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RECENT PROGRESS IN LOW TEMPERATURE MICROCALORIMETRY

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SUMMARY

The present situation of microcalorimetry below 120 K is critically reviewed. The construction and operation of a new differential-isoperibol-scanning calorimeter is described.
This contribution only consists of an extended abstract because many of the reported developments and techniques were discussed very recently or will be presented in detail elsewhere; references are indicated.

EXTENDED ABSTRACT

For a long time calorimetry below 120 K has been a domain of specialized laboratories and this technique was not available for other areas of research as chemistry, biology or medicine. The application of computer-controlled measurements, the use of modern and more sensitive electronics enabled higher calorimetric and thermometric resolution and better absolute accuracy, but did not essentially contribute to wider-spread application of low temperature calorimeters. In addition, the design of calorimeters was simplified and the necessary sample-masses were considerably reduced (ref. 1) to typically several hundred mg (instead of 10 - 100 g). However, it seems that the adiabatic calorimetry is restricted for ever to sample weights of greater than about 200 mg and temperatures above 2 K.

In recent years the development of adiabatic calorimetry and the experimental improvements were previously reviewed critically (ref. 2).

In order to determine heat capacities of very small samples (weights smaller than 50 mg) new experimental techniques are required. Recently, the actual trends and requirements of microcalorimetry at low temperatures have been outlined in ref. 3.

In the second section of this presentation (oral only) the basic principles, construction and operation of a new type of microcalorimeter was given: a differential, isoperibol, scanning calorimeter (DISC), which earlier was explained in ref. 2 and will be described in detail in the near future (ref. 4). This new microcalorimeter is fully automated; its cryogenic equipment consists of simplified, commercially available components (mainly a continuous flow

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cryostat). For thermometry Germanium-sensors and Au-Fe(0.03) vs. Chromel thermocouples are used. First experiences by calibration measurements with 50 mg copper-samples showed that the DISC can be used in the temperature range from 10 K to room temperature; above 70 K even in the presence of exchange gas within the calorimetric cell. So far, total heat capacities of 0.01 μJ/K have been resolved with a precision of 1 - 2%.

In a third part, a summary of recent developments (refs. 5,6) and trends in "small-sample-calorimetry" at low temperatures was presented and some future aspects, possibilities and necessities on this topic were discussed (see also ref. 3).

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REFERENCES

1 E. Gmelin, Thermochimica Acta 29 (1979) 1-39.
E. Gmelin and K. Ripka, Thermochimica Acta 85 (1985) 255-258.
2 E. Gmelin, Thermochimica Acta 110 (1986) 183-208.
3 E. Gmelin, Proc. of the 11th Int. Conf. on Cryogenic Engineering (Berlin, 22-25 April, 1986), Eds. G. Klipping and J. Klipping, Butterworth, London, 1986, pages 602-606.
4 Details will be presented in the Proceedings of ESTAC-3, "European Symposium on Thermal Analysis and Calorimetry", Jena (DDR), on August 23-27, 1987.
5 J.M. DePuydt and E.D. Dahlberg, Rev. Sci. Instrum. 57 (1986) 483-486.
6 M. Regelsberger, R. Wernhardt and M. Rosenberg, J. Phys. E: Sci. Instrum. 19 (1986) 525-531.