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Technique and Applications of High Pressure Differential Thermal Analysis

Study of Reactions of Dinitrotoluene and Tolylenediamine

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▶ **A technique of high pressure–high temperature differential thermal analysis has been developed. By utilizing a suitably designed sample container, temperature and pressure changes can be measured up to 3000 p.s.i.g. at 500° C. This procedure can be adapted to commercially available instrumentation and allows the safe study of dangerous reactions that present safety hazards on a macro scale. The application of this technique to the study of dinitrotoluene reactions has permitted the determination of the thermal stability of dinitrotoluene, reduction temperature of dinitrotoluene with Raney nickel, decomposition reactions of dinitrotoluene with tolylenediamine, and parameters affecting the violent decomposition of reduction mixtures.**

DIFFERENTIAL thermal analysis has been utilized in many applications to study inorganic (*4*, *7*, *22*) and organic reactions (*5*, *15*). This is in addition to the extensive use for characterization (*14*) and identification of many substances (*1*, *10*).

In spite of the usefulness of the DTA technique, relatively few applications have utilized high pressures. The types of apparatus that have appeared are limited in the usable pressure range (*13*, *23*, *25*). This sparsity of reporting could be due in part to limited applications and the highly specialized and limited equipment.

Bohon has described an apparatus specifically built for work with rocket propellants and capable of operating at several hundred pounds per square inch gauge (*2*). The main application of this apparatus has been for the study of heats of explosion of propellants (*3*).

Lodding and Hammell have utilized their apparatus to study reactions and phase changes of iron hydroxide and oxides up to 400 p.s.i.g. Provision has also been made to analyze the effluent vapors (*12*).

The present technique utilizes a suitably designed sample container, to achieve pressures up to 3000 p.s.i.g. at 500° C. This sample container is readily adaptable to many commercially available instruments and this technique can be applied to the study of organic reactions and transitions such as decomposition, melting, sublimation, coupling, and reduction. Provision has also been made to measure any sudden pressure surges up to 3000 p.s.i.g. as well as to transfer effluent vapors into a gas chromatograph.

We wished to show the utility of this technique for the safe study of the hazards of high pressure–high rate reactions under strenuous conditions of temperature and pressure that would be cumbersome, impractical, and dangerous to perform by methods other than DTA.

Since nitro compounds are of practical interest to the urethane industry (*18*), and they represent a potential safety hazard, we chose to investigate the thermal stability and reduction of dinitrotoluene in an attempt to define some of the hazardous parameters associated with these reactions.

In a previous, excellent, macro study by Havekoss (*9*) which inevitably suffered some of the limitations listed above, indications were obtained that dinitrotoluene did react exothermically with tolylenediamine. We wished to investigate this reaction, among the others mentioned above, utilizing the advantages of the DTA technique.