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THE USE OF COMPLEX TRIPLE THERMOPAIR IN THERMOANALITICAL PRACTICE

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ABSTRACT

Use of complex thermopair consisting of 3 thermoelements has promoted the raise of productivity of a thermoanalitical plant, as well as of precision increase of comparing thermal effects. This gives the possibility of simultaneous analysis of 2 models in different conditions, pressures, gas tamosphere, mixing with other components etc.

INTRODUCTION

Usually complex thermopair consisting of 2 individual thermopairs joined with each other according to adopted method is used in the process of differential thermal analysis, which allows to get thermal curves only for one analysing matter.

Separate complex thermopairs for each analised matter were, used with the aim of increase of a number of simultaneously analysing substances [3]. In such a combined way there may be shifting in thermal effects' temperature conditioned by different use of separate standards for each complex thermopair and, subsequently, unequal junture disposition of thermopair which measures the temperature. So, it's difficult to avoid errors characteristic of a separate complex thermopair. These factors may misrepresent the received results or may put under doubt their

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trustworthiness. The problem of 2 simultaneously comparing substances under analysis is settled, to our opinion, by using of complex thermopair consisting of 3 thermoelements (1, 2, 3) as on Fig. 1.

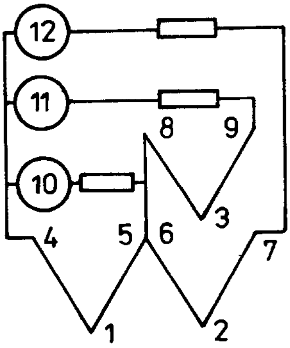

Fig. 1

Combining is realized by joining together of free ends (5, 6, 7) (Fig.1) of the same name thermoelectrodes of all the three identical thermopairs (1-3). Then, according to recommended methodics [1, 2] thermostatized cold ends (4-9) are joined to the thermals of galvanometer or many-channelled register apparatus.

In the suggested variant of complex thermopair the first one measures the temperature, which is recorded with the help of galvanometer 10; second and the third - make up separate differential thermocurrents of which are measured by the galvanometers 11, 12. According to the suggested method, thermopair joint which measures the temperature, should be places into the standard substance, the latter stays unchanged in different thermal transformations, while the other two of differential thermopairs should be placed into analysed substances. Though such a method is somehow different from an accepted one, but the experiments show some definite advantages of our above mentioned method. This method allows to reveal slightest differentiations in thermal effects´ temperature of one and the same substances differing with each other by synthesis conditions.

RESULTS AND DISCUSSION

Record of thermal curves when using complex thermopair was realized on photoregistration mechanism using chromel-alumel thermopairs. Heating velocity was 7^{o}/min.

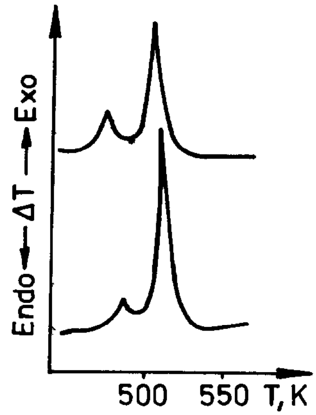

Fig. 2

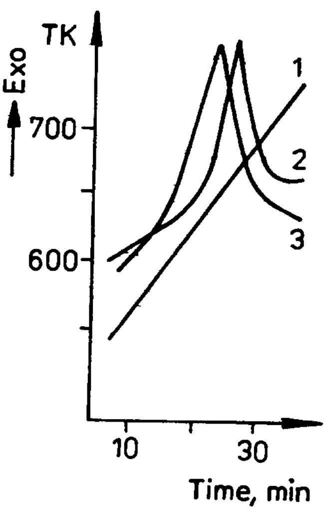

Fig. 3

For better reproduction of thermal curves the construction of the block is modernized. It s made of several individual discs of stainless steel with corresponding number of holes, centred thermopairs for the standard, the substance under analysis and the concretly taken substance in case of programmed heating. Fig. 2 shows the differential curves of complex combinations of palladium of one and the same composition obtained by different values of "pH", where one can clearly see shifting of thermal effects´ temperatures and also the quantity of peaks´ correlations. Fig. 3 shows thermal curves $T,(OH)_4$ obtained by $TiCL_4$ hydrolysis, recorded in condition of atmospheric pressure (curve 3) and 12 mm of mercury (curve 3), where temperature increase of exothermal effect of crystallization of amorphous dehydrotation product with drop in pressure is noted.

Observed differences in thermal effects´ temperature will be unnoticed

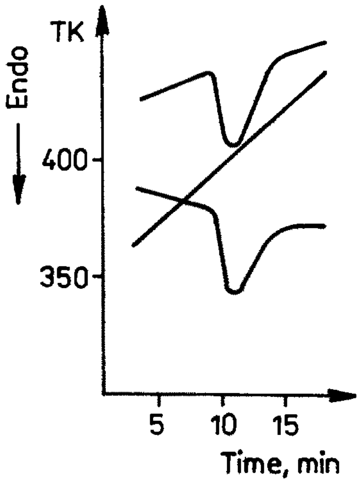

Fig. 4

in case of usual analysis methods the very differences may be regarded as errors of experiment as each substance is analysed by separate complex thermopair and this is followed by natural errors in temperature measurements : each complex thermopair has its own gradation.

According to this method, accurateness of temperature reproduction by both of differential thermopairs is evidently seen from the temperature coincidence of extreme points of differential curves of polymorphic transformation of KNO_3 as it's seen on Fig. 4. This gives way to confidence in objectivity of indices and temperature values of each differential curve taken separately.

CONCLUSIONS

Combined of 3 thermoelements thermopair affords : twice increase productivity of the plant; to limit the number of simultaneous use of thermopairs (three instead of fout); to improve compactness of the plant by using one standard substance instead of two; further quantity increase of simultaneous analysis is possible if subsequent number of additional differential thermopairs is increased.

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