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NAVAL SURFACE WEAPONS CENTER WHITE OAK LAB SILVER SP--ETC F/G 19/1  
ANALYSIS OF 2,2',4,4',6,6'-HEXANITROSTILBENE (HNS) BY HIGH PERF--ETC(U)  
MAR 78 E G KAYSER  
NSWC/WOL/TR-77-154

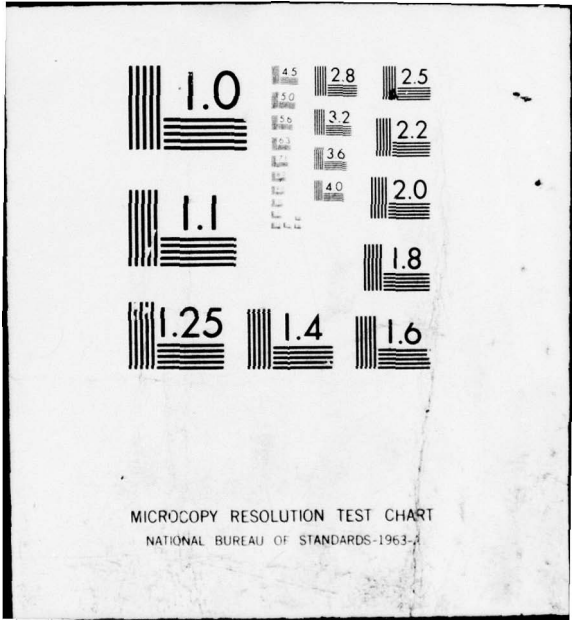
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**ANALYSIS OF 2,2', 4,4', 6,6' -  
HEXANITROSTILBENE (HNS) BY HIGH  
PERFORMANCE LIQUID CHROMATOGRAPHY**

BY ELEANORE G. KAYSER

RESEARCH AND TECHNOLOGY DEPARTMENT

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A high performance liquid chromatographic (HPLC) method for the determination of HNS is described. A reverse-phase micro column was used with a methanol/distilled water mobile phase in a ratio of 2:3 by volume. This method is capable of separating all known by-product impurities resulting from the preparation of HNS by the "Shipp Process". Assays of HNS and 2,2',4,4',6,6'-Hexanitrobibenzyl (HNBiB), the major impurity, can be obtained in less than 40 minutes. Accurate analyses ( $\pm 2\%$ ) of HNS and all known by-products can be made in about 90 minutes.		

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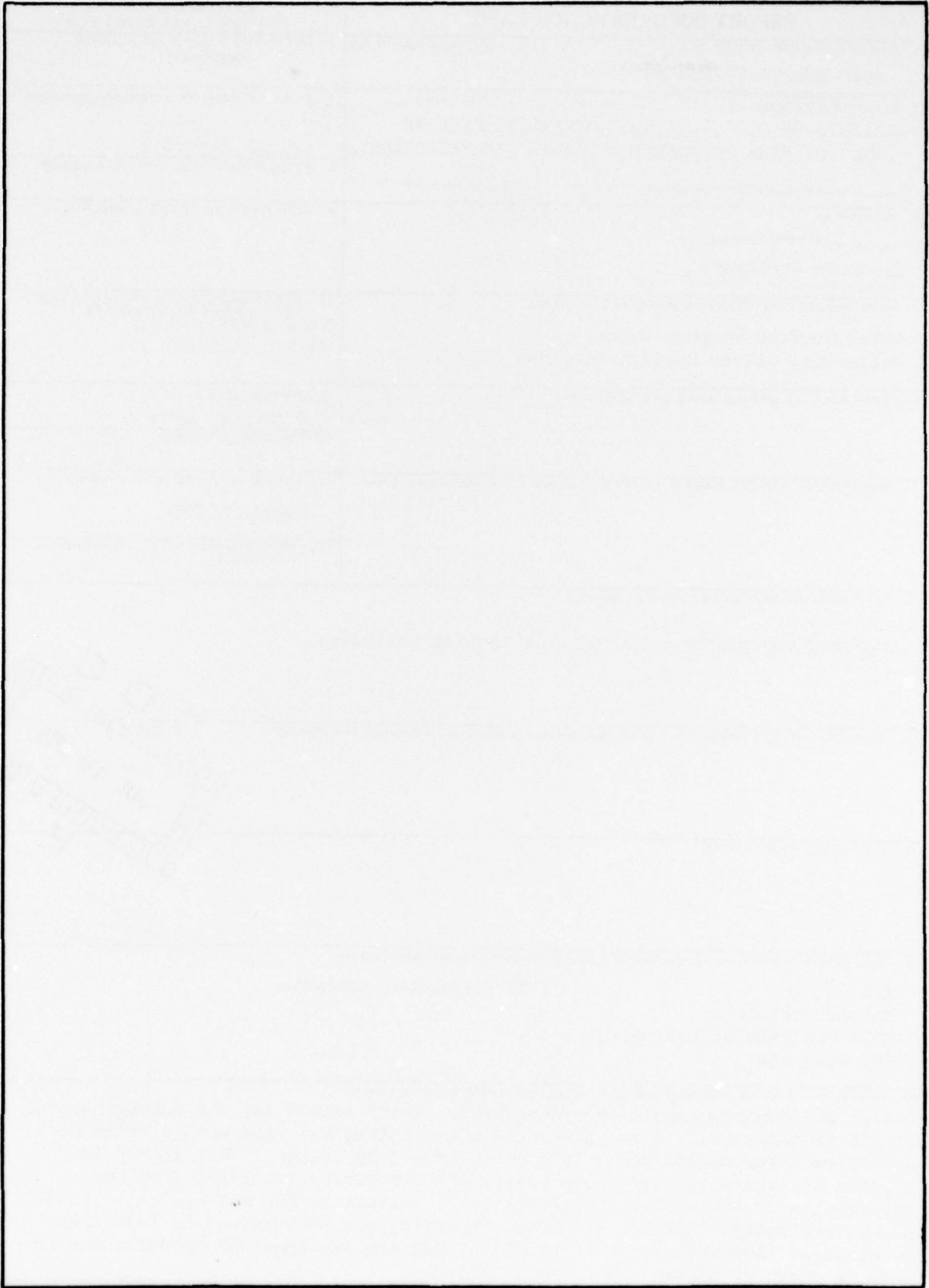
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## SUMMARY

This report describes an analytical procedure for the assay and analysis of HNS-I and HNS-II using HPLC. Assays of HNS and HNBiB can be obtained in less than 40 minutes. Accurate analyses of HNS and all other known by-products can be made in about 90 minutes. This work was carried out under two tasks, WR12KA, "HNS Explosive Evaluation" for the National Aeronautics and Space Administration, Lyndon B. Johnson Space Center, Houston, and CR12JC, "HNS Explosive Characterization" for the Naval Air Systems Command.

*Julius W. Enig*

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## CONTENTS

	Page
I. INTRODUCTION.....	5
II. EXPERIMENTAL.....	7
III. RESULTS AND DISCUSSION.....	9
IV. CONCLUSION.....	10

## TABLES

<u>Table</u>	<u>Page</u>
1 Compounds Identified as Products of the TNT-NaClO Reaction.....	11
2 Average Retention Times and Response Factors for Compounds Isolated from the TNT-NaClO Reaction.....	13
3 Quantitative HNS Data.....	14

## ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	HPLC Trace of DMSO.....	15
2	HPLC Trace of a Synthetic Mixture of Potential HNS Impurities.....	16
3	HPLC Trace of a Synthetic Mixture of Potential HNS Impurities.....	17
4	HPLC Trace of a Synthetic Mixture of Potential HNS Impurities.....	18
5	HPLC Trace of a Synthetic Mixture of Potential HNS Impurities.....	19



## I. INTRODUCTION

In recent years, research in polynitroaromatic chemistry at the Naval Surface Weapons Center, White Oak Laboratory, has produced several new high energy compounds that have found numerous applications in military ordnance items and in the National Aeronautics and Space Administration (NASA) space programs including the Space Shuttle. One of these compounds is 2,2',4,4',6,6'-hexanitrostilbene (HNS) which has been shown to be an exceptionally good explosive for high temperature applications in detonation transfer lines and cutting devices using flexible linear shaped charges.

HNS is commercially prepared by reacting 2,4,6-trinitrotoluene (TNT) with a 5 percent aqueous sodium hypochlorite solution (Clorox<sup>1</sup>) in the presence of tetrahydrofuran and methanol<sup>2</sup>. The yield of HNS from this reaction is less than 50 percent. The remainder of the TNT is converted to a red-tar fraction of which 80 weight percent has been separated and identified using a combination of thin layer chromatography (TLC) and nuclear magnetic resonance spectroscopy (NMR), Table 1<sup>3</sup>.

Two types of HNS are used; HNS-I (crude HNS) which is the direct reaction product with minimal purification, and HNS-II (recrystallized HNS). HNS-II has the larger particle size (therefore the smaller surface area) and is required by the Navy Specification WS-5003 to have a bulk density above 0.45 g/cm<sup>3</sup>,

<sup>1</sup>Commercial household bleach (5.25% sodium hypochlorite) manufactured by the Clorox Company.

<sup>2</sup>Shipp, K. G., and Kaplan, L. A., "Reaction of  $\alpha$ -Substituted Polynitrotoluenes," J. Org. Chem., Vol. 31, 857 (1966).

<sup>3</sup>Kayser, E. G., this Center, unpublished results.

whereas, HNS-I particles are small (large surface area) and have a bulk density ranging from 0.15 to 0.35 g/cm<sup>3</sup><sup>4</sup>. Methods for converting HNS-I to HNS-II include (a) extraction with a mixture of acetonitrile and toluene<sup>5</sup>, and (b) recrystallization by a nitric acid process<sup>6a</sup>, or from fuming nitric acid<sup>6b</sup>, or dimethylformide<sup>5</sup>.

Although HNS-I is used in a variety of military applications, no general assay method has been available due to (a) the relative insolubility of HNS in most solvents, and (b) the presence within the HNS crystals of trapped by-products from the TNT-NaClO reaction, which interfere with the usual spectrophotometric methods of analysis.

The absorption spectrum of HNS is nonspecific and difficult to distinguish from the absorption contributed by the known red-tar components. HNS can be analyzed spectrophotometrically by the ethylene diamine (EDA)/dimethylsulfoxide (DMSO) procedure<sup>7</sup> only after a preliminary purification, for example, by TLC.

At the present time the HNS specification WS-5003 establishes the only requirements for Navy procurement of HNS. The chemical and physical tests include melting point, melting point range, vacuum stability, surface moisture, volatile material, water soluble material, and bulk density. Performance tests include sensitivity and output compared to a "Standard Comparison Sample" (SCS) of either HNS-I or HNS-II. No assay or analysis of material purity is included.

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<sup>4</sup>O'Keefe, D. M., "Digestion as a Process Aid for Hexanitrostilbene," Sandia Corporation Report SAND-0330, Feb 1977.

<sup>5</sup>Taylor, F., and Oesterling, R. E., "Heat Resistant Explosives, XX. Production of Grade II HNS," NOLTR 65-142, 26 Aug 1965.

<sup>6</sup>(a) Such a process has been used at Teledyne McCormick Selph and at Chemtronics, Incorporated; (b) J. Dacons, unpublished results.

<sup>7</sup>Glover, D., and Kayser, E. G., "Quantitative Spectrophotometric Analysis of Polynitroaromatic Compounds by Reaction with Ethylenediamine," Analytical Chemistry, Vol. 40, 2055 (1968).



An HPLC method<sup>8</sup> for analyzing purified HNS samples and also a semi-quantitative method for analyzing crude samples has been developed using three columns packed with Corasil II<sup>9</sup> and a solvent system consisting of 20%  $\rho$ -dioxane/80% n-heptane. This report describes an HPLC method for the quantitative analysis of both HNS-I (crude HNS) and HNS-II (recrystallized HNS) using a reverse-phase micro column. The method does not require gradient elution and can differentiate HNS from any of the other known by-products (Table 1) formed in the "Shipp Process". This method is recommended for inclusion in the Navy HNS specification.

## II. EXPERIMENTAL

### A. Apparatus

A model ALC 202 Liquid chromatograph equipped with a model 6000 solvent delivery system and a U6K high pressure loop injector (Waters Associates, Milford, Massachusetts) was used for the analyses and the eluent was continuously monitored with a 254 nm UV detector. Qualitative data were obtained on a Waters C-18 reverse-phase Bondapak column (3.2 mm O.D. x 61 cm) with a 37-75 micron particle size. All quantitative data were obtained using a Whatman micro column. A Whatman HPLC guard column containing CO:PELL<sup>®</sup> ODS pellicular material was used as a pre-column to a Whatman Partisil<sup>™</sup> bonded reversed-phase column (25 cm x 4.6 mm x 1/4"). The typical efficiency of this analytical micro column is 18,000 plates per meter with octadecyl silane (ODS) functionality. The UV absorption signals were recorded on a Omniscribe strip chart recorder set at 0.5 cm/minute.

<sup>8</sup>Stull, T. W., "Synthesis of High Purity HNS," MHSMP-75-37, Sep 1975.

<sup>9</sup>Waters Associates, Inc., Framingham, Maine.

<sup>®</sup>Registered trademark of Whatman, Inc.

<sup>™</sup>trademark of Whatman, Inc.

#### B. Reagents and Chromatographic Conditions

The mobile phase consisted of methanol (Fisher Scientific, certified A.C.S. grade) and distilled water in a ratio of 2:3 by volume. Pressures at the column head were about 900 psi, 2,000 psi, and 3,000 psi for flow rates of 1 ml, 2 ml, and 3 ml per minute respectively. Column temperature was about 25°C or room temperature. All solvents were filtered twice through a #42 Whatman paper filter prior to use in the HPLC. Samples of 10 to 100  $\mu$ l were introduced by syringe injection through the septum.

#### C. Preparation of Standards and Samples

The compounds (Table 1) isolated from the red-oil produced in the preparation of HNS by the "Shipp Process" were recrystallized and purified by TLC prior to their use as HPLC standards. After drying, approximately 1 mg of each standard was accurately weighed using a Mettler Micro Gram-atic balance and placed in a 10 ml volumetric flask. Each standard and sample was diluted to volume with Fisher A.C.S. grade DMSO, just prior to analysis, to prevent possible solution photolysis. An HPLC trace of DMSO (Figure 1) was run to determine its retention time and the presence of any peaks which could interfere with this method of HNS analysis. p-dioxane was also tried as an HNS solvent but was found unsatisfactory. All sample and standard solutions were prefiltered through a 0.5  $\mu$ m filter (Waters Clarification Kit) prior to injection in the HPLC.

#### D. Calculations

The concentration of each material was determined by peak height as well as peak area. As expected, the percentage accuracy for the area method was found to be greater than that of the peak height method. Thus, for quantitative work the area method was used. The response factor for each compound was determined by manually integrating the area of the chromatographic peak of a standard of known concentration. These relative response factors are summarized in Table 2.

## III. RESULTS AND DISCUSSION

In preliminary experimental work with a Waters C-18 reverse-phase column, relatively good separations of hexanitrobibenzyl (HNBiB), hexanitrostilbene (HNS), trinitrobenzyl chloride (PiCH<sub>2</sub>Cl), and trinitrotoluene (TNT) were obtained (Figure 2). However, trinitrobenzene (TNB) was only partially resolved, and trinitrobenzaldehyde (PiCHO) and trinitrobenzyl alcohol (PiCH<sub>2</sub>OH) remained unresolved.

An improved separation of potential HNS impurities<sup>10</sup> was obtained with a Whatman micro column. As seen in Figures 3 and 4 as well as in Table 2, Picric Acid (PiOH), PiCHO, dinitroanthranil (Anil), TNT, Picryl chloride (PiCl), PiCH<sub>2</sub>Cl, HNS, and HNBiB are well separated and the peaks are easily quantified. However, the separation of PiCOOH and TNB was less satisfactory. The PiCOOH eluted just prior to the TNB peak resulting in only a partial resolution of the two components. At a flow rate of 1.0 ml per minute, the average retention time for the last compound, HNBiB, was about 165 minutes.

At a flow rate of 2 ml per minute (Figure 4) separation was still sufficient for quantitation of all compounds except, as expected, PiCOOH and TNB. The average retention time of HNBiB was decreased by about 75 minutes leading to a more rapid method of assay and analysis. When the flow rate was increased to 5 ml per minute, only TNT, PiCH<sub>2</sub>Cl, HNS, and HNBiB could be quantitatively determined (Figure 5), but the total sample retention time was decreased to about 40 minutes.

Decreasing or increasing the concentration of methanol in the mobile phase had the same effect on sample separation as a

---

<sup>10</sup>Synthetic mixture containing all compounds listed in Table 1 except chloropicrin.



flow rate change (i.e., increasing the methanol concentration decreased the average retention time of the sample peaks while a decrease in methanol concentration increased the retention time.) The most complete sample separation in the shortest time resulted with the 40:60 methanol/water eluent at a flow rate of 2 ml per minute. Data obtained from several production grade samples of HNS-I and HNS-II are given in Table 3. The percent composition of each HNS sample was obtained (a) from previously determined response factors, and (b) from the area of the peak alone without the use of comparison standards. For the production grade HNS samples tested, our results show that the area method can be used to determine with sufficient accuracy the percent composition of both HNS-I and HNS-II.

The HNS-I samples were found to contain about 5% HNBiB with minute amounts (<0.1 to 0.2%) of TNB and  $\text{PiCH}_2\text{OH}$ . Since only a partial separation of TNB and  $\text{PiCOOH}$  was achieved with HPLC, the presence of trace quantities of  $\text{PiCOOH}$  was determined by TLC; none was found. The HNS-II samples contained between 0 to 3% HNBiB and only possible trace quantities (<0.1%) of the other impurities. Neither TNT nor  $\text{PiCH}_2\text{Cl}$  was found in any of the production grade HNS samples tested.

#### IV. CONCLUSION

The method detailed in this report permits rapid quantitative determinations of HNS and its primary contaminant HNBiB. Sensitivity is sufficient for reliable determinations in small volumes (50 - 100  $\mu\text{l}$ ) of DMSO solutions. This method can also be used to quantitatively determine all other known by-products resulting from the preparation of HNS by the "Shipp Process".

TABLE I  
COMPOUNDS IDENTIFIED AS PRODUCTS OF THE TNT- $\text{NaClO}$  REACTION

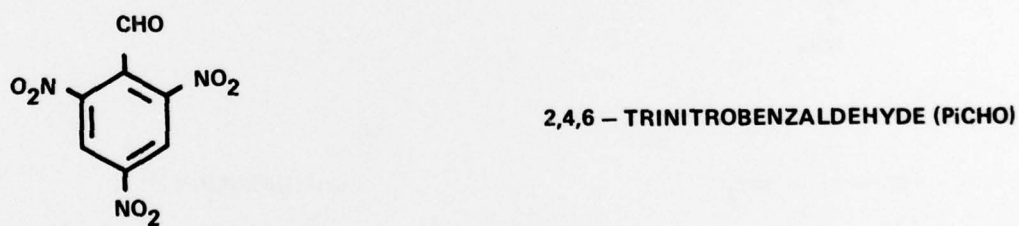
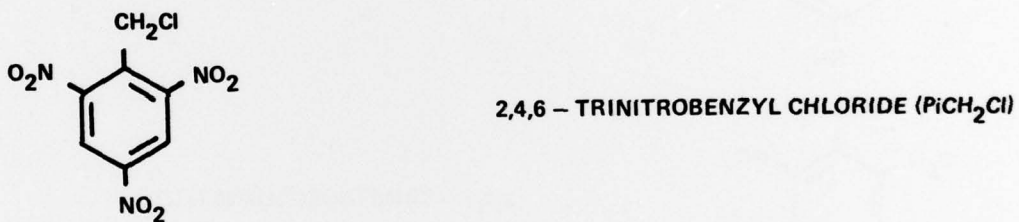
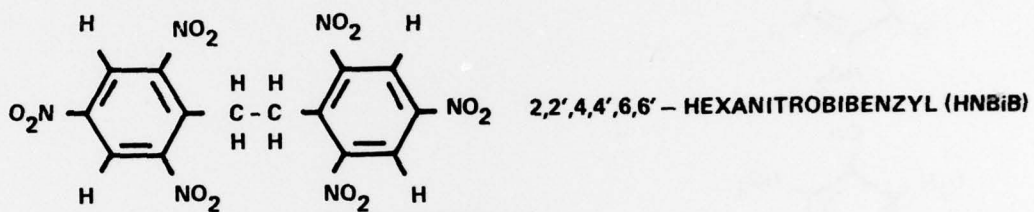
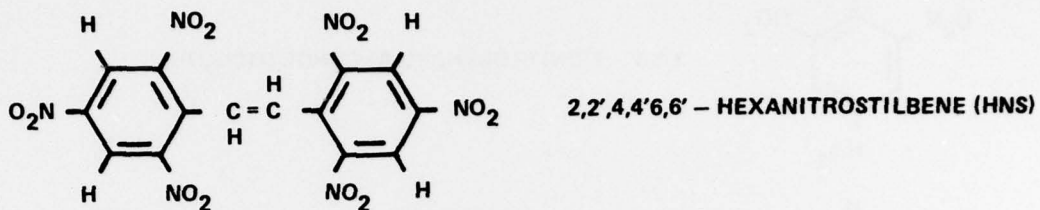
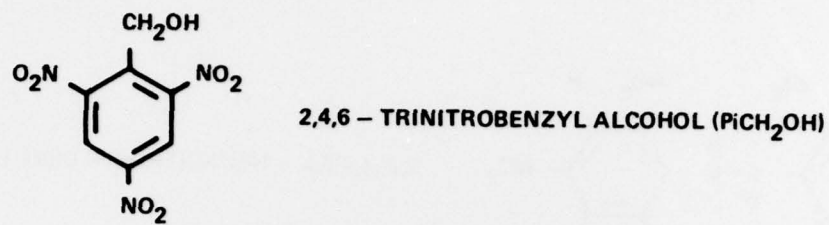


TABLE I (CONTINUED)



Pi = 2,4,6 - TRINITROPHENYL

(a) DETERMINED BY GAS CHROMATOGRAPHY (GC), ALL OTHER COMPOUNDS DETERMINED BY NMR.



TABLE 2  
 AVERAGE RETENTION TIMES AND RESPONSE FACTORS  
 FOR COMPOUNDS ISOLATED FROM THE TNT- $\text{NaClO}$  REACTION

COMPOUND	RETENTION TIME AT RATE OF			AVERAGE RESPONSE FACTOR <sup>(a)</sup>	AVERAGE RESPONSE FACTOR (NORMALIZED TO TNB = 1.0)
	1 ML/MINUTE	2 ML/MINUTE	3 ML/MINUTE		
PiOH	2.3	1.1	0.9	(b)	(b)
DMSO <sup>(c)</sup>	3.5	1.9	1.2	(b)	(b)
PiCH <sub>2</sub> OH	9.4	4.8	3.4	764,300	0.82
PiCHO	14.2	7.1	4.9	620,150	0.67
PiCOOH	16.8	8.3	6.0	(b)	(b)
TNB	17.4	8.5	6.0	927,100	1.00
ANIL	23.1	12.1	(b)	(b)	(b)
PiCl	31.8	15.9	10.9	(b)	(b)
TNT	33.6	17.3	12.2	808,950	0.87
PiCH <sub>2</sub> Cl	53.3	26.3	18.0	779,700	0.84
HNS	98.7	60.0	36.0	875,000	0.94
HNBiB	162.5	90.0	60.0	886,400	0.95

CONDITIONS:

COLUMN           PARTISIL-10 ODS-2 (REVERSED PHASE)  
 RANGE            08  
 SAMPLE VOLUME   5-50 $\mu$ l  
 SAMPLE           HPLC STANDARDS  
 SOLVENT          40% METHANOL/60% WATER

COLUMN PRESSURES

1 ML/MINUTE     900-1000 PSI  
 2 ML/MINUTE     2000 PSI  
 3 ML/MINUTE     3000 PSI

- (a) RESPONSE FACTOR DETERMINED FROM THE PEAK AREA OF THE STANDARD  
 (b) NOT DETERMINED  
 (c) HNS SOLVENT

TABLE 3  
QUANTITATIVE HNS DATA

EXPLOSIVE TYPE	IDENTIFICATION (a)	% COMPOSITION DETERMINED FROM RESPONSE FACTORS			
		% HNS	% HNBiB	% TNB (b)	% PiCH <sub>2</sub> OH
HNS-I	I.D. # 2297 (UTC LOT #3)	95.3	4.6	0.1	(c)
HNS-I	I.D. # 2294 (UTC LOT #1)	94.9	4.9	0.1	0.1
HNS-I	(d)	94.6	5.3	(c)	0.1
HNS-I	(d)	94.9	4.9	0.1	(c)
HNS	I.D. # 2276 (NSWC 96-8433-50 (e))	100.0	0.0	0.0	0.0
HNS-II	(d)	98.2	1.8	0.0	(c)
HNS-II	I.D. # 2299 (SILAS MASON HANGER - PANTEX LOT 6348-074-001)	100.0	0.0	(c)	(c)
HNS-I	(BRITISH - PERME 343 )	97.3	2.5	0.2	0.1

EXPLOSIVE TYPE	IDENTIFICATION (a)	% COMPOSITION DETERMINED FROM PEAK AREA			
		% HNS	% HNBiB	% TNB (b)	% PiCH <sub>2</sub> OH
HNS-I	I.D. # 2297 (UTC LOT #3)	94.4	5.5	(c)	0.1
HNS-I	I.D. # 2294 (UTC LOT #1)	94.6	5.2	0.1	0.1
HNS-I	(d)	94.8	5.1	0.1	0.1
HNS-I	(d)	95.1	4.9	(c)	0.1
HNS	I.D. # 2276 (NSWC 96-8433-50 (e))	100.0	0.0	0.0	0.0
HNS-II	(d)	98.3	1.7	(c)	(c)
HNS-II	I.D. # 2299 (SILAS MASON HANGER - PANTEX LOT 6348-074-001)	100.0	0.0	(c)	(c)
HNS-I	(BRITISH - PERME 343 )	97.2	2.5	0.1	0.1

(a) I.D. # IS NSWC IDENTIFICATION NUMBER

(b) UNRESOLVED PEAK AREAS OF TNB AND PiCOOH (NO PiCOOH DETECTED BY TLC)

(c) TRACE = < 0.1 %

(d) NSWC LABORATORY SAMPLE

(e) J.C. DACONS, THIS LABORATORY, UNPUBLISHED RESULTS-FINE PARTICLE SIZE  
HNS MADE FROM HNS-II BY PRECIPITATION FROM HNO<sub>3</sub>

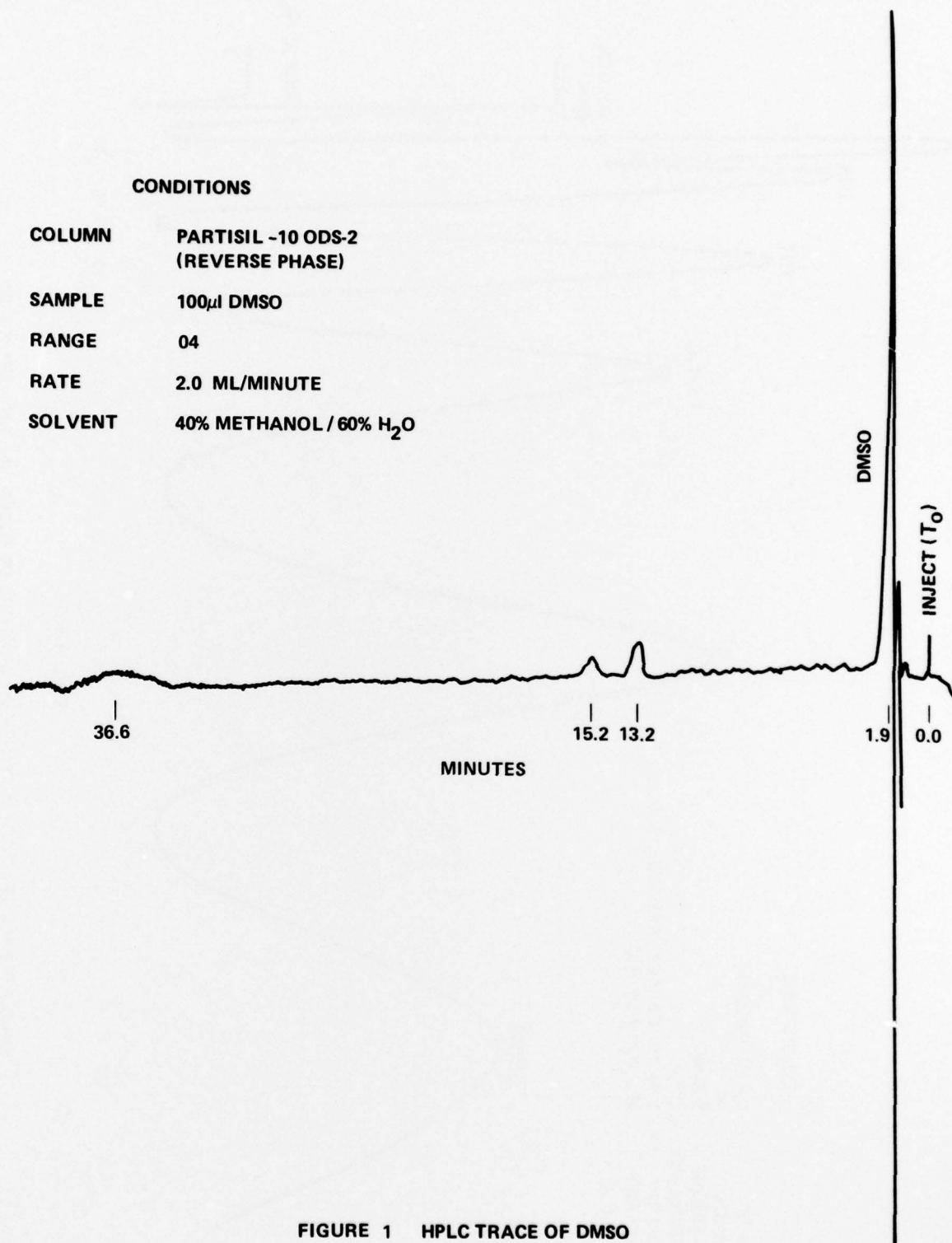
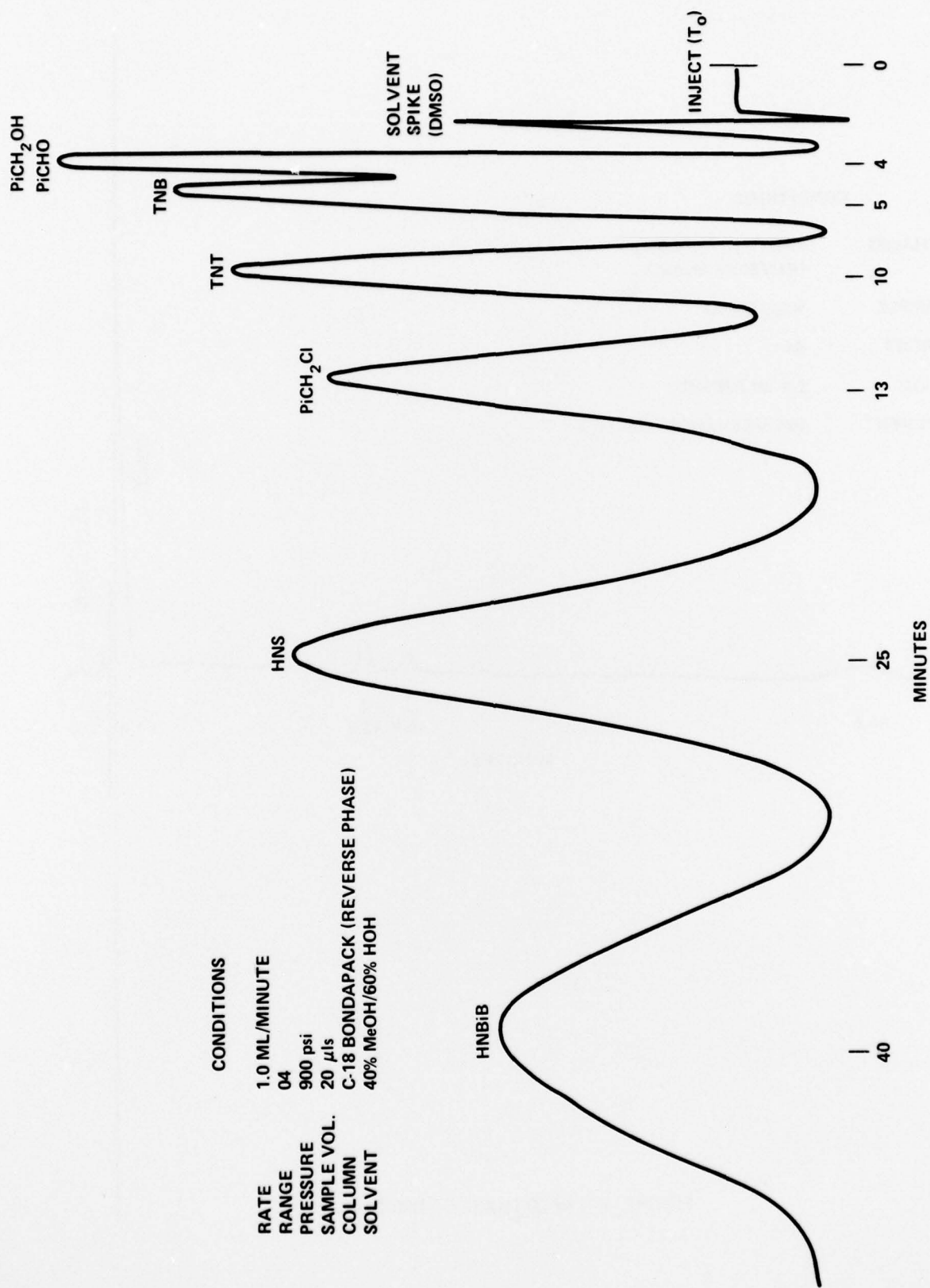


FIGURE 1 HPLC TRACE OF DMSO

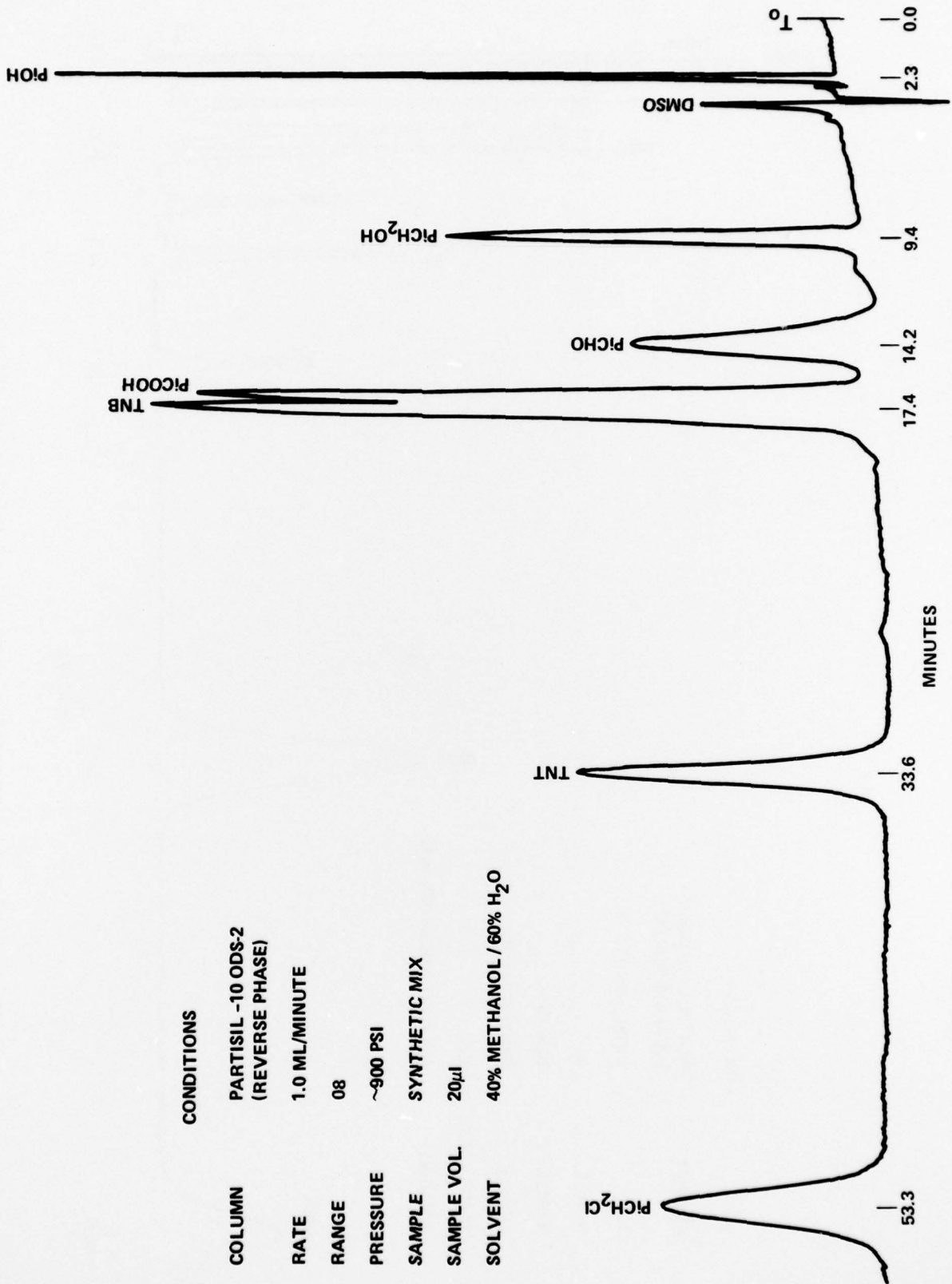


**CONDITIONS**

RATE 1.0 ML/MINUTE  
 RANGE 04  
 PRESSURE 900 psi  
 SAMPLE VOL. 20 µls  
 COLUMN C-18 BONDAPACK (REVERSE PHASE)  
 SOLVENT 40% MeOH/60% HOH

FIGURE 2 HPLC TRACE OF A SYNTHETIC MIXTURE OF POTENTIAL HNS IMPURITIES

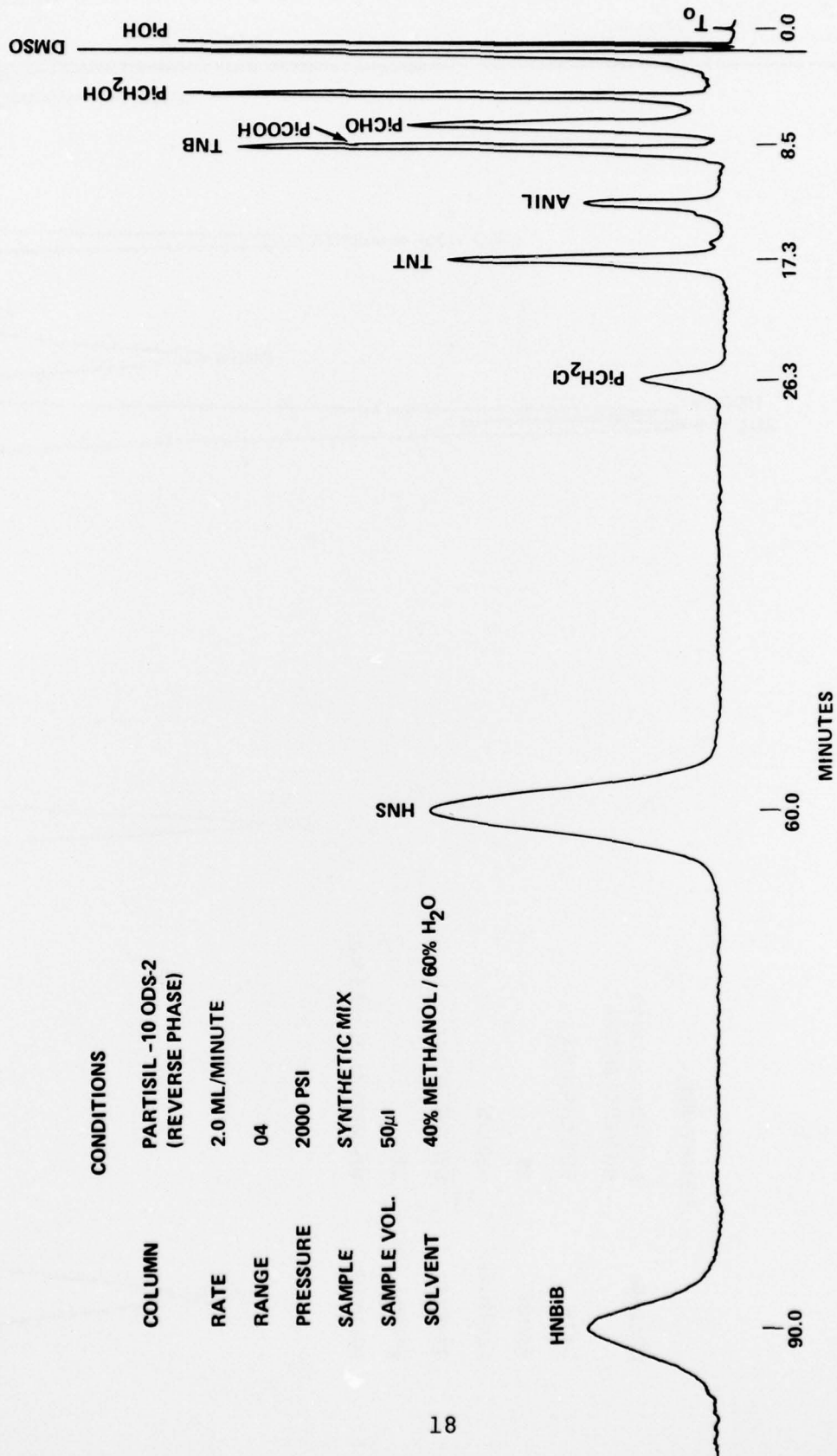




**CONDITIONS**

<b>COLUMN</b>	PARTISIL -10 ODS-2 (REVERSE PHASE)
<b>RATE</b>	1.0 ML/MINUTE
<b>RANGE</b>	08
<b>PRESSURE</b>	~900 PSI
<b>SAMPLE</b>	SYNTHETIC MIX
<b>SAMPLE VOL.</b>	20 $\mu$ l
<b>SOLVENT</b>	40% METHANOL / 60% H <sub>2</sub> O

FIGURE 3 HPLC TRACE OF A SYNTHETIC MIXTURE OF POTENTIAL HNS IMPURITIES



**CONDITIONS**

**COLUMN** PARTISIL-10 ODS-2 (REVERSE PHASE)  
**RATE** 2.0 ML/MINUTE  
**RANGE** 04  
**PRESSURE** 2000 PSI  
**SAMPLE** SYNTHETIC MIX  
**SAMPLE VOL.** 50µl  
**SOLVENT** 40% METHANOL / 60% H<sub>2</sub>O

FIGURE 4 HPLC TRACE OF A SYNTHETIC MIXTURE OF POTENTIAL HNS IMPURITIES



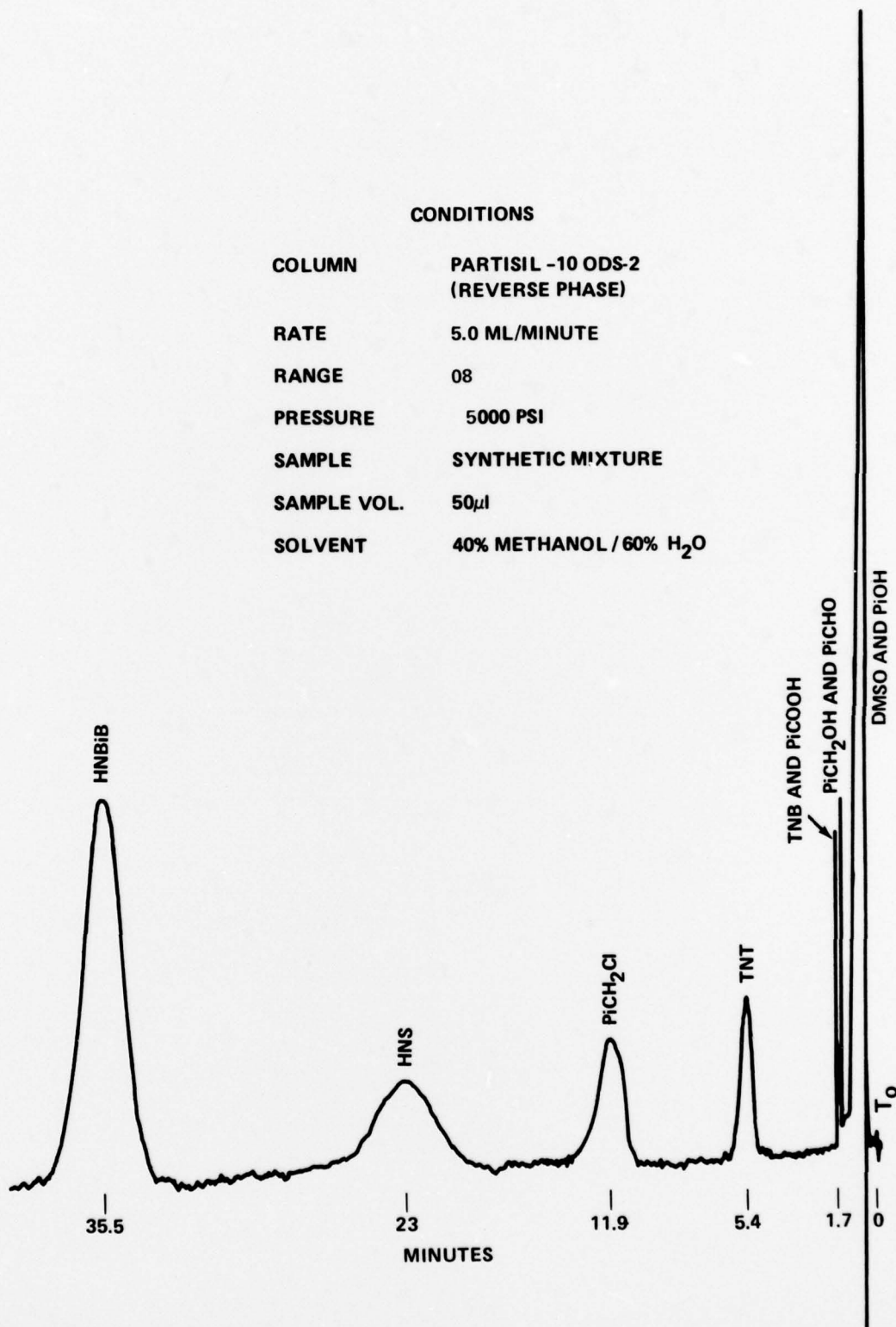


FIGURE 5 HPLC TRACE OF A SYNTHETIC MIXTURE OF POTENTIAL HNS IMPURITIES

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