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TECHNICAL REPORT RD-PR-86-3

SELECTIVE DETECTION OF MINIMUM SMOKE STABILIZERS:
FINAL REPORT

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MAY 28 1987
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Propulsion Directorate
Research, Development and Engineering Center

JULY 1986



U.S. ARMY MISSILE COMMAND

Redstone Arsenal, Alabama 35898-5000

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 This project has been accomplished as part of the US Army Material Testing Technical Program. Its objective was the timely establishment of test methods, procedures, or prototype equipment (in mechanical, chemical, or nondestructive testing) to enable efficient inspection methods for material produced by AMI.

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 This report describes the development of an electrochemical detector for a flame retardant in double base propellant. The detector is used to detect the presence of the flame retardant in the propellant. The detector is used to detect the presence of the flame retardant in the propellant. The detector is used to detect the presence of the flame retardant in the propellant.

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 Per Dr. James G. Carver, Army Missile,
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I. INTRODUCTION

The Armed services lately have been expressing an increased interest in the safe life of propellants. The Army has an Independent Review Team examining the status of gun and missile propellants, in storage, for present safety and remaining safe life. The Army has also placed a high priority on the evaluation of the safety of the M55 chemical rocket. There are over 600,000 rounds stored in several locations around the U. S. and in the Pacific which are planned for disposal. However, before these rounds can be shipped to a central location for destruction, it is necessary that their safety be evaluated.

Of the 21 Army missile systems currently in development or fielded, 12 use double base, composite modified double base, or minimum signature propellants as the main grain. Most of the 21 systems use 1 of these propellants in their ignitors or gas generators. Many other systems with these propellants are in storage under the demilitarization account awaiting disposition.

The safe life as well as the usable service life of these missiles is directly dependent upon the levels of the various stabilizers and their degradation products present in the propellant. Therefore, it is critical that these levels be accurately known at all times. Gas chromatography (GC) and liquid chromatography (LC) are the two procedures currently used in the propellant industry to determine stabilizer levels. The most frequently used detector in GC is the Flame Ionization Detector (FID), and the most favored LC detector is the UV detector. Both of these systems are used because of their ability to detect almost every ingredient in the propellant. This is a problem, however, with the complex mixtures being used in the newer minimum signature propellant formulations. It can be difficult to completely resolve the stabilizers from the other ingredients in these complex mixtures.

A liquid chromatographic detector, that has received some attention in forensic science, is the electrochemical detector. This detector has the ability to be selective and very sensitive. In the oxidative mode, it can be adjusted to detect only aromatic amines and phenols at ng levels. The most frequently used stabilizers in the United States are of this type. Therefore, a study to evaluate the use of the electrochemical detector in the detection of propellant stabilizers was conducted.

II. EXPERIMENTAL

Experiments were conducted using the liquid chromatographic system which consisted of a Waters Associates 720 Controller, an M6000A pump, and a Wisp 710B Automatic Sample Injector, a Perkin-Elmer LC85 variable wavelength UV detector with autocontrol, and a BAS LC-4B dual amperometric detector.

Separations were performed on a DuPont Zorbax 5 μm ODS 25 cm X 4.6 mm column or a Waters C-18 NOVAPAC 5 μm column. In both cases, a Brownlee Labs 3 cm X 4.6 mm ODS Guard Column was placed in front of the analytical column. The eluting solvents compositions varied with the propellant, but were mixtures of acetonitrile, methanol, tetrahydrofuran, and 0.1 M sodium acetate adjusted to a pH of 5 with perchloric acid. All ingredients were reagent grade. Solvents were UV grade HPLC quality, and water was distilled, deionized, and filtered.

The UV detector was typically set to 214 nm. The electrochemical detector could be arranged so that the working electrodes were parallel or in series. Most of this work was performed with the series conformation. The working electrodes were dual glassy carbon. The potentials were set relative to a silver/silver chloride reference electrode.

The analog output from each detector was digitized and sent to a Hewlett-Packard 3357 lab automation system for integration and data reduction.

Samples of propellant were prepared in two similar procedures. The propellant was hand chopped to a coarse powder. In one procedure the propellant powder (25 to 100 mg) is accurately weighed into a 10 mL test tube. The internal standard is then accurately weighed into a small aluminum pan and placed in the test tube. Five to seven mL of a 50 percent ACN solution is then added and the sample is sonicated with a microprobe for 10-15 min. In the alternate procedure 25 mg of the propellant powder was accurately weighed into a 5 mL vial. The internal standard was accurately weighed into a small aluminum pan and placed in the vial. Four milliliters of dichloroethane was pipeted into the vial. The vial was capped and the propellant soaked overnight.

Once the propellant had been extracted by one of the above procedures, the supernatant liquid was pipeted into a 5 mL syringe fitted with a 0.45 μm disposable filter and filtered into a sample vial.

III. RESULTS

One of the problems encountered when selecting the conditions to be used with an electrochemical (EC) detector was the choice of the mobile phase. For the detector to function properly, the eluting solvent must conduct electricity. This usually requires a minimum ion concentration of 0.05 M in the solvent. Thus, for a 50 percent organic phase solution the aqueous phase must be 0.1 M. The ions used are typically buffered salts of acetic, phosphoric, or citric acids. These give pH values within the safe operating limits of silica based columns. If a solution is not consumed in a few days, it should be discarded since it can act as a growing medium for biological species. Sodium acetate was selected for this study since it has a low UV cutoff and solutions have a longer shelf life than the phosphate and citrate salts.

Solutions prepared from sodium acetate and glacial acetic acid were found to give noisy background and a high UV cutoff. A solution of 0.1 M sodium acetate adjusted with perchloric acid to a pH of 5 was found to give the best results.

Solutions of the 32 propellant ingredients listed in Table 1 were prepared in acetonitrile. The EC detector was set to its highest recommended oxidative potential of 1.20 V and each ingredient was eluted through the system. As shown in Table 1, only the stabilizers which have an amine or hydroxyl on an aromatic ring are detected in the oxidative mode at 1.20 V. As a point of interest, when the upstream electrode is set to -0.9 V, (the reductive mode) and the down stream electrode to +1.0 V most of the materials with nitro groups could also be detected.

The response of the EC detector to a substance is dependent on the concentration of the substance, the half-wave potential of the substance and the potential of the working electrode. For a given concentration of a substance the response vs potential curve looks much like a pH curve for titration of a monoprotic acid. There is a portion of the curve below the threshold potential where no response above the background current is detected. Above the threshold potential the response increases as the oxidative potential is increased until a plateau is obtained. Applying higher potentials generally has little effect on the response to the ingredient but does increase the background current. The potential midway up the response curve is the halfwave potential.

The ingredients which responded to the EC detector at 1.20 V were analyzed at a series of oxidative potentials between 0.6 V and 1.20 V. The normalized response vs potential curves are given in Figures 1, 2, and 3. As shown in these figures, with careful selection of the oxidative potential it is possible to detect the parent stabilizer and not its degradation products. For example, in Figure 3 at 0.80 V diphenylamine is detected while nitroso DPA, 2-NDPA, 4-NDPA, and the dinitro and trinitro derivatives do not respond in the electrochemical detector. It is also evident that if the original stabilizer is 2-NDPA it can be detected at 0.9 V or 1.0 V while the dinitro- and trinitro-DPA derivatives do not respond. This might become useful if resolution becomes a problem.

TABLE 1. Detectability of Propellant Ingredients

Ingredient	UV (214 nm)	EC (1.20 V)
Resorcinol	Yes	Yes
2 Nitro Resorcinol	Yes	Yes
2,4 dinitro Resorcinol	Yes	Yes
Triacetin	Weakly	No
HMX	Yes	No
RDX	Yes	No
DEGDN	Yes	No
Nitroglycerin	Yes	No
BTTN	Yes	No
MNA	Yes	Yes
Nitroso MNA	Yes	Yes
Acaradite I	Yes	Yes
Acaradite II	Yes	Yes
2,4 DNT	Yes	No
2,6 DNT	Yes	No
2,4,6 TNT	Yes	No
Tetryl	Yes	No
TEGDN	Yes	No
TMETN	Yes	No
Methyl Centralite	Yes	Yes
Ethyl Centralite	Yes	Yes
Diphenylamine	Yes	Yes
Nitroso-DPA	Yes	Yes
2-NDPA	Yes	Yes
4-NDPA	Yes	Yes
2,2' DN-DPA	Yes	Yes
2,4' DN-DPA	Yes	Yes
2,4,4' TN-DPA	Yes	Yes
Methyl phthalate	Yes	No
Ethyl phthalate	Yes	No
Propyl phthalate	Yes	No
Propyl Adipate	Weakly	No

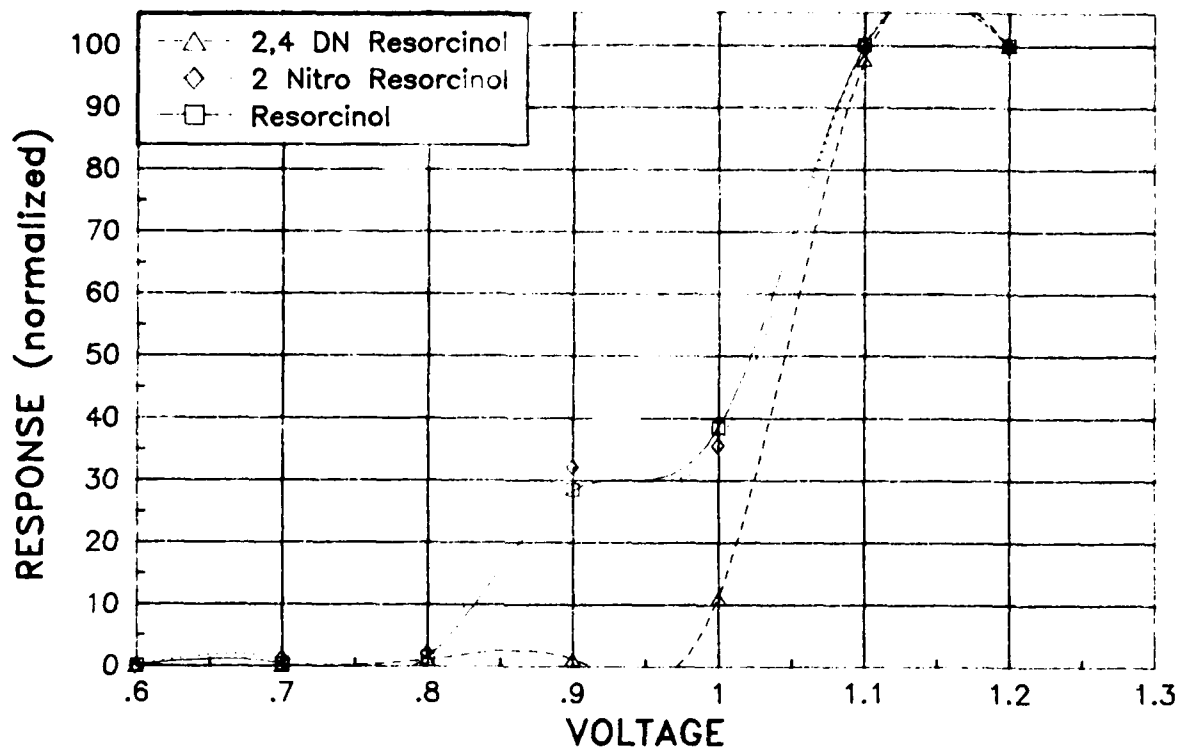


Figure 1. EC data - resorcinol.

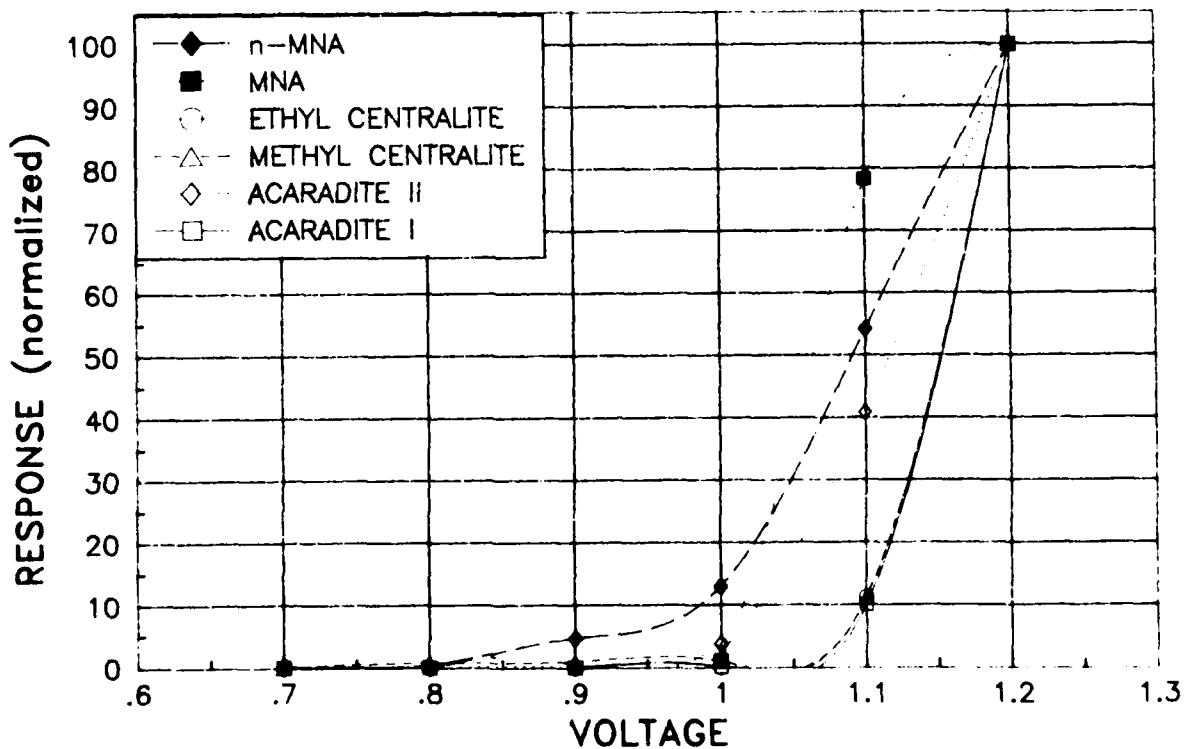


Figure 2. EC data - miscellaneous.

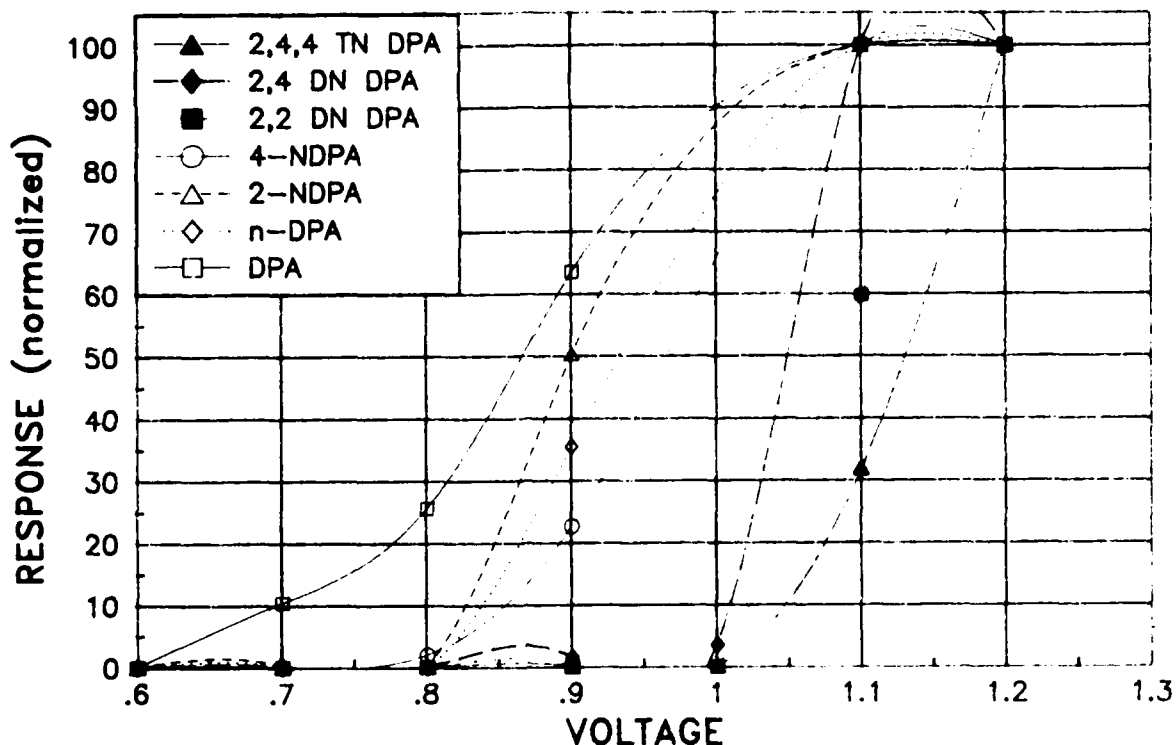


Figure 3. EC data - DPA.

The sensitivity of the EC detector to the stabilizers and their products was examined. Solutions of the 16 materials were prepared at 5 concentrations 1 mg/mL, 100 μ g/mL, 10 μ g/mL, 1 μ g/mL and 0.1 μ g/mL. The last concentration represents 0.001 percent in the propellant sample prepared by standard procedure. The results are shown in Figures 4, 5, and 6. The lowest detectable amount of material was assumed to have a response or peak height of twice the background noise level. Table 2 indicates the estimated minimum detectable amount of each material. The response of the detector is linear up to about 750 nA on the highest setting. Table 2 also indicates the upper limit of linearity for each compound. During this study, good results were not obtained from 2,4 Dinitro resorcinol. After the experiment was completed, it was found that a small baseline disturbance occurred at the same time that the material eluted. This disturbance was probably an injection phenomenon.

Comparison of the lower limits of detectability for the UV detector at 214 nm with the lower limits for the EC detector indicated that the EC was much more sensitive than the UV at 214 nm. This can be a problem if the sample is too concentrated. Originally, during this analyses poor accuracy was obtained because the linearity of the EC detector was exceeded. By preparing samples with one tenth to one half the concentrations typical for UV detection, this problem can be avoided.

(EC 1.20 V)

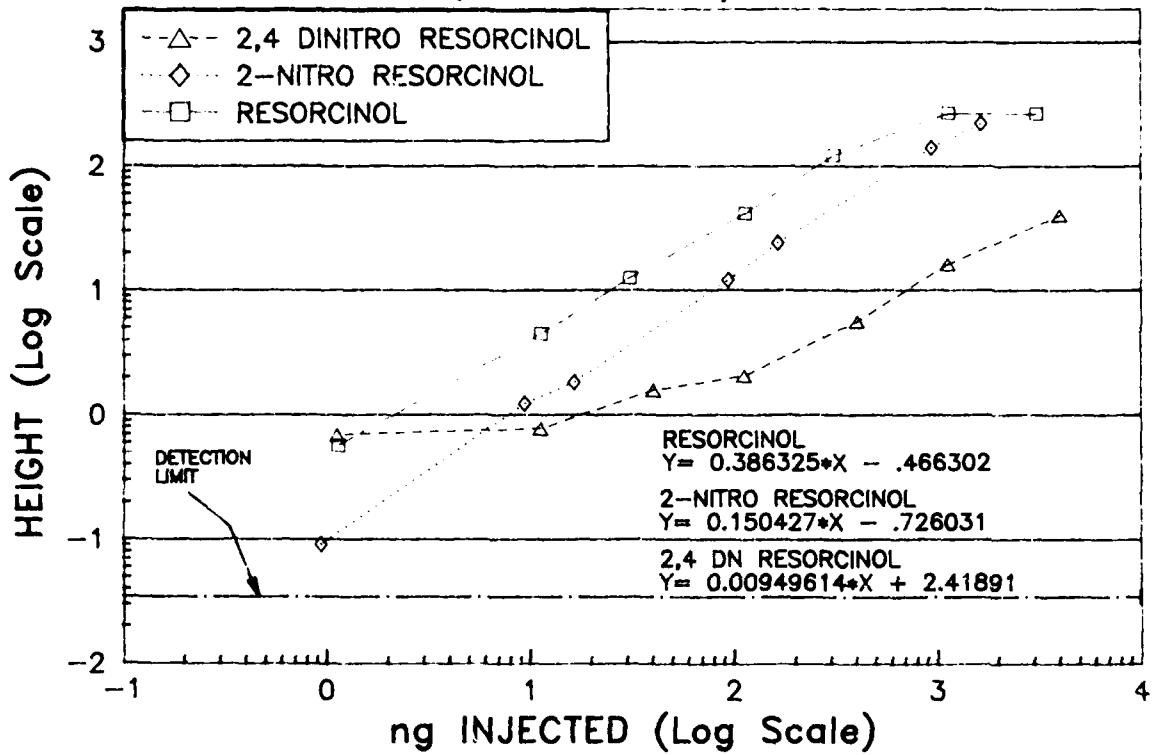


Figure 4. Propellant sample results - resorcinol.

(EC 1.20 V)

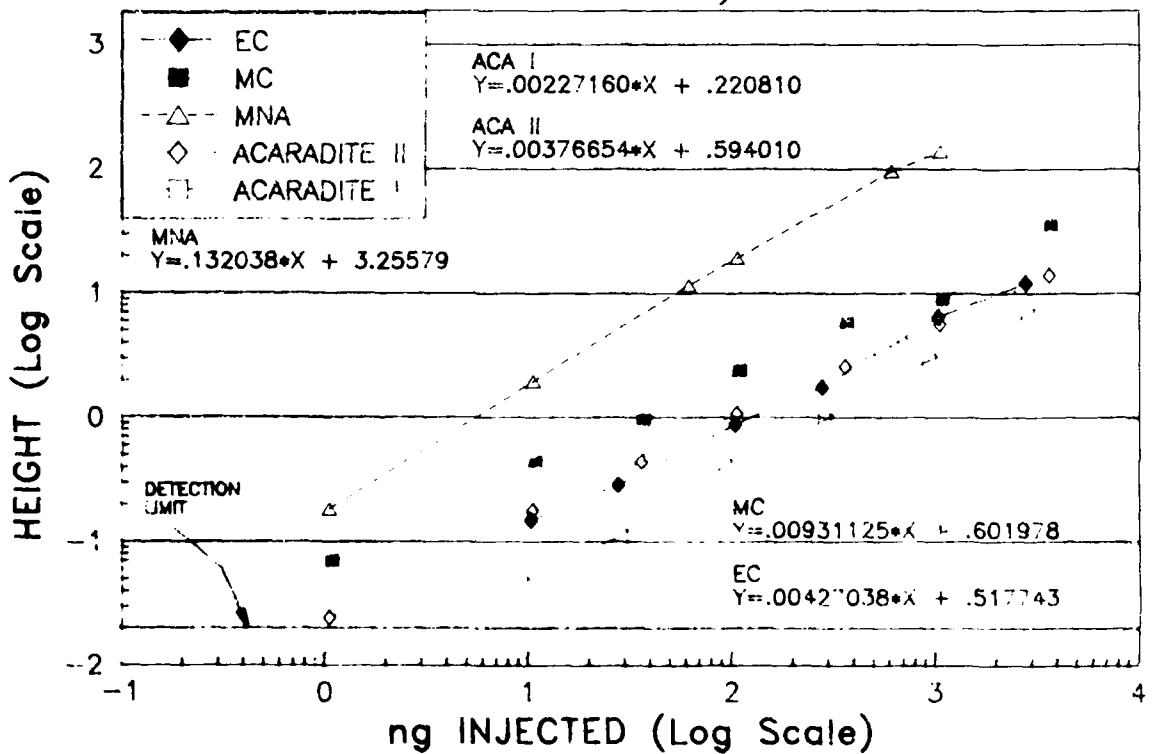


Figure 5. Propellant sample results - miscellaneous.

(EC 1.20 V)

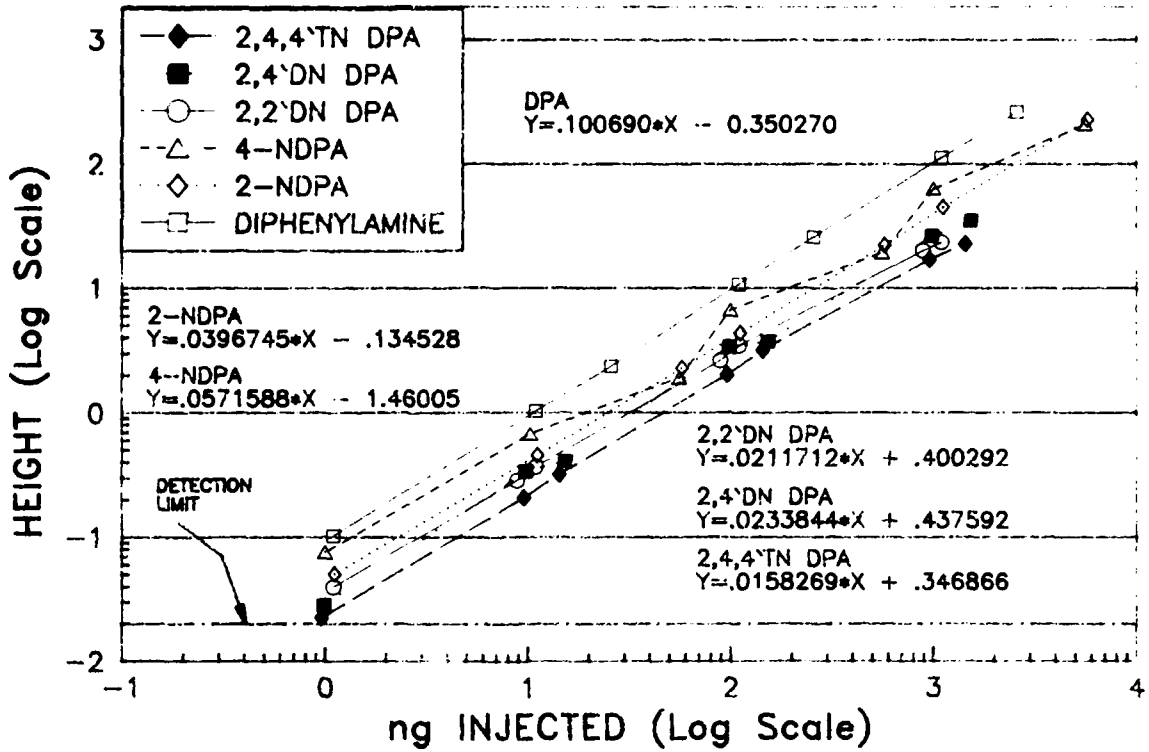


Figure 6. Propellant sample results - DPA.

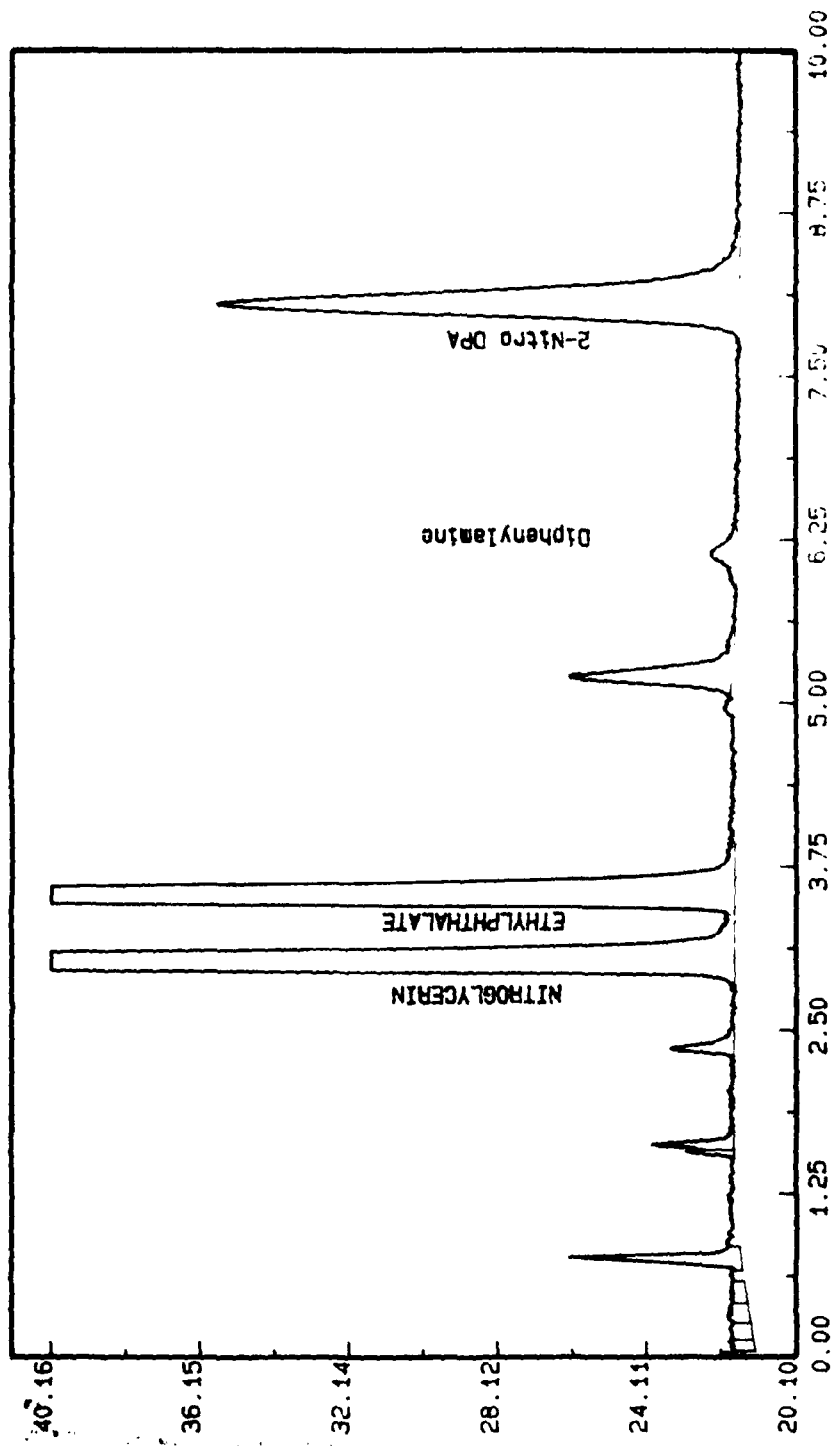
TABLE 2. Linearity and Detection Limits

Material	Linearity (EC 1.2 V)	Detection (EC 1.2 V)	Detection (UV 214 nm)
Resorcinol	260 ng	.05 ng	10 ng
2-Nitro Resorcinol	670 ng	.4 ng	80 ng
Acaradite I	44000 ng	5.0 ng	15 ng
Acaradite II	26000 ng	1.0 ng	20 ng
MNA	730 ng	.1 ng	25 ng
MC	10000 ng	.3 ng	35 ng
EC	23000 ng	1.0 ng	50 ng
DPA	990 ng	.2 ng	20 ng
2-NDPA	2500 ng	.5 ng	32 ng
4-NDPA	1800 ng	.3 ng	58 ng
2,2'DNDPA	4700 ng	.6 ng	10 ng
2,4'DNDPA	4300 ng	.8 ng	50 ng
2,4,4'TNDPA	6300 ng	1.0 ng	15 ng

During the course of this study, it was found that the precision and accuracy of propellant analyses were poor when compared to GC or LC-UV techniques. A study of the sources of error disclosed the following:

- Use of methanol in the eluting solvent, even at less than 10 percent concentrations, slowly desensitizes the glassy carbon electrodes. This changes the response of the detector to the materials. The effect can cause significant changes in calculated stabilizer levels over a period of even 1 hour.
- Flow reproducibility is very critical since the EC detector is a concentration sensitive detector like UV rather than a mass sensitive detector like FID. Higher flow rates, such as 2 to 4 mL/min, were found to result in greater precision than slow flow rates such as 0.5 to 1 mL/min. Proper solvent preparation such as, filtering, mixing, and degassing are also critical to reproducible retention time and area precision.
- When real world samples were used it was found that the propellant matrix can contain ingredients that slowly desensitize the glassy carbon electrodes. The rate is dependent on the sample concentration, volume injected, and the number of injections made. Typically, this required the electrodes to be cleaned once a week, a 5-minute operation. Heavier use, as would be expected in an active QA lab, might require electrode cleaning every other day.
- The precision and accuracy of the procedure was evaluated by analyzing a sample of HYDRA 70 propellant. Figures 7 and 8 represent the typical responses of the UV detector at 214 nm and EC detector at 1.05 V in the chromatographic analysis of HYDRA 70 propellant. Table 3 indicates the results of triplicate analyses of two samples of RAD84K003-018. Within sample precision is good with a standard deviation of 0.019 percent and 0.029 percent and a relative standard deviation of 0.98 percent and 1.44 percent. Between sample precision is also good with a standard deviation of 0.023 percent and a RSD of 1.14 percent. The accuracy is also excellent. Gas chromatographic analysis found a 2-NDPA level of 1.98 percent \pm 0.01, while the LC/EC found 2.00 percent \pm 0.02.

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RETENTION TIME (min.)

Figure 7. Typical responses of LC/UV detector at 214 nm.

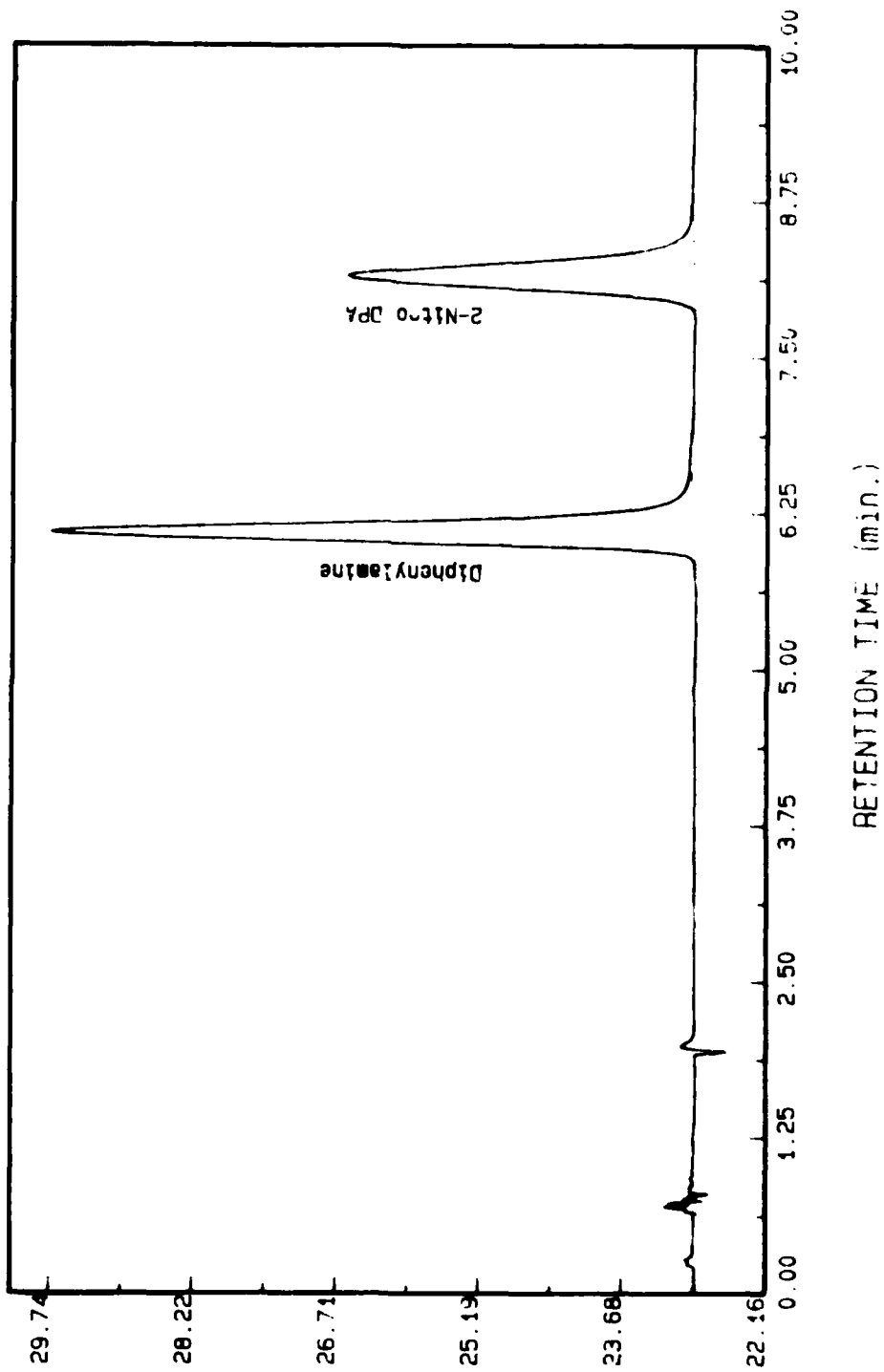


Figure 8. Typical responses of LC/EC detector at 1.05 V.

TABLE 3. HYDRA 70 2-NDPA Analysis by LC/EC 3 1.05 V

Results	SAMPLE 1	SAMPLE 2
	2.0181 percent	2.0419 percent
	1.9964 percent	1.9899 percent
	1.9788 percent	1.9939 percent
AVG	1.9979 percent	2.0086 percent
SD	0.0197	0.0289
RSD	0.985	1.411

IV. CONCLUSIONS

The EC detector has been demonstrated to be a sensitive detector that is selective for propellant stabilizers. Analytical chemists concerned only with the stabilizer levels of a propellant can select a solvent that will elute the stabilizers rapidly being only concerned with resolution of the stabilizer and its daughter products. This technique will be most useful for routine sample analysis procedures.

The internal standards used in this study were stabilizers themselves such as MNA, DPA, and 4-NDPA. As a follow-on study a variety of materials should be evaluated as internal standards. The optimum class of compounds would have a good UV response at 214 nm or at 254 nm and have a good response at above +0.9 V vs Ag/AgCl reference electrode. The class of compounds should be commercially available in high purity, have good stability in air, water, and most organic solvents, and have enough members to provide a wide selection of retention times.

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