

## DEVELOPMENTS IN VACUUM STABILITY TESTING - THE INCLUSION OF A DATA LOGGER

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### ABSTRACT

A description is given of the design and performance of the AWRE vacuum stability apparatus incorporating a transducer, data logger and teleprinter for producing a direct print-out of the volume of gas produced on heating an explosive or a compatibility test mix. Reference is made to the applicability of this apparatus to the proposed NATO compatibility test.

### 1. INTRODUCTION

This paper reports on the final stage of the development work in designing and testing a new apparatus for performing vacuum stability type tests to determine the stability of explosives or the compatibility of explosives with other materials. The test requires an estimate of the volume of gas evolved when a test sample is treated under specified conditions. The new apparatus updates the hardware used to do this so as to take advantage of modern technology.

The previous stages in the development programme have been published<sup>(1)(2)</sup>. An intermediate stage in updating the technology for the measurement of the gas consisted of replacing the mercury-in-glass manometer by a pressure transducer. This produces a millivolt output which is directly proportional to the gas pressure. The output was measured with a chart recorder<sup>(1)</sup>. In the same paper a description of a supplementary procedure was published which utilised a high vacuum apparatus to separate the non-condensable gas from products which are condensable (at -80°C). The gas was then sampled and the condensibles were collected and

weighed. Each could then be analysed. In the second paper<sup>(2)</sup> a review of existing and potential techniques for compatibility testing was given and it was argued that the vacuum stability approach was still the most sensitive and viable for making an assessment of the early stages of chemical reaction between a high explosive and an adulterant. For this reason further development work on updating the technique for gas assessment was considered to be worthwhile. This paper also quoted some results which demonstrated the value of the supplementary procedure in making a separate estimate of the condensable and non-condensable products. This work, in which the aim was to make the supplementary procedure quantitative, was complete at the time of this publication. The further work required was that concerning the transducerised approach for the gas measurement and this required the addition of a data logger and teleprinter. It is primarily this development work which is reported in this paper.

In the new technique a print-out of the volume of gas (in cm<sup>3</sup> corrected to STP) is obtained and no calculations are required as in the conventional manometer method. The calibration procedure to

enable this direct print-out of volume is simple. The method gives the same values for the gas volume as does the conventional method and it can therefore be used as a direct replacement for the conventional apparatus in performing compatibility tests to the AWRE test specification. It could be used as an apparatus for the compatibility procedure currently being standardised by NATO. It has been accepted in principle by all UK government establishments where the vacuum stability test is performed and is currently in various stages of commissioning with modifications according to local requirements. The AWRE version is versatile in the sense that the calibration technique, on which the direct read-out in true gas volume depends, can be applied to virtually any size or shape of apparatus. The method is more sensitive than the conventional one and is not subject to the errors intrinsic in manual readings. It offers potential for a test at lower and more realistic temperatures.

## 2. THE NEW APPARATUS FOR GAS VOLUME ASSESSMENT

### 2.1 THE COMPLETE ASSEMBLY

The test mixture is contained in a glass reaction tube which is immersed for all its effective length in a heating bath and which is connected, via a glass adapter and a glass connector, to a pressure transducer (Figure 1). The voltage output from the transducer is fed to a data logger which prints out on a teleprinter. Each channel of the logger is reserved and calibrated for a particular complete assembly. The individual parts of the complete assembly will now be described separately.

### 2.2 GLASSWARE

The general design of the glassware is as previously published<sup>(1)</sup> but the dimensions have been altered so that the total volume more closely conforms to that of the UK/AWRE conventional manometer apparatus. The glassware with transducer is shown in Figure 1.

The reaction tube. This is identical to that used in the conventional apparatus (Figure 2). It connects to the adapter with a ground glass joint which is greased with Dow Corning stopcock grease. It is provided with a cup to take a mercury seal which is necessary because this joint experiences a temperature differential.

The adapter. This carries the transducer vertically above the reaction tube and joins to a connector with a ground glass joint which is greased with the stopcock grease. It has a side arm with a vacuum tap, for evacuating the apparatus and for the introduction of air for calibration (Figure 3).

The connector. This joins the adapter to the transducer by a tube of outside diameter 5 mm which fits into the cone-shaped entry port of the transducer (Figure 4). Sealing with the transducer is produced by a brass nut, a brass olive, a "large" O ring and then two "small" O rings which are placed on the 5 mm tube in that order. Pressure is applied by the brass nut which is threaded to suit the thread on the outside of the transducer. The seal is provided by the last of the "small" O rings against the cone-shaped interior of the entry port. The second "small" O ring is a spacer to ensure that the "large" O ring is wholly outside and not in contact with the end of the transducer since this would produce an unintended but ineffective seal. This sealing system when properly assembled is completely effective and could be adapted to other makes of transducer provided that they have a cone-shaped entry port and an external thread. The only item in this sealing system which is in contact with the products of reaction during a test is the last of the two "small" O rings to be placed on the connector tube. It is made from silicone rubber to DTD 818 and has been shown to have no reactivity with a wide range of explosives. The detail of the O rings which suit the AWRE design of connector and the transducer specified are as follows:

Large O ring. Butadiene-acrylonitrile rubber of internal diameter 0.187 in (4.75 mm) and cross-section 0.103 in (2.62 mm).

Small O rings. Silicone rubber of internal diameter 0.176 in (4.47 mm) and cross-section 0.070 in (1.78 mm). The 4.47 mm internal diameter of this gives a good fit around the 5 mm outside diameter of the glass tubing of the connector.

The volume. The volume of the apparatus was designed to be around 24 cm<sup>3</sup> which is the mean of the range specified for the mercury manometer apparatus, namely 23 to 25 cm<sup>3</sup>. The range of this latter arises from the movement of the mercury column. The tolerances for the glass tubing from which the glassware is made, are half those quoted in the manufacturer's catalogue and the glassblowers were requested to use selected tubing. There will be a small variation around the nominal volume of 24 cm<sup>3</sup> as a result of the tolerances and also because of the necessity for hand glassblowing. The reaction tube has a volume of 15 cm<sup>3</sup> to the bottom of the ground glass joint and this is the same as that used in the conventional apparatus.

The design. The geometry of the glassware was chosen to provide a good design of high mechanical strength. It was also required to be as similar as possible to the conventional apparatus, consistent with accepting a transducer and having a port for evacuation and for air injection. The similarity to the conventional apparatus was achieved by keeping the reaction tube and the total volume of the apparatus unchanged. Hence, the same proportion of the apparatus is inside and outside the heating bath. This ensures that the temperature distribution during a test approximates to that in a conventional apparatus. If a condensible product is produced, which is gaseous at the test temperature and liquid at the ambient temperature within the safety cabinet, the product should experience a similar treatment in both designs of apparatus. If any of the volatile products are also catalytic, their effect on the volume of gas produced should again be similar in both designs.

## 2.3 HEATING BATH

The heating bath is that used for the conventional test and accepts ten reaction tubes which are wholly immersed to the bottom of the ground glass joint (Figure 5). The bath contains an electrically heated metal block which is well lagged on all sides, including the top. Electrical controls of large bulk must not intrude into the lagging so as to create uneven heat distribution.

Temperature controller. This should be a proportional controller and the minimum standard should be  $\pm 0.5^\circ\text{C}$  which is that proposed by the NATO committee which is currently endeavouring to standardise the vacuum stability type compatibility test.

Temperature measurement. The sensor should be sited within a replica of a typical test mix in a standard reaction tube within one of the holes of the heating bath. A platinum resistance thermometer immersed in sand, with read-out on a chart recorder, is used at AWRE. Exposed metal of the casing and sheath of any temperature sensing device must be thermally insulated so as to minimise cooling of the sensor by conduction. A standardised mercury-in-glass thermometer similarly immersed in a replica of a test mix and placed in an adjacent hole is a good check on the installation of the recording thermometer.

Temperature cut-out. A cut-out device, overriding the temperature controller, is included. It is set at a higher temperature than the test temperature. The device should be readily accessible and easy to re-set when the test temperature is changed.

## 2.4 TRANSDUCERS

Main characteristics. The transducers are manufactured by Bell and Howell Ltd. Pressure applied to the diaphragm is converted to a controlled strain in an excited Wheatstone bridge circuit of strain gauge windings. The construction

is such as to ensure that the voltage output is a linear function of the pressure applied to the diaphragm. The end of the transducer has two features which allow an effective seal to the glass connector. The outside is threaded and the inside, which constitutes the entry port to the diaphragm, is conical. The main characteristics of the transducers are as follows:

Type 4-366-002 with  $\frac{1}{2}$  in BSP 60° female cone; Pressure range 0 to 1 bar A; Rated excitation 10 V dc; Full range output 40 mV (+ 20% - 10%) open-circuit at rated excitation and 25°C; Residual unbalance  $\pm 5\%$  FRO at zero pressure and 25°C; Non-linearity and hysteresis combined shall not exceed  $\pm 0.5\%$  FRO; Pressure limits twice the rated pressure when applied for 3 min shall not cause a zero set to exceed 0.5% FRO; Pressure media 17-4 PH and 15-7 Mo stainless steel; Case all welded stainless steel AMCO 17-4 PH.

The transducers are normal production quality and are not "rationalised" to a standard output. In order to cover the variations in output from different transducers for a given pressure, the "RANGE" control of the data logger when operating in the "ENGINEERING" mode has been made wide. This provides for some economy in the purchase of transducers when a large number are required.

Overpressure. The transducers exhibit no change in output characteristics for pressures on the diaphragm up to  $1\frac{1}{2}$  atm. This is the maximum pressure likely to be encountered with the gas evolution apparatus as designed (see 2.6). Pressures between  $1\frac{1}{2}$  and 2 atm would have an effect on the zero offset but not on the FRO. The diaphragm is very vulnerable to misuse of mechanical pressure. It must not be cleaned with a brush or a swab or any such device. Cleaning should be by the use of an ultrasonic cleaning bath with a suitable fluid. For the same reason the connector tube which joins the transducer to the adapter must not be allowed to impinge on the diaphragm. The length of the connector tube is designed so that when used with the rubber O ring

seal this cannot occur. Additionally, these transducers have a restriction near the junction of the entry port and the diaphragm cavity of diameter slightly less than that of the connector tube.

Chemical integrity. Those parts of the transducers which come into contact with volatile products, namely, the diaphragm cavity and part of the entry port, are made from stainless steel. Many varieties of stainless steel have been tested for compatibility with explosives using the conventional all glass apparatus and there has never been any evidence of chemical reaction with any of the explosives used. These covered nitroaromatic, nitramine and nitric ester type secondary explosives. The tests were performed by the standard procedure in which the steel, in the form of swarf, was mixed with the powdered explosive and heated for 40 hours at 100°C or 120°C. This is a rigorous test and complements the further evidence from using the transducers in compatibility tests during the development programme that no corrosion has been seen on the inside of the diaphragm cavity.

Connection to the logger. Each transducer is fitted with a multi-point plug. It accepts the 10 V supply from the logger to energise the transducer bridge and transmits the millivolt output from the transducer to the logger.

## 2.5 LOGGER AND PRINTER

The main parameters of the data logger are quoted here. These indicate some of the main areas of choice which are of interest to the user and are dependent on the applications to which the logger is to be put. The AWRE logger is dual purpose. It is designed primarily for use with the standard test and also for other work in which apparatus of different volumes may be required.

Channels. 60.

Sensitivity. 0.01 mV - this is equivalent to less than 0.01 cm<sup>3</sup> of gas produced in the standardised 24 cm<sup>3</sup> apparatus.

Stability.  $\pm 0.01$  mV.

mV range. To 99.99 mV.

Read-out modes. The logger will display in mV or in "engineering units". The latter is used for direct read-out in  $\text{cm}^3$  of gas when used with a suitable calibration technique. The mV mode is fundamental and is useful for a variety of purposes, including setting up the system, calibration purposes and non routine experimental procedures.

Zero and range controls. Both read-out modes have independent zero and range controls. The range control in the engineering units mode is very wide to enable the economy previously referred to. It also facilitates flexibility in calibrating apparatus of differing volumes by the introduction of a standard volume of air rather than carefully adjusting the volume to suit the logger.

Clock. Real time is printed for each scan.

Interval timers. Two independent interval timers with a cross-over switch are provided. They can be set for regular time intervals.

Skip facilities. Skipping of channels is provided for in groups of ten, corresponding to ten test holes in the heating baths. The channels can be used independently in groups of ten in the mV or the engineering units mode.

Printer. 390 Data Dynamics teleprinter.

10 V supply. The transducers require a 10 V supply to energise the bridge circuit. This supply must be constant since the mV output is determined by it. Ideally, the value would be printed for each scan. In the AWRE logger a resistor is incorporated between the input and output terminals on a multi-point plug which can be inserted in one of the channels. The resistor reduces the 10 V supply to 10 mV which reads out on the logger.

## 2.6 BLOW OFF PRESSURE

The conventional mercury manometer vents to atmosphere automatically when the pressure reaches a little above atmospheric. It therefore acts as a safety valve. It was necessary to know at what pressure the new apparatus would "blow off", whether this pressure was too high for explosive safety and whether it was below the limit which would affect the transducer output characteristics. Six different pieces of apparatus were tested. In four of these the apparatus, full of air at ambient pressure, was heated until the pressure (as shown by the mV output to the logger) abruptly dropped back to atmospheric pressure. In two an incompatible mixture was placed in the apparatus which was then evacuated and heated to  $120^\circ\text{C}$  until it vented to atmosphere.

<u>Test</u>	<u>Blow off pressure (ambient atmospheres) <math>\equiv</math> approx. kPa <math>\times 10^{-2}</math></u>
Apparatus originally at atmospheric pressure	1.3 1.5 1.5 1.3
Apparatus evacuated and containing an incompatible mix	1.3 1.3

The apparatus always vented at the joint between the reaction tube and the adapter. These results, though few in number, confirm the author's observation from many unrecorded results obtained when using the original version of the test with a chart recorder that "blow off" always occurred at or below about  $1\frac{1}{2}$  atmospheres. They give sufficient confidence to cover explosive safety and protection of the transducer.

## 2.7 CALIBRATION

### 2.7.1 Principle

The principle involved in the calibration procedure is first to introduce into the evacuated apparatus (containing a solid filler of volume equal to that of the sample to be tested) a known volume of air

at ambient conditions and then place the apparatus in the heating bath for sufficient time for it to attain a stable thermal equilibrium. The RANGE CONTROL of the logger for the selected channel is then adjusted to a volume equal to that of the volume of air introduced (adjusted to standard temperature and pressure). When the apparatus is subsequently used in a stability test, the logger will register true gas volumes.

### 2.7.2 Methods of Introducing Air

The application of the classical method for this would be to measure the pressure of the ambient atmosphere and then to take a bulb of known volume full of air at atmospheric conditions and connect it, via a connecting link of known volume ("dead space"), to the evacuated apparatus. The connecting link in this apparatus would be that portion of the side arm of the adapter above the stopcock. The pressure of the total assembly would then be measured and the stopcock closed. The volume of the ambient air which had been introduced into the apparatus could then be calculated. In this particular application the procedure would be facilitated by the availability of the *in situ* transducer with its linear response and of the data logger. The mV outputs to the logger could therefore be used to compare the pressures. A variant on this which would not require a standard bulb would be to measure the volume of the apparatus and adjust the logger using this value for the volume of the air with the transducer open to the atmosphere. This method at first sight seems attractive in its simplicity but is in fact similar to the bulb method since a bulb technique or a volumetric or gravimetric technique would then be used to determine the volume of the apparatus. The method has the disadvantage that in calibration one would then heat the apparatus full of air at atmospheric pressure to the test conditions and the pressure would become above atmospheric with the likelihood of leaks.

A second method is to utilise a high quality gas-tight syringe by which means a predetermined quantity of air can be introduced. In the bulb technique the quantity introduced is determined by the volume of the apparatus into which the air is introduced and to a smaller extent by the volume of the dead space (which needs to be measured in each case). The syringe technique should therefore lead to a simpler calculation for routine purposes.

### 2.7.3 Introduction of Air with a Syringe

The use of a syringe not only allows different volumes of air to be introduced but allows a method which in this particular application eliminates the "dead space" problem. This is achieved by evacuating the apparatus, filling the dead space with mercury (the part of the side arm above the stopcock), sealing the end with a rubber septum, opening the stopcock to allow the mercury to drop into the reaction tube and then injecting a known amount of air with a syringe. The air injected is now contained in a space identical in volume (except for the additional tiny volume of the capillary bore of the stopcock) with that of the normal apparatus not containing the mercury and the stopcock closed. The pressure is therefore the required pressure. This technique when translated into a practical procedure using a 5 cm<sup>3</sup> syringe becomes as follows:

- (1) Assemble the complete glassware with transducer and include a solid filler of volume equal to that of the sample to be tested.
- (2) Fill the side arm of the adapter above the stopcock with mercury.
- (3) Level off the mercury using a metal washer of diameter equal to that of the end of the side arm.
- (4) Stretch a rubber septum over the end of the side arm and washer, press the septum on to the washer and manipulate to displace traces of entrapped air.
- (5) Adjust the syringe to 5.00 cm<sup>3</sup> and just pierce the septum with the needle.

(6) Open the stopcock and press the plunger to zero.

(7) Remove the syringe.

The apparatus is now placed in the heating bath until thermal equilibrium has been established and the logger appropriately adjusted. The time taken to reach equilibrium in a heating bath at 120°C was found to be 1½ hours.

#### 2.7.4 Errors Using a 5 cm<sup>3</sup> Syringe Technique

A number of factors in this procedure produce small errors and these have been investigated.

Air under the septum: accuracy of delivery from the syringe. Tests showed that the use of the metal washer as described in the above procedure helped greatly in reducing the quantity of air trapped under the septum. A determination of the quantity and its reproducibility was, however, essential. The accuracy of the calibration marking on the syringe was not doubted but this type of syringe was not designed for injection into a vacuum. Air within the needle and in any space below the plunger will be sucked into the evacuated system. Air might also enter momentarily as the septum is pierced by the needle. The total quantity would be additional to that to which the plunger was adjusted. Values for the above additional quantities of air delivered from a 5 cm<sup>3</sup> syringe were found by the following sequence of operations. Readings were taken on the logger in the millivolt mode:

- (1) Assemble the evacuated apparatus complete with mercury dead space filler and septum seal; the millivolts recorded (zero offset of transducer) are equivalent to the vacuum.
- (2) Open the stopcock; the additional millivolts recorded are equivalent to the air under the septum.
- (3) Insert the needle of the 5.00 cm<sup>3</sup> syringe with the plunger at zero; the additional millivolts are equivalent to the air associated with the syringe.

(4) Remove the syringe and septum; the millivolts recorded are equivalent to the atmospheric pressure and can be equated to the volume of the apparatus.

For these error determinations the nominal value for the volume of the apparatus can be used (24 cm<sup>3</sup>). The following values were obtained by two operators:

Operator	Air under septum, cm <sup>3</sup>		Air with syringe set at zero, cm <sup>3</sup>	
	A	B	A	B
Number of determinations	9	9	9	9
Mean	0.058	0.075	0.068	0.063
σ of the 9 values	0.012	0.018	0.001(1)	0.004(1)
Standard error (estimated σ of m)	0.004	0.006	0.000(4)	0.001(4)

The values found for these errors are small but very reproducible. The mean error from the two operators due to the septum was 0.067 cm<sup>3</sup> and that due to the syringe was 0.066 cm<sup>3</sup>. A check was made at this stage on the calibration of the syringe in a separate experiment using the same technique with the plunger of the syringe at 5.00 cm<sup>3</sup>. The total air delivered was found to be 5.12 cm<sup>3</sup>. When 0.13 cm<sup>3</sup> is subtracted for the sum of the errors referred to above, this confirmed within practical limits the 5.00 cm<sup>3</sup> marking on the syringe. For this estimation an accurate value for the volume of the apparatus was determined by an independent volumetric technique.

Capillary bore of stopcock. In the procedure for the calibration described above (2.7.3), the volume of the free space into which the air is injected is greater than the required volume by the volume of the capillary bore of the stopcock. No additional air is introduced because the capillary is evacuated. The volume of the capillary is 0.11 cm<sup>3</sup>. In a calibration involving a typical 5 g sample of volume 2.8 cm<sup>3</sup>, the free space would be 24.0 - 2.8 = 21.2 cm<sup>3</sup> and the volume of 0.11 cm<sup>3</sup> would be

additional to this. This is equivalent to a true gas evolution of 4.974 cm<sup>3</sup> being registered as 5.00 cm<sup>3</sup>.

#### Temperature differential during calibration.

There will be a slightly different temperature differential established during the heating stage of the calibration, when a quantity of mercury is in the reaction tube and the stopcock is open, than will exist during a stability test when no mercury is in the apparatus and the stopcock is closed. The effect of this can be estimated by determining the "MEAN EFFECTIVE TEMPERATURE" under the two conditions and the pressures produced will be in direct proportion to the two temperatures ( $\bar{A}$ ). The "MEAN EFFECTIVE TEMPERATURE" is that single hypothetical temperature which can be substituted for the temperature differential and which, together with the actual pressure, corresponds to the total gas volume. It can be determined by introducing any quantity of air at a known temperature into the apparatus, measuring the mV output on the logger and then heating the apparatus to equilibrium conditions (90 min for a test temperature of 120°C) and again measuring the mV output. The values shown below were determined with the inclusion of a filler to represent the sample and for a test temperature of 120°C:

Apparatus as normally used	91.9°C
Apparatus as used in calibration	86.0°C

The effect of this error in the calibration is that a true gas volume of 5.00 cm<sup>3</sup> will register as 5.083 cm<sup>3</sup>.

Summation of errors. The total error expressed as a correction factor to a 5.00 cm<sup>3</sup> nominal volume of air injected is the algebraic sum of the above. This factor requires to be added before the adjustment to standard temperature and pressure and subsequent setting of the logger.

Septum	+ 0.067
Syringe	+ 0.066
Stopcock	- 0.026
Temperature	- 0.083
TOTAL	+ 0.024

Values to which the logger requires to be set for typical ambient conditions can be pre-calculated and be available for reference so that no calculations are involved in this correction or indeed in the whole of the calibration process.

#### 2.7.5 Comparison of Bulb and Syringe Techniques

For routine purposes, using the apparatus of standard volume, the syringe technique has the advantage sought (2.7.2). In the bulb technique, additional operations would be necessary, namely (1) measurement of the dead space, (2) calculation of the volume of air injected, and (3) an additional Table for correction of different volumes to the standard conditions. For routine purposes at AWRE, the syringe technique is considered the more suitable for the reason stated, although both techniques are relatively simple.

For non-routine purposes, using apparatus of different volumes, the bulb technique is probably the more useful since it avoids the re-calculation of the correction factor. This would be different because of the different contribution by the stopcock and temperature differential errors.

#### 2.8 SENSITIVITY AND STABILITY

Figure 7 shows a stability run on an RDX + TNT type composition which, because of the limitation of sample available, was performed on 0.5 g. The test was performed before the septum sealing technique was as fully developed as at present and the entrapped air at the start of the run was 0.2 cm<sup>3</sup>. This has been subtracted for presentation in Figure 7. The top graph shows a plot of the figures printed on the teleprinter at 2 hourly intervals. The display on the logger showed a small scatter which the printer picked at random. The bottom graph shows a replot in which each five consecutive readings were averaged and these values plotted at 10 hourly intervals. This shows a good graphical presentation for a gas evolution of 0.10 cm<sup>3</sup> over a period of 70 hours. This stability enables reliable tests to be performed on



small quantities of sample and lends itself to a computer averaging technique.

## 2.9 COMPARISON WITH THE MERCURY MANOMETER APPARATUS

The technique for performing a stability or compatibility test by the vacuum stability method which is described in 2.1 to 2.8 is a great improvement on the original technique using a mercury manometer. The latter requires a complicated experimental and arithmetical procedure for the calibration of each piece of apparatus to be used, which results in the establishment of three factors which are specific for each piece of glass-ware. In testing, manual readings of the mercury levels are taken and for each test a further arithmetical calculation is required involving the three particular factors for the apparatus used. The necessity for manual readings determines the times over which tests can be performed and, in particular, tests cannot be started near the end of a working week unless weekend readings can be taken. The new technique requires no calculations in either the calibration or the test stage and the practical procedure for the calibration is simple.

## 2.10 VARIATIONS AT OTHER UK ESTABLISHMENTS

A number of variations on the ANRE design are in use or under development at different centres in the UK. These all use the same transducer but a variety of adaptors are in use or are being investigated. In some of the applications a data logger is incorporated and in some, where only a small amount of testing is performed, a digital voltmeter is used. A read-out in millivolts can be converted to a gas volume using rationalised transducers, together with the volume of the apparatus and a temperature function. This latter can be determined by the method described in 2.7.4 for the "MEAN EFFECTIVE TEMPERATURE".

## 2.11 APPLICATION TO THE NATO COMPATIBILITY TEST

The statistical analysis of results using the

transducer technique with an earlier design of the new apparatus, involving a chart recorder rather than a data logger, was very reassuring<sup>(1)</sup>. The nature of explosives and the quality of explosive plus sample mixes does not always enable identical replicates to be obtained, but the mean value for the gas evolutions from a considerable number of different test mixes was virtually identical with those from the same mixes tested by the mercury manometer method<sup>(1)</sup> (difference between the two means = 0.074 cm<sup>3</sup>). There seems to the author to be no reason why a version of the new test method should not be accepted in an approved method for the NATO version of the Vacuum Stability Test which is currently being standardised.

## 3. CONCLUSION

The new technique for performing the Vacuum Stability Test described in Section 2 substitutes a manometer and manual readings by a transducer and data logger. The procedure is suitable for use in a routine laboratory and has a number of advantages, namely, it is more efficient in operator time, it is more sensitive, and it facilitates the examination of the products of reaction. In combination with the procedures previously published, for the separation and examination of the condensible and non-condensable products, it provides for a more complete examination of a compatibility problem than the conventional procedure.

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#### BIOGRAPHY

Bill Merrick originates from Manchester, England, was elected an Associate of the Royal Institute of Chemistry in 1941 and is a Chartered Chemist. He is currently Head of a section which deals with the chemical analysis and compatibility of explosives at the Ministry of Defence, AWRE, Aldermaston, England.

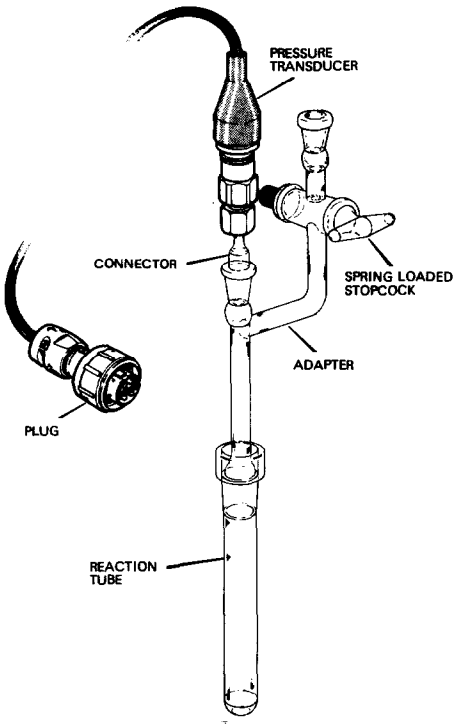


FIGURE 1 GLASSWARE WITH TRANSDUCER

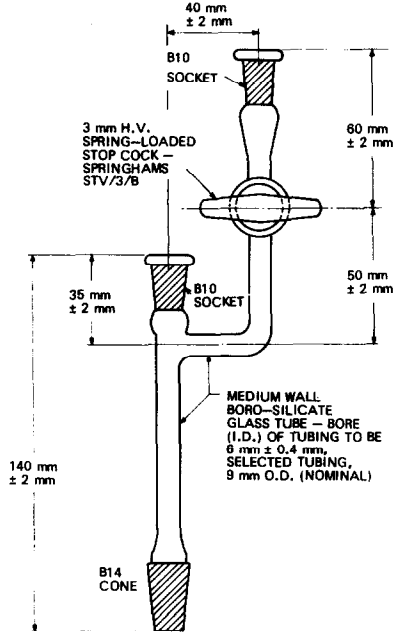


FIGURE 3 ADAPTER

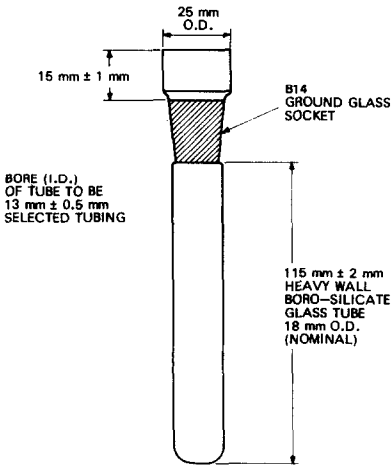


FIGURE 2 REACTION TUBE

BORO-SILICATE GLASS TUBE, 5 mm O.D.  
END TO BE FLAME POLISHED

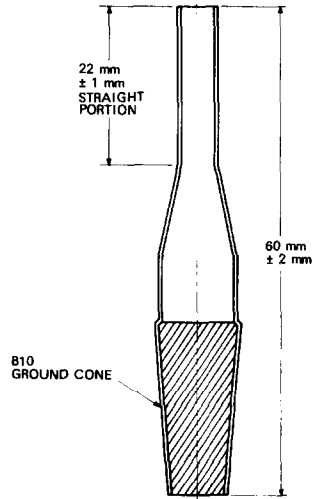


FIGURE 4 CONNECTOR

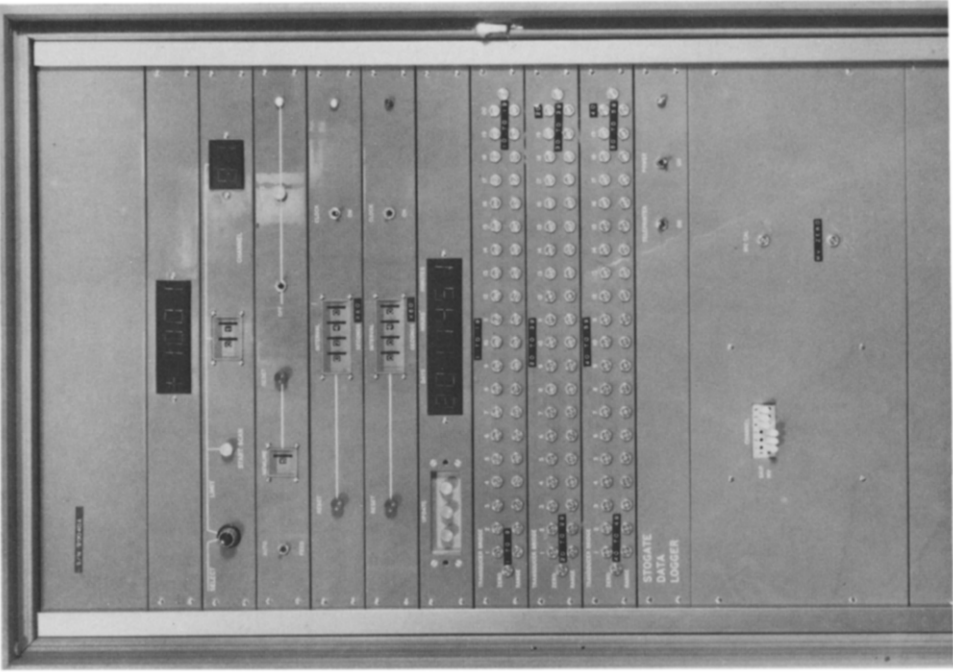


FIGURE 6 DATA LOGGER

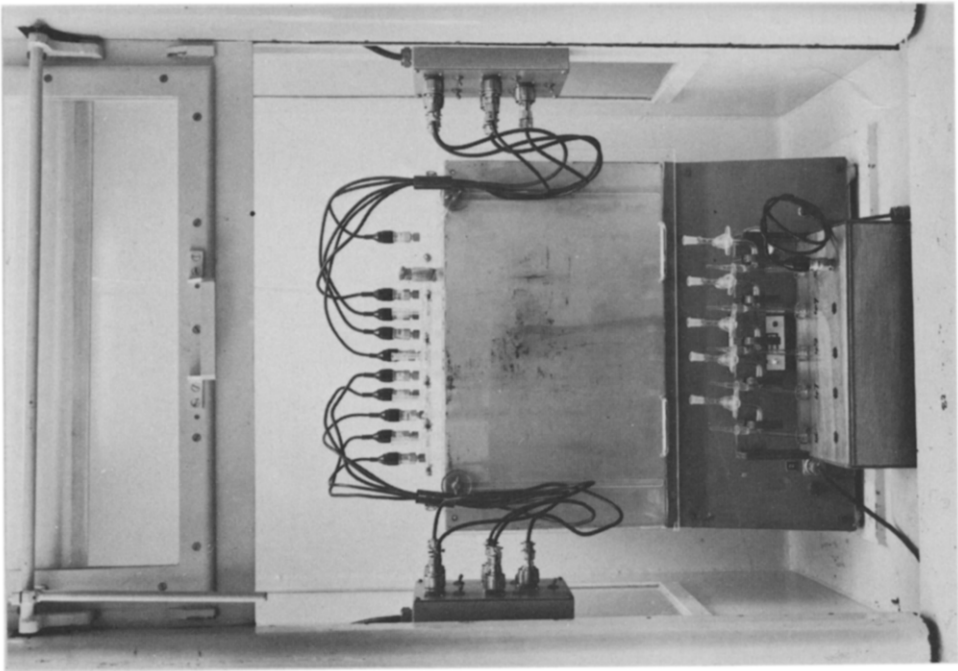


FIGURE 5 HEATING BATH AND TRANSDUCERS

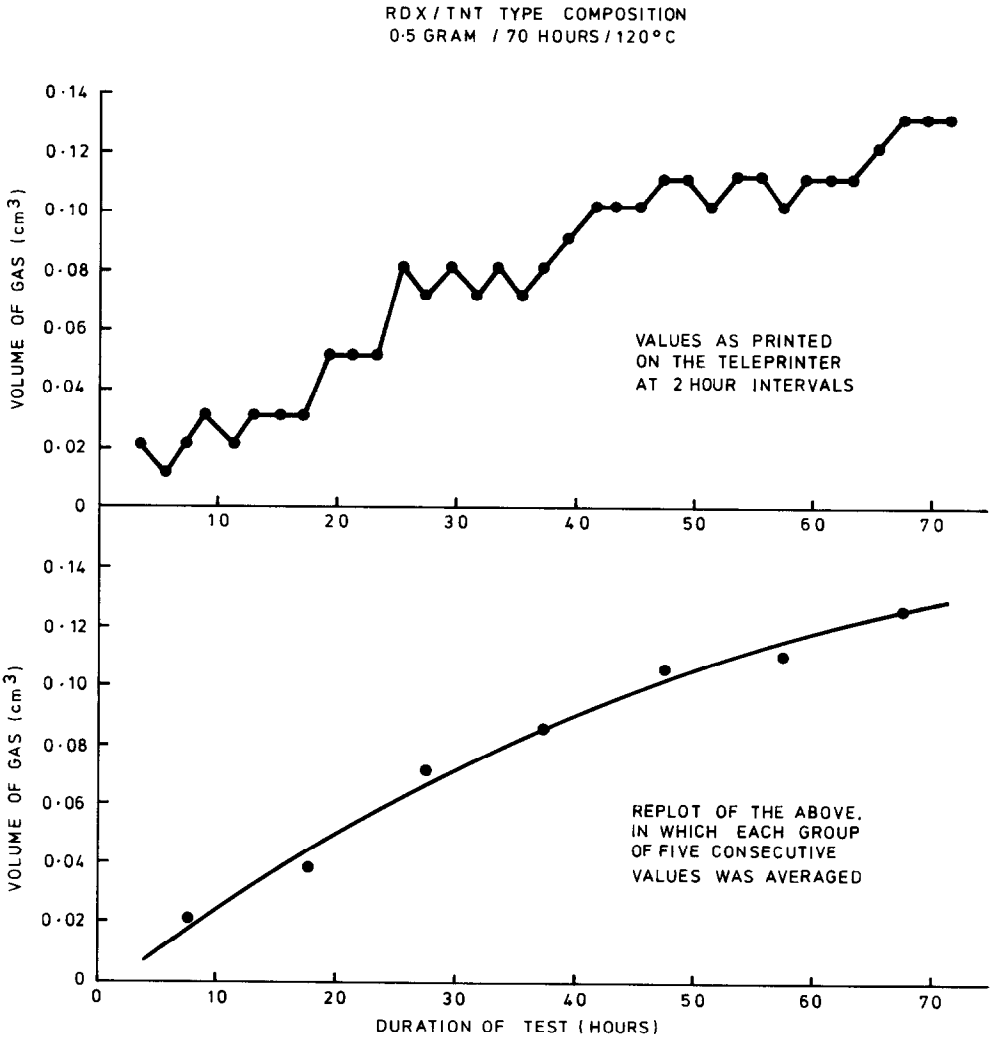


FIGURE 7 APPLICATION OF DATA AVERAGING TECHNIQUE