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The discontinuous vacuum stability test (DVST)

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GAS EVOLUTION ANALYSIS

I. THE DISCONTINUOUS VACUUM STABILITY TEST (DVST)

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ABSTRACT

A method consisting of a modified mercury-free vacuum stability test used in conjunction with gas chromatography and FT-IR spectroscopy has been developed in order to perform a complete gas evolution analysis. This method whose reliability is easily controlled, has the potential of simplicity, low cost of equipment and good accuracy.

INTRODUCTION

Although the vacuum stability technique has been in use for a long time and has provided a cheap and reliable test, it does have a number of disadvantages: toxicity due to mercury use; inaccuracy due to corrections and condensation during the aging period, and time and labour intensiveness. Alternative methods where the mercury

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manometer is replaced by a pressure transducer (PT) resulted in a cleaner and faster vacuum stability test (VST)[1,2] but did not necessarily give more accurate determination. (The mercury method is still used as a reference for accuracy). A permanent pressure detection with a PT, in the alternative VST, has several drawbacks: 1) the corrosive gases may alter the PT response, 2) a PT is required for each test tube and makes the method more expensive; 3) the connection between the test tube and the PT may be a source of leaks in long term testing. To overcome these problems, we developed a new method, where the sample is aged in a simple test tube. The PT is connected to the test tube only for the time required to measure the pressure. In this semi-continuous VST, only one pressure transducer is required regardless of the number of test tubes.

In addition, evolved gas analysis may be performed by gas chromatography and FT-IR spectroscopy in conjunction with the discontinuous vacuum stability test (DVST).

Description of the discontinuous method

The apparatus, shown in figure 1, consists of a glass tube equipped with a valve, a septum, and a pressure transducer (PT). The sample is aged in the vacuum tube, which is connected to the PT only for

pressure measurements. The free gaseous volume of the PT is evacuated before coupling. Unlike the mercury method and the alternative methods which provide continuous pressure measurements, this method provides discontinuous pressure measurements (figure 2).

This procedure has several advantages. First, only one pressure transducer is required regardless of the number of test tubes. Second, the PT remains free of any gas condensation. Third, the procedure provides absolute results whose accuracy is easily verifiable. Fourth, such a system minimizes leaks. And finally, this is an ideal method for a long-term stability test at low temperature; the contents of the stand-alone test tube may be aged for a long period without leaks and without immobilizing a PT.

Volume determination

Pressure may be converted into volume of gas either by means of a calibration curve[3] as illustrated in figure 3, or using equation 1:

$$V = V_f(P/P_{atm}) \quad (1)$$

with V_f = tube volume + dead volume - sample volume, V is the volume of gas evolved, V_f is the free

volume and P is the pressure measured by the transducer. This equation is more advantageous than the calibration curve because it is faster and provides absolute results. Since the tube and the PT are at the same room temperature when the pressure is measured, there is no need for a temperature correction. The only requirement is an accurate determination of the free volume.

Reliability

The reliability of the instrument, validity of equation 1, and accuracy of the PT are easily verified and controlled by injecting a known amount of gas into the vacuum tube. At the end of the test, the volume measured is equal to the volume of gas injected. Results obtained with equation 1 and those obtained from the calibration curve, table 1, are in very good agreement.

TABLE 1. Verification of equation 1 ($V=V_f(P/P_{atm})$) and comparison with the calibration curve. V_f=61.5 ml., P_{atm}=753.6 torr.

Amount of gas injected (ml)	Pressure (P) (torr)	Volume of gas (ml) determined with	
		Equation 1	Calibration curve
2.0	25.60	2.09	2.09
4.0	50.00	4.08	4.04
6.0	74.50	6.08	6.01
8.0	98.50	8.04	7.95
10.0	122.40	9.99	9.87

Comparison of the results obtained by the new and traditional methods

Various samples of energetic materials (liquids, solids and mixtures) were run by both the traditional method and the new technique. The results obtained by both methods are comparable. However, the mercury stability results (table 2) tend to be slightly higher than those obtained by the discontinuous stability test.

TABLE 2. Comparison of the new VST results to those obtained by the conventional technique. Volume evolved at 100°C for two days.

Samples	Mercury method	New method
GAP	0.32	0.29
DEGDN	0.39	0.40
TEGDN	0.35	0.30
GBP-082	2.81	1.96
AN/BDNPAF	4.90	4.24
Composite B	0.10	0.19
Propellant 2800	0.36	0.31

Evolved gas analysis

Determination of the composition of the evolved gas may be performed by gas chromatography and FT-IR spectroscopy in conjunction with the vacuum stability

test. A programmable gas chromatograph (HP 5890 with a thermal conductivity detector) equipped with a Carbosieve SII column is used to separate and identify the different gases. A typical chromatogram is shown in figure 4. The species O_2, N_2, CO, CO_2, and N_2O were well separated and identified. Nitrogen dioxide, however, was not found although it is considered a decomposition product. Since NO_2 has been identified by infra-red spectroscopy, its absence from the chromatograms is probably due to reaction with the chromatographic column packing.

A Michelson 100 FT-IR spectrometer (Bomem) was also used for gas analysis. A typical FT-IR spectrum of the gas evolved from aged TMETN is shown in figure 5, where gases such as CO, CO_2, N_2O, and NO_2 were identified. As nitrogen oxides are easily detected, Ft-IR spectroscopy appears to be an excellent complementary technique to the GC. It is also useful in the identification of the unknown peaks obtained by the GC.

Conclusions

This new concept of VST has several advantages. It respects the environment; has better reliability, precision, and lower cost; and is less labour-intensive than the traditional test. It also permits gas sampling for GC or FT-IR analysis for determination of gas

composition. The reliability of the discontinuous VST is easily controlled by injecting a known amount of gas into the vacuum tube. At the end of the test, the volume measured is equal to the volume of gas injected.

ACKNOWLEDGEMENTS

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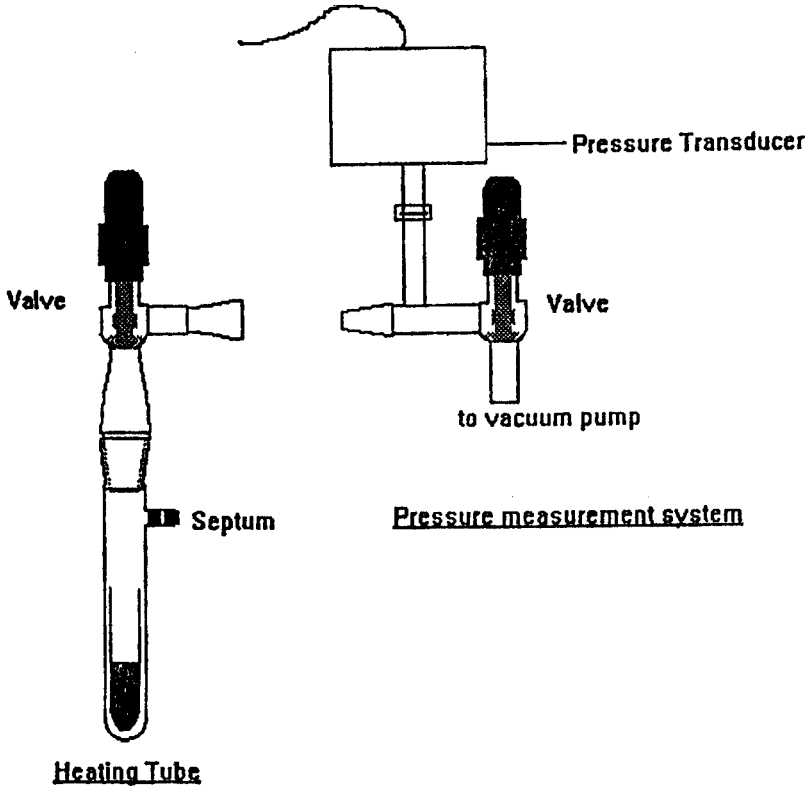

FIGURE 1
Discontinuous stability test apparatus.

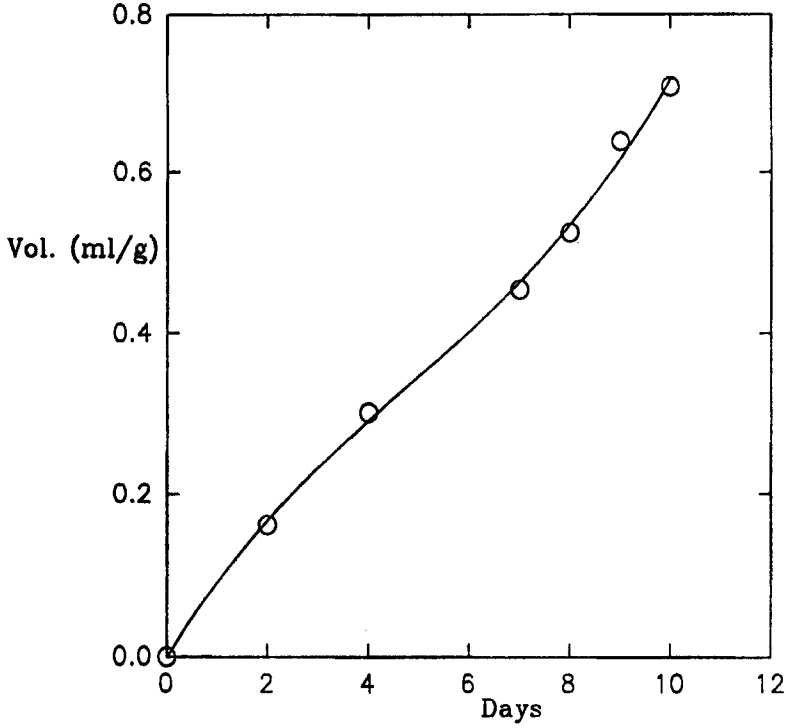

FIGURE 2
Gas evolution as a function of time for GAP based propellant at 100°C.

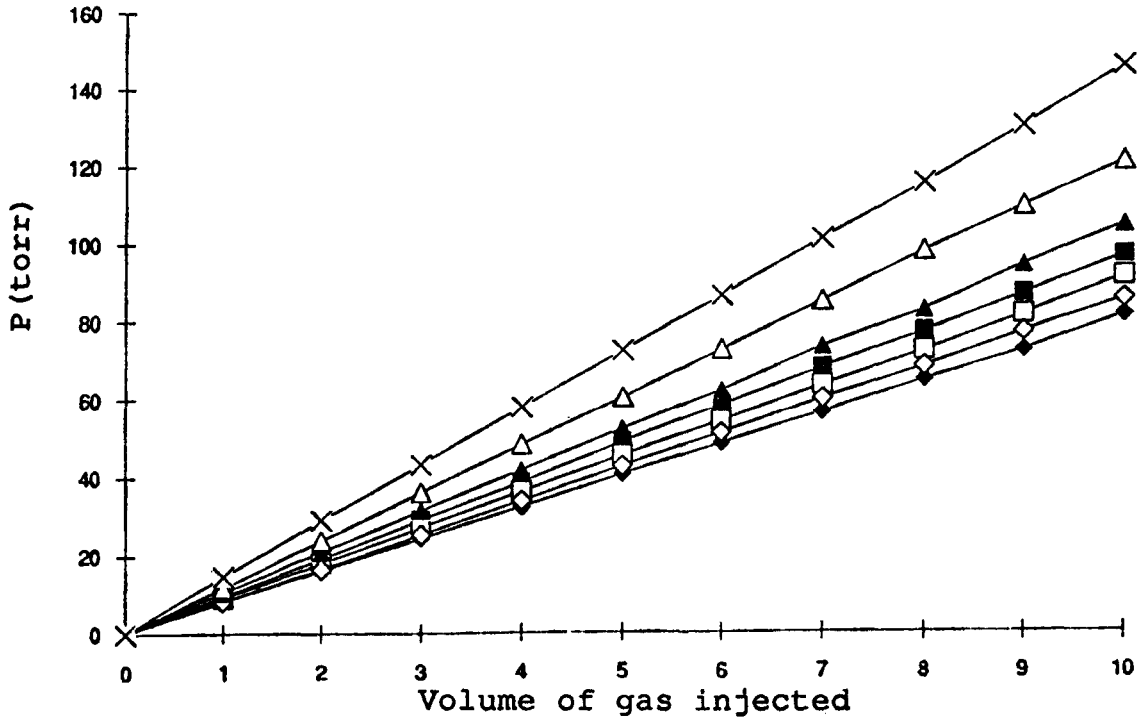

FIGURE 3
Calibration curves: Pressure as a function of volume for different tubes.

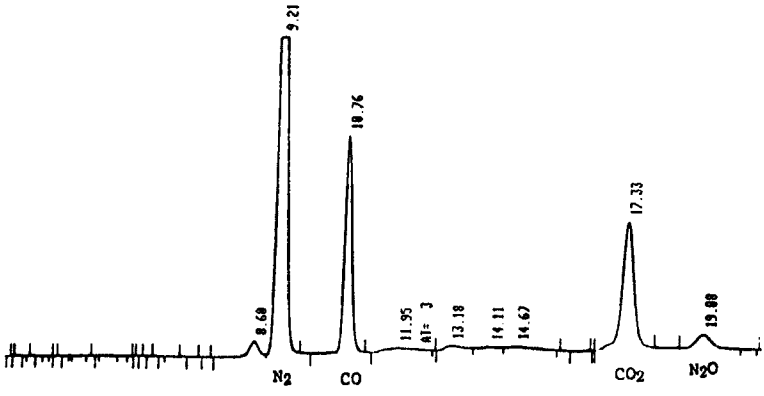

FIGURE 4
Typical chromatogram of evolved gases from TMETN after 2 days at 100°C.

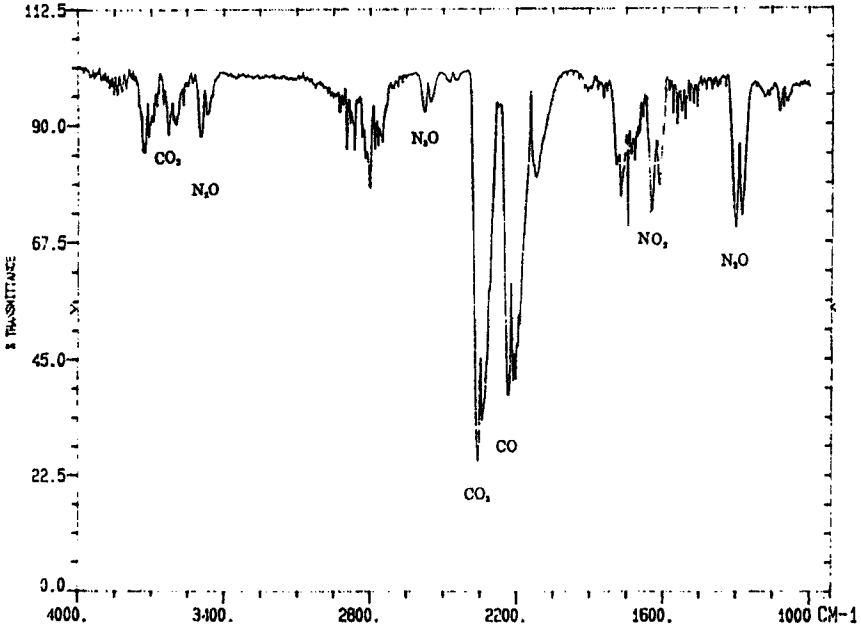

FIGURE 5
Typical FT-IR spectrum of evolved gases from TMETN after 5 days at 100°C.