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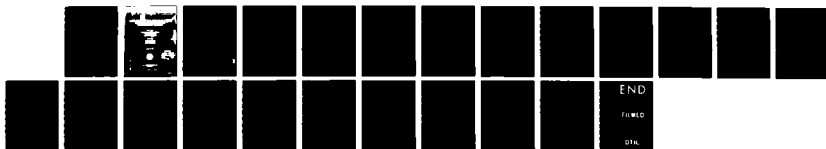
ANALYSIS TECHNIQUE FOR DETERMINING THE LEVELS OF
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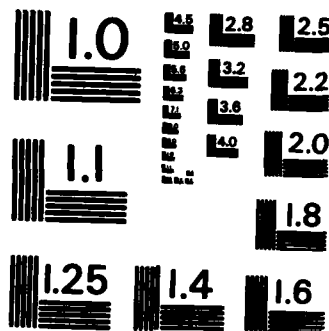
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**ANALYSIS TECHNIQUES FOR DETERMINING THE LEVELS OF ORGANIC ADDITIVES
IN AN EPDM RUBBER FOR USE IN UNDERWATER ACOUSTIC APPLICATIONS**

INTRODUCTION

Ethylene-propylene diene monomer (EPDM) rubber is being considered for applications in underwater electroacoustic transducers. This material is attractive for such applications because it is acoustically transparent over a wide range of frequencies with very low loss and has an excellent environmental resistance. An optimized Navy EPDM formulation for transducer applications has been developed at Underwater Sound Reference Detachment (USRD) under the support of the Office of Naval Research (ONR) Acoustic Transduction and Metrology Program (see Table 1) [1, 2]. In order to ensure that the Navy receives materials as specified according to this EPDM formulation, analytical procedures for determining the various ingredients in the EPDM rubber need to be developed for quality-control purposes. An analytical procedure for determining the organic additive levels in this EPDM formulation is given in this report. By using a liquid-solid chromatography (LSC) method, one can now measure reproducibly the percentages of dicumyl peroxide (DCP), trimethylolpropane trimethacrylate (TMPTMA), and polymerized 2,2,4-trimethyl-1,2-dihydroquinoline (TMDQ) in EPDM rubber. Chemical structures for these materials are shown in Fig. 1. This procedure was tested by conducting a blind study on three compounded EPDM rubbers and was proven to be satisfactory.

Table 1 - EPDM Rubber #259.3 Formulation.*

ADDITIVE	PARTS	WT%
Royalene 521	100	63.4
Carbon Black	45	28.5
Zinc Oxide	5.0	3.2
Di-Cup KE**	5.0	3.2
TMPTMA	2.0	1.3
TMDQ	0.8	0.5

* This formulation is now designated an NRL-USRD EPDM RLE.

** Di-Cup KE - 40% Di-Cup on Burgess KE Clay; therefore actual % of DCP is 1.28.

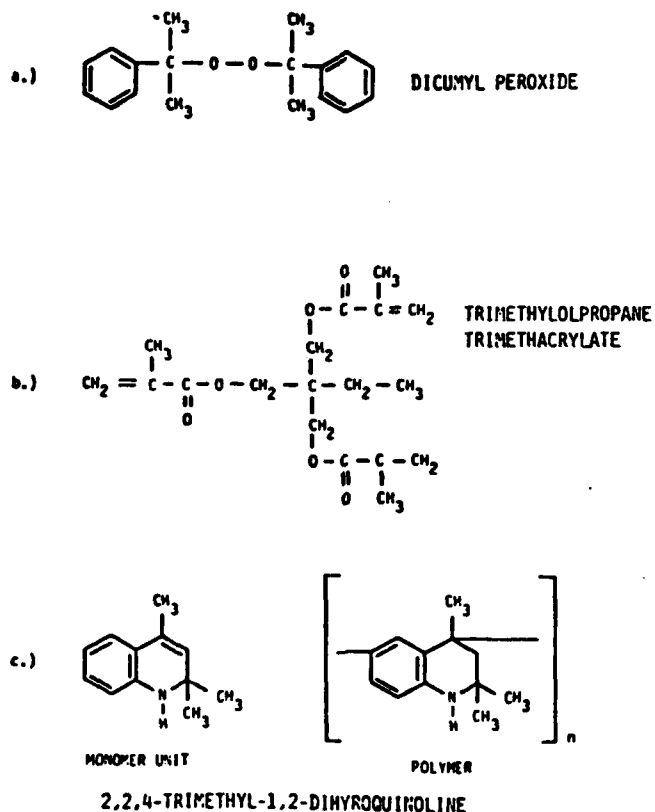


Fig. 1 - Chemical structures of (a) DCP, (b) TMPTMA, and (c) TMDQ.

EXPERIMENTAL PROCEDURE

Selection of HPLC Operating Conditions

By using guidelines suggested by Snyder and Kirkland [3], a flow chart was prepared which would be used to guide the development of analysis procedures for measuring the organic additives in EPDM rubber samples. This flow chart is shown in Fig. 2. Since the molecular weights of the additives are less than 2000, and they are non-ionizable, the following chromatography methods were investigated as potential liquid chromatography (LC) systems: bonded phase and adsorption. Schram [4] and McGee [5] provided a more detailed discussion on the basic aspects of liquid chromatography. Table 2 lists the conditions under which the various LC methods were evaluated. Liquid-solid chromatography was chosen as the LC system because it provided an adequate separation of the additives within a reasonable amount of time (i.e., less than 15 minutes). Figure 3 illustrates the LSC system.

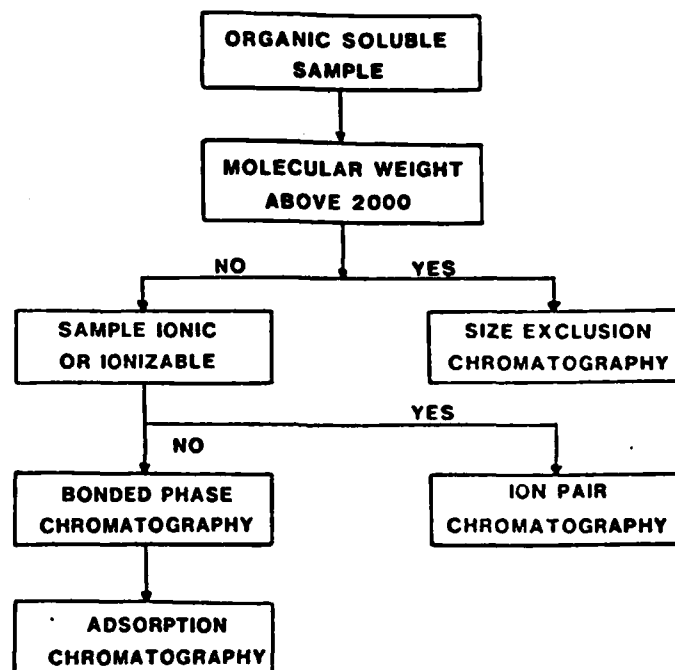


Fig. 2 - Flowchart for selecting possible LC methods.

Table 2 - Summary of LC Methods Investigated During Development Work

HPLC METHOD	COLUMN	MOBILE PHASE COMPOSITION
1. Bonded Phase		
a. Reverse	Waters C ₁₈ Phase	40-60% Tetrahydrofuran/Water 40-60% Methanol/Water 40-60% Acetonitrile/Water
b. Normal Phase	Waters C ₁₈	5, 10, 20% MeCl/hexane or cyclohexane* 5, 10, 20% EtCl/hexane or cyclohexane** 1, 5, 10% 2-PrOH/hexane or cyclohexane† 1, 2, 5, 10% THF/hexane or cyclohexane††
2. Adsorption		
a. LSC	Waters μPorasil	1, 2, 4% THF/hexane or cyclohexane

*MeCl:Methylene chloride

**EtCl:Ethylene chloride

†2-PrOH:2-Propanol

††THF:Tetrahydrofuran

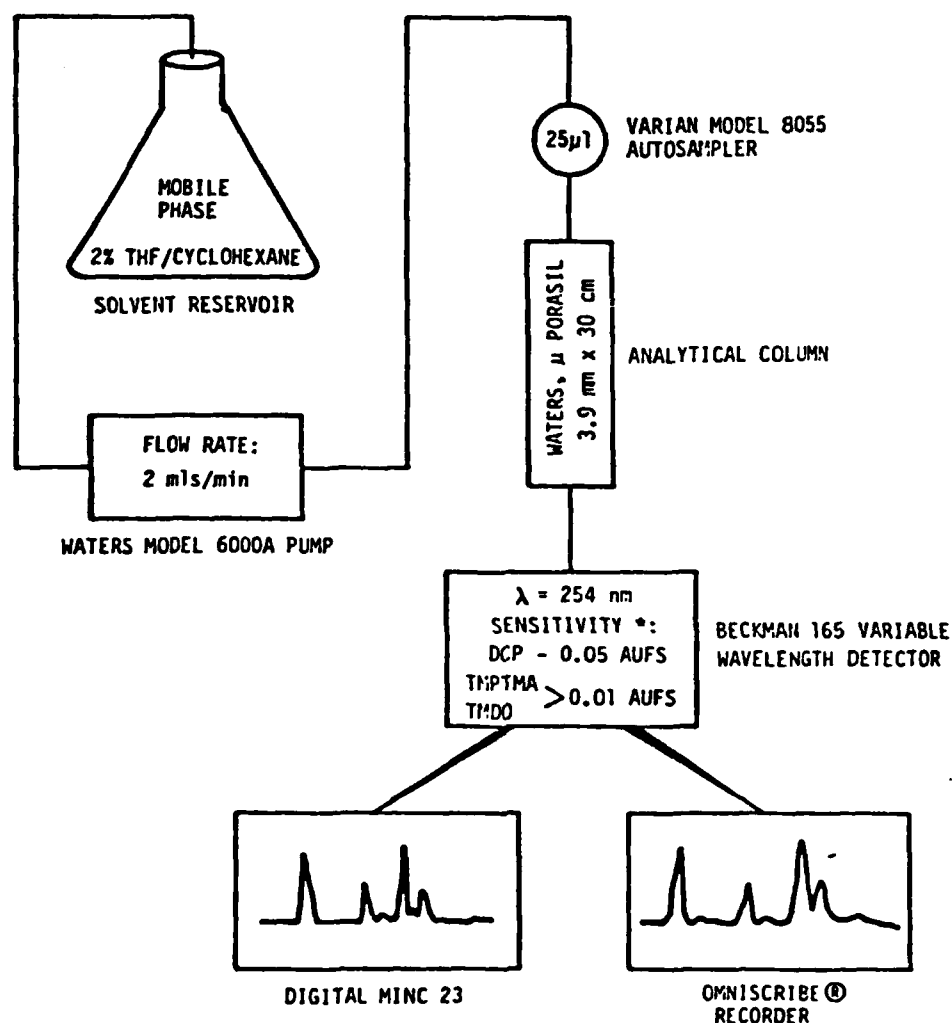


Fig. 3 - Block diagram of the LSC method.

The chromatograms shown in Figs. 4 through 8 were generated by using the LC operating parameters listed in Fig. 3. Figures 4, 5, and 6 are chromatograms of DCP, TMPTMA, and TMDQ at high concentrations, respectively. (Chromatograms of the standards at high concentrations were also run in order to check for impurities in the standards.) At high concentrations TMDQ shows several peaks which could interfere with the analysis of DCP and TMDQ. However, when the concentration of TMDQ is decreased to that expected in an EPDM rubber sample, these TMDQ peaks also decrease in height to levels below the sensitivity of the detector. Figure 7 is a chromatogram of a standard containing all three additives at concentration levels typical of those found in a compounded EPDM formulation. Figure 8 shows a chromatogram of additives recovered from a compounded EPDM sample. A comparison of Figs. 7 and 8 shows that the resulting peaks from the eluate of a rubber sample appear at the same positions on the retention time axis as the three additives of the standard. This implies that it is possible to separate and quantify the organic additives found in an EPDM rubber sample.

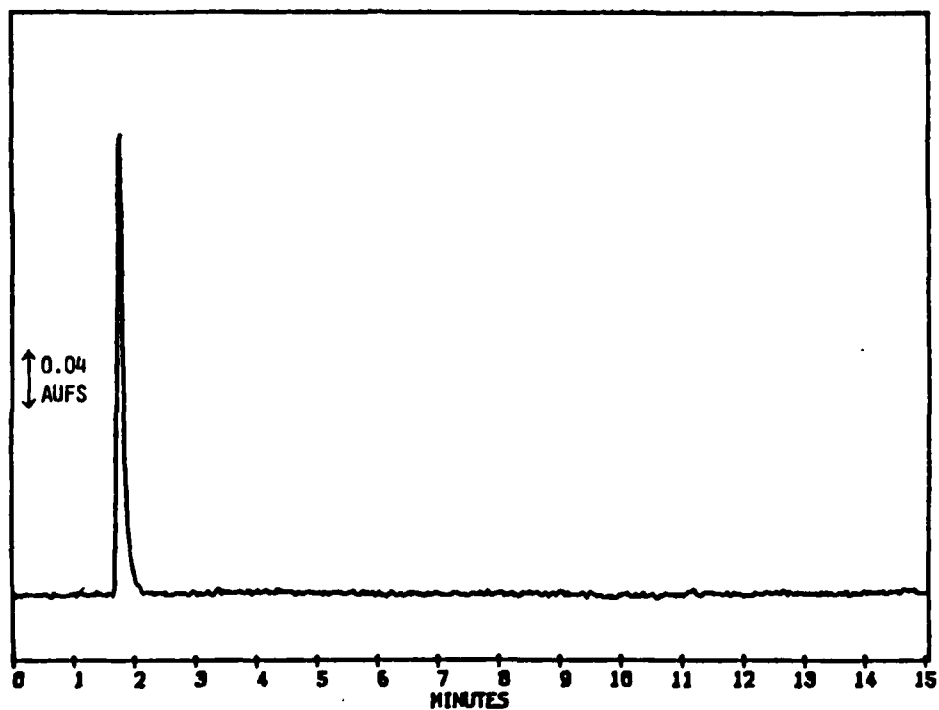


Fig. 4 - Chromatogram of DCP concentration: 0.07 mg/ml.

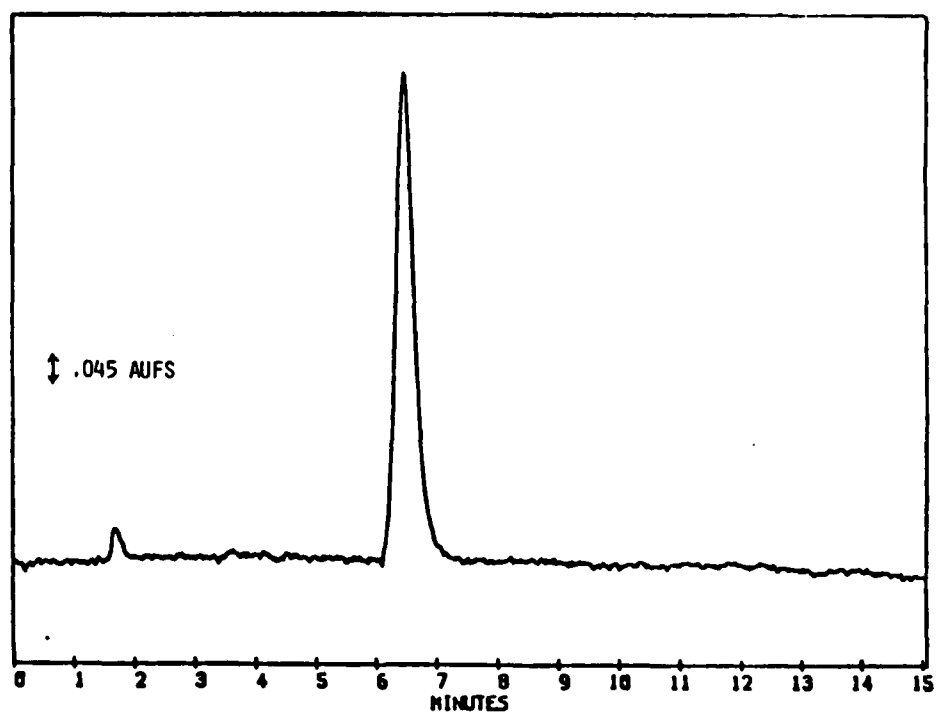


Fig. 5 - Chromatogram of TMPTMA concentration: 0.65 mg/ml.

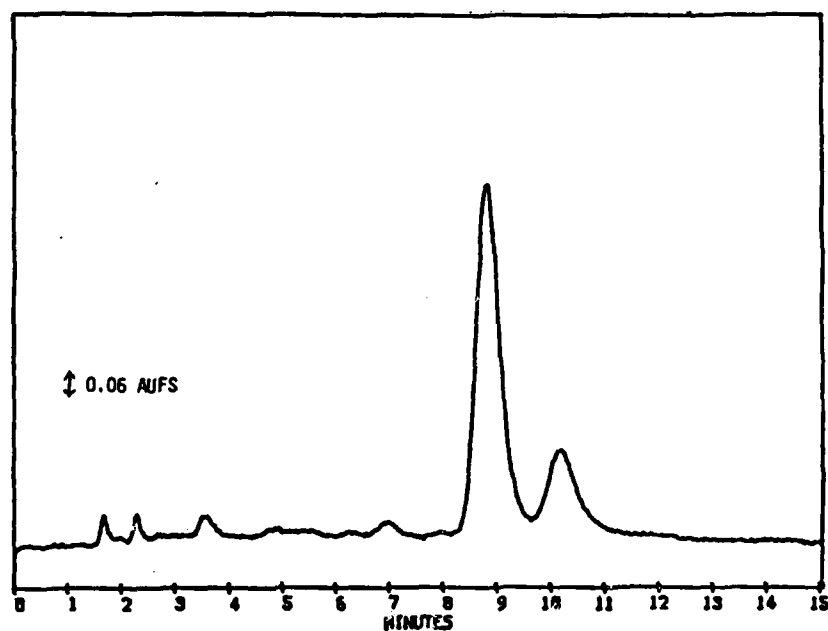


Fig. 6 - Chromatogram of TMDQ concentration: 0.18 mg/ml.

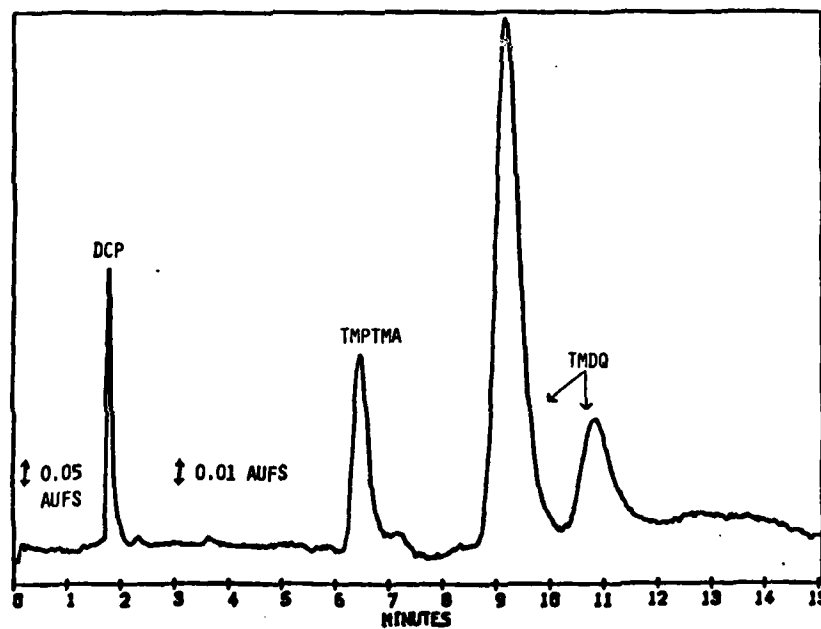


Fig. 7 - Chromatogram of a mixed standard solution
 Column: Water's μ PORASIL (#T22391D-78).
 Concentrations: DCP - 0.03 mg/ml
 TMPTMA - 0.12 mg/ml
 TMDQ - 0.05 mg/ml.

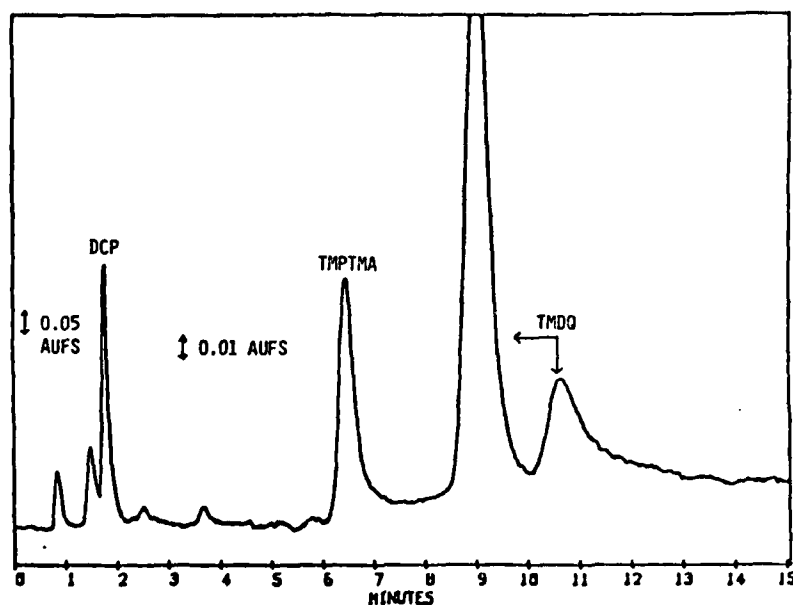


Fig. 8 - Chromatogram of an EPDM rubber sample.

Detector Response Calibration

A brief study was conducted to determine the optimum wavelength for monitoring the analysis. Absorbance spectra of the additives were obtained using a Beckman UV-VIS spectrophotometer and are shown in Fig. 9. Since all three of the additives showed an absorbance at 254 nm, this was selected as the monitoring wavelength. Further UV studies on the additives at a later date, using a Cary 219 spectrophotometer, suggest monitoring TMPTMA and TMDQ at 230 nm. However, in this report the chromatographic analysis was run at 254 nm.

The linear response range of the UV-detector to the additives at 254 nm was determined by injecting standards of varying concentrations and measuring their peak heights. Since TMDQ shows multiple peaks when chromatographed, only the peak at ten minutes was monitored (see Fig. 6). At the time the work was performed, a computer program for quantifying the peak heights or peak areas had not been developed. Therefore, peak heights were measured manually. Figure 10 shows the calibration curves for the additives. All three plots have a correlation coefficient of 0.9999, which implies the LC method can be used for quantitative analysis.

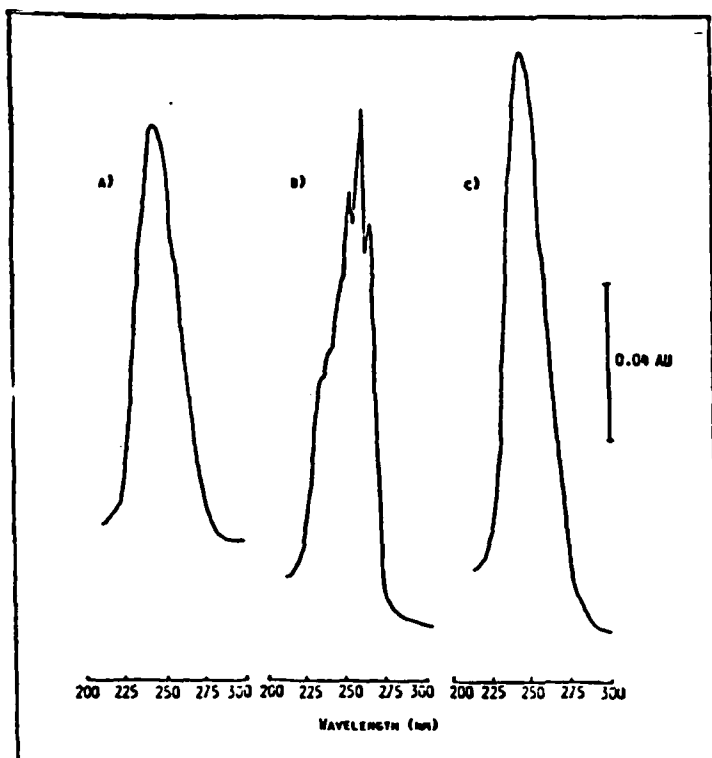


Fig. 9 - UV Scans of
 (a) TMPTMA,
 (b) DCP,
 (c) TMDQ.

Recovery of Additives from a Compounded EPDM Sample

Recovery Procedures Evaluated

Table 3 lists the various additive recovery techniques evaluated. Among the procedures tested, procedure no. 4 was finally adopted as the sample preparation method because:

- No extra peaks were added to the chromatogram which could interfere with interpretation of data.
- Gentle room-temperature extraction does not allow rubber to dissolve, thus the solution can be filtered without difficulty.
- Additives are extracted into mobile phase; therefore, no further sample pretreatment is necessary (i.e., evaporation of alcohol).
- Sources for error are kept to a minimum.

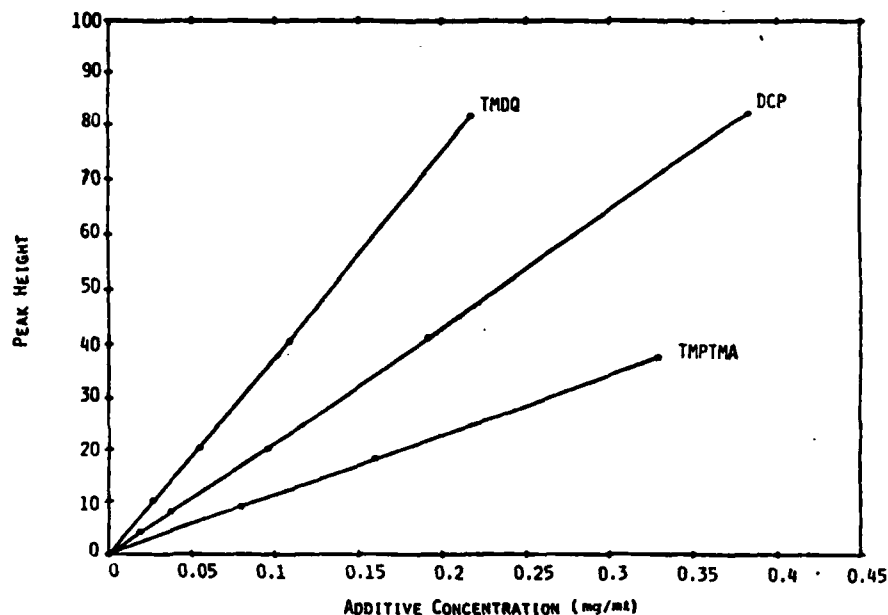


Fig. 10 - Calibration curves of EPDM additives.

Table 3 - Summary of Recovery Procedures.

PROCEDURE	SOLVENTS	COMMENTS
1. Waring blender: Finely chopped EPDM was placed in a semi-micro jar with solvent. Mixture was blended for 15 minutes.	1. Ethanol 2. Cyclohexane	Both solvents studied removed grease from the blender which added a peak to the chromatogram
2. Modified Extraction: Finely chopped EPDM was wrapped in felt and hung below a condenser. Solvent was placed in round-bottom flask, and mixture was refluxed for 1 to 3 hours.	1. Cyclohexane 2. Mobile Phase	Rubber dissolved which made it very difficult to filter.
3. Alcohol Reflux: Chopped EPDM was placed in a round-bottom flask with alcohol and refluxed 1 to 3 hours. An aliquot was removed and evaporated. The residue was dissolved in mobile phase.	1. Methanol 2. Ethanol	This procedure worked well but it was lengthy and a potential source of error existed at the evaporation step.
4. Rotator: (explained in detail in next section of report).	1. Mobile Phase	This procedure proved to be the most effective.

Final Procedure for Recovery of Organic Additives

- Approximately 150 mg of frozen, uncured, compounded EPDM rubber is cut from the sampling site and diced into small pieces (1 mm²).
- The diced rubber is placed in a dessicator for 30 minutes in order to remove the surface moisture and bring it to room temperature.
- A 100-mg sample is weighed and placed in a screw-top test tube containing 6 mls of 2% THF/cyclohexane. The test tube is capped and gently rotated for 1 hour. (The rubber swells during this process but does not disperse, and the additives are extracted into the solvent.)
- The sample is filtered into a 10-ml volumetric flask using a Millipore 47-mm stainless steel filter funnel (#XX409700) and a Rainin nylon filter (#38-114; 0.45 µm pore size). After adjusting the volume to 10.00 ml, the sample can be analyzed by LC.

Evaluation of Recovery Procedure

The additive extraction procedure was thoroughly tested by conducting an exhaustive extraction study on NRL-USRD EPDM rubber #259.3. The formulation for #259.3 is identical to that of EPDM-RLE, except the concentration of TMDQ was increased to 1.6 phr (see Table 1). Ten LSC samples were prepared from this rubber stock according to the extraction procedure, except that the extraction times were varied from 30 minutes to 4 hours, as shown in Table 4. Two samples were analyzed at each extraction time interval, except at 3 hours where only one sample was analyzed. The results, which are listed in Table 4, suggest that 1 hour is sufficient time to extract the additives. Due to the inhomogeneity of the additives in the rubber stock and the use of technical grade chemicals as LC standards, additive recoveries greater than 100% are seen in Table 4.

Table 4 - Exhaustive Extraction Study.

TIME INTERVAL (HOURS)	% ADDITIVE RECOVERED		
	DCP	TMPTMA	TMDQ
0.5	97.25	83.33	115.35
1	102.73	84.92	120.30
2	92.58	84.53	125.25
3	89.06	80.95	117.82
4	86.72	76.59	115.84

DISCUSSION AND CONCLUSIONS

Discussion of Problems

Reproducibility of Additive Retention Times

There was difficulty in achieving reproducible retention times for the additives. Table 5 shows the variation found in the retention time for TMPTMA. Similar variations were also seen for TMDQ and DCP. After several weeks this problem of unstable peaks was attributed to the inability of the Varian pump to regulate the solvent flow. In order for the Varian pump, a constant pressure pump, to maintain a constant flow rate, it is necessary for the column back pressure, mobile phase viscosity, and column temperature to remain constant. Apparently, the column back pressure changes as samples are injected resulting in reduced flow rates. For the LC separation to be reproducible, a constant flow rate pump; i.e., waters reciprocating piston pump, must be used as the solvent metering device.

Table 5 - Retention Times for TMPTMA.

DATE OF ANALYSIS	RETENTION TIME (MINUTES)
4-27-84	8.1
5-09-84	7.4
5-17-84	7.5
5-18-84 A*	7.0
5-18-84 B*	8.2
5-21-84	11.8
5-22-84 (1:00 pm)**	6.6
5-22-84 (3:30 pm)**	7.4

* Prepared two separate 2% THF/Cyclohexane mobile phases.

** Same mobile phase, chromatograms run at different times.

Column Sensitivity

While investigating the reproducibility of additive retention times, the condition of the μ Porasil column became a matter of concern because column deactivation is a problem in LSC [6]. The LSC columns are deactivated by the accumulation of polar compounds from impure solvents or dirty samples onto the adsorbent. A procedure for reconditioning the column after a day of analyses was therefore instituted. The procedure involved washing the column with 60 mls of ethylene chloride, followed by 60 mls of n-hexane, then allowing the n-hexane to remain in the column. This reconditioning scheme proved to be unsuitable as it resulted in the loss of TMDQ's peaks at 10.0 and 11.4 minutes. The peaks reappeared as the mobile phase (and subsequent injections) were run through the column.

The above observation was attributed to the solvents in the reconditioning scheme reactivating the column by removing chemicals that have accumulated on the adsorbent. In order to obtain reproducible results with this LSC analysis, it was necessary to deactivate the column by allowing only mobile phase and sample injections to come in contact with the column. The concept of deactivating a silica column to obtain reproducible chromatographic retention times is frequently used in LSC. Moriyasu and Hashimoto have discussed the use of deactivated columns for determining metal chelates by HPLC [7].

Column Variability

In addition to column sensitivity, column variability, i.e., the inability to reproduce the activity of the silica column packing material from one batch to the next, proved to be a problem [8]. Two silica columns were purchased in order to test the separation method:

- An Analytical Services Incorporated, ASI, silica column
- A new Waters μ Porasil column.

Figure 11 shows a chromatogram of a mixed standard solution separated using the ASI silica column. The TMDQ remains adsorbed to the column after 15 minutes. Although the ASI silica column is recommended as a direct replacement for Waters μ Porasil columns, it is not effective in analyzing the additives in an EPDM rubber sample.

Figures 12 and 13 show chromatograms of a mixed standard solution using a new Waters μ Porasil column (#T43191D-24). The flow rate was 2 mls/min for the chromatogram of Fig. 12. Comparison of Fig. 12 to a chromatogram generated by the Waters μ Porasil column (#T22391D-78), Fig. 7, shows the retention times of TMPTMA and TMDQ decreasing by approximately two minutes. By lowering the flow rate to 1.5 mls/min, as was done for the chromatogram in Fig. 13, it was possible to increase the retention times of the additives and better resolve TMDQ's peak at 10.0 minutes. This illustrates the necessity for manipulation of the conditions in Fig. 3, in order to achieve sufficient separation when a new column is received due to the variability among commercial LSC columns.

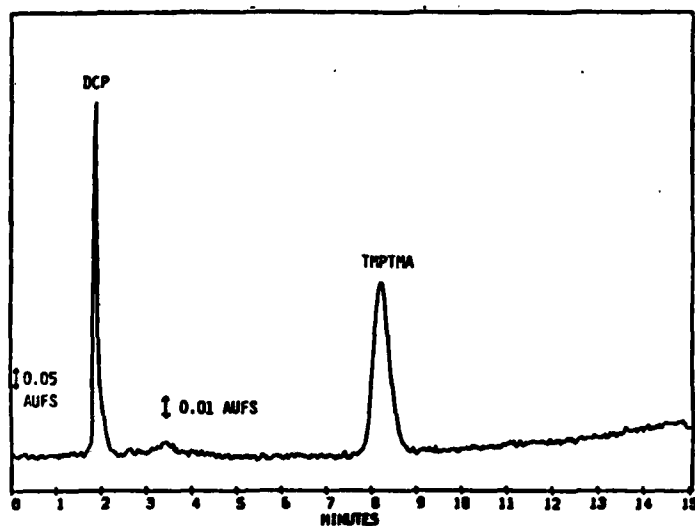


Fig. 11 - Chromatogram of a mixed standard solution. Column: ASI silica (#77023N).

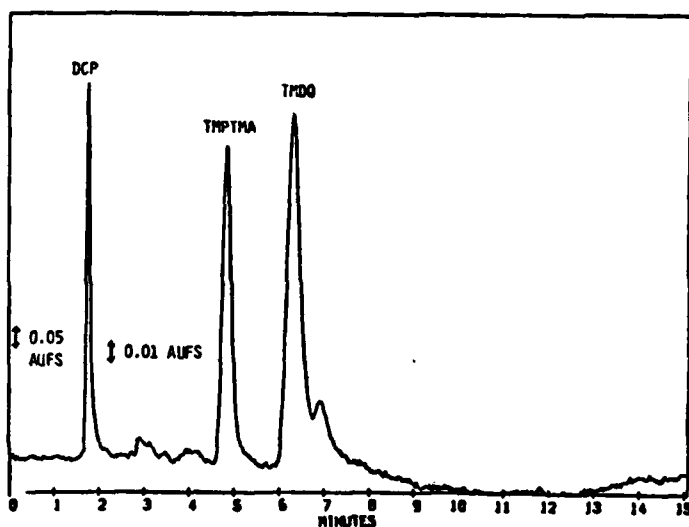


Fig. 12 - Chromatogram of a mixed standard solution. Column: Water's μ Porasil (#T43191D-24). Mobile Phase Flow Rate: 2.0 mls/min.

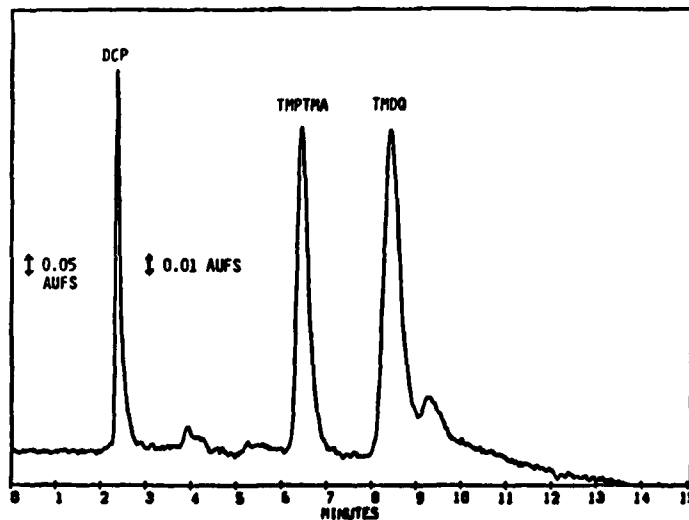


Fig. 13 - Chromatogram of a mixed standard solution. Column: Water's μ Porasil (#T431910-24). Mobile Phase Flow Rate: 1.5 mls/min.

TMDQ Variations

In the initial stages of this work, comparison of a TMDQ standard chromatogram to one of a rubber sample showed a marked difference in the TMDQ profile. Since TMDQ is synthesized by several manufacturers, samples were obtained from each supplier and a study was conducted to determine if variations could be found among the manufacturers' samples. The concentration of TMDQ was approximately 0.05 mg/ml in the mobile phase for this series of samples. Table 6 lists the manufacturers with their TMDQ trade names. Figures 14 through 18 are the chromatograms of the samples. Naugard Q (#0900400), Flectol Pastilles, and Ultranox 254 chromatographed similarly with two major peaks at 10.0 and 11.4 minutes. Naugard Q (#201811) and Flectol H exhibit an additional peak at 3.0 minutes. These differences could arise from the fact that TMDQ is a polymerized product and each manufacturer has its own specifications for the synthesis of TMDQ.

Because of this variation, it is necessary to obtain a sample of the TMDQ material that is actually used in mixing the rubber, in order to accurately determine the TMDQ concentration in an EPDM rubber sample.

Table 6 - TMDQ Manufacturers.

MANUFACTURER	TRADE NAME	LOT NUMBER
Uniroyal*	Naugard Q	201811;0900400
Monsanto	Flectol Pastilles	3L803
	Flectol H	N001-013
Borg-Warner	Ultranox 254	--

* Two samples of Naugard Q were obtained. #0900400 was received on 7-3-84; 201811 was received at an earlier date.

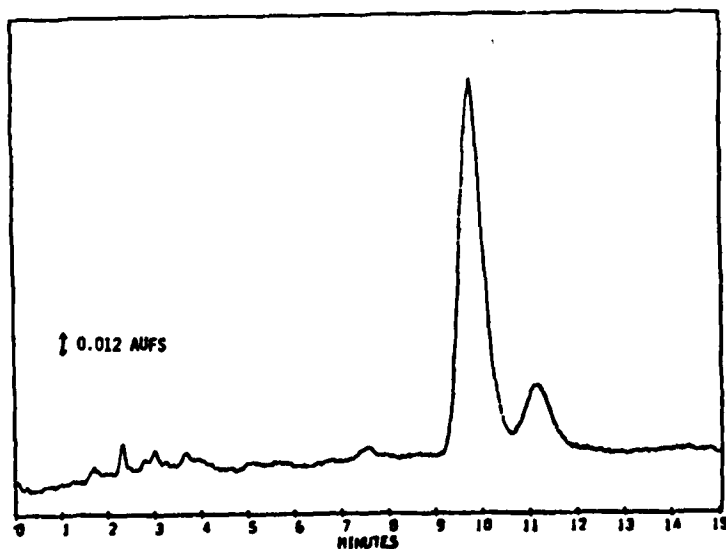


Fig. 14 - Chromatogram of a TMDQ sample.
Manufacturer: Uniroyal
Trade Name: Naugard Q
Lot No: 0900400.
Concentration: 0.051 mg/ml.

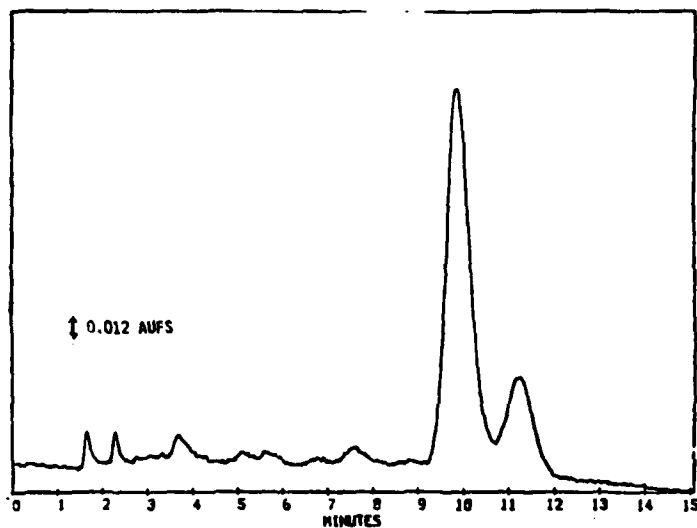


Fig. 15 - Chromatogram of a TMDQ sample. Manufacturer: Monsanto. Trade Name: Flectol Pastilles. Lot No.: 3L803. Concentration: 0.055 mg/ml.

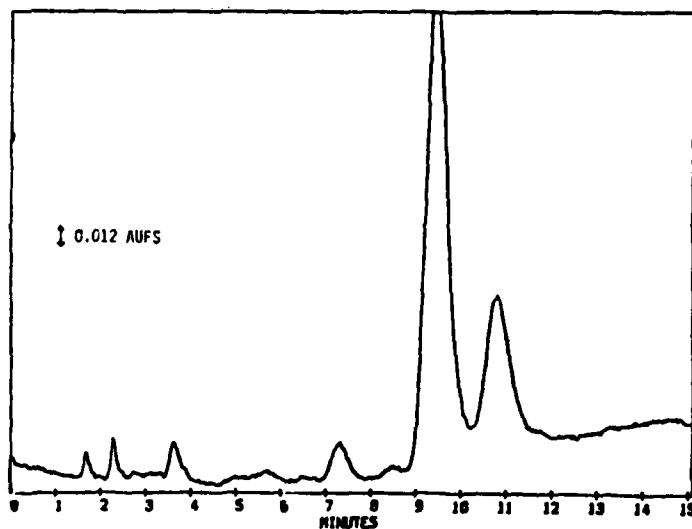


Fig. 16 - Chromatogram of a TMDQ sample. Manufacturer: Borg-Warner. Trade Name: Ultranox 254. Concentration: 0.068 mg/ml.

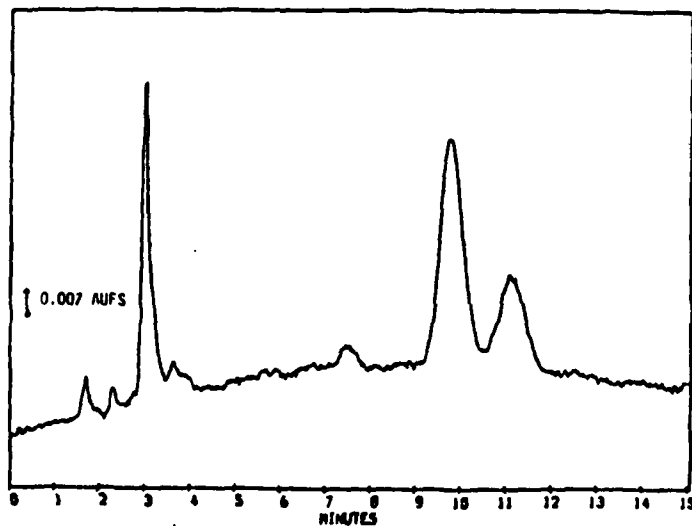


Fig. 17 - Chromatogram of a TMDQ sample. Manufacturer: Uniroyal. Trade Name: Naugard Q. Lot No.: 201811. Concentration: 0.050 mg/ml.

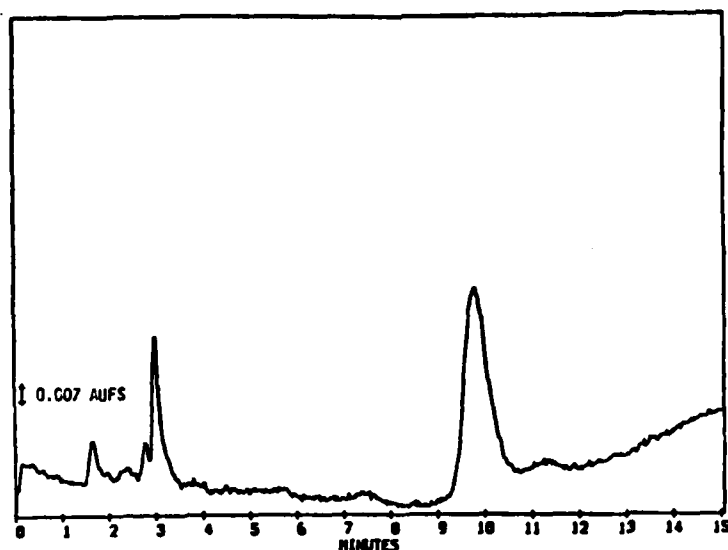


Fig. 18 - Chromatogram of a TMDQ sample. Manufacturer: Monsanto. Trade Name: Flectol H. Lot: NO01-013. Concentration: 0.049 mg/ml.

Single Blind Test Results

Three EPDM rubber samples, prepared separately at NRL-USRD, were analyzed for additive concentrations according to the methods described in this report. At the time of the analysis, the compounded formulations were not known to the LSC operator. The results are listed in Table 7. The values obtained indicate the LSC method is effective in reproducibly determining the levels of organic additives in uncured, compounded EPDM rubber.

Table 7 - Single Blind Test Results.

NRL-USRD SAMPLE NO.		WT % ADDITIVE		
		DCP	TMPTMA	TMDQ
259-14	Experimental*	1.44	0.55	0.54
	As compounded	1.76	0.63	0.50
	% Recovery**	81.8	87.3	108.0
259-1	Experimental	1.15	0.96	0.58
	As compounded	1.27	1.27	0.51
	% Recovery	90.6	75.6	113.7
259-10	Experimental	1.30	0.52	0.53
	As compounded	1.28	0.64	0.51
	% Recovery	101.6	80.5	102.9

*Two sampling sites were analyzed from each of the rubber samples.

** % Recovery = $\frac{\text{Experimental}}{\text{As compounded}} \times 100$

Concluding Remarks

An LSC procedure for reproducibly measuring the levels of organic additives in compounded, uncured EPDM rubber has been developed. For the quality-control method to be successful, these recommendations should be followed:

- A Waters μ Porasil column should be used as the stationary phase.
- Since it is necessary to deactivate the μ Porasil column, care must be taken to avoid contamination of the column with solvents other than the mobile phase.
- When obtaining a rubber sample from a manufacturer, a sample of the TMDQ used in compounding the rubber should also be sent to be used as an LC standard.
- A constant flow-rate-pump should be used as the solvent metering device to achieve reproducible separations.

As a result of this study, it is believed that the Navy is now in the position to measure, on a routine basis, the levels of organic additives in an EPDM rubber.

ACKNOWLEDGMENTS

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