CONF-9209325--,EGG-M-92417

EGG-M--92417

DE93 010622

DESIGN AND CALIBRATION OF PULSED VAPOR GENERATORS FOR TNT, RDX AND PETN

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ABSTRACT. Computer controlled explosive vapor generators for 2,4,6-trinitrotoluene (TNT), cyclo-1,3,5-trimethylene-2,4,6-trinitramine (RDX) and pentaerythritol tetranitrate (PETN) were built and calibrated to support an Independent Validation and Verification (IV&V) facility for Explosive Detection Systems (EDS) for the Federal Aviation Administration (FAA) at the Idaho National Engineering Laboratory. The generators were constructed using pure explosive suspended on quartz beads which were then loaded into a stainless steel tube. The tube was coiled and placed into a temperature controlled chamber. A carrier gas (ultra-pure air) was passed through the coil to carry the explosive molecules. The generators are capable of delivering a pulse of varying explosive mass through the control of coil temperature, air flow rate, and pulse width. Preliminary calibrations have been completed in the picogram to nanogram range using an Ion Mobility Spectrometer (IMS) as the calibrating instrument. The explosive vapor generators will be used as quantitative vapor standards to establish the lower limit of detection for EDS systems at the IV&V.

INTRODUCTION

Over the last 15 years explosive vapor generators have been constructed by a number of investigators. The first generators were of a basic design that included the explosive in a temperature controlled reservoir through which a carrier gas is passed to carry the explosive vapors. These include designs by Pella[1], Krzymien et al.[2], Carter et al.[3] and Hobbs et al.[4]. All of the above generators are continuous flow systems.

The generators needed in the IV&V tests are required to deliver a pulse of explosive vapors between 1 and 1,000 picograms in less than 5 seconds with a high degree of accuracy. The mass output will be varied during a test with only a few minutes between pulses. Pulsed systems have been reported by Reiner et al[5] and McGann et al[6]. Reiner's system uses a modified gas chromatograph (GC). An aliquot of

a standard is injected into the GC resulting in the emission of the vaporized explosive at the exit of the capillary column. The system by McGann uses a heated syringe which contains a reservoir of the explosive in the syringe barrel. The difficulty in preparing standards at the low pg level and the significant variability in the output from the GC generator[5] and the lack of controls in the syringe method led us to construct a new pulsed vapor generator. The system is based on the design of Carter et al[3].

In this paper the design and preliminary calibration of the explosive vapor generators will be discussed. An Ion Mobility Spectrometer (IMS) was used to calibrate the generators mass output. The calibration of the IMS will also be discussed.

EXPERIMENTAL

VAPOR GENERATOR CONSTRUCTION

A diagram of the vapor generator system is presented in Figure 1. The system is composed of four parts, a thermostated explosive vapor reservoir, a flow control manifold, a supply of thermostated air and a computer data acquisition/control system (not shown on the schematic).

Explosive reservoir. The explosive reservoir contains approximately 0.1 grams of solid explosive suspended on quartz beads. The explosive, either TNT, RDX or PETN, is dissolved in ~15 ml of methyl ethyl ketone. Between 20 and 22 g of 0.1 mm diameter quartz beads are added to the solution. The mixture is stirred during solvent evaporation to aid in homogeneous deposition of the explosive on the beads. The explosive coated beads are then loaded into a cleaned 10 foot long thin walled 1/8 inch OD stainless steel tube. A 1/2 inch long plug of quartz wool is packed into both ends of the tubing to hold the beads in place. In the case of the TNT and PETN tubes, 1/2 inch of empty tubing remains at both ends. The RDX coil is constructed with 1 foot of empty tubing at both ends (The reason for the latter tube design is discussed in the results section). After packing, the tubing is coiled around a 2" thin-walled aluminum tube and placed inside of a 3" thin walled outer aluminum tube with aluminum end caps. The temperature of the explosive reservoir is controlled to within ± 0.1°C by varying the flow rate of the heating/cooling air through the aluminum canister. The explosives reservoir temperature is monitored by two thermocouples. The thermocouples are positioned 2 1/2 inches from each end of the coil in the TNT and PETN generators and 1 foot from each end of the coil in the RDX generator. A nicrome wire heater maintains the last inch of the vapor generator coil at 4 ± 1°C higher than the rest of the generator to eliminate deposition of explosive vapors at the outlet. This last region is monitored by a third thermocouple positioned 1/2 inch from the outlet.

Flow control manifold. The carrier gas flow rate is set using a micrometer needle value (V-1). During the initial setup and between

explosive vapor pulses, the carrier gas is diverted using a three way solenoid valve (SV-1) to the setup manifold. The setup manifold is used to simulate the resistance of the generator by adjusting a needle valve (V-2) at the end of the manifold. The resistance adjustment is optimized by monitoring the differential pressure vs time (dPt) response across the generator coil during a pulse of air flow through the generator. A 0-3.6 psig differential pressure cell (Pt-2) is used to monitor the above. When the resistances of the coil and the setup manifold match, the leading edge of the dPt response will rise and level off rapidly. A resistance mismatch will result in either overshooting the final differential pressure (too large of manifold resistance) or a gradual rise to the final differential pressure value (too small of a manifold resistance). An additional pressure transducer (PT-3) is present in the manifold to check the system for leaks.

Data Acquisition/Control System (DACS). The DACS is used to monitor and control various parameters of the vapor generator. The DACS consists of an IBM PS/2 personal computer that utilizes National Instruments MC-MIO-16(L/H)-25 multi-function input/output data acquisition board.

The DACS monitors all 3 thermocouples in the vapor generator, the carrier gas flow rate and the pulse integral (dPt). Explosive vapor pulses are set and controlled on the DACS. During a pulse the carrier gas is switched from the setup side of the vapor generator system to the vapor generator coil via the three-way solenoid valve (valve SV-1). When the injection is completed the flow is switched back to the setup side of the system and a 2-way solenoid valve (SV-2) is opened to relieve the back pressure in the vapor generator line to reduce trailing of the explosive vapor out the generator. The output of the generator is monitored using the change in the dPt across the coil because the response of the flowmeters is too slow to be used. During a typical calibration the flow rate is changed over the range of 20 to 100 ccm and the variability in the dPt over this range is less than ± 1%.

VAPOR COLLECTION TUBES

A preconcentrator is used to collect explosive vapors from the vapor generator for subsequent quantification. These preconcentrators are also used to inject standards into the IMS. A preconcentrator consists of a 3.8 mm long X 5.7 mm OD X 4.0 mm ID quarts glass tube (Richland Glass Co.) containing 5.0 ± 1.0 mg of quartz wool packed into a 13 mm plug positioned 13 mm from the front end of the tube. D. W. Hannum[7] at Sandia National Laboratories has characterized similar preconcentrators with respect to the collection and release of explosive vapors. Hannum reported a quantitative release of adsorbed explosives and a collection efficiency of ~100%.

MATERIALS

Quartz tubing is cleaned with methanol then baked at 225°C. Quartz wool in the preconcentrators is baked after the preconcentrator is assembled at 225°C. All stainless steel tubing is first rinsed with acetone then methanol. The tubing is then baked at 300°C with a nitrogen purge. All teflon tubing is baked at 100°C with a nitrogen purge. Refrigerator grade copper tubing for the heating and cooling air is used as received. Swagelok brand fittings connecting the stainless steel as well as the teflon tubing are stainless steel and pre-cleaned in acetone followed by methanol followed by baking at 225°C.

Solvents are used as received. Methyl ethyl ketone is Fisher Certified grade. Methanol is Fisher Optima grade. The explosives are used as received from the FAA. Gas chromatofraphy with an electron capture detector (GC-ECD) and gas chromatography with a mass spectrometer detector (GC-MS) analysis did not reveal any significant impurities in the explosives.

EXPLOSIVE STANDARDS

Standards are prepared from dilutions of stock solution of a few mg of pure explosive (TNT, RDX or PETN) to 1 ml of methyl ethyl ketone. This stock solution is kept in an amber vial with a teflon lined cap and stored in a refrigerator to reduce sample degradation. Serial dilutions from the stock are made using methanol. These dilutions are prepared in amber vials with teflon lined crimp tops. To reduce loss of explosive molecules to the walls of the vials, dilutions are prepared six times in the same vial and discarded. The seventh dilution becomes the standard. These standards are also kept refrigerated. The dilution standards are only valid for 24 hours.

ION MOBILITY SPECTROMETER

IMS operating conditions. The IMS used in this study is a PCP model 110. In the IMS vaporized molecules are carried into a Ni-63 source where they are ionized and injected as a pulse (500 μs wide in this study) into a drift region. Ions are separated in space within the drift tube, due to charge, mass and size differences. A typical spectra generated by the IMS for TNT, RDX and PETN with NO_2 as the reagent gas is shown in Figure 2. An IMS spectrum is a plot of voltage vs time. The voltage is related to the ion current arriving at the electrometer plate at the end of the drift tube and thereby to the quantity of explosive molecules entering the source.

To enhance the selectivity of the IMS for the detection of explosives the ion source can be seeded with molecules that either form adducts with the target analyte or reduce the background noise from interferrents in the room air. In this study the ion source is seeded with methylene chloride (CH_2Cl_2) for TNT and RDX detection and nitrogen dioxide (NO_2) for PETN detection. These reagent gases are added using permeation tubes (VCI Metronics Inc.). The typical

concentration of the seed compound is 2.6 ±0.6 ppm for the CH_2Cl_2 and 1.3 ±0.3 ppm for NO_2 (based on a temperature of 20 °C and a carrier gas flow of 250 ccm). The analyte ions detected under the above conditions were TNT-H^-, RDX•Cl^- and PETN•NO_3^-. A general review of IMS and the ion molecule chemistry of the Ni-63 source can be found in Plasma Chromatography[8] and a recent review by St. Louis et al[9].

Purified room air or bottled ultra high purity air is used as the carrier and drift gas in the IMS. A water bubbler is used in the carrier manifold in all of the experiments to add water vapor to the carrier gas[10]. This approach results in shorter instrument clearance times for the explosives and therefore increased sensitivities. The concentration of water is approximately 6 ppt. (based on the saturated vapor pressure of water in air at 20 °C and a carrier gas flow rate of 250 ccm).

Calibration. Calibration data relating IMS response to the mass of explosives is generated by loading measured quantities of explosive onto the quartz wool plug in a preconcentrator. A typical calibration contains 30 points, 5 at each of 6 quantities (1000, 500, 100, 25, 5 and 0 pg). To reduce the variability in sample preparation, a modified Hewlett Packard autosampler (HP model 7673) is used to load the explosives onto the wool. Each injection is 2.5 μl in volume. After allowing the solvent to evaporate (5 minutes) the preconcentrator is inserted into the inlet of the IMS. An external heater which is held at 100 °C is then slid over the preconcentrator. The heater supplies a flow of heated purified air at a flow rate typically 50 ccm greater than the flow rate at which the IMS is drawing air through the preconcentrator. The heated air desorbes the explosives from the preconcentrator and carries the vapors into the IMS. The method of quantification of the explosives is outlined in Figure 3.

The output voltage of the IMS is monitored in a time window (typically 550 μs wide) centered on the peak associated with the explosive to be quantified. The voltage in this window is integrated using a boxcar integrator (PCP model BC-10) and then subtracted from the integrated voltage in a background window. A new IMS scan is acquired every 26 msec (gate rep rate of 39 Hz). The signal - background voltage is next sent to an HP model 7673 series II integrator. The 7673 integrates the output voltage vs time. This final step is also performed using a software package called "Chrom-Perfect" by Justice Innovations. The software allows storage and post analysis inspection and manipulation of the data.

Vapor Generator Calibration

The vapor generator calibration procedure involves three steps, first the calibration of the IMS [precalibration] then the vapor generator and finally a second IMS calibration [post-calibration]. This process takes approximately 12 to 15 hours. The IMS calibration is repeated to assure that no significant drift in IMS response has occurred during the vapor generator calibration. The points from both IMS calibrations are combined to calculate the IMS response vs explosive

mass equation. This equation is then used to estimate the vapor generator mass output. A typical vapor generator calibration is performed at a set coil temperature and pulse width (5 seconds in this study) with the mass output varied by changing the carrier gas flow rate. The output could also be changed by varying the pulse width or both the carrier flow rate and pulse width. Varying the coil temperature to adjust the vapor generator mass output is not feasible in the IV&V tests due to the long equilibration time needed to reach the steady state saturated vapor concentration for the explosive in the coil.

Explosive vapors from the generator are collected using the quartz preconcentrators. The preconcentrator is positioned at the exit of the generator. A teflon washer is placed between the preconcentrator and the generator to assure that all of the vapors enter the preconcentrator. The typical DACS sampling method included a 5 sec presampling time followed by a 5 sec sample pulse and a 2 sec post sampling time. After the 12 seconds the preconcentrator is removed from the vapor generator outlet and placed immediately into the IMS inlet where a heater is slid over the preconcentrator to desorbed the explosives into the IMS. Quantification of the sample is accomplished by integration of the IMS response as described in the IMS calibration section.

RESULTS AND DISCUSSION

ION MOBILITY SPECTROMETER CALIBRATION

The IMS calibrations (combined points from the pre and post-calibrations) for TNT, RDX, and PETN are shown in Figures 4, 5 and 6 respectively. The sensitivity of the IMS for TNT and RDX is similar, with both explosive calibration curves having slopes of approximately 0.02 (IMS response $X10^6$ / pg explosive, 0.023 for TNT and 0.020 for RDX). The intercept for the combined TNT calibrations is 119,000 while the intercept for RDX averaged 296,000. The IMS response for PETN was significantly less with a slope of 0.0065 and an average intercept of 5,000. Linear regression analysis of the IMS calibrations typically had R^2 values of >0.99. Blanks were not included in the calculation of the calibration curves. The blanks did yield a measurable response however, therefor they dictate the baseline above which the IMS lower limit of detection for each explosive is calculated. The average response of the IMS for blanks is equivalent to 5 - 10 pg for TNT or RDX and 10 - 20 pg for PETN. The above quantities were estimated from the IMS calibration curves. Since preconcentrators are randomly selected during the calibration process, typically from a set of 5, preconcentrators used in the blank injections were used previously in an explosive injection. A possible scenario for the origion of the blank signal may be the following. Explosives are taken up in the methanol then redeposited at sites that are more thermally labile. Explosive molecules may also be released from the IMS inlet/source region if residual methanol exists on the preconcentrator quarts wool.

VAPOR GENERATOR CALIBRATION

As was mentioned in the experimental section the explosive output from the generator is monitored indirectly using the differential pressure change vs time (dPt) across the generator during a pulse. At this time the output volume is estimated using the product of nominal flow and pulse width. We are currently deriving the relationship between dPt and output volume. The curves resulting from the regression analysis of dPt vs nominal flow for the TNT, RDX, and PETN vapor generators are presented in Figure 7. A linear relation existed between dPt and flow rate for all of the vapor generators tested (typical $R^2 \geq 0.996$). Significant differences exist, however, between the slopes of the curves between vapor generators. The variation in the slopes could be the result of differences in resistance between the three coils, due to physical construction, changing resistance of the air with temperature (higher resistance at higher temperatures) and variations in the setup from day to day of the VG (i.e., flow rate resistance of the setup side of the vapor generator).

The IMS response vs dPt at a specific vapor generator coil temperature for each explosive are plotted in Figures 8 through 12. The mass output from the explosive vapor generators at the average dPt values associated with each of the nominal flow rates used in the calibrations (20, 40, 60, 80 and 100 ccm) are presented in Table I.

TABLE I

Calculated Vapor Generator Output in Picograms vs dPt, 5 Second Pulse, Preconcentrator Injection.

TNT, 18°C Coil Temperature:

dPt	Picograms	Standard Deviation	% Error (2σ)
1.26	55.4	19.9	72
2.34	103	19.5	38
3.51	155	19.5	25
4.42	196	19.6	20
5.97	264	20.4	15

TNT, 22°C Coil Temperature:

dPt	Picograms	Standard Deviation	% Error (2σ)
1.25	83.7	22.7	54
2.49	171	22.3	26
3.74	258	22.3	17
5.00	346	22.7	13
6.53	453	23.7	10

TABLE I, cont.

RDX, 61.9°C Coil Temperature dPt	Picograms	Standard Deviation	% Error (2σ)
1.93	112	20.7	37
3.79	222	21.8	20
5.68	333	24.1	14
7.60	446	27.5	12
9.61	564	31.8	11

RDX, 75.8°C Coil Temperature Dpt	Picograms	Standard Deviation	% Error (2σ)
1.99	557	187	67
3.79	850	185	44
5.77	1170	187	32
8.01	1540	194	25
10.1	1870	203	22

PETN, 64.4°C Coil Temperature dPt	Picograms	Standard Deviation	% Error (2σ)
2.55	340	164	96
4.76	571	161	56
7.01	807	159	39
9.44	1060	161	30
12.1	1340	165	25

The largest uncertainty in the vapor generator calibration is associated with the sample collection method. This point is illustrated by comparing the calibration for the PETN generator done using preconcentrators (Figure 12 and Table I) with the calibration done using direct transfer of the explosive vapors into the IMS through a heated quartz lined transfer tube (Figure 13 and Table II). The direct transfer calibration was done within two hours of the preconcentrator calibration with the PETN vapor generator's coil at the same temperature. A significant decrease in the standard deviation is observed, from 160 to 26. The direct transfer method was tried for the other 2 generators with similar improvement in the precision of the calibrations. While it reduces the variability in response the direct transfer method can not be used at this time because the temporal profile of the injection of the explosives is significantly shortened and not representative of a calibration injection. Figure 14 shows a comparison of the temporal IMS response for the two types of injection methods (preconcentrator and direct injection) along with an injection of 1,000 pg of PETN from a standard. The resulting increased concentration of explosives in the IMS source with the direct transfer method is out of the linear response region of the IMS and therefor results in a decreased sensitivity. This decreased sensitivity is indicated by the lower mass quantities observed in the direct transfer

vapor generator calibration vs the preconcentrator method (ie 1,060 vs 1340 pg PETN output at 64.4 °C coil temperature, 100 ccm flow rate and a 5 second pulse width). This phenomena of an apparent reduction in the vapor generator output was also observed in the other 2 generators when calibrated using the direct transfer line.

Table II

Calculated Vapor Generator Output in Picograms vs dPt, 5 second Pulse, Direct Injection, PETN

dPt	Picograms	Standard Deviation	% Error (2σ)
2.47	309	26.4	17
4.77	490	26.0	11
7.06	670	26.1	8
9.52	864	26.8	6
12.0	1060	28.1	5

A comparison of the measured mass output from all 3 of the generators with the estimated mass output is reported in Table III. The mass output from the generators is calculated using the following equilibrium vapor pressure equations reported by Dionne et al[11] with the resulting vapor pressure incorporated in Equation 4.

$$(1) \quad TNT, \quad Log\ P_{(ppb)} = \frac{-5481}{T(K)} + 19.37$$

$$(2) \quad RDX, \quad Log\ P_{(ppT)} = \frac{-6473}{T(K)} + 22.50$$

$$(3) \quad PETN, \quad Log\ P_{(ppT)} = \frac{-7243}{T(K)} + 25.56$$

$$(4) \quad pg_{(output)} = 2.03x10^{-4}\ K \min\ ml^{-1}\ mole^{-1}\ \sec^{-1}\ (flow,\ ml\ m\ (pulse,\ \sec)\ (P_{(ppT)})\ (mw,\ g\ mole^{-1})\ (T^{-1}_{(amb)}$$

flow = flow rate of the carrier gas
pulse = width of explosive vapor pulse
$P_{(ppT)}$ = equilibrium vapor pressure of explosive, parts per trillion
mw = molecular weight of explosive
$T_{(amb)}$ = ambient temperature

TABLE III

Comparison of Predicted vs Observed Vapor Generator Mass Output in Picograms. Vapor Generator condition, 5 second Pulse, 20 and 100 ccm Flow Rates.

Explosive	VG Temp., °C	Predicted	Observed
TNT	18	54-270	52-257
TNT	22	97-486	80-445
RDX	61.9	23-114	109-561
RDX	75.8	135-673	531-1865
PETN	64.4	271-1356	326-1323

Referring to Table III, the observed output of the TNT and PETN generators were within ± 20% of the expected value. The RDX generator output, however, was up to 393% greater than expected. The reason for this discrepancy may be due to the RDX coil design (see experimental). The last foot of tubing was maintained at 5°C above the coil temperature to reduce the loss of explosives to the tubing walls. One scenario for the elevated mass output is the presence of solid explosive in this last foot. This would result in the generator output being a sum of two sources of RDX vapors. A temperature rise of 5°C above the coil temperature of 61.9°C would increase the mass output at 100 ccm with a 5 sec pulse width from 114 pg to 193 pg. Clearly some other mechanism is causing this anomaly.

A graphical comparison of the vapor pressure of TNT, RDX and PETN as reported by a number of investigators is shown in Figures 15 through 17. These graphs were copied from an article by Dionne et al[10]. The vapor pressure of the explosives in this study estimated from the mass output from the generator are included in the above graphs. The graphs show that even the RDX data from this study is consistent with that previously reported.

Conclusions

Preliminary calibrations have been performed for all three vapor generators (TNT, RDX, and PETN) using the IMS. Linear regression analysis for the vapor generators typically resulted in $R^2>0.85$. The generators are calibrated in the following ranges: TNT, 52 to 445 pg; RDX, 109 to 1,000 pg; and PETN, 326 to 1000 pg. The calibrations have shown that the vapor generators are capable of delivering a mass of explosives very close to that predicted from vapor pressure calculations. What remains to be done is to repeat the calibrations

with the aim of lowering the calibration to 1 pg and to verify that the generator can reproducibly emit a known quantity of explosives by control of flow, pulse width, and VG temperature.

Calibration of the vapor generator in the 1 pg limit will be possible only with an increase in the sensitivity of the calibration standard, the IMS. The lower limit of detection of the IMS in the case of preconcentrator injection is dictated by the chemical noise associated with the preconcentrator. Therefor the preconcentrator introduction method should be replaced in both the IMS and the vapor generator calibrations with an introduction method of higher precision and one that results in a higher sensativity. A possible alternative would be a GC introduction system. GC-IMS systems have been reported to have sensitivities in the sub-picogram (900 femtograms) range for TNT.

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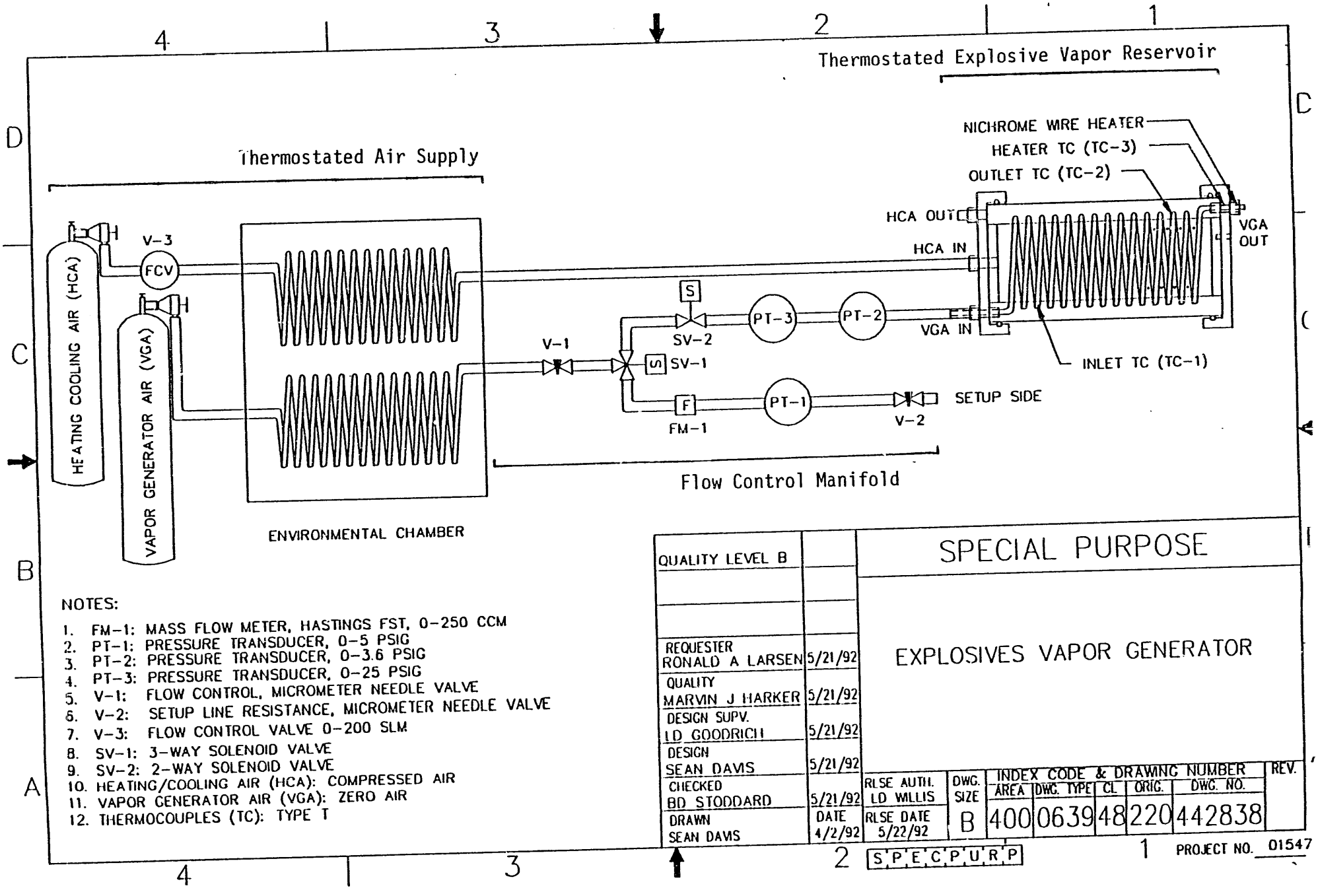
Figure 1. Explosive Vapor Generator



NOTES:

1. FM-1: MASS FLOW METER, HASTINGS FST, 0-250 CCM
2. PT-1: PRESSURE TRANSDUCER, 0-5 PSIG
3. PT-2: PRESSURE TRANSDUCER, 0-3.6 PSIG
4. PT-3: PRESSURE TRANSDUCER, 0-25 PSIG
5. V-1: FLOW CONTROL, MICROMETER NEEDLE VALVE
6. V-2: SETUP LINE RESISTANCE, MICROMETER NEEDLE VALVE
7. V-3: FLOW CONTROL VALVE 0-200 SLM
8. SV-1: 3-WAY SOLENOID VALVE
9. SV-2: 2-WAY SOLENOID VALVE
10. HEATING/COOLING AIR (HCA): COMPRESSED AIR
11. VAPOR GENERATOR AIR (VGA): ZERO AIR
12. THERMOCOUPLES (TC): TYPE T

QUALITY LEVEL B		
REQUESTER RONALD A LARSEN	5/21/92	
QUALITY MARVIN J HARKER	5/21/92	
DESIGN SUPV. LD GOODRICH	5/21/92	
DESIGN SEAN DAVIS	5/21/92	
CHECKED BD STODDARD	5/21/92	
DRAWN SEAN DAVIS	DATE 4/2/92	

SPECIAL PURPOSE

EXPLOSIVES VAPOR GENERATOR

RLSE AUTH.	DWG. SIZE	INDEX CODE & DRAWING NUMBER					REV.
		AREA	DWG. TYPE	CL	ORIG.	DWG. NO.	
LD WILLIS	B	400	0639	48	220	442838	
RLSE DATE 5/22/92							

S P E C P U R P

PROJECT NO. 01547

IMS Spectrum from 50 picograms TNT, 50 picograms RDX, and 75 picograms PETN

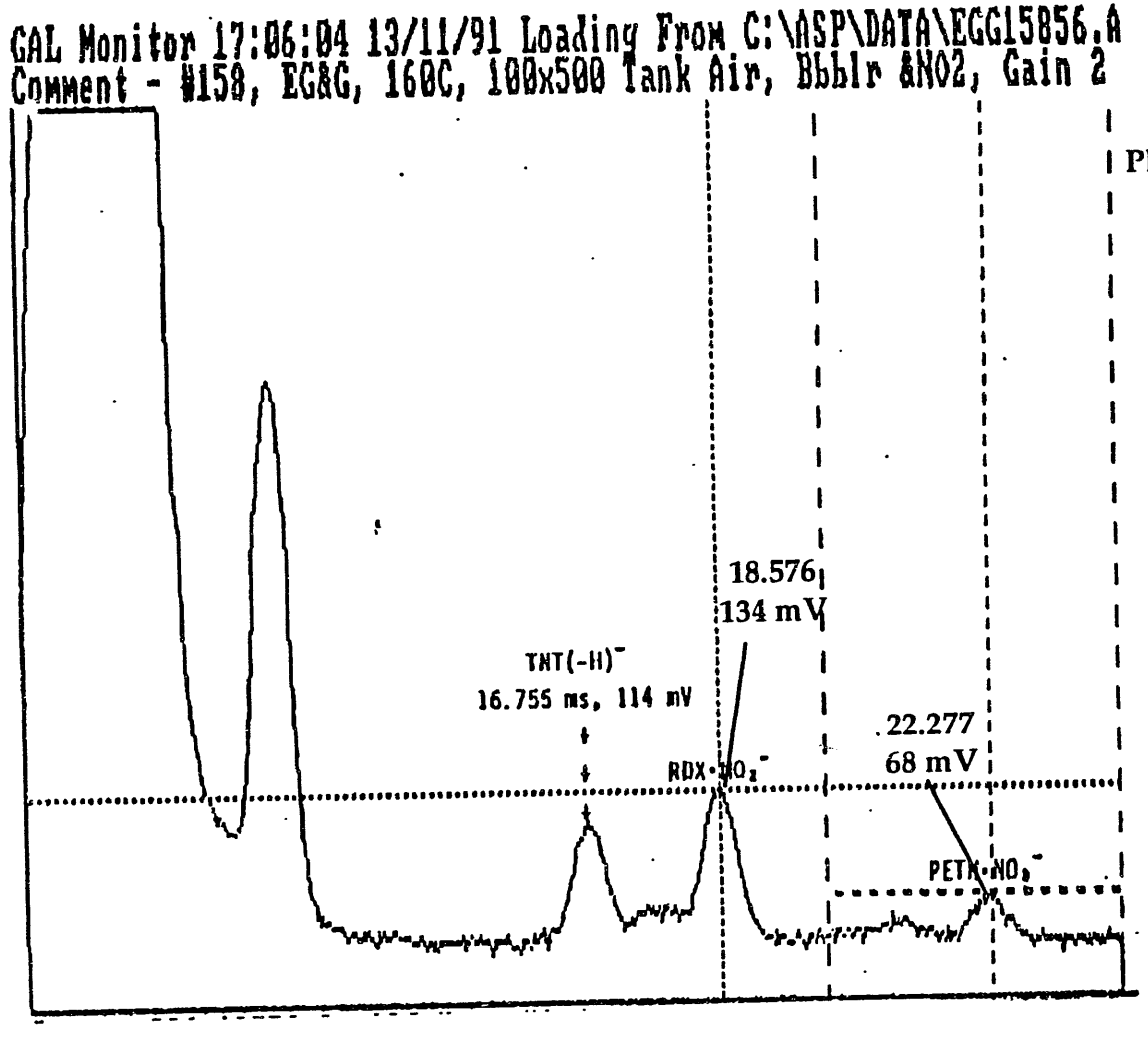


FIGURE 2.

EXPLOSIVE VAPOR GENERATOR CALIBRATION FLOW DIAGRAM

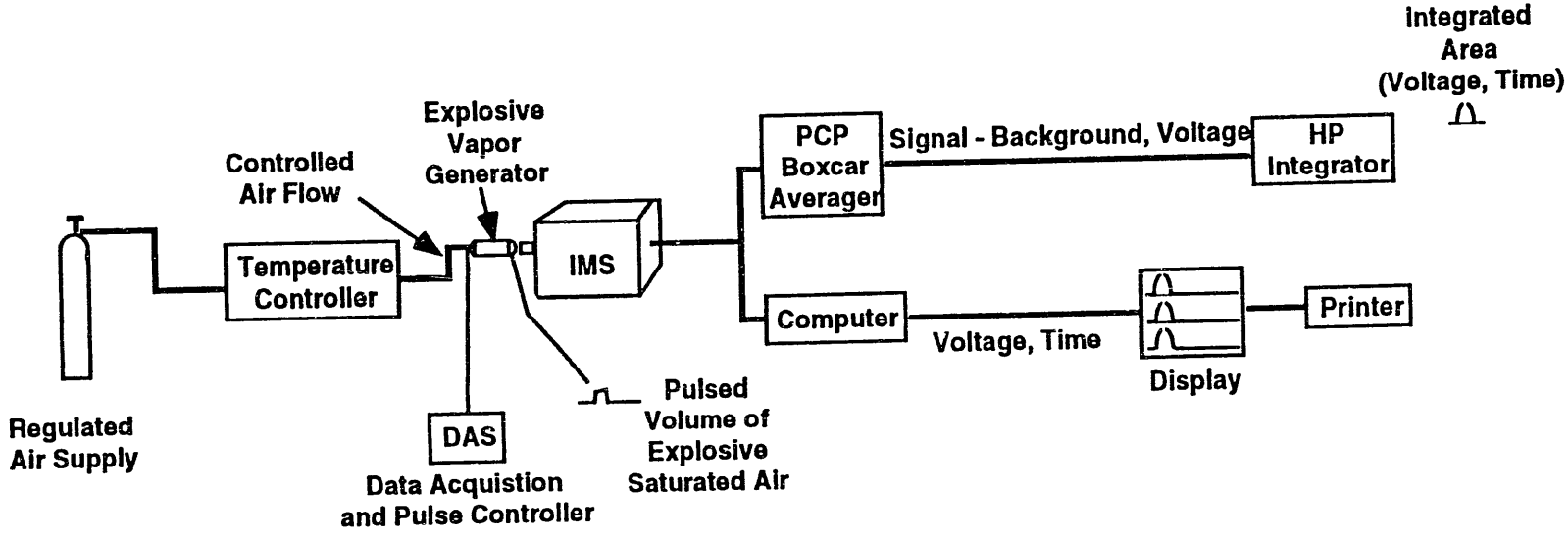


FIGURE 3.

TNT Combined

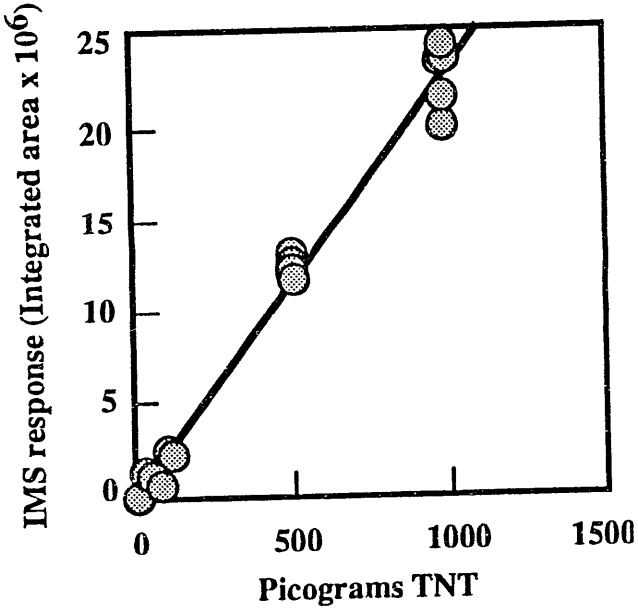
1st and 2nd IMS Calibration

Regression Equation (WLS):
IMS response = .119 + (.0231)(picograms TNT)

Inverse Equation:
Picograms TNT = -5.15 + (43.3)(IMS response)

R^2 = .994

N = 50



Picograms TNT	Repeatiblity C.V (%)	95% Prediction Interval (± picograms)
5	26	5 ± 8.5
25	16	25 ± 18
100	2.8	100 ± 37
500	3.6	500 ± 83
1000	5.5	1000 ± 119

FIGURE 4.

RDX COMBINED
1ST AND 2ND IMS CALIBRATION

Regression Equation (WLS):

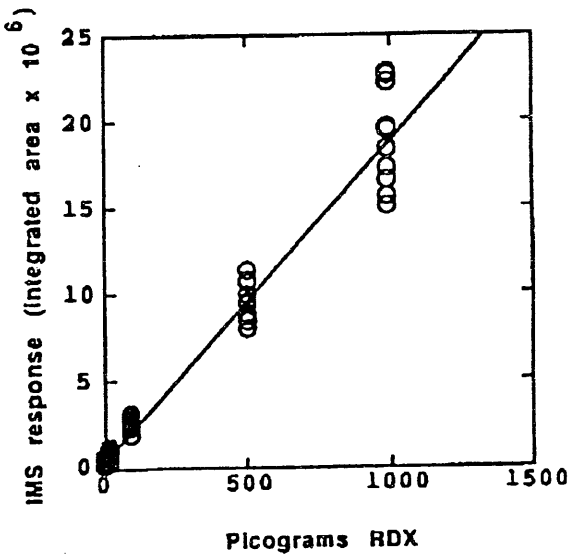
IMS response = 0.296 + (.0199)(picograms RDX)

Inverse Equation:

Picograms RDX = -1.48 + (50.3)(IMS response)

$R^2 = .965$

N = 50



Picograms RDX	Repeatability C.V. (%)	95% Prediction Interval (± picograms)
5	44	5 ± 14
25	33	25 ± 22
100	13	100 ± 54
500	12	500 ± 229
1000	15	1000 ± 449

FIGURE 5.

PETN COMBINED
1ST AND 2ND IMS CALIBRATION

Regression Equation (WLS):

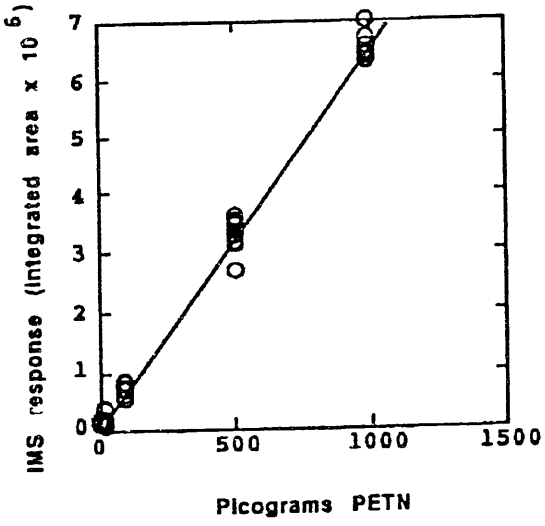
IMS Response = .00494 + (.00652)(picograms PETN)

Inverse Equation:

Picograms PETN = -.757 + (153.3)(IMS response)

$R^2 = .995$

N = 43



Picograms RDX	Repeatability C.V. (%)	95% Prediction Interval (± picograms)
5	75	5 ± 36
25	66	25 ± 37
100	16	100 ± 41
500	7.9	500 ± 57
1000	3.1	1000 ± 75

FIGURE 6.

REGRESSION ANALYSIS OF NOMINAL FLOW VS INTEGRATED FLOW VAPOR GENERATOR CALIBRATION RUNS

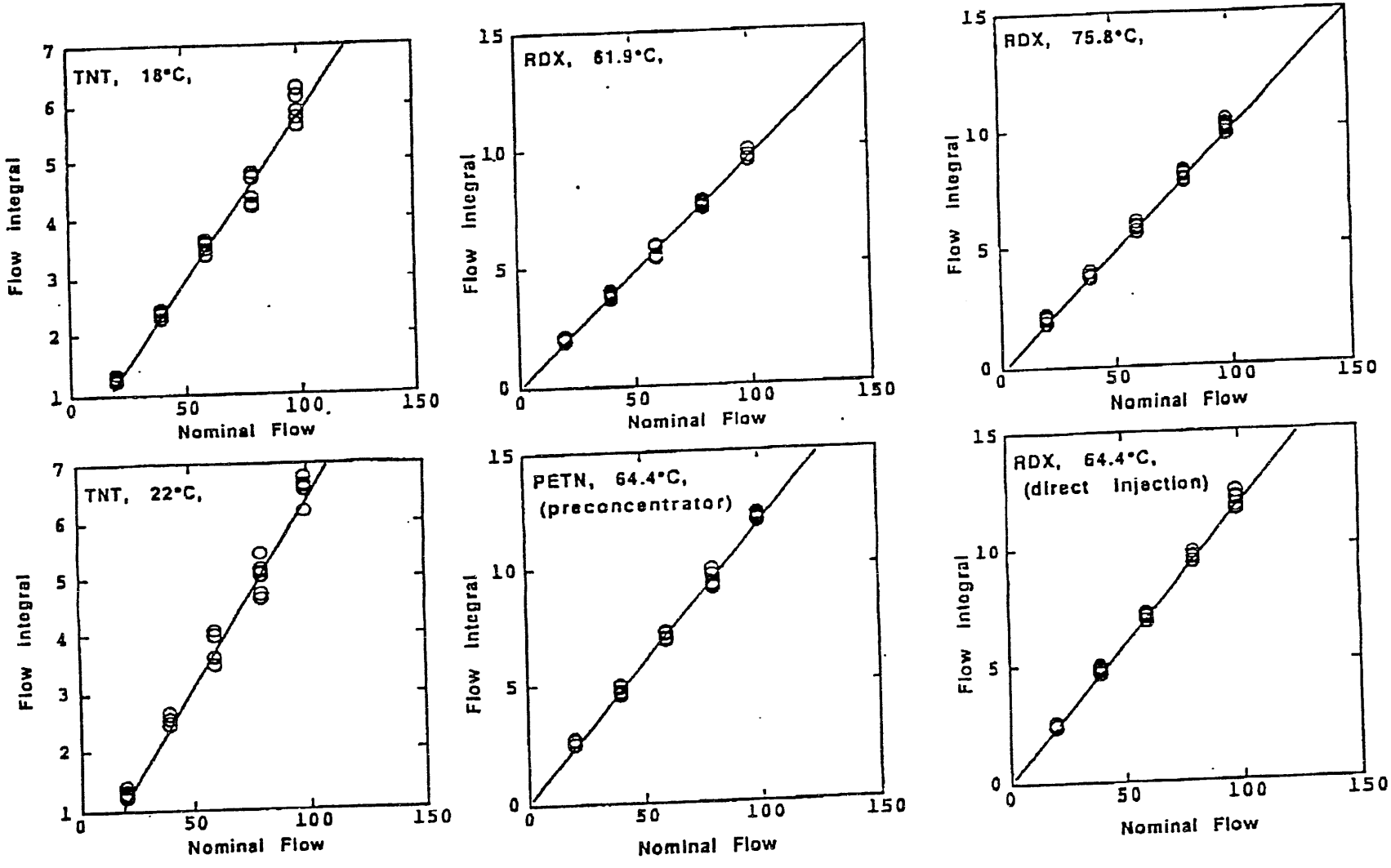
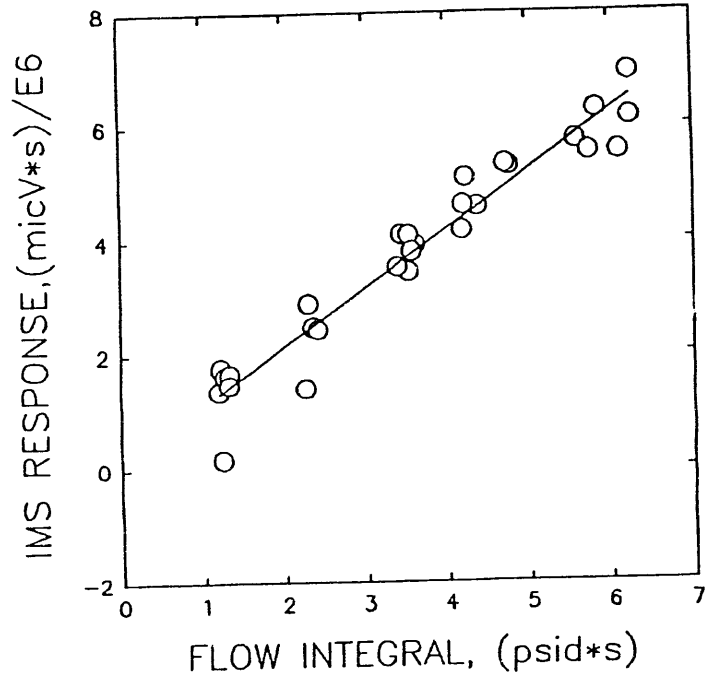


FIGURE 7.

TNT VAPOR GENERATOR CALIBRATION PRECONCENTRATOR INJECTION, 5 SEC PULSE, 18 C

Regression
Equation: IMS Response = 0.107 + 1.027 * (VGFI)
R^2 = 0.940
N = 30



Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	1.26	1.40 (0.94)
40	2.34	2.51 (0.92)
60	3.51	3.71 (0.92)
80	4.42	4.65 (0.92)
100	5.97	6.24 (0.95)

FIGURE 8.

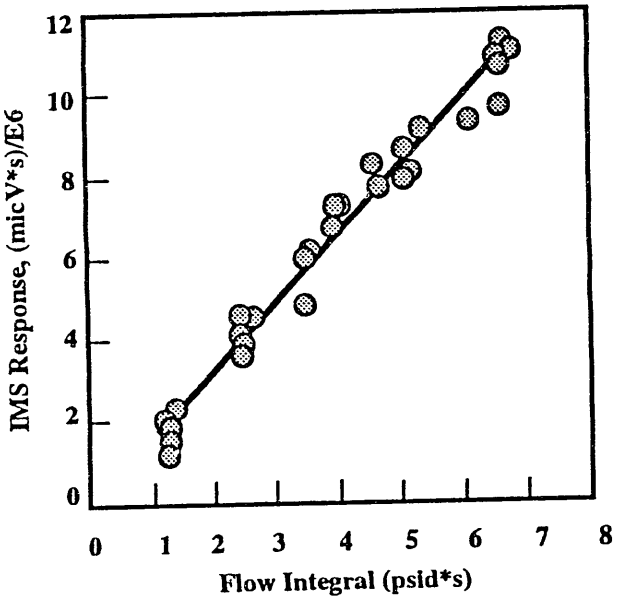
TNT Vapor Generator Calibration, Preconcentrator Injection 5 second pulse

Regression Equation:

IMS response = 0.031 + 1.620*(VGFI)

R^2 = 0.975

N = 30

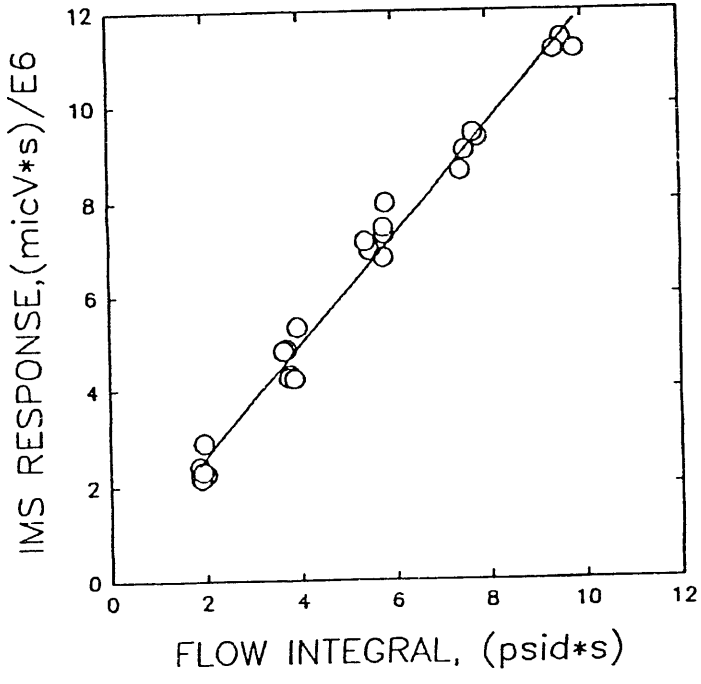


Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	1.25	2.06 (1.08)
40	2.49	4.07 (1.05)
60	3.74	6.09 (1.04)
80	5.00	8.13 (1.05)
100	6.53	10.61 (1.08)

FIGURE 9.

RDX VAPOR GENERATOR CALIBRATION, PRECONCENTRATOR INJECTION, 5 SEC PULSE, 61.9 C

Regression Equation:
IMS Response = 0.265 + 1.171* (VGFI)
R^2 = 0.985
N = 25(4 outliers eliminated from analysis)

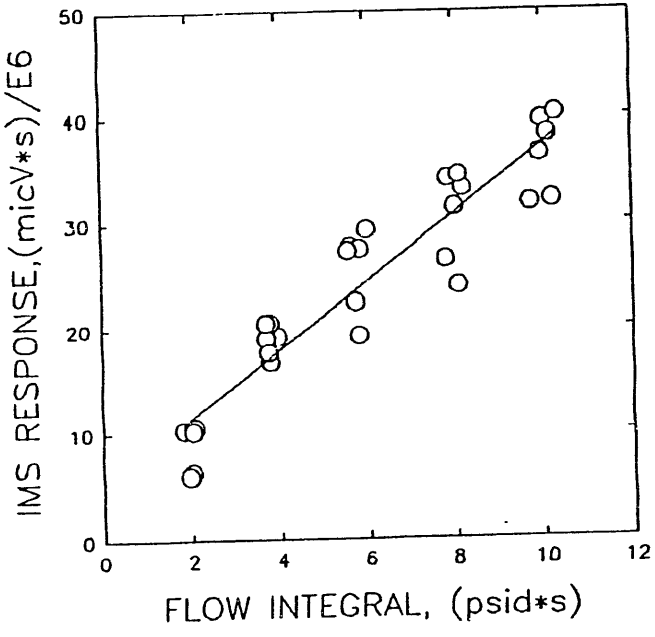


Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	1.93	2.52 (0.83)
40	3.79	4.70 (0.81)
60	5.68	6.91 (0.81)
80	7.60	9.16 (0.82)
100	9.61	11.52 (0.86)

FIGURE 10.

RDX VAPOR GENERATOR CALIBRATION PRECONCENTRATOR INJECTION, 5 SEC PULSE, 75.8 C

Regression Equation:
IMS response = 4.920 + 3.239 * (VGFI)
R^2 = 0.883
N = 30

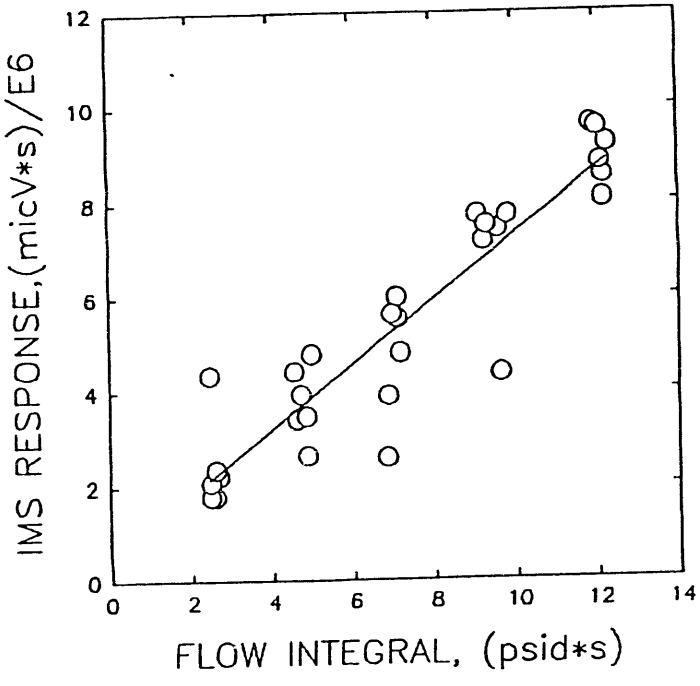


Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	1.99	11.36 (7.56)
40	3.79	17.20 (7.41)
60	5.77	23.61 (7.34)
80	8.01	30.86 (7.40)
100	10.06	37.50 (7.58)

FIGURE 11.

PETN VAPOR GENERATOR CALIBRATION PRECONCENTRATOR INJECTION, 64.4 C

Regression Equation:
IMS Response = 0.480 + 0.683*(VGFI)
R^2 = 0.845
N = 30

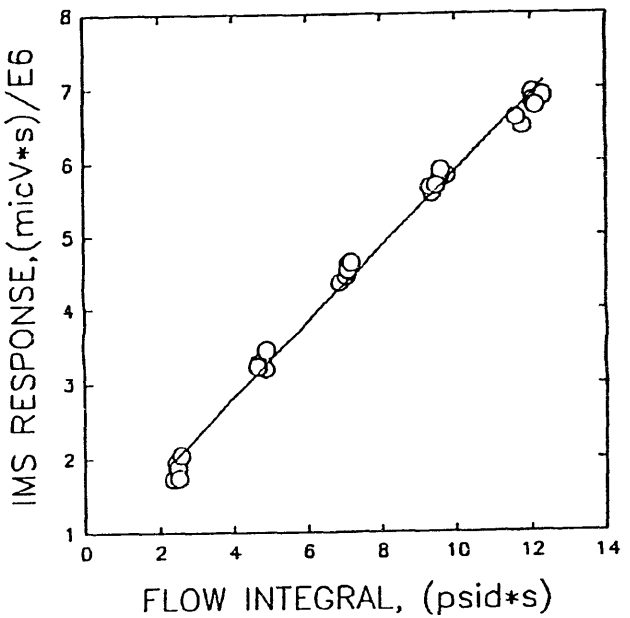


Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	2.55	2.22 (2.19)
40	4.76	3.73 (2.14)
60	7.01	5.27 (2.13)
80	9.44	6.93 (2.14)
100	12.11	8.76 (2.20)

FIGURE 12.

PETN VAPOR GENERATOR CALIBRATION, DIRECT INJECTION, 5 SEC PULSE, 64.4 C

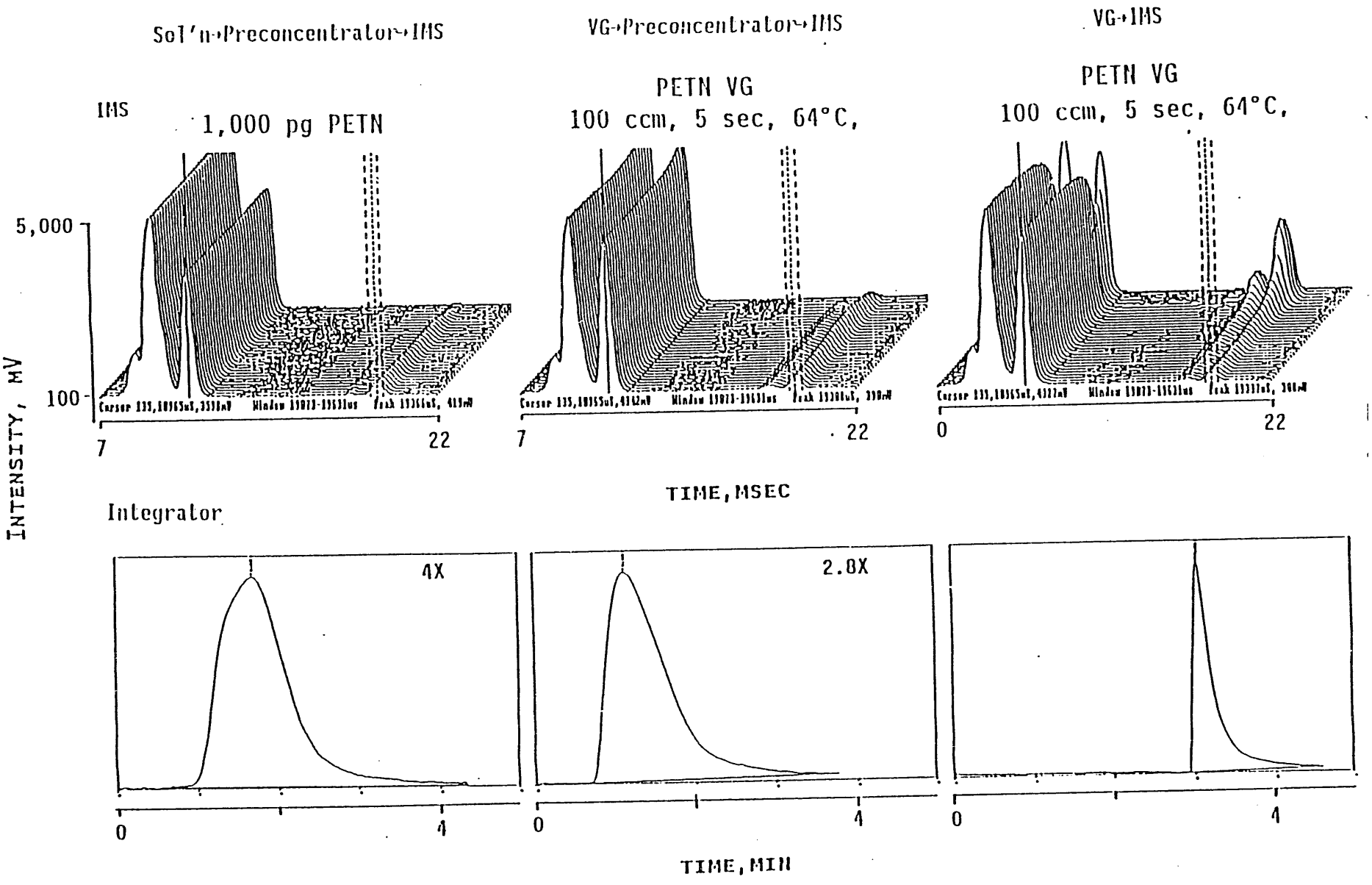
Regression Equation:
IMS Response = 0.753 + 0.513*(VGFI)
R^2 = 0.992
N = 30



Vapor Generator Flow	Average Flow Integral	95% Prediction Interval (IMS area ± uncertainty)
20	2.47	2.02 (0.35)
40	4.77	3.20 (0.34)
60	7.06	4.38 (0.34)
80	9.52	5.64 (0.34)
100	12.00	6.91 (0.35)

FIGURE 13.

FIGURE 14. IMS Response vs Injection Methods



Vapor Pressure of TNT

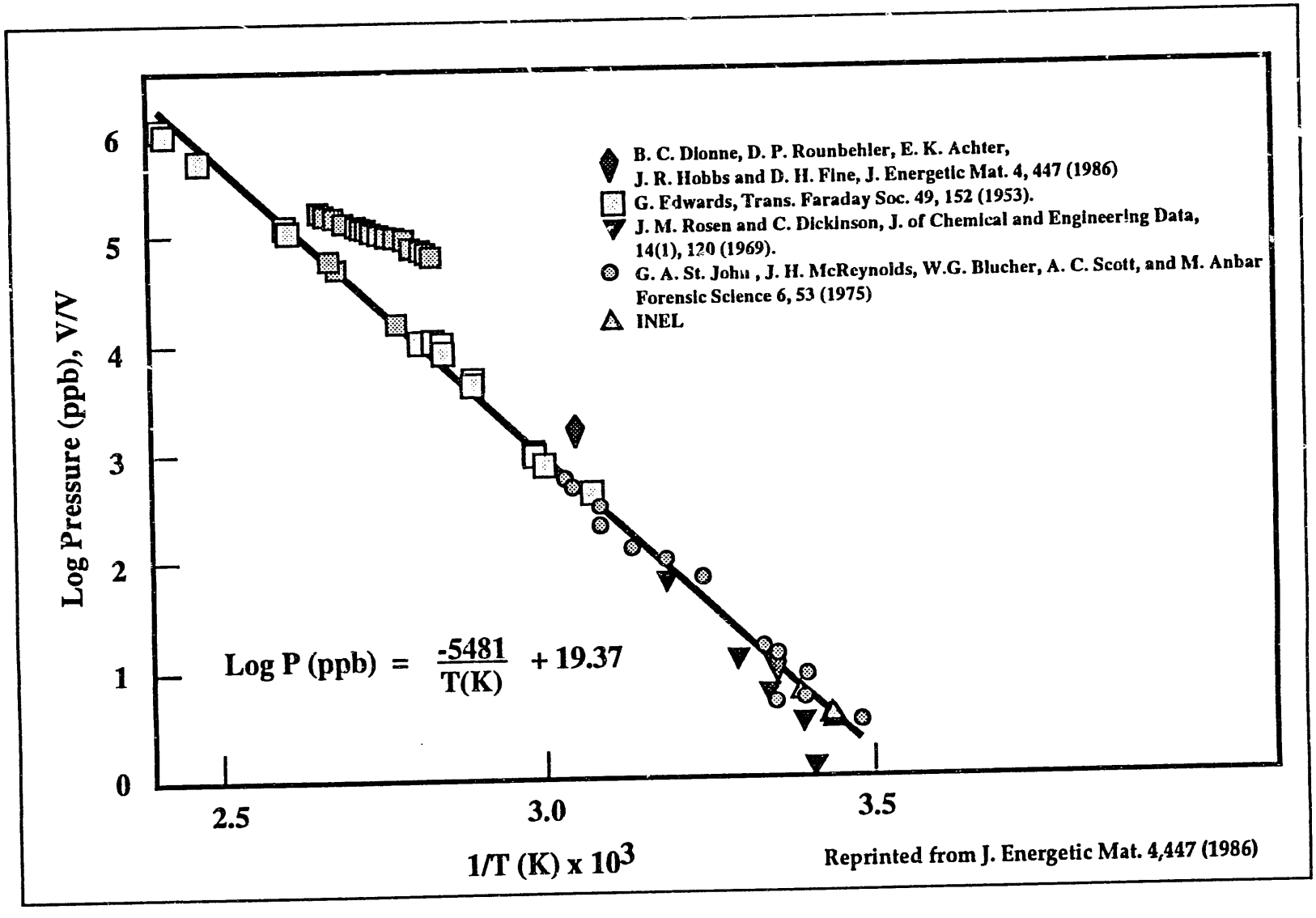


FIGURE 15.

Vapor Pressure of RDX

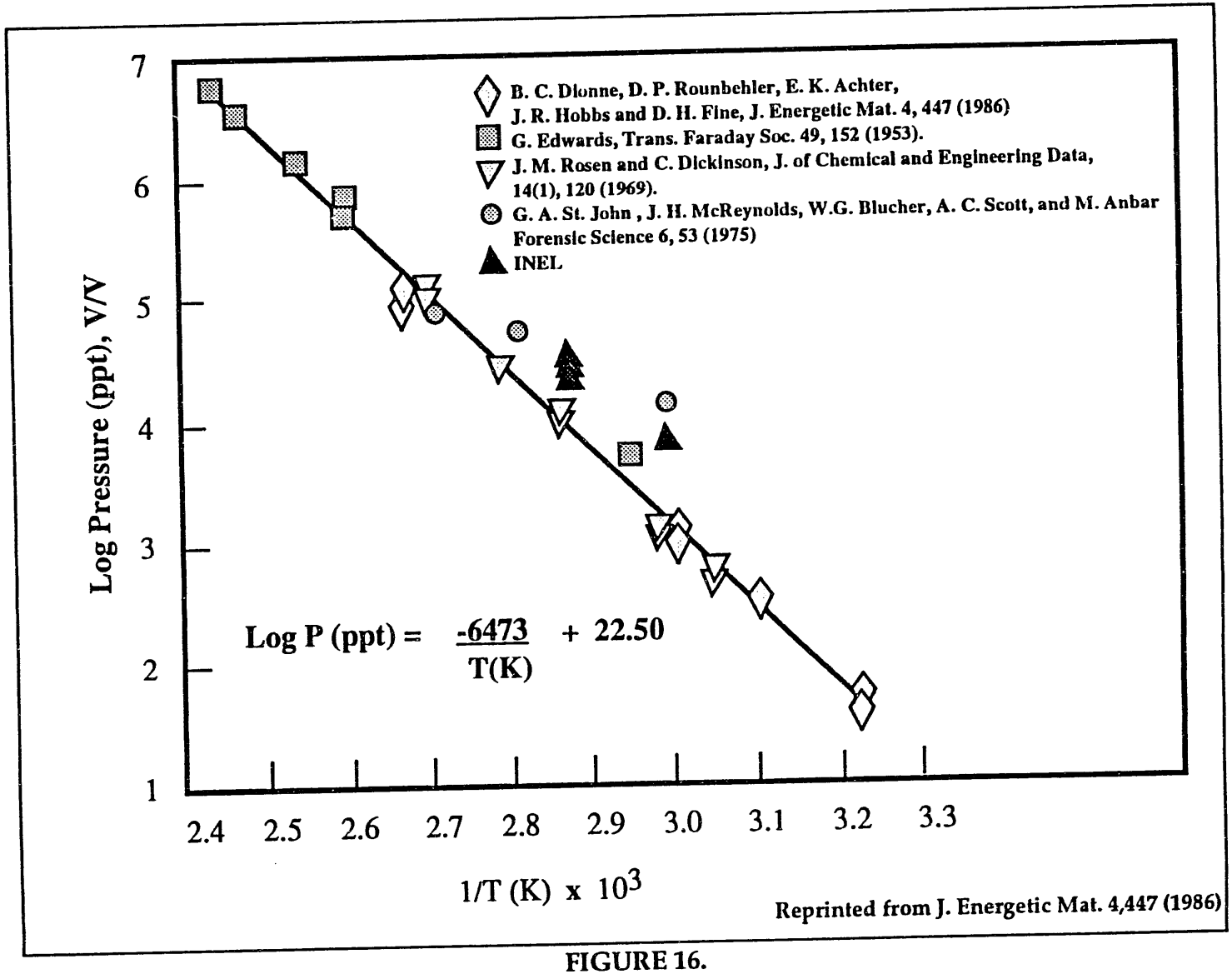


FIGURE 16.

Vapor Pressure of PETN

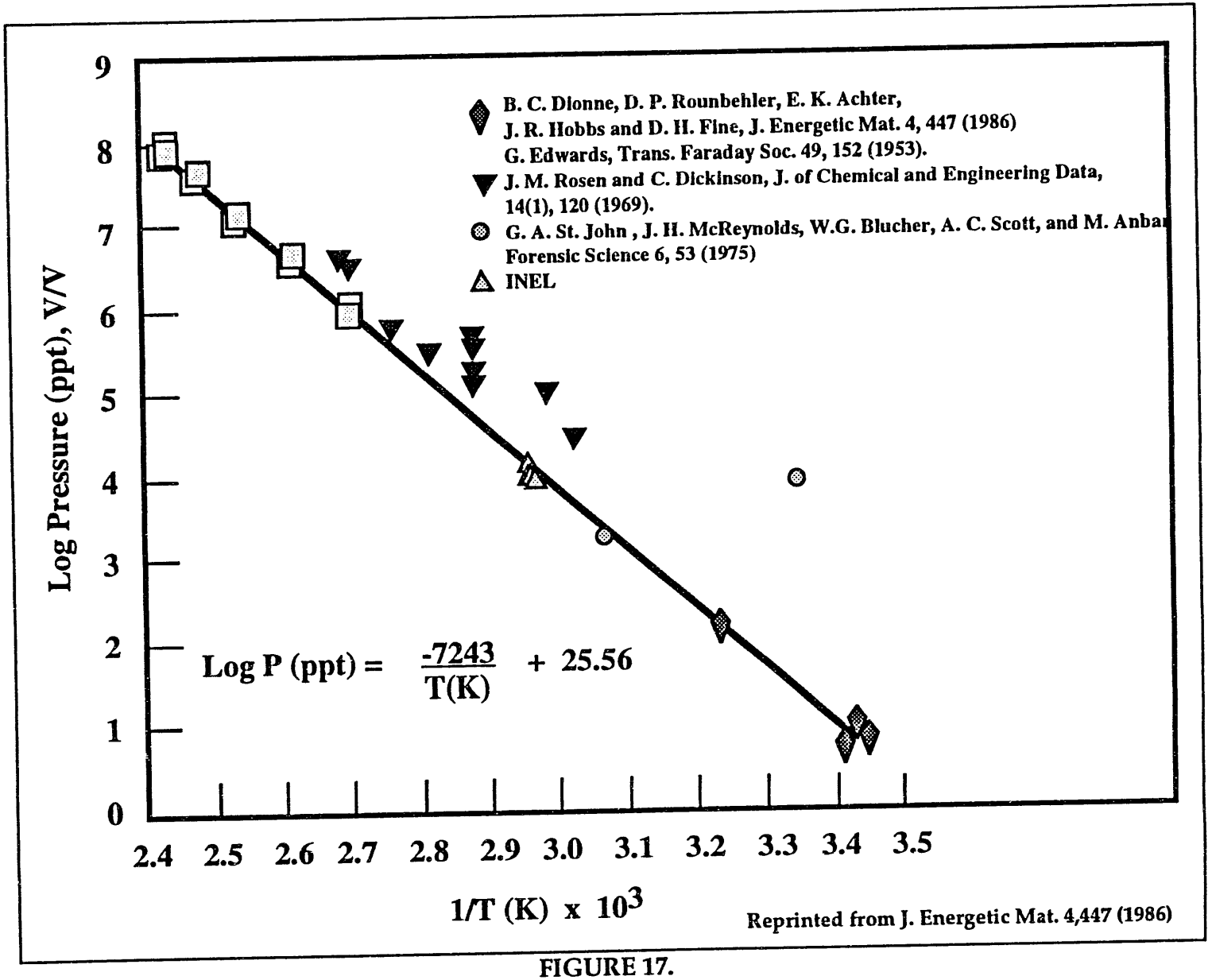


FIGURE 17.

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