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# Gel Permeation Chromatography Analysis of Coal Tar-Based Joint Sealants

*by Rogers T. Graham, Larry N. Lynch*



Prepared for Federal Aviation Administration
and Air Force Civil Engineering Support Agency

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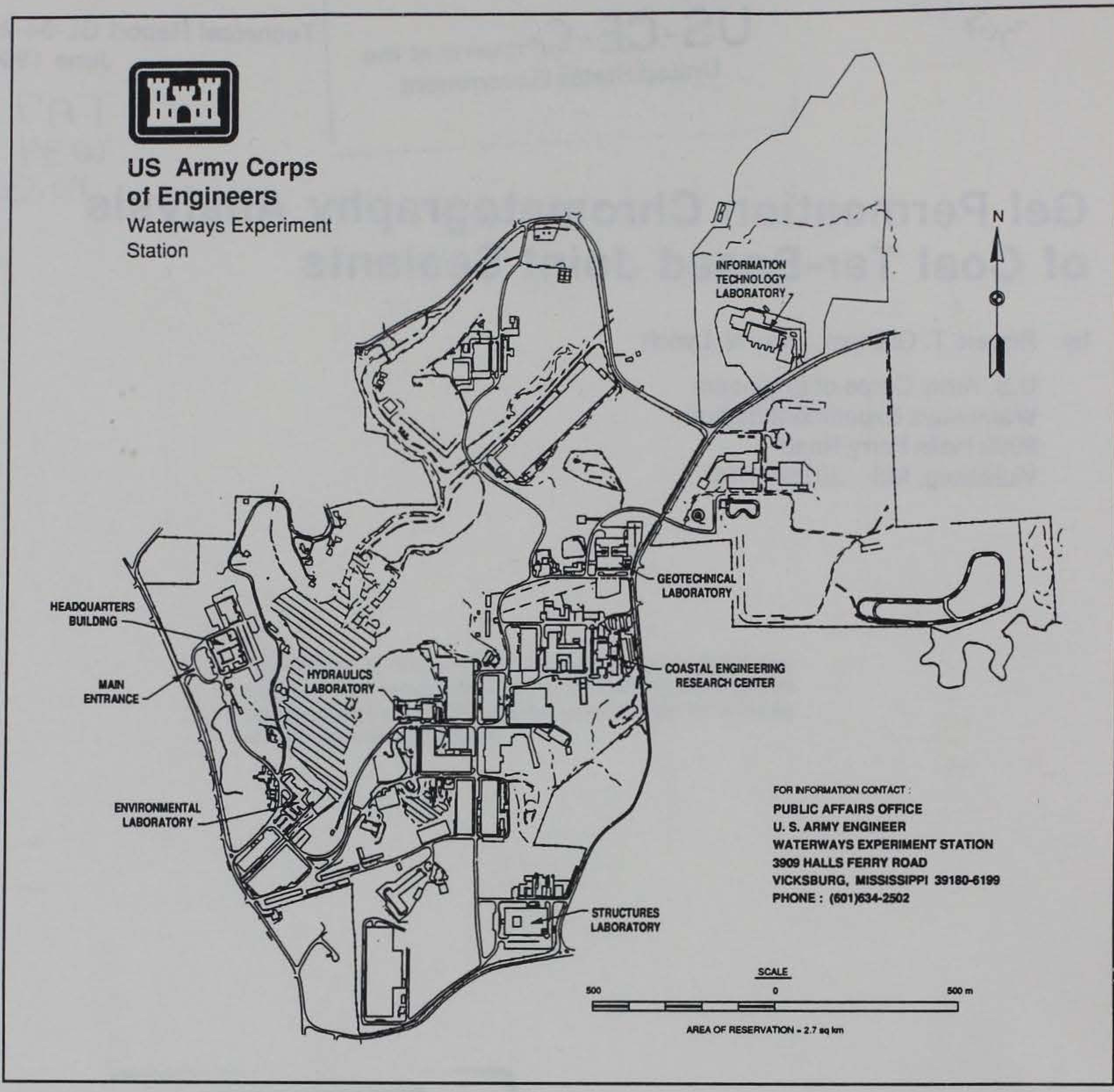


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# Preface

This investigation was conducted by the Pavement System Division of the Geotechnical Laboratory (GL), at the U.S. Army Engineer Waterways Experiment Station (WES), Vicksburg, MS, for the Federal Aviation Administration (FAA) and the Air Force Engineering Support Agency from April through December 1991.

The study was conducted under the general supervision of Dr. William F. Marcuson III, Director, GL, and Mr. Harry H. Ulery, Jr., former Chief, Pavement System Division (PSD) and Dr. George M. Hammitt II, Chief, PSD. This report was written by Messrs. Rogers T. Graham and Larry N. Lynch under the direct supervision of Mr. Timothy W. Vollor, Chief, Materials Research and Construction Technology Branch, PSD. PSD personnel in addition to the authors, engaged in the sampling, testing, analyzing, and evaluation of this project included Mr. Herbert McKnight, and CPT Laurand Lewandowski.

The Director of WES during the publication of this report was Dr. Robert W. Whalin. The Commander was COL Bruce K. Howard, EN.

# Conversion Factors, Non-SI to SI Units of Measurement

Non-SI units of measurement used in this report can be converted to SI units as follows:

| Multiply | By | To Obtain |
|---|---|---|
| degrees (angle) | 0.01745329 | radians |
| Fahrenheit degrees | 5/9 | Celsius degrees or kelvins[1] |
| feet | 0.3048 | meters |
| inches | 2.54 | centimeters |
| ounces (mass) | 28.34952 | grams |
| pounds (force) | 4.448222 | newtons |
| pounds (force) per square inch | 6.894857 | kilopascals |
| pounds (mass) per cubic foot | 16.01846 | kilograms per cubic meter |
| square inches | 6.4516 | square centimeters |

[1] To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: C = (5/9) (F - 32). To obtain kelvin (K) readings, use: K = (5/9) (F - 32) + 273.15.

# 1 Introduction

## Background

The field performance of most field molded pavement joint sealant materials (those sealants that are liquid at the time of installation and mold to the shape of the joint reservoir) has been less than desirable and is becoming an ever increasing focus of the pavement engineer. The increased focus has been generated by two factors. First, joint sealant materials can extend the life of a pavement by protecting the pavement structure. Secondly, fiscal problems at the city, state, and the federal levels have greatly reduced the amount of funds available to the pavement engineer for infrastructure maintenance. Therefore, maintenance funds that are expended have to provide a high-quality, long-term solution to the problem being solved; whether it is patching potholes or sealing joints and cracks.

Pavement joint sealant materials are designed to perform two basic functions; prevent the retention of incompressible debris in the joint and prevent or minimize the infiltration of water through the joint into moisture susceptible base and subbase materials. There are other functions, such as fuel resistance, that are designed into some sealants, but all sealants must perform the two basic functions to provide satisfactory field performance. If a sealant does not prevent the retention of incompressible debris, the thermal stress relief provided to the pavement through the joint will be negated often causing the joint edges to spall. Water infiltration through the joint can cause a weakening of the pavement structure by softening a moisture susceptible base or subbase material and creating voids under the pavement. Pumping of pavement slabs is a typical example of a weakened pavement structure that could be caused by water infiltration.

Field surveys conducted by the U.S. Army Engineer Waterways Experiment Station (WES) (Lynch 1989) and the Naval Civil Engineering Laboratory (NCEL) (Inaba, Hironaka, and Novison 1988) indicate that portland cement concrete (PCC) pavement joint sealant materials are generally performing their designed function for approximately 1 to 3 years. Some user agencies are reporting joint sealant failures within 6 months after application. This is a considerable difference from the verbal claims of some manufacturers who state their material will perform satisfactorily for 10 to 15 years when "properly installed." One manufacturer offered to substantiate the performance claims of their material by offering a 5 to 10 years warranty (Gaus

1984), but this offer has not gained wide acceptance in the sealant industry or in Government procurement actions.

The suspected reason for the discrepancy between the verbal performance claims, the proposed warranty period, and the actual field performance experienced in the field will vary depending upon the party providing the explanation. From the manufacturer's view point, the natural explanation of poor joint sealant field performance is poor workmanship or poorly written project specifications. The user agency will cite the reason for failure as poor workmanship or poor quality material, and the contractor will cite inferior materials or flawed project specifications.

The actual cause of poor field performance could be any one or all of the above mentioned explanations, but whatever the reason, the premature sealant failure affects the bottom line of the user agency. For example, it is estimated that the U.S. Department of the Navy spends $12 million annually resealing joints in PCC pavements (Inaba, Hironaka, and Novison 1988). Expenditures for the U.S. Departments of the Army and Air Force are not as easy to estimate because joint resealing projects are often included in maintenance contracts or performed by in-house crews; however, it is expected that similar funding would be required by both agencies. Therefore, an estimated $36 million is being spent annually by the Department of Defense (DoD) to reseal PCC pavement with joint sealant materials whose life cycles are less than half of that claimed by the manufacturers. If a method could be found to double the actual field performance of pavement joint sealants, the DoD alone could save an estimated $18 million annually.

To examine the potential benefits of increased joint sealant field performance in the civilian sector instead of focusing on the narrower DoD use of sealants, it is necessary to determine the quantity of sealant materials used on an annual basis. Joint sealant manufacturers estimate that the total United States market for pavement joint sealant materials is 100 to 125 million lb per year. If one assumes a joint reservoir size of 3/4 in.[1] wide by 3/4 in. deep, the total linear feet of joints sealed each year would be 356 to 445 million. The actual cost savings to the user agencies is difficult to estimate because the material cost for joint sealant materials range from 40 to 60 cents per lb for hot-applied joint sealants up to $3.20 per lb for some cold-applied sealants. Regardless of the actual material cost, the potential savings to user agencies could be astronomical.

To improve the field performance of pavement joint sealants, the three suspected causes of poor field performance should be investigated. Workmanship could be improved by implementing quality control and quality assurance measures. Educating contractor personnel and user agency inspectors on proper joint preparation and sealant application techniques and why these procedures are important would be one method of accomplishing this goal.

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[1] A table of factors for converting non-SI units of measurement to SI units is presented on page x.

Comparing project specifications with recently updated U.S. Army Corps of Engineers Guide Specifications, technical manuals, and manufacturers's literature could reduce the defects contained within project specifications, and testing the sealant material to the appropriate material specification will minimize the use of inferior materials (assuming material specification conformance implies a superior material). However, there are joint sealing projects in which all three areas of concern were reported to be correct, but the field performance of the sealant was still unsatisfactory.

Forensic analysis of some of these perfect projects indicate that problem areas exist in determining the exact cause of sealant failure. The largest deficiency is the fact that satisfactory field tests are not available to determine if the physical properties evaluated in the laboratory are being obtained in the field (Lynch 1989).

Field tests which are currently available include a coin test, twist test, and peel test. These tests are used to indicate a sealant's resilience and/or adhesion to the joint face. The coin test is conducted by pressing a coin, usually a quarter, into the sealant material to a depth of approximately 1/4 in. Then coin is then released to allow the sealant to rebound. If the sealant rebounds to its original shape and the coin is completely pushed out of the sealant, the sealant is considered to have satisfactory resilience. The twist test is conducted by pressing a flat piece of metal (generally 1/2 in. wide by 1/8 in. thick by 12 in. long) into the sealant material to a depth of approximately 1/4 in. and twisting it 90 deg or until it touches the joint face. If the sealant does not crack or pull loose from the joint face, it is considered to be in satisfactory condition. The peel test is conducted by cutting loose a 6-in. piece of the in-place sealant material. Two marks are placed 2 in. apart on the sealant and the loose end of the sealant is stretched to a specified elongation. This test has been modified by some user agencies to include a scale. The scale is attached to the loose end of the sealant so that the force required to stretch the sealant to the specified elongation can be measured.

The limited field tests that do exist are highly dependent upon the operator conducting the test, the environmental conditions during the test, and the shape factor of the sealant being tested. Because of these factors, the tests are not reproducible and cannot necessarily distinguish between properly and improperly applied sealants. The only method currently available to determine if a sealant has been properly prepared and installed is for user agency to require 100 percent inspection during the project. Due to manpower shortages and lack of funding, this type of inspection is difficult, if not impossible, to obtain. It would therefore be advantageous to develop or modify a test procedure that could identify improperly prepared and/or applied joint sealant materials.

One method that has been used to identify different types of asphalt cements and provide an indication of the physical properties of the asphalt cement is gel permeation chromatography (GPC). In this method, the asphalt cement is dissolved in a solvent and is injected into a GPC device. The injected sample travels through a series of columns which separates the sample based on molecular size. The larger molecular size particles exit the columns first and are detected by the system's detectors. The smaller molecular size particles travel into the pores of the columns, and therefore, have longer retention times. A molecular size distribution (which can be thought of as analogous to a type of sieve analysis of the sample) is obtained. One study (Price 1988) indicates that asphalt cements which have a higher concentration of large molecular size particles are more brittle than asphalts which contain high concentrations of small molecular size particles.

It is expected that similar trends may be found in joint sealant materials and that the change can be detected by the GPC, especially for hot-applied joint sealant materials. As the sealant is heated before application, volatiles (small molecular size particles) will be driven off. If the sealant is over heated or exposed to prolonged heating, a greater amount of the volatiles will be driven off causing the sealant to become brittle.

## Objective

The objective of this research was to determine if a laboratory test method could be used to identify joint sealant materials that have been improperly prepared and to predict how the improper preparation will affect the physical properties of the sealant.

## Scope

This research study was a continuation of Gel Permeation Chromatography Analysis of Asphalt Based Joint Sealants (Graham and Lynch 1992). The scope included a three-phase laboratory study, and an analysis of the laboratory data. Phase I of the laboratory study consisted of testing two different joint sealant materials in accordance with federal specifications. Two hot-applied, coal tar-based sealants were tested in accordance with Federal Specification SS-S-1614A (Federal Specification SS-S-1614A 1984).

Phase II of the laboratory study was a modification of Phase I. In Phase II, the heating times of the hot-applied sealants were changed to simulate different exposures to application temperatures. Test criteria outlined in the federal specification were then conducted to determine the physical property changes caused by the preparation conditions.

Additional tests were conducted on each sealant in both the Phase I and Phase II portions of the laboratory study. The additional tests consisted of penetration and resilience tests on artificially aged specimens. The penetration and resilience tests were conducted in accordance with Federal Specification SS-S-1401C (Federal Specification SS-S-1401C 1984) except that the specimens were conditioned in the Weather-O-Meter for 160 hr as specified in

Federal Specification SS-S-200E, Amendment 1, (Federal Specification SS-S-200E, Amendment 1 1988).

Phase III of the laboratory study was the GPC analysis of each of the conditioned sealant materials. Samples of the as-received sealants were also analyzed using the GPC to establish a base line fingerprint of each sealant. The fingerprint was then compared to the chromatograms obtained from the conditioned sealants to determine if a significant change had occurred. Replicates of each GPC sample were conducted to verify reproducibility.

# 2 Laboratory Test Plan

## Phase I

Phase I of the laboratory study consisted of testing the joint sealant materials to the appropriate Federal Specification, in this case SS-S-1614A. The Federal Specification has several criteria requirements that the sealant must meet in order to conform to the specification. Failure of any one of the criteria is classified as nonconformance. Table 1 lists the criteria and their requirements for the specifications.

Additional tests were established and conducted on the sealants to test the physical characteristics under various aging conditions. For example, notice in Table 1 that the test criteria for Federal Specification SS-S-1614A includes a penetration and a fuel immersed penetration. The additional tests chosen for the SS-S-1614A sealants were an aged penetration, a penetration conditioned in the weather-o-meter, an unaged resilience, an aged resilience, and a resilience conditioned in the weather-o-meter. The specific additional tests conducted on each sealant are provided in Table 2.

Federal Specification SS-S-1614A requires the penetration test to be conducted by filling a 6-oz container flush with sealant material. The specifications require that the penetration be conducted in accordance with ASTM D 5 (American Society for Testing and Materials 1986) using the penetrometer and optional cone described in ASTM D 217 (ASTM 1988). All unaged penetrations were prepared and tested using this procedure. The weather-o-meter and oven-aged penetration specimens were prepared and tested in accordance with the Federal Specifications except they were conditioned in a twin-enclosed, carbon arc weather-o-meter or a forced-draft oven, respectively.

The penetration specimens conditioned in the twin-enclosed carbon arc weather-o-meter, were exposed to 160 cycles of 51 min of ultraviolet (UV) radiation with a controlled specimen temperature of 140°F and 9 min of UV combined with a water spray as described in Federal Specification SS-S-200E. This conditioning was conducted to simulate exposure to the natural weathering conditions of sunlight and rain. The penetration specimens conditioned in the forced-draft oven were exposed to 158°F for 7 days. This conditioning was conducted to accelerate the aging of the sealant.

**Table 1**
**Federal Specification SS-S-1614A Test Requirements**

| Test | Requirement |
|---|---|
| Application temperature (°F)[1] | Pouring temperature shall be the safe heating temperature and shall be determined by the manufacturer. |
| Melting time | 3 hr |
| Penetration, 77°F (mm) | |
| Nonimmersed[2] | Shall not exceed 13.0 |
| Fuel immersed[3] | Shall not exceed 15.5 |
| Change | Increase shall not be more than 2.5 over nonimmersed |
| Change in weight, percent | Shall not exceed 2.0 |
| Flow at 140°F (mm) | Shall not exceed 30.0 |
| Bond to concrete (0°F) | |
| Nonimmersed[2] | Not more than 1 specimen out of 3 shall develop any crack, separation, or other opening in the sealing compound or between the sealing compound and the concrete blocks. |
| Fuel immersed[3] | None of the three samples shall evidence a complete cohesive failure of the material and the gross area of bare concrete exposed on the face of any one concrete block shall not exceed an area of 1/4 sq in. |
| Water immersed[4] | Same as nonimmersed |

[1] The application temperature is the highest used temperature permitted by the manufacturer and is a temperature to which the sealant can be heated for a duration of at least 3 hr and still conform to all of the requirements specified.
[2] Nonimmersed specimens were prepared in accordance with ASTM D 5.
[3] Fuel immersed specimens were immersed for 24 hr in 500 ml of clean test fuel maintained at 120°F plus or minus 2°F before testing. Test fuel shall be made up of 70 percent iso-octane and 30 percent toluene. This is the same as Reference Fuel B as described in ASTM D 471. Testing was conducted the same as nonimmersed specimens.
[4] Water immersed specimens were immersed for 96 hr in 500 ml of distilled or deionized water before testing. Testing was conducted the same as nonimmersed specimens.

All resiliences were run in accordance with Federal Specification SS-S-1401C. The specifications require a 6-oz container to be filled flush with the sealant material. The penetrometer specified in ASTM D 217 using a ball penetration tool substituted for the penetration needle is used to conduct the test. The ball is placed in contact with the sealant surface, then released and allowed to penetrate the specimen for 5 sec. The penetration at 5 sec is recorded in 0.1-mm units. The ball is then pressed into the sealant material an additional 100 units (10 mm) within 10 sec at a uniform rate. The ball is held at the additional depth for 5 sec and is then allowed to rebound for 20 sec. The final reading is subtracted from 100 plus the initial reading to determine the resilience. The unaged specimens were conditioned at standard laboratory conditions for 24 hr before testing. The oven-aged and weather-o-meter aged resilience specimens were conditioned in the same manner as the oven-aged and weather-o-meter aged penetration specimens.

**Table 2**
**Additional Tests Conducted on Each Sealant Type**

| Sealant Type | Additional Test |
|---|---|
| SS-S-161A | 2 - penetrations, 1 oven-aged, and 1 conditioned in the weather-o-meter[1]<br><br>3 - resiliences, 1 conditioned at standard laboratory conditions, 1 oven-aged, and weather-o-meter conditioned |

[1] Standard laboratory conditioned specimens are conditioned for 24 hr at 73 ± 4°F temperature and 50 ± 5 percent relative humidity. Oven-aged specimens are cured for 24 hr at standard conditions (73 ± 4°F temperature and 50 ± 5 percent humidity), oven-aged in a forced-draft oven at 158 ± 2°F for 168 ± 2 hr, cooled under standard conditions for 1 hr, then conditioned for 1 hr in a water bath at 77 ± 0.5°F prior to testing.
[2] Weather-o-meter conditioned specimens are cured for 24 hr at standard conditions, exposed to 160 cycles of 51 min of UV radiation with a controlled black panel temperature of 140°F and 9 min of UV combined with a water spray, then conditioned for 1 hr in a water bath at 77 ± 0.5° prior to testing.

The Phase I testing data were used as the base line against which all other test data were compared. The Federal Specification testing provided an indication of the physical characteristics and the additional testing provided an indication of how various artificial aging techniques affect selected physical properties.

## Phase II

Phase II of the laboratory study used the same type of conditioning and testing procedures as Phase I; however, the heating times for hot-applied sealants were varied. The sealants were exposed to the manufacturer's recommended safe heating temperature for the following times:

*a.* Heating Time A - Sealant was poured into the specimen molds as soon as the sealant reached the safe heating temperature.

*b.* Heating Time B - Sealant was maintained at the safe heating temperature for 90 min and then poured into the specimen molds.

*c.* Heating Time C - Sealant was maintained at the safe heating temperature for 3 hr and then poured into the specimen molds. This is the heating time used in Phase I per Federal Specification SS-S-1614A test requirements; therefore, it was not repeated in Phase II.

*d.* Heating Time D - Sealant was maintained at the safe heating temperature for 6 hr and then poured into the specimen molds.

By exposing the sealant to the various heating times, the affects of prolonged heating on the sealant's physical properties could be evaluated.

## Phase III

Phase III of the laboratory study consisted of the GPC analysis of the joint, sealant materials. To conduct this portion of the study, the same solvent, equipment and test parameters that were used for Asphalt Based Joint Sealants was used (Graham and Lynch 1992).

*a.* Mobile phase and solvent tetrahydrofuran.

*b.* 3 columns in series, an Ultrastyragel 1000Å, an Ultrastyragel 500Å, and an Ultrastyragel 100Å.

*c.* Column temp 104°F.

*d.* Injection concentration 1.5% m/v.

*e.* Flow rate 0.8 ml/min.

*f.* Injection volume 0.25 ml.

*g.* Sample time in solution was less than 2 hr.

*h.* Samples were filtered using a prefilter and 2.005 $\mu\ell$ filter.

*i.* UV detection wavelength 254 n.m.

An as-received sample of each sealant was analyzed using the GPC to establish a before conditioning base line or fingerprint of each material. Samples were then prepared from each of the unaged, oven-aged, and weather-o-meter conditioned penetration samples from Phase I and II of the laboratory study to evaluate GPC as a test method to determine the effects of prolonged heating and aging in sealants.

# 3 Phase I and II - Presentation and Analysis of Data

Phase I and II data will be discussed together since the only difference between the two phases is a change in the length of heating time before the samples were poured into the molds. Phase I data are designated by a C following the sealant identification, and Phase II data are designated by an A, B, or D following the sealant identification. Table 3 provides the nomenclature used to describe the sealants which were evaluated.

**Table 3**
**Nomenclature Used for Sealant Analysis**

| Nomenclature | Definition |
|---|---|
| 6523 | Sealant manufactured to meet Federal Specification SS-S-1614A obtained from Manufacturer A. |
| 6540 | Sealant manufactured to meet Federal Specification SS-S-1614A obtained from Manufacturer B. |
| 6523-A<br>6540-A | Sealant samples poured as soon as the material reached the safe heating temperature. |
| 6523-B<br>6540-B | Sealant samples poured after 90 min of heating at the safe heating temperature. |
| 6523-C<br>6540-C | Sealant samples poured after 3 hr of heating at the safe heating temperature. |
| 6523-D<br>6540-D | Sealant samples poured after 6 hr of heating at the safe heating temperature. |

Two sealants, one each from two manufacturers, were obtained for the Phase I and Phase II analysis. These two sealants were manufactured to meet the requirements of Federal Specification SS-S-1614A and were selected based on the fact that the two materials are often submitted for specification conformance testing for use on military projects. Sealants manufactured to meet Federal Specification SS-S-1614A can be supplied by the manufacturer in either solid or liquid form. The solid form behaves like a thermoplastic material in the sense that the solid must be heated to insert it into the joint. The liquid form is a thermoset material. The liquid sealant must be heated before it is inserted into the joint, but the heating process converts the sealant to a solid as it cools in the joint. The two sealants used for this analysis were supplied in liquid form. The liquid form was selected because hand stirring of the as-received samples could be easily accomplished prior to placing the sealant in the melter, thereby minimizing any segregation that may have occurred during shipment.

When the sealants arrived at the laboratory, they were logged in using a flexible pavement laboratory (FPL) number. The FPL number is an in-house method used to track materials that have been received for testing. The FPL number will be used to represent the different sealants instead of using product names. The FPL numbers for the sealants are 6523 and 6540.

The as-received samples of both FPL 6523 and FPL 6540 met requirements of Federal Specification SS-S-1614A. Summaries of the Federal Specification SS-S-1614A test results for the two materials are provided in column C of Tables 4 and 5.

**Table 4**
**Federal Specification SS-S-1614A Test Results of FPL 6523**

| Test/Heat Time | A | B | C | D |
|---|---|---|---|---|
| Safe Heating Temperature (Deg F) | Used | Used | Used | Used |
| Penetration (cm) | | | | |
| Nonimmersed | 1.40 | 1.61 | 1.23 | 0.97 |
| Fuel-immersed | 1.45 | 1.14 | 0.92 | 0.83 |
| Oven-aged[1] | 0.92 | 0.86 | 0.48 | 0.40 |
| Weather-o-meter aged[1] | 0.99 | 1.11 | 0.98 | 0.91 |
| Flow (cm) | 0.0 | 0.0 | 0.0 | 0.0 |
| Change in Weight (percent) | 0.4 | 0.4 | 0.3 | 0.3 |
| Resilience[1] | | | | |
| Unaged | | | | |
| Initial indentation (cm) | 0.27 | 0.32 | 0.24 | 0.20 |
| Percent recovery | 63 percent | 58 percent | 66 percent | 66 percent |
| Oven-aged | | | | |
| Initial indentation (cm) | 0.21 | 0.23 | 0.19 | 0.18 |
| Percent recovery | 51 percent | 48 percent | 48 percent | 48 percent |
| Weather-o-meter aged | | | | |
| Initial indentation (cm) | 0.25 | 0.30 | 0.24 | 0.21 |
| Percent recovery | 53 percent | 54 percent | 60 percent | 64 percent |
| Bond to concrete (-20 Deg F) | | | | |
| Nonimmersed | Sat. | Sat. | Sat. | Sat. |
| Water-immersed | Sat. | Sat. | Sat. | Sat. |
| Fuel-immersed | Sat. | Sat. | Sat. | Sat. |

[1] Additional tests not included in SS-S-1614A specification.

**Table 5**
**Federal Specification SS-S-1614A Test Results of FPL 6540**

| Test/Heat Time | A | B | C | D |
|---|---|---|---|---|
| Safe Heating Temperature (Deg F) | Used | Used | Used | Used |
| Penetration (cm) | | | | |
| Nonimmersed | 1.24 | 1.21 | 1.10 | 0.89 |
| Fuel-immersed | 1.01 | 1.96 | 0.82 | 0.65 |
| Oven-aged[1] | 0.45 | 0.40 | 0.45 | 0.38 |
| Weather-o-meter aged[1] | 1.11 | 1.00 | 0.69 | 0.43 |
| Flow (cm) | 0.0 | 0.0 | 0.0 | 0.0 |
| Change in Weight (percent) | 0.3 | 0.3 | 0.3 | 0.2 |
| Resilience[1] | | | | |
| Unaged | | | | |
| Initial indentation (cm) | 0.26 | 0.24 | 0.20 | 0.16 |
| Percent recovery | 71 percent | 69 percent | 70 percent | 73 percent |
| Oven-aged | | | | |
| Initial indentation (cm) | 0.21 | 0.19 | 0.17 | 0.11 |
| Percent recovery | 46 percent | 43 percent | 49 percent | 53 percent |
| Weather-o-meter aged | | | | |
| Initial indentation (cm) | 0.27 | 0.28 | 0.20 | 0.13 |
| Percent recovery | 53 percent | 57 percent | 58 percent | 62 percent |
| Bond to concrete (-20 Deg F) | | | | |
| Nonimmersed | Sat. | Sat. | Sat. | Sat. |
| Water-immersed | Sat. | Sat. | Sat. | Sat. |
| Fuel-immersed | Sat. | Sat. | Sat. | Sat. |

[1] Additional tests not included in SS-S-1614A specifications.

## FPL 6523

The FPL 6523 penetration test results as shown in Table 4 indicate a hardening of the material with aging within all four heating times. The penetration results indicated that the oven-aging hardened the sealant more than the weather-o-meter aging. Also, with the exception of 6523-B nonimmersed or unaged penetration and 6523-B weather-o-meter aged specimens, the sealant exhibited hardening as the heating time was increased. FPL 6523-D showed the largest amount of hardening followed by FPL 6523-C with a lesser amount of hardening as indicated by the penetration values. This hardening trend would be expected because as a sealant ages it should become more brittle. The smallest amount of change among samples heated for different times occurred in the weather-o-meter aged samples. This indicated that the effects of the weather-o-meter aging were reduced as the heating time of the sealant was increased. The oven-aging, because of its severity, continued to age the sealant even at the extended heating times.

The fuel-immersed penetration specimens were conditioned and tested in the same manner as the unaged and nonimmersed specimens except they were immersed in reference fuel B as specified in Federal Specification SS-S-1614A for 24 hr. It would be expected that the fuel-immersed penetration would exhibit the same trend as the nonimmersed penetration results between the four heating times. This was in fact what occurred with the exception of FPL 6523-B. The penetration values decrease as the heating time was increased indicating that the sealant hardened.

The resilience results did not indicate the hardening trend as consistently as the penetration results. The differences between the unaged and weather-o-meter aged samples indicated inconsistencies to the hardening trend. The weather-o-meter specimens for two of the heating times, FPL 6523-A and B, exhibited a decrease in initial indentation with a corresponding decrease in the percent recovery as compared to the unaged specimens. The weather-o-meter initial indentation result of FPL 6523-C was the same as the unaged results, but a decrease did occur in the percent recovery. A decrease in the percent recovery of the weather-o-meter aged specimen also occurred with FPL 6523-D, but had a slight increase in the initial indentation value.

Comparing the initial indentation resilience results for the different aged conditions versus heating time indicated that hardening occurred as the heating time was extended with the exception of heating time B. As the heating time was increased, the initial indentation decreased, but there was not a clear trend in the percent recovery results. The unaged resilience percent recovery exhibited a decrease from FPL 6523-A to B, 63 percent versus 58 percent, but the percent recovery for FPL 6523-C and D increased to 66 percent. The oven-aged results indicated a decrease from FPL 6523-A to B and then remained constant at 48 percent for FPL 6523-B, C, and D.

The extended heating times used for FPL 6523 did not affect the percent change-in-weight on fuel-immersion, the flow, or the bond to concrete tests. The fact that these other test results were not affected indicate that the sealant fuel resistant and adhesive properties of the sealant may not be adversely affected if the sealant is heated at or below the safe heating temperature in the field for extended periods of up to 6 hr.

The penetration results appeared to provide an indication of the hardening a sealant undergoes as it is exposed to extended heating. The resilience initial indentation results indicated a similar hardening trend as the penetration results but not as pronounced as the penetration results. The resilience percent recovery test results indicated a decrease in resilience within a specific heating time when comparing unaged versus aged but yielded inconsistent results when comparing the four heating times.

## FPL 6540

The penetration results indicated a decrease as the sample was aged within a specific heating time and a decrease as the heating time was increased. The summarized test results are provided in Table 5. The one exception to the decrease in penetration value between heating times was the oven-aged specimen of FPL 6540-C. The FPL 6540-C oven-aged penetration value was the same as the FPL 6540-A value. The oven-aged specimens showed a greater amount of hardening than the weather-o-meter specimens that were very similar to the trend exhibited in FPL 6523. The weather-o-meter penetration results continued to decrease with an increase in heating time indicating that FPL 6540 was more susceptible to hardening caused by UV radiation than FPL 6523.

The resilience initial indentation test results for a given heating time indicated a hardening of the sealant when it was aged in the oven. The weather-o-meter initial indentation results were less consistent. The weather-o-meter initial indentation test results for given heating time exhibited either a slight increase, a slight decrease, or remained constant when compared to the unaged results. This indicated that the weather-o-meter aging did not affect the resilience initial indentation results as much as the penetration results. The resilience percent recovery within a specific heating time exhibited a decrease with both the oven-aged and weather-o-meter aged samples as compared to the unaged samples. As with the penetration results, the oven-aged materials exhibited the largest change.

The resilience initial indentation testing for the four heating times indicated that the sealant hardened as the heating time was increased. The FPL 6540-B weather-o-meter specimen was the one exception to this trend. The resilience percent recovery results for the four heating times exhibited an increase as the heating time was increased with the exception of the 6540-B unaged and oven-aged results. The increase in the percent recovery indicated that the sealant's ability to reject incompressibles seemed to increase as the heating time was extended. The increase in the percent recovery may be attributed to the extenders which have been added to the sealant contributing more to the physical characteristics of the sealant than the amount of volatiles that are removed during heating.

The percent change-in-weight on fuel-immersion, the flow, and the bond to concrete tests did not seem to be affected by the extended heating times. Therefore, even though FPL 6540 appeared to be more susceptible to UV radiation than FPL 6523, the fuel resistant and adhesive properties should not be adversely affected by heating as long as 6 hr in the field.

# 4 Phase III - Presentation and Analysis of Data

Physical testing of the pavement joint sealant materials conducted in Phase I and II indicated that the inconsistent changes that occur due to extended heating and artificial aging can be physically detected. The objective of Phase III was to determine if those changes could be detected using GPC. The same nomenclature provided in Table 3 was used to described the evaluated sealants.

Two specimens were prepared for each sealant condition, and two injections were made from each of the prepared specimens. For example, two pieces of sealant were obtained from the unaged penetration test specimen of FPL 6523-A. Each piece of sealant was placed into a separate vial and the appropriate amount of tetrahydrofuran (THF) was added to the vial. After the sealant dissolved in the THF, the sealant/THF solution was filtered to remove the inert fillers from the specimen. Two samples were taken from each vial and injected into the GPC, thus providing four runs for each sealant condition. Figure 1 illustrates typical results obtained from the four GPC runs. In this figure, samples 1 and 2 were taken from the first vial, and samples 3 and 4 were taken from the second vial. If the GPC is used to provide meaningful comparative data, then all four runs should overlay directly on each other as illustrated in Figure 1. Unfortunately, not all of the chromatograms for each of the sealant conditions yielded the near ideal results depicted in Figure 1.

The GPC analysis indicated considerable inconsistencies between the four runs of some of the sealant samples. Because of the inconsistencies, average chromatograms were calculated to assist in the comparative analysis. Standard deviations of the chromatograms were also calculated so that a 95 percent confidence region chromatogram could be constructed. The 95 percent confidence region was constructed by taking the average chromatogram and then adding and subtracting two standard deviations from the average. The $\pm$ 2.0 standard deviations provided the upper and lower limit for the 95 percent confidence region.

The chromatograms for the two sealants which were manufactured to meet the requirements of Federal Specification SS-S-1614A had similar profiles.

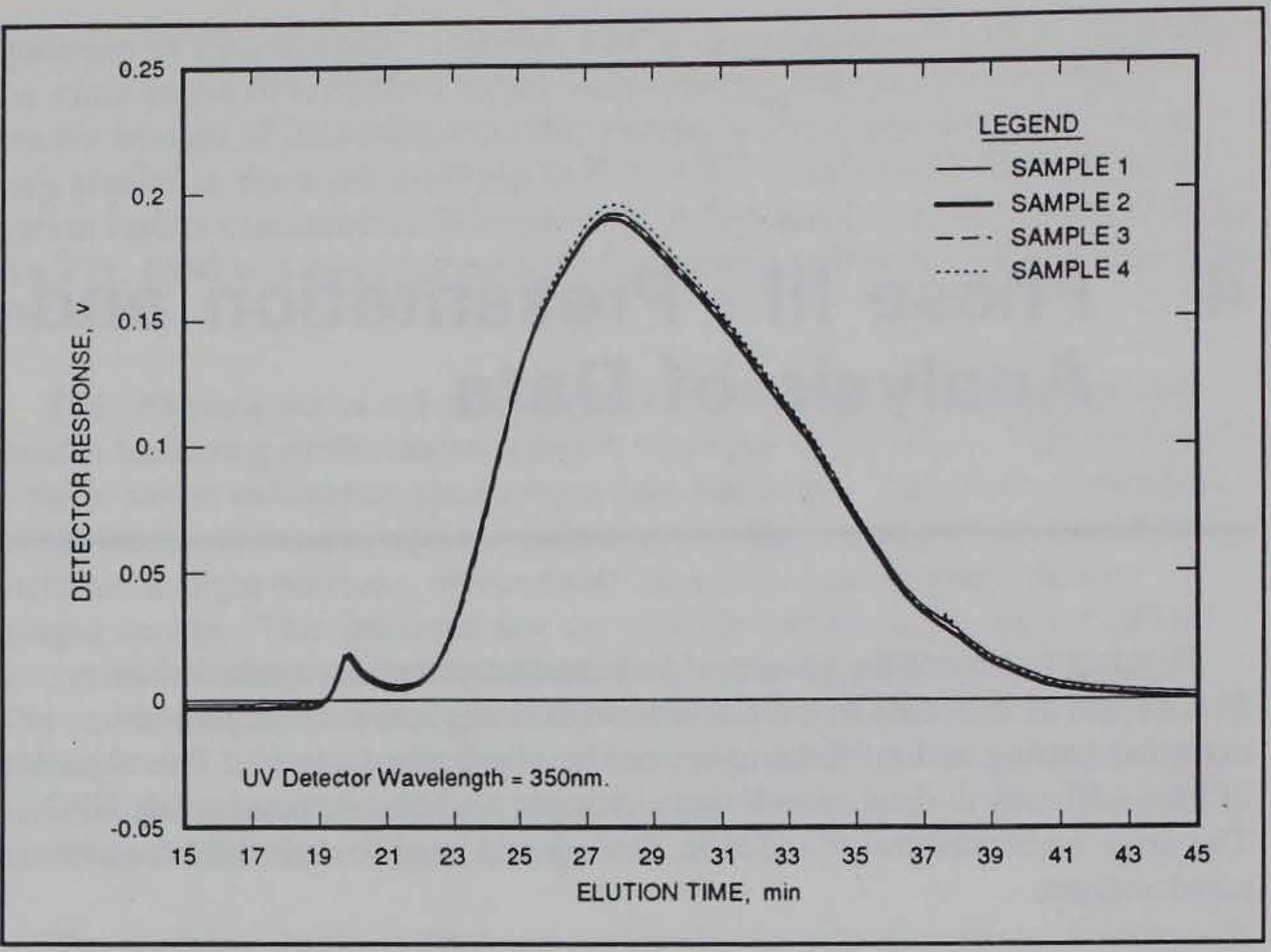


Figure 1. Chromatograms obtained from one sealant penetration specimen

The as-received samples had a single broad peak. A second peak was formed at an elution time of approximately 18 or 19 min as the sealant was heated in preparation for specification testing. In the remainder of the chromatographic analysis, the small peak which was formed only after heating the sealant will be referred to as the first peak. The chromatograms returned to the base line after this first small peak at an elution time of approximately 21 to 22 min. The broad peak which begin eluting at approximately 26 to 28 min exhibited a bimodal distribution. The first shoulder occurred at approximately 30 to 33 min and the second peak occurred at approximately 35 to 37 min. Most of these chromatograms then returned to the base line at approximately 45 min. However, some of the sealant chromatograms indicated that the sealant was absorbing onto the GPC columns. The absorption was evident by the fact that the chromatogram did not return to the base line within 50 min. The 50 min time limit should be more than sufficient to allow all of the sample to elute from the column if only mechanical forces are acting on the samples. Since the first small peak did not occur in the as-received sealants, it was believed to be caused by a chemical reaction, perhaps a type of vulcanization that causes the sealant to solidify in the joint after installation. The second peak was believed to be that of the base coal tar material of the sealant.

## FPL 6523

A total of 13 different FPL 6523 sealant conditions were analyzed using the GPC. Two sealant conditions, the FPL 6523-A and B weather-o-meter aged specimens, experienced sample absorption on the column. None of the FPL 6523-A weather-o-meter samples were used in additional analysis because only one of the sample runs did not experience absorption. Only one run of the FPL 6523-B weather-o-meter aged exhibited absorption so it was eliminated, and the other three chromatograms were used for continued analysis.

Six of the 95 percent confidence regions of FPL 6523 were less than 0.02 at the widest point, but the remaining confidence regions has maximum widths ranging up to 0.5. The confidence regions of FPL 6523 overlapped allowing only generalized trends to be discussed. Another factor which influences the overlapping of the confidence regions was the fact that the three columns in the GPC had to be replaced. The column used in the GPC analysis of some of the FPL 6523 samples became damaged or plugged. The damage was indicated by a steady increase in the pressure required to pump the sample and solvent through the system. The chromatograms obtained using the new columns exhibited a shift toward longer elution times. For example, the first peak of the FPL 6523-B weather-o-meter aged average chromatogram was first detected at approximately 20 min while the first peak of the FPL 6523-B unaged and oven-aged average chromatograms were detected at approximately 19 min. The shift of approximately 1 min increased the difficult of establishing an aging index for sealants and indicated that the GPC technique for analyzing joint sealants may not be repeatable between laboratories. The individual chromatograms for the FPL 6523 sealant conditions and the corresponding 95 percent confidence regions are provided in Appendix A.

The FPL 6523-A average chromatograms are illustrated in Figure 2. The as-received, unaged, and oven-aged samples were analyzed using the same columns; therefore, the samples eluted at similar times. The chromatograms exhibited a shift toward the early fraction as the sealant was aged. A bimodal profile was created as the sealant was heated to pour into the joints. The bimodal profile became more pronounced as the sealant was aged in the oven. The first peak illustrated in Figure 3 also exhibited an increase in height as the sealant was heated and aged. One would expect that the sealant should become harder with the development of the bimodal distribution and the corresponding increase in height of the first peak. The FPL 6523-A penetration values listed in Table 4 agreed with the trend observed in the chromatograms.

Even though the FPL 6523-A weather-o-meter aged chromatograms absorbed onto the columns, the chromatograms exhibited the development of a peak in the 18 to 22 min region. The development of the first peak indicated that the sealant should become harder as it was weather-o-meter aged. The weather-o-meter aged penetration value agrees with the assumption based on the first peak of the chromatogram.

The FPL 6523-B sealant analysis was conducted on two different sets of columns. The as-received, unaged, and oven-aged samples were analyzed on the original column bank and the weather-o-meter aged samples were analyzed on the new column bank. The average chromatograms of FPL 6523-B in Figure 4 illustrated the differences observed between the two different column banks. The weather-o-meter aged chromatogram had the same profile as the

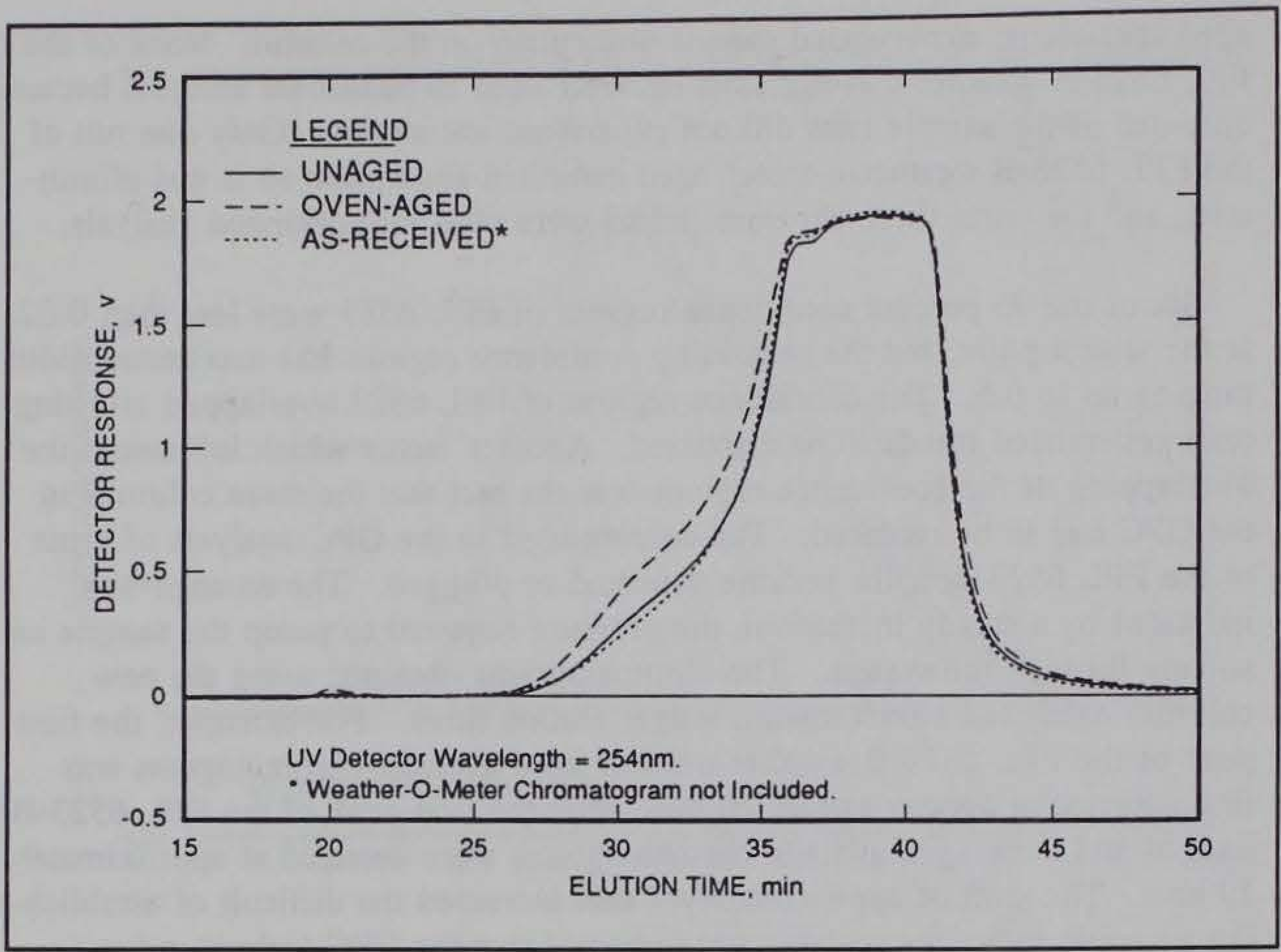


Figure 2. FPL 6523-A average chromatograms

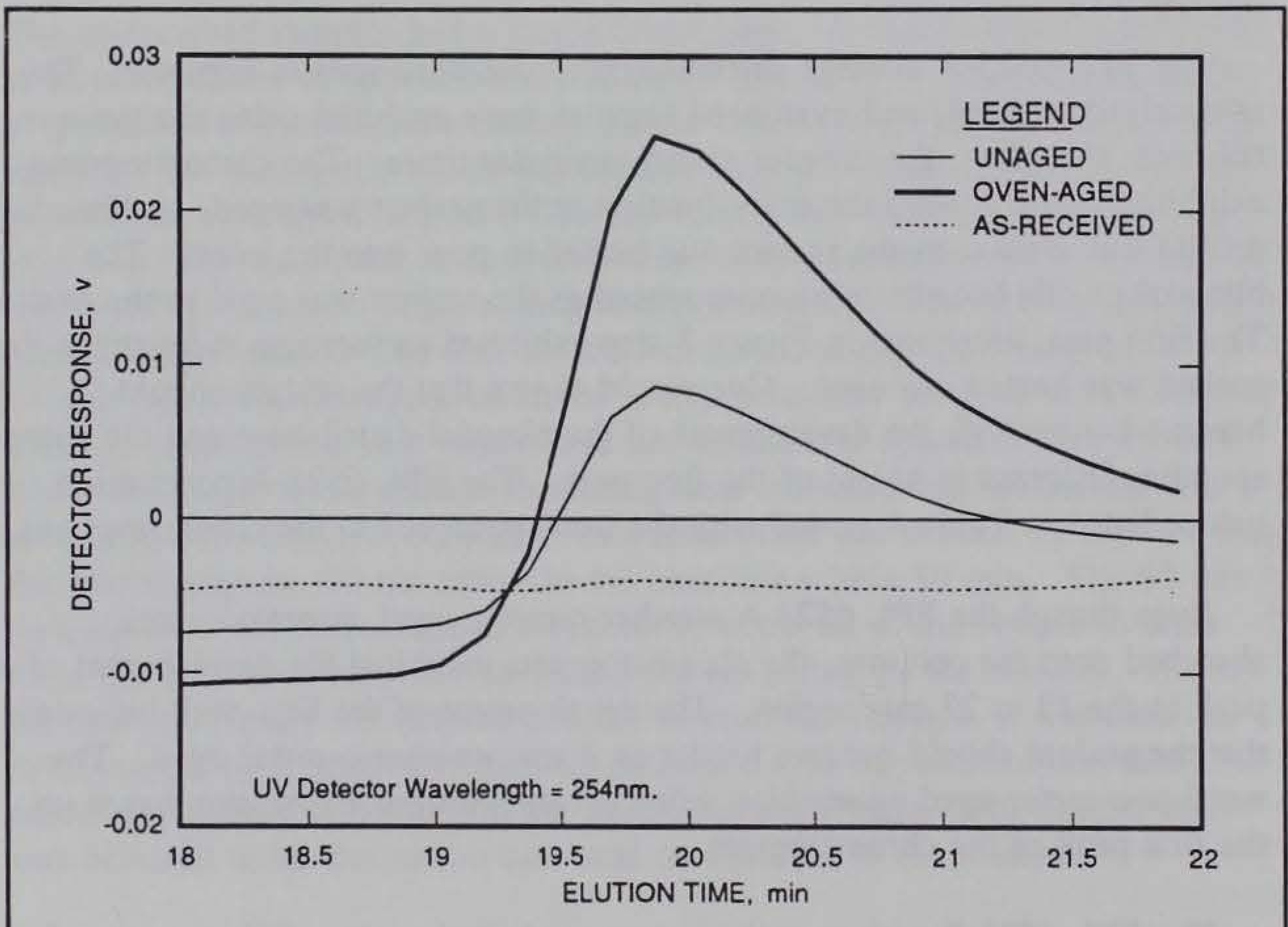


Figure 3. FPL 6523-A average chromatograms from 18 to 22 min

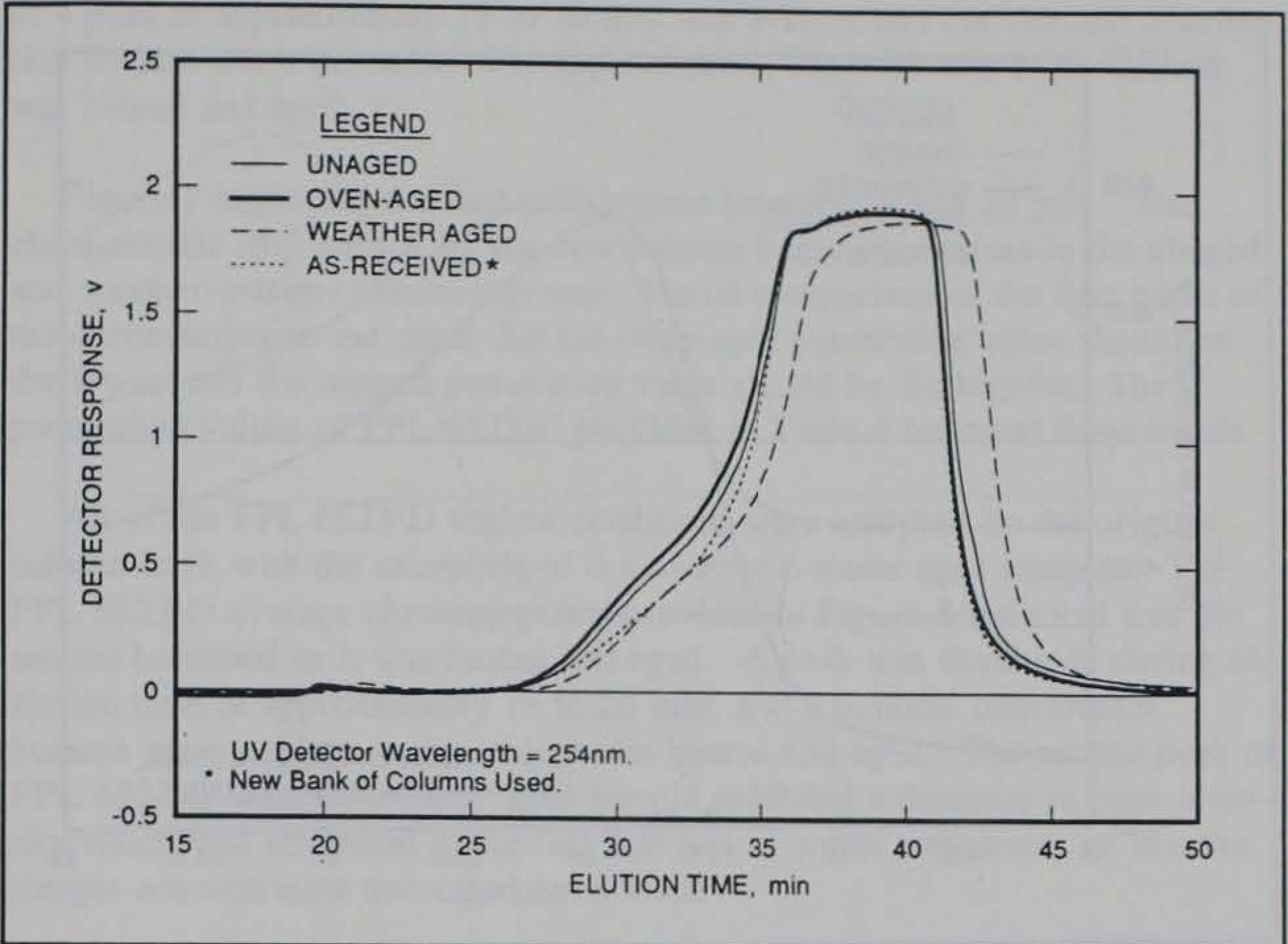


Figure 4. FPL 6523-B average chromatograms

unaged and oven-aged sample but the chromatogram has shifted to longer elution times. Figure 5, which provides the average chromatograms for the first peak, verified that the initial elution time increased. Normally, a shift toward the late fraction would be indicative of a decrease in the molecular size of the sample. However, a similar shift toward the late fraction was also noticed in the calibration curve of the new columns indicating that the sample was not actually experiencing a decrease in molecular size.

Because the FPL 6523-B weather-o-meter aged chromatogram did not decrease in molecular size but simply eluted at a longer time because of the change in columns, the weather-o-meter chromatogram was transposed to an initial elution time of approximately 19 min allowing a more direct comparison between the chromatograms. Visual comparisons of the first peak indicated that the oven-aged material should have the lowest penetration value and the unaged penetration value should be the highest. The conclusions were not based on the maximum peak height of the chromatograms, but on the total change in height from the sample base line. The penetration values for FPL 6523-B provided in Table 4 followed these trends.

Two of the FPL 6523-C chromatograms, the unaged and weather-o-meter aged samples, were obtained using the new column bank. These two chromatograms exhibited the same characteristic shift toward longer initial elution times as experienced by the FPL 6523-B weather-o-meter aged chromatogram. The average chromatograms of FPL 6523-C illustrated in Figure 6 have had similar profiles to the FPL 6523-A and B chromatograms. The development

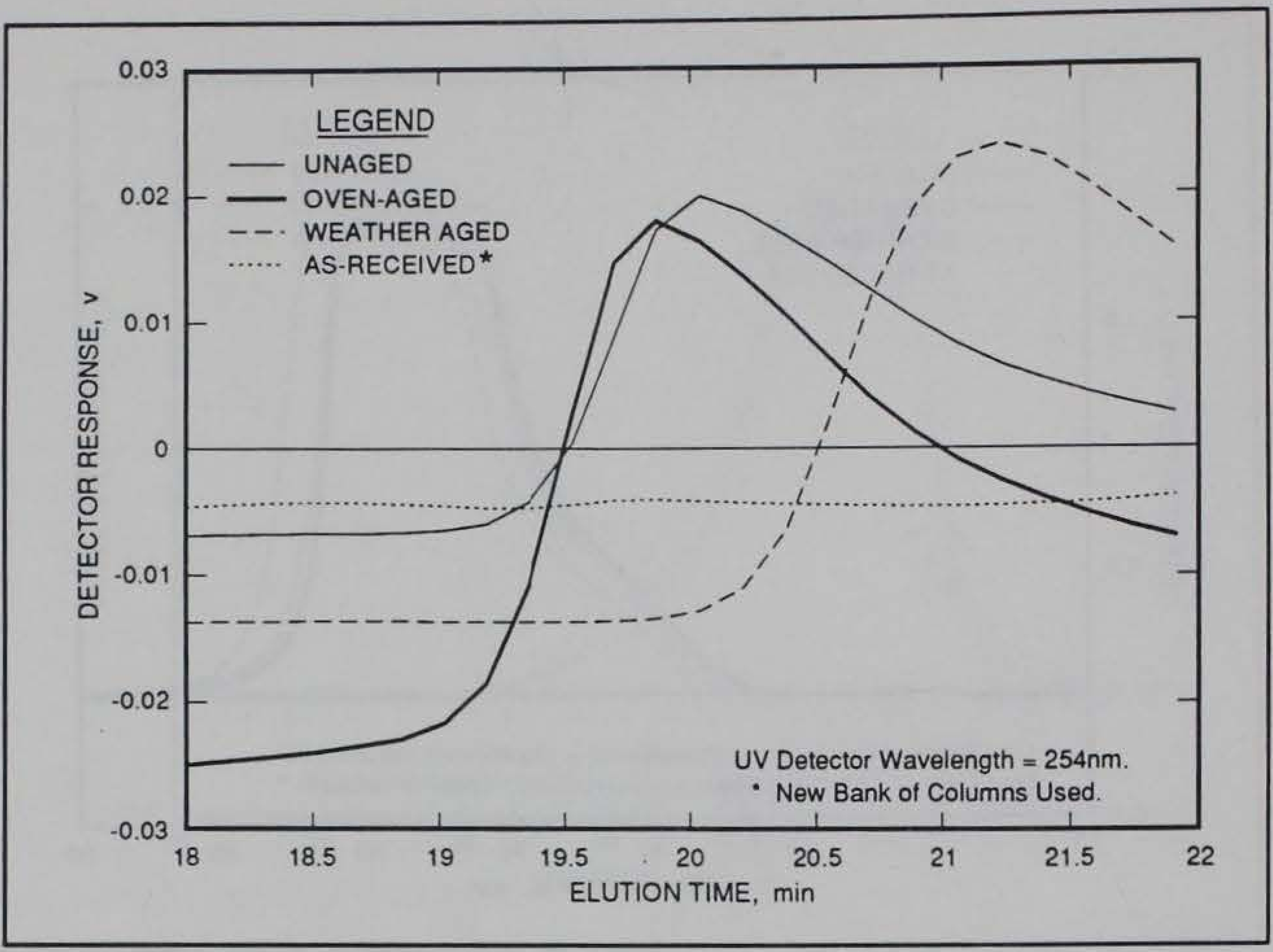


Figure 5. FPL 6523-B average chromatograms from 18 to 22 min

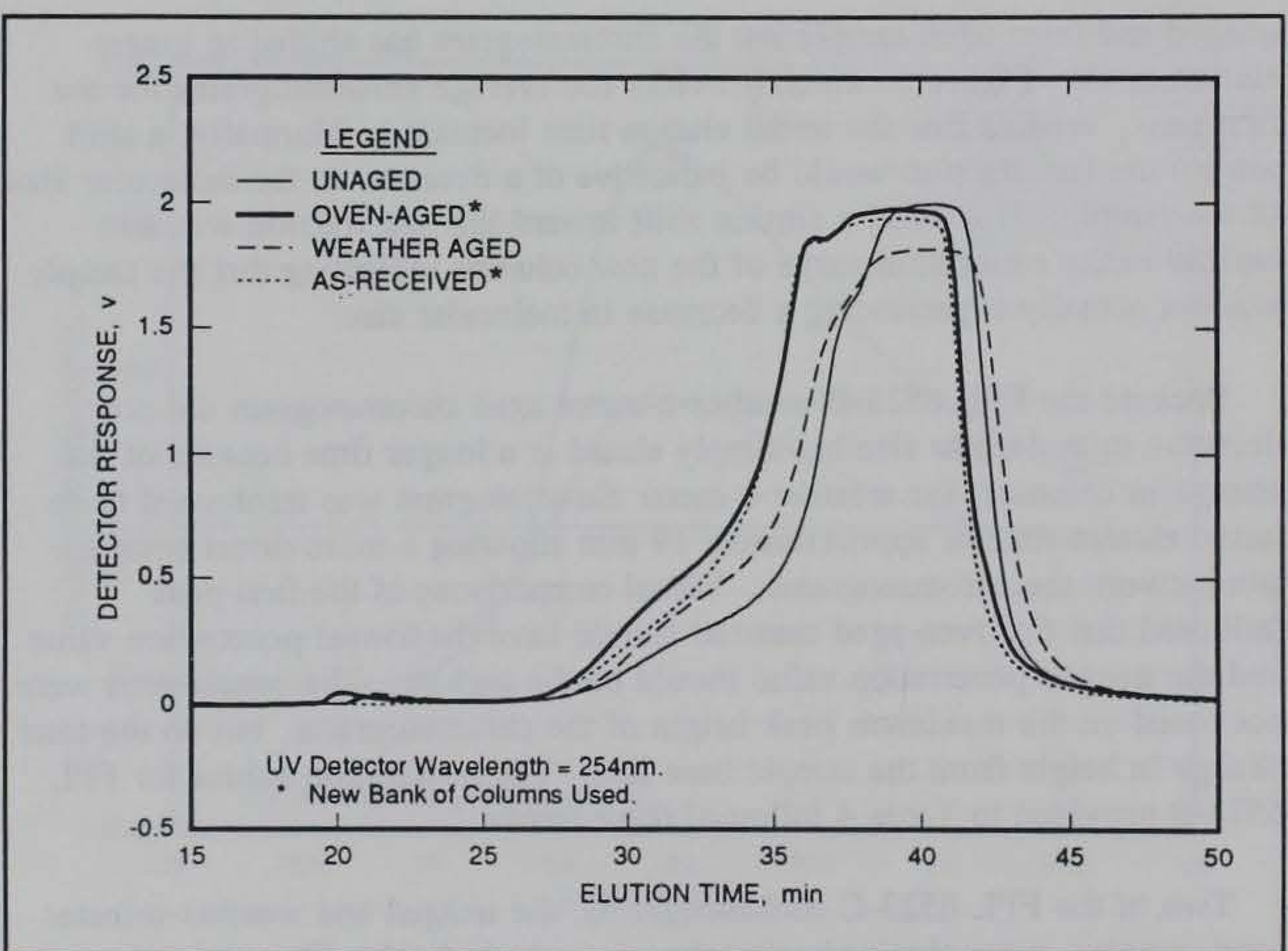


Figure 6. FPL 6523-C average chromatograms

of a peak at approximately 19 to 20 min was evident and the bimodal distribution became more pronounced at approximately 29 to 30 min as the sealant was heated and aged.

Figure 7 highlights the first peak region between 18 and 22 min. The characteristic shift caused by the new column bank was evident in the unaged and weather-o-meter chromatograms. Visual comparison of the first peaks of the chromatograms indicated that the oven-aged penetration value should be the lowest and the unaged penetration value should be the highest. The penetration values of FPL 6523-C provided in Table 4 followed these trends.

All of the FPL 6523-D sealant conditions were analyzed on the original column bank with the exception of the weather-o-meter aged samples. The FPL 6523-D average chromatograms provided in Figure 8 indicated that the sealant hardened as it was heated and aged. A peak was developed during an elution time of approximately 19 to 20 min, and a bimodal distribution became more evident as the sealant was heated and aged. The second peak of FPL 6523-D weather-o-meter aged sample exhibited a decrease in peak intensity which was attributed to the fact that new columns were used, or that the sample concentration decreased.

The development of the first peak of FPL 6523-D is illustrated in Figure 9. The weather-o-meter aged chromatogram exhibited the typical shift caused by the use of the new column bank. The visual comparison of the first peak region indicated that the penetration values of the sealant conditions should be similar. But the penetration value for the weather-o-meter aged sample should be the lowest and the unaged penetration value should be the highest. The FPL 6523-D penetration values provided in Table 4 did not agree with this trend. Instead, the penetration values follow the trend established in FPL 6523-A, B, and C with the oven-aged sample having the lowest penetration value and the unaged sample having the highest penetration value. The FPL 6523-D penetration also indicate that the unaged and weather-o-meter aged values were similar, but the oven-aged sample exhibits a significant decrease in penetration.

Comparing the first peaks of the different sealant conditions indicated that as the sealant was exposed to longer heating times an increase in hardening occurred. The hardening was evidenced by an increase in the maximum peak heights of the first peak ranging from approximately 0.025 volts for FPL 6523-A to approximately 0.055 volts for FPL 6523-D. The penetration values follow the trend exhibited by the chromatograms with the exception of the FPL 6523-B unaged and weather-o-meter aged penetration results which had a slightly higher penetration value than their FPL 6523-A counterpart.

The percentage of the total area of the first two slices were arbitrarily selected and added together to establish an aging index. By taking the percentage of the total area of the first two slices or a normalized area, effects caused by changes in sample concentrations are minimized. The calculated aging index for each of the FPL 6523 sealant conditions are illustrated in Figure 10. The slice data and slice data summaries for each sealant condition

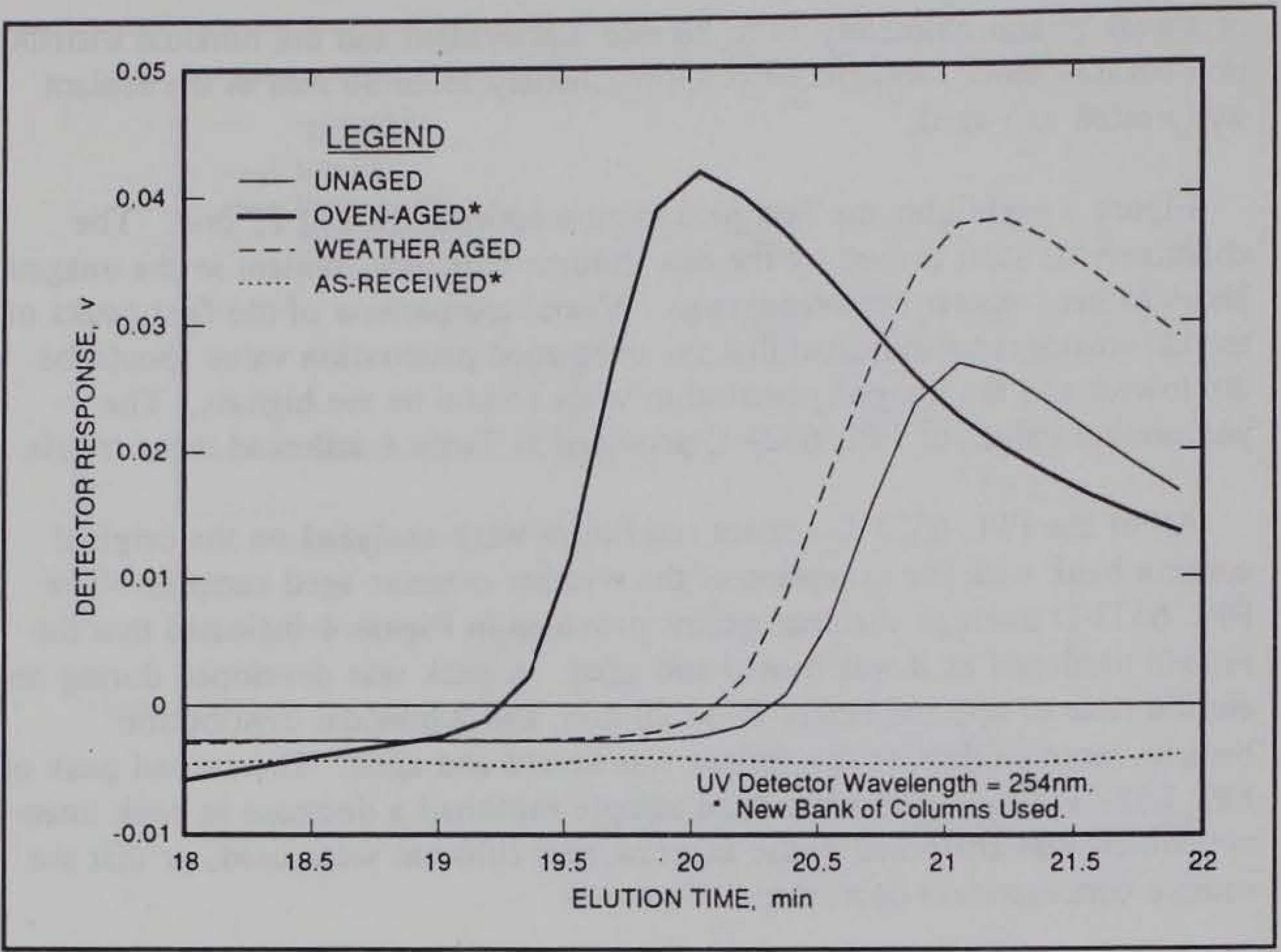


Figure 7. FPL 6523-C average chromatograms from 18 to 22 min

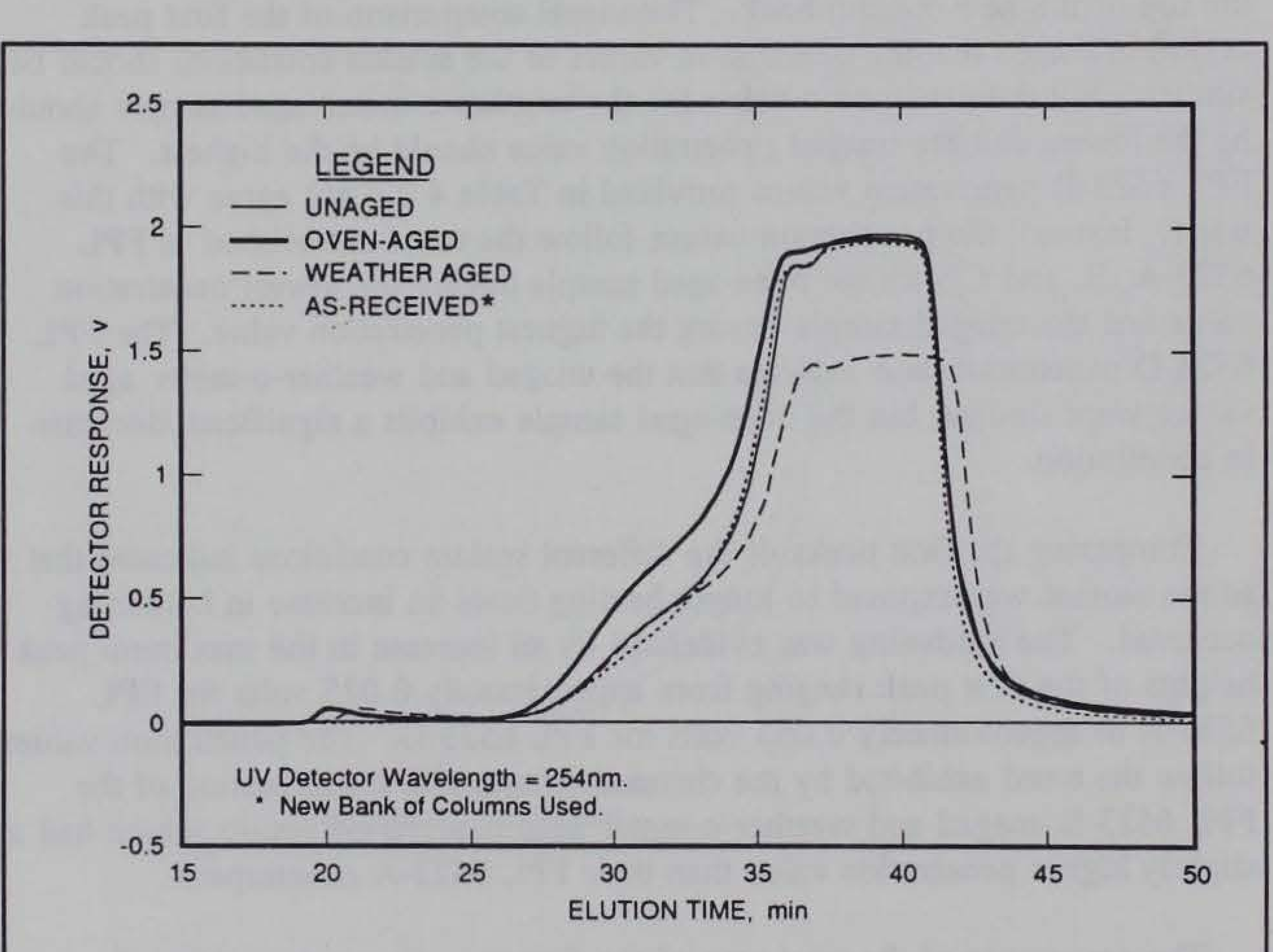


Figure 8. FPL 6523-D average chromatograms

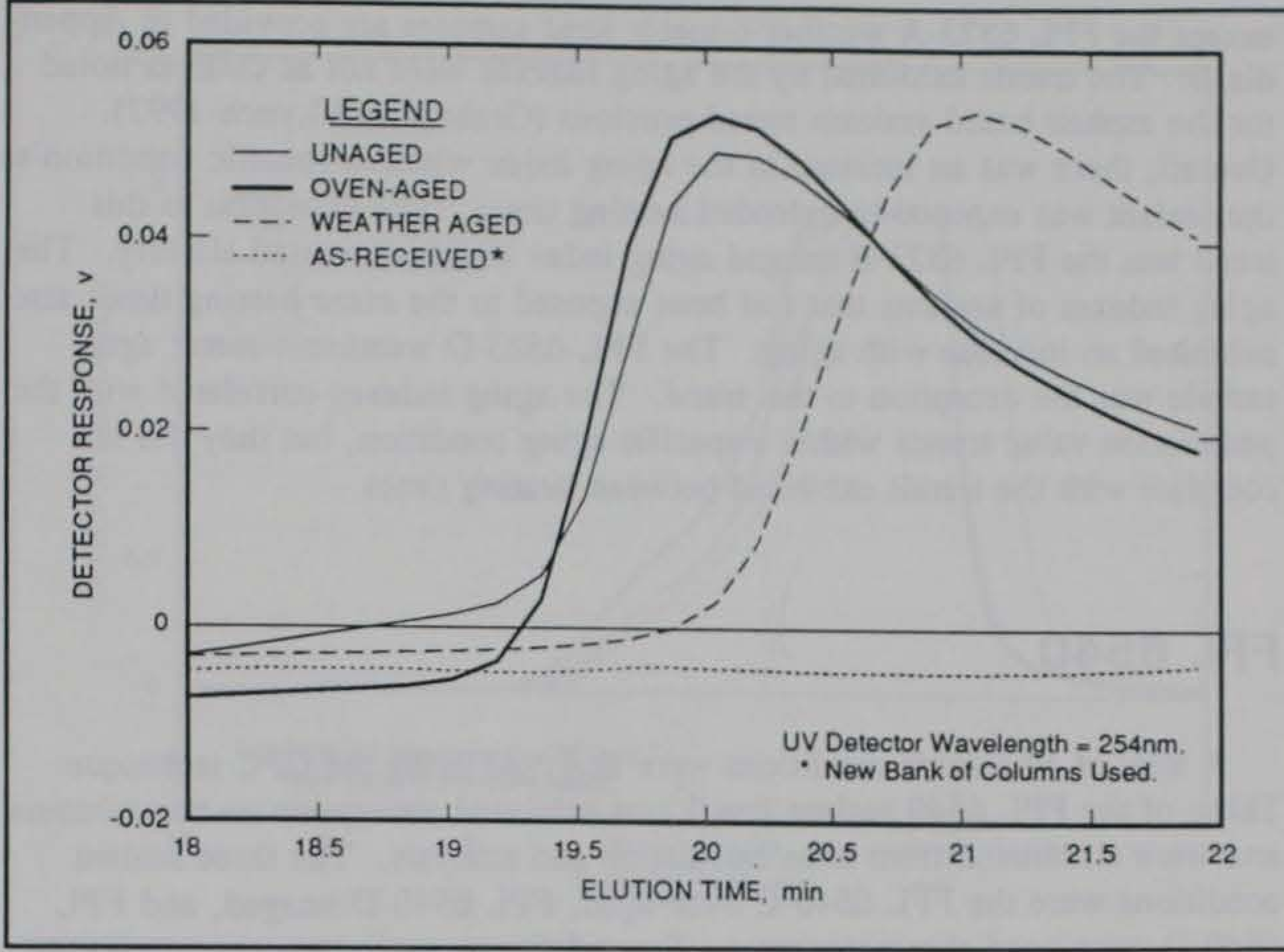


Figure 9. FPL 6523-D average chromatograms from 18 to 22 min

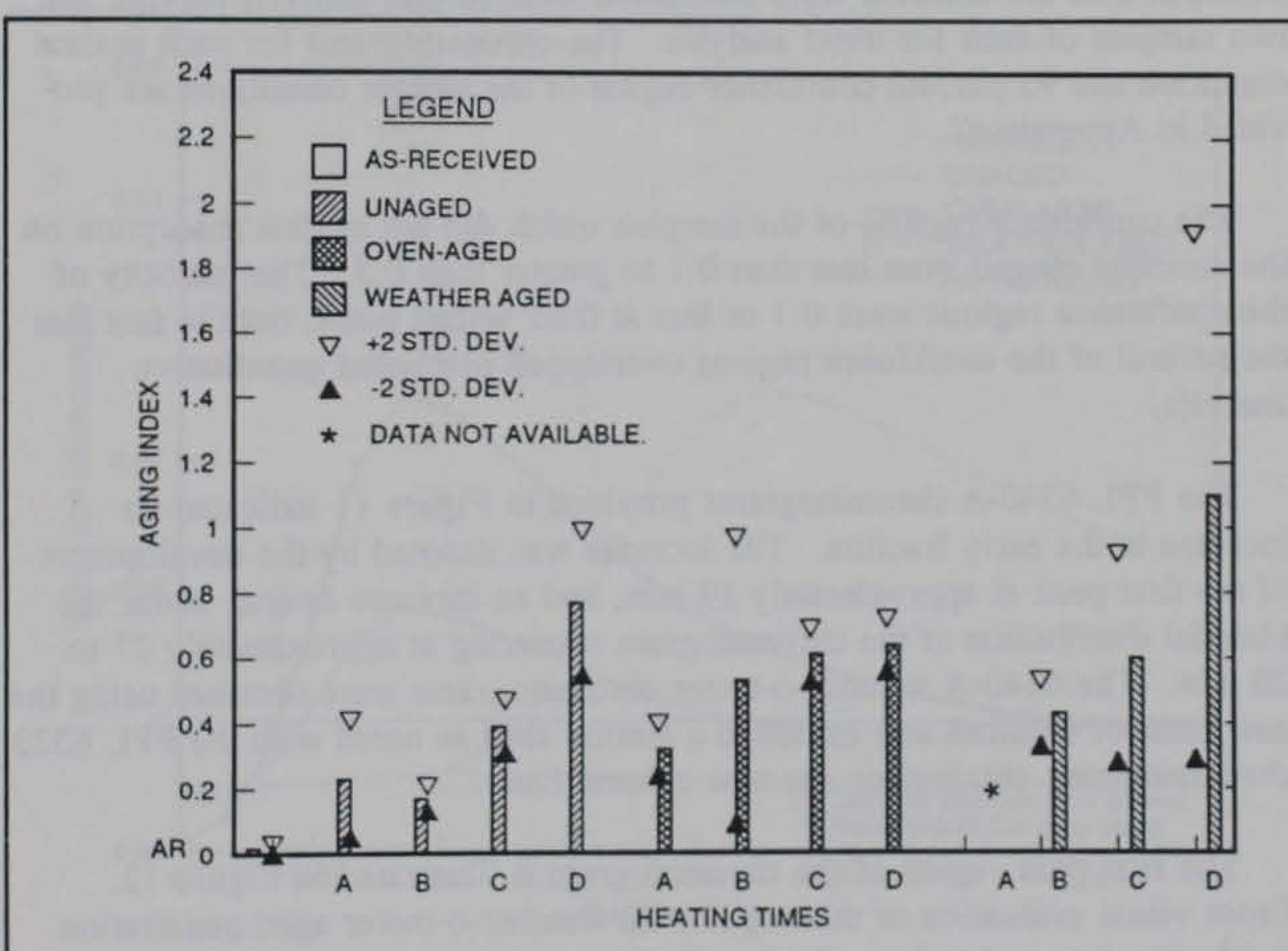


Figure 10. Aging index of FPL 6523 using area percentages of Slices 1 and 2

except the FPL 6523-A weather-o-meter aged samples are provided in Appendix B. The trends exhibited by the aging indexes were not as clear as noted for the asphalt based sealants tested previous (Graham and Lynch 1992). Overall, there was an increase in the aging index within a specific condition as the sealant was exposed to extended heating times. The exception to this trend was the FPL 6523-B unaged aging index which decreased slightly. The aging indexes of sealants that had been exposed to the same heating times also exhibited an increase with aging. The FPL 6523-D weather-o-meter aged sample was the exception to this trend. The aging indexes correlated with the penetration value trends within a specific aging condition, but they did not correlate with the trends exhibited between heating times.

## FPL 6540

A total of 13 sealant conditions were analyzed using the GPC technique. Three of the FPL 6540 sealant conditions exhibited absorption on the columns and were eliminated from slice calculation and analysis. The three sealant conditions were the FPL 6540-C oven-aged, FPL 6540-D unaged, and FPL 6540-D oven-aged chromatograms. Two of the remaining sealant conditions, the FPL 6540-A weather-o-meter aged and 6540-B unaged material, had two samples each that experienced absorption. The two samples that exhibited absorption on the columns were eliminated from further analysis leaving just two samples of each for trend analysis. The chromatograms for each sealant condition and 95 percent confidence region of the sealant conditions are provided in Appendix C.

The confidence regions of the samples which did not exhibit absorption on the columns ranged from less than 0.1 to greater than 0.5. The majority of the confidence regions were 0.1 or less at their widest point, but the fact that the several of the confidence regions overlapped precluded quantitative analysis.

The FPL 6540-A chromatograms provided in Figure 11 indicated an increase in the early fraction. The increase was denoted by the development of the first peak at approximately 19 min, and an increase in area under the bimodal distribution of the chromatogram occurring at approximately 27 to 28 min. The 6540-A weather-o-meter chromatograms were obtained using the new bank of columns and exhibited a similar shift as noted with the FPL 6523 chromatograms obtained on the new column bank.

The first peak region of the chromatogram is illustrated in Figure 12. From visual evaluation of this region, the weather-o-meter aged penetration value was expected to have the lowest value and the unaged penetration value should be the highest. The actual FPL 6540-A penetration values provided in Table 5 did not correspond to the trend expected from the chromatograms. The penetration values indicated that the oven-aged sample was significantly harder than the weather-o-meter aged sample. The chromatograms and penetration data agreed in a general way that aging hardened the sealant.

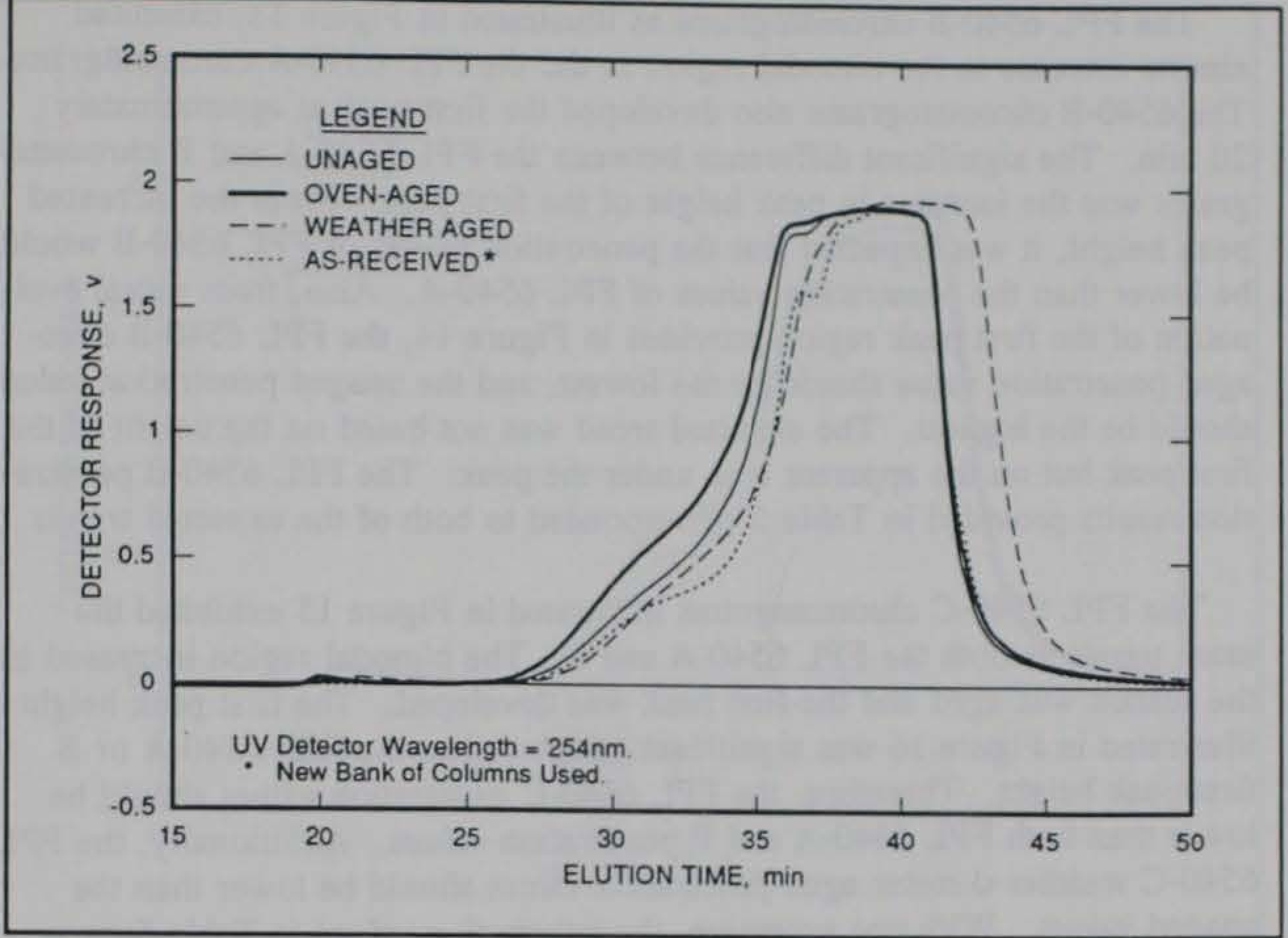


Figure 11. FPL 6540-A average chromatograms

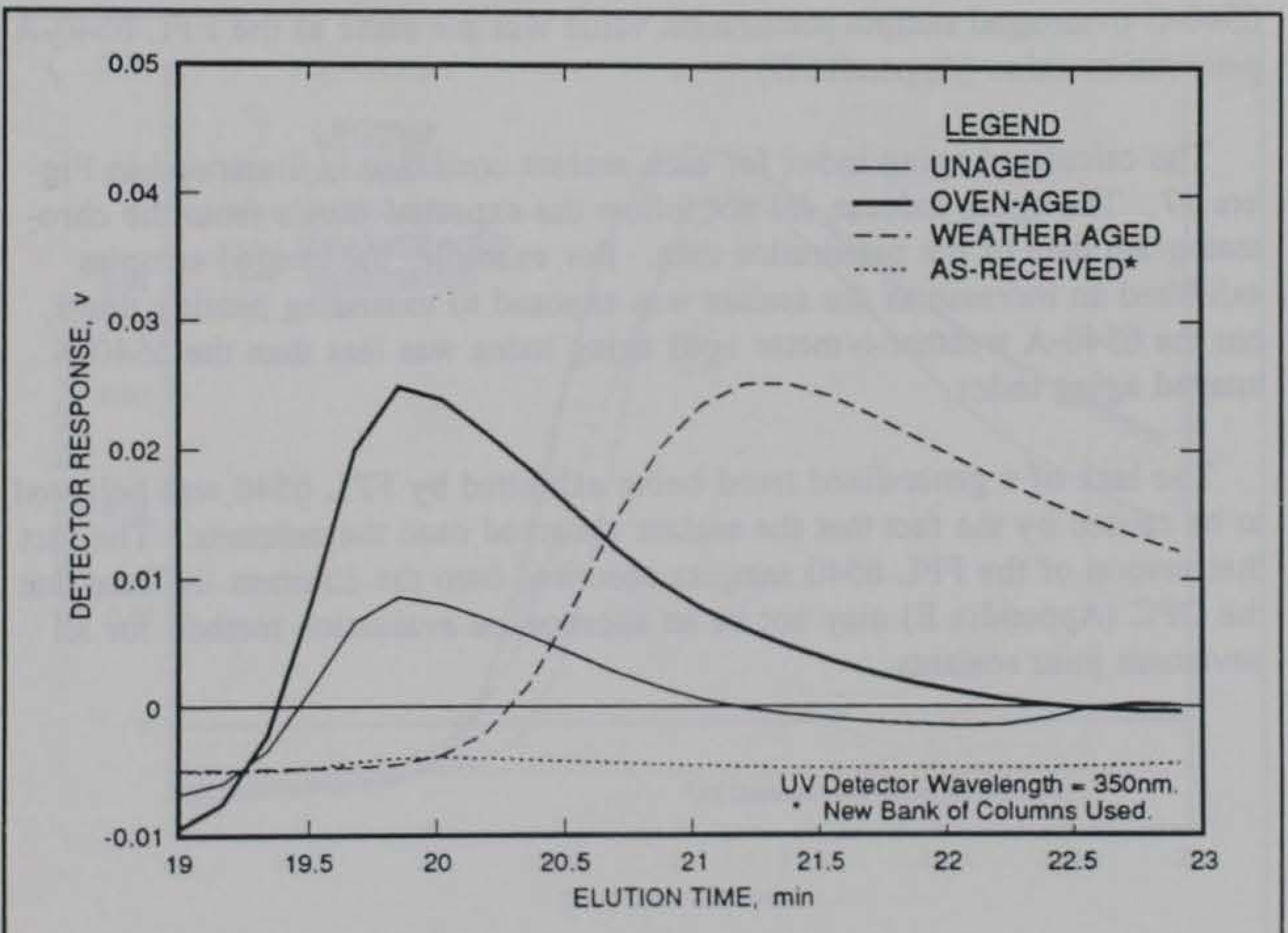


Figure 12. FPL 6540-A average chromatograms from 19 to 23 min

The FPL 6540-B chromatograms as illustrated in Figure 13, exhibited similar increase in the bimodal region as did the FPL 6540-A chromatograms. The 6540-B chromatograms also developed the first peak at approximately 20 min. The significant difference between the FPL 6540-A and B chromatograms was the increase in peak height of the first peak. From the increased peak height, it was expected that the penetration values of FPL 6540-B would be lower than the penetration values of FPL 6540-A. Also, from visual evaluation of the first peak region provided in Figure 14, the FPL 6540-B oven-aged penetration value should be the lowest, and the unaged penetration value should be the highest. The expected trend was not based on the height of the first peak but on the apparent area under the peak. The FPL 6540-B penetration results provided in Table 5 corresponded to both of the expected trends.

The FPL 6540-C chromatograms illustrated in Figure 15 exhibited the same trends as both the FPL 6540-A and B. The bimodal region increased as the sealant was aged and the first peak was developed. The first peak height illustrated in Figure 16 was significantly higher than the FPL 6540-A or B first peak height. Therefore, the FPL 6540-C penetration values should be lower than both FPL 6540-A and B penetration values. Additionally, the FPL 6540-C weather-o-meter aged penetration values should be lower than the unaged values. With one exception, the penetration values in Table 5 corresponded with the trends expected by the chromatograms. The FPL 6540-C unaged and weather-o-meter aged sample penetration values were lower than the corresponding penetration values of FPL 6540-A and B, but the FPL 6540-C oven-aged sample penetration value was the same as the FPL 6540-A penetration value (Appendix D).

The calculated aging index for each sealant condition is illustrated in Figure 17. The aging indexes did not follow the expected trends from the chromatogram data or the penetration data. For example, the unaged samples exhibited an increase as the sealant was exposed to extending heating times, but the 6540-A weather-o-meter aged aging index was less than the 6540-A unaged aging index.

The lack of a generalized trend being exhibited by FPL 6540 was believed to be caused by the fact that the sealant absorbed onto the columns. The fact that several of the FPL 6540 samples absorbed onto the columns indicate that the GPC (Appendix E) may not be an appropriate evaluation method for all pavement joint sealants.

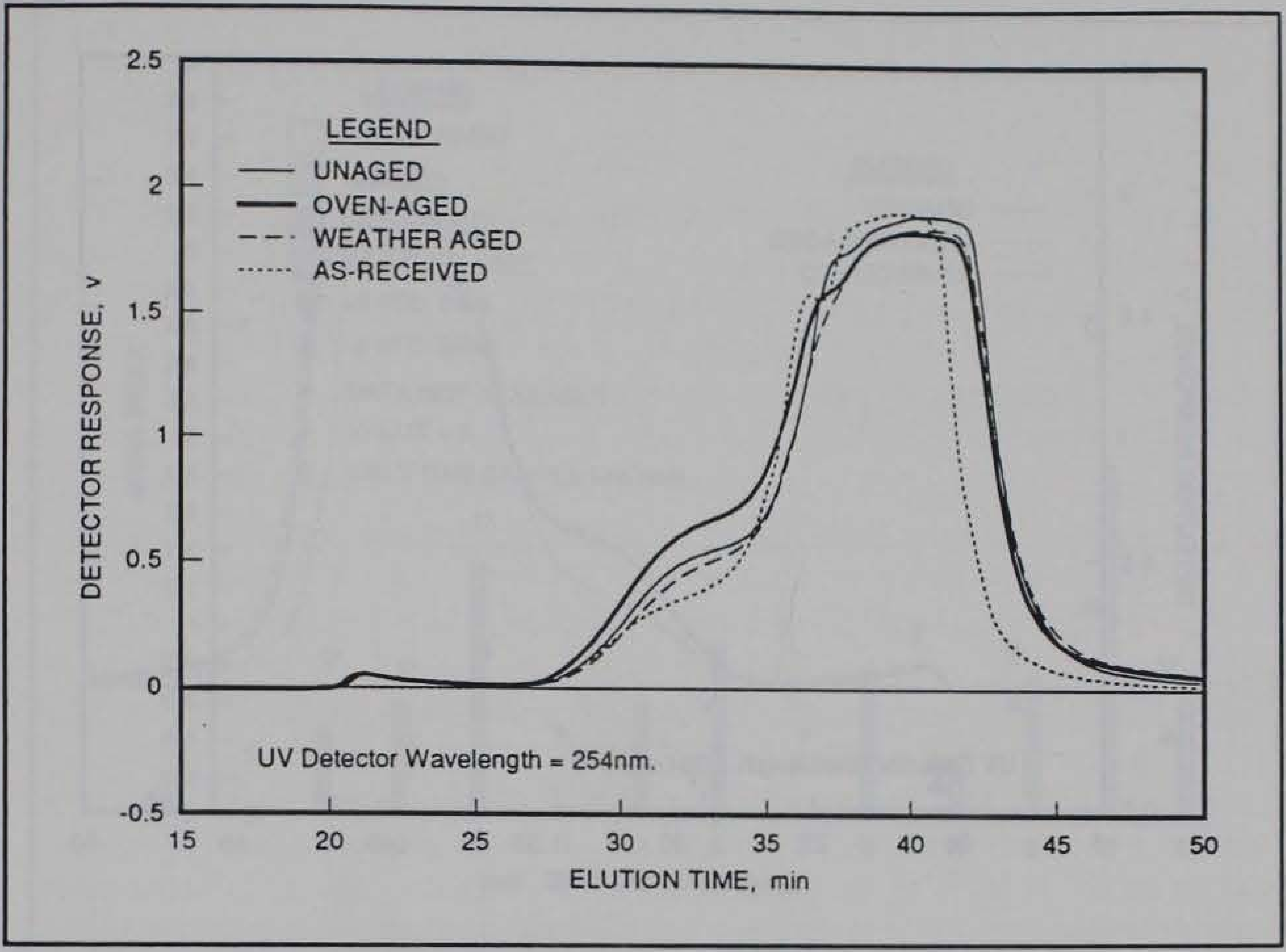


Figure 13. FPL 6540-B average chromatograms

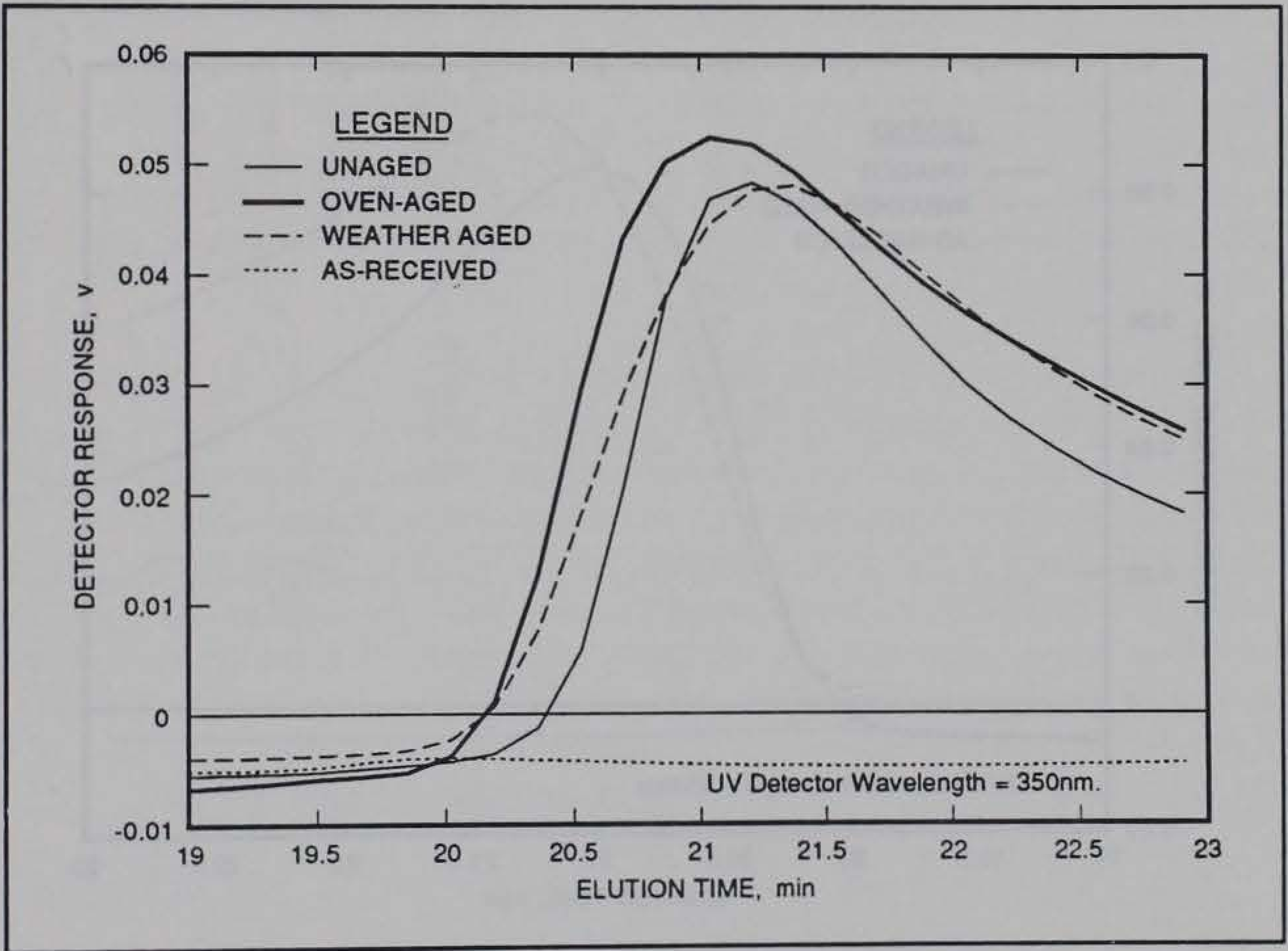


Figure 14. FPL 6540-B average chromatograms from 19 to 23 min

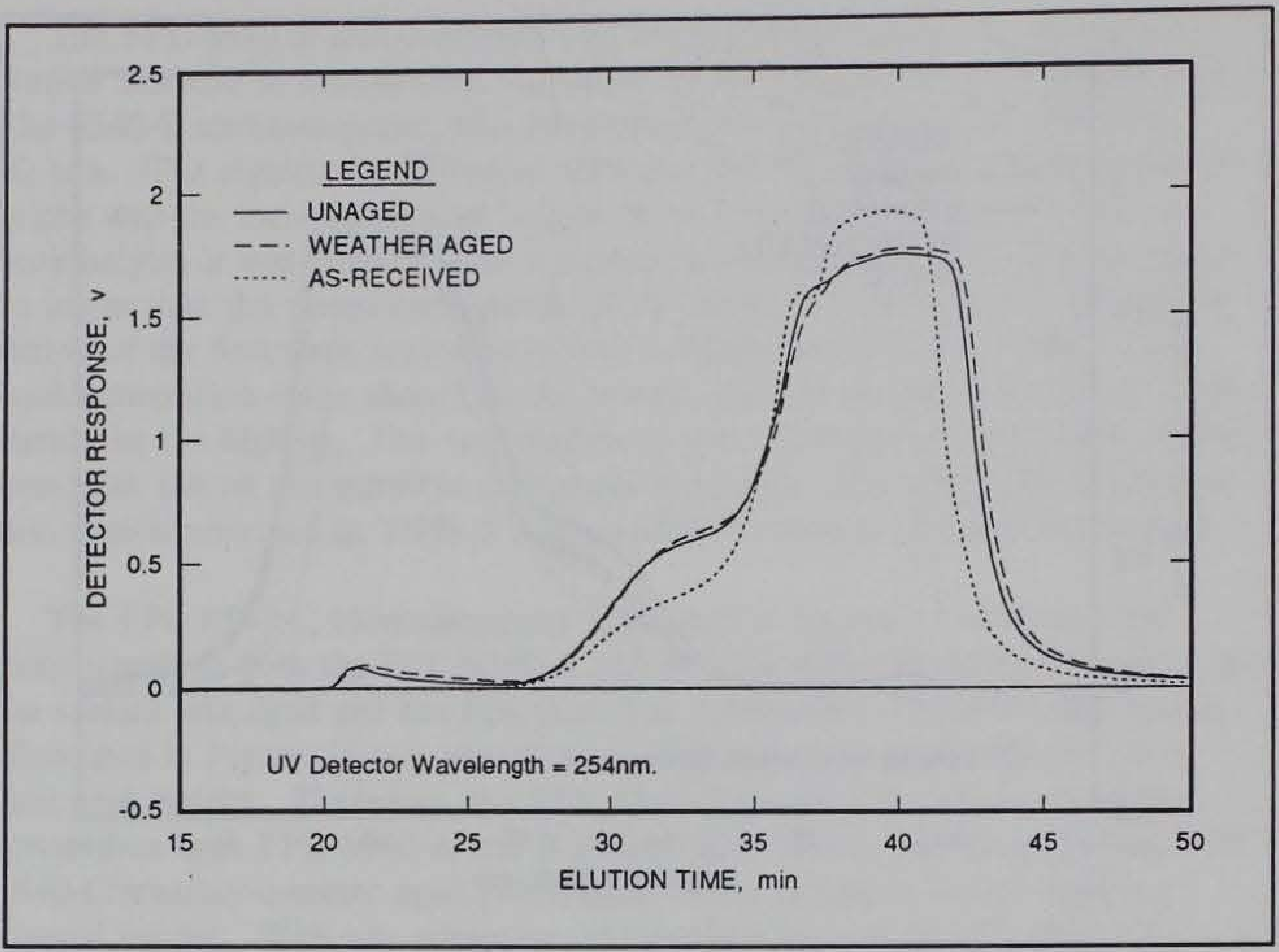


Figure 15. FPL 6540-C average chromatograms

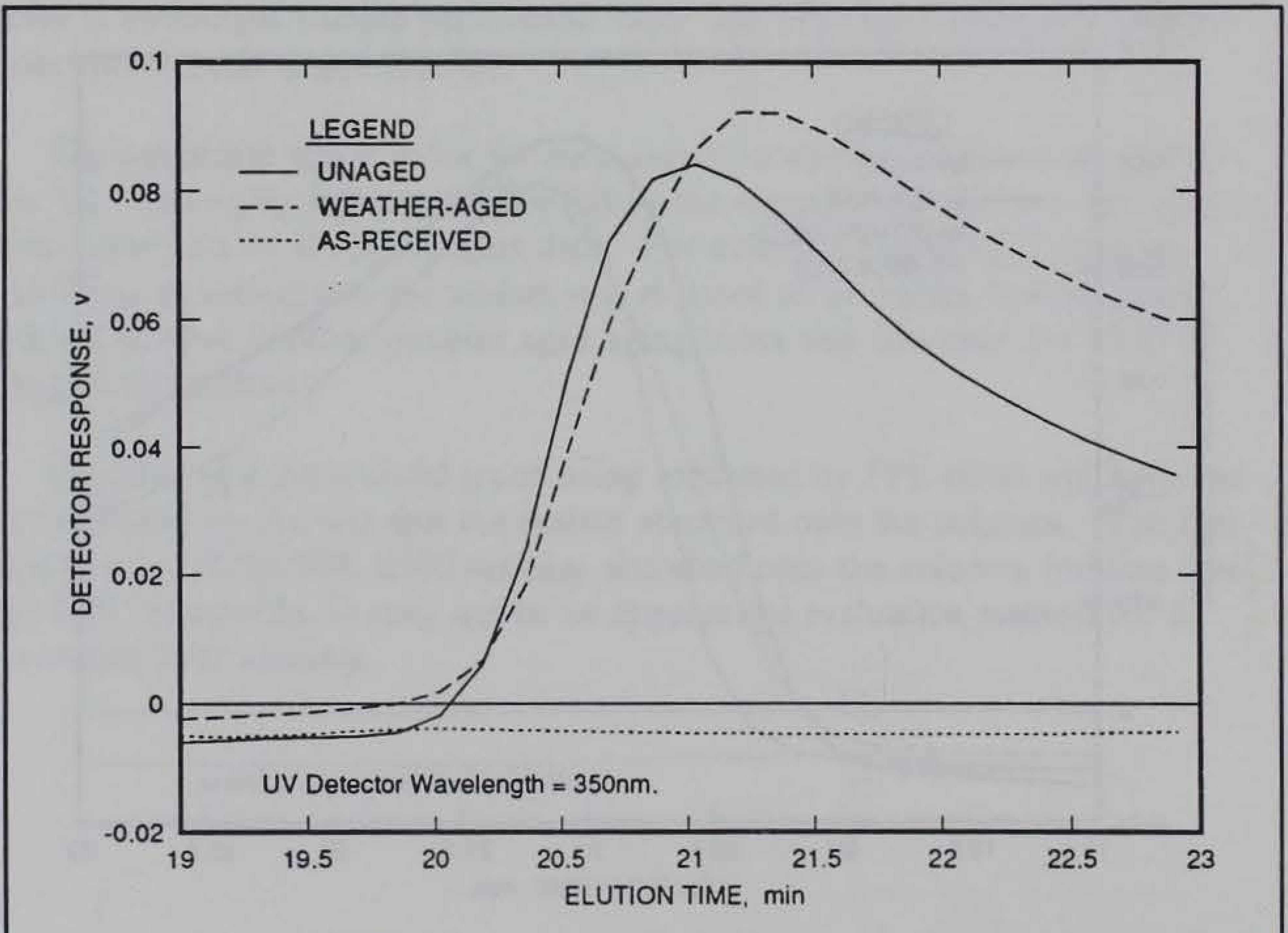


Figure 16. FPL 6540-B average chromatograms from 19 to 23 min

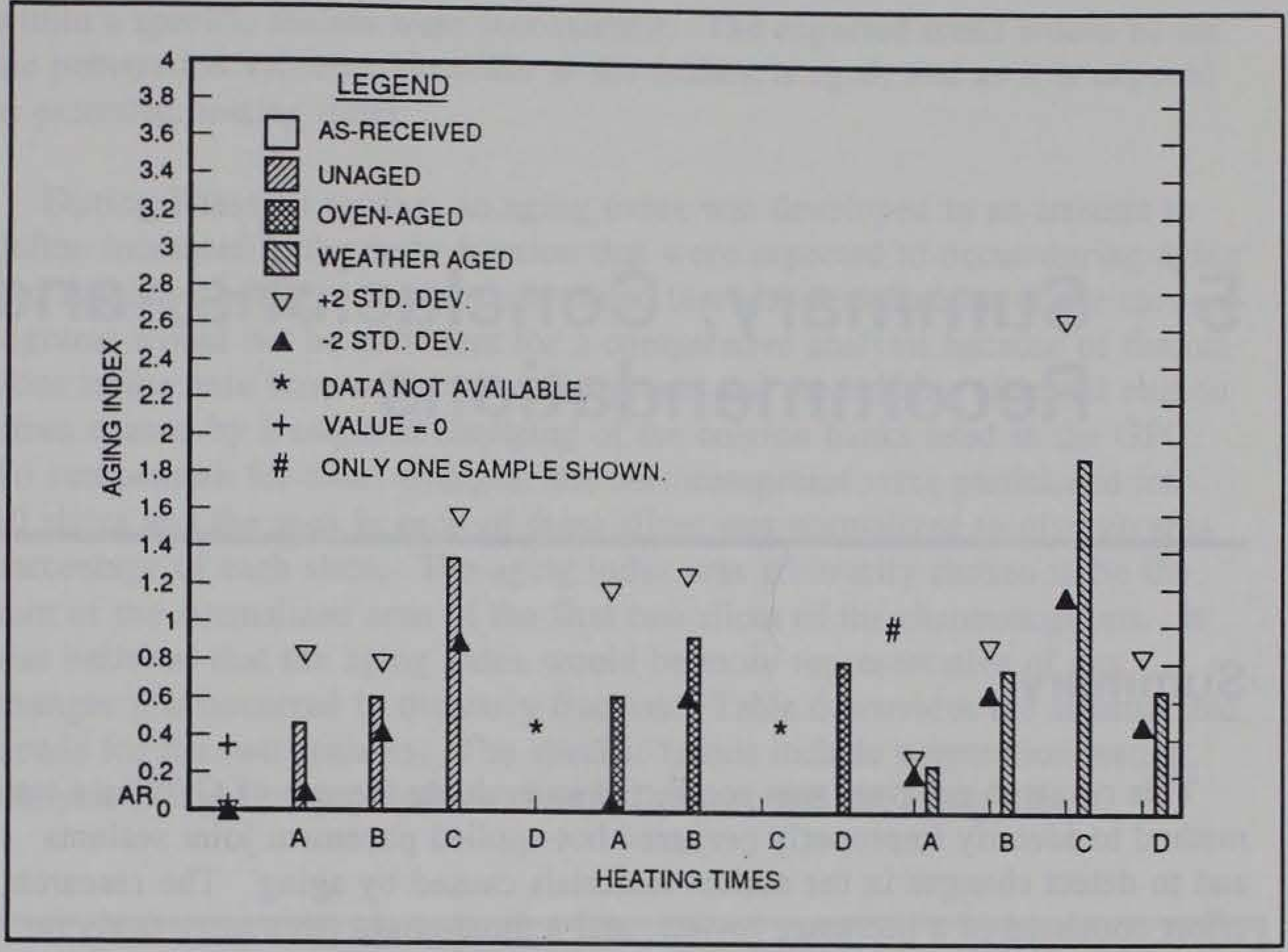


**Figure 17.** Aging index of FPL 6540 using area percentages of Slices 1 and 2

# 5 Summary, Conclusions, and Recommendations

## Summary

This research program was conducted to evaluate the use of GPC as a test method to identify improperly prepared hot-applied pavement joint sealants and to detect changes in the sealant materials caused by aging. The research effort consisted of a literature review, and a three-phase laboratory study on laboratory prepared and aged specimens. Phases I and II of the laboratory study involved material property testing of two pavement joint sealant materials. The two sealants tested were coal tar-based sealants manufactured to meet the requirements of Federal Specification SS-S-1614A. Phase III testing was the GPC analysis of sealants tested in Phases I and II. The objective of this research was to determine if GPC could be used to identify or detect changes in the sealants caused by prolonged heating and/or aging. The research objective was partially achieved. The GPC technique did detect changes in the various sealant samples, but the changes were inconsistent and did not correlate to physical test data.

The review of the literature indicated that GPC had been used extensively to analyze polymeric materials, and attempts had been made to analyze nonpolymeric materials such as asphalt cements and coal tar pitches. However, no data were found concerning the GPC analysis of pavement joint sealant materials. The majority of the literature concluded that changes in the analyzed materials which were caused by aging could be detected using GPC. Opinions varied concerning the significance of changes that are detected when analyzing nonpolymeric materials using GPC, but the literature (Graham and Lynch 1992) generally agreed that an increase in the early fraction portion of the chromatogram indicates a hardening of the material being analyzed.

Phase I testing indicated that both of the sealants manufactured to meet the requirements of Federal Specification SS-S-1614A did meet the test requirements. Phase II testing was designed to determine the effects different heating times and various types of artificial aging on the physical properties specified in the Federal Specifications. Phase II testing indicated that changes could be detected in the penetration results of most of the sealants, but the changes did not necessarily follow expected trends, and the changes detected within a specific sealant were inconsistent. The expected trend would be for the penetration values to decrease as the sealant is aged, and as it is exposed to extended heating times.

During Phase III testing, an aging index was developed in an attempt to define increases in the early fraction that were expected to occur during aging and extended heating. It was determined that visual evaluation of the chromatograms would not be sufficient for a comparative analysis because of fluctuations in the base lines of the chromatograms and the shifting of initial elution times caused by a required changing of the column banks used in the GPC. To compensate for these changes, the chromatograms were partitioned into 10 slices and the area in each of these slices was normalized to give an area percentage of each slice. The aging index was arbitrarily chosen to be the sum of the normalized area of the first two slices of the chromatograms. It was believed that the aging index would be more representative of any changes that occurred in the early fraction. Table 6 provides the summarized trends for the two sealants. The specific trends include penetration testing, analysis of the first peak of the chromatograms, and the aging index.

**Table 6**
**Summarized Trend Analysis[1]**

| Sealant Material | Penetration Results | First Peak | Aging Index |
|---|---|---|---|
| FPL 6523 | General decrease as sealant was aged and as heating time was extended. | General increase as sealant was aged and as heating time was extended. | General increase as sealant was aged and as heating time increased. |
| FPL 6540 | General decrease as sealant was aged as heating time as extended. | General increase as sealant was aged. Large increase as heating time was extended. Some material absorbed on columns. | General increase as heating time increased but no consistent trend as sealant was aged. |

[1] Only generalized trends are presented here. There were several exceptions to the trends, and overlapping of the 95 percent confidence region occurred between specimens.

## Conclusions

Based on the results of the investigation including the literature review and the three-phase laboratory study, the following conclusions were made concerning the use of GPC as a method to identify joint sealants which have been improperly prepared, and how the improper preparation affects the physical properties of the sealant:

*a.* GPC can be used to detect changes in a sealant caused by extended heating or aging if the original material has been evaluated. The changes that were detected are qualitative not quantitative and the changes did not correlate with physical tests such as penetration.

*b.* The aging index established for the joint sealant materials during the GPC analysis exhibited a trend which indicated that extended heating and aging of the sealants increased the early fraction of the chromatogram. However, the trend was not consistent for both joint sealant materials.

*c.* The chromatograms obtained by analyzing the joint sealant materials had significant variability. When 95 percent confidence regions were established for individual sealant conditions, several of them overlapped. Therefore, GPC analysis of unknown sealant samples would be difficult.

*d.* The procedures used to conduct the GPC analysis are extremely important. The procedures must be carefully thought out and patiently followed. Any deviation from the prescribed procedures will adversely affect the resulting chromatograms.

*e.* Any changes in equipment, especially in the columns, will affect the resulting chromatograms. Therefore, reproducibility of GPC analysis of pavement joint sealants between laboratories will be difficult.

*f.* Penetration values of the sealants generally exhibited a change as the sealants were exposed to extended heating or to artificial aging. The changes in penetration were not consistent with expected trends, and the amount of change varied with each sealant.

*g.* Changes experienced in the penetration values did not appear to affect the various sealants' ability to adhere to concrete as demonstrated by satisfactory results of almost all of the bond testing. Subsequently, correlations between field performance characteristics such as adhesion and the penetration results were not exhibited.

## Recommendations

Based on the literature review and laboratory analysis of this research effort, the following recommendations were made:

*a.* To maximize the potential use of GPC in the analysis of sealants, alternate detection methods such as a photodiode array (PDA) should be investigated. The PDA can analyze several wavelengths at the same time, and, therefore, may provide a clearer "fingerprint" of the sealant being evaluated. The PDA could also allow the variability of the sealant materials to be more readily defined.

*b.* Investigations which combine techniques such as Fourier Transform Infrared Spectroscopy (FTIR) with GPC should be conducted. This type of analysis should provide a more quantitative analysis of the sealants.

*c.* Round robin testing should be conducted on the laboratory test procedures used to evaluate the physical properties of joint sealants and on GPC analysis of nonpolymeric materials. The round robin testing would allow significant changes in penetration and resilience testing, and chromatograms to be defined.

*d.* Research is still required to develop a test method that more accurately detects sealant materials that have been improperly prepared and installed. Investigations are also required to determine realistic life cycles and working ranges of pavement joint sealant materials.

*e.* There is a need to standardize a sampling procedure for joint sealing materials that are solid at room temperature to assure that the same material as that which is mixed in the melter in the field is tested.

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# Appendix A
# FPL 6523 Chromatograms

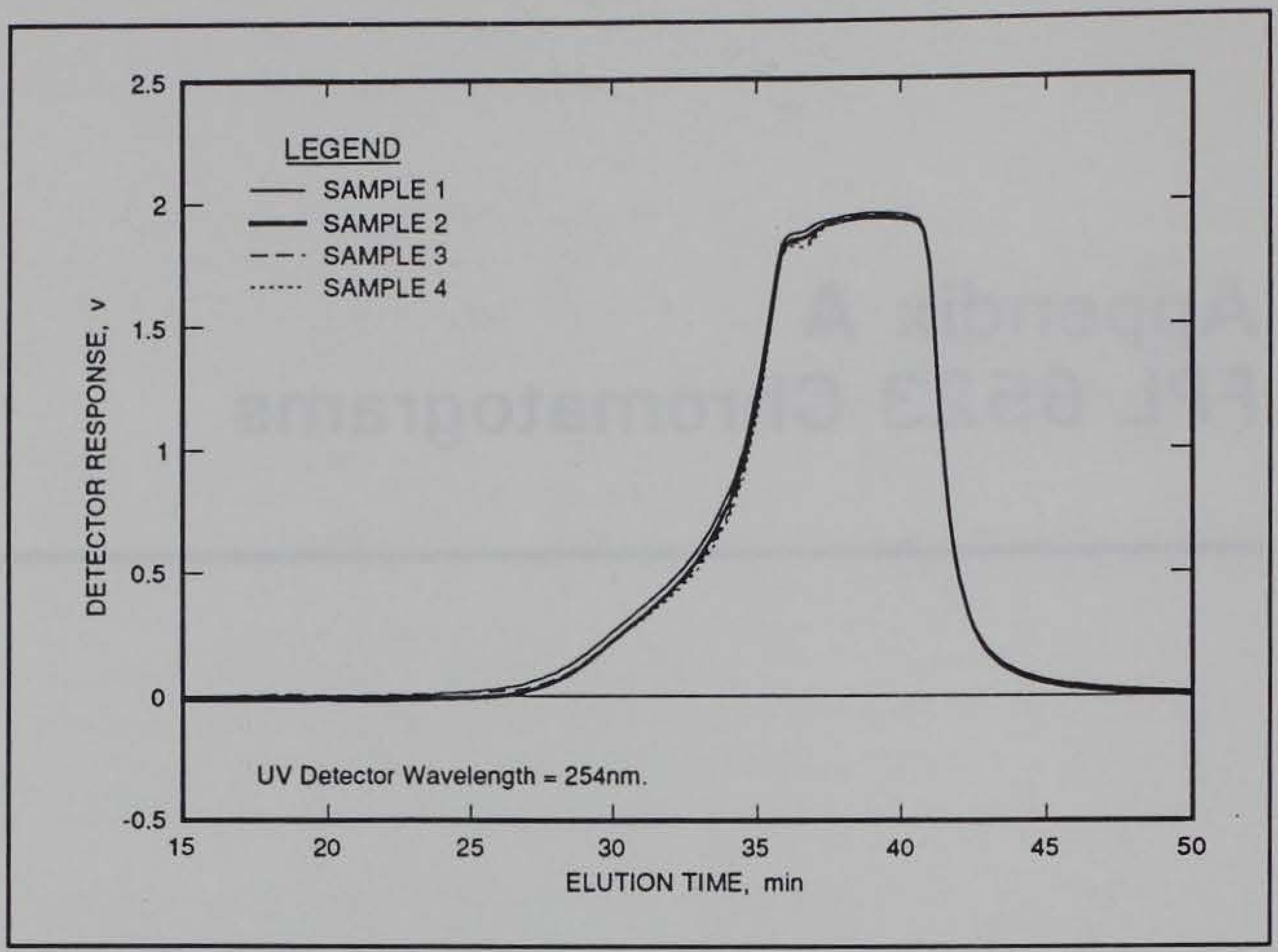


Figure A1. FPL 6523 as-received chromatograms

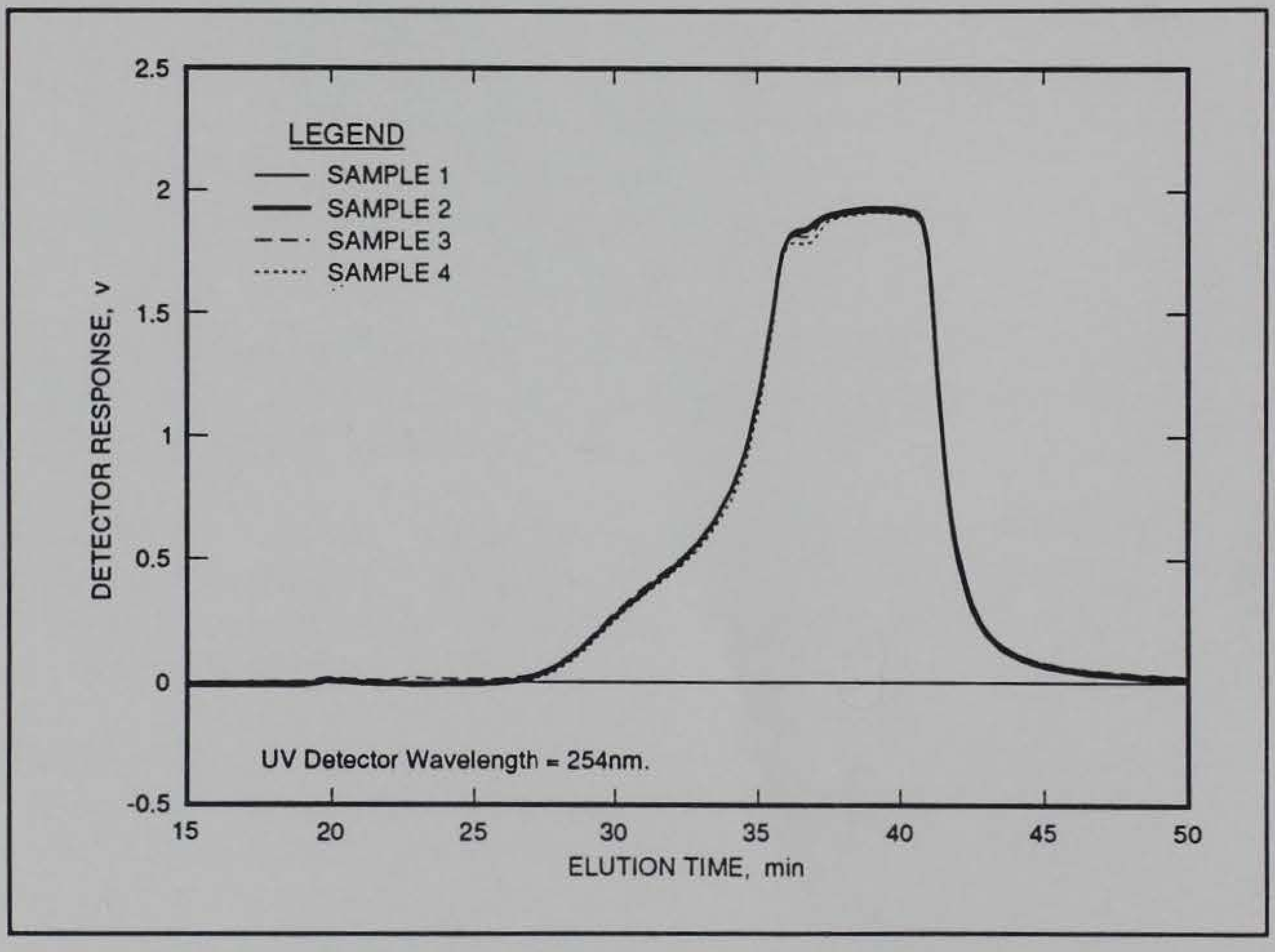


Figure A2. FPL 6523-A unaged chromatograms

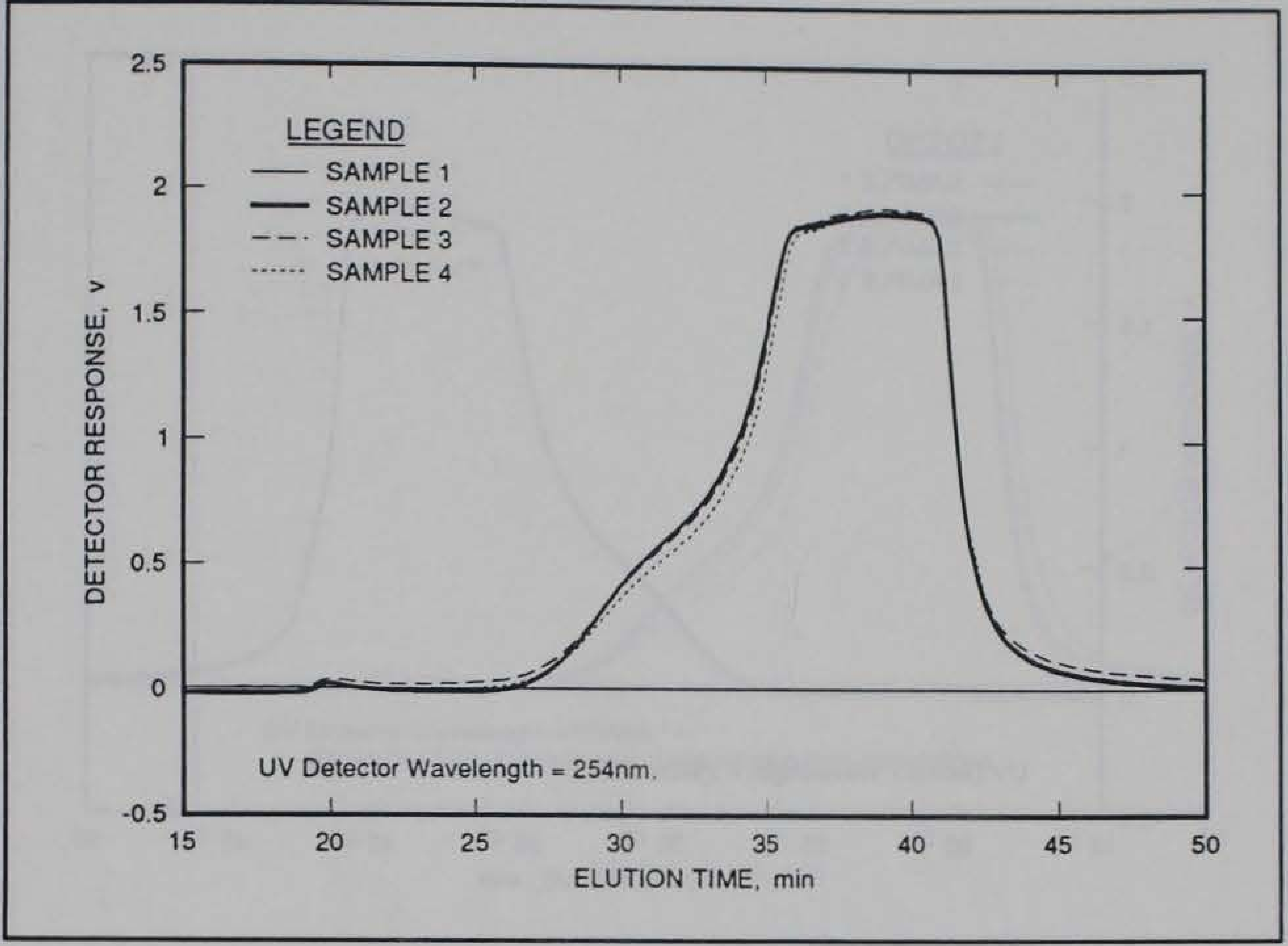


Figure A3. FPL 6523-A oven-aged chromatograms

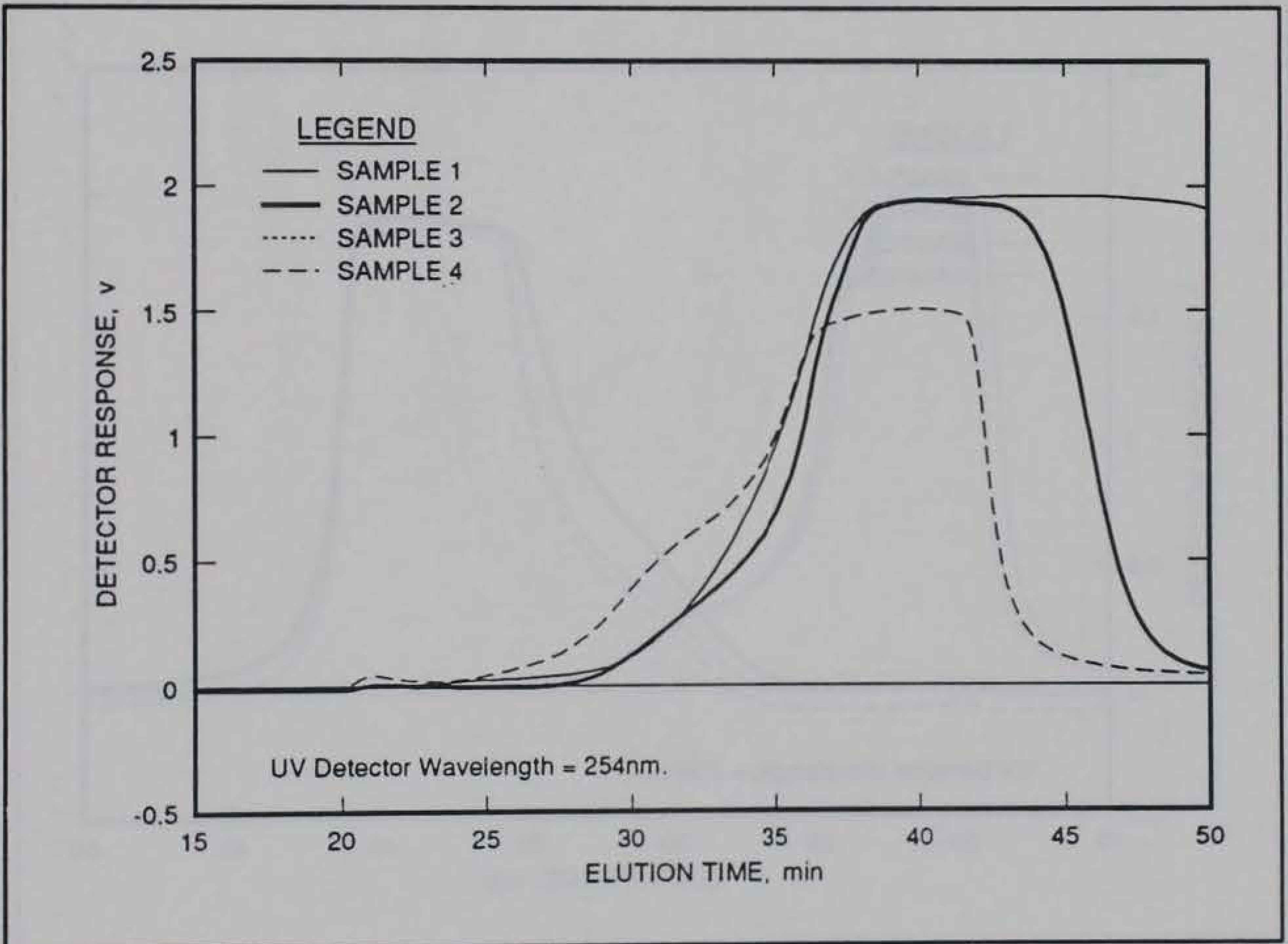


Figure A4. FPL 6523-A weather-o-meter aged chromatograms

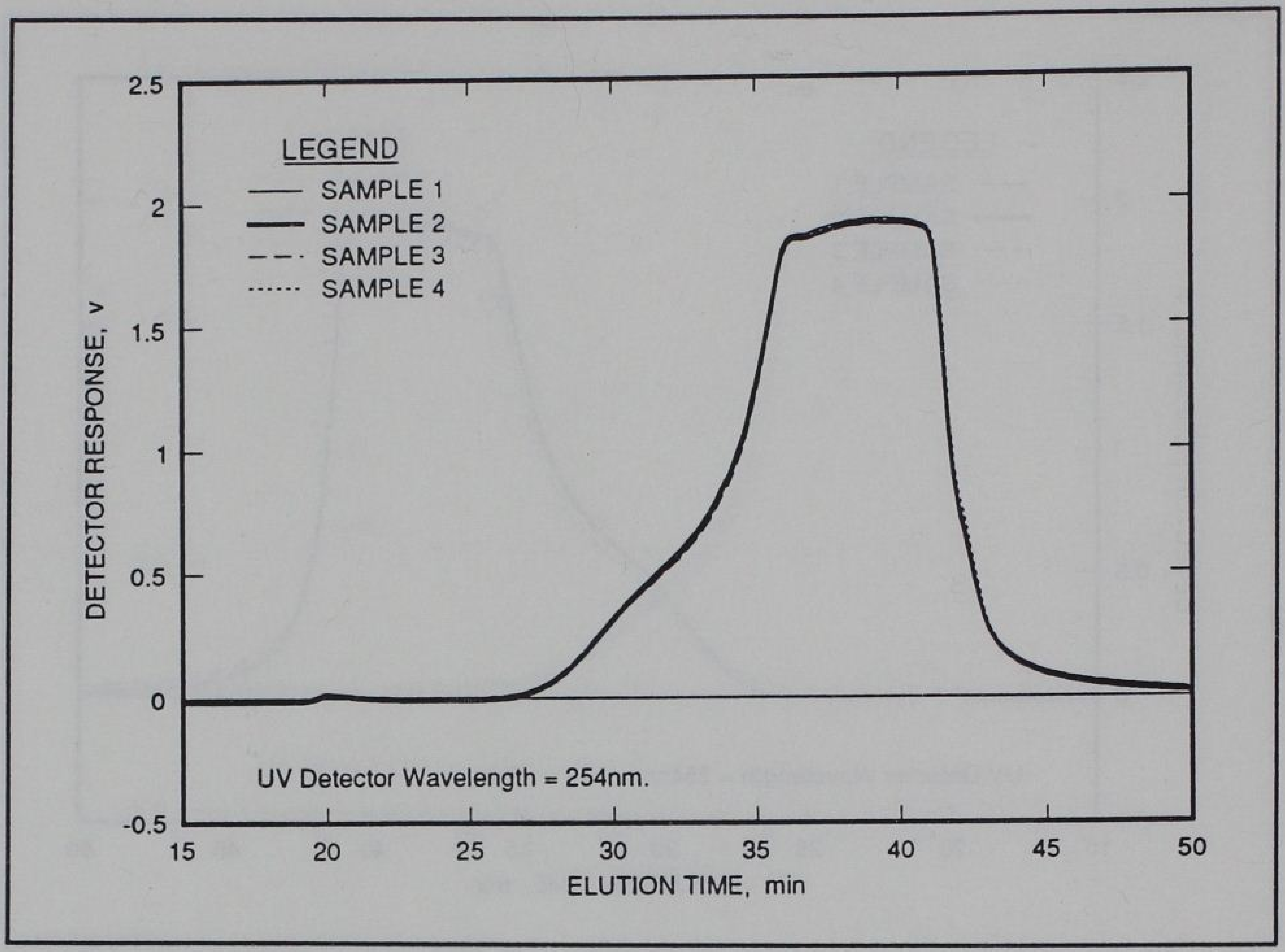


Figure A5. FPL 6523-B unaged chromatograms

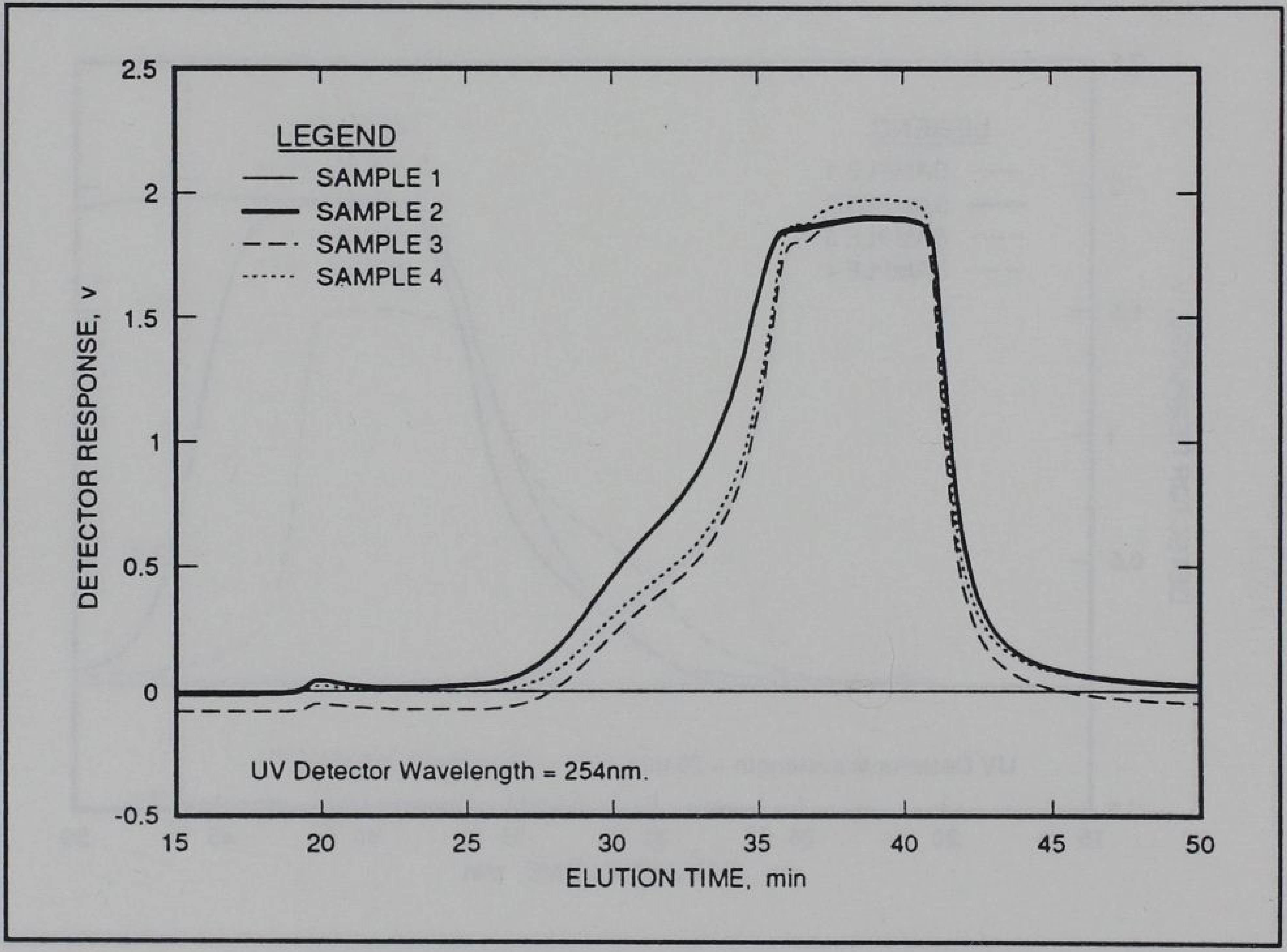


Figure A6. FPL 6523-B oven-aged chromatograms

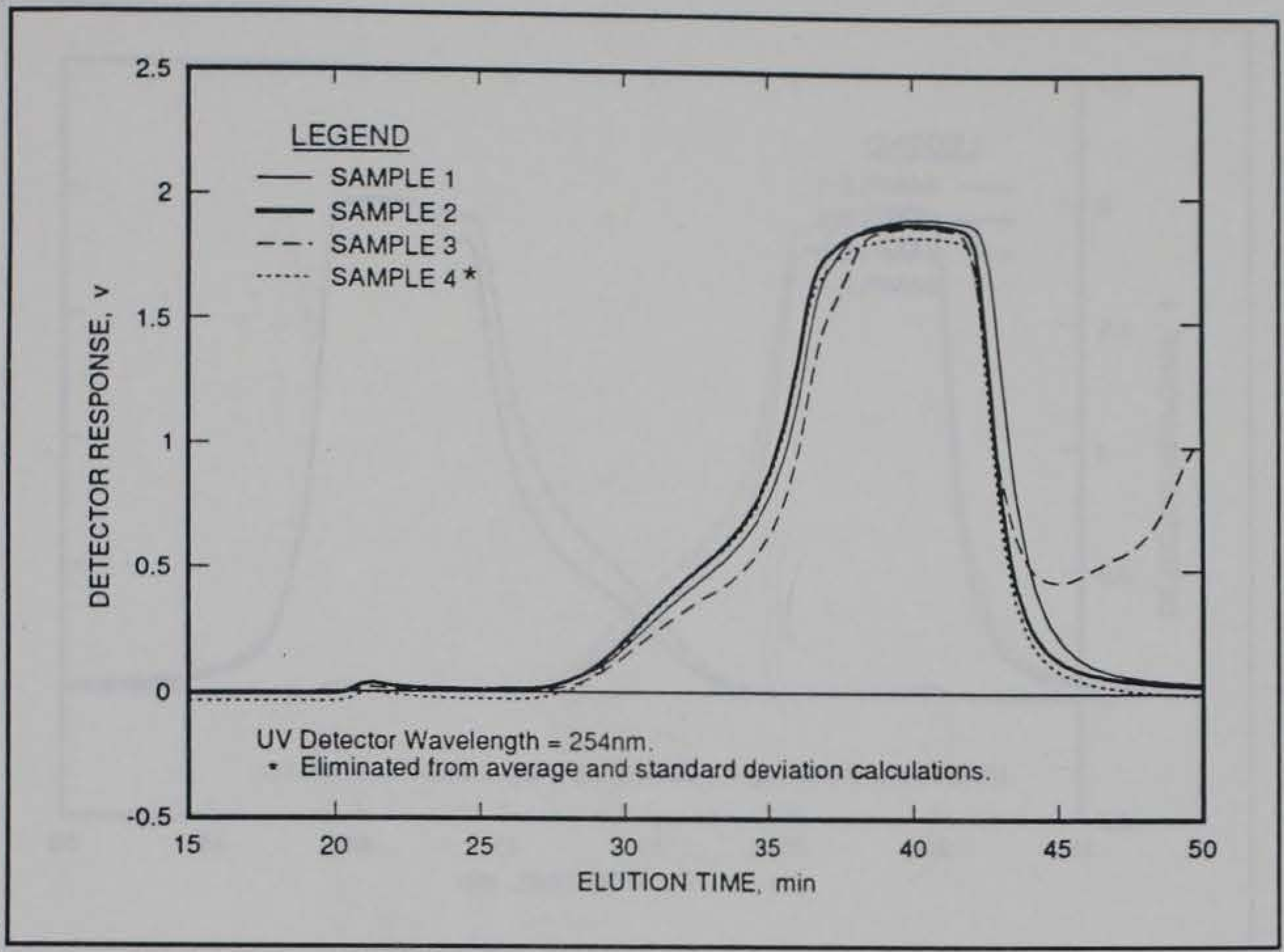


Figure A7. FPL 6523-B weather-o-meter aged chromatograms

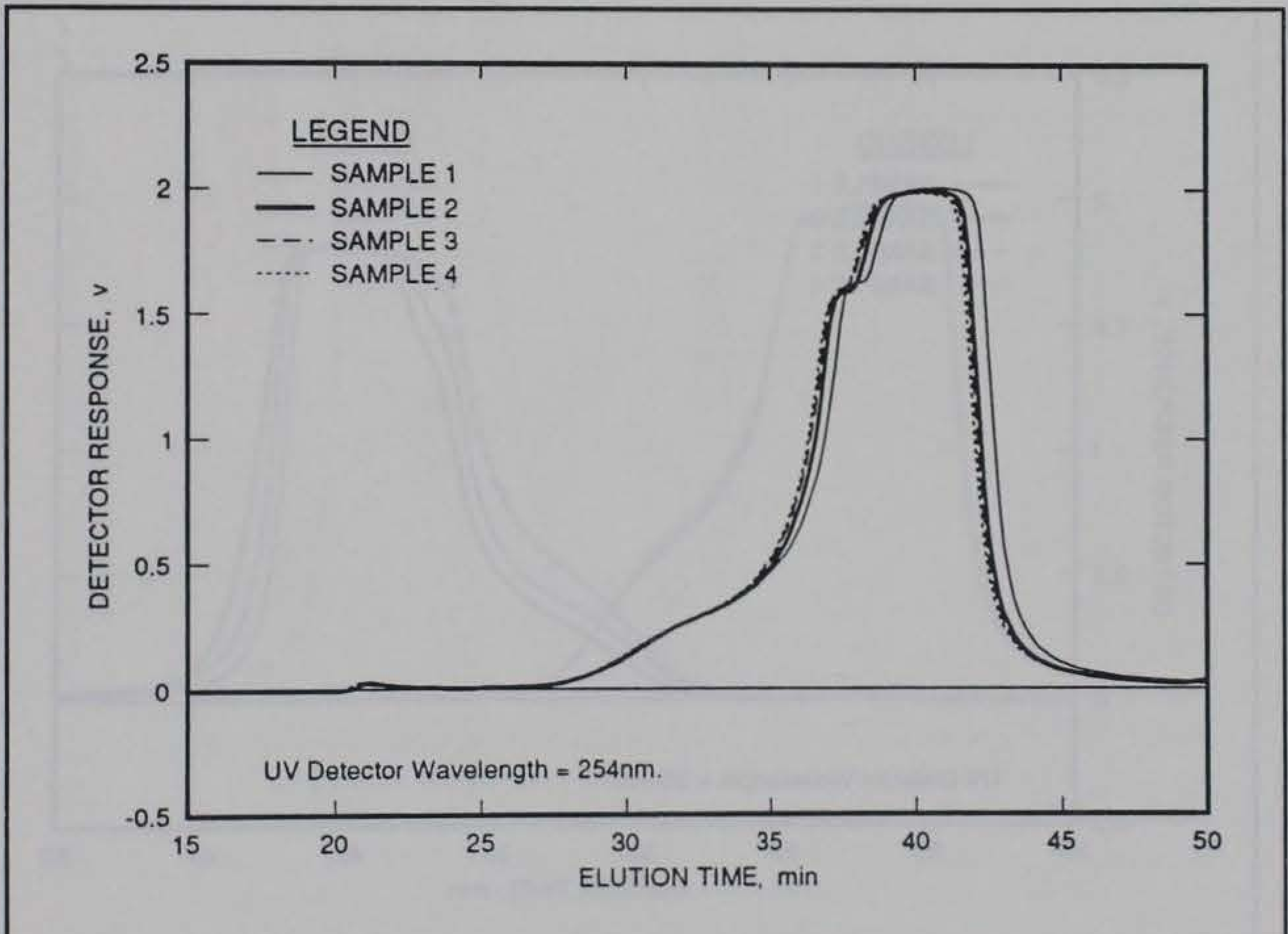


Figure A8. FPL 6523-C unaged chromatograms

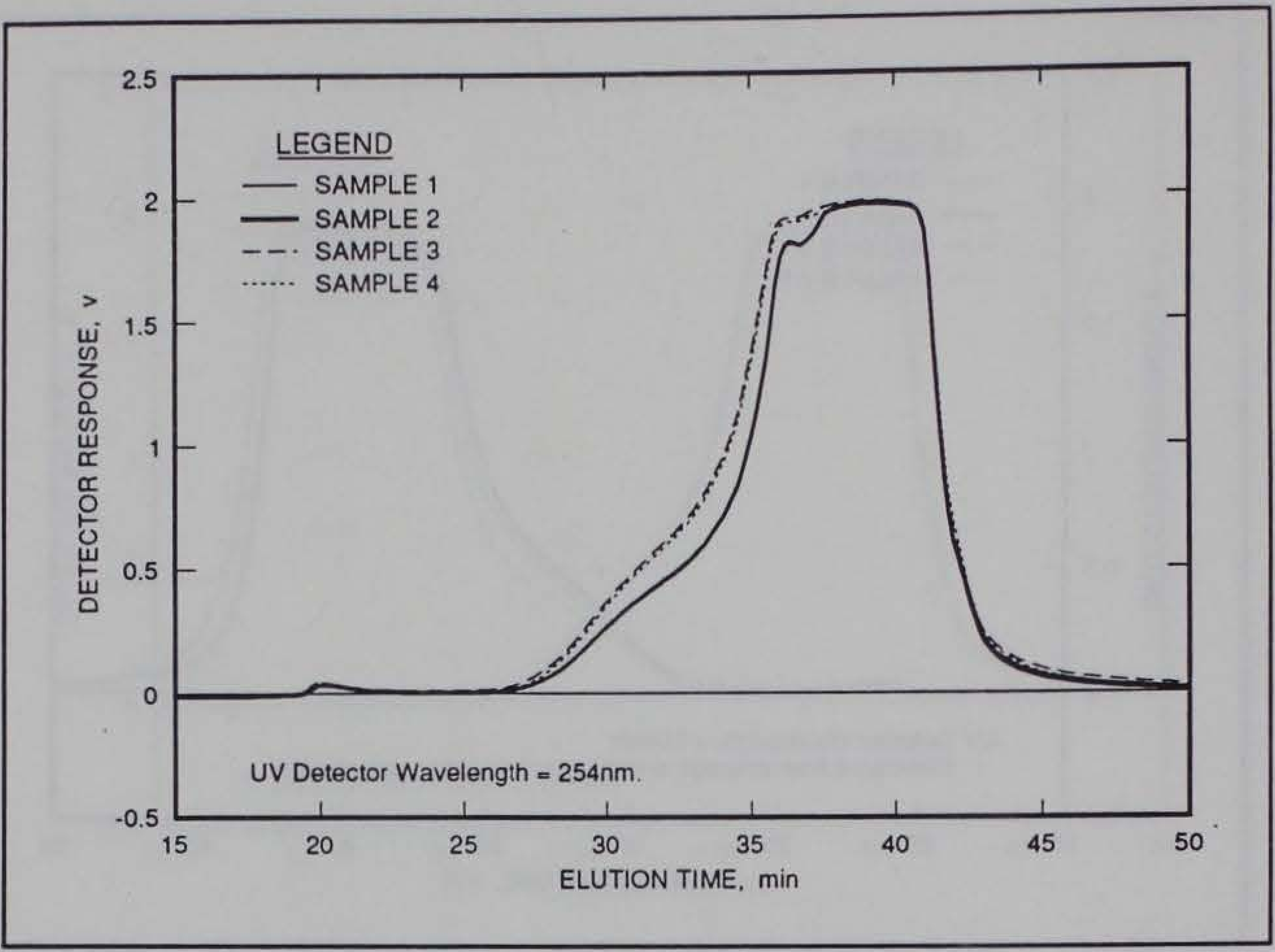


Figure A9. FPL 6523-C oven-aged chromatograms

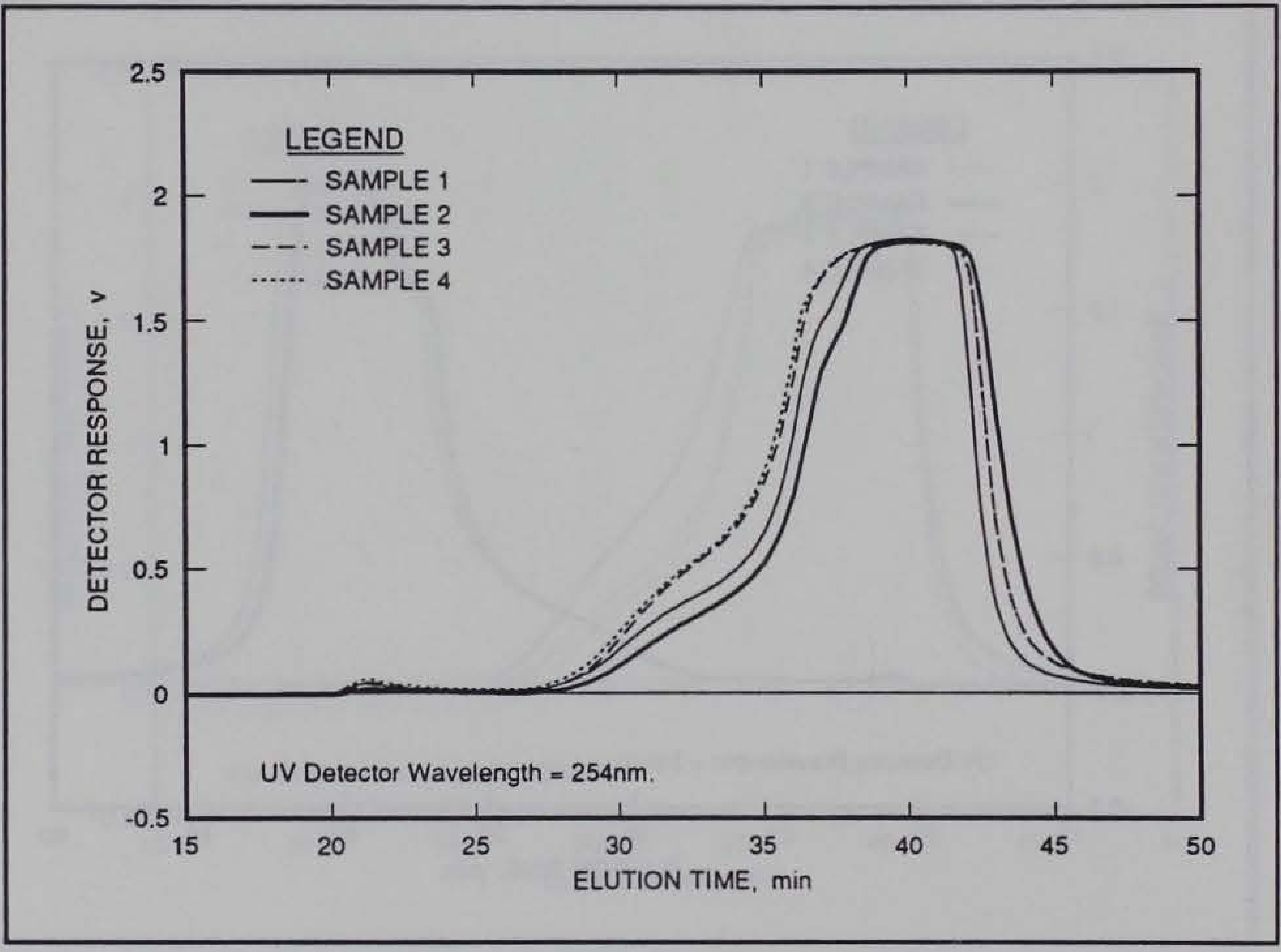


Figure A10. FPL 6523-C weather-o-meter aged chromatograms

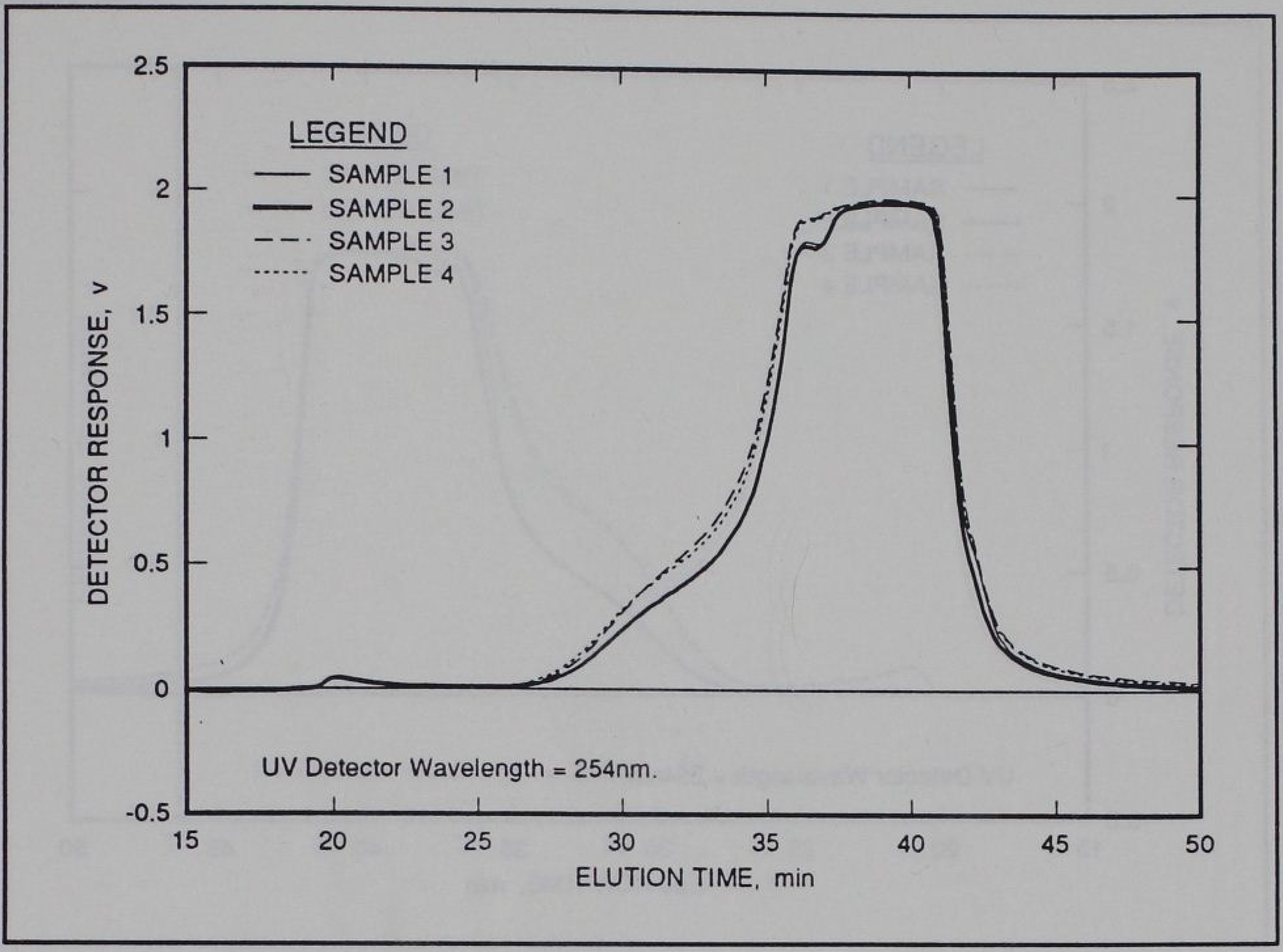


Figure A11. FPL 6523-D unaged chromatograms

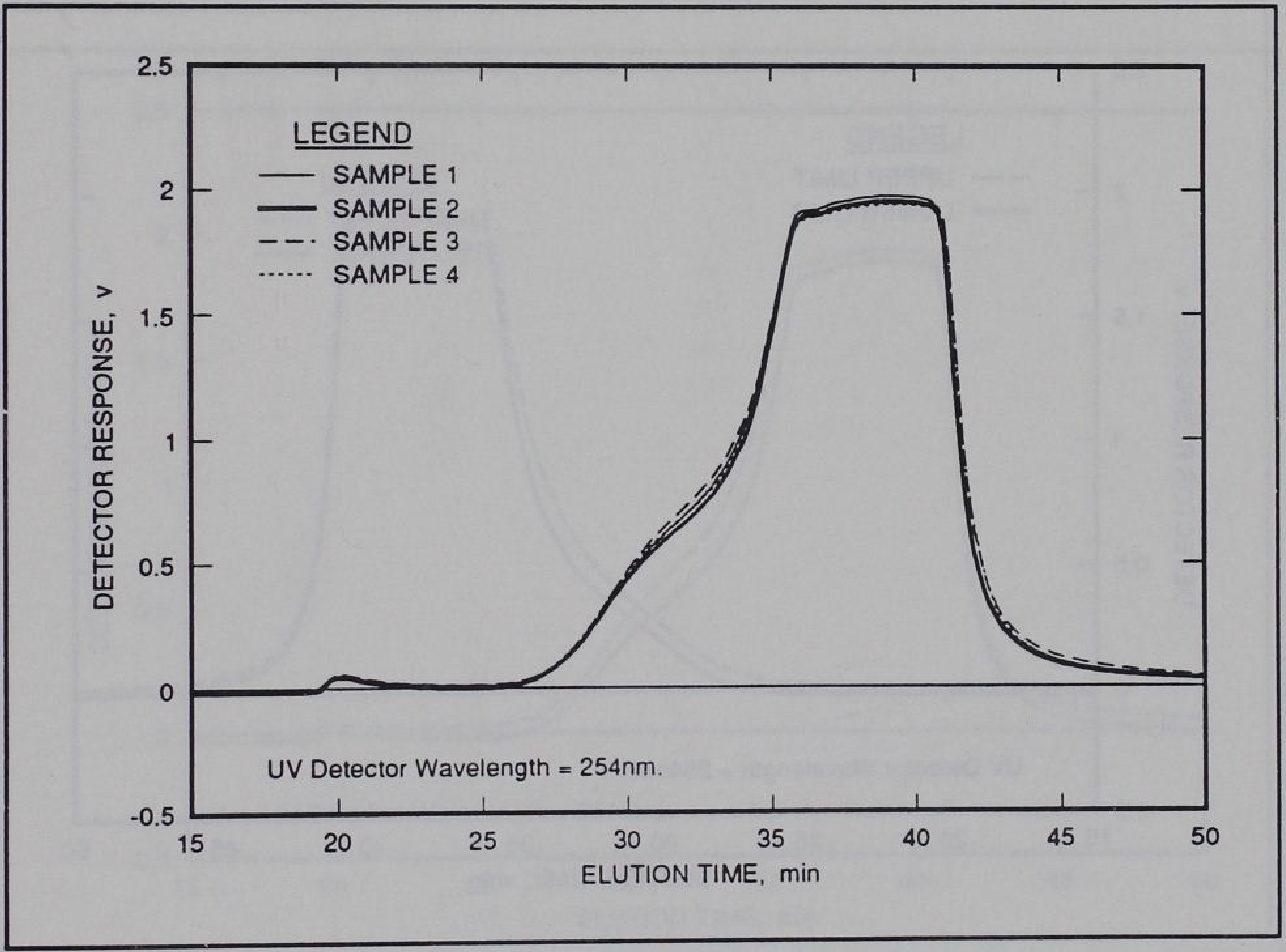


Figure A12. FPL 6523-D oven-aged chromatograms

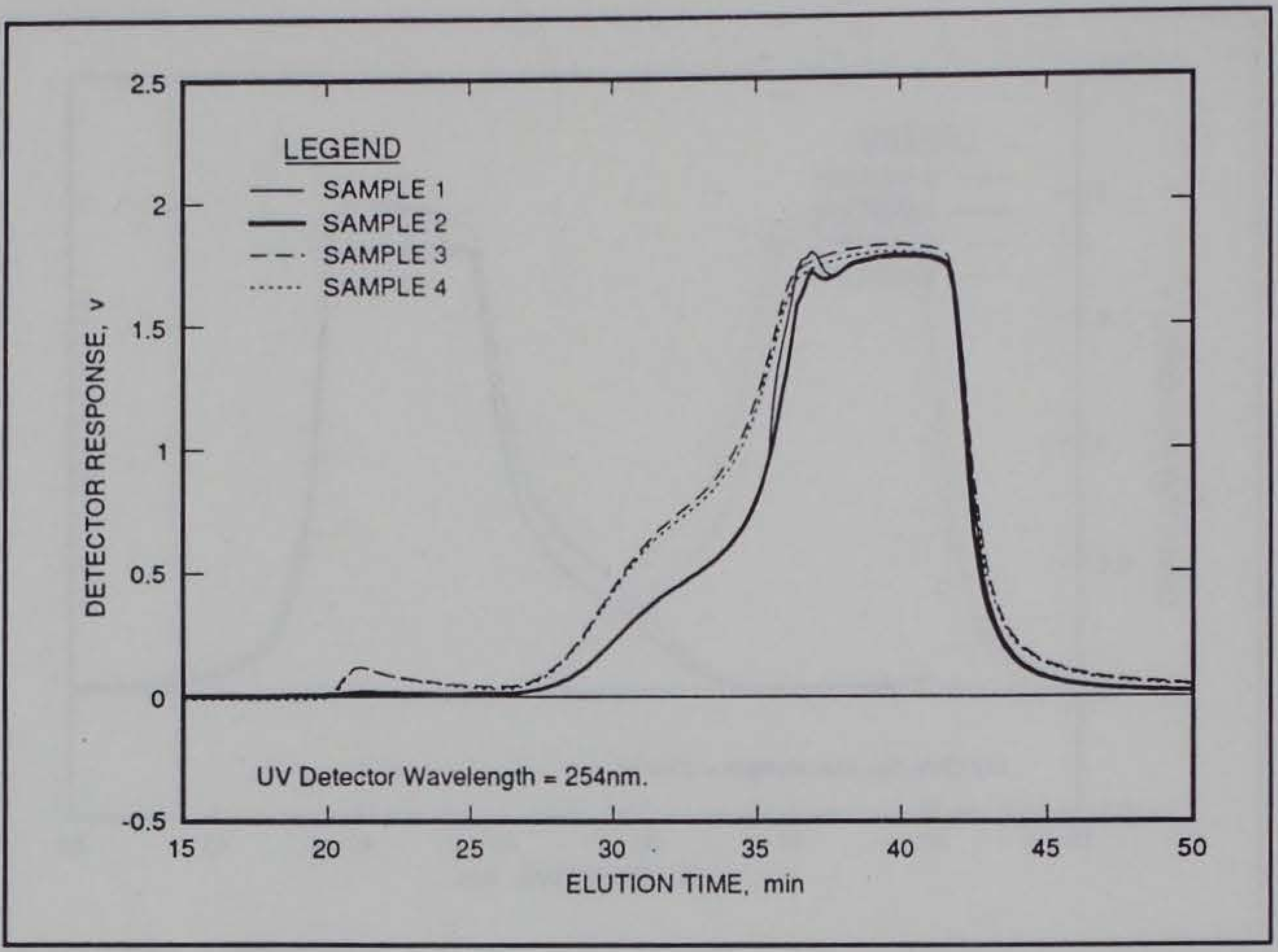


Figure A13. FPL 6523-D weather-o-meter aged chromatograms

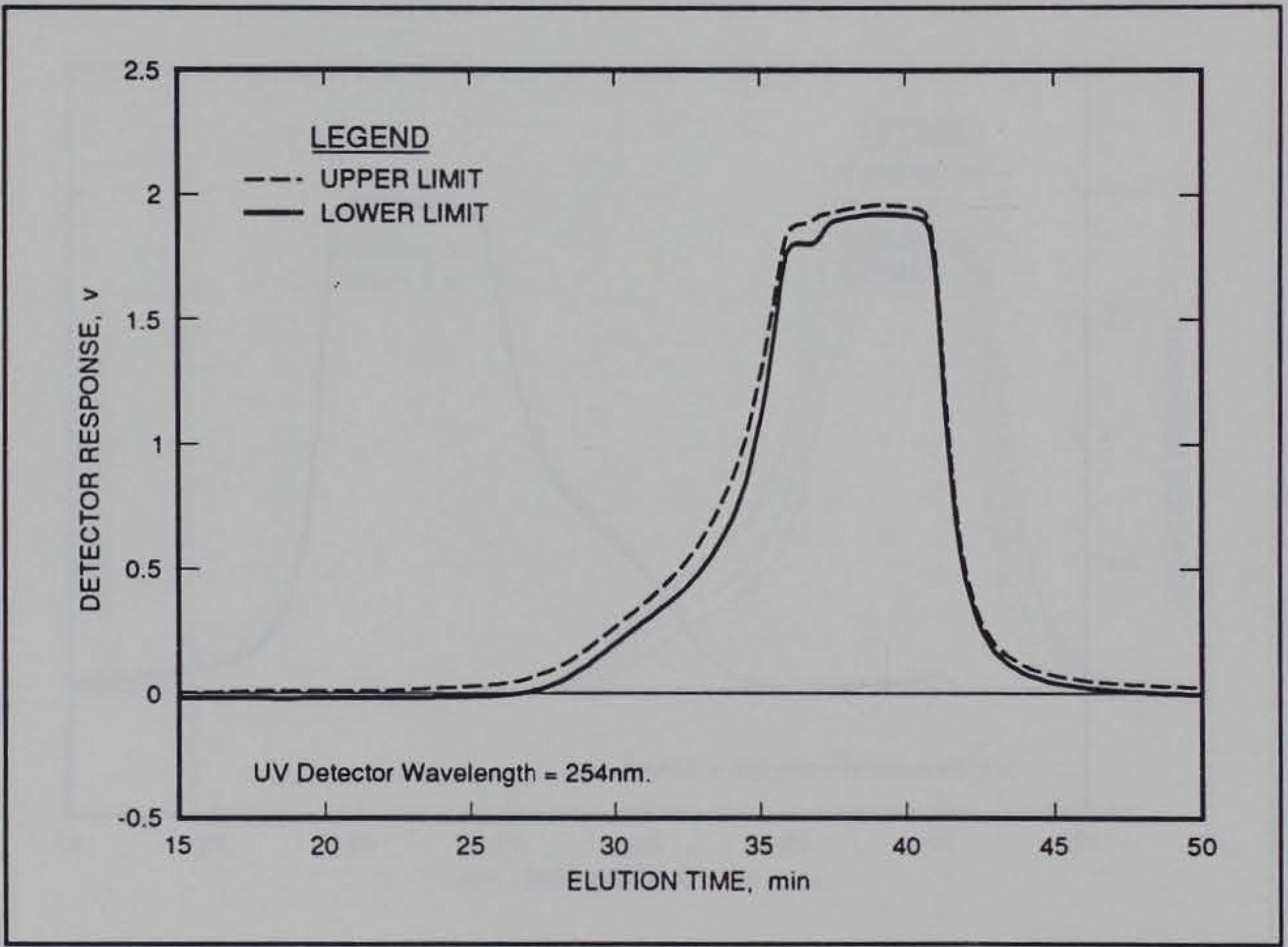


Figure A14. FPL 6523 as-received chromatogram 95 percent confidence region

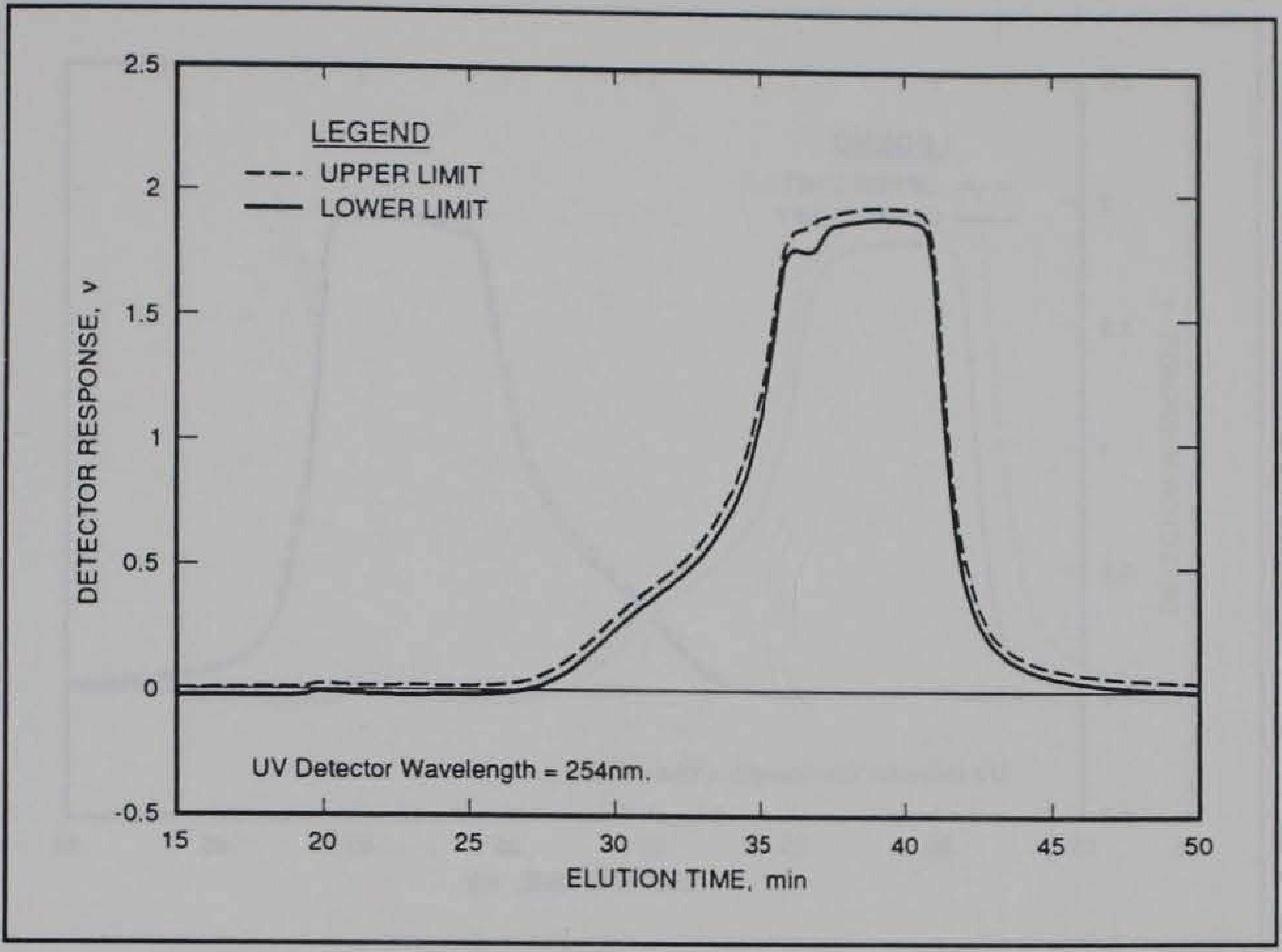


Figure A15. FPL 6523-A unaged chromatogram 95 percent confidence region

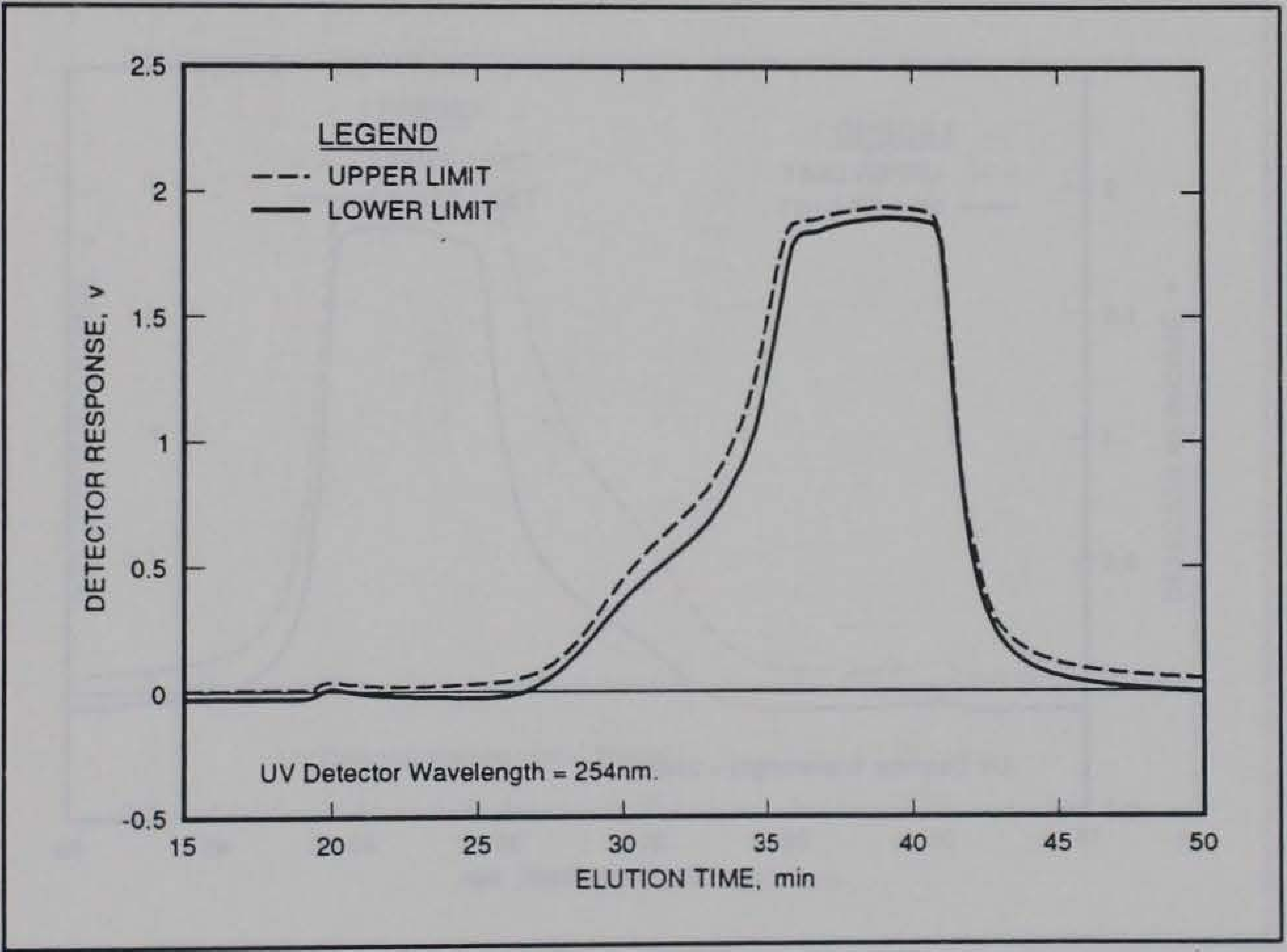


Figure A16. FPL 6523-A oven-aged chromatogram 95 percent confidence region

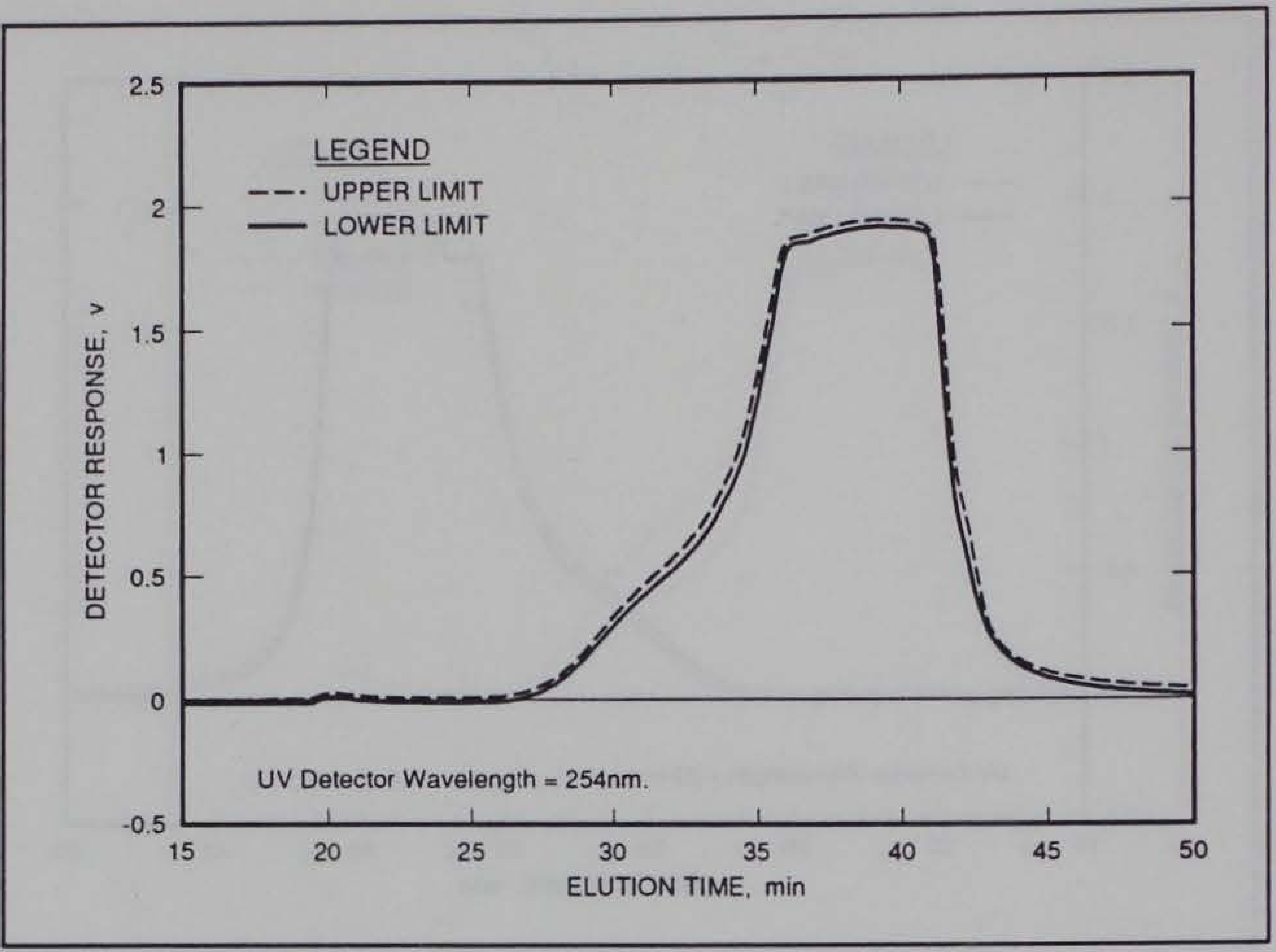


Figure A17. FPL 6523-B unaged chromatogram 95 percent confidence region

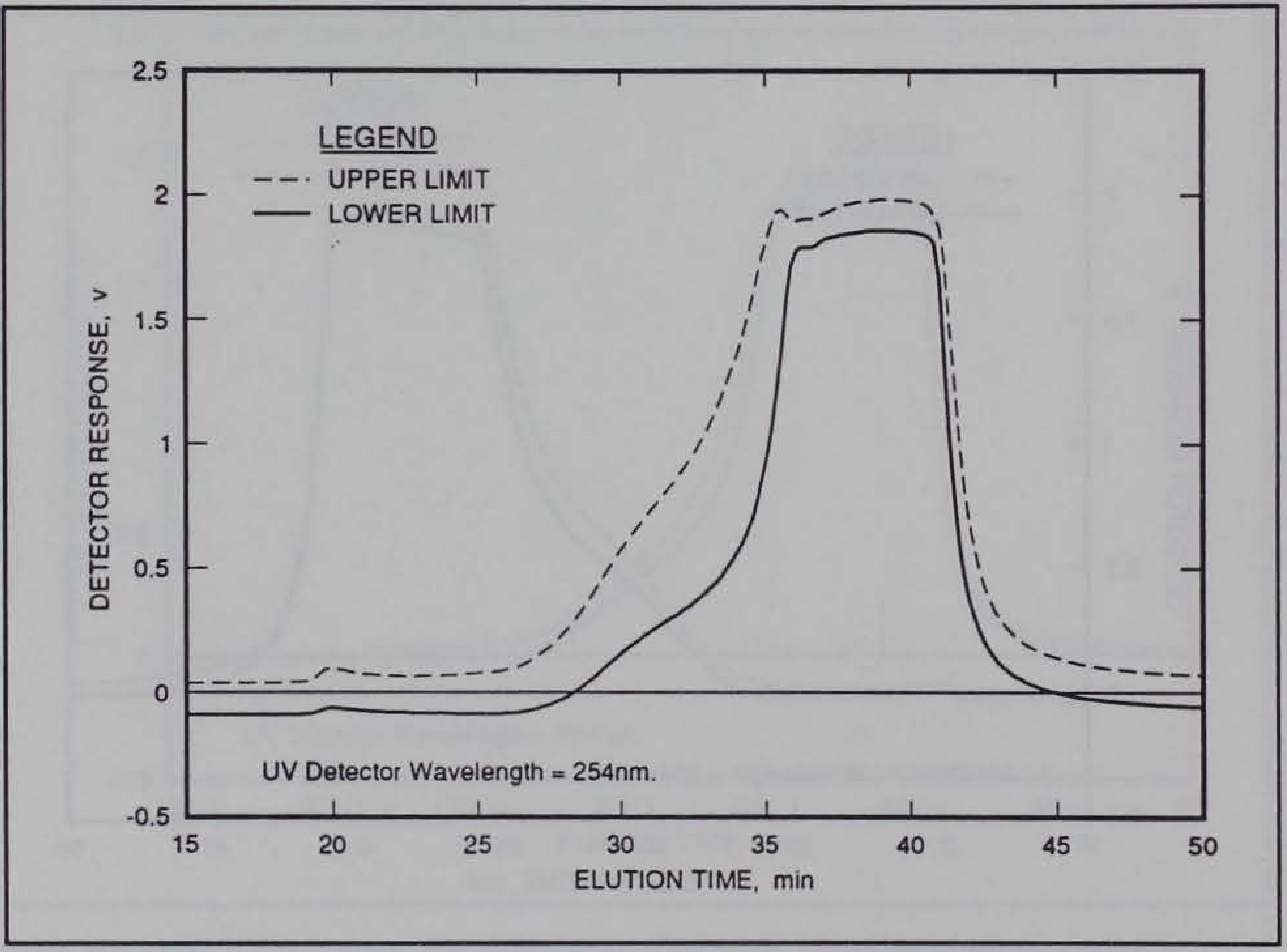


Figure A18. FPL 6523-B oven-aged chromatogram 95 percent confidence region

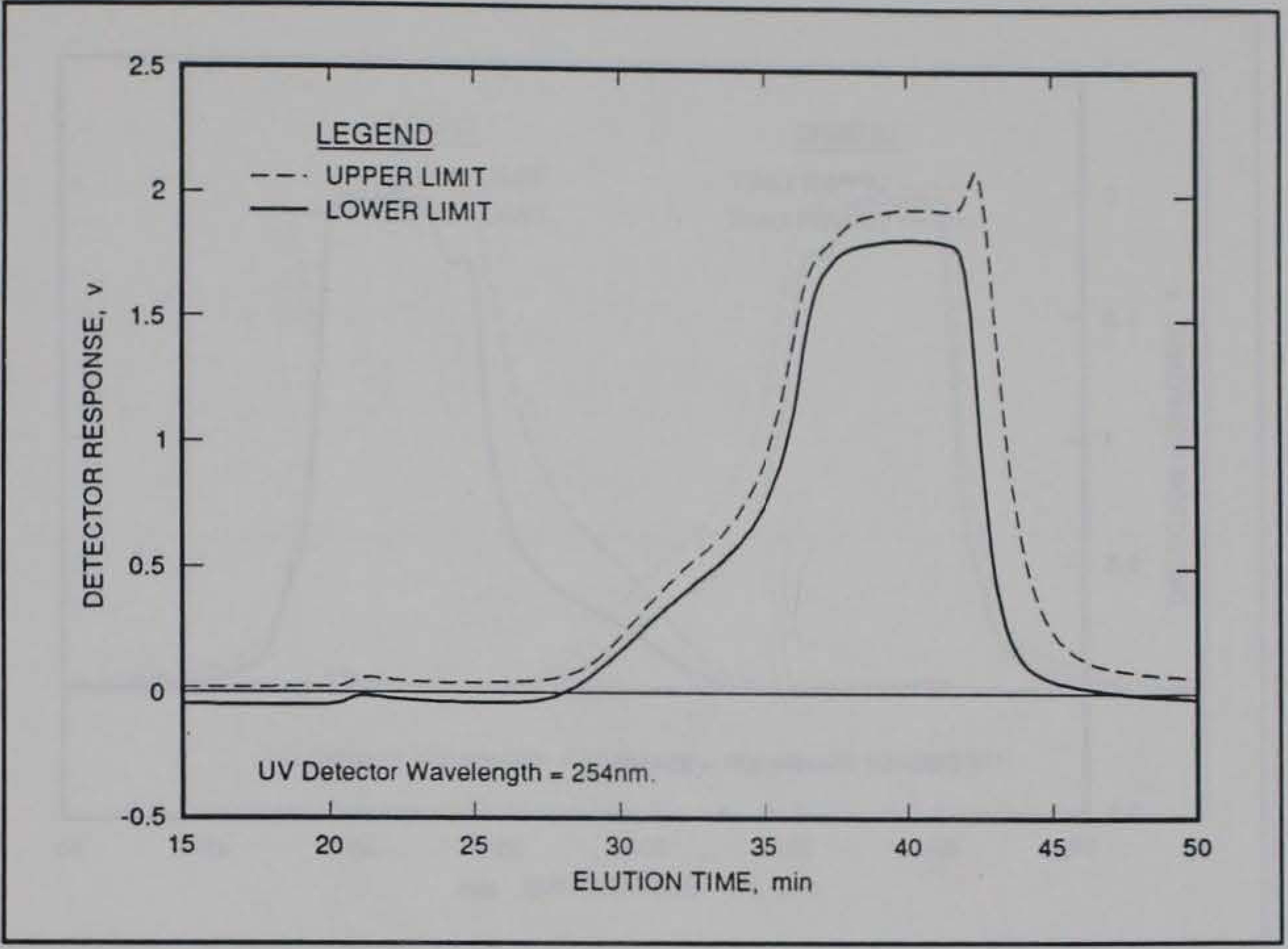


Figure A19. FPL 6523-B weather-o-meter chromatogram 95 percent confidence region

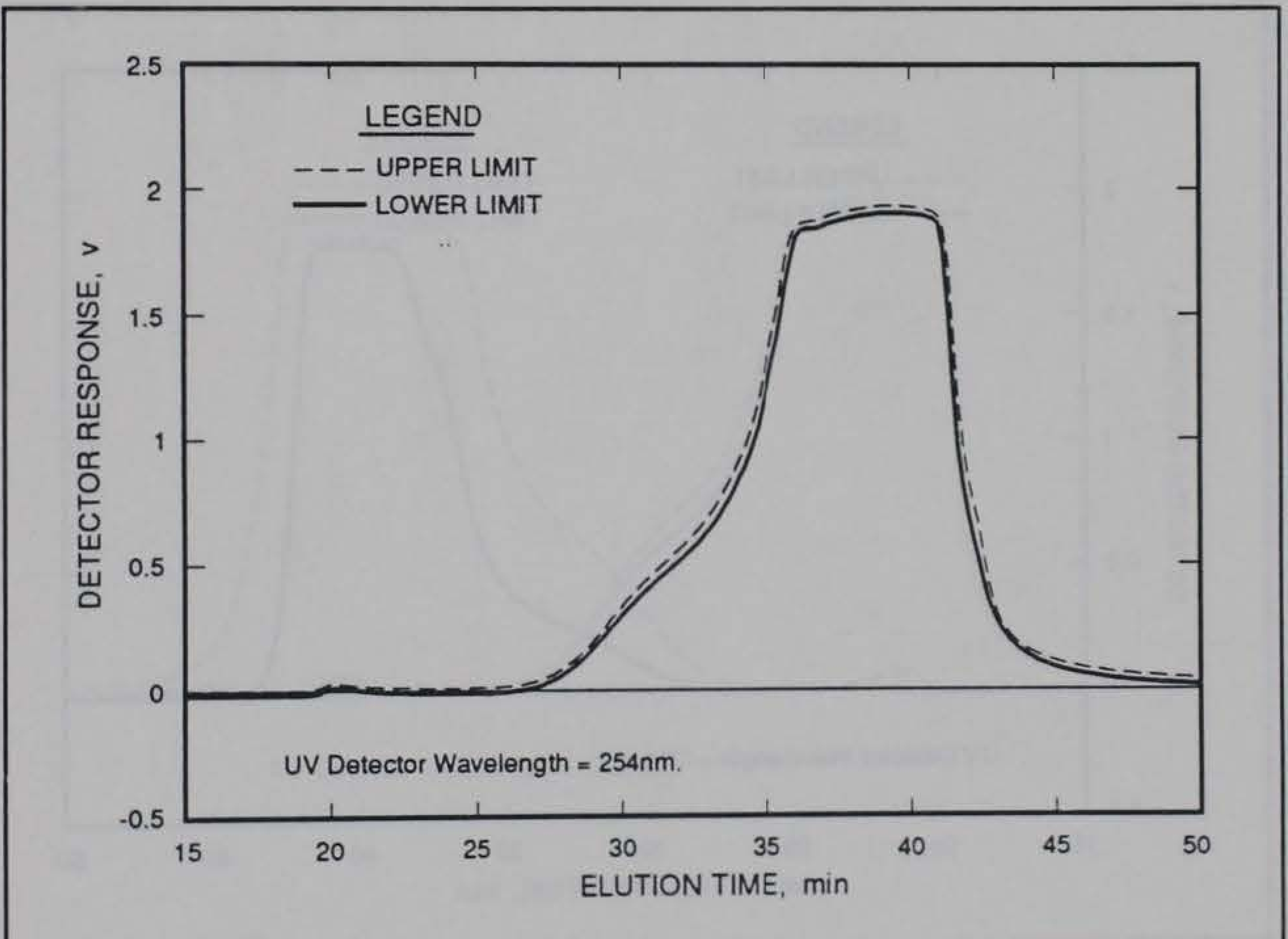


Figure A20. FPL 6523-C unaged chromatogram 95 percent confidence region

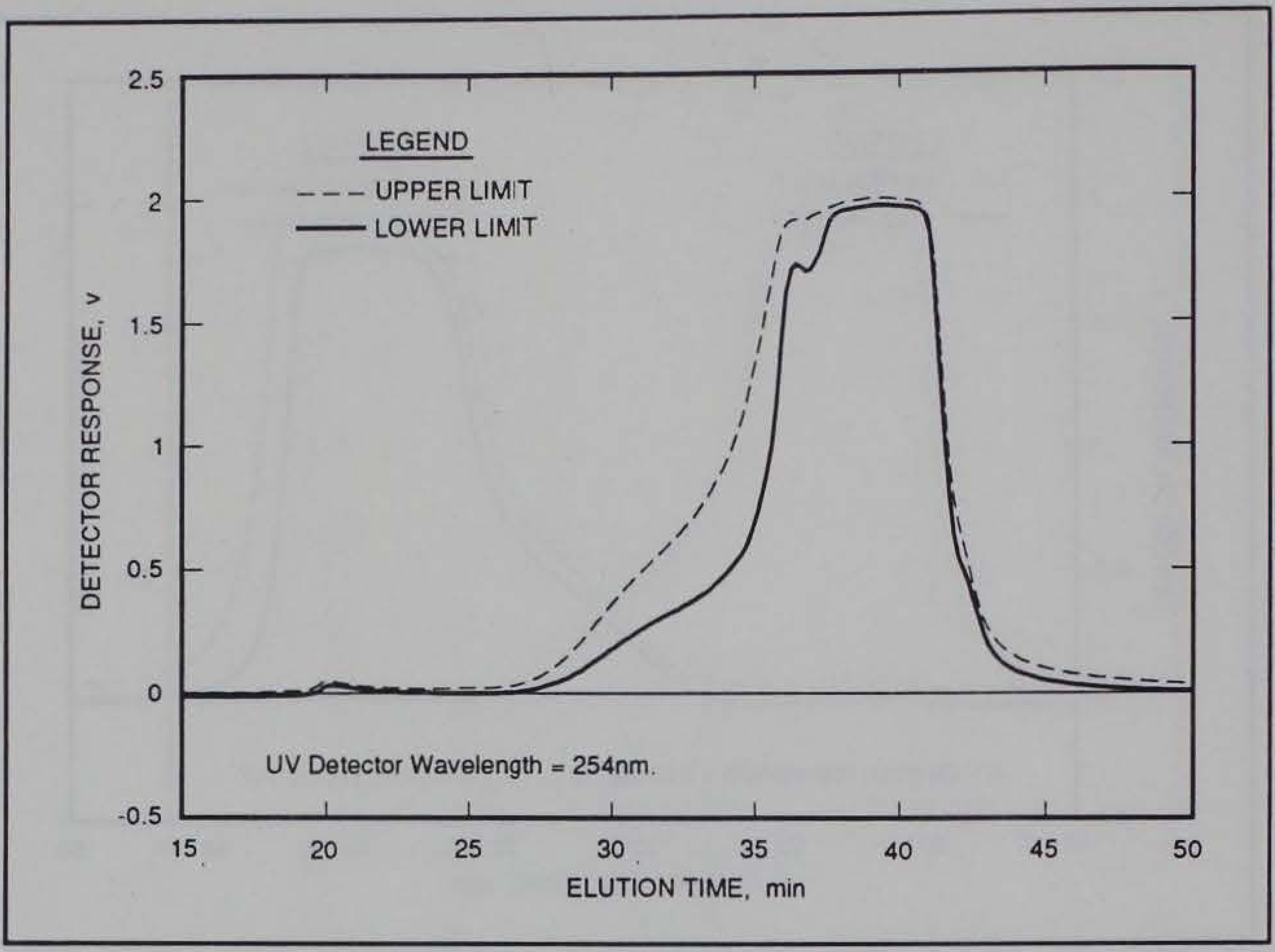


Figure A21. FPL 6523-C oven-aged chromatogram 95 percent confidence region

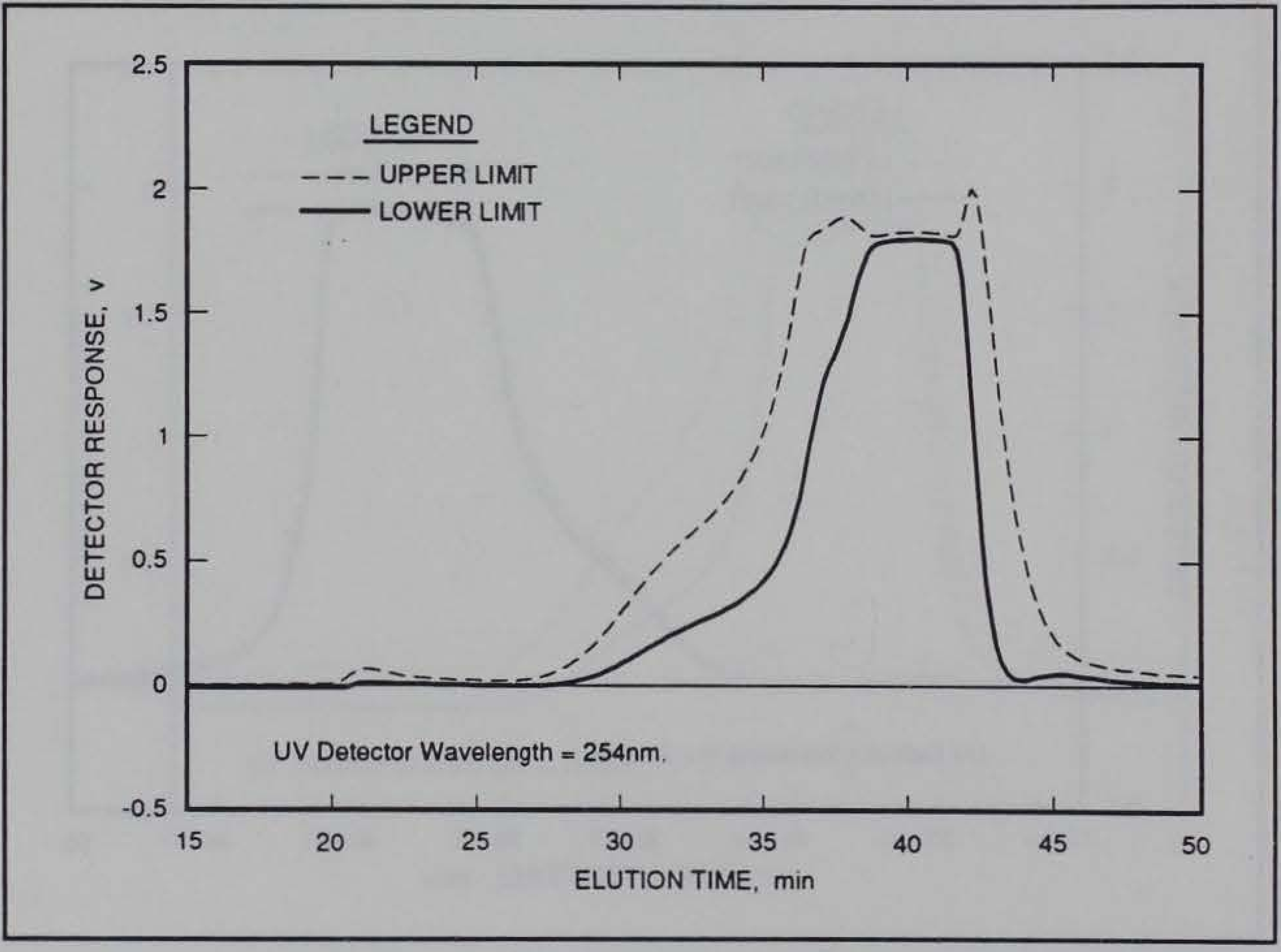


Figure A22. FPL 6523-C weather-o-meter chromatogram 95 percent confidence region

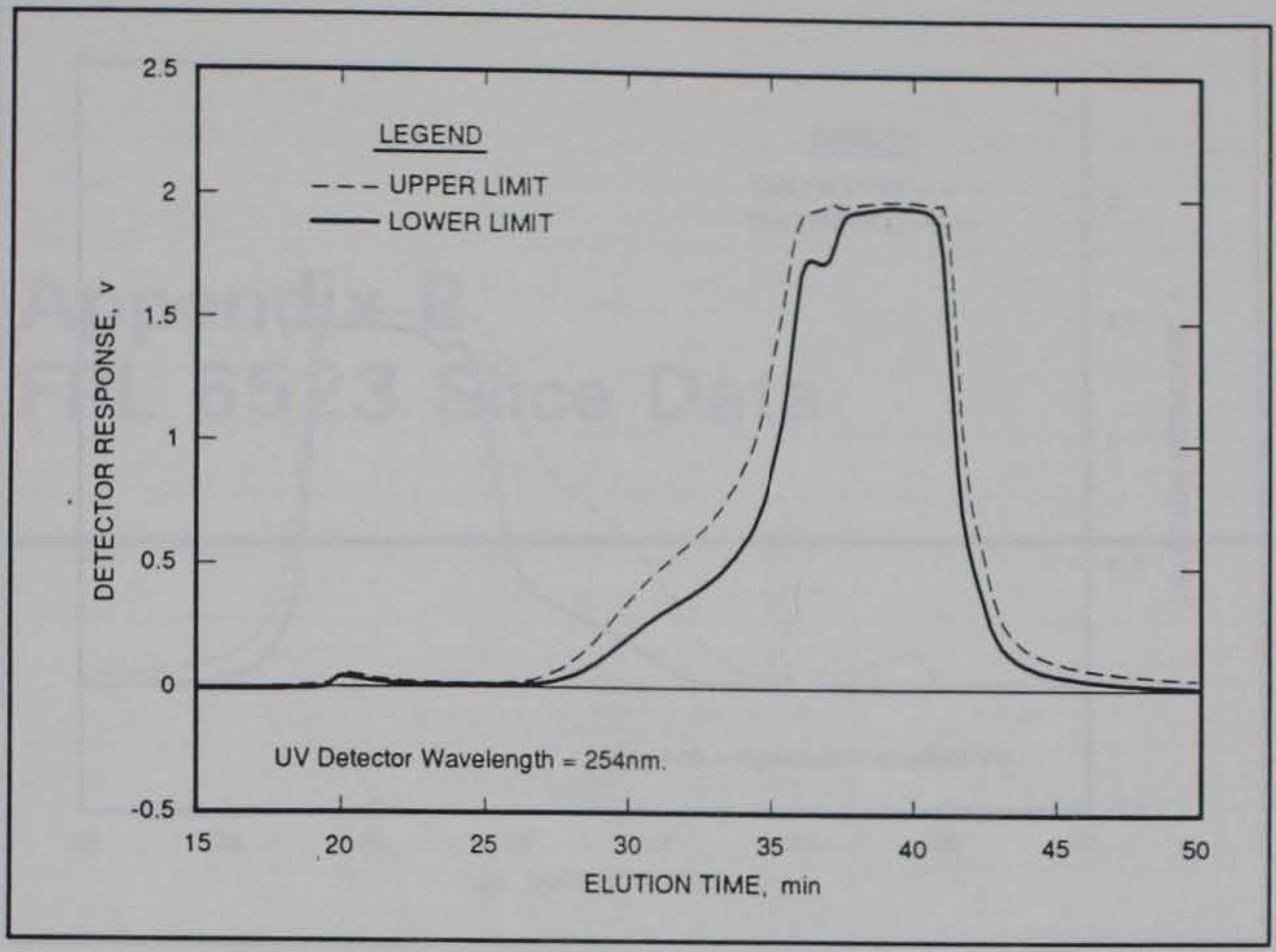


Figure A23. FPL 6523-D unaged chromatogram 95 percent confidence region

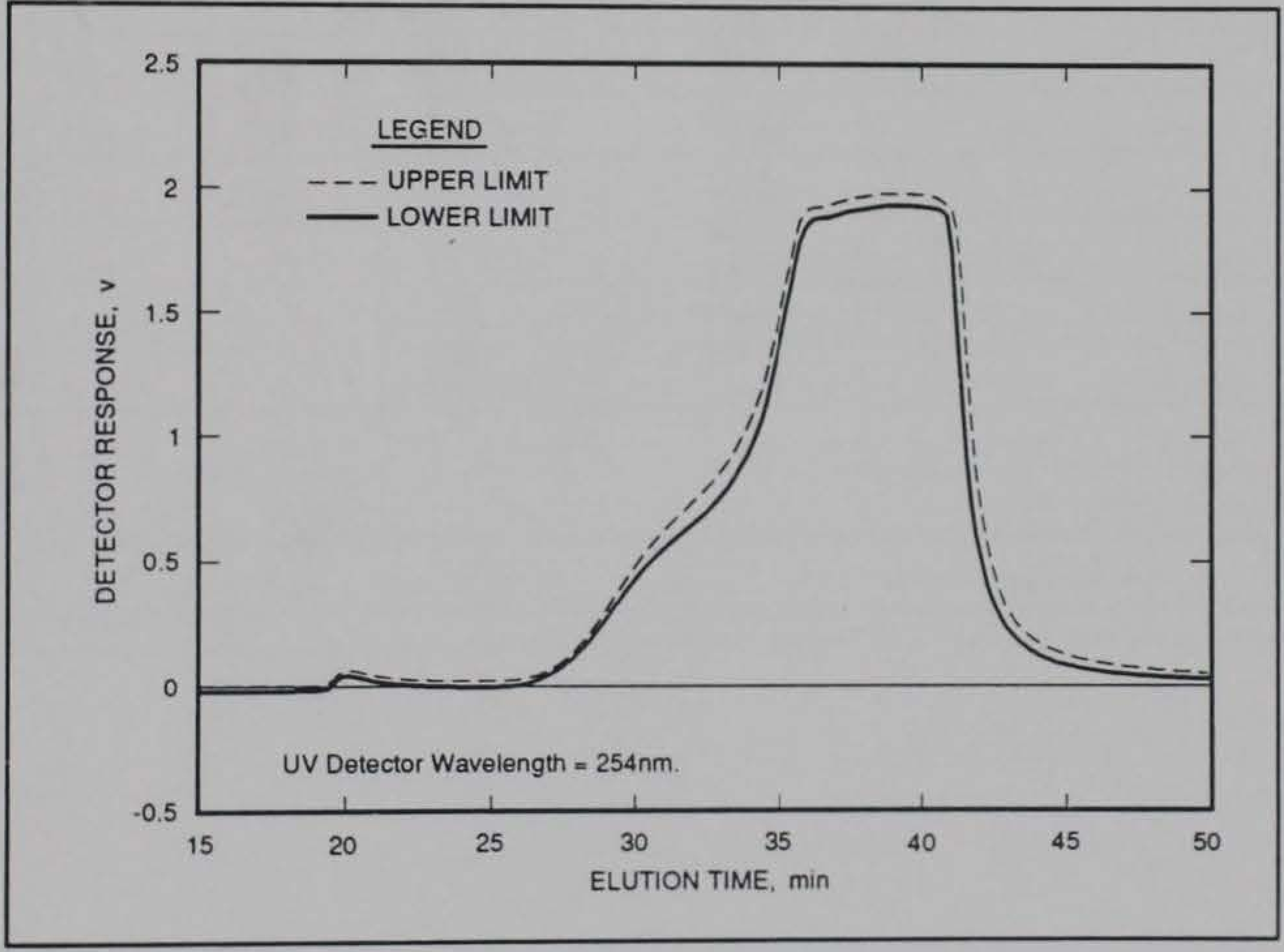


Figure A24. FPL 6523-D oven-aged chromatogram 95 percent confidence region

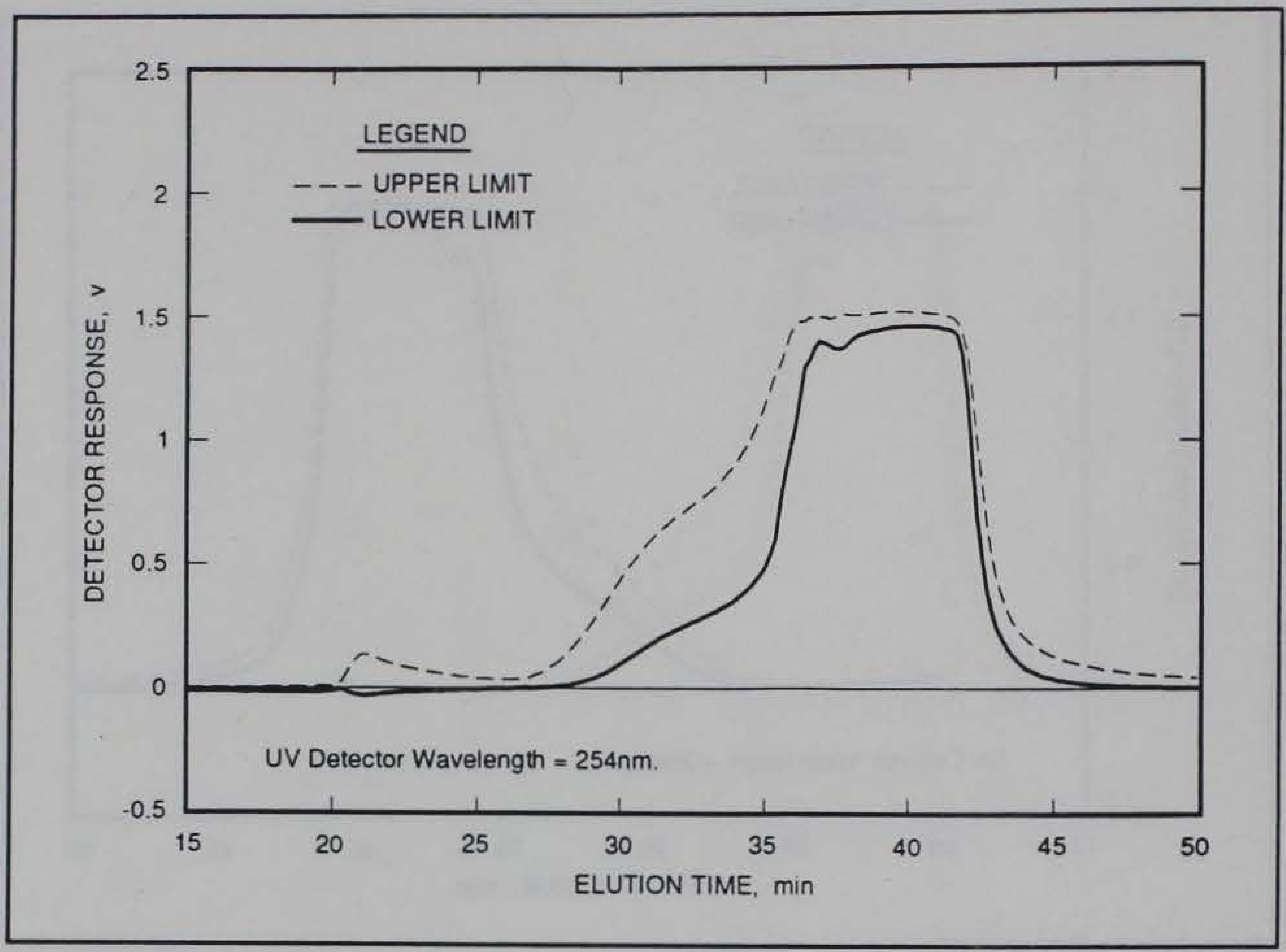


Figure A25. FPL 6523-D weather-o-meter chromatogram 95 percent confidence region

# Appendix B
# FPL 6523 Slice Data

## Table B1
## Slice Data For FPL 6523 As-Received Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 23,431 | 33,136 | 141.42 |
| Slice 2 | 68,555 | 96,951 | 141.42 |
| Slice 3 | 1,477,043 | 1,175,332 | 79.57 |
| Slice 4 | 17,754,005 | 2,018,092 | 11.37 |
| Slice 5 | 58,365,447 | 2,761,684 | 4.73 |
| Slice 6 | 145,398,308 | 5,236,541 | 3.60 |
| Slice 7 | 303,534,682 | 1,821,534 | 0.60 |
| Slice 8 | 310,063,777 | 909,242 | 0.29 |
| Slice 9 | 62,765,901 | 1,688,235 | 2.69 |
| Slice 10 | 3,227,134 | 518,896 | 16.08 |
| Total | 902,678,282 | 14,890,401 | 1.65 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.00 | 0.00 | 141.42 |
| Slice 2 | 0.01 | 0.01 | 141.42 |
| Slice 3 | 0.16 | 0.13 | 78.50 |
| Slice 4 | 1.96 | 0.19 | 9.75 |
| Slice 5 | 6.46 | 0.20 | 3.06 |
| Slice 6 | 16.10 | 0.32 | 2.00 |
| Slice 7 | 33.63 | 0.35 | 1.04 |
| Slice 8 | 34.36 | 0.46 | 1.35 |
| Slice 9 | 6.95 | 0.12 | 1.80 |
| Slice 10 | 0.36 | 0.05 | 14.70 |

[1] Average of three samples.

## Table B2
## Slice Data For FPL 6523-A Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 1,295,066 | 315,586 | 24.37 |
| Slice 2 | 804,413 | 593,259 | 73.75 |
| Slice 3 | 1,408,527 | 293,407 | 20.83 |
| Slice 4 | 20,500,091 | 708,243 | 3.45 |
| Slice 5 | 65,837,478 | 934,572 | 1.42 |
| Slice 6 | 142,382,080 | 2,305,629 | 1.62 |
| Slice 7 | 300,715,692 | 1,834,800 | 0.61 |
| Slice 8 | 311,755,489 | 1,114,353 | 0.36 |
| Slice 9 | 71,571,587 | 2,591,249 | 3.62 |
| Slice 10 | 9,541,202 | 78,191 | 0.82 |
| Total | 925,811,626 | 7,658,984 | 0.83 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.14 | 0.03 | 24.62 |
| Slice 2 | 0.09 | 0.06 | 73.53 |
| Slice 3 | 0.15 | 0.03 | 20.93 |
| Slice 4 | 2.21 | 0.06 | 2.86 |
| Slice 5 | 7.11 | 0.07 | 1.03 |
| Slice 6 | 15.38 | 0.12 | 0.80 |
| Slice 7 | 32.48 | 0.08 | 0.23 |
| Slice 8 | 33.68 | 0.17 | 0.50 |
| Slice 9 | 7.73 | 0.24 | 3.06 |
| Slice 10 | 1.03 | 0.02 | 1.61 |

[1] Average of three samples. Sample 3 was omitted from the calculations because it had a negative area.

## Table B3
## Slice Data For FPL 6523-A Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,482,723 | 237,806 | 9.58 |
| Slice 2 | 810,513 | 212,356 | 26.20 |
| Slice 3 | 2,847,679 | 907,035 | 31.85 |
| Slice 4 | 34,148,382 | 1,614,566 | 4.73 |
| Slice 5 | 92,352,917 | 4,918,894 | 5.33 |
| Slice 6 | 178,260,494 | 7,941,619 | 4.46 |
| Slice 7 | 306,597,980 | 2,016,282 | 0.66 |
| Slice 8 | 311,317,038 | 1,148,715 | 0.37 |
| Slice 9 | 82,086,771 | 1,669,165 | 2.03 |
| Slice 10 | 11,202,924 | 275,431 | 2.46 |
| Total | 1,022,107,427 | 16,164,108 | 1.58 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.24 | 0.02 | 8.10 |
| Slice 2 | 0.08 | 0.02 | 27.15 |
| Slice 3 | 0.28 | 0.09 | 33.00 |
| Slice 4 | 3.34 | 0.11 | 3.24 |
| Slice 5 | 9.03 | 0.34 | 3.77 |
| Slice 6 | 17.43 | 0.51 | 2.90 |
| Slice 7 | 30.00 | 0.28 | 0.94 |
| Slice 8 | 30.46 | 0.38 | 1.25 |
| Slice 9 | 8.03 | 0.23 | 2.91 |
| Slice 10 | 1.10 | 0.04 | 3.86 |

[1] Average of four samples.

## Table B4
## Slice Data For FPL 6523-B Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 1,549,660 | 137,274 | 8.86 |
| Slice 2 | 111,911 | 95,929 | 85.72 |
| Slice 3 | 543,969 | 146,741 | 26.98 |
| Slice 4 | 23,080,514 | 801,424 | 3.47 |
| Slice 5 | 76,570,383 | 1,842,575 | 2.41 |
| Slice 6 | 159,387,970 | 3,399,717 | 2.13 |
| Slice 7 | 302,671,139 | 716,582 | 0.24 |
| Slice 8 | 312,304,777 | 227,420 | 0.07 |
| Slice 9 | 100,200,706 | 4,227,488 | 4.22 |
| Slice 10 | 11,781,622 | 129,782 | 1.10 |
| Total | 988,202,650 | 7,419,327 | 0.75 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.16 | 0.01 | 8.23 |
| Slice 2 | 0.01 | 0.01 | 85.46 |
| Slice 3 | 0.05 | 0.01 | 26.51 |
| Slice 4 | 2.34 | 0.07 | 2.79 |
| Slice 5 | 7.75 | 0.14 | 1.81 |
| Slice 6 | 16.13 | 0.27 | 1.68 |
| Slice 7 | 30.63 | 0.20 | 0.65 |
| Slice 8 | 31.61 | 0.23 | 0.74 |
| Slice 9 | 10.14 | 0.40 | 3.94 |
| Slice 10 | 1.19 | 0.01 | 0.79 |

[1] Average of four samples.

## Table B5
## Slice Data For FPL 6523-B Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,482,732 | 237,806 | 9.58 |
| Slice 2 | 810,513 | 212,356 | 26.20 |
| Slice 3 | 2,847,679 | 907,035 | 31.85 |
| Slice 4 | 34,148,382 | 1,614,566 | 4.73 |
| Slice 5 | 92,352,917 | 4,918,894 | 5.33 |
| Slice 6 | 178,260,494 | 7,941,619 | 4.46 |
| Slice 7 | 306,597,980 | 2,016,282 | 0.66 |
| Slice 8 | 311,317,038 | 1,148,715 | 0.37 |
| Slice 9 | 82,086,771 | 1,669,165 | 2.03 |
| Slice 10 | 11,202,924 | 275,431 | 2.46 |
| Total | 1,022,107,427 | 16,164,108 | 1.58 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.35 | 0.10 | 28.82 |
| Slice 2 | 0.18 | 0.12 | 65.07 |
| Slice 3 | 0.39 | 0.24 | 62.02 |
| Slice 4 | 3.16 | 0.65 | 20.66 |
| Slice 5 | 8.67 | 1.18 | 13.66 |
| Slice 6 | 17.46 | 1.75 | 10.00 |
| Slice 7 | 30.33 | 1.93 | 6.37 |
| Slice 8 | 30.85 | 2.26 | 7.33 |
| Slice 9 | 7.56 | 0.22 | 2.96 |
| Slice 10 | 1.06 | 0.02 | 1.48 |

[1] Average of four samples.

**Table B6**
**Slice Data For FPL 6523-B Weather-O-Meter Aged Chromatograms**

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 3,276,796 | 313,855 | 9.58 |
| Slice 2 | 1,033,878 | 220,008 | 21.28 |
| Slice 3 | 3,460,274 | 337,684 | 9.76 |
| Slice 4 | 34,611,801 | 3,777,213 | 10.91 |
| Slice 5 | 85,507,921 | 6,245,364 | 7.30 |
| Slice 6 | 189,378,641 | 9,807,492 | 5.18 |
| Slice 7 | 302,355,871 | 1,099,391 | 0.36 |
| Slice 8 | 299,191,889 | 6,256,670 | 2.09 |
| Slice 9 | 71,499,367 | 20,782,425 | 29.07 |
| Slice 10 | 7,552,593 | 1,076,189 | 14.25 |
| Total | 997,869,030 | 8,384,038 | 0.84 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.33 | 0.03 | 9.90 |
| Slice 2 | 0.10 | 0.02 | 20.62 |
| Slice 3 | 0.35 | 0.03 | 10.00 |
| Slice 4 | 3.47 | 0.41 | 11.69 |
| Slice 5 | 8.57 | 0.69 | 8.10 |
| Slice 6 | 18.99 | 1.12 | 5.90 |
| Slice 7 | 30.30 | 0.25 | 0.83 |
| Slice 8 | 29.98 | 0.38 | 1.25 |
| Slice 9 | 7.15 | 2.02 | 28.21 |
| Slice 10 | 0.76 | 0.10 | 13.43 |

[1] Average of three samples.

## Table B7
## Slice Data For FPL 6523-C Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,281,125 | 187,363 | 8.21 |
| Slice 2 | 822,340 | 182,659 | 22.21 |
| Slice 3 | 2,887,460 | 187,501 | 6.49 |
| Slice 4 | 21,941,551 | 927,713 | 4.23 |
| Slice 5 | 50,711,048 | 755,295 | 1.49 |
| Slice 6 | 119,489,635 | 11,771,363 | 9.85 |
| Slice 7 | 293,898,134 | 7,316,858 | 2.49 |
| Slice 8 | 276,892,600 | 21,223,532 | 7.66 |
| Slice 9 | 26,865,274 | 8,924,808 | 33.22 |
| Slice 10 | 4,113,543 | 953,944 | 23.19 |
| Total | 799,902,709 | 11,831,590 | 1.48 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.29 | 0.02 | 8.61 |
| Slice 2 | 0.10 | 0.02 | 21.38 |
| Slice 3 | 0.36 | 0.02 | 6.79 |
| Slice 4 | 2.75 | 0.15 | 5.50 |
| Slice 5 | 6.34 | 0.15 | 2.37 |
| Slice 6 | 14.96 | 1.63 | 10.88 |
| Slice 7 | 36.76 | 1.38 | 3.75 |
| Slice 8 | 34.59 | 2.19 | 6.34 |
| Slice 9 | 3.34 | 1.05 | 31.49 |
| Slice 10 | 0.51 | 0.11 | 21.71 |

[1] Average of four samples.

## Table B8
## Slice Data For FPL 6523-C Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,033,869 | 256,077 | 6.35 |
| Slice 2 | 1,944,748 | 71,174 | 3.66 |
| Slice 3 | 2,107,364 | 202,164 | 9.59 |
| Slice 4 | 23,999,331 | 3,751,821 | 15.63 |
| Slice 5 | 74,896,381 | 10,030,163 | 13.39 |
| Slice 6 | 151,058,602 | 20,186,985 | 13.36 |
| Slice 7 | 305,764,297 | 7,591,842 | 2.48 |
| Slice 8 | 320,588,555 | 353,032 | 0.11 |
| Slice 9 | 84,373,035 | 3,386,303 | 4.01 |
| Slice 10 | 9,747,176 | 1,198,900 | 12.30 |
| Total | 978,513,358 | 45,126,576 | 4.61 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.41 | 0.02 | 3.74 |
| Slice 2 | 0.20 | 0.02 | 7.74 |
| Slice 3 | 0.22 | 0.02 | 7.18 |
| Slice 4 | 2.44 | 0.27 | 11.12 |
| Slice 5 | 7.62 | 0.67 | 8.84 |
| Slice 6 | 15.38 | 1.36 | 8.82 |
| Slice 7 | 31.28 | 0.67 | 2.15 |
| Slice 8 | 32.83 | 1.53 | 4.67 |
| Slice 9 | 8.63 | 0.28 | 3.21 |
| Slice 10 | 0.99 | 0.08 | 8.21 |

[1] Average of four samples.

**Table B9**
**Slice Data For FPL 6523-C Weather-O-Meter Aged Chromatograms**

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,010,780 | 1,291,813 | 32.21 |
| Slice 2 | 1,473,156 | 426,074 | 28.92 |
| Slice 3 | 3,116,920 | 1,260,093 | 40.43 |
| Slice 4 | 31,377,576 | 8,038,295 | 25.62 |
| Slice 5 | 73,994,208 | 15,667,501 | 21.17 |
| Slice 6 | 162,732,235 | 28,341,996 | 17.42 |
| Slice 7 | 283,284,455 | 8,407,118 | 2.97 |
| Slice 8 | 278,619,981 | 10,713,423 | 3.85 |
| Slice 9 | 53,662,423 | 21,970,903 | 40.94 |
| Slice 10 | 5,741,303 | 1,153,633 | 20.09 |
| Total | 898,013,037 | 56,446,349 | 6.29 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.44 | 0.12 | 27.86 |
| Slice 2 | 0.16 | 0.04 | 24.79 |
| Slice 3 | 0.34 | 0.13 | 37.91 |
| Slice 4 | 3.46 | 0.74 | 21.24 |
| Slice 5 | 8.17 | 1.31 | 16.00 |
| Slice 6 | 18.02 | 2.27 | 12.62 |
| Slice 7 | 31.63 | 1.55 | 4.91 |
| Slice 8 | 31.12 | 1.96 | 6.29 |
| Slice 9 | 6.00 | 2.63 | 43.77 |
| Slice 10 | 0.64 | 0.12 | 18.51 |

[1] Average of four samples.

## Table B10
## Slice Data For FPL 6523-D Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,650,370 | 350,899 | 7.55 |
| Slice 2 | 2,651,453 | 481,870 | 18.17 |
| Slice 3 | 2,305,204 | 360,792 | 15.65 |
| Slice 4 | 20,822,556 | 2,898,364 | 13.92 |
| Slice 5 | 66,788,887 | 7,172,240 | 10.74 |
| Slice 6 | 138,178,092 | 12,962,845 | 9.38 |
| Slice 7 | 303,130,685 | 6,141,558 | 2.03 |
| Slice 8 | 320,457,149 | 1,039,581 | 0.32 |
| Slice 9 | 84,981,328 | 7,193,757 | 8.47 |
| Slice 10 | 9,980,283 | 977,931 | 9.80 |
| Total | 953,946,007 | 35,511,825 | 3.72 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.49 | 0.05 | 11.14 |
| Slice 2 | 0.28 | 0.06 | 21.62 |
| Slice 3 | 0.24 | 0.05 | 18.66 |
| Slice 4 | 2.18 | 0.23 | 10.70 |
| Slice 5 | 6.98 | 0.49 | 7.08 |
| Slice 6 | 14.46 | 0.83 | 5.76 |
| Slice 7 | 31.80 | 0.57 | 1.79 |
| Slice 8 | 33.64 | 1.17 | 3.47 |
| Slice 9 | 8.90 | 0.50 | 5.65 |
| Slice 10 | 1.04 | 0.06 | 6.14 |

[1] Average of four samples.

## Table B11
## Slice Data For FPL 6523-D Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,782,886 | 225,380 | 4.71 |
| Slice 2 | 2,040,856 | 270,063 | 13.23 |
| Slice 3 | 2,754,865 | 320,627 | 11.64 |
| Slice 4 | 37,413,296 | 1,118,154 | 2.99 |
| Slice 5 | 101,016,874 | 2,514,713 | 2.49 |
| Slice 6 | 185,819,728 | 3,594,974 | 1.93 |
| Slice 7 | 312,093,668 | 1,880,315 | 0.60 |
| Slice 8 | 315,846,581 | 929,563 | 0.29 |
| Slice 9 | 82,310,841 | 6,427,249 | 7.81 |
| Slice 10 | 11,633,305 | 958,179 | 8.24 |
| Total | 1,055,712,899 | 12,445,467 | 1.18 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.45 | 0.02 | 4.06 |
| Slice 2 | 0.19 | 0.02 | 12.39 |
| Slice 3 | 0.26 | 0.03 | 10.86 |
| Slice 4 | 3.54 | 0.09 | 2.62 |
| Slice 5 | 9.57 | 0.13 | 1.33 |
| Slice 6 | 17.60 | 0.23 | 1.30 |
| Slice 7 | 29.57 | 0.43 | 1.46 |
| Slice 8 | 29.92 | 0.34 | 1.14 |
| Slice 9 | 7.79 | 0.53 | 6.86 |
| Slice 10 | 1.10 | 0.08 | 7.11 |

[1] Average of four samples.

## Table B12
## Slice Data For FPL 6523-D Weather-O-Meter Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 6,363,207 | 4,999,987 | 78.58 |
| Slice 2 | 3,097,646 | 2,336,216 | 75.42 |
| Slice 3 | 6,736,527 | 3,080,045 | 45.72 |
| Slice 4 | 44,103,475 | 13,427,216 | 30.44 |
| Slice 5 | 86,533,013 | 19,824,230 | 22.91 |
| Slice 6 | 177,612,656 | 16,169,011 | 9.10 |
| Slice 7 | 239,936,342 | 2,677,829 | 1.12 |
| Slice 8 | 215,449,520 | 3,577,736 | 1.66 |
| Slice 9 | 26,978,653 | 4,204,575 | 15.58 |
| Slice 10 | 4,546,784 | 1,374,536 | 30.23 |
| Total | 811,357,822 | 69,824,797 | 8.61 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.74 | 0.55 | 75.26 |
| Slice 2 | 0.36 | 0.26 | 71.70 |
| Slice 3 | 0.80 | 0.31 | 38.72 |
| Slice 4 | 5.33 | 1.20 | 22.50 |
| Slice 5 | 10.54 | 1.55 | 14.69 |
| Slice 6 | 21.88 | 0.25 | 1.14 |
| Slice 7 | 29.77 | 2.26 | 7.60 |
| Slice 8 | 26.73 | 2.03 | 7.59 |
| Slice 9 | 3.31 | 0.25 | 7.44 |
| Slice 10 | 0.55 | 0.12 | 22.31 |

[1] Average of four samples.

**Table B13**
**Percent Average Area Per Slice for FPL 6523 As-Received and Unaged Chromatograms**

| Slice Number | As-Received | Heating Time | | | |
|---|---|---|---|---|---|
| | | A | B | C | D |
| Slice 1 | 0.00 | 0.14 | 0.16 | 0.29 | 0.49 |
| Slice 2 | 0.01 | 0.09 | 0.01 | 0.10 | 0.28 |
| Slice 3 | 0.16 | 0.15 | 0.05 | 0.36 | 0.24 |
| Slice 4 | 1.96 | 2.21 | 2.34 | 2.75 | 2.18 |
| Slice 5 | 6.46 | 7.11 | 7.75 | 6.34 | 6.98 |
| Slice 6 | 16.10 | 15.38 | 16.13 | 14.96 | 14.46 |
| Slice 7 | 33.63 | 32.48 | 30.63 | 36.76 | 31.80 |
| Slice 8 | 34.36 | 33.68 | 31.61 | 34.59 | 33.64 |
| Slice 9 | 6.95 | 7.73 | 10.14 | 3.34 | 8.90 |
| Slice 10 | 0.36 | 1.03 | 1.19 | 0.51 | 1.04 |

**Table B14**
**Percent Average Area Per Slice For FPL 6523 Oven-Aged Chromatograms.**

| Slice Number | Heating Time | | | |
|---|---|---|---|---|
| | A | B | C | D |
| Slice 1 | 0.24 | 0.35 | 0.41 | 0.45 |
| Slice 2 | 0.08 | 0.18 | 0.20 | 0.19 |
| Slice 3 | 0.28 | 0.39 | 0.22 | 0.26 |
| Slice 4 | 3.34 | 3.16 | 2.44 | 3.54 |
| Slice 5 | 9.03 | 8.67 | 7.62 | 9.57 |
| Slice 6 | 17.43 | 17.46 | 15.38 | 17.60 |
| Slice 7 | 30.00 | 30.33 | 31.28 | 29.57 |
| Slice 8 | 30.46 | 30.85 | 32.83 | 29.92 |
| Slice 9 | 8.03 | 7.56 | 8.63 | 7.79 |
| Slice 10 | 1.10 | 1.06 | 0.99 | 1.10 |

**Table B15**
**Percent Average Area Per Slice for FPL 6523**
**Weather-O-Meter Aged Chromatograms**

| Slice Number | Heating Time | | | |
| --- | --- | --- | --- | --- |
| | A | B | C | D |
| Slice 1 | [1] | 0.33 | 0.44 | 0.74 |
| Slice 2 | [1] | 0.10 | 0.16 | 0.36 |
| Slice 3 | [1] | 0.35 | 0.34 | 0.80 |
| Slice 4 | [1] | 3.47 | 3.46 | 5.33 |
| Slice 5 | [1] | 8.57 | 8.17 | 10.54 |
| Slice 6 | [1] | 18.99 | 18.02 | 21.88 |
| Slice 7 | [1] | 30.30 | 31.63 | 29.77 |
| Slice 8 | [1] | 29.98 | 31.12 | 26.73 |
| Slice 9 | [1] | 7.15 | 6.00 | 3.31 |
| Slice 10 | [1] | 0.76 | 0.64 | 0.55 |

[1] Slice data not available.

# Appendix C
# FPL 6540 Chromatograms

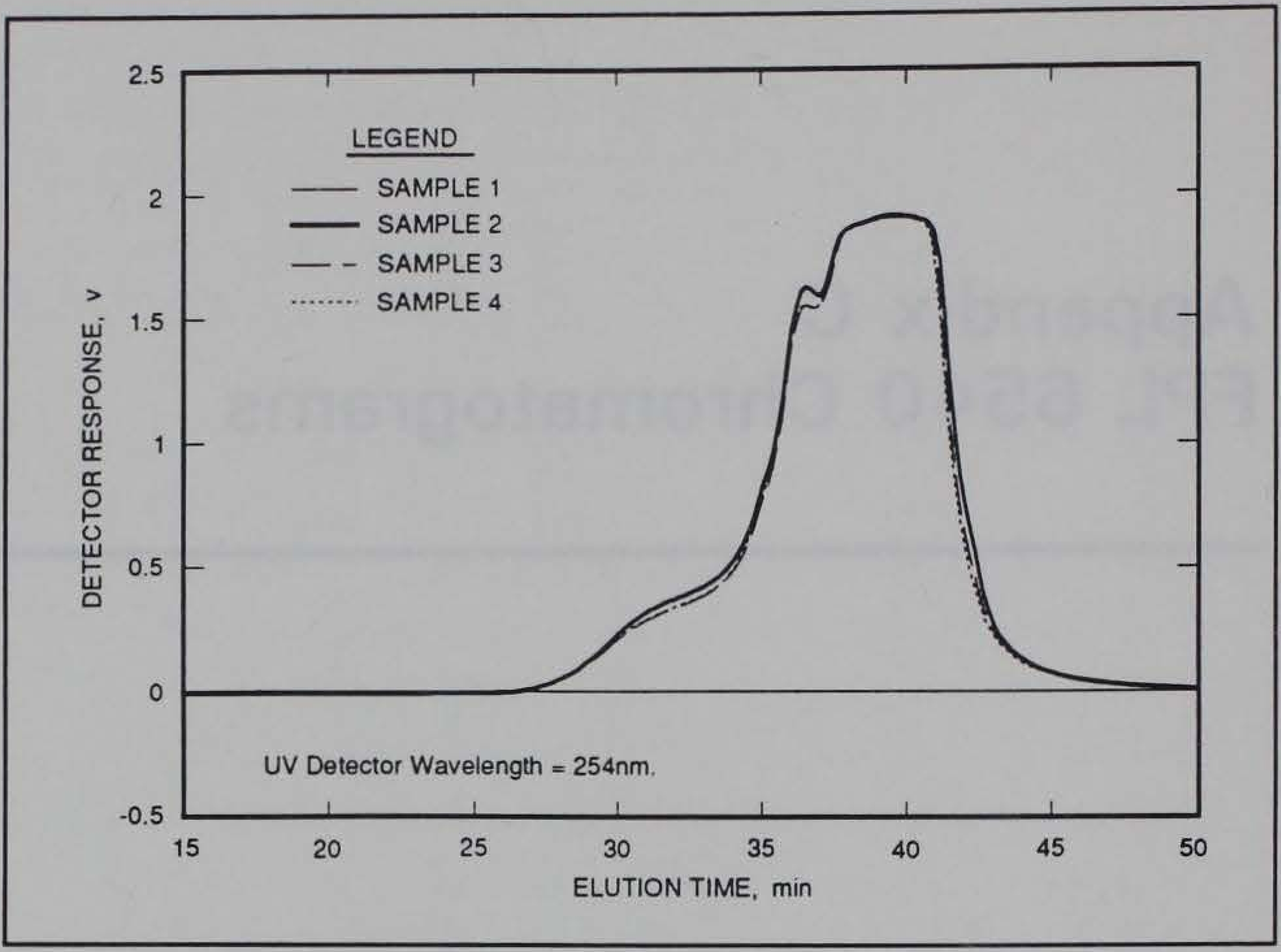


Figure C1. FPL 6540 as-received chromatograms

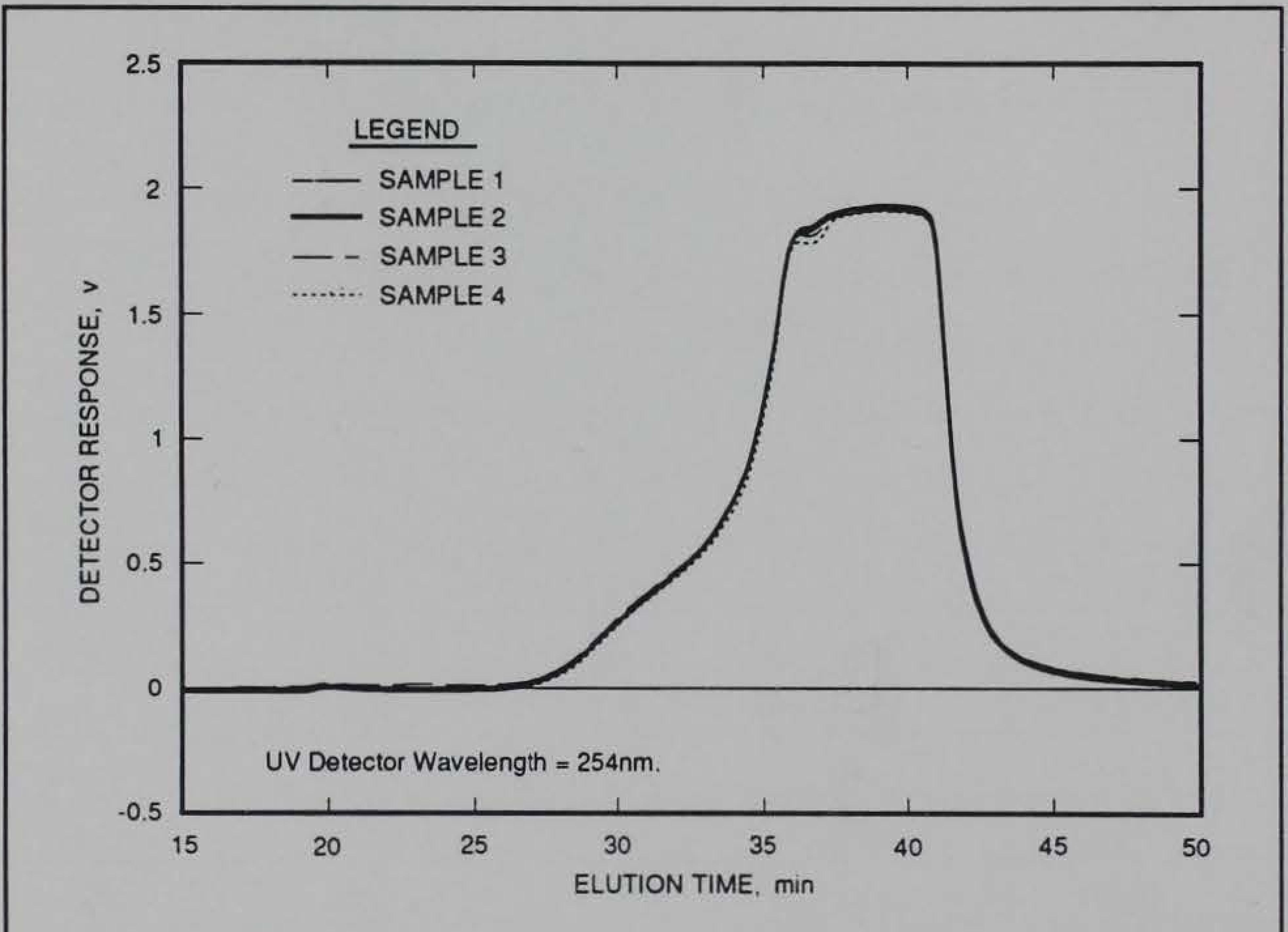


Figure C2. FPL 6540-A unaged chromatograms

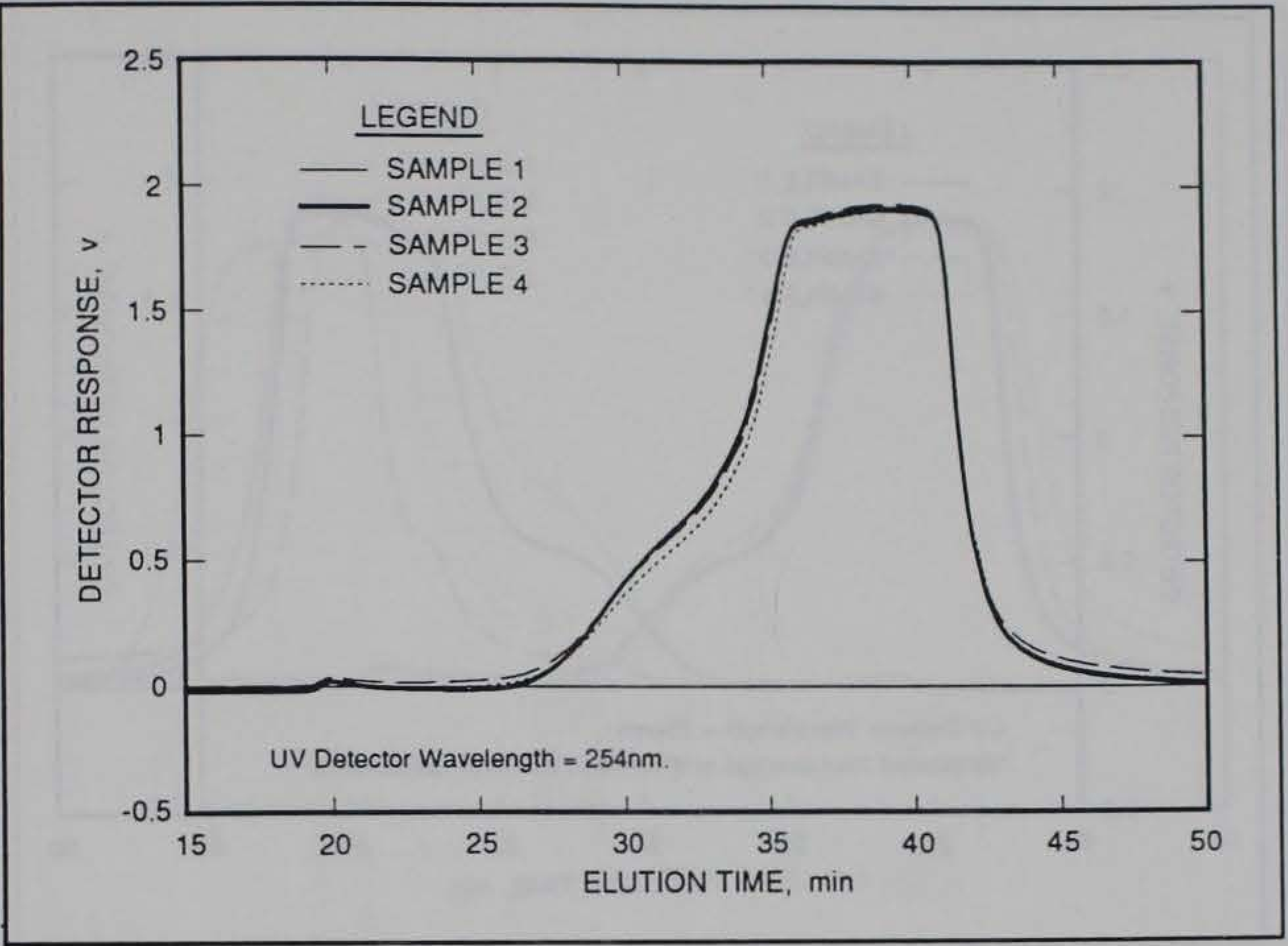


Figure C3. FPL 6540-A oven-aged chromatograms

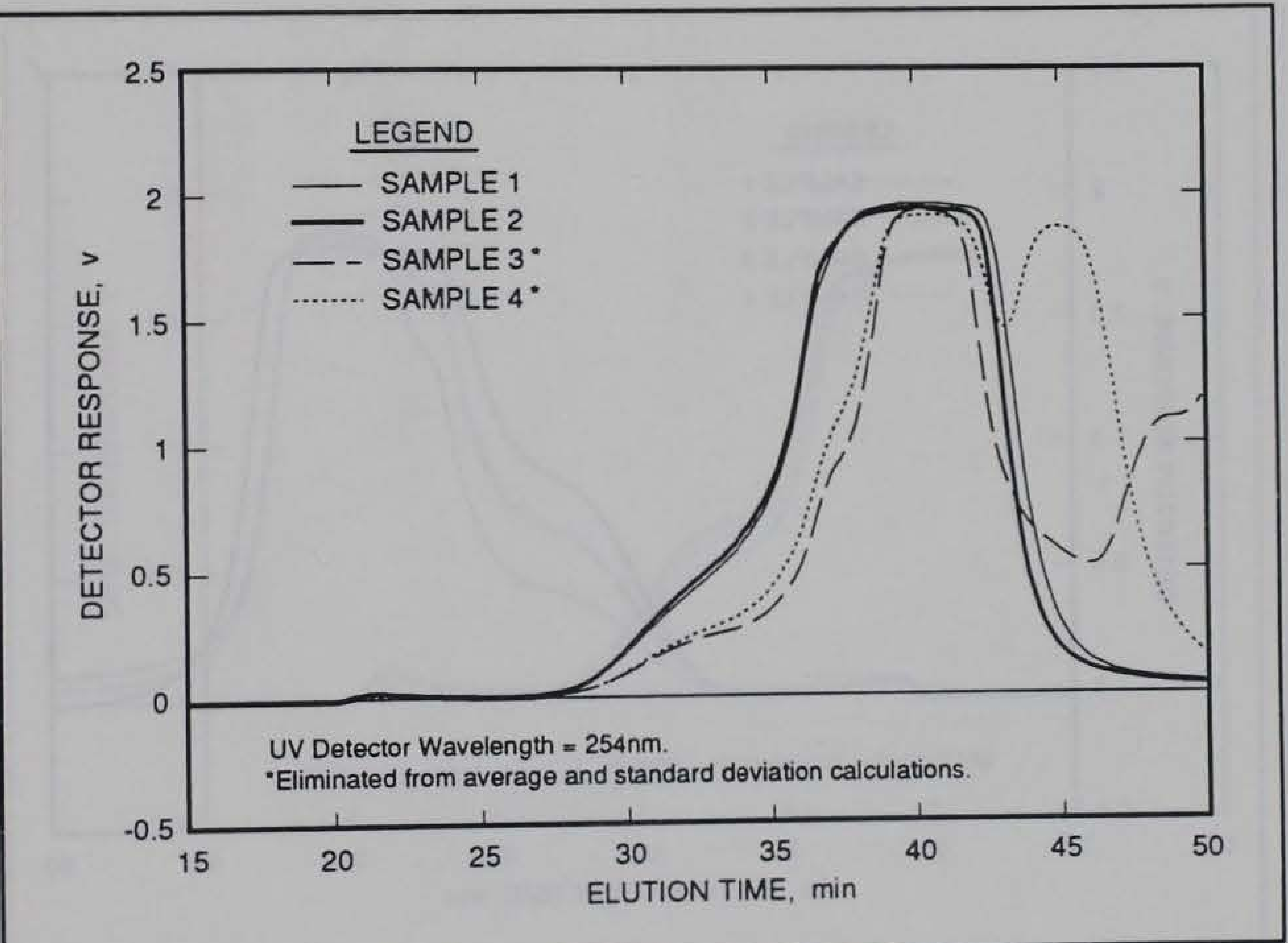


Figure C4. FPL 6540-A weather-o-meter aged chromatograms

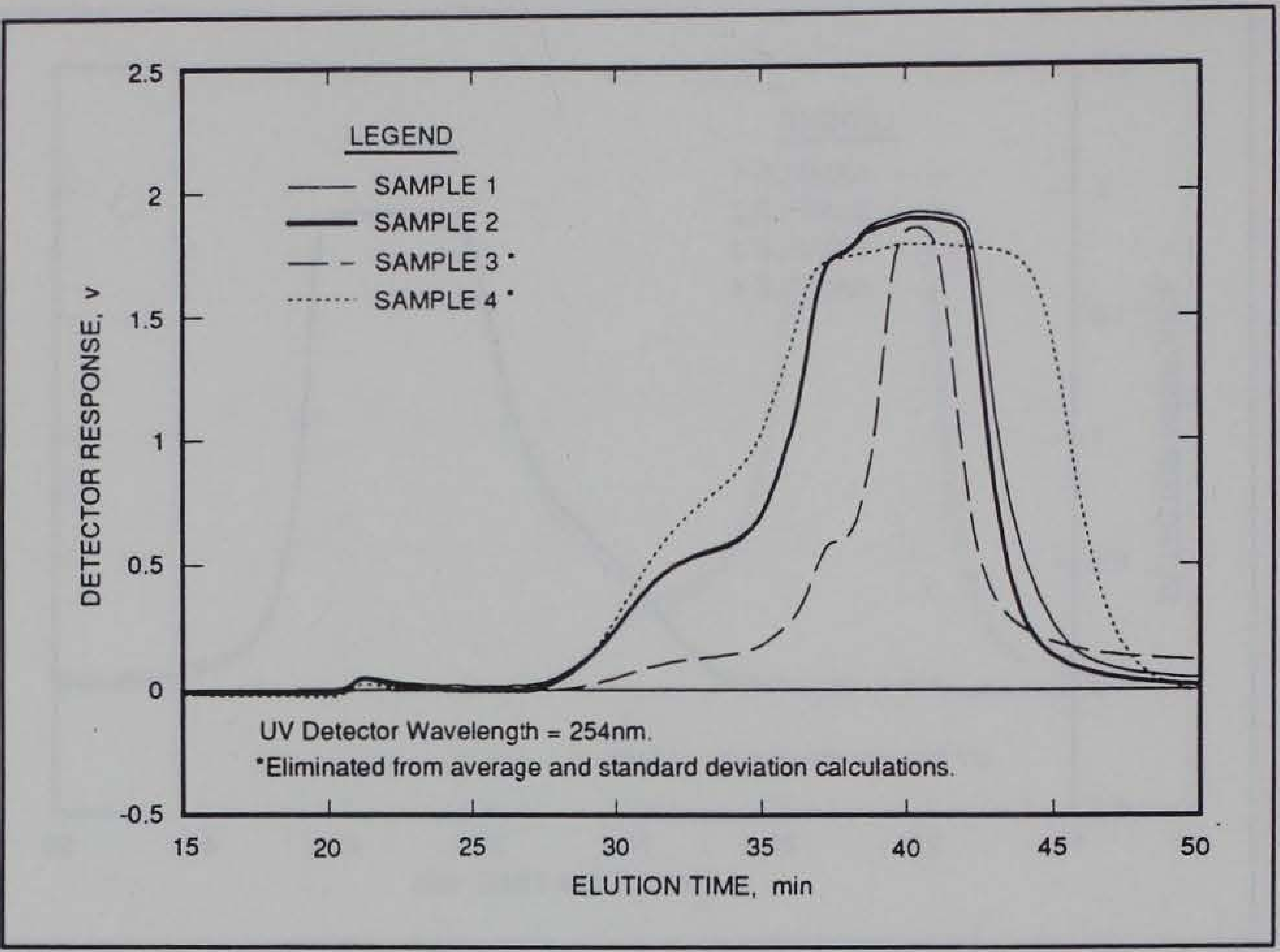


Figure C5. FPL 6540-B unaged chromatograms

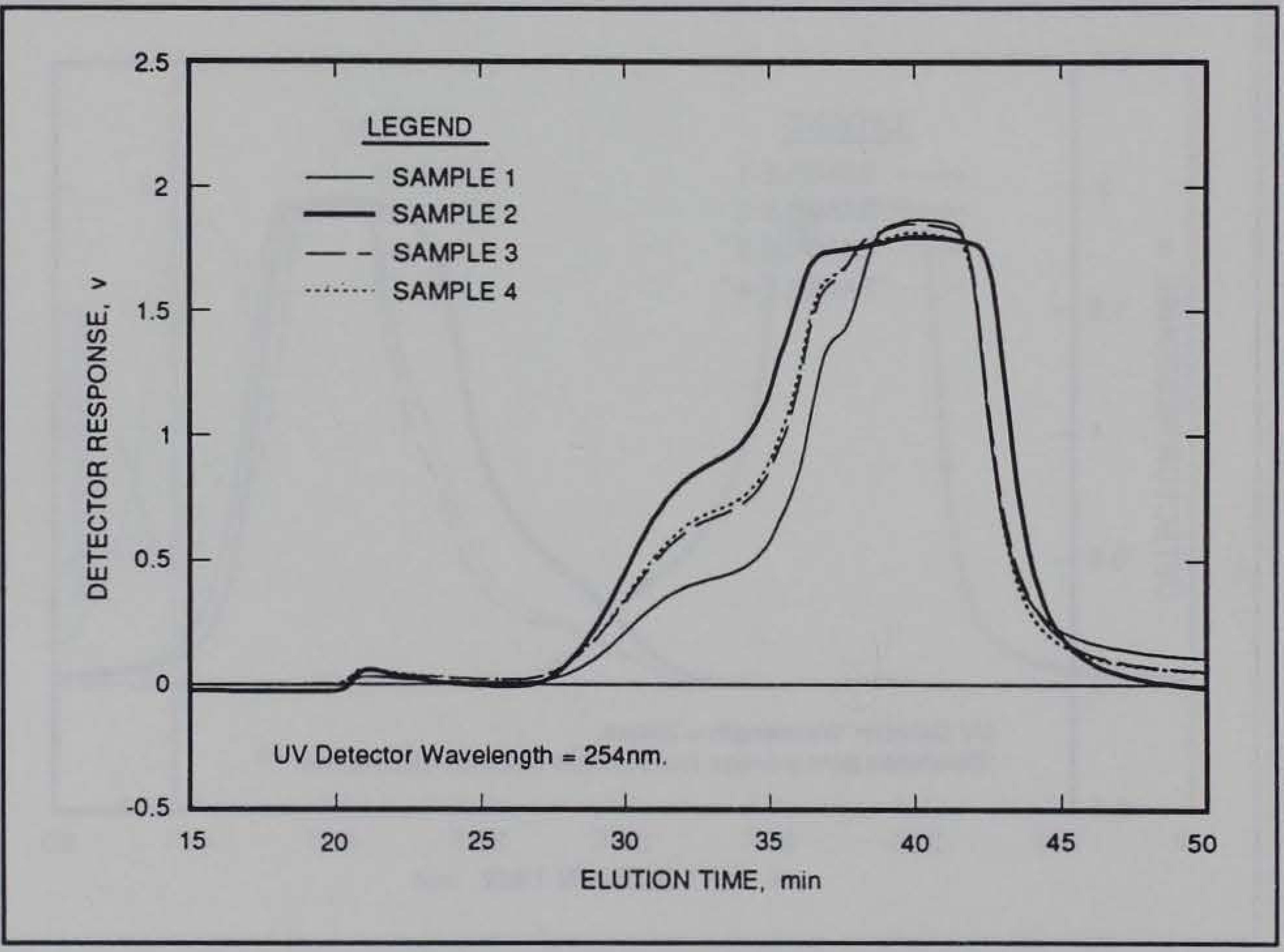


Figure C6. FPL 6540-B oven-aged chromatograms

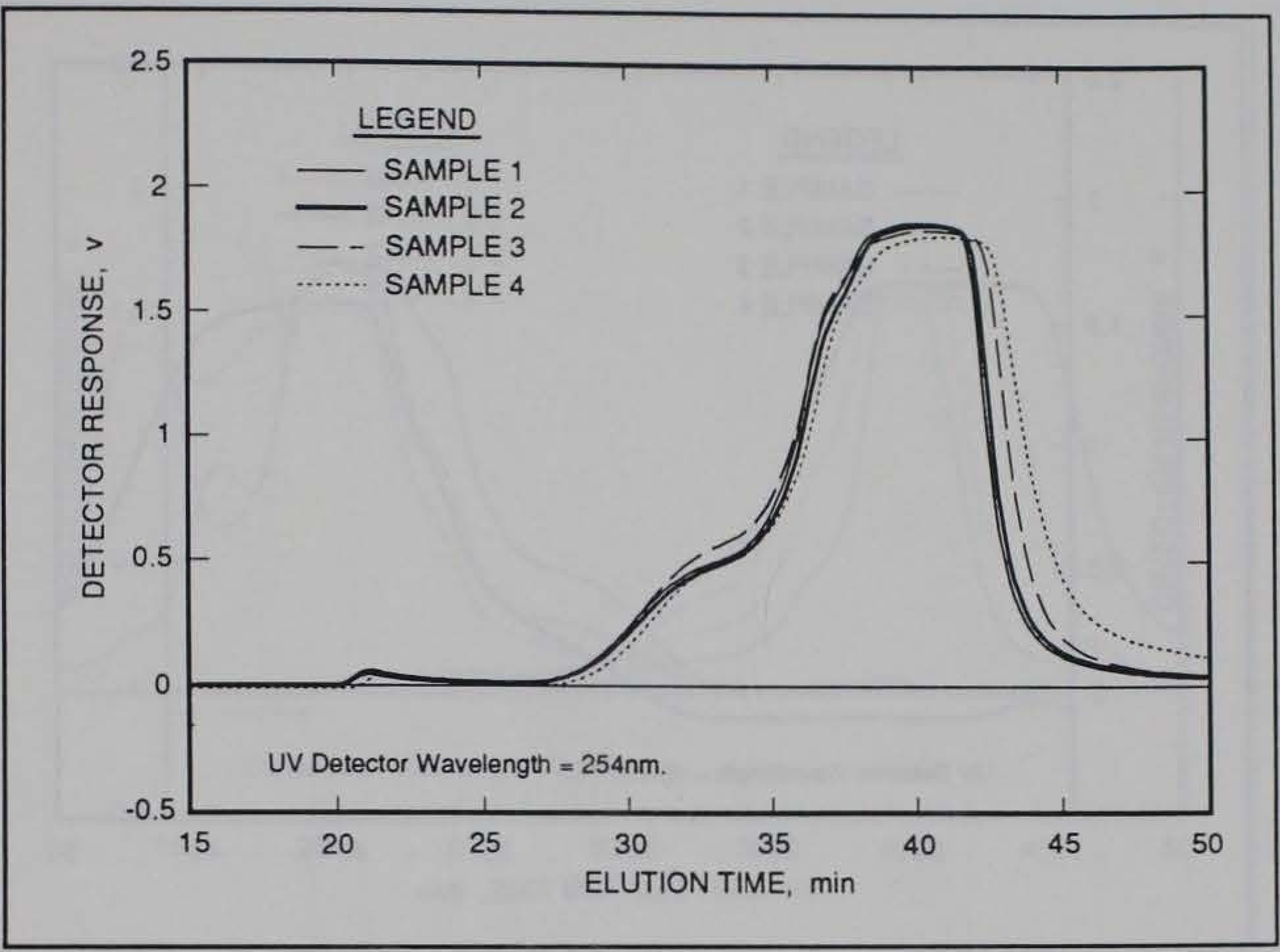


Figure C7. FPL 6540-B weather-o-meter aged chromatograms

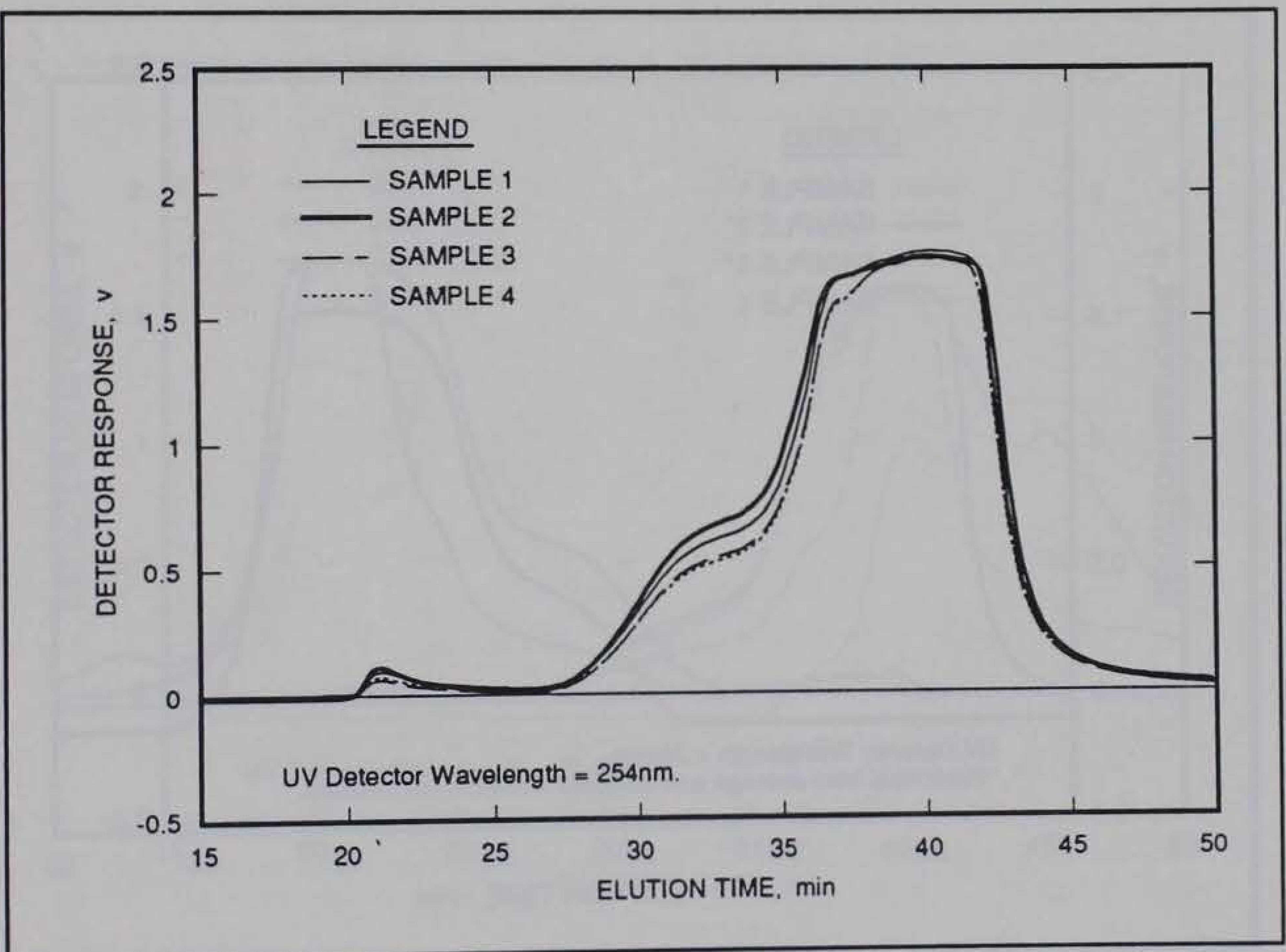


Figure C8. FPL 6540-C unaged chromatograms

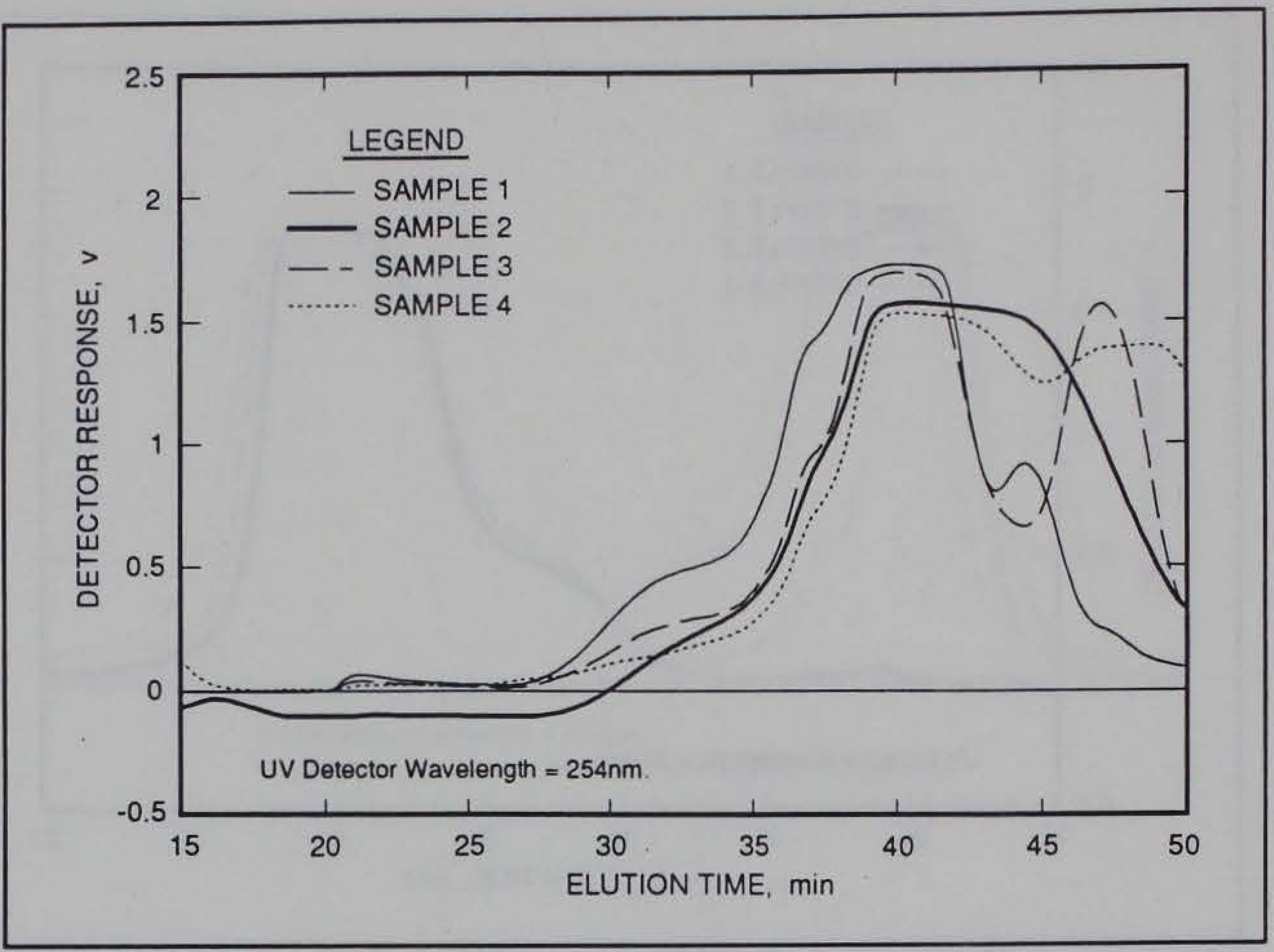


Figure C9. FPL 6540-C oven-aged chromatograms

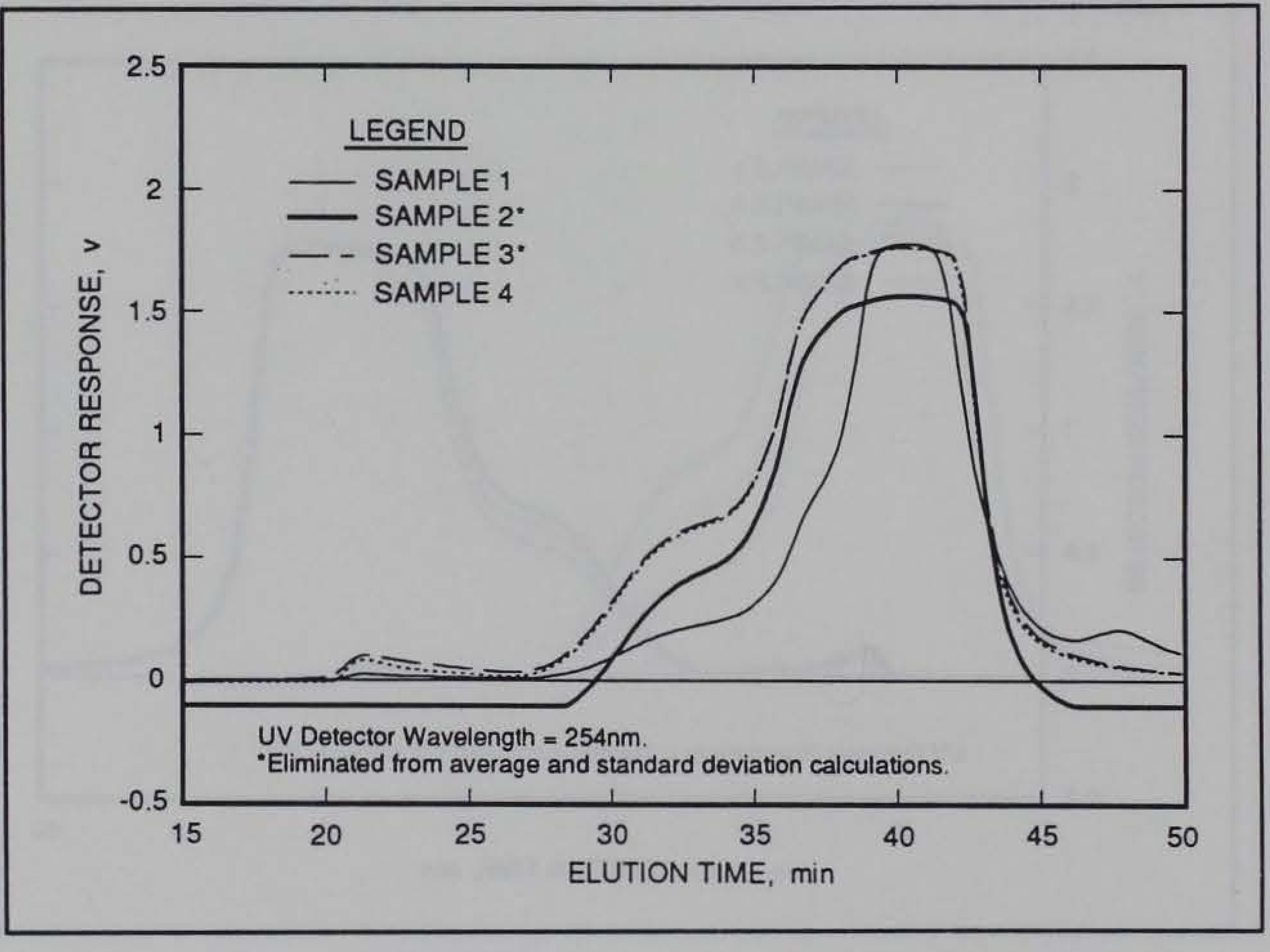


Figure C10. FPL 6540-C weather-o-meter aged chromatograms

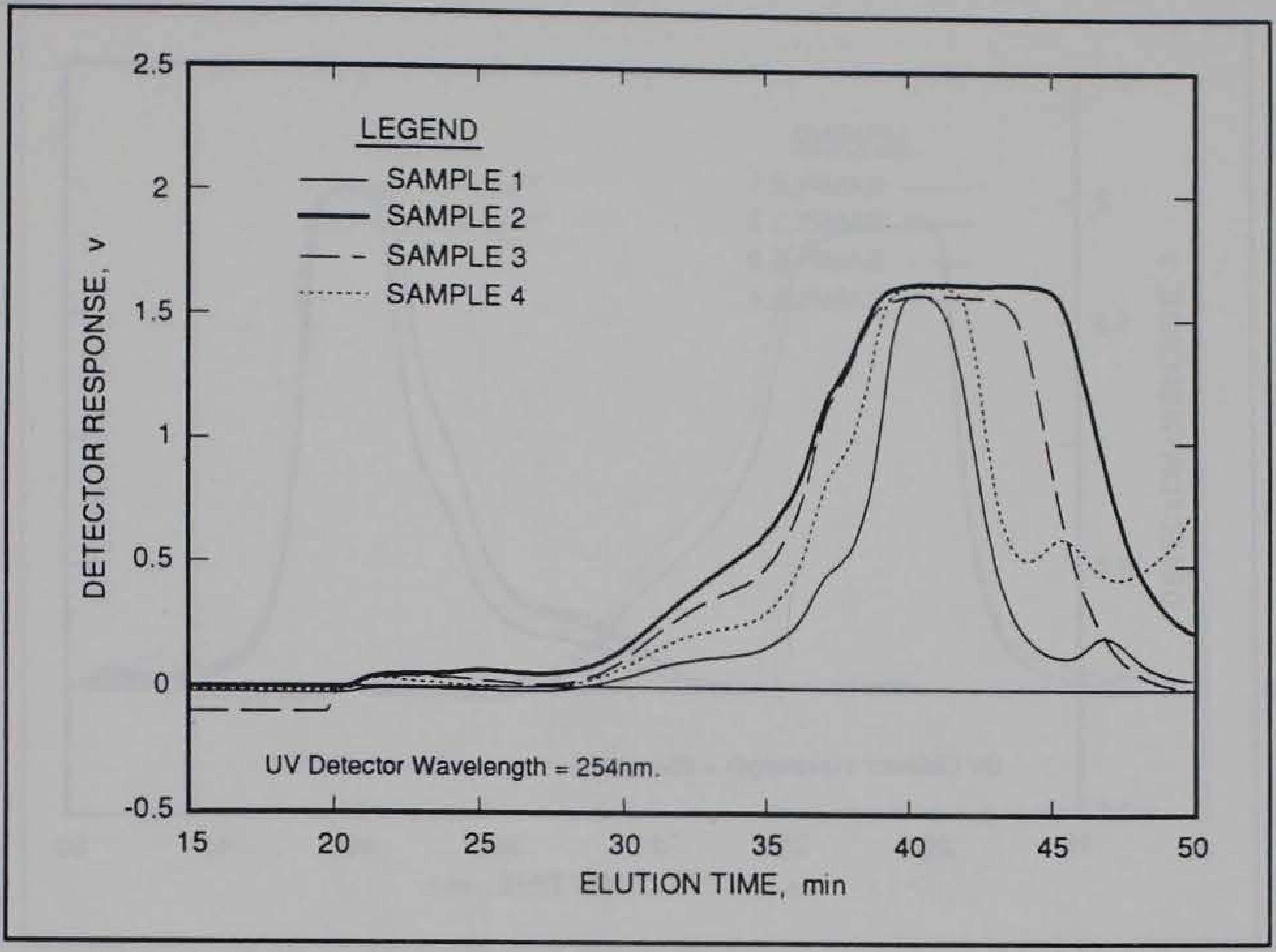


Figure C11. FPL 6540-D unaged chromatograms

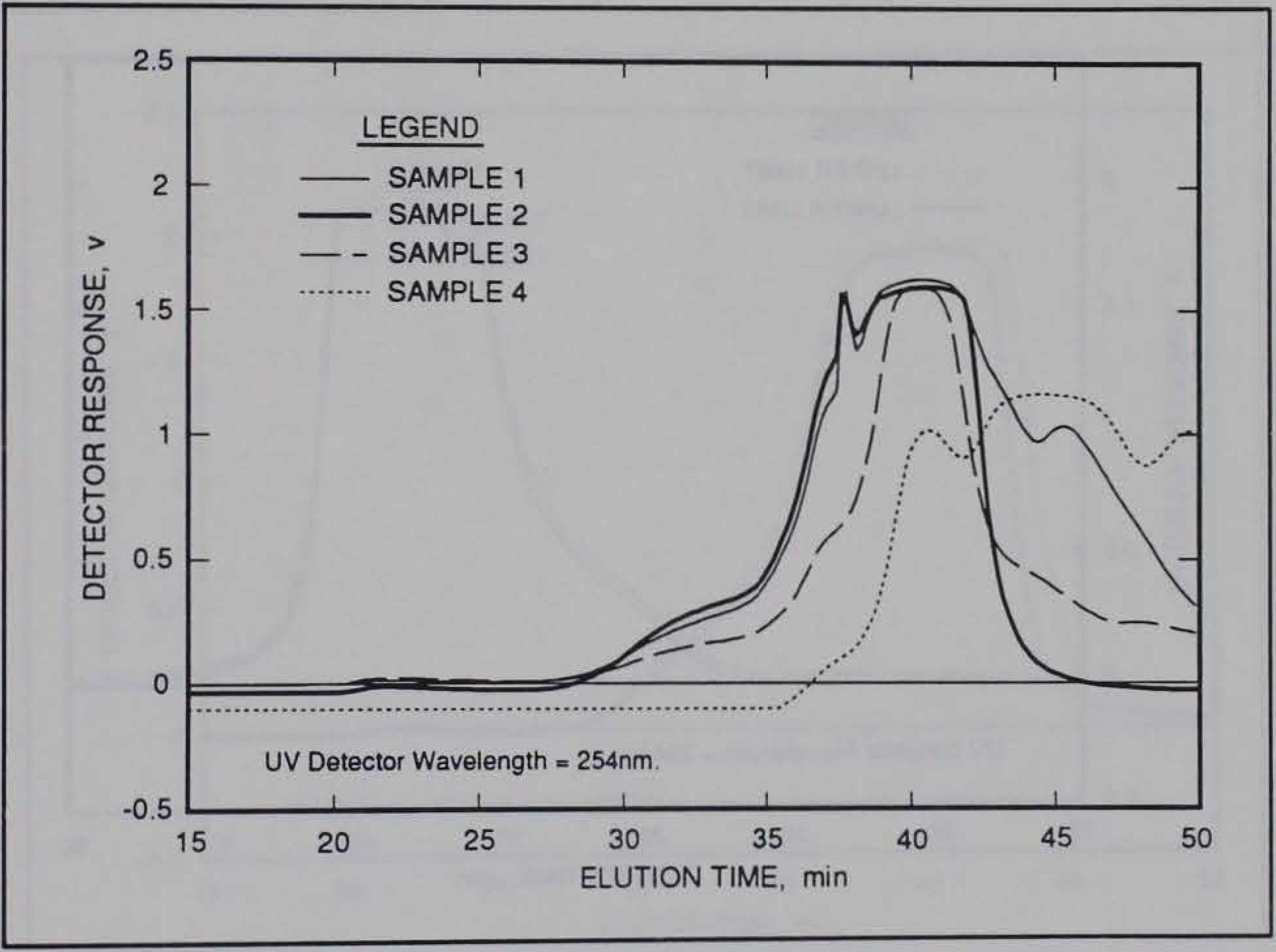


Figure C12. FPL 6540-D oven-aged chromatograms

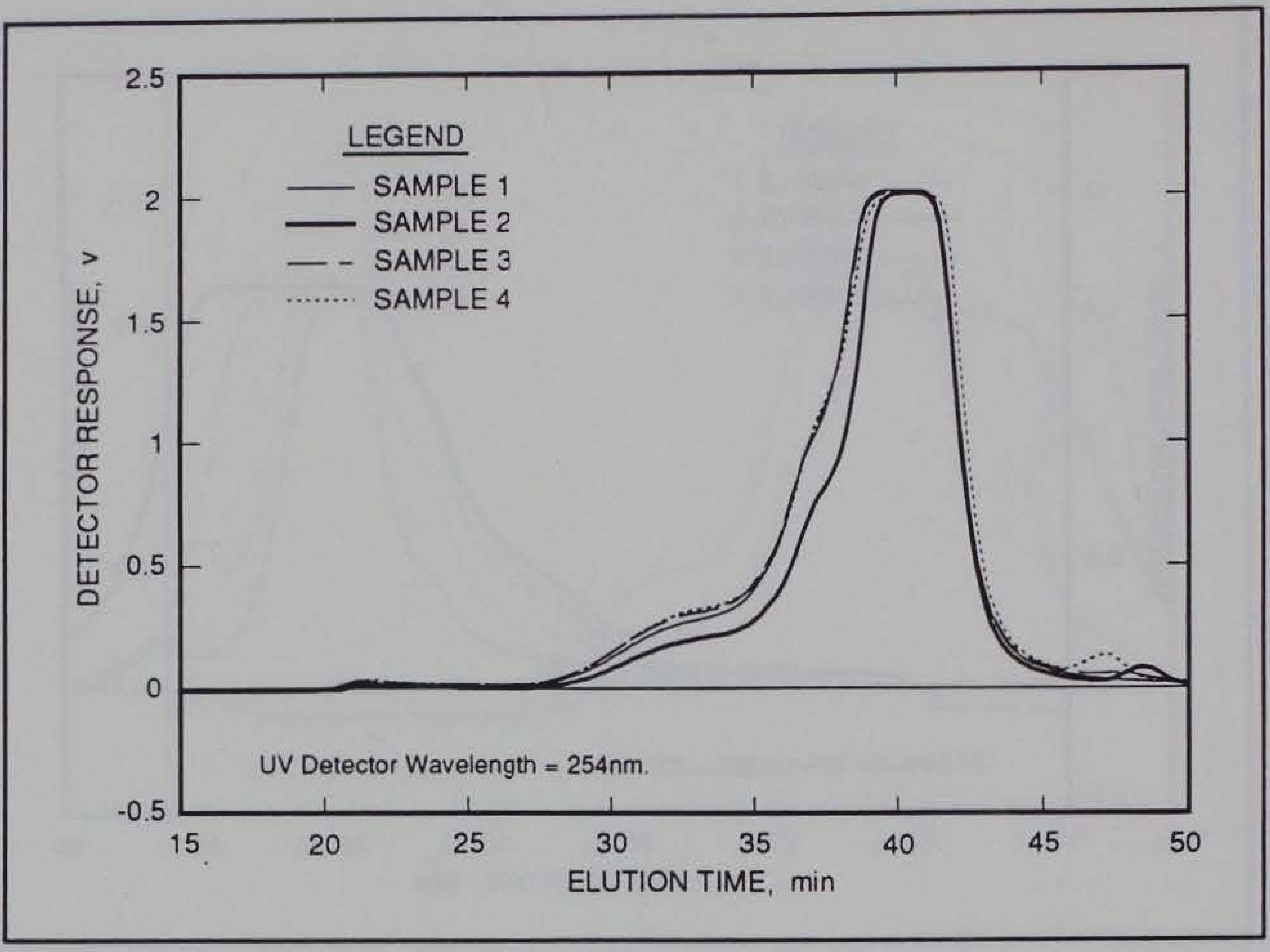


Figure C13. FPL 6540-D weather-o-meter aged chromatograms

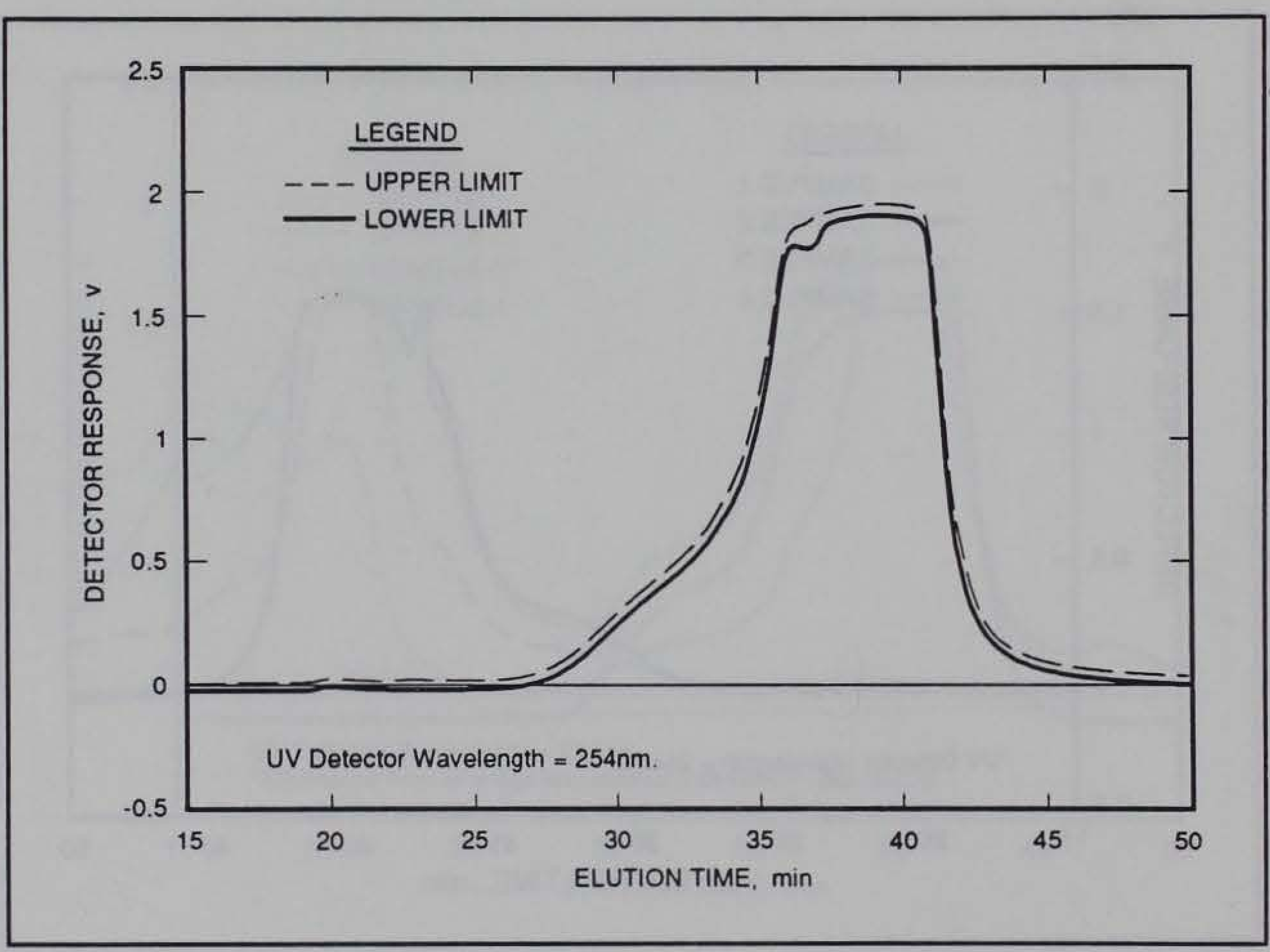


Figure C14. FPL 6540-A unaged chromatogram 95 percent confidence region

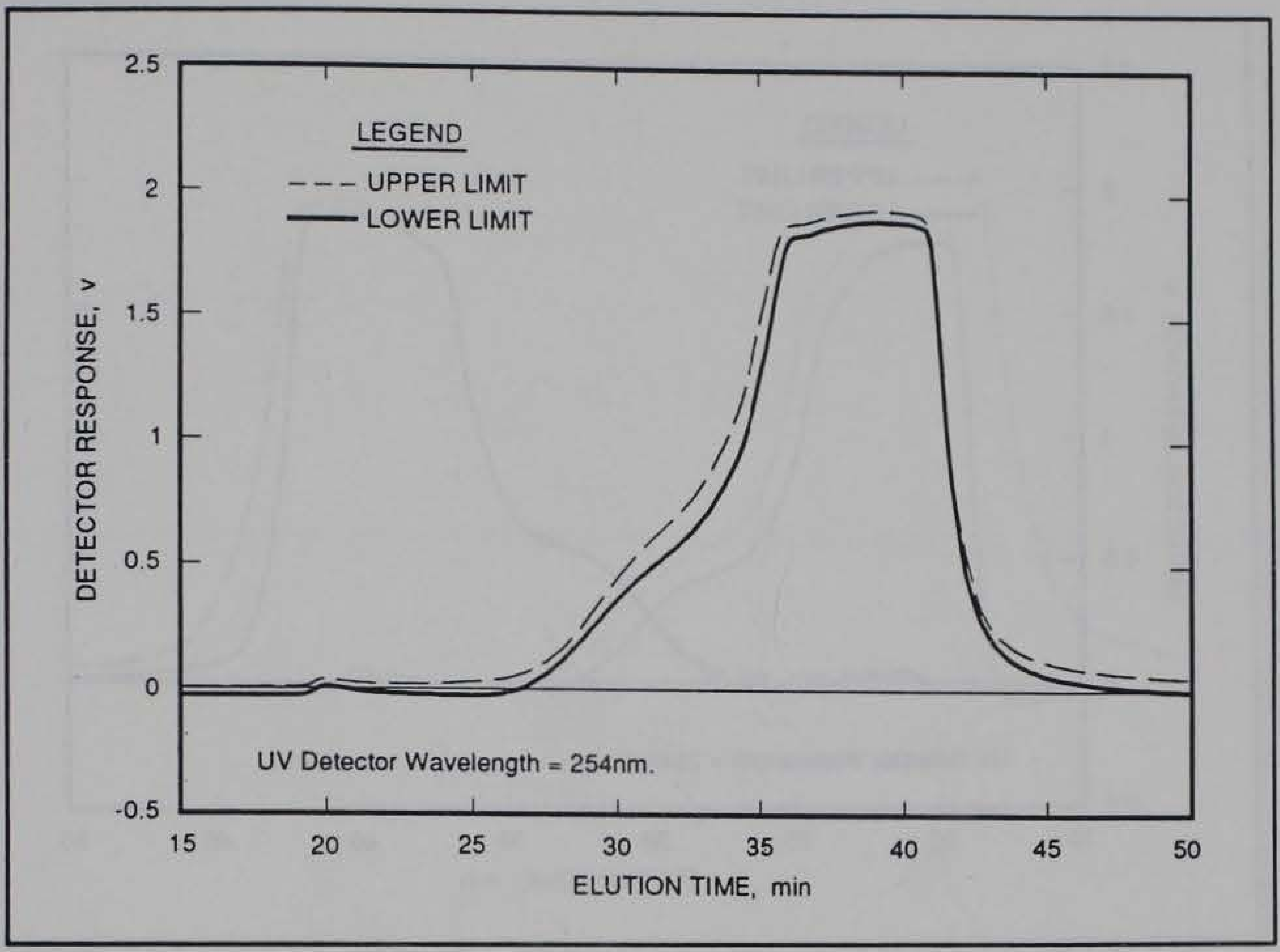


Figure C15. FPL 6540-A oven-aged chromatogram 95 percent confidence region

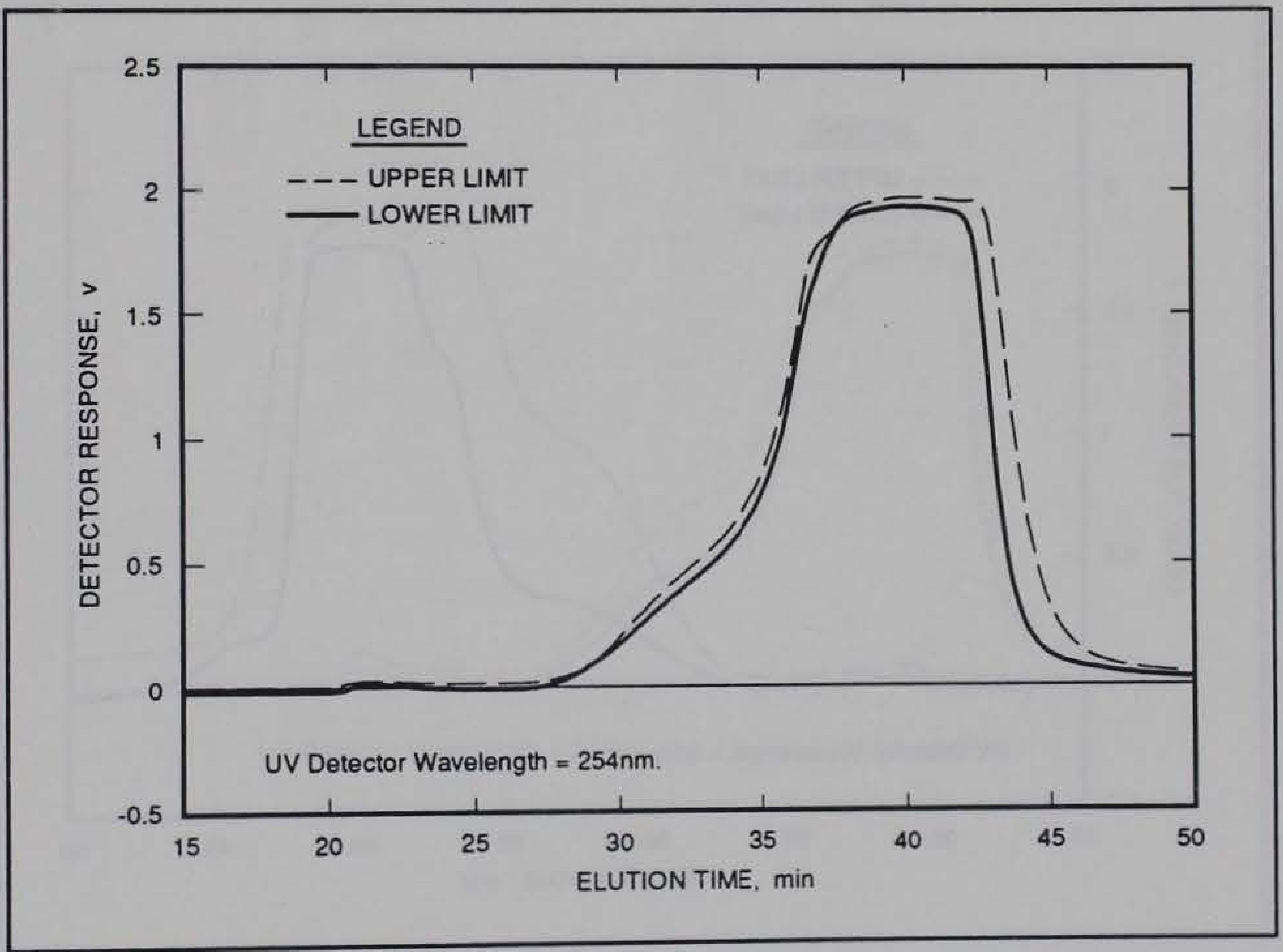


Figure C16. FPL 6540-A weather-o-meter aged chromatogram 95 percent confidence region

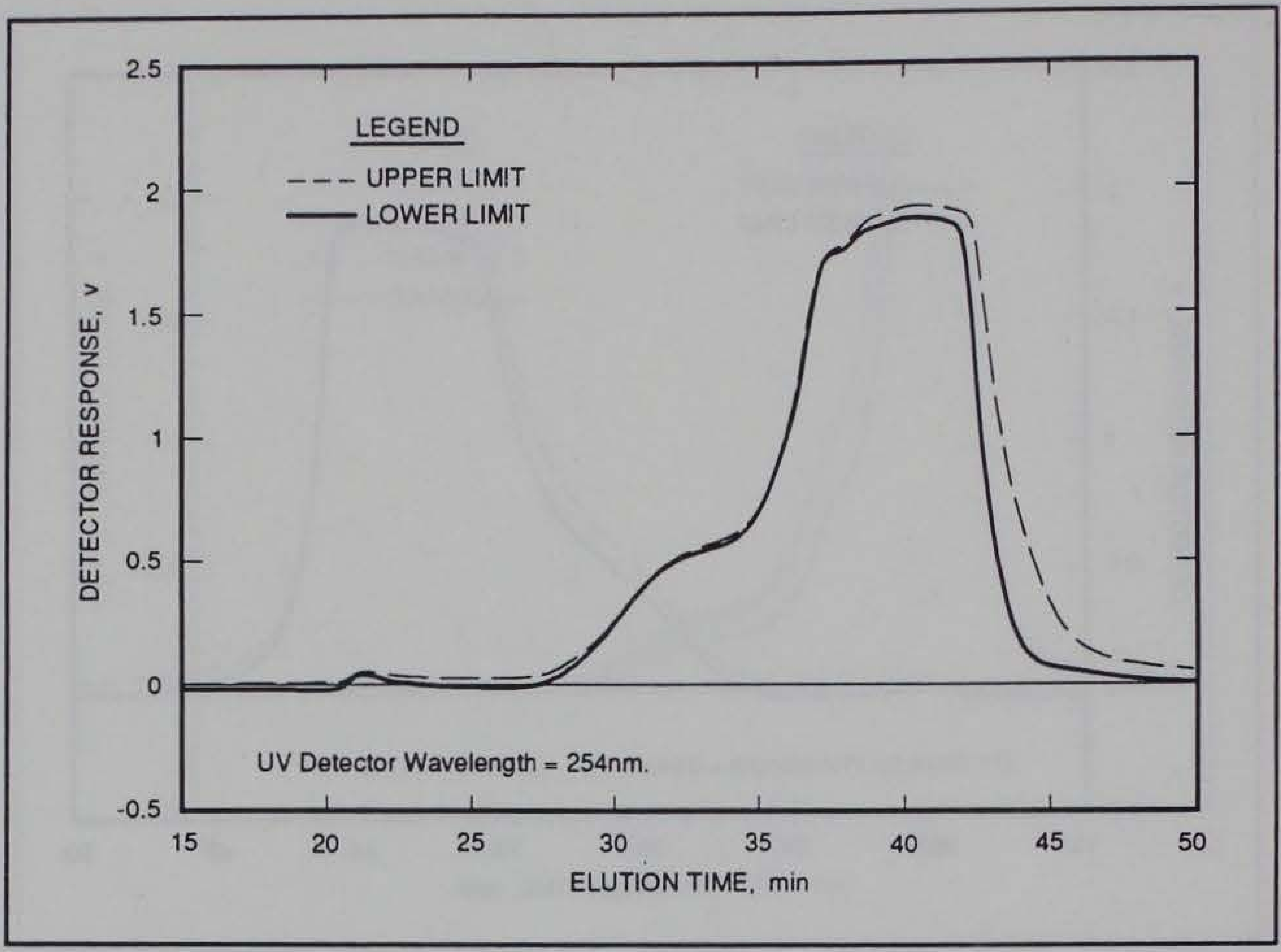


Figure C17. FPL 6540-B unaged chromatogram 95 percent confidence region

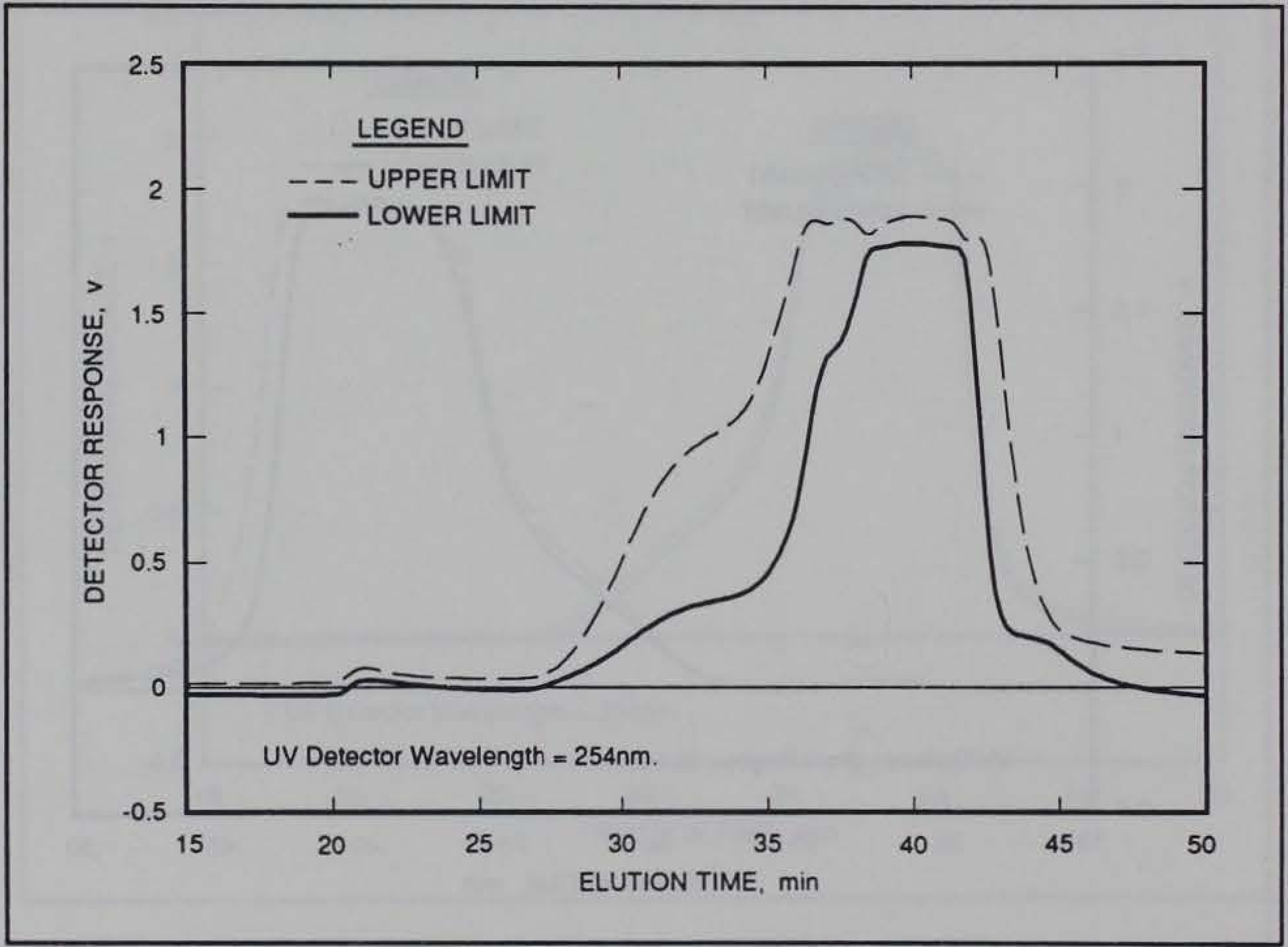


Figure C18. FPL 6540-B oven-aged chromatogram 95 percent confidence region

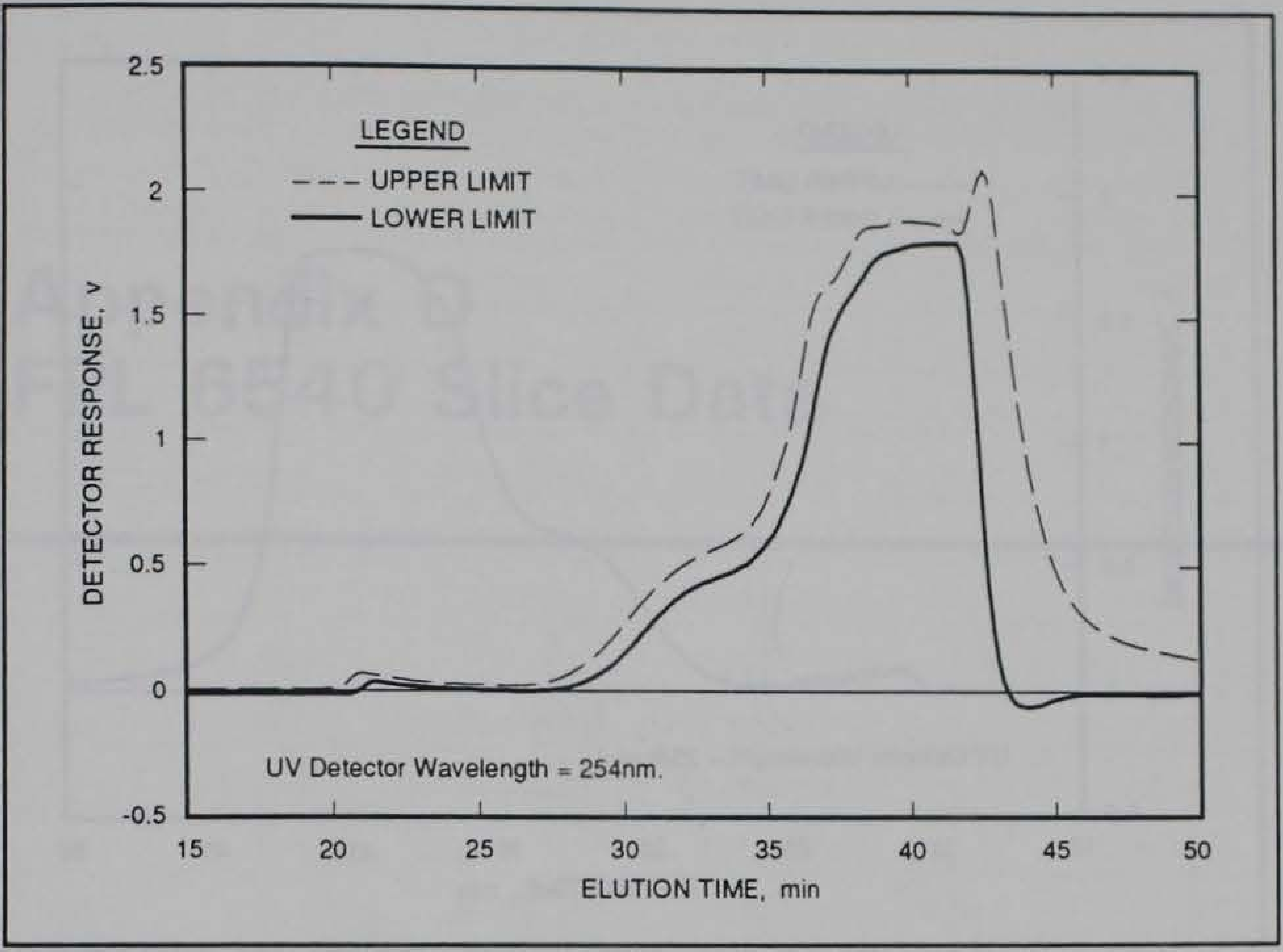


Figure C19. FPL 6540-B weather-o-meter aged chromatogram 95 percent confidence region

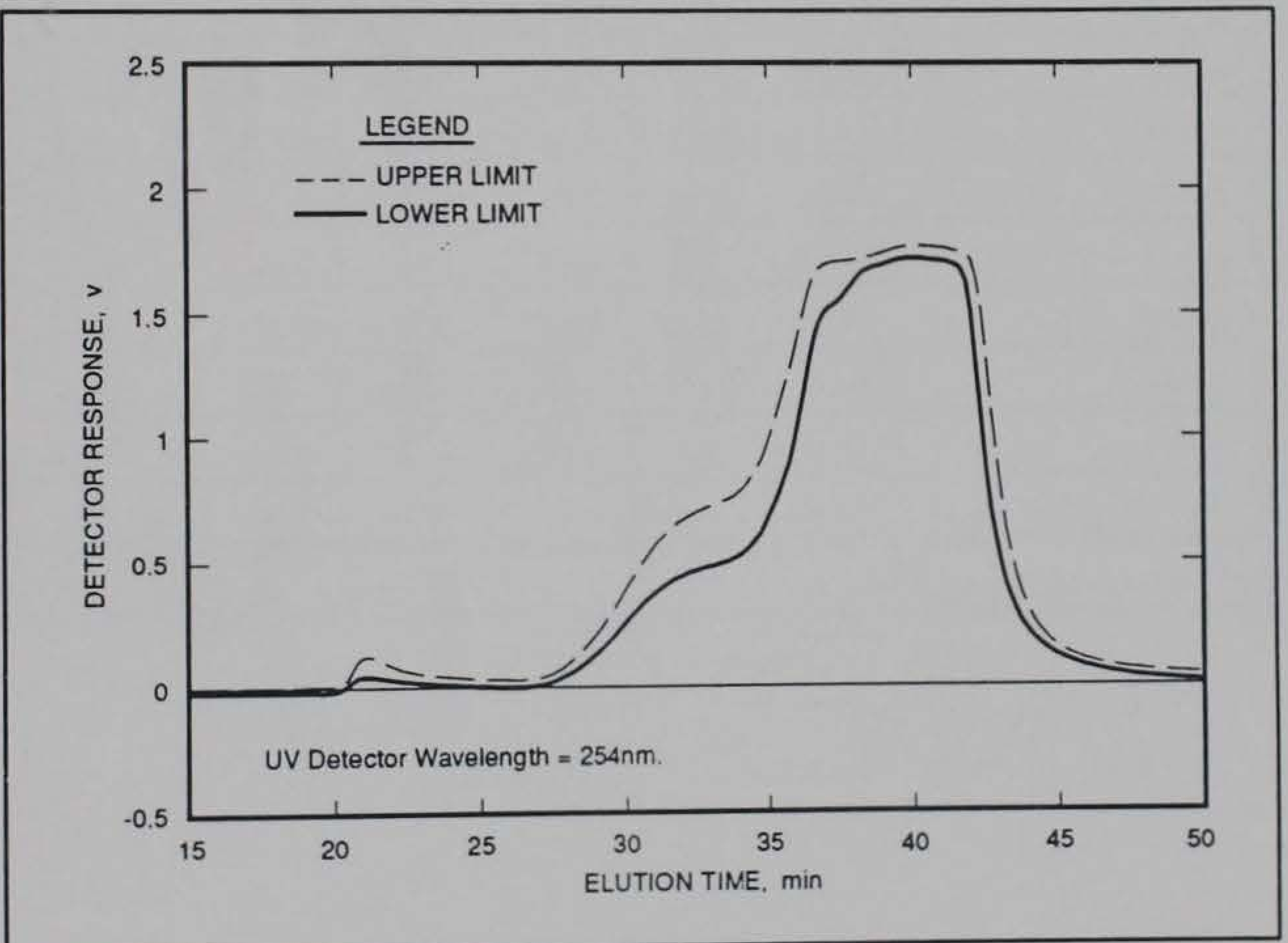


Figure C20. FPL 6540-C unaged chromatogram 95 percent confidence region

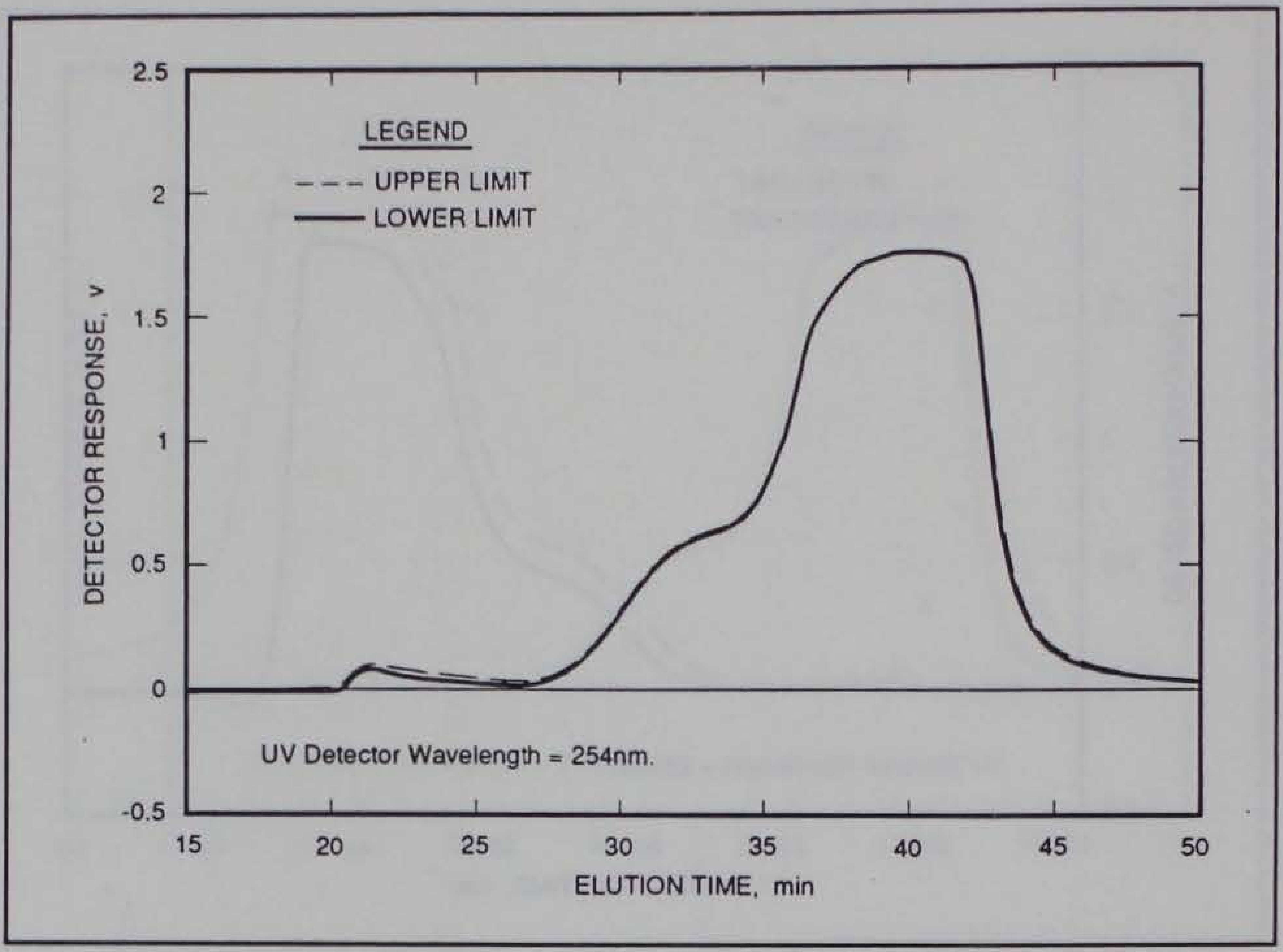


Figure C21. FPL 6540-C weather-o-meter aged chromatogram 95 percent confidence region

# Appendix D
# FPL 6540 Slice Data

## Table D1
## Slice Data For FPL 6540 As-Received Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 0 | 0 | NA |
| Slice 2 | 0 | 0 | NA |
| Slice 3 | 4,588,098 | 296,376 | 6.46 |
| Slice 4 | 35,721,633 | 1,770,298 | 4.96 |
| Slice 5 | 67,314,353 | 3,017,480 | 4.48 |
| Slice 6 | 186,546,371 | 4,465,436 | 2.39 |
| Slice 7 | 303,075,410 | 753,016 | 0.25 |
| Slice 8 | 213,402,917 | 9,064,051 | 4.25 |
| Slice 9 | 22,155,274 | 1,579,779 | 7.13 |
| Slice 10 | 4,603,212 | 173,196 | 3.76 |
| Total | 837,407,269 | 20,304,091 | 2.42 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.00 | 0.00 | NA |
| Slice 2 | 0.00 | 0.00 | NA |
| Slice 3 | 0.55 | 0.02 | 4.42 |
| Slice 4 | 4.26 | 0.12 | 2.81 |
| Slice 5 | 8.03 | 0.17 | 2.14 |
| Slice 6 | 22.28 | 0.15 | 0.69 |
| Slice 7 | 36.21 | 0.80 | 2.21 |
| Slice 8 | 25.47 | 0.52 | 2.03 |
| Slice 9 | 2.64 | 0.13 | 4.80 |
| Slice 10 | 0.55 | 0.01 | 1.60 |

[1] Average of three samples.

## Table D2
## Slice Data For FPL 6540-A Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 3,127,024 | 1,494,691 | 47.80 |
| Slice 2 | 1,405,194 | 1,026,377 | 73.04 |
| Slice 3 | 5,495,796 | 3,385,121 | 61.59 |
| Slice 4 | 41,428,593 | 18,754,891 | 45.27 |
| Slice 5 | 82,129,446 | 33,676,195 | 41.00 |
| Slice 6 | 152,034,643 | 45,802,359 | 30.13 |
| Slice 7 | 292,414,811 | 11,484,045 | 3.93 |
| Slice 8 | 288,184,696 | 9,370,239 | 3.25 |
| Slice 9 | 48,177,271 | 12,290,624 | 25.51 |
| Slice 10 | 6,826,502 | 2,194,783 | 32.15 |
| Total | 921,223,976 | 139,348,692 | 15.13 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.32 | 0.10 | 32.27 |
| Slice 2 | 0.14 | 0.08 | 58.32 |
| Slice 3 | 0.56 | 0.26 | 45.85 |
| Slice 4 | 4.31 | 1.25 | 29.03 |
| Slice 5 | 8.59 | 2.13 | 24.77 |
| Slice 6 | 16.16 | 2.29 | 14.14 |
| Slice 7 | 32.24 | 3.28 | 10.18 |
| Slice 8 | 31.80 | 3.43 | 10.80 |
| Slice 9 | 5.15 | 0.51 | 9.95 |
| Slice 10 | 0.72 | 0.12 | 16.09 |

[1] Average of two samples.

## Table D3
## Slice Data For FPL 6540-A Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,760,531 | 1,888,997 | 39.68 |
| Slice 2 | 1,636,324 | 1,319,541 | 80.64 |
| Slice 3 | 4,893,687 | 4,408,669 | 90.09 |
| Slice 4 | 44,508,273 | 15,305,647 | 34.39 |
| Slice 5 | 89,042,337 | 26,235,103 | 29.46 |
| Slice 6 | 161,081,886 | 38,190,139 | 23.71 |
| Slice 7 | 281,771,758 | 18,395,580 | 6.53 |
| Slice 8 | 294,718,307 | 4,234,115 | 1.44 |
| Slice 9 | 120,251,616 | 47,033,470 | 39.11 |
| Slice 10 | 14,071,555 | 4,273,704 | 30.37 |
| Total | 1,016,736,271 | 58,670,615 | 5.77 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.46 | 0.16 | 34.70 |
| Slice 2 | 0.15 | 0.12 | 78.52 |
| Slice 3 | 0.46 | 0.41 | 88.94 |
| Slice 4 | 4.31 | 1.26 | 29.20 |
| Slice 5 | 8.64 | 2.08 | 24.10 |
| Slice 6 | 15.68 | 2.85 | 18.19 |
| Slice 7 | 27.70 | 0.21 | 0.76 |
| Slice 8 | 29.06 | 1.26 | 4.34 |
| Slice 9 | 12.13 | 5.33 | 43.89 |
| Slice 10 | 1.41 | 0.50 | 35.52 |

[1] Average of two samples.

**Table D4**
**Slice Data For FPL 6540-A Weather-O-Meter Aged Chromatograms**

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,419,216 | 97,607 | 4.03 |
| Slice 2 | 233,289 | 122,363 | 52.45 |
| Slice 3 | 2,009,436 | 21,371 | 1.06 |
| Slice 4 | 31,267,662 | 1,797,740 | 5.75 |
| Slice 5 | 80,098,074 | 3,192,014 | 3.99 |
| Slice 6 | 183,851,930 | 4,682,794 | 2.55 |
| Slice 7 | 309,590,707 | 52,271 | 0.02 |
| Slice 8 | 313,532,860 | 2,302,495 | 0.73 |
| Slice 9 | 106,174,021 | 18,530,137 | 17.45 |
| Slice 10 | 9,530,436 | 1,166,222 | 12.24 |
| Total | 1,038,707,629 | 12,320,161 | 1.19 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.23 | 0.01 | 5.22 |
| Slice 2 | 0.02 | 0.01 | 51.59 |
| Slice 3 | 0.19 | 0.00 | 0.12 |
| Slice 4 | 3.01 | 0.21 | 6.93 |
| Slice 5 | 7.72 | 0.40 | 5.17 |
| Slice 6 | 17.71 | 0.66 | 3.73 |
| Slice 7 | 29.81 | 0.36 | 1.20 |
| Slice 8 | 30.19 | 0.14 | 0.45 |
| Slice 9 | 10.20 | 1.66 | 16.30 |
| Slice 10 | 0.92 | 0.10 | 11.07 |

[1] Average of two samples.

## Table D5
## Slice Data For FPL 6540-B Unaged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 4,786,560 | 309,040 | 6.46 |
| Slice 2 | 1,829,301 | 780,046 | 42.64 |
| Slice 3 | 4,737,317 | 1,010,282 | 21.33 |
| Slice 4 | 46,935,454 | 6,002,143 | 12.79 |
| Slice 5 | 99,613,494 | 18,029,710 | 18.10 |
| Slice 6 | 177,305,837 | 26,543,506 | 14.97 |
| Slice 7 | 294,857,804 | 1,556,443 | 0.53 |
| Slice 8 | 298,809,827 | 2,952,525 | 0.99 |
| Slice 9 | 140,684,461 | 90,698,234 | 64.47 |
| Slice 10 | 28,487,987 | 27,133,981 | 95.25 |
| Total | 1,098,048,042 | 167,910,228 | 15.29 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.44 | 0.05 | 11.02 |
| Slice 2 | 0.16 | 0.04 | 27.06 |
| Slice 3 | 0.43 | 0.02 | 5.73 |
| Slice 4 | 4.29 | 0.16 | 3.84 |
| Slice 5 | 9.04 | 0.30 | 3.37 |
| Slice 6 | 16.16 | 0.42 | 2.60 |
| Slice 7 | 27.45 | 3.93 | 14.32 |
| Slice 8 | 27.80 | 3.87 | 13.91 |
| Slice 9 | 11.92 | 5.88 | 49.32 |
| Slice 10 | 2.30 | 1.92 | 83.26 |

[1] Average of three samples.

## Table D6
## Slice Data For FPL 6540-B Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 7,158,865 | 1,824,294 | 25.48 |
| Slice 2 | 2,787,705 | 994,560 | 35.68 |
| Slice 3 | 7,972,461 | 2,061,622 | 25.86 |
| Slice 4 | 63,893,726 | 12,249,385 | 19.17 |
| Slice 5 | 120,543,073 | 20,027,996 | 16.61 |
| Slice 6 | 201,842,718 | 24,906,466 | 12.34 |
| Slice 7 | 288,720,211 | 4,919,084 | 1.70 |
| Slice 8 | 280,953,946 | 11,590,361 | 4.13 |
| Slice 9 | 70,667,470 | 23,923,628 | 33.85 |
| Slice 10 | 9,720,842 | 1,941,668 | 19.97 |
| Total | 1,054,261,015 | 103,599,160 | 9.83 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.67 | 0.10 | 15.58 |
| Slice 2 | 0.26 | 0.06 | 25.00 |
| Slice 3 | 0.74 | 0.11 | 15.37 |
| Slice 4 | 6.01 | 0.54 | 8.99 |
| Slice 5 | 11.36 | 0.75 | 6.60 |
| Slice 6 | 19.10 | 1.52 | 2.75 |
| Slice 7 | 27.59 | 2.11 | 7.65 |
| Slice 8 | 26.79 | 1.47 | 5.48 |
| Slice 9 | 6.55 | 1.53 | 23.36 |
| Slice 10 | 0.91 | 0.09 | 10.05 |

[1] Average of three samples.

**Table D7**
**Slice Data For FPL 6540-B Weather-O-Meter Aged Chromatograms**

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 5,225,328 | 309,058 | 5.91 |
| Slice 2 | 1,716,285 | 298,642 | 17.40 |
| Slice 3 | 3,475,049 | 323,334 | 9.30 |
| Slice 4 | 37,529,948 | 2,070,640 | 5.52 |
| Slice 5 | 80,177,554 | 4,813,827 | 6.00 |
| Slice 6 | 157,502,491 | 7,037,160 | 4.47 |
| Slice 7 | 285,325,807 | 2,843,825 | 1.00 |
| Slice 8 | 283,769,513 | 8,111,077 | 2.86 |
| Slice 9 | 58,302,523 | 19,720,927 | 33.83 |
| Slice 10 | 7,780,843 | 1,273,986 | 16.37 |
| Total | 920,805,340 | 38,625,482 | 4.19 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.57 | 0.04 | 6.33 |
| Slice 2 | 0.19 | 0.02 | 13.34 |
| Slice 3 | 0.38 | 0.04 | 9.77 |
| Slice 4 | 4.08 | 0.21 | 5.05 |
| Slice 5 | 8.70 | 0.19 | 2.23 |
| Slice 6 | 17.11 | 0.47 | 2.74 |
| Slice 7 | 31.04 | 1.34 | 4.33 |
| Slice 8 | 30.84 | 0.68 | 2.19 |
| Slice 9 | 6.26 | 1.84 | 29.38 |
| Slice 10 | 0.84 | 0.10 | 12.26 |

[1] Average of three samples.

**Table D8**
**Slice Data For FPL 6540-C Unaged Chromatograms**

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 9,329,386 | 1,972,585 | 21.14 |
| Slice 2 | 3,396,909 | 744,131 | 21.91 |
| Slice 3 | 6,976,865 | 1,067,382 | 15.30 |
| Slice 4 | 53,623,827 | 5,725,651 | 10.68 |
| Slice 5 | 97,068,025 | 9,904,747 | 10.20 |
| Slice 6 | 182,659,520 | 12,425,041 | 6.80 |
| Slice 7 | 276,331,744 | 2,647,502 | 0.96 |
| Slice 8 | 264,097,015 | 5,070,600 | 1.92 |
| Slice 9 | 48,412,318 | 5,022,278 | 10.37 |
| Slice 10 | 7,314,935 | 424,754 | 5.81 |
| Total | 949,210,542 | 44,413,291 | 4.68 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.98 | 0.16 | 16.31 |
| Slice 2 | 0.36 | 0.06 | 17.76 |
| Slice 3 | 0.73 | 0.08 | 10.69 |
| Slice 4 | 5.63 | 0.33 | 5.89 |
| Slice 5 | 10.20 | 0.56 | 5.44 |
| Slice 6 | 19.22 | 0.40 | 2.09 |
| Slice 7 | 29.16 | 1.09 | 3.72 |
| Slice 8 | 27.86 | 0.75 | 2.71 |
| Slice 9 | 5.09 | 0.29 | 5.76 |
| Slice 10 | 0.77 | 0.02 | 2.83 |

[1] Average of four samples.

## Table D9
## Slice Data For FPL 6540-C Weather-O-Meter Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 11,725,847 | 1,965,621 | 16.76 |
| Slice 2 | 6,966,293 | 1,980,202 | 28.43 |
| Slice 3 | 7,767,650 | 1,290,387 | 16.61 |
| Slice 4 | 52,367,745 | 1,222,985 | 2.34 |
| Slice 5 | 100,312,470 | 950,335 | 0.95 |
| Slice 6 | 174,992,382 | 513,448 | 0.29 |
| Slice 7 | 277,250,231 | 335,427 | 0.12 |
| Slice 8 | 277,275,740 | 1,337,208 | 0.48 |
| Slice 9 | 62,660,162 | 2,991,357 | 4.77 |
| Slice 10 | 9,218,061 | 794,985 | 8.62 |
| Total | 490,268,289 | 490,359,596 | 100.00 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 1.19 | 0.18 | 15.43 |
| Slice 2 | 0.71 | 0.19 | 27.17 |
| Slice 3 | 0.79 | 0.12 | 15.28 |
| Slice 4 | 5.34 | 0.05 | 0.97 |
| Slice 5 | 10.23 | 0.04 | 0.42 |
| Slice 6 | 17.85 | 0.19 | 1.07 |
| Slice 7 | 28.28 | 0.35 | 1.24 |
| Slice 8 | 28.28 | 0.25 | 0.88 |
| Slice 9 | 6.39 | 0.22 | 3.41 |
| Slice 10 | 0.94 | 0.07 | 7.27 |

[1] Average of two samples.

## Table D10
## Slice Data For FPL 6540-D Oven-Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,714,722 | NA | NA |
| Slice 2 | 1,896,929 | NA | NA |
| Slice 3 | 2,740,251 | NA | NA |
| Slice 4 | 24,891,045 | NA | NA |
| Slice 5 | 55,022,724 | NA | NA |
| Slice 6 | 119,672,066 | NA | NA |
| Slice 7 | 250,535,081 | NA | NA |
| Slice 8 | 246,640,496 | NA | NA |
| Slice 9 | 35,182,056 | NA | NA |
| Slice 10 | 3,795,935 | NA | NA |
| Total | 743,091,305 | NA | NA |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.62 | NA | NA |
| Slice 2 | 0.18 | NA | NA |
| Slice 3 | 0.41 | NA | NA |
| Slice 4 | 4.35 | NA | NA |
| Slice 5 | 7.92 | NA | NA |
| Slice 6 | 15.42 | NA | NA |
| Slice 7 | 27.09 | NA | NA |
| Slice 8 | 26.58 | NA | NA |
| Slice 9 | 13.49 | NA | NA |
| Slice 10 | 3.95 | NA | NA |

[1] Only one sample used.

## Table D11
## Slice Data For FPL 6540-D Weather-O-Meter Aged Chromatograms

| Slice Number | Average Area[1] (microvolt-sec) | Standard Deviation | Coefficient of Variance (Percent) |
|---|---|---|---|
| Slice 1 | 2,757,851 | 624,458 | 22.64 |
| Slice 2 | 1,954,585 | 353,008 | 18.06 |
| Slice 3 | 2,787,185 | 634,719 | 22.77 |
| Slice 4 | 20,275,711 | 3,777,569 | 18.63 |
| Slice 5 | 43,163,237 | 6,862,392 | 15.90 |
| Slice 6 | 90,474,815 | 13,634,286 | 15.07 |
| Slice 7 | 250,963,540 | 19,455,198 | 7.75 |
| Slice 8 | 274,789,070 | 6,528,027 | 2.38 |
| Slice 9 | 29,049,143 | 3,229,410 | 11.12 |
| Slice 10 | 7,203,145 | 3,140,733 | 43.60 |
| Total | 723,418,280 | 52,552,236 | 7.26 |
| **Slice Number** | **Average Area (Percent)** | **Standard Deviation** | **Coefficient of Variance (Percent)** |
| Slice 1 | 0.38 | 0.06 | 16.78 |
| Slice 2 | 0.27 | 0.03 | 11.82 |
| Slice 3 | 0.38 | 0.07 | 17.11 |
| Slice 4 | 2.78 | 0.35 | 12.60 |
| Slice 5 | 5.93 | 0.56 | 9.49 |
| Slice 6 | 12.43 | 1.11 | 8.94 |
| Slice 7 | 34.69 | 0.85 | 2.44 |
| Slice 8 | 38.15 | 2.42 | 6.34 |
| Slice 9 | 4.01 | 0.22 | 5.58 |
| Slice 10 | 0.99 | 0.39 | 39.34 |

[1] Average of four samples.

## Table D12
**Percent Average Area Per Slice for FPL 6540 As-Received and Unaged Chromatograms**

| Slice Number | As-Received | Heating Time | | | |
|---|---|---|---|---|---|
| | | A | B | C | D |
| Slice 1 | 0.00 | 0.32 | 0.44 | 0.98 | • |
| Slice 2 | 0.00 | 0.14 | 0.16 | 0.36 | • |
| Slice 3 | 0.55 | 0.56 | 0.43 | 0.73 | • |
| Slice 4 | 4.26 | 4.31 | 4.29 | 5.63 | • |
| Slice 5 | 8.03 | 8.59 | 9.04 | 10.20 | • |
| Slice 6 | 22.28 | 16.16 | 16.16 | 19.22 | • |
| Slice 7 | 36.21 | 32.24 | 27.45 | 29.16 | • |
| Slice 8 | 25.47 | 31.80 | 27.80 | 27.86 | • |
| Slice 9 | 2.64 | 5.15 | 11.92 | 5.09 | • |
| Slice 10 | 0.55 | 0.72 | 2.30 | 0.77 | • |

[1] Slice data not available.

## Table D13
**Percent Average Area Per Slice For FPL 6540 Oven-Aged Chromatograms**

| Slice Number | Heating Time | | | |
|---|---|---|---|---|
| | A | B | C | D |
| Slice 1 | 0.46 | 0.67 | • | 0.62 |
| Slice 2 | 0.15 | 0.26 | • | 0.18 |
| Slice 3 | 0.46 | 0.74 | • | 0.41 |
| Slice 4 | 4.31 | 6.01 | • | 4.35 |
| Slice 5 | 8.64 | 11.36 | • | 7.92 |
| Slice 6 | 15.68 | 19.10 | • | 15.42 |
| Slice 7 | 27.70 | 27.59 | • | 27.09 |
| Slice 8 | 29.06 | 26.79 | • | 26.58 |
| Slice 9 | 12.13 | 6.55 | • | 13.49 |
| Slice 10 | 1.41 | 0.91 | • | 3.95 |

[1] Slice data not available.

**Table D14**
**Percent Average Area Per Slice For FPL 6540 Weather-O-Meter Aged Chromatograms**

| Slice Number | Heating Time | | | |
|---|---|---|---|---|
| | A | B | C | D |
| Slice 1 | 0.23 | 0.57 | 1.19 | 0.38 |
| Slice 2 | 0.02 | 0.19 | 0.71 | 0.27 |
| Slice 3 | 0.19 | 0.38 | 0.79 | 0.38 |
| Slice 4 | 3.01 | 4.08 | 5.34 | 2.78 |
| Slice 5 | 7.72 | 8.70 | 10.23 | 5.93 |
| Slice 6 | 17.71 | 17.11 | 17.85 | 12.43 |
| Slice 7 | 29.81 | 31.04 | 28.28 | 34.69 |
| Slice 8 | 30.19 | 30.84 | 28.28 | 38.15 |
| Slice 9 | 10.20 | 6.26 | 6.39 | 4.01 |
| Slice 10 | 0.92 | 0.84 | 0.94 | 0.99 |

# Appendix E
# Analyzing Chromatograms

The work conducted on asphalt cements using GPC has not answered many questions and often the answers that have been provided are contradictory (Brule 1980). One of the main reasons for the contradictory results could be that the molecular types and sizes vary widely with chain and ring shapes found in asphalt cements. Therefore, it is very difficult, if not impossible, to develop a consistent relationship between molecular size and molecular weight (Stock 1986). But if the chromatograms obtained from asphalt cements are analyzed using a comparative analysis, the actual molecular weight is not as important as the differences between different chromatograms. Therefore, a method to systematically analyze the chromatograms is needed. It would also be desirable that the systematic evaluation method be extrapolated to predict the field performance of the material being analyzed (Price and Burati 1989).

One of the most common methods of analyzing asphalt cement chromatograms is to divide them into three sections or partitions (Stock 1986, Price 1988, Leite et al 1989, Jennings 1985). The partitions are normally established by dividing the chromatogram into equal sections based on elution time. The area in each section is then calculated by slice integration.

The labeling or terminology used to classify the three partitions has also been standardized. The section which elutes first has traditionally been referred to as the large molecular size fraction, the second fraction as the intermediate or medium molecular size fraction, and the last fraction as the small molecular size fraction. However, since the solvent may not separate all of the aggregates and micelles in an asphalt, it is probable that some of the material that elutes in the large or intermediate size molecular fraction is actually a grouping of small molecules. A more appropriate method of labeling these sections would be to eliminate the molecular size wording and replace it with early fraction for the portion of the chromatogram that elutes as the large molecular size, intermediate fraction for intermediate molecular size, and late fraction for small molecular size (Stock 1986). Figure 3 illustrates a chromatogram which has been divided into three partitions and the terminology used to describe the sections. Early, intermediate, and late fractions will be used to describe portions of chromatograms for the joint sealant analysis.

A second possibility for the contradictory results sometimes obtained when analyzing the chromatograms could be that it was only divided into three partitions. Price (1988) performed studies in which the chromatograms were first divided into thirds as described by earlier researchers (Figure E1). The chromatograms were then reevaluated by dividing them into fourths and tenths (Figure E2). By dividing the chromatograms into fourths, the asphaltene fraction of the asphalt cement was better approximated by the upper fourth or early fraction. Dividing the chromatograms into tenths provided better resolution for analysis and thus provided an improved statistical correlation with physical data.

Each partition of the chromatogram was divided by the total area under the curve to normalize the comparisons of one chromatogram with another. The chromatograms that were partitioned into tenths indicated that different sections correlate to different rheological tests such as specific gravity, kinematic viscosity, thin film oven loss, pen-vis number, viscosity-temperature susceptibility, and kinetic viscosity of thin-film oven residue (Price and Burati 1989). The equations obtained from the analysis were validated using a different asphalt cement (AC-20). In five of the six models tested, the predicted values were within 10 percent of the actual value. This strengthens the hypothesis that HP-GPC characterization of asphalt can be used to predict performance in physical properties regardless of source or crude oil origin (Price and Burati 1989). The joint sealant chromatograms in this study will be analyzed by determining the initial detection and final detection and partitioning them into tenths to provide better resolution.

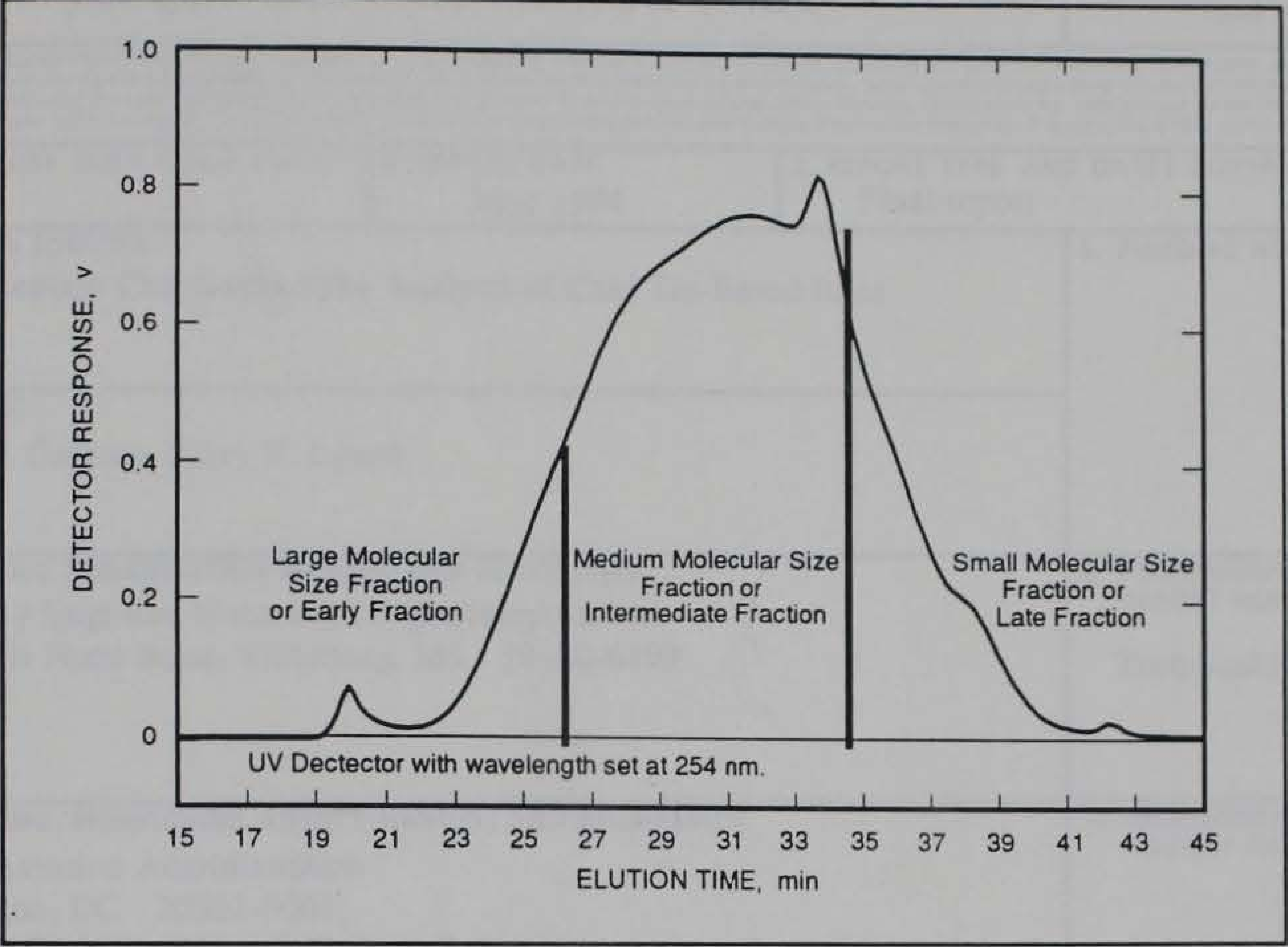


**Figure E1.** Chromatogram partitioned into thirds with descriptive terminology

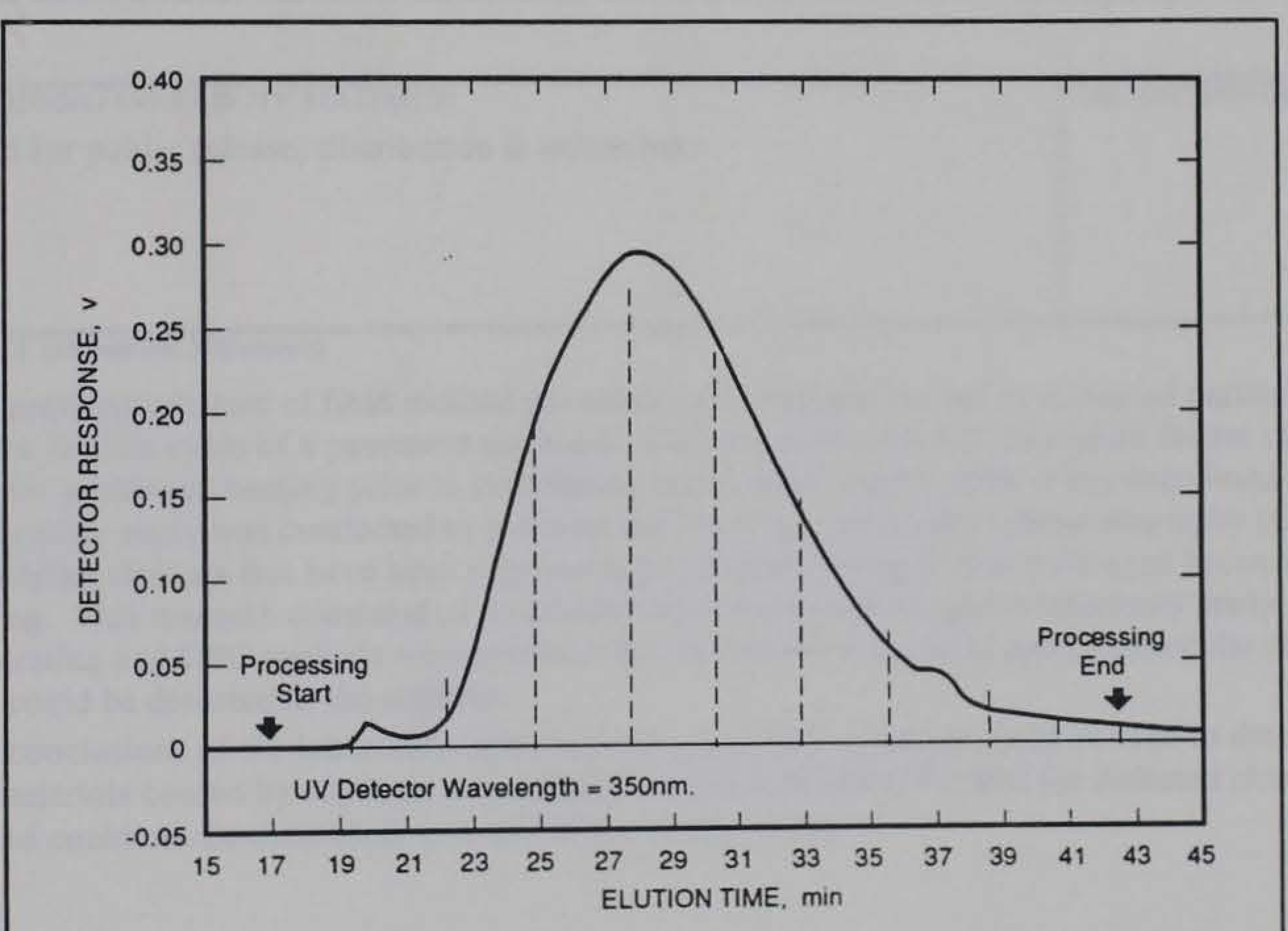


**Figure E2.** Chromatogram partitioned into tenths

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The premature failure of field molded pavement joint sealants has led to increased maintenance cost and can reduce the life cycle of a pavement structure. Two possible causes of premature failure of pavement joint sealants are prolonged heating prior to installation and excessive aging after it has been installed into the joint. This laboratory study was conducted to evaluate the use of gel permeation chromatography (GPC) as a method for identifying sealants that have been exposed to prolonged heating or that have aged because of natural weathering. This research consisted of a literature review and a three-phase laboratory study. Material specification testing and GPC analysis were conducted to determine if physical and/or molecular size distribution changes could be detected in the sealants.

The conclusions of the laboratory study indicated that GPC analysis could be used to detect changes in sealant materials caused by exposure to extended heating and aging, but that the detected changes were inconsistent and could not be correlated with physical test properties.

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