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A SIMPLE LOW-TEMPERATURE DTA MODEL FOR TEACHING PURPOSES

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ABSTRACT

A simple model for low-temperature DTA for teaching purposes is described, and its utility is demonstrated by measurements on mercury, pentane, cyclohexane and ammonium nitrate.

INTRODUCTION

DTA is an important thermoanalytical method which should be used in school courses, too. In a recent publication (ref. 1) a simple DTA apparatus for teaching purposes was described. The apparatus consists of an electric soldering pot as furnace and an aluminum cylinder with borings for reproducible positioning and symmetrical heating of the substances.

Substance and reference substance are placed into glass tubes; then two cromel-alumel thermocouples are inserted. A plotter is used to record thermal voltage of ΔT and T. The apparatus is capable of operating from ambient temperature to about 550 ^{o}C.

To demonstrate the broad range of application for DTA even at low-temperatures for teaching purposes, a very simple low-temperature DTA model is proposed and its capabilities are demonstrated by some examples.

EXPERIMENTAL

Apparatus: Aluminum cylinder (19,5 mm ID, height 60 mm) with three symmetrical borings (5,2 mm ID and 30 mm deep) on the top for holding samples and occasionally an additional temperature sensitizer and, on the bottom, a boring for attaching a conic aluminum body in order to regulate temperature increase.

As sample holder: Glass tubes, one end sealed, 5 mm ID, height 35 mm.

As temperature sensitizer: Two mantel thermocouples, Cromel-Alumel 1 mm D, Nr. 4AD 100/2000 (Philips)

As furnace: Electric soldering pot 0-120 W, 220 V Model ZWMS-R (ZEVA, D-3548 Arolsen, West Germany), heating plate, water bath

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Aluminum cylinder, sample holder, thermocouples and furnace (MAUER Lehrmittel und Labortechnik GmbH, D-6238 Hofheim am Taunus 7, West Germany)
Clamps and supports
Two channel y/t plotter

CHEMICALS

liquid nitrogen
Al_2O_3 reference material
1. Mercury
2. Pentane
3. Cyclohexane
4. Ammonium nitrate

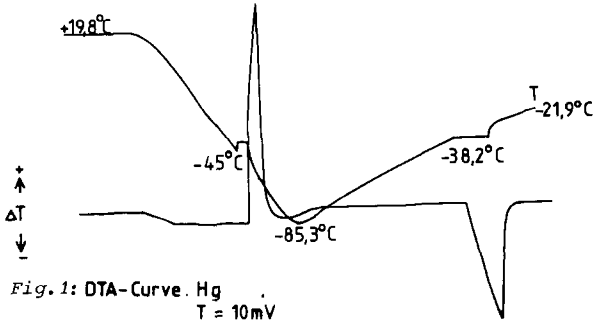

Fig. 1: DTA-Curve. Hg
T = 10mV
ΔT = 1mV
200mm/h

Execution of the experiments

The aluminum cylinder with the filled glass tubes and inserted thermocouples is put into place and the thermocouples are connected to the plotter. A Dewar filled with liquid nitrogen is positioned underneath the aluminum cylinder. In about one minute the substances are cooled to -196 ^{o}C. Then the liquid nitrogen is removed. We use three techniques for heating the aluminum cylinder and samples:
First, the surrounding air heats up the substance, e.g. mercury.
Second, after the aluminum cylinder has reached about -70 ^{o}C, it is then heated with a hot water bath, e.g. pentane, cyclohexane.
Third, the liquid nitrogen is substituted by the soldering pot. The furnace is heated up immediately with the dimmer, position 5, e.g. ammonium nitrate.

PARAMETERS

Substances: ca. 100 mg
Surrounding atmosphere: Air, static
Two channel plotter: ΔT-signal field width: 1 mV
T-signal field width: 10 mV (ammonium nitrate 50 mV)
paper speed: 20 cm/h

DISCUSSION

1. DTA investiqation of mercury (Fig. 1)

The ΔT curve shows an exothermic peak upon soldification while cooling the mercury and an endothermic peak upon heating to the melting point.

2. DTA investigation of pentane (Fig. 2)

While cooling pentane the DTA curve shows one exothermic peak whereas two endothermic peaks appear upon heating. The first peak demonstrates the freezing point, the second the melting point and the last the boiling point. Note that

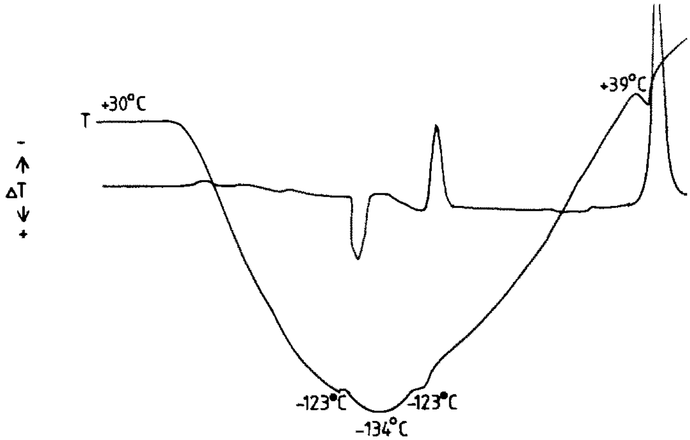

Fig. 2:

DTA-Curve. n-Pentane
T = 10 mV
ΔT = 1 mV
200 mm/h

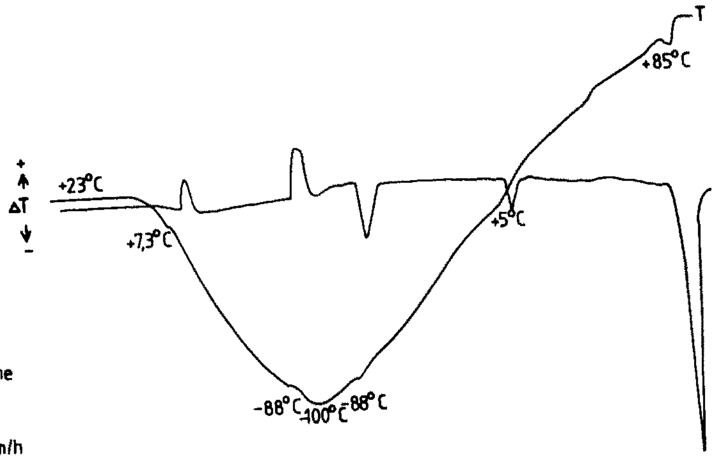

Fig. 3:

DTA-Curve. Cyclohexane
T = 10 mV
ΔT = 1 mV
200 mm/h

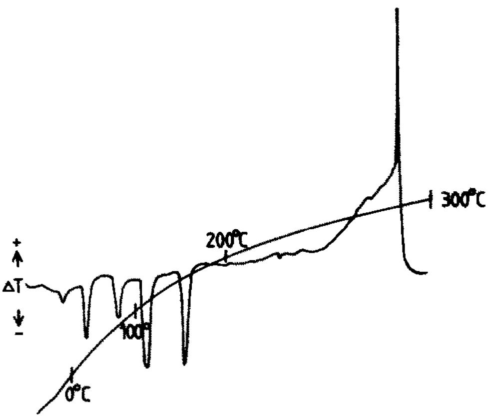

Fig. 4:

DTA-Curve: NH_4NO_3
T = 50 mV
ΔT = 1 mV
200 mm/h

the freezing point is a little lower than the melting point.

3. DTA investigation of cyclohexane (Fig. 3)

The cooling curve of cyclohexane shows two exothermic peaks. The first is the freezing point and the second is the transition point.

While heating from -100 ^{o}C up to 100 ^{o}C you will find three peaks: transition point, melting point and boiling point.

4. DTA investigation of ammonium nitrate (Fig. 4)

Upon heating NH_4NO_3 from room-temperature, one exothermic and four endothermic peaks were found (ref. 2) which correspond to three solid-state transitions between four different crystalline phases, the melting point and the decomposition point - the last depending on the heating rate. Employing the low-temperature model, you will find five endothermic peaks - the additional feature being a solid-state transition at -18 ^{o}C.

CONCLUSION

Although it is not possible to measure the exact temperatures at which endothermic and exothermic changes occur, you can detect all enthalpic changes in low-temperature DTA with a very simple model.

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