

MILLENNIUM APPLICATION BRIEF

TOPIC: Paraquat, Diquat Analysis Using PDA and Symmetry

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Date: 9/2/94

Introduction:

Analysis of paraquat chloride and diquat dibromide currently analyzed using EPA method 549.1 was done using photodiode array (PDA) detection and a symmetry C₁₈ column. The current method specifies the use of a PDA since the HPLC method provided does not fully resolve all of the peaks. Since each of the two components has a different lambda max (paraquat = 257nm, and diquat = 308) monitoring at two different wavelengths is required for quantitation. The method uses a C₁₈ column and a mobile phase containing diethylamine (DEA) to maintain good peak shape. Several different methods have been reported and are used to analyze for paraquat and diquat but all use some sort of amine in the mobile phase. A symmetry column was used for this analysis since it has the advantage of not needing any amine modifier to prevent tailing. Due to its high level of retention for most analytes a 50 mm column was used as well. The column was heated above room temperature so as to provide a rapid and reproducible analysis. Run times for both analytes were less than 12 minutes. A high (figure 1) and low (figure 2) standard were run; spectrum index plots for both are shown as well in figures 3 and 4. The overlaid spectra of each component at the high and low level are shown in figures 5 and 6. As seen the spectra do match even with the low sample at an absorbance of less than 0.0015 AU.

EXPERIMENTAL:

| | |
|---------------|--|
| System: | 600 Pump w/ column heater, 717+ autosampler, 996 PDA |
| Mobile phase: | Pic B-7, MeOH, pH = 3.0 |
| Flow Rate: | 1.0 mL/min |
| Column: | Symmetry C ₁₈ 3.9 X 50 mm |
| Detection: | 230-350 |
| Data: | Millennium 2010 Chromatography Manager, Ver. 2.01 |

FIGURES:

- Figure 1: Maxplot chromatogram of high standard (5 ppm)
- Figure 2: Maxplot chromatogram of low standard (25 ppb)
- Figure 3: Spectrum Index plot of high standard
- Figure 4: Spectrum Index plot of low standard
- Figure 5: Overlaid spectra for high and low std of paraquat
- Figure 6: Overlaid spectra for high and low std of diquat

For Sample: PD Std A-10uL Vial: 2 Inj: 1 Chan: 996

Date Processed 09/02/94 11:50 AM

Channel Descr: PDA MaxPlot(240.0 nm to 350.0 nm)

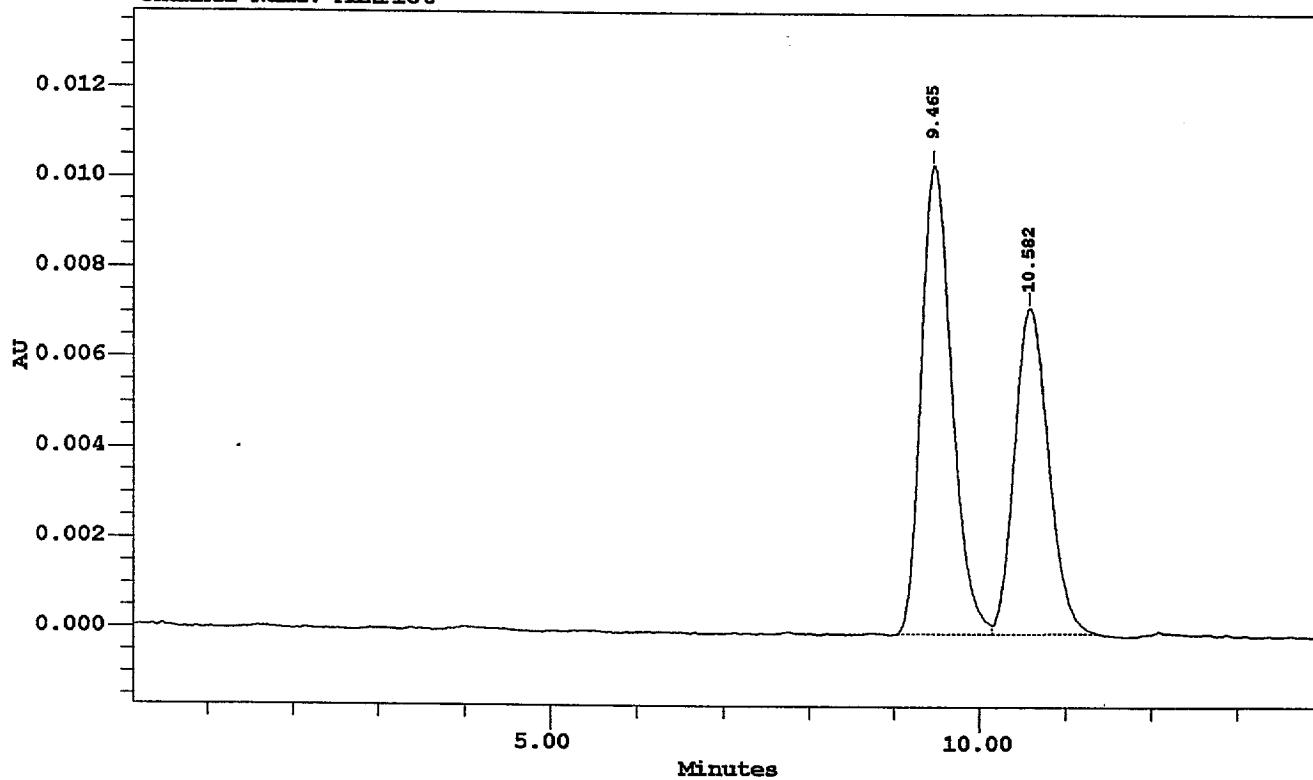
Northern Kentucky Laboratory

Sample Information

Project Name: Enviro_01
 SampleName: PD Std A-10uL
 Vial: 2
 Injection: 1
 Channel: 996
 Date Acquired: 08/05/94 05:40 PM
 Scale Factor: 1.00
 Acq Meth Set: Paraquat_Diquat01b
 Processing Method: Paraquat_Diquat01

Sample Type: Standard
 Volume: 10.00
 Run Time: 14.0 min
 Date Processed: 09/02/94 11:50 AM
 Dilution: 1.00000

SampleName: PD Std A-10uL Comments: Symmetry 50mm-Pic B7/MeOH-pH=3.0, 30C
 Channel Name: MaxPlot



Peak Data

| # | Retention Time (min) | Name | Height (uV) | Area (uV*sec) | Amount-ppb |
|---|----------------------|----------|-------------|---------------|------------|
| 1 | 9.465 | Paraquat | 10420 | 261483 | 5000.000 |
| 2 | 10.582 | Diquat | 7249 | 200818 | 5000.000 |

fig. 1

Millennium Results Report: Chrom_02

Proc Chan: MaxPlot

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For Sample: PD Std2-200uL Vial: 4 Inj: 1 Chan: 996

Date Processed 09/02/94 10:02 AM

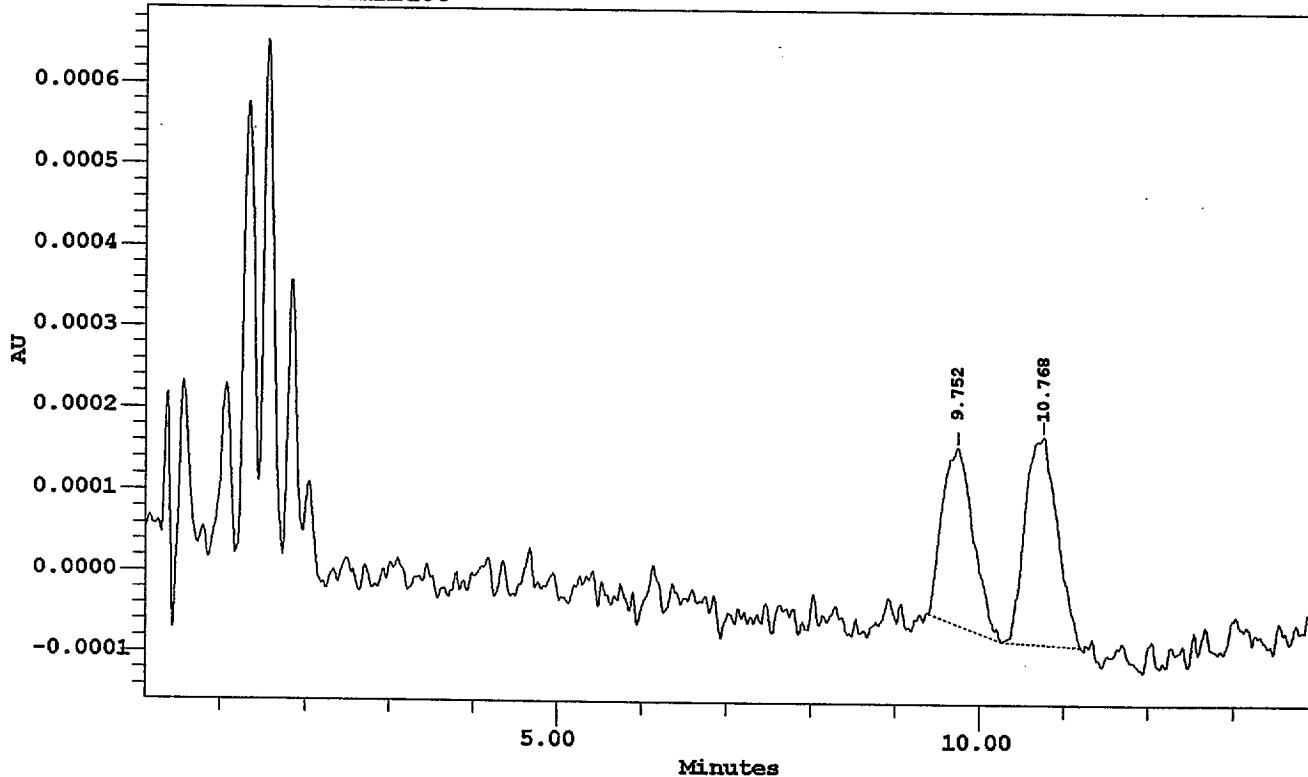
Channel Descr: PDA MaxPlot(240.0 nm to 350.0 nm)

N o r t h e r n K e n t u c k y L a b o r a t o r y
S a m p l e I n f o r m a t i o n

Project Name: Enviro_01
SampleName: PD Std2-200uL
Vial: 4
Injection: 1
Channel: 996
Date Acquired: 08/05/94 05:25 PM
Scale Factor: 1.00
Acq Meth Set: Paraquat_Diquat01b
Processing Method: Paraquat_Diquat01s

Sample Type: Standard
Volume: 200.00
Run Time: 14.0 min
Date Processed: 09/02/94 10:02 AM
Dilution: 1.00000

SampleName: PD Std2-200uL Comments: Symmetry 50mm-Pic B7/MeOH-pH=3.0, 30C
Channel Name: MaxPlot



Peak Data

| # | Retention Time (min) | Name | Height (uV) | Area (uV*sec) | Amount-ppb |
|---|----------------------|----------|-------------|---------------|------------|
| 1 | 9.752 | Paraquat | 220 | 5664 | 25.000 |
| 2 | 10.768 | Diquat | 256 | 6844 | 25.000 |

Fig. 2

Millennium Results Report: Spectrum_Index

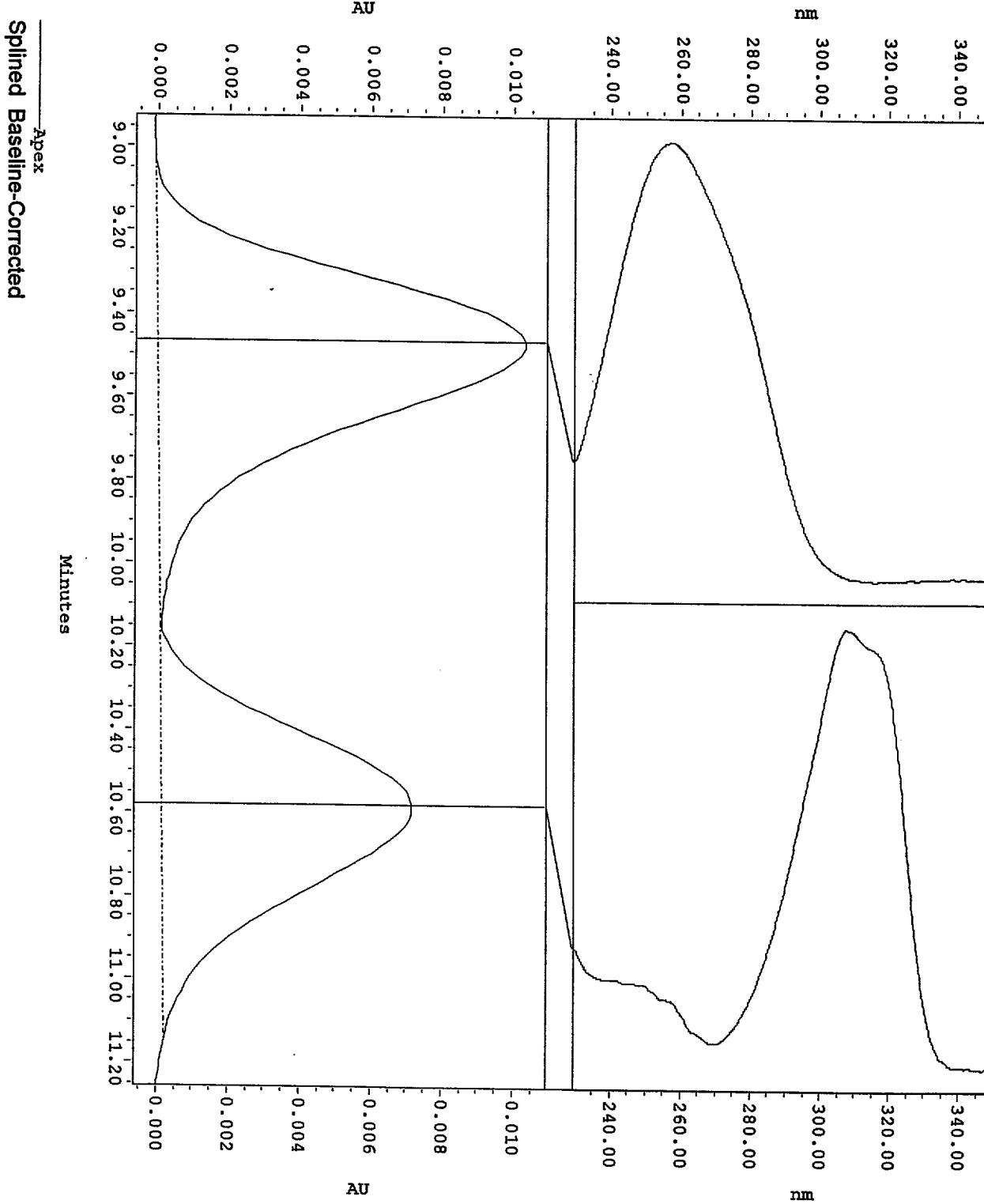
Proc Chan: MaxPlot

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For Sample: PD Std A-10uL Vial: 2 Inj: 1 Chan: 996

Date Processed 09/02/94 09:55 AM

Channel Descr: PDA MaxPlot(240.0 nm to 350.0 nm)



Millennium Results Report: Spectrum_Index

Proc Chan: MaxPlot

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For Sample: PD Std2-200uL Vial: 4 Inj: 1 Chan: 996

Date Processed 09/02/94 10:02 AM

Channel Descr: PDA MaxPlot(240.0 nm to 350.0 nm)

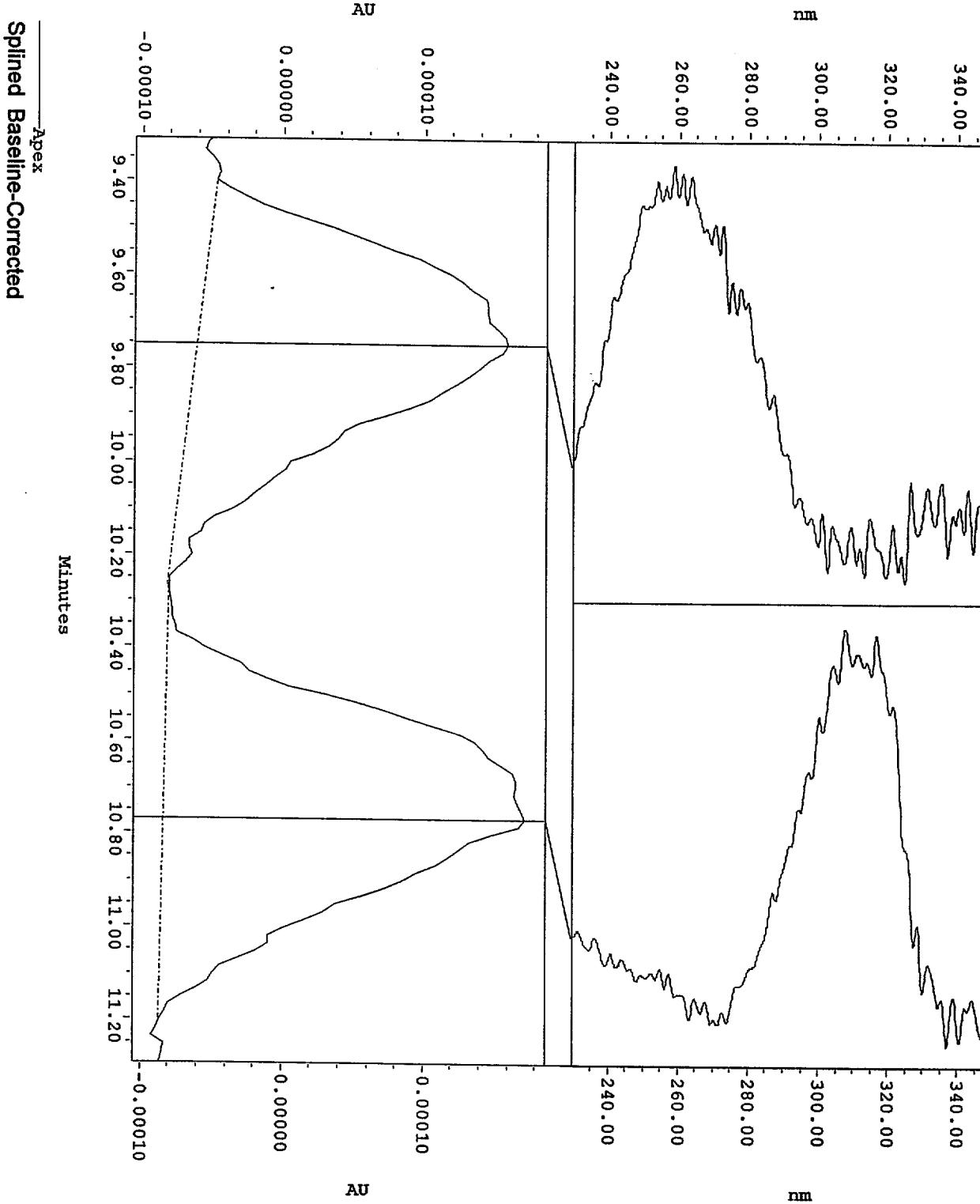
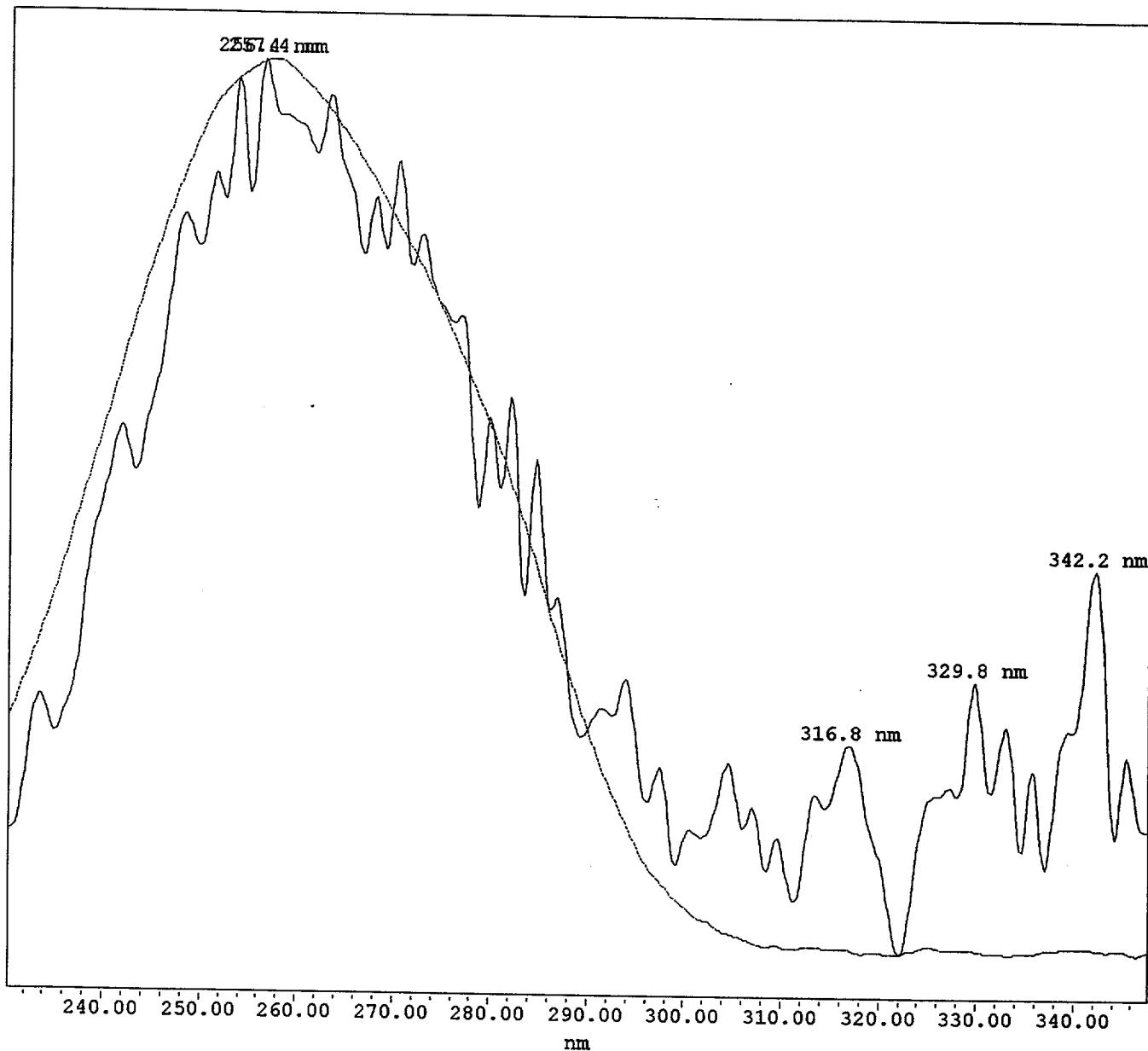


Fig. 4

Millennium Spectrum Review Report

Millennium v2.00

Date Printed: 9:30:01 am, September 6, 1994



— 9.706 minutes, 230 - 350 @ 1.2 nm, from SampleName PD Std2-200uL, Vial 4, Inj. 1
- - - Paraquat 9.496 minutes, 230 - 350 @ 1.2 nm, from SampleName PD Std A-10uL, Vial 2, Inj. 1

② fig 5

Millennium Spectrum Review Report

Millennium v2.00

Date Printed: 9:30:26 am, September 6, 1994

Spectral Table

| # | Retention Time | Lambda Max | Maximum Absorbance | Start Wvln | End Wvln | Smooth | Derivative | Spline | Traceable |
|---|----------------|------------|--------------------|------------|----------|--------|------------|--------|-----------|
| 1 | 9.706 | 256.4 | 0.000 | 230 | 350 | None | None | On | Yes |
| 2 | 9.496 | 257.4 | 0.010 | 230 | 350 | None | None | On | Yes |

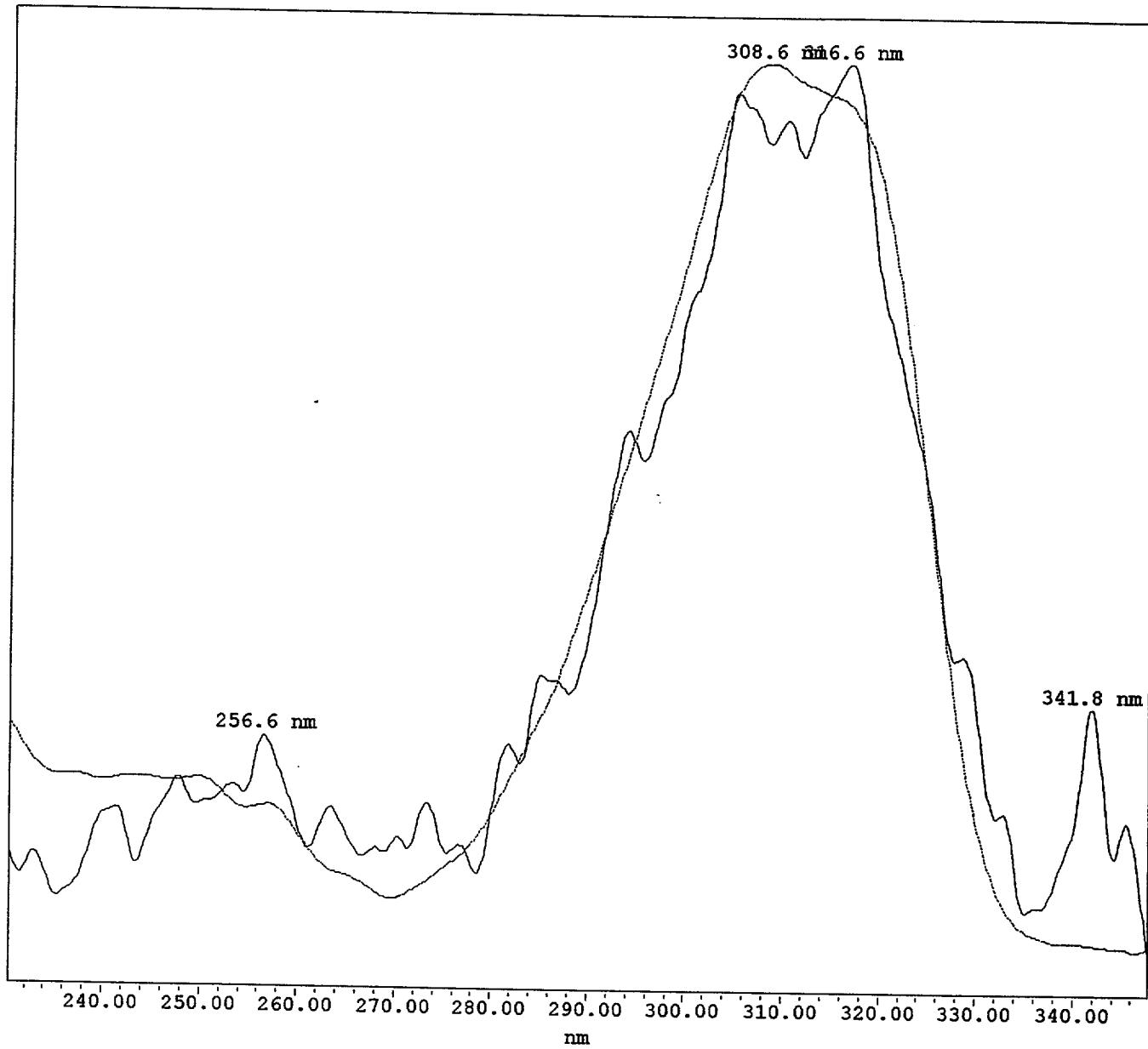
Spectral Table

| # | Searchable | Resolution | Baseline Correct |
|---|------------|------------|------------------|
| 1 | No | 1.2 | No |
| 2 | No | 1.2 | No |

Millennium Spectrum Review Report

Millennium v2.00

Date Printed: 9:28:09 am, September 6, 1994



Millennium Spectrum Review Report

Millennium v2.00

Date Printed: 9:28:33 am, September 6, 1994

Spectral Table

| # | Retention Time | Lambda Max | Maximum Absorbance | Start Wvln | End Wvln | Smooth | Derivative | Spline | Traceable |
|---|----------------|------------|--------------------|------------|----------|--------|------------|--------|-----------|
| 1 | 10.718 | 316.6 | 0.000 | 230 | 350 | 5 | None | On | Yes |
| 2 | 10.579 | 308.6 | 0.007 | 230 | 350 | 5 | None | On | Yes |

Spectral Table

| # | Searchable | Resolution | Baseline Correct |
|---|------------|------------|------------------|
| 1 | No | 1.2 | No |
| 2 | No | 1.2 | No |