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THERMAL BEHAVIOUR OF CARBOHYDRATES STUDIED BY HEAT FLOW CALORIMETRY

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The technique of heat flow calorimetry was used to study the thermal behaviour of different carbohydrates between 20°C and 270°C. The samples were analyzed by heating in sealed cells. The temperature range in which exothermic reactions, due to thermal decomposition, occurred varied widely depending on the type of carbohydrate investigated. Reaction enthalpies of 44 sugars and polysaccharides are given. Endothermic phenomena, such as fusion or vaporization of crystallized water, were also observed: fusion temperatures and enthalpies of 34 sugars and sugar alcohols are listed. Calorimetric curves showing crystallization of amorphous sucrose, cellobiose and lactose are also presented.

Carbohydrates upon heating undergo several physical changes and chemical transformations which can be studied by thermal analysis techniques.

Early investigations on the thermal behaviour of carbohydrates were focused above all on the chemical aspects of the pyrolysis of glucose [1, 2], starch [3, 4], cellulose [5, 6] and cellulose-rich materials such as wood [7], paper [8] and cottonwood [9]. The experimental design of most of these studies led to sample vaporization and elimination of the volatiles from the reaction zone. It thus precluded detection of early exothermic phenomena, which are important for the food manufacturer [10–13] and for which carbohydrates have been shown recently to be largely responsible in cereals [11], in coffee and chicory products [12] and in milk powders [13]. Differential thermal analysis (DTA) or other calorimetric techniques have also been used to study the physical state of lactose and sucrose in their pure form [14–16] and of lactose in milk powders [13, 17–19].

The present study is a systematic investigation into the thermal behaviour – decomposition, fusion, crystallization – of a range of sugars and polysaccharides. Heat flow calorimetry was used in order to obtain quantitative data concerning the net heat changes produced by carbohydrates during their heating. Under the working conditions chosen, the volatiles formed cannot leave the heated cell. The net heat release measured during carbohydrate decomposition represents therefore the sum of many reactions and includes secondary reactions which are generally carefully avoided in

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studies focusing on the mechanistic aspects of pyrolysis. As a consequence, our method does not allow the elucidation of details of the decomposition reactions, but still provides useful information about the overall process taking place when carbohydrates are heated. This information is important for safely directing high-temperature processing operations and understanding deterioration phenomena in foods.

Experimental

The instruments used were a special high-pressure DTA apparatus (constructed by Netzsch) and a conventional heat flow calorimeter (Setaram C 80) of Calvet type. These instruments have been described in detail in a previous paper [10]. A hot stage microscope (Stanton Redcroft HSM-5) was used to assist in the interpretation of the calorimetric curves, especially in those cases where more than one endothermic phenomenon was observed. Gas-chromatography (GC) was carried out on a Hewlett Packard 5710 A model connected to an automatic integrator 3380 A, using trimethylsilyl (TMS) derivatives [20] on a 90 cm glass column packed with 5% SE 30 on Chromosorb W-AW (DMCS) 80–100 mesh.

With the heat flow calorimeter, sealed cells capable of supporting a pressure increase of up to 100 bar were used. Temperature was programmed at 1 deg/min from ambient to 270°. The samples were heated in an atmosphere of air and of their own volatiles. Sample weights of 1g were generally chosen.

On the obtained calorimetric curves, onset temperatures of endothermic or exothermic transitions were determined somewhat arbitrarily by measuring the first deviation from horizontal base line or horizontal base line prolongation. This procedure gives only approximate values in cases where endothermic and exothermic peaks are incompletely resolved. The reaction enthalpies indicated correspond to the surface of the peaks under or above horizontal base line continuation. The peak temperatures are given without any correction.

Samples

Sugars and polyols were from Fluka AG (Buchs, Switzerland) except glucoheptose and cellobiose (from Sigma Chemicals, St Louis, USA), maltitol and isomaltitol (gifts from Anic, Milan, Italy and from Süddeutsche-Zucker AG, Mannheim, Germany, respectively). Polysaccharides were from Sigma Chemicals except inulin (from Fluka AG), pullulan (from Hayashibara Ltd, Okayama, Japan) and maltodextrins (Roquette Frères, Lestrem, France). All samples were of the purest grade available. Anhydrous α-lactose was prepared by refluxing its monohydrate in dry methanol, filtering and drying in vacuo. β-lactose was obtained by crystallization at 95° from water. Amorphous α- and β-lactose were prepared by rapidly freezing cold dilute solutions of the corresponding anomers followed by freeze-drying. They contained 94% of the desired anomer when checked by GC. An amorphous mixture of α- and β-lactose in the ratio 1:1.5 was obtained by spray-drying a concentrated lactose solution.

Results and discussion

The shape of the calorimetric curves was widely different depending on the type of carbohydrates analyzed. As expected, all polysaccharides studied, except inulin, exhibited only exothermic peaks, corresponding to their decomposition. In the case of the mono- and oligosaccharides the decomposition exotherm was preceeded by at least one endotherm corresponding to their fusion and in some cases to loss of water. Most sugar alcohols and the cyclitol myo-inositol did not decompose in the studied temperature range and exhibited only an endothermic peak due to melting. Finally, calorimetric curves of amorphous sugars showed an early exothermic peak due to crystallization. These different phenomena will be dealt with individually in the subsequent paragraphs.

(a) Exothermic decomposition

The onset temperatures of the exothermic peaks due to decomposition varied between 100° and 230° and the peak temperatures between about 150° and 250°. The observed enthalpies ranged from 300 to 800 Jg^{-1}. Table 1 shows the calorimetric data obtained for selected monosaccharides, di- and oligosaccharides and polysaccharides.

The ketohexoses, fructose and sorbose, had peak temperatures 25–35° below all the other monosaccharides, which otherwise exhibited very similar degradation temperatures. This lower stability of the fructose moiety is probably responsible for the relatively early onset of decomposition and low peak temperature of 215° for the oligosaccharides sucrose, lactulose and raffinose, which all have a 1 → 2–linked terminal fructose unit. Turanose and melezitose however were again slightly more stable, probably because of the 1 → 3–linkage between glucose and fructose. β-1 → 4–linked disaccharides seem to be slightly more stable than α-1.4 or α-1.6 linked, the α-1.1 linked trehalose being in-between. These observations partially disagree with the findings of Pavlath and Gregorski [21]. However, such structural considerations cannot have a definitive value in view of the small number of samples with similar structure. Unfortunately, planteose and plantebiose were not available for study, whereas gentianose has a melting point of 211°, which would cause serious distortion of its exothermic peak. Already for sucrose the determined onset temperature is 20° above the expected 170°, when compared to raffinose and lactulose, since melting endotherm and decomposition exotherm were slightly overlapping. Accordingly, onset temperature and reaction enthalpies of sucrose and especially of cellobiose (cf. Fig. 1) must be considered as rough estimates.

The reaction enthalpies associated to the decomposition of sugars were in general between 600 and 800 Jg^{-1}. Exceptionally low enthalpies were found for rhamnose,

Table 1 Exothermic reaction temperatures and enthalpies of selected carbohydrates

Sample	Onset temp., °C	Peak temp., °C	Enthalpy, Jg^{-1}
Monosaccharides			
D-Arabinose	190	225	740
D-Xylose	195	225	620
D-Ribose	190	220	780
α-D-Glucose • H_2O	185	230	630
α-D-Glucose	200	230	700
β-D-Glucose	200	235	710
D-Galactose	200	230	700
D-Fructose	170	195	700
L-Sorbose	175	195	700
L-Rhamnose • H_2O	195	225	420
L-Fucose	175	225	560
D-Glucoheptose	195	220	660
D-Galacturonic acid • H_2O	135	150	600
D-Glucuronic acid	155	190	670
D-Glucurono-δ-lactone	175	200	770
Disaccharides			
α-Lactose • H_2O	195	235	680
α-Lactose	220	235	700
β-Lactose	230	245	800
Maltose • H_2O	190	220	750
Cellobiose	230	240	600
Melibiose • H_2O	185	225	780
Sucrose	190	215	770
Turanose	185	220	740
Trehalose • 2 H_2O	215	235	710
Lactulose	170	215	370
Maltitol	215	240	320
Isomaltitol	230	250	330
Oligosaccharides			
Melezitose • 2 H_2O	195	225	710
Raffinose • 5 H_2O	175	215	670

Table 1 (cont.)

Sample	Onset temp., °C	Peak temp., °C	Enthalpy, Jg^{-1}
Polysaccharides			
Inulin	170	200	700
Pullulan	160	210	700
Maltodextrin MD01	180	225	720
Maltodextrin MD33	180	220	720
Amylose	180	225	690
Amylopectin	200	230	630
Cellulose	220	245	650
Guar gum	130	210	680
Carob gum	125	205	680
ι-Carrageenan	150	155	490
λ-Carrageenan	175	180	440
κ-Carrageenan	155	160	590
Agar (double peak)	140	170	740
	190	205	
Alginic acid	100	150	620
Polygalacturonic acid	130	170	540

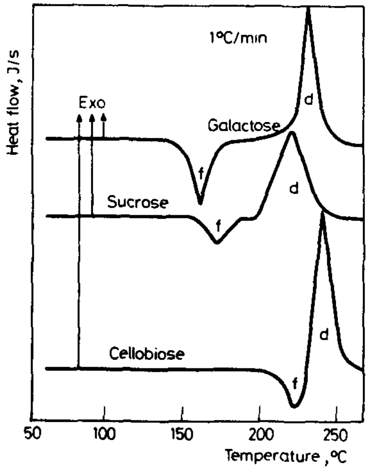

Fig. 1 Calorimetric curves of galactose, sucrose and cellobiose (all three heated in sealed cells). f: fusion, d: decomposition

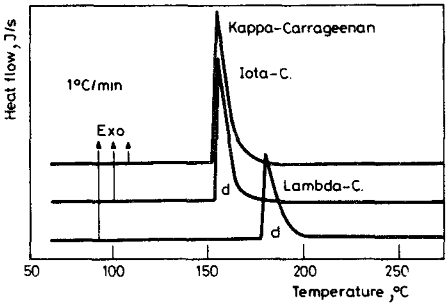

Fig. 2 Calorimetric curves of kappa- and lambda-carrageenans (all three heated in sealed cells). d: decomposition

lactulose, maltitol and isomaltitol. For the latter two substances, this can be easily explained by assuming that only the non-hydrogenated half of the molecule decomposed after scission of the glycosidic bond. Hydrogenation of maltose retarded the onset of decomposition by 25°. It is not fully clear if the determined enthalpies do necessarily reflect the totality of exothermic reactions occurring in this temperature range. Thus it has been shown by DTA combined with TG measurements that sucrose [22] and xylose [7] shortly after melting undergo condensation reactions involving loss of water and resulting in endotherms at about 210°.

Among the polysaccharides, inulin was less stable than all the glucans, when judging from the peak temperatures. This is in agreement with the order of stability observed in the corresponding monomers fructose and glucose. Within the glucans, the order of stability was pullulan < maltodextrins = amylose < amylopectin < cellulose. For the last three products this is in agreement with previous reports [3] and with the stability of the building units maltose < cellobiose and probably also maltose < isomaltose concluding from the order of stability of their hydrogenated analogues, i.e. maltitol < isomaltitol.

Among the other polysaccharides screened, all three types of carrageenans (iota, lambda, kappa) showed extremely fast exothermic reactions, the leading edges of the peaks being almost vertical (cf. Fig. 2). Iota- and kappa-carrageenan which contain about 30% of 3.6-anhydrogalactose decomposed 20° earlier than lambda-carrageenan which is virtually devoid of such building units. In agreement with this interpretation is the still earlier decomposition onset of agar, which contains about 50% of 3.6-anhydrogalactose. The rather low enthalpies of reaction might be due to the high sulfate ester content of these polysaccharides. Guar gum and carob gum showed near identical calorimetric curves; the different substitution pattern of the mannan skeleton by galactose units was thus not reflected by our measurements. The two polyuronides included in this study, alginic acid and polygalacturonic acid, were among the least stable, in agreement with the low stability of the uronic acids.

When comparing our values with those reported in the literature, where measurements were performed in open cells under a stream of air or inert gas, it is obvious that the onsets of exothermic decomposition observed in our experimental conditions (sealed cells) were considerably lower. Thus Morita, by DTA, found no exotherms for starches, amylose or amylopectin before 375°, the early decomposition phenomena under these conditions being characterized by endotherms [4]. Similarly, for cellulose, the first exotherms have most often been reported to appear above 350°. The early exotherms reported in the present study are associated with considerable enthalpies when considering the small mass losses occurring at these temperatures. Thus, for anhydrous glucose, we have recovered 88% of the sample weight at the end of an experiment, although no more glucose could be detected. It is worth noting that after heating D-glucose for 30 min to 175°, which is 10° below our onset temperature, the residue contained still 81% of glucose and 96% of the original weight [1].

We found an excellent agreement between the exothermic decomposition phenomena observed by heat flow calorimetry and the results of TG measurements. Thus, for xylose, Shafizadeh [7] found weight loss started at about 195° with a first maximum in the rate of decomposition at 225°. These values correspond exactly to our onset temperature and peak temperature respectively. Similarly, for cellulose, Dollimore and Hoath [23, 24] showed that major mass losses began at 240°, which corresponds to our peak temperature, more than 90% of the mass however being retained at 300°.

Thus, it is obvious that calorimetric techniques, TG and evolved gas analysis are complementary thermal analysis methods.

(b) Endothermic phenomena

The endothermic phenomena observed correspond to water loss and fusion. For selected carbohydrates and for some sugar alcohols, onset temperatures, peak temperatures, literature values of melting points and fusion enthalpies are given in Table 2.

With sugars containing crystallized water, three different types of behaviour could be distinguished: melting appears before water vaporization (e.g. for glucose monohydrate as shown in Fig. 3, for rhamnose monohydrate and for melezitose dihydrate),

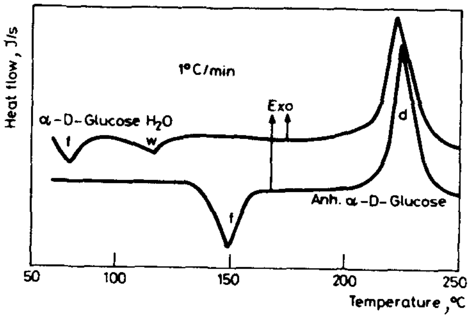

Fig. 3 Calorimetric curves of anhydrous α-D-glucose and α-D-glucose monohydrate (both heated in sealed cells). f: fusion, d: decomposition, w: water desorption

Table 2 Fusion temperatures and enthalpies of selected sugars and sugar alcohols

Sample	Onset, °C	Extrapolated onset, °C	Peak temperature, °C	Literature melting point, °C	Literature Reference, N°	Enthalpy, Jg^{-1}
Monosaccharides						
L-Arabinose	135	145	155	158–160	40	260
D-Xylose	135	140	150	145	40	280
D-Ribose	60	75	90	87	40	150
α-D-Glucose • H_2O	65	70	75	83	40	60
α-D-Glucose	135	140	150	146	40	180
β-D-Glucose	130	140	150	148–150	40	150
D-Galactose	140	155	165	167	40	280
D-Fructose	80	105	115	102–104	40	180
L-Sorbose	140	150	160	159–161	40	250
L-Rhamnose • H_2O	85	90	100	93–94	40	210
L-Fucose	115	125	130	145	40	190
D-Glucoheptose	130	170	180	193	41	270
D-Galacturonic acid •	80	100	105	110–115	40	70
H_2O	120	125	130	159–160	40	50
D-Glucuronic acid	120	130	140	156	40	280
D-Glucurono-δ-lactone	135	150	165	163–165	40	120
Disaccharides						
α-Lactose	160	185	195	223	41	220
β-Lactose	195	210	220	229.5	25	250
Cellobiose	195	210	220	225	40	160
Sucrose	160	175	185	188	40	120
Turanose	145	155	165	157	40	150
Lactulose	130	145	155	158	40	110
Oligosaccharides						
Melezitose • 2 H_2O	135	155	165	153–154	40	140
Raffinose • 5 H_2O	70	80	85	77–78	40	150
Polyols						
Meso-erythritol	95	115	125	118–120	40	330
L-Arabinitol	75	90	105	101–102	40	230
Xylitol	65	90	100	93–94	40	250
Ribitol	85	100	110	102	40	250

Table 2 (cont.)

Sample	Onset, °C	Extrapolated onset, °C	Peak temperature, °C	Literature melting point, °C	Literature Reference, N°	Enthalpy, Jg^{-1}
D-Glucitol (Sorbitol)	60	85	95	97	42	150
D-Mannitol	140	155	170	166	40	290
Galactitol (Dulcitol)	130	175	190	186–187	40	330
Maltitol	115	140	150	146–147	43	150
Isomaltitol	150	160	175	–	–	180
Cyclitol						
Myo-inositol	200	215	230	225–227	40	260
Polysaccharide						
Inulin	140	145	150	178	41	40

melting appears after water vaporization (e.g. for lactose monohydrate), the sugar melts as hydrate (e.g. for raffinose pentahydrate). In the second case, the fusion peaks were distorted because of the use of sealed cells. These data were not included in Table 2. In the last case, only one endothermic peak was observed in the calorimetric curves.

It is worth noting, that the observed fusion temperatures of sugars do not always correspond to the traditionally accepted melting points such as in the case of β-lactose [25], and it seems that for organic compounds in general, more objective values should be obtained by calorimetric techniques than by the standard optical methods [26, 27]. The great differences between onset, extrapolated onset and peak temperatures (cf. Table 2) are certainly due to the large sample amount used [26] in our study and to the relatively weak thermal conductivity of such powders. It is however obvious that, in some cases, low melting temperatures could also be due to insufficient sample purity.

In general, the surface of the endotherms in the calorimetric curves should correspond to the fusion enthalpy of the compound. As already mentioned for the exothermic reaction enthalpies, problems arise in quantifying when endothermic and exothermic peaks are poorly resolved. In addition, reducing sugars anomerize during fusion [2], but the thermal contribution of the anomerization is unknown. Such difficulties do not exist for sugar alcohols as shown in Fig. 4, which presents the calorimetric curves of one sugar alcohol (xylitol) and of the cyclitol myo-inositol: both exhibit only an endothermic peak due to melting and do not anomerize.

The fusion enthalpy (ΔH_f) values determined for α-glucose, mesoerythritol, xylitol, mannitol and maltitol correspond to the literature data. Curiously, however, a great

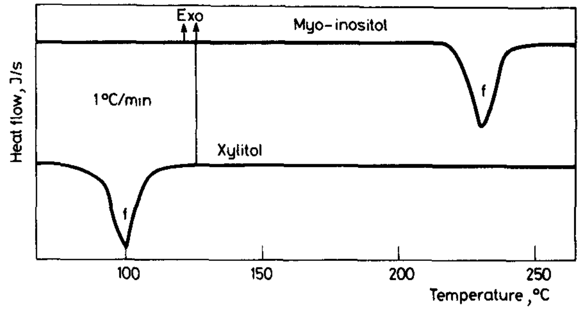

Fig. 4 Calorimetric curves of xylitol and myo-inositol (both heated in sealed cells). f: fusion

discrepancy appears for sucrose between ΔH_f of 56 Jg^{-1} indicated by Kohlrausch [28] and our value of 120 Jg^{-1}. We have however consistently found this higher value, when measuring sucrose samples from different sources. In other cases, the enthalpy values listed in Table 2 are higher than those found in the literature [17, 25, 29]. This may be due to the low heating rate chosen here and to the better base line obtained with modern instruments. These reasons may, for example, explain the differences found for the fusion enthalpy of β-lactose: 203 Jg^{-1} by Berlin [15], 229 Jg^{-1} by Ross [17] and 250 Jg^{-1} in the present work.

(c) Crystallization

Amorphous sugars may crystallize when heated [15, 16, 30] and a corresponding exothermic peak is found in the calorimetric curves often between 50° and 130°. Figure 5 shows the calorimetric curves of amorphous sucrose and cellobiose with the corresponding crystallization peaks. The interest of this calorimetric information is explained in more detail for the case of lactose.

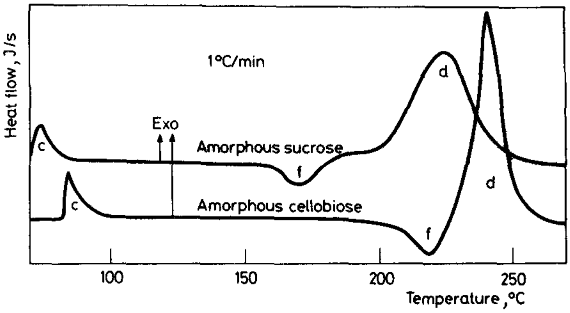

Fig. 5 Calorimetric curves of amorphous sucrose and cellobiose (both heated in sealed cells). f: fusion, d: decomposition, c: crystallization

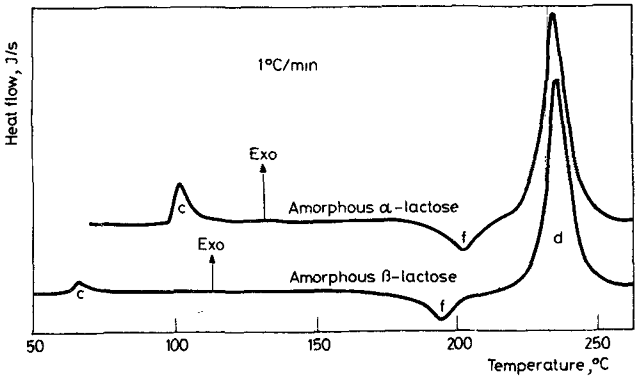

Fig. 6 Calorimetric curves of amorphous α- and β-lactose (both heated in sealed cells). f: fusion, d: decomposition, c: crystallization

Lactose exists in two amorphous (α or β) and in three crystalline states (anhydrous α or β, hydrated α). In dry milk products, depending on the processing or storage conditions, all these states of lactose may be present and can affect the homogeneity and solubility of dairy products [18, 31].

The thermal behaviour of amorphous α- and β-lactose is shown in Fig. 6. Both anomers partially crystallized, under retention of their anomeric state. This was concluded from the ratios of the anomers obtained by GC after interrupting the heating cycle just after the crystallization exotherm. The temperature range of the crystallization peaks for α- and β-lactose were clearly distinct: this also allowed the characterization of the anomeric state of the lactose sample. However, a sample of amorphous lactose containing both anomers in the ratio $\alpha:\beta = 1:1.5$ failed to crystallize and consequently showed no fusion either.

In principle, the proportion of crystallized lactose in a sample is given by:

$$\frac{\Delta H_{fp} - \Delta H_{cp}}{\Delta H_f} 100$$

where ΔH_{fp} and ΔH_{cp} are the measured enthalpies of fusion and crystallization, respectively, and ΔH_f the heat of fusion of an entirely crystalline sample. Correspondingly, the part of lactose crystallized during heating would be

$$\frac{\Delta H_{cp}}{\Delta H_f} 100$$

The literature concerning thermal behaviour of the different forms of lactose and its interpretation is controversial [14, 15, 17, 32]. Our observations on the behaviour of amorphous lactoses demonstrate that it is necessary to make sure that all lactose *in milk powders is crystalline before attempting to determine the amounts of α- and* β-lactose from their respective fusion enthalpies according to the method of Ross [17].

For α-lactose hydrate it is certain, as shown by Berlin [15], that the calorimetric curves can be perturbed after water desorption by undesired secondary phenomena such as water adsorption or sample dissolution. Because of the working conditions chosen in the present work, a more precise comparison with the literature concerning lactose characterization is difficult.

Conclusion

This study has shown that heat flow calorimetry can bring important contributions in characterizing the thermal behaviour of carbohydrates, when the samples are heated in sealed cells. In particular, the measurements of the early decomposition reactions should help in defining the role of carbohydrates in self-heating and thermal explosions of foodstuffs. The study of crystallization, water desorption and fusion of carbohydrates will, no doubt, become increasingly important for controlling physicochemical properties of foods, e.g. in relation with texture, storage stability, solubility characteristics and even flavour retention. By using new methods and techniques of thermal analysis [33–38] more complementary information will be obtained.

Part of this work has already been presented in a premilinary form [39].

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Zusammenfassung — Die Technik der Wärmeleitungs-Kalorimetrie wurde zur Untersuchung des thermischen Verhaltens von verschiedenen Kohlenhydraten zwischen 20°C und 270°C eingesetzt. Die Muster wurden in dicht verschlossenen Kapseln erhitzt. Der Temperaturbereich, in welchem exotherme Reaktionen infolge thermischer Zersetzung abliefen, änderte sich stark in Abhängigkeit vom untersuchten Kohlenhydrattyp. Reaktionsenthalpien von 44 Zuckern und Polysacchariden wurden ermittelt. Endotherme Phänomene, wie das Schmelzen oder das Verdampfen von Kristallwasser wurden auch beobachtet. Schmelztemperaturen und Schmelzenthalpien von 34 Zuckern und Zuckeralkoholen sind tabelliert. Kalorimetrische Daten der Kristallisation amorpher Saccharose, Cellobiose und Lactose werden diskutiert.

Резюме — Метод калориметрии теплового потока был использован для изучения термического поведения различных углеводов между 20 и 270 °C. Образцы анализировались нагревом в закрытых ячейках. Вследствии термического разложения, интервал температур экзотермических реакций изменялся в широких пределах в зависимости от типа исследуемого углевода. Приведены энтальпии реакций для 44 сахаров и полисахаридов. Наблюдались такие эндотермические явления, как плавление или испарение кристаллизационной воды. Приведены температуры плавления и энтальпии для 34 сахаров и многоатомных спиртов. Представлены калориметрические кривые, показывающие кристаллизацию аморфной сахарозы, целлобиозы и лактозы.