OPERATIONAL GUIDELINE: TENAX TA® COLLECTION AND IN-INJECTION PORT THERMAL DESORPTION ANALYSIS OF TRACE LEVELS OF ORGANIC EXPLOSIVE VAPORS

SEPTEMBER 1, 2001

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ORNL-27 (4-00)

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Michael E. Sigman and Ralph H. Ilgner

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Foreword: This document contains operational guidelines for an organic explosives vapor sampling and analysis method developed in the Chemical and Analytical Sciences Division at Oak Ridge National Laboratory under funding provided by the Defense Advanced Research Projects Agency. This document is written in a format resembling that of a standard operating procedure (SOP); however, it is intended only as an operational guideline for those wishing to implement the method.

1. **Scope and Application**

1.1. This procedure describes a method of preparing Tenax TA sorbent traps for collecting vapor samples of trace levels of organic explosives and organics related to the production and decomposition of organic explosives.

1.2. This procedure also describes a gas chromatographic method utilizing thermal desorption (TD) of Tenax TA sorption tubes within a split/splitless injection port, followed by gas chromatographic analysis using negative ion chemical ionization (GC/NICI) mass spectrometric detection.

1.3. The method is suitable for sampling explosives vapors from open or confined spaces.

1.4. This method has been validated for the organic explosives and organics related to the manufacture or decomposition of organic explosives listed in Table 1.

1.5. Refer to Reference Section 12.1 for sources of supply for explosive and related standards.

2. **Summary of Method**

2.1. Trace explosives vapors are collected on Tenax TA sorbent tubes by pulling an air sample across the sorbent bed with the aid of a portable mechanical pump.

2.2. The explosives are thermally desorbed from the sorbent tube within the split/splitless injection port of a GC and focused onto the head of a capillary column.

2.3. Quantification of desorbed explosives is carried out by gas chromatographic analysis with negative ion chemical ionization mass spectrometry.

3. **Precision, linearity and limit of detection**

3.1. The mean precision (defined as percent relative standard deviation, %RSD) of the method ranges from 7 to 56% for 15 analytes determined by GC/NICI with loading range of 0.3-436.4 ng.

3.2. The method limit of detection (LOD, defined in Section 12.2) ranges from 0.1 to 35.4 ng for 15 analytes determined by GC/NICI.

3.3. The method limit of quantitation (LOQ, defined in Section 12.3) ranges from 0.33 to 118.03 ng for 15 explosives analyzed by GC/NICI.

4. Hazards

4.1. Benzene is a carcinogen. Wear nitrile gloves when handling it.

4.2. Methanol and acetonitrile are flammable. Wear gloves when handling them and avoid ignition sources.

4.3. Properly dispose of spent solvents according to appropriate waste handling procedures.

4.4. Insulated (thermal) gloves and eye protection should be worn when handling liquid nitrogen.

4.5. Desorptions are performed at high temperatures (ca. 170 °C). Avoid contact with the thermal desorption injector while a desorption is in progress.

4.6. The gases used in the analytical operation are contained in cylinders under high pressure. Be sure all cylinders are securely strapped. Safety glasses should always be worn when changing cylinders.

4.7. Guidelines for handling explosives at the user's specific site should be followed (see Section 12.5, or explosives handling guidelines at your specific site).

5. Interferences

5.1. Since a short column with a thin film thickness is used in this method, excessive quantities of sample will eventually diminish the column performance.

5.2. Non-target components (such as phthalates and halogenated compounds), that respond to NICI detection, may interfere with the analysis of explosives.

6. Personnel Qualifications

6.1. Personnel with adequate instrumentation analysis background can be trained to perform this method.

6.2. It is recommended that personnel demonstrate proficiency before attempting to perform this method without supervision.

6.3. It is mandatory to have site-specific explosive handling guidelines implemented before using this method.

7. Material and Instrumentation

7.1. Procedure for Manufacturing and Conditioning Tenax TA Sorbent Tubes.

7.1.1. Stainless steel tubing, 6mm OD and 4 mm ID is used to hold the bed of Tenax TA sorbent material.

7.1.2. The tubing is cut to 76 mm in length and washed by sonication with Micro® laboratory cleaning solution, methanol and methylene chloride.

7.1.3. The tubing is packed with a bed of 0.1-0.3 g of Tenax TA sorbent and silanized glass wool (Supelco Inc.) is used hold the sorbent bed in the tube. (Figure 1).

7.1.4. Swagelok® ¼ inch fittings with vespel-graphite ferrules are used to cap the ends of the tube when they are not being analyzed or used for sample collection.

7.1.5. The sorbent-containing tubes are heated at 325 °C under a flow of helium at 100 ml/min for at least 2 hours (Figure 2).

7.1.6. The resulting tubes should have little or no detectable background signal by NICI when thermally desorbed using the procedure described below.

7.1.7. When analyzing explosive residue, a freshly conditioned Tenax TA tube should be used for collecting the vapor sample. A known quantity of internal standard should be spiked onto the Tenax TA tube after collecting the vapor sample and before analysis.

7.2. Preparation of calibration standards.

7.2.1. The stock solutions for each of the 15 compounds (as listed in Table 1) can be prepared in either methanol or acetonitrile at a nominal concentration of 1 mg/ml. The stock solutions can be stored in a flamable-storage approved refrigerator for at least two months.

7.2.2. Calibration standard mixtures are prepared in benzene from the individual stock solutions. Calibration standard mixtures are prepared at concentrations such that a 2 µl spike is sufficient to give alanyte levels reported in Table 3.

7.2.3. A stock solution of internal standard (1,8-DNN, Table 3) is prepared in benzene at a concentration such that a 2 µl spike on a "real" sample trap gives 50 ng of internal standard.

7.3. GC column sppecifications.

7.3.1. An HT-5 column (5% phenyl polycarborane siloxane, Scientific Glass Engineering, Inc., Austin, TX) or an equivalent column (such as DB-5 or Rtx-5) can be used in this method.

7.3.2. A short (12 m), narrow bore (0.22 mm ID) column, coated with thin film (0.1 µm) is recommended for the optimal analytical separation and recovery of explosives.

7.3.3. With vacuum compensation, a flow rate of greater than 1 ml/min is recommended for GC/NICI analysis. Slower flow rates or longer column length can lead to degradation of PETN on the column.

7.4. Instrumental conditions for GC/NICI analysis.

7.4.1. Hewlett-Packard 5989B Gas Chromatograph/Mass Spectrometer with dual ion source or an equivalent instrument can be used for this method.

7.4.2. The analysis should be performed in electron capture negative ion chemical ionization mode.

7.4.3. With methane as reagent gas, the source pressure (typically 0.8 to 1.6 Torr) should be optimized for maximum sensitivity and mass accuracy (see Quality Control section for details).

7.4.4. Source temperature should be set at 150 ^{0}C, quadrupole temperature at 100 ^{0}C.

7.4.5. With the electron energy set at 230 eV, and emission current at 300μA, full scan (50-550 amu) spectral data should be acquired at a rate as predetermined by the manufacture's software.

7.5. Water removal from wet traps.

7.5.1. Traps containing water as the result of sample collection should be purged to constant weight with an ambient temperature stream of dry argon (c.a. 200 mlL/min). Water purge flow should be in the same direction as sample collection flow. Tenax TA has a water breakthrough volume of 65 mL/g at 20 °C.

8. **Calibration and Sample Analysis**

8.1. Spiking internal standard and calibration solutions.

8.1.1. A welded ¼ inch nut was fabricated in-laboratory so that a sorbent trap could be directly connected to the base of the injecton port (Figure 3) of a gas chromatograph (Hewlett Packard 5890 used in the work discusssed here).

8.1.2. On/off valves were attached to the split vent and septum purge exits (Figure 4).

8.1.3. The injection port was heated at 170 °C, the split vent and septum purge exits closed, and the gas flow through the injection port was set at 100 ml/min.

8.1.4. A sorbent trap is attached to the injection port and a *calibration* solution (2 μl) containing internal standard injected through a glass injector liner. The sample is

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purged with 200 mL of helium carrier gas (2 min at 100 mL min⁻¹) to ensure all the analytes were concentrated on the upstream end of the trap.

8.1.5. The trap is removed from the injection port, capped and held for analysis.

8.1.6. Step 7.5.4 can be used to spike internal standard onto a trap that has been used to collect analytes for a "real" sample.

8.2. Calibration.

8.2.1. A five-point calibration curve with replicate measurements at each point should be constructed for each target analyte over a pre-determined concentration range.

8.2.2. In a typical calibration (or sample) analysis, a 2 µl sample of calibration solution (or internal standard solution) should be loaded onto a Tenax TA tube by injection of the sample through a standard injection port (Figure 5) which has been modified as described above.

8.2.3. The trap is purged with 200 mL of helium carrier gas (2 min at 100 mL min⁻¹) to ensure all the analytes concentrate on the upstream end of the trap.

8.2.4. The trap is removed from the injection port, capped and held for analysis.

8.3. Thermal desorption and gas chromatographic analysis.

8.3.1. The injection port temperature should be reduced to less than 50 ^{0}C to allow the Tenax trap to be inserted into the injection port. This step can be facilitated by blowing with a stream of house air or cold nitrogen drawn from a liquid nitrogen dewar (see Hazard section for proper handling of liquid nitrogen).

8.3.2. Prior to inserting the Tenax trap inside the injection port, the split purge valve should be turned off and the split vent should be blocked by closing the on/off valve. The GC oven temperature should be lowered to c.a. 30 °C.

8.3.3. After inserting the Tenax TA tube inside the injection port (Figure 6), the port should be closed securely and heated to 170 °C. The carrier flow during desorption process should be increased to at least 12-13 ml/min (with vacuum compensation) for GC/NICI analysis. Failure of the carrier flow to reach the pre-determined rate may indicate a leak in the system.

8.3.4. Thermal desorption should be allowed to proceed for 7 minutes, including 3.5 - 4 minutes injector heating time.

8.3.5. The GC oven should be held near 30 °C during thermal desorption to allow the desorbed target analytes to be condensed and focused at the head of the column.

8.3.6. A two-tiered oven program is recommended for optimal separation of the analytes. When the oven is turned on after the desorption, the initial oven temperature (70 °C) will be attained quickly and should be held for a total of 5 min, not including the warm-up time. The oven is then heated to 185 °C, at a rate of 7 °C/min, in the first tier of the program. The oven temperature should then be increased to 280 °C at a rate of 20 °C/min and held at 280 °C for 10 minutes.

9. Data reduction

9.1.1. Calibration curves can be constructed by plotting the concentration ratios of target analytes relative to the internal standard versus their corresponding area ratios.

9.1.2. The integrated area for each analyte should be obtained from an integrated area of the most abundant ion as analyzed by TD/GC/NICI.

9.1.3. The concentration and area ratios can be used to perform linear regression with, or without, intercept to obtain the Response Factor (slope) and correlation coefficient (r) for each target analyte. The Response Factor is defined as the slope of a plot of Area Ratio (ordinate) versus the Concentration Ratio (abscissa). A standard t-test should be used to determine whether the intercept from each calibration curve is significantly different from zero at the 95% confidence level.

9.1.4. The quantity of explosives collected on the Tenax trap can be calculated using one of the following equations:

Quantity (ng) = (Area ratio /Response factor) * (ng of IS spiked)
Quantity (ng) = [(Area ratio – Intercept)/Response factor]*(ng of IS spiked)

10. Sample Collection

10.1. Air samples to be analyzed are collected on Tenax TA adsorbent traps with the aid of a mechanical sampling pump.

10.2. Sampling rates and volumes are dictated by the pump capacity/speed and analyte concentration. Safe sampling volumes (explosives breakthrough volumes on

Tenax TA) are not determined. The low vapor pressures of most explosives make it reasonable to expect that the breakthrough volumes would be quite large.

10.3. Samples are collected without the aid of upstream water management (drying agent) traps.

10.4. After collection, sample traps are stored below 0 °C, and warmed to ambient temperature before spiking with internal standard and subsequent analysis.

11. **Quality Control and Quality Assurance**

11.1. Reproducibility of Tenax trap preparation.

11.1.1. The reproducibility of tube preparation should be ensured for Tenax traps prepared in-house, so that the method accuracy and precision are independent of the sorbent tube preparation.

11.1.2. Three traps, randomly selected from the batch of prepared traps, should be spiked with quantities of explosive greater than 75 ng per component and analyzed for their recovery by TD/GC/NICI.

11.1.3. The spiking and analysis should be repeated in triplicate for each trap, and the average analyte recovery based on an established calibration should be determined. The resulting average recovery should be used to calculate the percent relative standard deviation (%RSD) for each trap.

11.1.4. In general, the mean %RSD should be less than 10% for every analyte as listed in Section 2.5 except for PETN. The thermal instability of PETN will result in a higher %RSD (ca. 15%) for this analyte.

11.2. Column Performance.

11.2.1. By using a short capillary column coated with thin film thickness of moderately polar liquid phase (as recommended in Section 7.3.1), baseline separation should be achieved for all the explosives and related compounds including internal standards.

11.2.2. The order of gas chromatographic elution should be as the following (Figure 7): beyond 7 min, [1] DEGN (13.7 ng), [2] NG (16.1 ng), [3] 2,6-DNT (14.5 ng), [4] 1,3-DNB (16.1 ng), [5] 2,5-DNT (14.5 ng), [6] 2,4-DNT (21.7 ng), [7] 2,3-DNT (13.4 ng), [8] 3,4-DNT (12.4 ng), [9] TNT (14.3 ng), [10] 1,3,5-TNB (16.1 ng),

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[11] PETN (48.3 ng), [12] 4-A-2,6-DNT (17.4 ng), [13] RDX (77.3 ng), [14] 2-A-4,6-DNT (16.0 ng), [15] tetryl (16.1 ng), and [16] 1,8-DNN (IS, 50.0 ng)..

11.2.3. If the gas chromatograph is equipped with an electronic pressure control for maintaining a constant column flow throughout the entire GC program, then a baseline resolution should be achieved with the pair of 2,3- and 2,4-dinitrotoluene isomers. Otherwise, partial resolution of this pair of isomers is acceptable.

11.2.4. As part of QA/QC procedure, GC column performance (peak resolution, elution order, etc.) check should be carried out prior to calibration and/or sample analysis.

12. Method Performance

12.1. The following procedure is recommended for checking the NICI tune:

12.1.1. 300 pg of decachlorobiphenyl and 100 pg of hexachlorobenzene should be injected onto the column and analyzed with the following temperature program: 100 °C hold for 3 min, ramp to 280 °C at 20 °C/min.

12.1.2. The resulting spectra should be compared with reference spectra (Reference 13.8): for decachlorobiphenyl m/z 498 (100%), 464 (>27%), and 430 (>5%); for hexachlorobenzene m/z 284 (100%), 286 (>70%), 250 (>10%).

12.1.3. If the relative abundance is out of range, the instrument should be re-tuned. The GC peak height for both compounds acquired over the full scan (100-550 amu) must be at least three times greater than the noise.

12.1.4. The eight most abundant ions (m/z) and their relative intensities (% abundance) for the nine target analytes are listed in Table 2.

13. Precision and Linearity of the Methodology

13.1. Tenax traps should be spiked in triplicate with calibration standard mixtures that include 15 target analytes and an internal standard at minimal of five concentrations ranging from approximately 0.3 to 500 ng/tube, as shown in Table 3.

13.2. The spiked Tenax traps should be analyzed TD/GC/NICI. The spiking and analysis should take place in a random order over a period of a week.

13.3. The relative response and relative concentration for each analyte at each concentration should be calculated:

Relative response = Area of analyte/Area of IS

Relative concentration = Concentration of analyte/Concentration of IS

13.4. The relative responses obtained from triplicate analysis for each analyte at each concentration should be used to calculate the average response, which in turn should be used to calculate the percent relative standard deviation (%RSD).

13.5. The ranges for %RSD should be comparable to those listed in Table 3 for the specified loading range.

13.6. A linear regression curve should be fitted between the arrays of relative responses and relative concentrations to construct a five-point calibration.

13.7. The linearity (r^2) resulting from linear regression should be comparable to those listed in Table 4 for the specified loading range.

14. Limit of Detection and Limit of Quantitation

14.1. The data used for the precision and linearity should also be employed to

determined the method limit of detection (LOD) and limit of quantitation (LOQ)

for each analyte.

14.2. The LOD should be determined as:

LOD (ng) = 3.3 (SD/S) * (ng of IS spiked)

Where SD is the standard deviation of the intercept and S is the slope of the

calibration curve

14.3. The LOQ should be determined as :

LOQ = 10 (SD/S) * (ng of IS spiked)

14.4. The resulting LOD and LOQ for each analyte determined for TD/GC/NICI should be comparable to those listed in Table 4.

15. References

15.1. Michael E. Sigman, Cheng-Yu Ma and Ralph H. Ilgner "Performance Evaluation of an In-Injection Port Thermal Desorption/Gas Chromatographic/Negative Ion

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Chemical Ionization Mass Spectrometric Method for Trace Explosive Vapor Analysis" *Analytical Chemistry*, **2001**, *73(4)*, 792.

15.2. Michael E. Sigman and Cheng Yu Ma. "Detection Limits for GC/MS Analysis of Organic Explosives, *J. Forensic Sci.*, in press, 2001.

15.3. Michael E. Sigman, and Cheng Yu Ma, "In-Injection Port Thermal Desorption for Explosives Trace Evidence Analysis" Analytical Chemistry, *71*, pp 4119-4124, 1999.

15.4. Hewlett Packard Gas Chromatograph and Mass Spectrometer 5989B system Operating Manuals, including HP 5989B Hardware Manual (Copyright 1993, Part # 05989-90071), MS Chemstation User's Guide (Copyright 1993, HP G 1034C, Part # HP G 1034-90043).

15.5. Hewlett-Packard 5890 Series II Gas Chromatograph Operating Manual (Copyright 1989, Part # 05890-90260).

15.6. Microsoft Excel 97 Spreadsheet Program (Microsoft, 1997, Part # 98722).

15.7. Lockheed Martin Energy Research Corporation ORNL Explosives Program, number ORNL-SH-P05, Rev. 1, dated 10/5/98.

15.8. E. A. Stemmler; R. A. Hites, *Electron Capture Negative Ion Mass Spectra of Environmental Contaminants and Related Compounds*, PP 39, and 253, VCH Publisher, New York, 1988.

Table 1. List of analytes (explosives and explosives related compounds), abbreviations and CAS numbers.

No.	Analyte	Abbreviation	CAS Number
1	Diethyleneglycol dinitrate	(DEGN)	[693-21-0]
2	Trinitroglycerin	(NG)	[55-63-0]
3	2,6-Dinitrotoluene	(2,6-DNT)	[606-20-2]
4	1,3-Dinitrobenzene	(1,3-DNB)	[99-65-0]
5	2,5-Dinitrotoluene	(2,5-DNT)	[619-15-8]
6	2,4-Dinitrotoluene	(2,4-DNT)	[121-14-2]
7	2,3-Dinitrotoluene	(2,3-DNT)	[602-01-7]
8	3,4-Dinitrotoluene	(3,4-DNT)	[610-39-9]
9	2,4,6-Trinitrotoluene	(TNT)	[118-96-7]
10	1,3,5-Trinitrobenzene	(1,3,5-TNB)	[99-35-4]
11	Pentaerythritol tetranitrate	(PETN)	[78-11-5]
12	4-Amino-2,6-dinitrotoluene	(4-A-2,6-DNT)	[1946-51-0]
13	Hexahydro-1,3,5-trinitro-1,3,5-triazine	(RDX)	[121-82-4]
14	2-amino-4,6-dinitrotoluene	(2-A-4,6-DNT)	[35572-78-2]
15	Methyl-2,4,6-trinitrophenylnitramine	(Tetryl)	[479-45-8]
16	1,8-Dinitronaphthalene (*internal standard, IS*)	(1,8-DNN)	[606-37-1]

Table 2. The eight most abundant NICI ions (m/z) and their relative intensities (% abundance) for the 15 target analytes.

Analyte	m/z	% Abundance	Analyte	m/z	% Abundance
DEGN	62	100	NG	62	100
	46	91		59	23
	242	71		58	14
	258	20		57	3
	90	10		71	2
	93	10		73	1
	103	9			
	109	5			
2,6-DNT	182	100	1,3-DNB	168	100
	183	9		182	9
	152	8		169	8
	184	1		152	4
	166	1		137	3
	153	1		170	1
	150	1		164	1
	164	1		183	1
2,5-DNT	182	100	2,4-DNT	182	100
	183	9		152	12
	152	3		183	8
	166	2		166	2
	184	1		165	1
	153	1		150	1
				184	1
				164	1
2,3-DNT	182	100	3,4-DNT	182	100
	152	9		183	9
	166	1		152	6
	184	1		184	1
	153	1		166	1
				153	1
				134	1
TNT	227	100	1,3,5-TNB	213	100
	197	22		183	18
	210	16		214	8
	228	10		196	3
	198	3		182	2
	167	3		181	2
	195	2		215	2
	229	2		184	2

Table 2 continued.

Analyte	m/z	% Abundance	Analyte	m/z	% Abundance
PETN	62	100	4-A-2,6-DNT	197	100
	46	19		198	10
	101	16		167	2
	99	9		181	2
	84	8		199	1
	240	6			
	256	5			
	378	5			
RDX	102	100	2-A-4,6-DNT	197	100
	129	80		198	10
	176	30		181	3
	268	25		167	1
	46	17		199	1
	324	15		180	1
	130	4		179	1
	103	4			
Tetryl	242(M-46+2)	100	1,8-DNN	218	100
	212	34		220	17
	225	28		219	14
	243	9		172	3
	226	7		188	1
	191	5			
	196	3			
	208	3			

Table 3. Thermal desorption efficiency and reproducibility determined by TD/GC/NICI.

Analyte	Precision (%RSD)			Calibration Range (ng)
	Median	Mean	Range	
DEGN	19	24	13-49	1.20-77.28
NG	28	56	8-173	0.36 - 90.90
2,6-DNT	8	8	2-15	0.32 - 81.82
1,3-DNB	7	9	1-17	0.36 - 90.90
2,5-DNT	7	10	1-25	0.32 - 81.82
2,4-DNT	6	7	2-18	0.48 - 122.72
2,3-DNT	6	9	3-25	0.30 - 75.46
3,4-DNT	7	7	2-15	0.28 - 70.00
TNT	10	14	7-28	0.32 - 80.90
1,3,5-TNB	10	20	6-54	0.36 - 90.90
PETN	16	34	3-87	4.16 - 272.72
4-A-2,6-DNT	14	26	6-90	1.54 - 98.18
RDX	35	38	6-69	1.70 - 436.36
2-A-4,6-DNT	16	28	7-87	0.38 - 90.90
TETRYL	30	50	7-173	0.38 - 90.90

Table 4. Limit of detection and limit of quantitation determined by TD/GC/NICI.

Analyte	Calibration Linearity (r^2)	Method LOD (ng)	Method LOQ (ng)
DEGN	0.9712	7.82	26.07
NG	0.9672	9.65	32.17
2,6-DNT	0.9854	0.10	0.33
1,3-DNB	0.9875	2.57	8.57
2,5-DNT	0.9481	2.25	7.50
2,4-DNT	0.9339	3.30	11.00
2,3-DNT	0.9136	2.29	7.63
3,4-DNT	0.9426	1.58	5.27
TNT	0.9668	2.32	7.73
1,3,5-TNB	0.9639	4.86	16.20
PETN	0.9711	25.97	86.57
4-A-2,6-DNT	0.9836	4.63	15.43
RDX	0.9549	35.41	118.03
2-A-4,6-DNT	0.9856	5.14	17.13
TETRYL	0.9706	9.70	32.33

Figure 1: Steel tubing is packed with a bed of 0.1-0.3 g of Tenax TA sorbent . Silanized glass wool (Supelco Inc.) is used hold the sorbent bed in the tube.
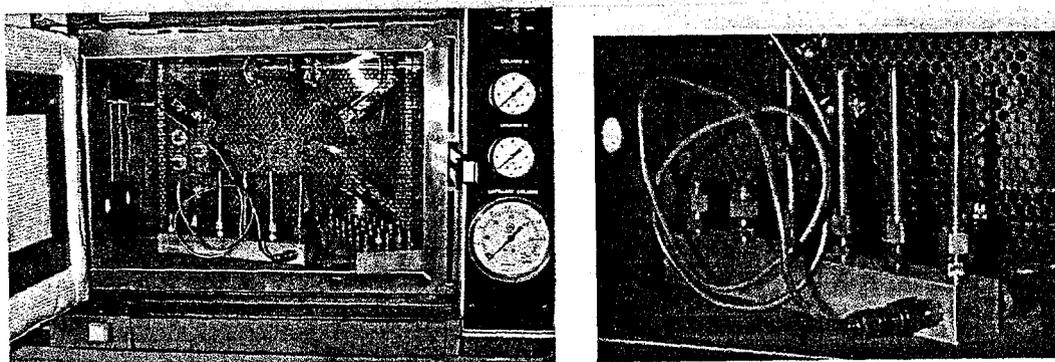
Figure 2: The sorbent-containing tubes are heated at 325 ^{0}C under a flow of helium at 100 ml/min for at least 2 hours.
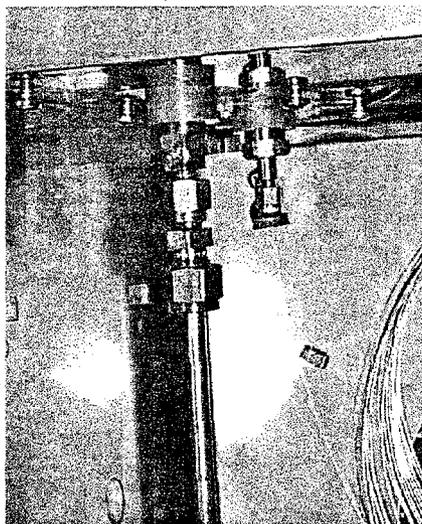
Figure 3: A welded ¼ inch nut was fabricated in-laboratory so that a sorbent trap could be directly connected to the base of the injecton port.
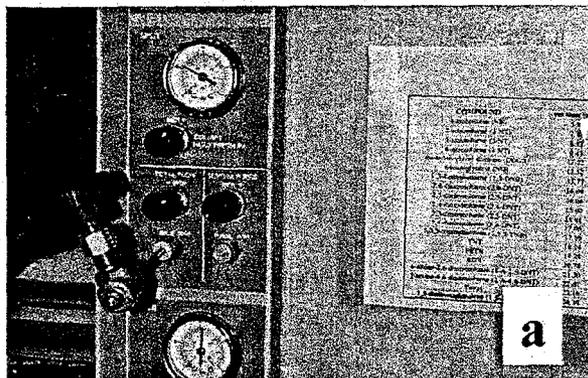
Figure 4: On/off valve attached to split-vent on the front of the gas chromatograph. Septum purge is also capped.
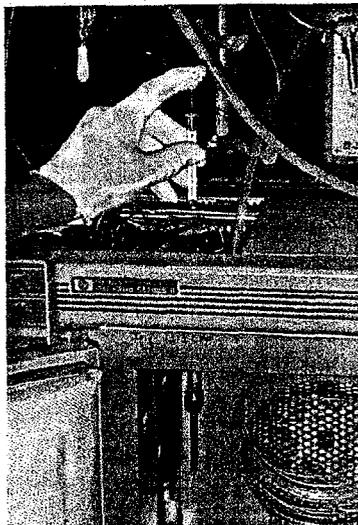
Figure 5: Deposition of calibration solution (or IS) on a Tenax trap by injection through a modified injection port.
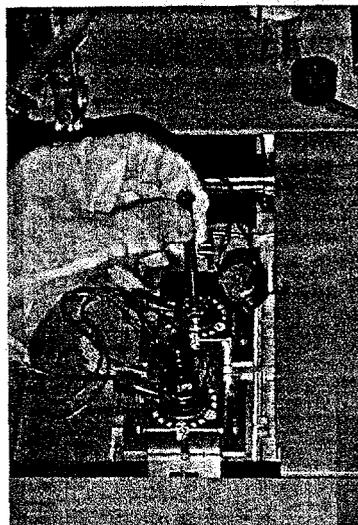
Figure 6: Inserting a Tenax trap inside a GC injection port for thermal desorption.
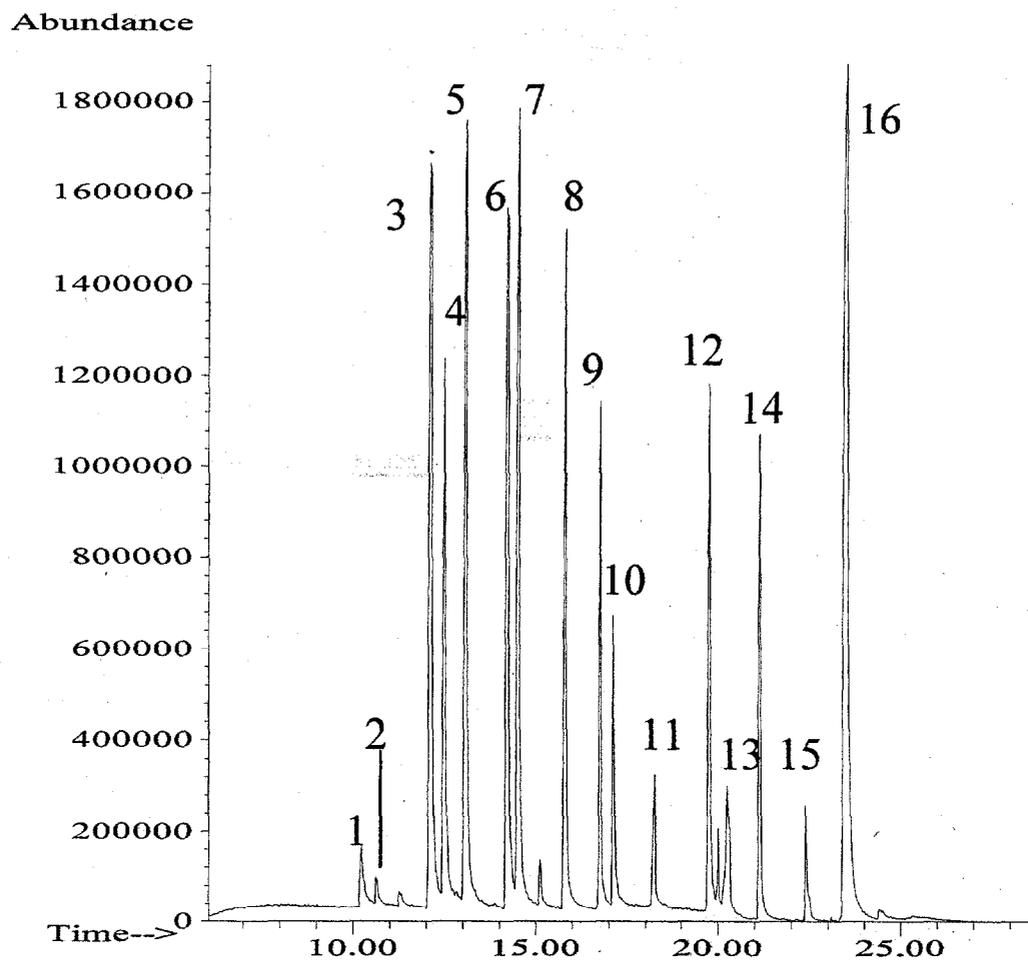
Figure 7: In-injection port thermal desorption GC/NICI chromatogram of a standard mix of explosives. The peak identities (starting at 10 min) are listed in Table 1.

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