Application News

SHIMADZU

THERMAL ANALYSIS T104

Evaluation of Dangerous Materials Using Self-Reactivity Evaluation Program

The dangerous objects defined as the Class 5 Material in the fire prevention law must be handled, stored and transported in accordance with standards stipulated in the law , as they are self-reactive substances that may cause combustion or explosion by exothermic decomposition without air.
The fire prevention law specifies two methods for determination whether a substance belongs to the Class 5 Material or not. The two are (1) thermal analysis method and (2) pressure vessel test method.
This report introduces a self-reactivity evaluation program for dangerous objects, using the thermal analysis method.
The fire prevention law specifies the testing procedure (thermal analysis) as described below for determining dangerous objects.

1) Measuring method
   A specimen shall be tested in a sealed pressure cell made of stainless steel with a differential thermal analyzer (DTA) or a differential scanning calorimeter (DSC) at a heating rate of 10˚C/min.
2) Method for analyzing and evaluating test results
   (1) Determine calorific value and its onset temperature of a decomposition reaction using benzoyl peroxide (BPO) and 2, 4-dinitrotoluene (DNT) as the reference samples.
   (2) Plot the logarithm of the calorific value multiplied by a factor of 0.8 for BPO or by 0.7 for DNT along the vertical axis, plot the logarithms of the onset temperature deducted by a factor of 25˚C along the horizontal axis, and finally draw a straight line through the two plotted points.
   (3) Measure a test specimen in the same way. If the curve of test specimen lies on or above the plotted line for BPO and DNT, the specimen is deemed a dangerous object.

The self-reactivity evaluation program automatically determines calorific value and its onset temperature if two points including exothermic peak point are designated on the obtained DSC (or DTA) curve, and it automatically produces a critical line on the basis of several reference values if specified for averaging purpose.
In addition, this program graphically indicates the status of a tested specimen with a mark visually showing its measured value against the critical line if the name of specimen is given.

Measurement of standard sample

Fig.1 shows a DSC curve of BPO, which is one of standard samples. If the two points A (for onset point of calorific peak) and B (for its final point) are designated, the onset temperature (108.8˚C) of exothermic peak and the calorific values (1306.43J/g) are automatically determined stored in the disk for producing the boundary line for qualification.
Fig.2 shows a DSC curve of DNT, another standard sample, in which, just as with BPO, the onset temperature (320.4˚C) of exothermic peak and calorific values (3403.17J/g) can be determined automatically if the two points are specified.

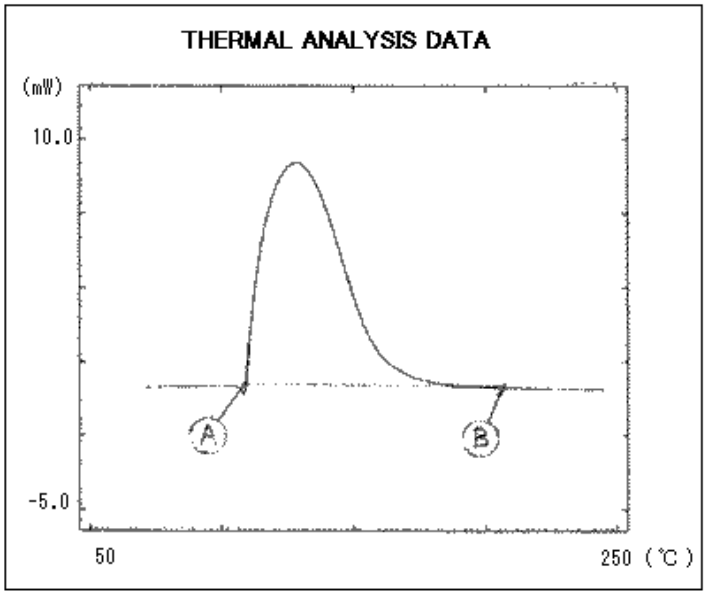

Fig.1 Onset Temperature and Calorific Value of Exothermic Peak of BPO

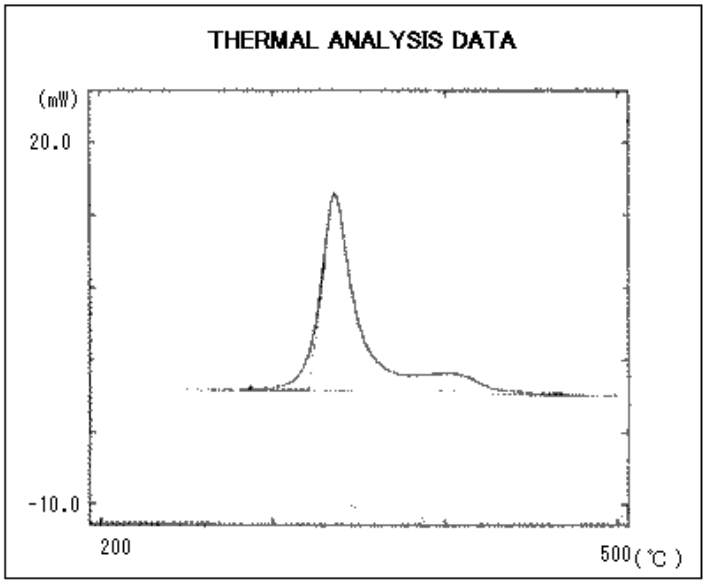

Fig.2 Onset Temperature and Calorific Value of Exothermic Peak of DNT

Critical line

Fig 3 shows a critical line using standard samples of BPO and DNT. A maximum of ten standard samples can be averaged for plotting.

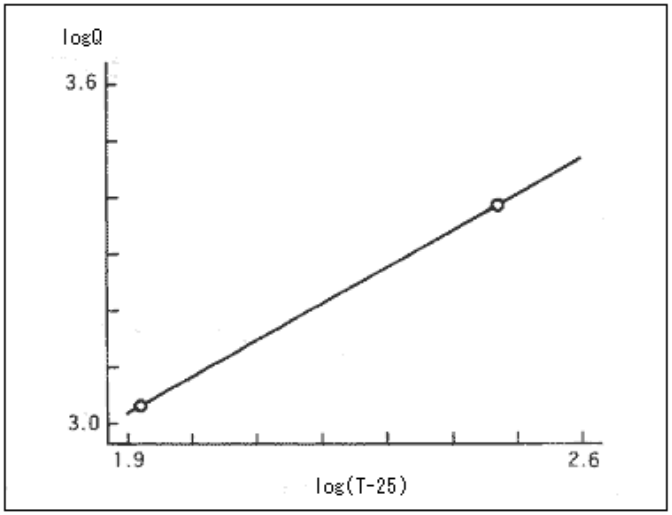

Fig.3 Critical Line

Evaluation of test results of dangerous objects

Fig.4 through Fig.7 are examples of evaluation of dangerous objects. The measured value of the tested specimen is shown with a mark "D" respectively. In Fig.4 through Fig.6, the D marks lie above the critical line, which means that all of these specimens belong to the Class 5 Material.
The data of dinitrodiphenyl sulphone, which is not considered a dangerous object so far in the criteria of thermal analysis as the mark D lies below the critical line, must clear another condition of pressure vessel test before final decision.

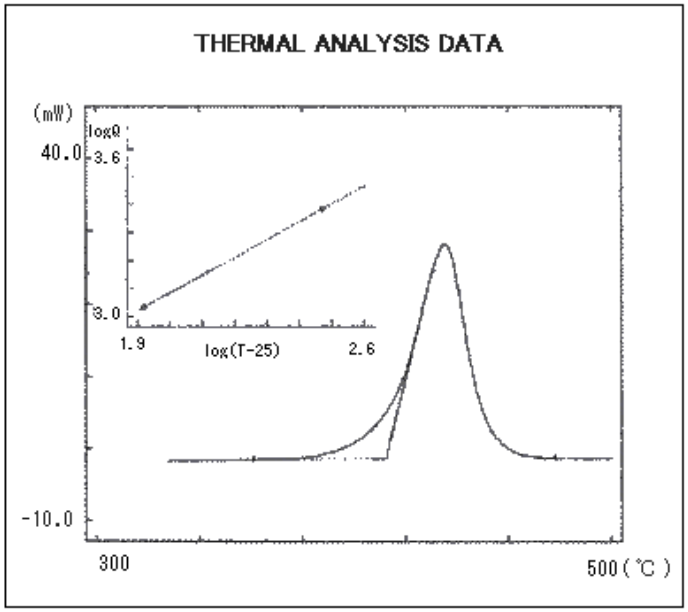

Fig.4 m-Dinitobenzene

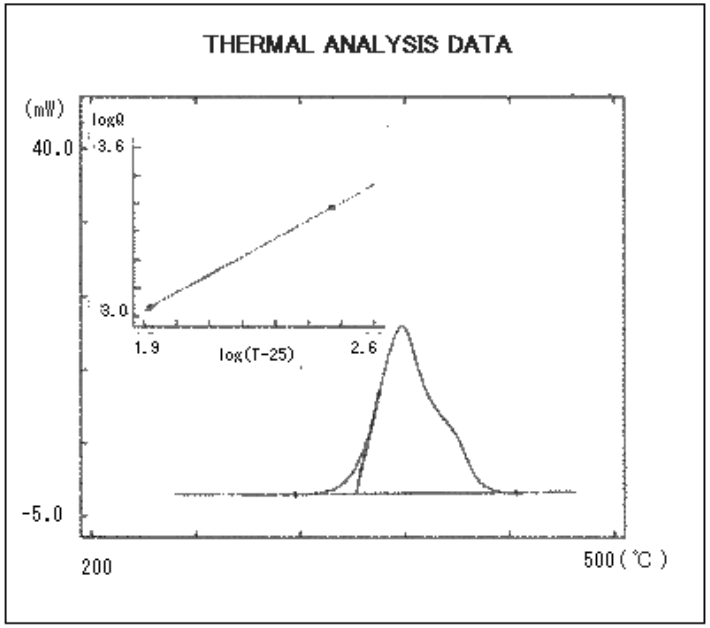

Fig.5 2, 4-Dinitroaniline

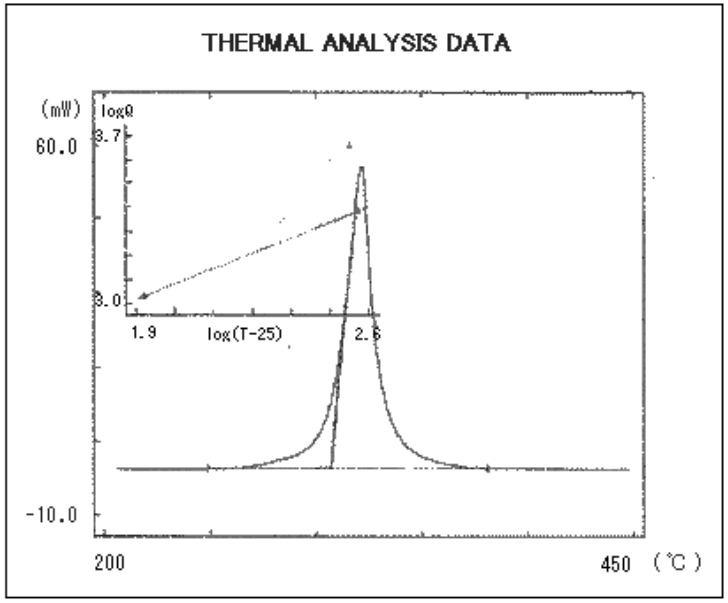

Fig.6 2, 4, 6-Trinitrophenol (picric acids)

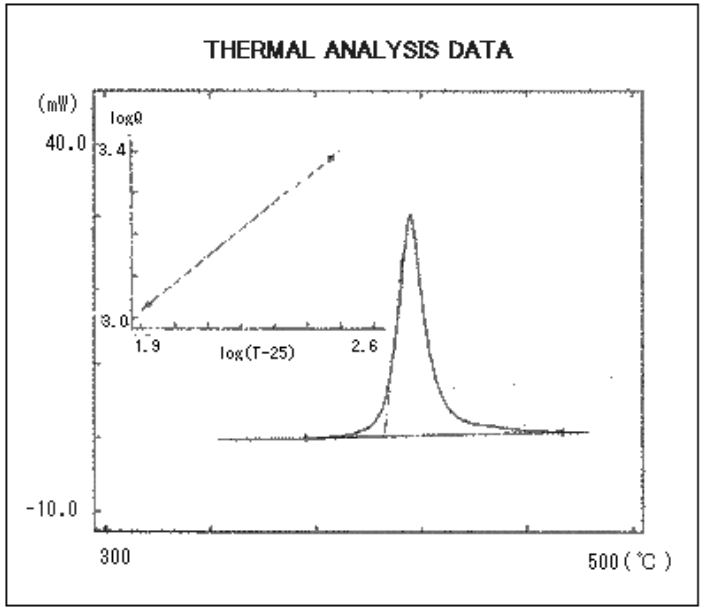

Fig.7 3, 3-Dinitrodiphenyl Sulfone

* Please be advised that data obtained before the implementation of the current Weights and Measures Law may be presented in terms of gravimetric unit.

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