e.g. krypton, is previously built into the crystal lattice of the substance to be studied. The radioactive isotope evolved from the surface under the effect of heat is measured with a GM tube and yields information regarding crystal structure, crystal rearrangement etc. [15].

A combination of X-ray-TG-MS [16] is known. The sample is placed into a heatable X-ray cell and the diffraction pattern is taken in the usual manner. The evolved decomposition products are then transferred through a special vacuum-proof valve to the thermobalance which is under high vacuum. The so-called "molecular beam" formed of the decomposition products strikes against the balance pans and displaces the balance. The displacement is proportional to the rate of decomposition, consequently its integral yields the TG curve. MS-curves can be obtained simultaneously.

Belgian researchers [17] studied the decomposition of Ca, $Mg(HCO_3)_2$ using the combination DTA-EGA. Others [18] used a similar combination for investigations of the decomposition of sodium and calcium dithionate-hydrates.

Theoretical research

In this field, numerous papers deal with the kinetic interpretation of solid-phase reactions, since apparent kinetic parameters, namely activation energy, order of reaction, activation enthropy and frequency factor can be calculated from

thermal curves, and used for characterizing the reactions. Part of these papers deal with theoretical considerations [19, 21], mathematical calculation methods [22–24], computer programs [25], studies of the effect of experimental conditions [26]. Other papers report investigations concerning the decomposition kinetics of coordination compounds, cadmium dipyridyl bromide [27], glucose, fructose [28], malonic acid [29], polymers containing polyacetylene groups [30], polyethylene and polypropylene [31], poly(pyromellite-imide) and its derivatives [32], sodium and potassium polymethacrylate [33], isobutene-butadiene and styrene-indene copolymers [34], Cr_2O_3/Al_2O_3 catalyst [35], ZnO/Al_2O_3 [36], glycine, DL-valine, DL-alanine, L-phenylalanine [37], cadmium thiourea reineckates [38], p-toluene sulpnonamide, chloroamine-T, dichloroamine-T [39]. Hungarian authors have studied the evaporation kinetics of water, benzene, toluene, *n*-heptane and cyclohexane in the presence of non-volatile *n*-paraffins [40].

Although most theoretical papers dealing with kinetics point out the theoretical and experimental difficulties arising in the computation of solid-phase kinetic parameters, only few authors have come to the final conclusion that the characteristics adopted from the kinetics of homogeneous reactions can only formally be applied under the conditions of heterogeneous reactions [41].

Inorganic compounds

Fundamental and structure research

The decomposition of $Mn(CO)_{10}$ and $Re_2(CO)_{10}$ has been studied [42]. The result indicated that the dissociation energy of the metal–metal bond is lower in the case of the Mn salt. A study of Pd, Rh, Re chlorides and oxides allowed to elucidate the structure of the intermediate products formed in the thermal reaction [43]. The thermal analysis of AgF and of a mixture of AgF with alkali halogenides demonstrated that the presence of the alkali metal compounds substantially affects the decomposition of silver fluoride [44]. Potassium chlorite undergoes a disproportioning reaction into chlorate and chloride, without evolving oxygen. The reaction is indicated by an exothermic maximum at 140° on the DTA curve [45]. Thermal analysis of periodic acid, iodic acid, iodic anhydride, iodine pentoxide has been reported [46]. The conditions of spinel formation with the compositions $2ZnO.3Al_2O_3$ [47] and $MgFe_2O_4$ [48] and the decomposition of $ZnCrO_4.2H_2O$ with the TG-DTG-MS technique [49] have been studied. The properties of $Ni(OH)_2$ precipitate and turbostratic $Ni(OH)_2$ have been compared [50], the formation of α-Cr_2O_3 from Cr(III) hydroxide [51] and the reactions of MbO_3 with manganese oxide and manganese carbonate [52] have been investigated. Other objects of studies were the thermal properties of magnesium propionate dihydrate [53], the thermal decomposition of precipitates containing Co and Ti [54], the reactions of boric acid with polyols and pentaerythrite under the effect of heating [55–56] solid-phase reactions between Ni(II) sulphide and manganese oxide [57] and between thallium nitrate and vanadium pentoxide [58].

The dehydratation processes of oxalate hydrates of Mg, Ca, Mn, Co, Ni, Cu, Zn and Cd have been investigated, indicating that the tetrahydrates lose their water of crystallization readier than the monohydrates. This was explained by a more stable structure of the latter [59]. The dehydratation of $CuSO_4.5H_2O$ [60], natural zeolites [61], synthetic zeolites [62], double salt hydrates [63], selenate hydrates of Mb(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) [64] and $CaHPO_4.2H_2O$ [65], and the desolvatation of RbF.HAc solvate [66] have been studied by thermal analysis.

Results of applied research

The hydratation of cements of different origin was studied depending on the amount and type of the flue-dust added [67]. Thermal analysis of lime, flue-dust and gypsum mixtures was carried out [68]. The corrosion of concrete stacks in power plants was followed by thermal analysis [69]. Corrosion of concrete caused by sulphate, alcohols and esters was studied [70].

In the field of industrial catalysts the following studies have been reported: activity measurement of active carbon in the presence of a nickel catalyst [71], utilization of Y-type zeolites as catalyst [72], non-pyrophoric nickel skeleton catalysts for hydrogenation reactions [73]. A Hungarian researcher works on the problems of pyrotechnical mixtures and retarded detonation [74, 75]. The study of the thermal properties of mechanically activated (ultra-finely ground) bauxite demonstrated that phase transitions take place at lower temperatures [76]. CO_2 and S content in pyritic and carbonatic bauxite was determined by combining the derivatograph and titrimetry of the evolved gas [77].

The main constituents of red clay have been identified [78], phase transitions in sulphide- and carbonate-containing ores from Silesian copper mines were investigated [79], Zn-dolomites from Silesia have been subjected to thermal analysis [80]. In the field of environment protection, the testing of effluents is of great importance. Thermal analysis was utilized for measuring the amounts of volatiles, of suspended petroleum and of $CaCO_3$ [81].

Organic compounds

Fundamental and applied research results

Thermal analysis has been frequently applied in investigations on the thermal stability and structure of coordination compounds, since the decomposition of these compounds, the order of splitting off individual ligands characterizes – as a first approach – the relative bond strengths.

The decomposition of Cu(II), Cd(II), Zn(II) and Mn(II) isoquinoline thiocyanates proceeds through the sulphides to the final oxides [82]. The melting points vapour pressures and heats of sublimation of Ga, In, Y, La, Gd, Fe, Zn, Pb, Li 2,2,6,6-tetramethyl-3,5-heptanedion were measured [83]. Heterogeneous reactions

between solid Ni(II) complexes and ammonia were investigated [84], the stability of the pyridine germanium catecholate complex was studied [85]. The derivatograph was applied for studying the decomposition of oxytricobalto acetate [86]. Thermal analysis of $Hg(CN)_2$, $K_2Hg(CN)_2$ and $KHg(Cb)_2.Cl.H_2O$ indicated that the complex compounds decompose at lower temperatures than $Hg(CN)_2$ [87]. The thermal stability of metal complexes of HET acid was studied using a derivatograph complemented by a titrimetric EGA device [88]. Further coordination compounds reported in the literature to have been subjected to thermal analysis are $Co(diox.H)_2(amine)_2.X$ [89], $Cu(C_2O_4)(NH_3)_2$ α, β, γ isomers [90], Cu(II), Ni(II), Co(II) pyridine, isoquinoline, α,α-dipyridyl or o-phenanthroline [91], $MHgJ_4$ complexes ($M = Ag^+, Cu^+, Hg_2^{2+}, Tl^+, Pb^{2+}$) [92], V, Cr, Mn, Co, Ni, Cu, Zn biguanide complexes [93], Zn(II), Cd(II), Mn(II), Hg(I), Hg(II), Th(IV), Ln(III) and Fe(III) complexes with pyridine, isoquinoline, α,α-dipyridyl and o-phenanthroline [94]. The study of seven different Cu(II) dithiocarbonate complexes indicated that the course of the TG curves was independent of the atmosphere applied [95]. Coordination compounds of Ln(III) nitrates and thiocyanates with cyclic polyethers were studied [96]. The phenomenon of thermal isomerization was observed with *trans*-$CoBr_2py_2(H_5O_2)Br$ which, in the solid phase, is transformed under the effect of heat into the *cis* form [97, 98]. The thermal analysis of coordination compounds of the $M(CO)_n.Cl_2L_2$ type allowed to determine the stability sequence (M = W or Mo, n = 2 or 3 and L = $P(C_6H_5)_3$ or $As(C_6H_5)_3$. $.OP(C_6H_5)_3$) [99]. Soviet researchers studied the thermodynamics of coordination compounds [100] and the structures of Fe(III), Rh(III) β diketone complexes [101]. Thermal properties of novel complexes (hexakis-DMSO – Cr(III)) have been investigated [102].

Thermal analysis is often being applied to plastics. The thermal stability of glycidyl ether and glycidyl ester type epoxy resins [103] and of polycarbonates [104] has been studied. Polyethylene studies demonstrated differences in DTA curves of aged samples, samples having been in use as extruded electric

Table 1

Statistical data on the publications dealing with thermal analysis and their temporal distribution.

(1970–1972)		(1973–1975)	
List of references	Number of publications	List of references	Number of publications
J. Thermal Anal.	132	J. Thermal Anal.	240
Thermochim. Acta	208	Thermochim. Acta	381
ICTA Proceedings (Davos) 1971	180	ICTA Proceedings (Budapest) 1974	277
Total	520		900

insulation and virgin material, respectively normal polyethylene [105]. The reactions taking place when coating a surface with polyester have been simulated by studying the thermal properties of polyesters and modified polyesters [106]. The derivatograph has been used for studying cross-linking taking place in polymers under the effect of heat [107]. Simultaneously with the thermal analysis of PVC, the evolved hydrochloric acid was determined by conductometry [108]. Carbonization processes of various polymers, simultaneously with the study of the structure of the formed carbon were investigated [109]. Combined thermal analysis and mass spectrometry was applied for analyzing polymers [110]. Polyurethane foams were studied using a quadrupol mass spectrometer aggregated with TG equipment [111]. Thermal analysis of aromatic polyesterimide type insulating resins [112] and acrylonitrite copolymers [113] has been reported. The stability of the β-alkylaryl ether bond characteristic for macromolecules in wood has been determined by thermal analysis, allowing to state that the stability of the bond depends on the conditions of lignin recovery [114]. A presumable model of the structure of oxycellulose has been established, based on the products of pyrolysis [115].

Many papers deal with thermal analysis of biopolymers. Stability studies of the structure of myosin and myofibrilla indicated that the former is decomposed in the temperature range of 370 to 285°, the latter at 490 to 520° [116]. A derivatograph was used for the thermal analysis of bones and for detecting biochemical changes resulting from disc injuries in the knee-joint [117]. The main constituents of bilestones, similarly to renal stones, can be determined by thermal analysis [118]. In thermal analysis of DL-β-phenylalanine and DL-β-aminophenyl-α-alanine, water is split off and dioxy-piperazine derivatives are formed [119]. Thermal analysis of tetrahydroperparin (THP) and its derivatives, being biologically active compounds, was performed [120]. Thermal analysis of plant leaves and leaves treated with nitric acid was reported [121].

In disubstituted phenyl-*p*-benzoylbenzoxybenzoate, the phase transitions solid crystal → liquid crystal → isotropic liquid were determined [122]. The action of flame-retardant additives on rayon fibres has been studied [123]. Thermal analysis of Li, Na, K, Rb and Cs formiates demonstrated that the ratio of carbonates and oxalates formed depends on the atmosphere [124]. Thermal analysis of some arylsulphanylamines containing benzene and naphthalene rings, resp., indicated that the compounds containing benzene rings have higher thermal stability [125]. Polish researchers report investigations of pseudo-homogeneous two-phase ion exchange membranes [126].

Numerous papers deal with thermal analysis of fuels. It was found that the thermal decomposition picture is very similar for peats of different origin [127]. On the TG curves of peat, xylite, lignite, brown coal, bituminous coal and anthracite, an abrupt weight-change precedes ignition [128]. The mechanism of the reaction taking place between coal and oxygen was studied in the temperature range of 20 to 300° [129]. The gasification process of coal was studied from the technological point of view [130].

Thermal analysis of the distillation residues of various crude oils has been performed [131].

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RÉSUMÉ — Mise au point sur le développement des méthodes thermiques associées au cours des trois dernières années et discussion des résultats obtenus.

ZUSAMMENFASSUNG — Es wird ein Überblick der vielseitigen thermischen Methoden gegeben und die wesentlichsten in den letzten drei Jahren erzielten Ergebnisse vorgestellt.

Резюме — Обсуждено развитие мультиплетных термических методов и результатов, полученных в течении последних трех лет.