Thermochimica Acta, 83 (1985) 113–116
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

DTA IN A SIMPLE APPARATUS FOR TEACHING PURPOSES

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ABSTRACT

The usefulness of DTA for teaching purposes is demonstrated with a simple DTA apparatus. There are three main reasons to introduce DTA into chemical education:

1. to investigate a known substance, e.g., $CuSO_4 \cdot 5H_2O$, in order to demonstrate the efficiency of the method,
2. to indicate a chemical reaction, e.g., the "solid-state" reaction $BaO + CuSO_4 \longrightarrow BaSO_4 + CuO$
3. to investigate an unknown substance, e.g., "tin foil" was identified as lead.

INTRODUCTION

A cheap easy way to build a DTA apparatus for teaching purposes was devised by converting an electric hammer soldering iron into a DTA furnace (ref. 1). An improved model by using a soldering pot as furnace is described in this periodical (ref. 2). The apparatus consists of an electrically heated aluminum cylinder with two borings for reproducible positioning and symmetrical heating of the substances.

After having placed two glass tubes with substance and reference material into the borings the two cromel-alumel thermocouples are inserted and exchanged with another, the temperature differences as thermal voltages being recorded with a plotter.

The DTA furnace is equipped with a dimmer to receive different heating curves. These are strongly reproducible but linear only in the lower part.

With such a simple DTA model it is possible to not only demonstrate the principle of the method but also to illustrate the efficiency of this thermoanalytical method in given examples.

This simple apparatus should help to introduce a thermoanalytical method in chemical education so that a later user has at least some familiarity with DTA.

To demonstrate a new method and to compare its efficiency with that of other techniques a known substance is considered. The DTA investigation of $CuSO_4 \cdot 5H_2O$ has been well described by Borchard and Daniels (ref. 3), so this example is also a test for the DTA apparatus.

A student may know whether $CuSO_4 \cdot 5H_2O$ is dehydrated in one step or not, as

0040-6031/85/$03.30

well as the temperatures of dehydration.

Each chemical reaction combines a substance and enthalpy change. Normally only the consumed and developed substances are regarded. The "solid-state" reaction of $BaO + CuSO_4 \longrightarrow BaSO_4 + CuO$ (ref. 4) is easily carried out by heating a mixture of both dried substances. DTA is an excellent and optimally suited method to indicate a chemical reaction by a temperature effect, as well as the temperature at which the reaction starts. Sometimes it is possible to detect participating substances by characteristic DTA peaks, e.g. in the reaction of $AgNO_3 + KI \longrightarrow AgI + KNO_3$ (ref. 5).

Once the DTA method is known to the student, it can then be employed to investigate unknown substances. For students it is more attractive to recognize an unknown substance than to analyse and identify a known one.

Because in schools it would be desirable for students to be able to gain chemical experience more frequently by investigating metals, we were looking for a metal with a melting point under 500 ^{o}C because of the DTA apparatus.

Expensive bottles of wine often have a capsule of soft metal to protect the cork and to embellish the bottle, while cheaper ones have nothing or at most an aluminum or plastic capsule. The soft material appears to be tin foil. To compare the unknown metal you only need to compare with pure tin.

EXPERIMENTAL

<u>Apparatus:</u>

DTA apparatus: Modell 8307 (Fa. Mauer, D-6238 Hofheim, West Germany)
recording apparatus: two channel y/t plotter, Linoscript Nr. 2388 (Fa. Linseis, D-8672 Selb, West Germany)

<u>Chemicals:</u>

Al_2O_3 reference material
1. $CuSO_4 \cdot 5H_2O$
2. $CuSO_4$, CuO, $BaSO_4$, BaO
3. "tin foil", Sn, Pb

<u>Parameters:</u>

Substances: ca. 100 mg
Surrounding atmosphere: Air, static
Dimmer setting: 7
Two channel plotter: Δ T-signal field width: 10 mV
T-signal field width: 50 mV
paper speed: 500 mm/h, 200 mm/h, 500 mm/h

DISCUSSION

The DTA curve of $CuSO_4 \cdot 5H_2O$ (see Fig. 1) shows four endothermic peaks. The thermocouple that was inserted in the $CuSO_4 \cdot 5H_2O$ also measured the absolute temperature. You can see the four stopping points:

$$CuSO_4 \cdot 5H_2O\ (s) \xrightarrow{95\ ^{o}C} CuSO_4 \cdot 3H_2O\ (s) + 2H_2O\ (l)$$
$$2H_2O\ (l) \xrightarrow{100\ ^{o}C} 2H_2O\ (g)$$
$$CuSO_4 \cdot 3H_2O\ (s) \xrightarrow{115\ ^{o}C} CuSO_4 \cdot H_2O\ (s) + 2H_2O\ (g)$$
$$CuSO_4 \cdot H_2O\ (s) \xrightarrow{240\ ^{o}C} CuSO_4\ (s) + H_2O\ (g)$$

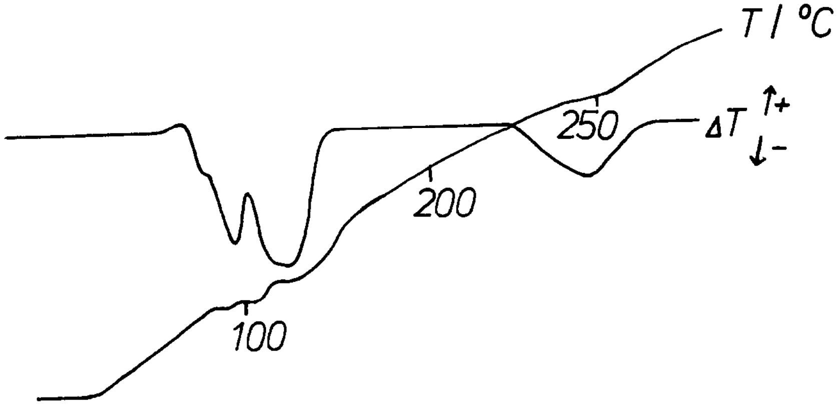

Fig. 1. DTA curve of $CuSO_4$.

The substances BaO, $BaSO_4$, CuO and $CuSO_4$ show no DTA signal from ambient to ca. 350 ^{o}C, correspondingly no physical changes or chemical reactions. The mixture of BaO with $CuSO_4$ shows a strong exothermic peak (see Fig. 2) indicating the "solid-state" reaction

$$BaO + CuSO_4 \longrightarrow BaSO_4 + CuO$$

The exothermic effect is so large that you don't need the DTA signal, as even the temperature curve shows an exothermic peak. You also see the mixture has become black by virtue of the CuO produced.

When this experiment is repeated by heating up the mixture for a second time or investigating the mixture of $BaSO_4$ and CuO by DTA no thermoanalytic signal appears. In this example you can not record the individual substances whereas you do find signals in the case of:

$$AgNO_3 + KI \longrightarrow AgI + KNO_3 \qquad \text{(ref. 5).}$$

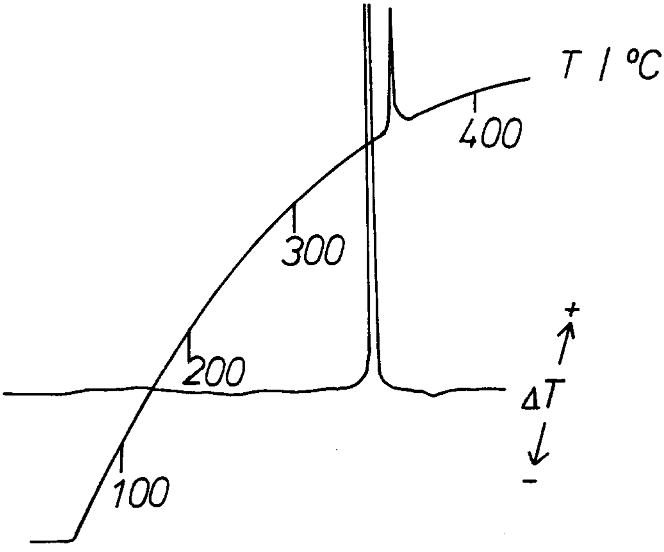

Fig. 2. DTA curve of BaO + $CuSO_4$.

The DTA curve of "tin foil" shows one endothermic peak at the melting point (see Fig. 3). The temperature from ca. 320 ^{0}C shows that the metals is not tin but rather lead. A parallel investigation by atom absorption showed more then 98% lead.

More than a dozen different capsules from wine bottles from Germany, France, Italy and Spain never showed tin but always lead.

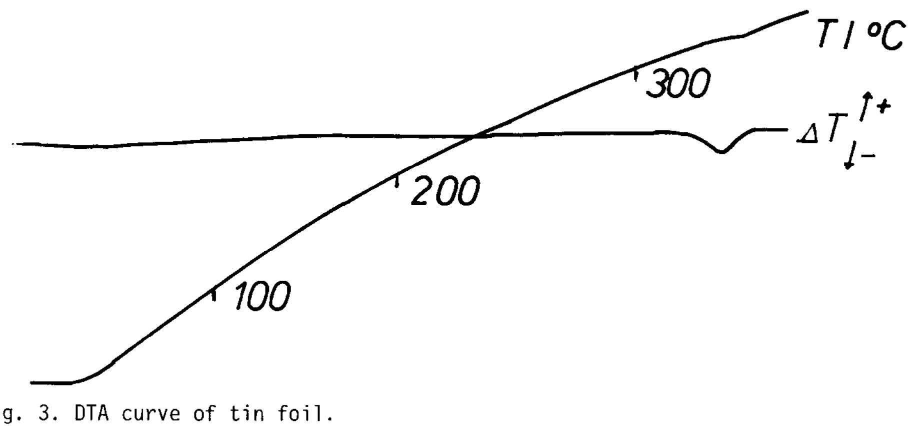

Fig. 3. DTA curve of tin foil.

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