Thermochimica Acta, 93 (1985) 49-52
Elsevier Science Publishers B.V., Amsterdam

DEKRYPTONATION THERMAL ANALYSIS OF SOME COMPLEXONES

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ABSTRACT

Dekryptonation thermal analysis of five complexones /racemic 2,4-diaminopentane-N,N,N',N'-tetraacetic acid; 2,3-diaminobutane-N,N,N',N'-mesotetraacetic acid; 2-methyl-1,2-diaminopropane-N,N,N',N'-tetraacetic acid; racemic 2,3-diaminobutane-N,N,N',N'-tetraacetic acid; 1,2-diaminobutane-N,N,N',N'-tetraacetic acid/, used as analytical reagent, is described.

INTRODUCTION

Dekryptonation thermal analysis /DkTA/ is based on the incorporation of ^{85}Kr into a solid substance and following its release at different temperatures. The characteristics of DkTA are set on the one hand by the properties of the indicator used, particularly its chemical inertness, and on the other hand, by the possibility of incorporating atoms of krypton into different positions in the solid carrier. It is possible to vary the type of ^{85}Kr incorporation by using different techniques /bombardment, diffusion, sublimation/ as well as by changing the condition of these techniques /accelerating potential, temperature, pressure, dose, etc./ [1].

Every device for DkTA consists of two main parts: dekryptonation and detection. The dekryptonation section consists of a suitable arranged small oven of which the temperature can be controlled. In this oven is a holder of the radioactive Kryptonate sample and gas can flow through it as well. The detection part consists of the detector, rate-meter and plotter.

Dekryptonation studies of complexones, used as analytical rea-

Proceedings of ICTA 85, Bratislava

gents, enabled us to study a new aspect of the structure, as well as the physicochemical and analytical properties of these materials.

The following complexones were studied:

1. racemic 2,4-diaminopentane-N,N,N',N'-tetraacetic acid /sample 1/,
2. 2,3-diaminobutane-N,N,N',N'-mesotetraacetic acid /sample 2/,
3. 2-methyl-1,2-diaminopropane-N,N,N',N'-tetraacetic acid /sample 3/
4. racemic 2,3-diaminobutane-N,N,N',N'-tetraacetic acid /sample 4/,
5. 1,2-diaminobutane-N,N,N',N'-tetraacetic acid /sample 5/.

EXPERIMENTAL

The incorporation of ^{85}Kr into complexones have been carried out by bombardment of the samples with accelerated ^{85}Kr ions [2].

A Mettler Thermoanalyzer TA 2 was used for the DTA, TG and DTG measurements, viz: heating rate: 6 $^{o}C.min^{-1}$, nitrogen flow rate: 100 $ml.min^{-1}$.

The dekryptonation thermal analysis was made with the device described in [3]. The diagram of device used is shown in Fig.1.

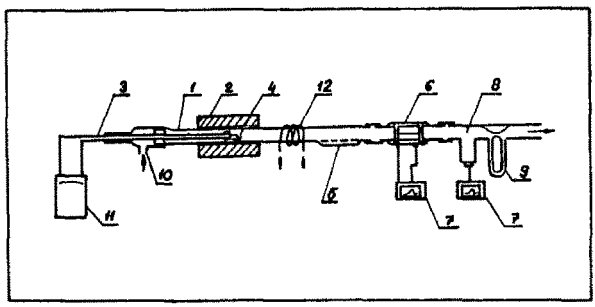

Fig.1 The diagram of the device for dekryptonation thermal analysis with flow-type GM-tube /1 - Ground quartz joint, 2 - oven, 3 - thermometer, 4 - platinum vessel for a sample, 5 - container with ethanol, 6 - flow-type GM-tube, 7 - rate-meter with plotter, 8 - window-type GM-tube, 9 - flow-meter, 10 - gas inlet, 11 - millivoltmeter, 12 - water cooler/

The carrier gas which is also counting gas of the GM-tube flows from a pressure tank through a reduction valve and manometer to a small oven /dekryptonation chamber/ containing the kryptonated complexone. The released ^{85}Kr is carried from the oven by the carrier gas to a flow-type GM tube and then to a flow-meter. The plotter joined to the rate-meter records the intensity of radioactivity detected by the GM-tube. The dekryptonation chamber consists of ground quartz joint; the shorter part contains a thin ceramic rod together with a thermocouple and inlet for the carrier gas. At the end of the ceramic rod is a platinum vessel for the sample. It is so arranged that the joint of the thermocouple is just above the sample, and therefore the measured temperature in the oven is the same as that of the sample. The temperature in the oven is regulated by a transformer which is controlled by an electric motor with constant rate of revolution.

The results obtained by thermal and dekryptonation thermal analyses of complexones are shown in Fig.2 - 6. The DkTA curves have one or several maximums, which correspond to the process of water releasing or to the structural changes /most often melting process/.

Sample 1 shows one maximum at 140 °C, which corresponds to the melting point temperature, determined in Kofler block /130-140 °C/ /Fig.2/. Krypton is released from the temperature of 120 °C.

Sample 2 /Fig.3/ shows 3 steeply decreasing maximums at temperatures 130 °C, 160 °C and 220 °C. Maximum at 130 °C corresponds to the release of the first molecule of crystal water. In this process the maximum amounts of radioactive krypton-85 is released. At 160 °C the second molecule of crystal water is released and maximum at 220 °C corresponds to the melting of the sample. Similar course of DkTA curve shows sample 4 /Fig.5/, however, here is the maximum at 160 °C instead of at 130 °C /the release of crystal water/

The DkTA curve of the sample 3 /Fig.4/ has a high and wide maximum at 190 °C with the beginning of ^{85}Kr release at ~ 150 °C. The melting point determined in Kofler block was found to be 185 - 207 °C. In the case of sample 5 /Fig.6/ an increased background at temperatures up to 150 °C without any expressive maximum is observed. The maximum lies at 280 °C, which corresponds to the melting of the sample.

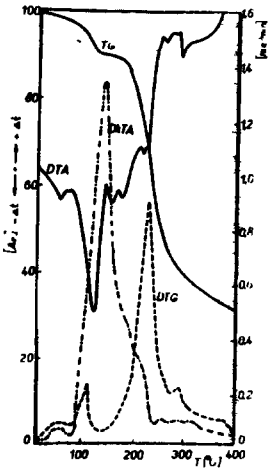

Fig.2
Sample 1

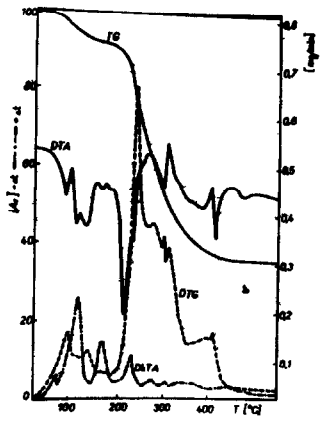

Fig.3
Sample 2

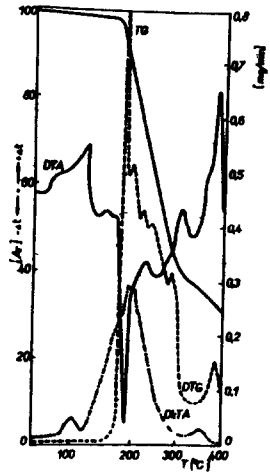

Fig.4
Sample 3

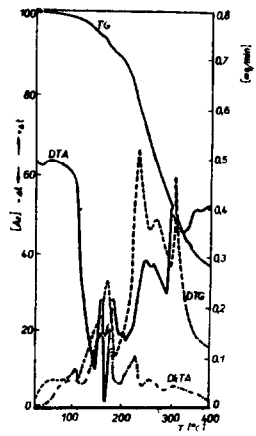

Fig.5
Sample 4

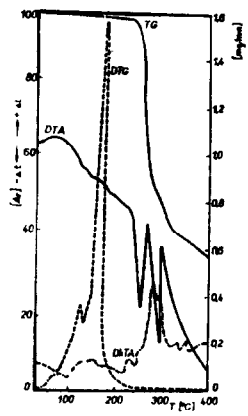

Fig.6
Sample 5

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