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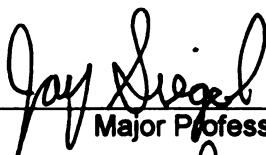
**UV MICROSPECTROPHOTOMETRY OF FIBERS FROM
APPARENTLY WHITE TEXTILES ENCOUNTERED IN
FORENSIC CASEWORK**

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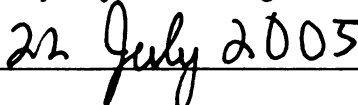
ERIN CUNNANE FARR

has been accepted towards fulfillment
of the requirements for the

**Master of Science degree in Criminal Justice
with a specialization in
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**UV MICROSPECTROPHOTOMETRY OF FIBERS FROM APPARENTLY WHITE
TEXTILES ENCOUNTERED IN FORENSIC CASEWORK**

By

Erin Cunnane Farr

A THESIS

**Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of**

MASTER OF SCIENCE

Department of Criminal Justice

2005

ABSTRACT

UV MICROSPECTROPHOTOMETRY OF FIBERS FROM APPARENTLY WHITE TEXTILES ENCOUNTERED IN FORENSIC CASEWORK

By

Erin Cunnane Farr

The purpose of this study is to determine whether similar looking undyed fibers can be differentiated by microspectrophotometry. Several classes of undyed fibers will be analyzed independently, and their UV transmittance properties will be examined.

Undyed fibers are very commonly encountered by forensic fiber examiners. Identification of the undyed fiber is fairly routine, but the question becomes how to differentiate between fibers within a class, such as polyester or cotton, for example. One possibility is that optical brighteners may play an important role in distinguishing fibers, similar to the way textile dyes are used to differentiate dyed fibers. Textile dyes can be differentiated through various analytical techniques, and a UV-visible microspectrophotometer is but one example. A microspectrophotometer allows the forensic scientist to measure the transmission, reflectance, or fluorescence characteristics of fibers.

Optical brighteners are often used on fibers during the manufacturing process or acquired through commercial detergents. Perhaps these optical brighteners will cause similar looking fibers, originating from different sources, to behave differently when exposed to electromagnetic radiation.

To my father, Jim Cunnane,
who has been constant support throughout my education,
and who taught me never to forget what the solution is:
the value of the variable that makes the equation true.
Here is the solution to my master's degree.
Thank you for all of your help along the way.

ACKNOWLEDGMENTS

Throughout this project I have received assistance and direction in various forms from many individuals. A special thanks to Larry Peterson and Tammy Jergovich, as well as other members of the Georgia Bureau of Identification Trace Evidence Section. I would also like to thank my advisor at Michigan State University, Dr. Jay Siegel, who accepted me into his program and helped me fulfill my dream of becoming a forensic scientist. A generous thank you to my parents who helped me get where I am today, with their continuous support and encouragement—it meant the world to me. To my husband, Matt, who believed in me in the midst of my frequent procrastination, and to all of my friends who have encouraged me throughout the progression of this project, I am very grateful!

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INTRODUCTION

The importance of forensic fiber analysis relies heavily upon an examiner's ability to establish associations between fibers. Fibers are submitted to the laboratory in the form of both questioned and known. Questioned fibers are collected by an investigator, and they are typically recovered from, but certainly not limited to, either a victim of a crime, a crime scene, or even an object such as a weapon. Known fibers are typically collected from a person who is believed to be involved in a crime, or from that person's textile environment. The forensic scientist will examine the questioned fibers and the known fibers to see if they can establish any associations between these samples. The general approach to the examination will consist of both identification and comparison of the questioned and known fiber samples.

Identification of Fibers

Identification of fibers in a forensic examination is a twofold process. First, the examiner must ascertain the type of fiber with which they are dealing. Second, they must identify as many significant characteristics as possible, such as the color, morphology, and diameter of the fiber, along with surface detail, amount of delustrant, and any other distinguishing characteristics. The examiner will generally start with a microscopic examination, looking for physical features that are characteristic of a particular fiber type. In addition, it is often helpful to compare the questioned fibers to standards from a reference collection. Generally, natural fibers will show more identifying features through bright field microscopy than will manufactured fibers. This is because natural fibers are highly variable whereas manufactured fibers are much more uniform. Thus,

polarized-light microscopy is used to reveal optical properties (i.e. refractive index, sign of elongation, birefringence) that are useful for the identification of manufactured fibers.^{1,2} These optical properties are sufficient for the identification of several synthetic fiber types, whereas other types may need further analysis. If need be, the examiner has many additional techniques (such as infrared spectroscopy, cross-section analysis, hot-stage microscopy, pyrolysis gas chromatography, and solubility tests) available for the purpose of aiding in the identification of the fiber.

Comparison of Fibers

Comparison of fibers is somewhat more involved than identification, because the examiner must examine questioned fibers and known fibers in a case until significant differences are found, or until all available tests are exhausted. As the number of associations between the two fiber samples increases, the more likely the possibility that they originated from the same source. Gaudette says that “if, after conducting several types of comparative examinations and looking at a large number of comparison characteristics, no significant differences between the questioned fiber and the known sample are found, they are said to be consistent with having had a common origin.”³ Several techniques are available to the fiber examiner for comparison purposes. The fiber type (or identification of the fiber) is the first comparison feature between questioned and known samples. If the fibers are determined to be of the same type, other microscopical characteristics should be compared with a comparison microscope. As mentioned above, microscopic differences can be found in various forms such as color, morphology, and diameter of the fiber, along with surface detail and amount of delustrant.¹ These differences are important because they can lead to further

differentiation within one specific class of fibers, allowing an examiner to say, for example, that one polyester fiber is different from another polyester fiber. Additional tests such as FTIR microscopy, UV-visible microspectrophotometry, dye analysis by thin-layer chromatography, and tests that reveal optical and fluorescent properties of the fibers are available to the examiner. It is important to remember that not all of these techniques need to be used in every case. The examiner must consider the instrumentation available combined with the discriminating power of the techniques used, to try to obtain as much comparative information as possible.

The Importance of Color

The color of the fiber is believed to be a very important and distinguishing characteristic of a fiber.⁴ There is a vast array of colors that are produced by textile dye manufacturers. After a fiber is dyed with a unique dye formula, it has a special chemical feature that becomes a foundation for distinguishing it from many other fibers within its same class.

Because of its sensitivity, the human eye is very good at evaluating color differences during the initial stages of a fiber examination. It has the ability to detect very slight differences in shades of the same color from sample to sample. Thus, the eye becomes a useful tool for both visually screening samples and for the side by side examinations of fibers under a comparison microscope. However, during later stages of a fiber examination the human eye is not as reliable due to subjectivity of the examiner; each influenced by personal opinion and experience.^{4,5,6} Furthermore, many textile dyes have colors that appear visually indistinguishable, but are made up of entirely different chemical formulas. This causes some hues of textile dyes to appear to be identical under

one set of lighting conditions, but very different under other conditions. For example, an examiner may find that fibers from two different garments appear to be similar in color when viewed through a comparison microscope; however, the UV transmittance data from the same two fibers will show different characteristics. This phenomenon is known as metamerism.⁶ Spectroscopic analysis is a quick and easy way to prevent metamerism from going undetected.

Textile dyes are complex mixtures—their chemical compositions become important when the colors are indistinguishable with the naked eye. A microspectrophotometer is an instrument that has the capability of detecting these differences in chemical composition with a degree of objectivity; thereby eliminating the subjectivity of the human eye and identifying any possibility of metamerism.

Microspectrophotometry

The method of microspectrophotometry (MSP) has been used for more than seventy years, and has been used in forensic applications for more than forty.⁷ MSP has been used for a variety of microscopic samples (such as biological samples, crystals and minerals, inks, microchips, paints, fibers and explosives) within a wide range of disciplines (such as biochemical studies, geology, document analysis, the semiconductor industry, and forensic trace evidence).⁸

Microspectrophotometry is one method of UV spectroscopy. A microspectrophotometer is essentially a spectrophotometer adapted with microscopic capabilities. When a sample is too small to be analyzed through ordinary spectroscopic analysis, it can usually be analyzed with a microspectrophotometer. As electromagnetic radiation interacts with a microscopic sample, some of the light is absorbed by the

sample, and the rest is emitted to the spectrophotometer. The instrument measures the change in light intensities, relative to wavelength.^{6,7,8} The light intensity will be measured at each wavelength over the ultra-violet (UV), visible (vis), and/or near infrared (NIR) regions. The plotted data will reflect the transmission, reflectance, or fluorescent light properties of these samples. As electromagnetic radiation interacts with the dyed fiber, excitation of electrons occurs in the UV region, making UV-vis MSP particularly useful in resolving the phenomena of metamerism in dyed fibers.⁷

Microspectrophotometry is convenient for forensic scientists because it is non-destructive, quick, and requires very little sample preparation. For example, it is possible to collect data from fiber samples without even removing the samples from the microscope slide. The slide is placed on the microscope stage, and that in turn becomes the sample compartment of the spectrophotometer.

Undyed Fibers

Many evidentiary fibers are undyed. This limits the usefulness of microspectrophotometry. Although the scientist can identify the type of fiber present, there are limitations to finding significant differences between fibers of the same class if no identifying textile dyes are present. Previous studies have shown that fibers treated with chemicals such as optical brighteners and bleaching agents will show different spectral characteristics in the UV range, even if not discriminated in the visible range.^{4,9,10} The purpose of this research is to simulate real casework by analyzing undyed fibers obtained from clothing samples associated with actual cases. The samples will be analyzed in the UV range with a microspectrophotometer, and the focus will be to see if the differences in UV spectra within groupings of fibers (cotton, polyester, nylon, rayon,

and acetate) are statistically different. The objective of this work is threefold. First, the UV microspectral response (transmittance in the approximate range of 250nm to 475nm, where nm = nanometers) of undyed fibers will be examined, and the data will be collected. Second, the data will be categorized by visual analysis as well as statistical analysis based on the observed response of the fibers. And third, it will be determined whether the transmittance data in the range examined reveals enough information for evaluating similarities/dissimilarities of fibers to be useful for forensic purposes. This is a preliminary, exploratory study, and by no means all-inclusive. It is simply a process of discovery to see what features are in the data.

REVIEW OF LITERATURE

In the field of forensic fiber analysis, there has been little investigation into the analysis of ultraviolet spectral characteristics of undyed fibers using a microspectrophotometer. A few studies, to date, have researched the ultraviolet spectral characteristics of fibers at different stages of production or at different stages of chemical bleaching. This study, on the contrary, has many uncontrolled variables such as the number of times the garment was washed, the detergents used, and the treatment of the fibers during manufacturing. Thus, this research is an exploratory study of the effects of chemical treatments (such as detergents or optical brighteners) on undyed fibers, along with an attempt to simulate real casework samples.

In one previous study, Martin used an S.E.E.2100 microspectrometer to analyze the ultraviolet characteristics of wool fibers at several different stages of the chemical bleaching and/or dyeing process.⁹ The purpose was to determine what kind of spectral characteristics are imparted by the fiber structure, as well as the dyes. Six wool samples, each with different chemical treatments, were tested to see what effects the chemical treatments would have on the absorption spectra. The first sample was untreated wool, the second, third and fourth samples were treated with different bleach solutions for various times and/or pHs. The fifth sample was dyed, and the sixth sample was dyed then bleached. None of the samples showed any meaningful data in the visible range spectrum, but all of the samples showed notable differences in their UV spectra, allowing for separation of the samples based on treatment. Thus, Martin concludes that “not only can the SEE 2100 microspectrometer detect changes in dyes in fibers, it can also detect changes in the fibers themselves when they are treated and their structure changes.”⁹

In another previous study, Desrosiers and Martin used an S.E.E.2100 microspectrometer to analyze the ultraviolet characteristics of nylon fibers at different stages of production.¹⁰ Again, the purpose was to determine what kind of spectral characteristics are imparted by the fiber structure, as well as the dyes. In this study, four samples of undyed fibers were treated with different chemicals and analyzed with the microspectrometer to determine the effects that the chemical treatments have on the transmission spectra. Some of the samples were difficult to distinguish when looking at the visible through short-wave NIR regions because they showed little difference in the peak positions. However, the UV spectra of these same samples showed very different spectral characteristics, allowing for clear distinction.

Thus, it is clear that analysis of some fibers in the ultraviolet region has some advantages. The goal of this study is to determine whether these previous findings are confirmed with undyed fibers when simulating real casework samples; and furthermore, if it would be advantageous to incorporate UV microspectrophotometry into real casework samples involving undyed fibers.

MATERIALS AND METHODS

The undyed fibers for this research were taken from thirty-two samples of apparently white clothing collected from a morgue. All clothing samples were collected with as little prescreening as possible, in hopes of simulating real casework. Thus, the samples had several uncontrolled variables such as environmental exposure (i.e. sunlight, heat, washing, dry cleaning), manufacturing stress, and the composition of the raw material. In theory, the number of times a sample has been washed may impart differences in the UV spectra of samples due to brighteners or bleaching agents on the fibers coming from detergents. The only prescreening performed on these samples was a visual exam to ensure that the samples had no obvious stains on them, and to ensure that the fibers had no significant colorant dyes or surface dyes present. The fibers used for this study were collected from samples as “white” as white cotton T-shirt fabric, which is known to be undyed. In addition, these fibers were selected from whole fabrics which would be noticeably discolored even if only a light beige colorant were present. For this reason, there is a level of confidence that the fibers used in this testing were undyed as opposed to white dyed fibers. For the purposes of this paper, the word “undyed” will be used to describe the fibers as defined above.

Sample Preparation

Using tweezers, fibers were teased out of different areas of a sample to ensure that a representative sample was collected. The fibers from each sample were then mounted on individual microscope slides. Traditional means of mounting fibers require glass microscope slides and cover slips and synthetic mounting media such as XAM and

Permunt. Unfortunately, these materials will adsorb too much in the UV range, interfering with data collection. Thus, collection of transmission spectra in the UV range requires samples to be mounted on quartz microscope slides in glycerin mounting media under quartz cover slips. Quartz and glycerin adsorb minimally in the UV range, allowing for more accurate data collection. Samples for this testing were mounted on quartz slides under quartz cover slips; both purchased from McCrone Accessories and Components (Westmont, IL). The glycerin mounting media was purchased from Fisher Scientific (certified grade).

Representative samples were also collected from each sample for the purpose of measuring the fiber thickness. These samples were mounted in 99.9% mineral oil on glass microscope slides under glass cover slips. Microscope slides and cover slips were purchased from Fisher Scientific, and the mineral oil was purchased from Safeway.

Microscopes and Instrumentation

Fibers from each sample were identified and measured using an Olympus BH-2 microscope. The BH-2 microscope is a comparison compound light microscope with polarizing light capabilities. Each quartz slide was mounted on the microscope stage, and the fiber types within each sample were identified. It is important to note that the identification scheme was limited to a generic classification of fiber types only, thus no subtypes were addressed. In the following table, the composition of each sample is recorded along with the sample origin (See Table 1).

Table 1: Undyed Fiber Samples

#	ORIGIN	FIBER COMPOSITION
1	Warm-up pants	Polyester, acrylic
2	Sweatshirt	Cotton, polyester
3	Sweatshirt	Acrylic
4	Sweatshirt	Cotton, polyester
5	Sweatshirt	Cotton, polyester
6	Panties	Nylon
7	Sweatpants	Cotton, polyester, acrylic
8	Button-up shirt	Cotton, polyester
9	Sweatshirt	Cotton, polyester
10	Towel	Cotton, polyester
11	T-shirt	Cotton, polyester
12	T-shirt	Cotton
13	Pull-over blouse	Polyester, some cotton
14	Jacket	Cotton, polyester
15	Long johns	Cotton
16	Socks	Cotton, nylon
17	Athletic socks	Cotton, nylon
18	Towel	Cotton, rayon (high & moderate delustrance)
19	Bed sheet	Cotton
20	Blanket	Polyester, nylon
21	Bed sheet	Cotton, polyester
22	Pull-over shirt	Cotton, polyester
23	Slacks	Polyester
24	Blouse	Cotton, polyester
25	Blouse	Rayon
26	Nightgown	Nylon
27	Socks	Acrylic, polyester
28	Button-up shirt	Cotton, polyester
29	Blouse	Polyester
30	Skirt	Rayon (heavy & semi-delustrance)
31	Nightshirt	Polyester
32	Stretch pants	acrylic

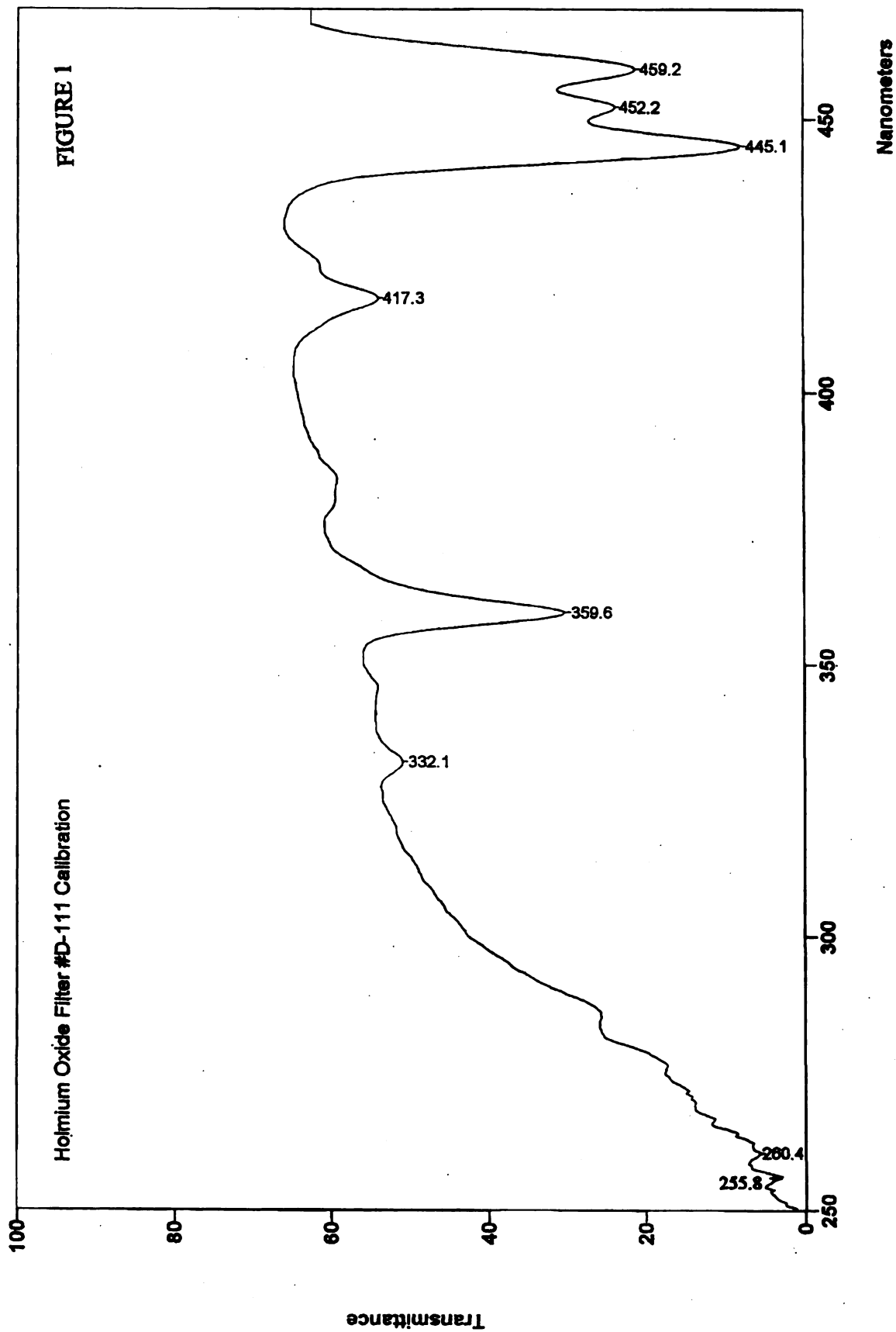
The availability of samples set boundaries to this research. Cotton and polyester fibers were by far the most abundant within the samples, so 20 of each of those fiber types were analyzed. Nylon, acetate, and rayon fibers were more limited, so only 5 of each of those types were analyzed. Although these fiber types were very limited, they were still analyzed in an attempt to get some preliminary data.

The S.E.E. 2000 microspectrophotometer was used for this research. This microspectrophotometer allows transmission spectra in the 250-850nm range. The power source is a 100-watt xenon lamp attached to a stabilized power source. The spectrometer is a dual CCD array detector with a total of 2048 detection wells. The first system has 1024 wells with a spectral range of 400-850nm. The second system has 1024 wells with a spectral range of 250-400nm. The estimated sensitivity is at approximately 86 photons per count, with a Signal-to-Noise ratio of 250:1. The 50x objective was used in this research, allowing a sampling area of 5 microns by 5 microns.¹¹

All fibers, with the exception of cotton, appeared round during identification, so fiber thicknesses were measured using a 10X objective with a reticle. Measurements for the cotton fibers were taken from the widest part of the untwisted areas. Ten fibers were measured for each fiber type within a sample, and the measurements were averaged.

Instrument Calibration

Calibration of the S.E.E. 2000 Microspectrophotometer requires a NIST traceable holmium oxide filter set, which has well documented absorption peaks.⁶ The filter is placed on the sample stage, and absorbance data is collected versus wavelength. Accuracy is reflected if the instrument calls several of the appropriate peaks at low, medium and high wavelength values. Specifically, the absorption peaks observed with the holmium oxide filter should be called over a wide range of the following wavelengths for UV data collection: 255.5nm, 286.5nm, 332.5nm, 360nm, 385nm, 417.5nm, 445.5nm, 452.5nm, and 459.5nm. The Holmium Oxide Filter #D-111 was used to calibrate the S.E.E. 2000 microspectrophotometer used for this data collection. The calibration data is included, and the peaks are labeled (See Figure 1).



Data Collection

A UV transmittance plot (transmittance vs. wavelength in nanometers) was taken for each run, consisting of 25 scans averaged per fiber, with a sampling frequency of 5. Before collecting the sample data, it is necessary to run a dark scan and a reference scan for each sample. A dark scan is collected by blocking the light path to the spectrometer. It is a measure of the instrument noise and interference. A reference scan is a measure of the lamp, slide, and mounting media without the sample of interest. Both the dark scan and the reference scan for each sample are stored in the instrument's memory so that they can be automatically subtracted from the sample run.

After collecting the necessary reference and dark scans, data were collected for the sample fibers. The data consisted of transmittance spectra (i.e. relative transmittance vs. wavelength) for ten randomly selected fibers within the same fiber class for every sample. For each sample, an overlay of ten runs from ten randomly selected individual fibers of the same class was collected. The overlays are also considered the raw data for each fiber type within a sample. For example, sample 1 originated from warm-up pants with a composition of polyester and acrylic. First, the data were collected for ten randomly selected polyester fibers from sample 1, and the spectra for all ten runs (the raw data) were plotted as an overlay on one graph. This process was then repeated for the data collection of the acrylic fibers in sample 1. Additionally, means and standard deviations of the overlay spectra were collected with the S.E.E. software. Step-by-step instructions for the collection of Transmittance data and operation of the S.E.E. 2000 are detailed in Appendix A. Appendix B contains the raw data, and Appendix C contains the means and standard deviations for all fiber types within each sample.

RESULTS AND DISCUSSION

The data were analyzed both qualitatively and quantitatively to identify potentially useful characteristics for separation. Qualitative analysis was based on visual analysis of the mean spectra, and quantitative analysis was based on statistical analysis of the mean spectra. First and foremost, the data (transmittance spectra overlays and sample mean spectra) were separated and organized by fiber type. The purpose of this separation was to facilitate a direct comparison between fibers of the same class that originated from different samples. The results and discussion section is separated to address each of the fiber classes individually. Each section will discuss regions of interest in the spectra established by visual examination. In addition, the statistical analysis is addressed by excel spreadsheets showing mathematical analysis of the regions of interest. Finally, the average fiber thicknesses (in microns¹²) for each fiber type will be included in the excel spreadsheets to facilitate an assessment of whether or not the fiber thickness could be responsible for imparting differences in the spectrographic data. Unless otherwise noted, the discussion section will refer to the mean data spectra, located in Appendix C.

Polyester

Visual examination of the UV spectral features of the polyester fibers shows three main regions of interest. The first region is at wavelength $< \sim 310\text{nm}$. The second region of interest is at a wavelength of approximately 310nm, and the final region is at wavelength $> \sim 310\text{nm}$.

Within the first region ($\lambda < \sim 310\text{nm}$), all of the polyester fibers tested showed a relatively flat spectrum with about 20%-30% fluctuations. The fluctuations in this region

could be rather insignificant, and attributed to light scattering or noise on the baseline. However, the features in this region seem to be very similar from one fiber to another within a sample. Further, when comparing this region to polyester fibers from different sources, there are obvious differences in the fine features. This suggests that this region may be useful for comparison or exclusionary purposes. Unfortunately, this hypothesis would need to be the focus of a further study with an updated S.E.E. that allows exploration of UV transmittance below 250nm. Thus, no statistical analysis of this region was attempted in this study.

The second region of significance for the polyester fibers is around the wavelength of approximately 310nm. At this wavelength, there is a rapid and very dramatic change in transmittance where the transmittance suddenly increases. This region of interest seems to be unique to polyester fibers, as none of the other fiber classes tested showed this consistent and obvious jump in transmittance. This region was analyzed quantitatively with a Microsoft Excel spreadsheet, and the goal was to determine if the jump in transmittance at wavelength 310 was statistically significant, allowing for separation of any of the polyester fibers. The results are tabulated in Table 2. The values for all spreadsheets were obtained from the mean spectra so that the data could be reduced to characteristics of the mean population.

The statistical analysis for region two ($\lambda \sim 310\text{nm}$) was calculated as follows. The mean transmittance and mean standard deviations (SD) for each polyester sample were recorded at wavelengths 300nm (T_1) and 350nm (T_2). Wavelengths of 300nm and 350nm were chosen because they were fairly stable areas of the spectra that bracketed the region of interest. The transmittance values at 300nm and 350nm were used to calculate the

change in transmittance ($T_2 - T_1$) at 310nm. To determine the standard deviations (SD) over the change in transmittance (ΔT), the following equation was used:

$\sqrt{(\text{SD of } T_1)^2 + (\text{SD of } T_2)^2}$. For the purposes of interpreting the statistics, the average ΔT and the average SD over the ΔT were calculated. Any sample that had a ΔT outside of the 95% confidence limit (two average standard deviations) was considered statistically significant. Six of the twenty polyester samples tested were shown to be statistically different from the others, and they are highlighted yellow in Table 2. Sample 20 has an asterisk next to it because the data is questionable. The ΔT for this sample is at the upper most limits of the 95% confidence limit. Since the value is still within two average standard deviations, and to retain a conservative approach to the statistical analysis in this study, the data for sample 20 will not be considered statistically significant.

Although the statistical analysis of region two lends some credence to significant quantitative differences between undyed polyester fibers, it is important to consider how variations in fiber thickness could be affecting the data. Samples within the 95% confidence limit have polyester fibers ranging in average thickness from 10.0 microns to 26.5 microns. Samples that were calculated to be statistically different had polyester fibers ranging in average thickness from 10.5 microns to 18.5 microns. The data collected shows that there is considerable overlap in fiber thickness between samples within the 95% confidence limit and samples outside of the 95% confidence limit. This data indicates that variations in fiber thickness between samples are not a cause of the statistically significant findings in this study.

Table 2: Quantitative Analysis of Polyester Fibers (Region 2)

Sample #	Transmittance @ 300nm (T ₁)	T ₁ Standard Deviation	Transmittance @ 350nm (T ₂)	T ₂ Standard Deviation	T ₂ - T ₁	(T ₂ - T ₁) Standard Deviation	Average Fiber Thickness (microns)
1	7.5	0.4	40.3	1.8	32.8	1.8	16.3
2	12.8	1.8	48.9	3.7	36.1	4.1	12.0
5	13.6	1.2	45.3	2.6	31.7	2.9	15.8
7	22.6	3.8	59.2	6.3	36.6	7.4	10.0
8	14.3	1.3	45.6	1.1	31.3	1.7	12.3
9	13.8	2.0	48.9	2.8	35.1	3.4	11.5
11	10.6	1.3	41.6	1.7	31.0	2.1	11.5
20 *	5.5	0.1	43.2	1.4	37.7	1.4	10.8
21	18.1	1.7	48.1	0.9	30.0	1.9	11.0
22	10.0	1.0	39.0	2.2	29.0	2.4	10.5
23	6.3	0.4	35.6	2.9	29.3	2.9	26.5
24	9.5	0.4	38.7	1.1	29.2	1.2	11.3
27	7.1	0.6	38.5	1.4	31.4	1.5	16.5
31	9.5	0.2	42.2	5.0	32.7	5.0	17.5
4	8.4	0.4	46.9	1.8	38.5	1.8	15.5
10	10.0	3.7	52.1	3.7	42.1	5.2	12.8
13	8.3	0.2	48.6	1.1	40.3	1.1	15.0
14	11.9	1.7	37.5	1.5	25.6	2.3	11.5
28	11.9	1.0	38.6	1.7	26.7	2.0	10.5
29	7.3	0.2	25.7	1.5	18.4	1.5	18.5
Averages of above columns—>					32.4	2.8	
LEGEND							
* Borderline statistically significant data							
Statistically significant data							
Average fiber thickness (microns)							
Images in this table are presented in color							

For the purposes of this study, the most discriminating data seems to be in region three. Region three focuses on the area of the spectrum with wavelength greater than 310nm. This region consistently shows a slight gradual increase in transmittance as the wavelength increases. Visual inspection of the samples shows that as the wavelength increases, some samples show one or more absorbances (dips in the spectrum, or regions of decreased transmittance). This observation became the basis for initial binning of the polyester data. The data were separated into groups according to the number of

absorbances (0, 1, and more than 1) in region three. Group P0 has zero absorbances, P1 has one absorbance, and P2 has more than 1.

Region three was also analyzed quantitatively with a Microsoft Excel spreadsheet, and the results are tabulated in Table 3. The goal was to see if quantitative analysis of region three justified the initial method of binning based on visual characteristics of the data. If necessary, the groups were reorganized after quantitative analysis. All anomalies are marked with an asterisk on the spreadsheet, and some of those samples were re-grouped in accordance with the criteria for quantitative separation. All samples that were grouped differently after quantitative analysis will be addressed.

The statistical analysis for region three ($\lambda > 310\text{nm}$) was calculated as follows. The mean transmittance and standard deviations (SD) for each polyester sample were recorded at wavelengths 350nm (T_2), 390nm (T_3), and 425nm (T_4). Wavelengths of 350nm, 390nm, and 425nm were chosen because they were fairly stable areas of the spectra that bracketed the major areas of absorbance that were of interest for the statistical analysis. The transmittance values at 350nm and 390nm were used to calculate the change in transmittance ($T_3 - T_2$) at one of the major absorbances, and the transmittance values at 390nm and 425nm were used to calculate the change in transmittance ($T_4 - T_3$) at a second major absorbance. To determine the standard deviations over the change in transmittance (ΔT) at the first and second major absorbances, the following equations were used: $\sqrt{(\text{SD of } T_2)^2 + (\text{SD of } T_3)^2}$ and $\sqrt{(\text{SD of } T_3)^2 + (\text{SD of } T_4)^2}$ respectively.

The above calculations were performed for all of the polyester samples, and the results are listed in Table 3. Generally, the statistical analysis justified the initial binning

based on visual characteristics. Comparison of the statistical results to the initial grouping based on visual inspection reveals the following:

1. Group P0 can be identified by looking at the values of $T_4 - T_3$ and $T_3 - T_2$. All samples that had statistical values below 6 at $T_4 - T_3$ and a positive value at $T_3 - T_2$ also showed no visual absorbances in the spectra. Thus, Group P0 could be separated from the rest of the polyester fibers by either visual examination or statistical analysis. These samples are highlighted yellow in Table 3. Sample 22 was the only sample that was not initially placed in Group P0 based on visual inspection because two shallow absorbances were observed visually. The raw data for Sample 22 reveals that the absorbances are not consistent between all of the individual fibers. Perhaps this sample has a mixture of polyester fibers that were treated differently before manufacturing the item, or perhaps the agent that is causing the absorbances was not evenly distributed throughout the fibers. Whatever the reason, the absorbances that are seen visually are not detected statistically because the absorbances are so shallow. Therefore, this sample was grouped based upon the statistical evaluation in attempt to be as conservative as possible in the separation of samples.

2. Group P1 can be identified by looking at the value of $T_3 - T_2$. All samples that had negative values at $T_3 - T_2$ also visually showed one large absorbance with a minimum value at wavelength $\sim 375\text{nm}$. Thus, Group P1 could be separated from the rest of the polyester fibers by either visual examination or statistical analysis. These samples are highlighted blue in Table 3. Sample 20 is the only sample that was not placed in Group P1 based on visual inspection, but statistically should have been re-grouped into Group P1. Reevaluation of Sample 20 showed that all individual polyester fibers within the sample had consistent features. Sample 20 is a true anomaly because visually, the

data shows three regions of absorbance, but statistically only one region of absorbance is captured. Because the visual features are so drastically different from what the statistical analysis captures, and because the features in the raw data are so consistent, it was determined that Sample 20 could be separated from the rest of the polyester samples based on visual analysis instead of statistical analysis. Sample 20 was placed into group P2 with other samples that showed more than one region of absorbance in the data.

3. Group P2 can be identified by values that do not fit into group P0 or P1. All samples that had a statistical value above 6 at $T_4 - T_3$ in addition to a positive value at $T_3 - T_2$ (with the exception of Sample 20, which had a negative value, as discussed above) showed more than one absorbance in the spectra. This proves that Group P2 could be separated from the rest of the polyester fibers by either visual examination or statistical analysis. These samples are highlighted pink in Table 3. Sample 10 is the only sample that was not initially placed in Group P2 based on visual inspection. Although sample 10 seems to have only one absorbance in region three by visual inspection, the statistics are placing sample 10 into group P2 because the transmittance value is much higher at one end of the absorbance at 425nm than at the other end of the absorbance at 350nm. Thus the statistics are still able to show a value above 6 at $T_4 - T_3$ and a positive value at $T_3 - T_2$ despite having only one obvious absorbance. Upon closer examination, it does appear that sample 10 could possibly have two very slight absorbances that are very close together.

Table 3: Quantitative Analysis of Polyester Fibers (Region 3)

Sample #	Transmittance @ 350nm (T ₂)	T ₂ Standard Deviation	Transmittance @ 390nm (T ₃)	T ₃ Standard Deviation	Transmittance @ 425nm (T ₄)	T ₄ Standard Deviation	T ₄ - T ₃ Standard Deviation	T ₄ - T ₃ Standard Deviation	(T ₃ - T ₂) Standard Deviation	Average Fiber Thickness (microns)
1	40.3	1.8	40.3	1.7	42.6	1.8	2.3	2.5	0.0	16.3
2	48.9	3.7	49.4	3.9	54.7	3.7	5.3	5.4	0.5	12.0
13	48.6	1.1	50.3	1.6	53.7	1.4	3.4	2.1	1.7	15.0
22 *	39.0	2.2	40.5	2.2	44.0	2.6	3.5	3.4	1.5	10.5
23	35.6	2.9	37.1	2.8	40.8	2.9	3.7	4.0	1.5	4.0
27	38.5	1.4	40.7	1.3	45.1	1.5	4.4	2.0	2.2	19.0
28	38.6	1.7	39.8	1.8	42.5	1.8	2.7	2.5	1.2	10.5
31	42.2	5.0	44.3	4.9	47.3	4.7	3.0	6.8	2.1	7.0
4	46.9	1.8	45.7	1.6	55.6	1.8	9.9	2.4	-1.2	2.4
5	45.3	2.6	45.2	2.4	53.1	2.7	7.9	3.6	-0.1	3.5
7	59.2	6.4	58.8	6.4	66.4	6.7	7.6	9.3	-0.4	9.1
9	48.9	2.8	48.6	2.8	55.4	3.3	6.8	4.3	-0.3	4.0
11	41.6	1.7	40.9	1.7	46.7	1.7	5.8	2.4	-0.7	2.4
24	38.7	1.1	37.8	1.0	43.3	1.1	5.5	1.5	-0.9	1.5
8	45.6	1.1	46.3	1.3	52.9	1.8	6.6	2.2	0.7	1.7
10 *	52.1	3.7	52.7	3.7	60.1	4.1	7.4	5.5	0.6	5.2
14	37.5	1.5	39.0	1.9	46.7	1.9	7.7	2.7	1.5	2.4
20 *	44.0	2.9	40.2	3.4	57.5	1.8	17.3	3.8	-3.8	4.5
21	48.1	0.9	48.4	0.6	55.0	0.8	6.6	1.0	0.3	1.1
29	25.8	1.5	27.8	0.6	36.1	1.9	8.3	2.0	2.0	1.6

LEGEND	
* Differences in data (Qualitative vs. Quantitative)	
	Group P0--zero absorbance
	Group P1--one absorbance
	Group P2--more than one absorbance
	Average fiber thickness (microns)
Images in this table are presented in color	

Although the statistical analysis of region three lends some credence to significant differences between the UV spectra of undyed polyester fibers, it is important to consider how variations in fiber thickness could be affecting the data. Samples in group P0 have polyester fibers ranging in average thickness from 10.5 microns to 26.5 microns. Samples in group P1 had polyester fibers ranging in average thickness from 10.0 microns to 15.8 microns. Samples in group P2 had polyester fibers ranging in average thickness from 10.8 microns to 18.5 microns. The data collected shows that there is considerable overlap in fiber thickness between groups P0, P1, and P2. This data indicates that variations in fiber thickness have no impact on the criteria used for the grouping of polyester fibers in this study.

Cotton

Visual examination of the UV spectral features of the cotton fibers shows two main regions of interest. The first region is at wavelength $< \sim 275\text{nm}$, and the second region of interest is at a wavelength $> \sim 275\text{nm}$.

Within the first region ($\lambda < \sim 275\text{nm}$), all of the cotton fibers tested showed an area of fluctuations, similar to the polyester fibers. Again, the fluctuations in this region could be rather insignificant, and attributed to light scattering or noise on the baseline. However, the features in this region seem to be very similar from one fiber to another within a sample. Further, when comparing this region to cotton fibers from different sources, there are obvious differences in the fine features. This suggests that this region may be useful for comparison or exclusionary purposes. Unfortunately, this hypothesis would need to be the focus of a further study with an updated S.E.E. that allows

exploration of UV transmittance below 250nm. Thus, no statistical analysis of this region was attempted in this study.

For the purposes of this study, the most discriminating data for the cotton samples seems to be in region two. This region focuses on the area of the spectrum with wavelength greater than 275nm. Visual inspection of the samples shows that as the wavelength increases, some samples show an area of absorbance (a dip in the spectrum, a region of decreased transmittance). This observation became the basis for initial binning of the cotton data. The data was separated into groups according to whether or not the spectra showed an absorbance in region two. Group C0 has zero absorbances, C1 has one shallow absorbance, and C2 has one deep absorbance. (Note: The mechanism for grouping these samples will change after statistical analysis.)

Region two of the cotton fibers was analyzed quantitatively with a Microsoft Excel spreadsheet, and the results are tabulated in Table 4. The goal was to see if quantitative analysis of region two justified the initial method of binning based on visual characteristics of the data. If necessary, the groups were reorganized after quantitative analysis. All anomalies are marked with an asterisk on the spreadsheet, and those samples were re-grouped in accordance with the criteria for quantitative separation. All samples that were grouped differently after quantitative analysis will be addressed.

The statistical analysis for region two ($\lambda > 275\text{nm}$) was calculated as follows. The mean transmittance and standard deviations (SD) for each cotton sample were recorded at wavelengths 375nm (T_1) and 450nm (T_2). Wavelengths of 375nm and 450nm were chosen because they were fairly stable areas of the spectra that bracketed the major area of absorbance that was of interest for the statistical analysis. The transmittance

values at 375nm and 450nm were used to calculate the change in transmittance ($T_2 - T_1$) at the absorbance. To determine the standard deviations over the change in transmittance (ΔT) at the absorbance, the following equation was used: $\sqrt{(\text{SD of } T_1)^2 + (\text{SD of } T_2)^2}$.

The above calculations were performed for all of the cotton samples, and the results are listed in Table 4. Initially, the cotton samples were separated into three groups based on visual analysis. As stated earlier, the groups consisted of group C0 which had zero absorbances, group C1 which had one shallow absorbance, and group C2 which had one deep absorbance. On the other hand, statistical analysis of the cotton samples revealed that they could only be separated into two groups with total confidence (Group C0 and Group C1). Statistically, the groups were identified by the value of $T_2 - T_1$. None of the samples had a $T_2 - T_1$ value of $5.3 < x < 8.3$. Thus all samples with a value of 5.3 or lower were placed into group C0, and all samples with a value of 8.3 or higher were placed into group C1. Although many of these samples had fairly high standard deviations, this is due to the high variability of fiber thickness in natural fibers such as cotton. Accordingly, not much weight was placed on the values of the standard deviations for these samples; the value of $T_2 - T_1$ was sufficient for purposes of separation of the cotton samples.

Comparison of the statistical results to the initial grouping based on visual inspection reveals the following:

1. All samples initially placed into group C0 by visual inspection of the data remained in group C0. These samples showed no visual evidence of a region of absorbance, and statistically, the $T_2 - T_1$ value was relatively low (below 3.5). These samples are highlighted yellow in Table 4.

2. All samples initially grouped together according to a region of one shallow absorbance were regrouped into group C0 after statistical analysis. These samples are highlighted yellow in Table 4 and identified with an asterisk. The reason these samples were regrouped with samples that showed no visual absorbance is because $T_2 - T_1$ values for all of these samples were fairly continuous (anywhere from 1.3 – 5.3) with no clear value that could be used for separation purposes. Yet it is interesting to note that the three samples with no visual absorbance do have the three lowest values for $T_2 - T_1$. In any case, the absorbances that are seen visually in these samples are not detected statistically because the absorbances are so shallow. For this reason, the statistical values were used for separation purposes instead of the visual characteristics. Samples that were initially separated according to either no visual absorbance or one shallow absorbance were grouped together after statistical analysis in attempt to be as conservative as possible in the separation of samples. Again, sample size for this project was limited, and perhaps a more distinct cut-off value for separation purposes would become apparent with further testing.

3. All samples initially grouped together according to a region of one deep absorbance, remained in the same group after statistical analysis. These samples all had $T_2 - T_1$ values of 8.3 or higher. Although this group was initially named Group C2 based on visual analysis, the group name changed to Group C1 after statistical analysis of the rest of the samples placed them all into Group C0. Samples with one deep absorbance, belonging to group C1, are highlighted blue in Table 4.

Table 4: Quantitative Analysis of Cotton Fibers (Region 2)

Sample #	Transmittance @ 375nm (T ₁)	T ₁ Standard Deviation	Transmittance @ 450nm (T ₂)	T ₂ Standard Deviation	T ₂ - T ₁	(T ₂ - T ₁) Standard Deviation	Average Fiber Thickness (microns)
4 *	76.0	7.2	79.4	7.1	3.4	10.1	21.3
7 *	71.6	2.2	76.3	2.3	4.7	3.2	19.5
8	78.9	3.7	81.0	3.8	2.1	5.3	20.0
10 *	79.3	4.3	82.9	4.6	3.6	6.3	18.3
12 *	80.0	5.6	84.3	5.3	4.3	7.7	20.5
13 *	69.6	5.3	74.9	5.7	5.3	7.8	19.8
14 *	77.4	1.7	80.8	2.2	3.4	2.8	19.8
16 *	74.8	3.3	79.4	3.1	4.6	4.5	19.8
17 *	69.3	2.6	73.6	2.9	4.3	3.9	18.3
19	74.2	4.6	77.3	5.0	3.1	6.8	18.3
21 *	72.7	3.7	75.9	3.4	3.2	5.0	16.3
22 *	56.6	3.6	61.0	3.2	4.4	4.8	15.8
24 *	57.7	2.2	62.7	2.2	5.0	3.1	18.3
28	65.8	4.7	67.1	4.7	1.3	6.6	19.8
2	67.5	5.6	75.8	6.3	8.3	8.4	21.0
5	70.7	2.2	80.9	3.3	10.2	4.0	21.5
9	80.1	5.4	90.9	5.6	10.8	7.8	19.3
11	67.5	5.8	78.7	4.7	11.2	7.5	20.0
15	75.2	3.0	84.0	4.6	8.8	5.5	19.3
18	63.7	2.4	74.0	2.8	10.3	3.7	17.8

LEGEND	
	Group C0—zero visible absorbances
*	Group C0—one visible shallow absorbance
	Group C1—one deep absorbance
	Average Fiber Thickness (microns)
Images in this table are presented in color	

Although the statistical analysis of region two lends some credence to significant differences between the UV spectra of undyed cotton fibers, it is important to consider how variations in fiber thickness could be affecting the data. Samples in group C0 have polyester fibers ranging in average thickness from 15.8 microns to 21.3 microns. Samples in group C1 had polyester fibers ranging in average thickness from 17.8 microns to 21.5 microns. The data collected shows that there is considerable overlap in fiber

thickness between groups C0 and C1. This data indicates that variations in thickness have no impact on the criteria used for the grouping of cotton fibers in this study.

Rayon

As mentioned earlier, the availability of samples set boundaries to this research. Samples made up of polyester and cotton were fairly abundant, whereas the samples made up of rayon, nylon, and acetate were more limited. Accordingly, only 5 samples for each of the rayon, nylon, and acetate fibers were available for analysis. Furthermore, there were actually only three rayon samples available, but two of the samples had both high and low delusterance fibers within the sample. These were treated as different samples even though they originated from the same source. Although these fiber types were very limited, they were still run in attempt to get some preliminary data.

Visual examination of the UV spectral features of the rayon fibers shows two main regions of interest. The first region is at wavelength $< \sim 275\text{nm}$, and the second region of interest is at a wavelength $> \sim 275\text{nm}$.

Within the first region ($\lambda < \sim 275\text{nm}$), all of the rayon fibers tested showed an area of fluctuations, similar to the polyester and cotton fibers. Again, the fluctuations in this region could be rather insignificant, and attributed to light scattering or noise on the baseline. However, the features in this region seem to be very similar from one fiber to another within a sample. Further, when comparing this region to rayon fibers from different sources, there are obvious differences in the fine features. This suggests that this region may be useful for comparison or exclusionary purposes. Unfortunately, this hypothesis would need to be the focus of a further study with an updated S.E.E. that

allows exploration of UV transmittance below 250nm. Thus, no statistical analysis of this region was attempted in this study.

For the purposes of this study, the most discriminating data for the rayon samples seems to be in region two. This region focuses on the area of the spectrum with wavelength greater than 275nm. Visual inspection of the samples shows that as the wavelength increases, some samples show an area with a very shallow absorbance (a dip in the spectrum, a region of decreased transmittance). This observation became the basis for initial binning of the rayon data. The data was separated into groups according to whether or not the spectra showed an absorbance in region two. Group R0 has zero absorbances and consisted of sample number 30 (high and low delust.). Group R1 has one very shallow absorbance and consisted of sample numbers 25 and 18 (high and low delust.). (Note: The mechanism for grouping these samples will change after statistical analysis.)

Region two of the rayon samples was analyzed quantitatively with a Microsoft Excel spreadsheet, and the results are tabulated in Table 5. The goal was to see if quantitative analysis of region two justified the initial method of binning based on visual characteristics of the data. The statistical analysis for region two ($\lambda > 275\text{nm}$) was calculated as follows. The mean transmittance and standard deviations (SD) for each rayon sample were recorded at wavelengths 375nm (T_1) and 425nm (T_2). Wavelengths of 375nm and 425nm were chosen because they were fairly stable areas of the spectra that bracketed the major area of absorbance that was of interest for the statistical analysis. The transmittance values at 375nm and 425nm were used to calculate the change in transmittance ($T_2 - T_1$) at the absorbance. To determine the standard deviations over the

change in transmittance (ΔT) at the absorbance, the following equation was used:

$$\sqrt{(\text{SD of } T_1)^2 + (\text{SD of } T_2)^2}.$$

The above calculations were performed for all of the rayon samples, and the results are listed in Table 5. Initially, the rayon samples were separated into two groups based on visual analysis. As stated earlier, the groups consisted of group R0, which had zero absorbances, and group R1, which had one very shallow absorbance. However, statistical analysis of the rayon samples revealed that they could not clearly be separated into two groups, because there was no clear demarcation in the $T_2 - T_1$ values. The values ranged from $0.1 \leq x \leq 4.2$. However, it is interesting to note that the two samples, which were placed into group R0 based on visual examination (no absorbance), do have the two lowest $T_2 - T_1$ values. This indicates that statistical analysis may have the ability to separate these samples, but clearly, there needs to be more data to look at before this could be supported. The data for fiber thickness is included in the table but will not be discussed further since the samples could not be separated into groups for the purposes of this study.

The rayon samples that had both high and low delusterance fibers showed the higher delusterance samples having a lower transmittance (or higher absorbance) as would be expected. The interesting point to make about these samples is that visual examination of region one ($<275\text{nm}$) between high and low delusterance fibers within a sample is significantly different. This indicates that the delusterance is imparting some of the UV characteristics. However, comparison of high delusterance fibers or low delusterance fibers from different samples is also significantly different. This indicates that some of the UV characteristics may be imparted by environmental factors. It is important to remember that this seems to be the trend with the preliminary data, but

clearly, more data would need to be collected before any definitive conclusions could be made.

Table 5: Quantitative Analysis of Rayon Fibers (Region 2)

Sample #	Transmittance @ 375nm (T_1)	T_1 Standard Deviation	Transmittance @ 425nm (T_2)	T_2 Standard Deviation	$T_2 - T_1$	$(T_2 - T_1)$ Standard Deviation	Average Fiber Thickness (microns)
18 (High)	46.6	2.6	50.8	2.8	4.2	3.8	12.5
18 (Low)	61.9	3.9	63.9	3.9	2.0	5.5	13.3
25	64.7	3.3	67.4	3.3	2.7	4.7	18.8
30 (High)	22.6	2.5	22.7	2.5	0.1	3.5	14.5
30 (Low)	65.6	3.9	67.3	4.0	1.7	5.6	13.8

LEGEND	
	Average Fiber Thickness (microns)
Images in this table are presented in color	

Nylon

Again, the availability of samples set boundaries to this research, so only 5 samples of nylon fibers were available for analysis. Although the nylon samples were very limited, they were still run in attempt to get some preliminary data.

Visual examination of the UV spectral features of the nylon fibers shows three main regions of interest. The first region of interest is at wavelengths $< \sim 300\text{nm}$, the second region of interest is around wavelength $\sim 300\text{nm}$, and the third region of interest is at wavelengths $> \sim 300\text{nm}$.

Within the first region ($\lambda < \sim 300\text{nm}$), all of the nylon fibers tested showed an area of fluctuations, similar to the polyester, cotton, and rayon fibers. Again, the fluctuations in this region could be rather insignificant, and attributed to light scattering or noise on the baseline. However, the features in this region seem to be very similar from one fiber to another within a sample. Further, when comparing this region to nylon fibers from different sources, there are obvious differences in the fine features. This suggests that

this region may be useful for comparison or exclusionary purposes. Unfortunately, this hypothesis would need to be the focus of further study with an updated S.E.E. that allows exploration of UV transmittance below 250nm. Thus, no statistical analysis of this region was attempted in this study.

For the purposes of this study, the most discriminating data for the nylon samples seems to be in region two. This region focuses on the area of the spectrum around wavelength $\sim 300\text{nm}$. Visual inspection of the samples shows that some samples show a very gradual increase in transmittance around this region, while others show a very drastic increase in transmittance around this region. This observation became the basis for initial binning of the nylon data. The data was separated into groups according to whether the spectra showed a gradual or a drastic increase in transmittance in region two. Group N0 shows a gradual increase in transmittance and consisted of sample numbers 16, 17 and 26, and group N1 shows a drastic increase in transmittance, consisting of sample numbers 6 and 20.

Region two of the nylon fibers was analyzed quantitatively with a Microsoft Excel spreadsheet, and the results are tabulated in Table 6. The goal was to see if quantitative analysis of region two justified the initial method of binning based on visual characteristics of the data. The statistical analysis for region two ($\lambda > \sim 300\text{nm}$) was calculated as follows. The mean transmittance and standard deviations (SD) for each nylon sample were recorded at wavelengths 275nm (T_1) and 325nm (T_2). Wavelengths of 275nm and 325nm were chosen because they were fairly stable areas of the spectra that bracketed the area of increased transmittance that was of interest for the statistical analysis. The transmittance values at 275nm and 325nm were used to calculate the

change in transmittance ($T_2 - T_1$) for region two. To determine the standard deviations over the change in transmittance (ΔT) in region two, the following equation was used:

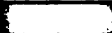
$$\sqrt{(\text{SD of } T_1)^2 + (\text{SD of } T_2)^2}.$$

The above calculations were performed for all of the nylon samples, and the results are listed in Table 6. Statistically, the groups were identified by the value of $T_2 - T_1$, and the statistics seemed to support the original binning based on visual characteristics. None of the samples had a $T_2 - T_1$ value of $9.3 < x < 22.1$. Thus all samples with a value less than or equal to 9.3 were placed into group N0, and all samples with a value greater than or equal to 22.1 were placed into group N1. Although many of these samples had fairly high standard deviations, this is due to the high variability of fiber thickness in these nylon fibers. Accordingly, not much weight was placed on the values of the standard deviations for these samples; the value of $T_2 - T_1$ was sufficient for purposes of separation of the nylon samples.

All samples initially placed into group N0 by visual inspection of the data remained in group N0. These samples showed a gradual increase in transmittance around the 300nm wavelength, and statistically, the $T_2 - T_1$ value was relatively low (less than or equal to 9.3). These samples are highlighted yellow in Table 6. Furthermore, all samples initially placed into group N1 by visual inspection of the data remained in group N1. These samples showed a drastic increase in transmittance around the 300nm wavelength region, and statistically, the $T_2 - T_1$ value was relatively high (greater than or equal to 22.1). These samples are highlighted blue in Table 6.

Table 6: Quantitative Analysis of Nylon Fibers (Region 2)

Sample #	Transmittance @ 275nm (T_1)	T_1 Standard Deviation	Transmittance @ 325nm (T_2)	T_2 Standard Deviation	$T_2 - T_1$	$(T_2 - T_1)$ Standard Deviation	Average Fiber Thickness (microns)
16	51.9	2.2	60.8	1.9	8.9	2.9	20.0
17	60.2	3.6	66.9	3.8	6.7	5.2	18.5
26	41.6	3.3	50.9	2.9	9.3	4.4	20.0
6	38.2	3.2	60.3	3.3	22.1	4.6	20.0
20	9.3	1	38.8	2.5	29.5	2.7	19.0

LEGEND	
	Group N0—gradual increase in Transmittance
	Group N1—dramatic increase in Transmittance
	Average Fiber Thickness (microns)
Images in this table are presented in color	

Although the statistical analysis of region two lends some credence to significant differences between the UV spectra of undyed nylon fibers, it is important to consider how variations in fiber thickness could be affecting the data. Samples in group N0 have polyester fibers ranging in average thickness from 18.5 microns to 20.0 microns. Samples in group N1 had polyester fibers ranging in average thickness from 19.0 microns to 20.0 microns. The data collected shows that there is considerable overlap in fiber thickness between groups N0 and N1. Although the limitations in availability of nylon samples set boundaries to this research, this preliminary data indicates that variations in fiber thickness have no impact on the criteria used for the grouping of nylon fibers in this study.

Region three of the nylon fibers focuses on the area of the spectrum with wavelength $> \sim 300\text{nm}$. Visual inspection of the samples reveals that all samples have a very gradual, constant increase in transmittance through this region, but only some of the samples have an absorbance in this region. Initially, this was believed to be good criteria for separation purposes. However, the absorbances in nylon samples 16 and 17 were so

nominal, that statistical analysis would not even be beneficial. Perhaps this region may be more useful for statistical analysis if a larger quantity of nylon samples is available for future studies.

Acrylic

As mentioned earlier, the availability of samples set boundaries to this research, so only 5 samples of acrylic fibers were available for analysis. Although the acrylic samples were very limited, they were still run in attempt to get some preliminary data.

All five of the acrylic fibers had different UV spectral features that were noticeable through visual examination alone. Because the spectra were so different from each other, there was no common link between any of them to be able to easily separate them into groups. However, all of the acrylic fibers did show the same area of fluctuations that the polyester, cotton, rayon and nylon fibers did below wavelength 300nm. Again, the fluctuations in this region could be rather insignificant, and attributed to light scattering or noise on the baseline. However, the features in this region seem to be very similar from one fiber to another within a sample. Further, when comparing this region to acrylic fibers from different sources, there are obvious differences in the fine features. This suggests that this region may be useful for comparison or exclusionary purposes. Unfortunately, this hypothesis would need to be the focus of further study with an updated S.E.E. that allows exploration of UV transmittance below 250nm. Thus, no statistical analysis of this region was attempted in this study.

At wavelengths above 300nm, the acrylic fibers were analyzed quantitatively with a Microsoft Excel spreadsheet, and the results are tabulated in Table 7. The goal was to see if quantitative analysis of this region would justify binning since visual examination

did not. The statistical analysis was calculated as follows. The mean transmittance and standard deviations (SD) for each acrylic sample were recorded at wavelengths 375nm (T_1) and 450nm (T_2). Wavelengths of 375nm and 450nm were chosen because they captured a region that had a very large absorbance in one of the samples. The transmittance values at 375nm and 450nm were used to calculate the change in transmittance ($T_2 - T_1$) over this large absorbance. To determine the standard deviations over the change in transmittance (ΔT) in this region, the following equation was used:

$$\sqrt{(\text{SD of } T_1)^2 + (\text{SD of } T_2)^2}.$$

The above calculations were performed for all of the acrylic samples, and the results are listed in Table 7. Statistically, the groups were identified by the value of $T_2 - T_1$, and the statistics seemed to place the samples into three groups. Acrylic samples 1 and 32 had a $T_2 - T_1$ value close to zero, which means that they had a fairly constant transmittance between 300nm and 450nm. These samples are highlighted blue in the table. Acrylic samples 7 and 27 had a slightly larger $T_2 - T_1$ value, close to 6, which indicates that there was an increase in transmittance between 300nm and 450nm. These samples are highlighted pink in the table. Acrylic sample 3 had a very large $T_2 - T_1$ value at 33.8. The large change in transmittance is due to the large absorbance seen in the spectrum. This sample (highlighted yellow in the table) is clearly very different from the others both visually and statistically. Although many of these samples had fairly high standard deviations, this is due to the high variability of fiber thickness in these acrylic fibers. Accordingly, not much weight was placed on the values of the standard deviations for these samples; the value of $T_2 - T_1$ was sufficient for purposes of separation of the acrylic samples.

Table 7: Quantitative Analysis of Acrylic Fibers (Region 2)

Sample #	Transmittance @ 375nm (T_1)	T_1 Standard Deviation	Transmittance @ 450nm (T_2)	T_2 Standard Deviation	$T_2 - T_1$	$(T_2 - T_1)$ Standard Deviation	Average Fiber Thickness (microns)
1	69.2	2.3	68.6	2.3	-0.6	3.3	17.8
32	69.3	2.7	70.1	2.6	0.8	3.7	17.8
7	70.6	2	76.1	2.4	5.5	3.1	23.5
27	70.4	2.3	76.5	2.5	6.1	3.4	20.0
3	45.8	2.9	79.6	3.3	33.8	4.4	22.0

LEGEND	
	Group A1-- $\Delta T \approx 0$
	Group A2-- $\Delta T \approx 6$
	Group A3--large ΔT
	Average Fiber Thickness (microns)
Images in this table are presented in color	

Although the statistical analysis lends some credence to significant differences between the UV spectra of undyed acrylic fibers, it is important to consider how variations in fiber thickness could be affecting the data. Both samples in group A1 had an average thickness of 17.8 microns. The two samples in group A2 had average thicknesses of 20.0 microns and 23.5 microns. The sample in group A3 had an average thickness of 22.0 microns. The data collected shows that there is overlap in fiber thickness between groups A2 and A3, while group A1 had the smallest diameters. However, the limitations in availability of acrylic samples set boundaries to this research, and it would be beneficial to gather more data in future studies before determining whether variations in fiber thickness had an impact on the criteria used for the grouping of acrylic fibers in this study.

Although all of the data mentioned herein seems fairly promising for separating undyed fibers, it must be emphasized that this technique is only in the beginning stages. Many more samples will need to be tested before any definitive conclusions can be made.

Additionally, it is important to remember that the data thus far seems to be more relevant for exclusionary purposes since the data can only be put into groups and not individualized.

CONCLUSIONS AND RECOMMENDATIONS

The data shows many features in the UV transmittance spectra. Some examples include a rapid variation in transmittance with wavelength $< \sim 300\text{nm}$, a steep change in transmittance at wavelength $\sim 300\text{nm}$, and / or some characteristic variations with wavelength $> \sim 300\text{nm}$. These features became the basis for visual inspection of the data, and the spectra were analyzed as follows: First, the spectra were organized by fiber type (i.e. polyester, cotton, rayon, etc.). Second, distinguishing characteristics for each spectrum were noted, and the samples were grouped based on these characteristics. Third, quantitative (statistical) analysis was performed on these distinguishing characteristics based upon a mean response of the data from each fiber type, and samples were re-grouped accordingly, if needed.

The data from this study indicates that there are potential uses and applications for this work. One potential use for this data is the ability to analyze the characteristics of the sample mean with the intention of initial binning. For example, this study indicated that undyed polyester fibers could be separated into three different groups. With additional research, it is possible that the average characteristics of these fiber types could lead to automation by computer in the future. Obviously, in the early stages, this application would be used as a guide only. Additionally, several caveats should be kept in mind. For example, averaging within a sample may have a large standard deviation due to fiber to fiber variation within a sample. Further, there is always the possibility for a significant amount of information to be lost in averaging the transmittance response. Normalization of the data should be investigated as a way to minimize the effects of physical characteristics (i.e. fiber thickness) on the characteristics of a sample mean

(inter-sample variation). This would also improve the potential automation of binning. Finally, a more exhaustive study could be performed on the variation of fiber thicknesses between samples (intra-sample variation). Beer's Law could be applied to the statistics to determine whether variation in fiber thickness had any significant affect on the grouping system used in this study that was not captured through measuring the fibers.

A recommendation for future studies is to use an instrument that has the capability of collecting data extending into the wavelength region around 200nm in order to take full advantage of the data in the region below 300nm. Exploring the data in this region, which may be rich in features, could potentially hold answers to further separation capabilities.

Another intriguing research topic would be to explore the fluorescent properties of undyed fibers, using a microspectrophotometer. Optical brighteners and other bleaching agents, which may be responsible for the differences in the UV characteristics observed in this study, may also cause differences in fluorescent properties. It would be interesting to see whether fluorescence data would be beneficial to use in conjunction with UV data. Perhaps this joint approach would allow for further separation within the groups already mentioned herein.

The final recommendation would be to perform a study that has more controlled variables (i.e. number of times a garment is washed, detergents used, exposure to UV light) to see if specific UV characteristics are associated with those particular factors. A study of these controlled variables, in addition to other studies mentioned above, may give more insight to whether there is any validity to using UV characteristics of optical brighteners and bleaching agents as a means of definitively excluding (or more

importantly, relating) fibers. Again, it will be emphasized that these are merely speculations for what may happen in the future, and that the data within this study alone will not support these speculations without further research. This is a preliminary, exploratory study to see what kinds of features are in the data. Hopefully the data gathered herein, as well as the aforementioned applications and recommendations, will be used as a stepping stone for future studies.

APPENDICES

APPENDIX A

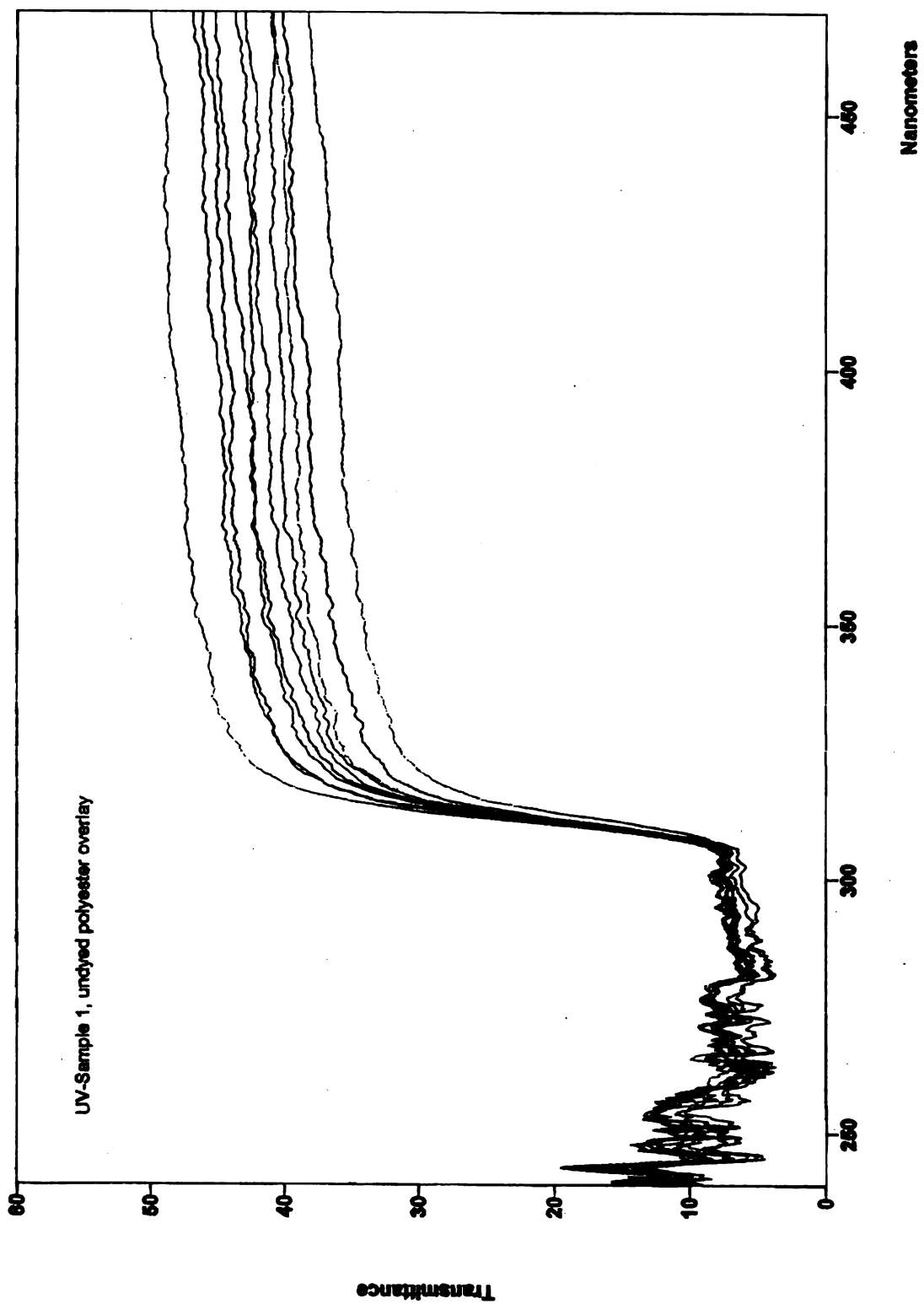
OPERATION INSTRUCTIONS FOR THE S.E.E. 2000

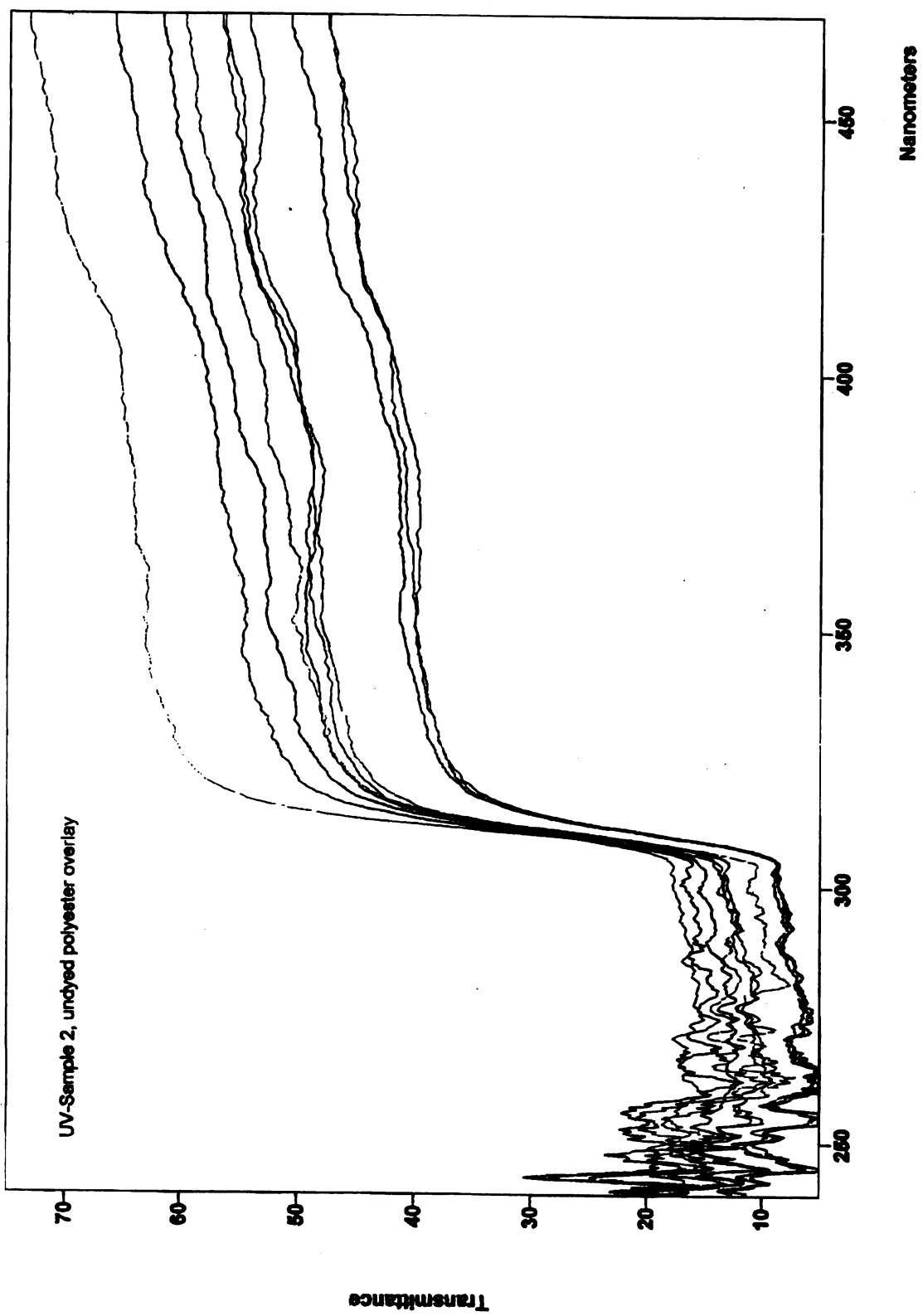
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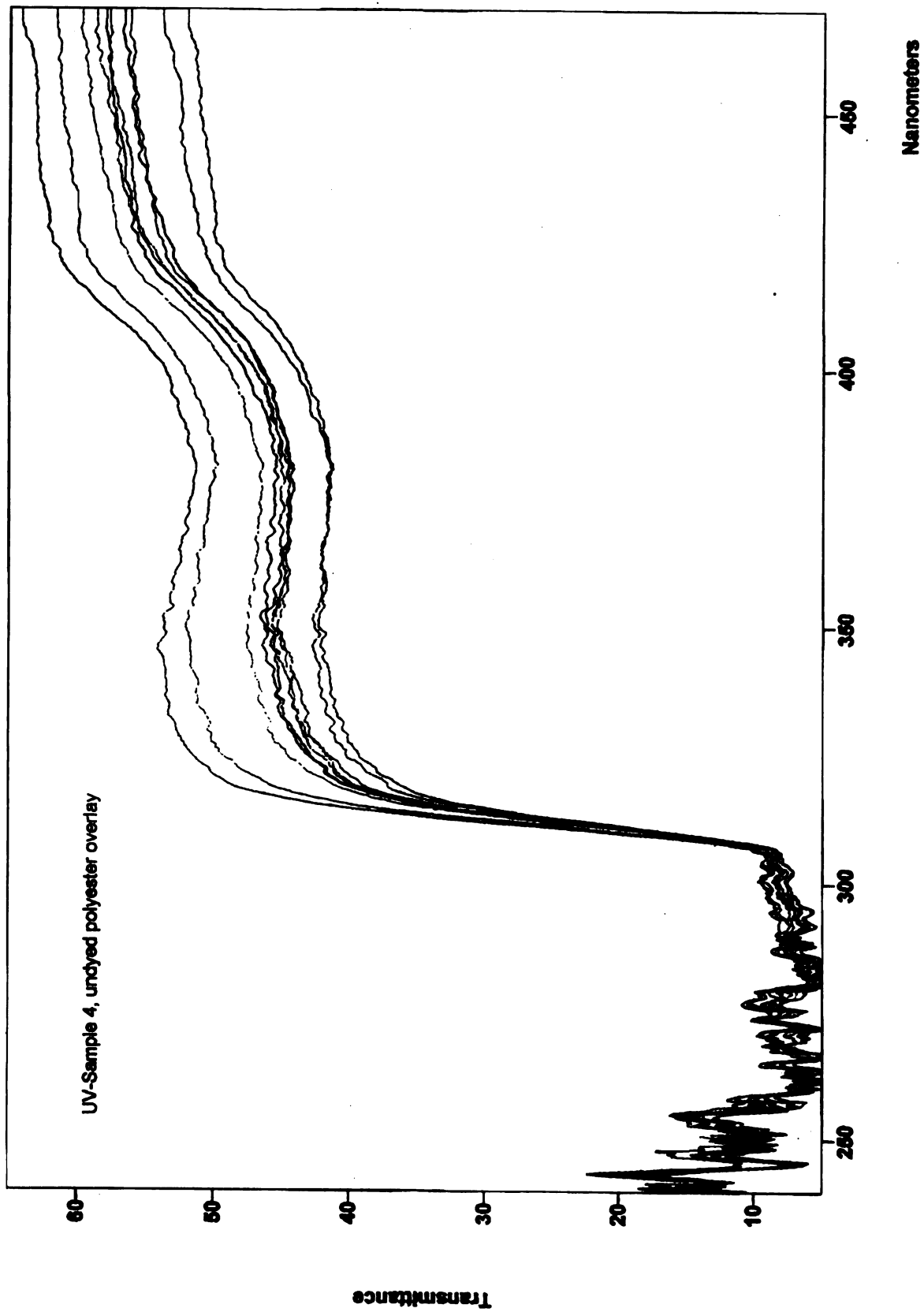
1. Turn on “Transmission” xenon lamp power supply. After approximately 2 minute warm-up period, observe the digital display on the power supply, and check that the reading is approximately “75” and stable.
2. Turn on the power to the video monitor and computer. Open the “S.E.E.” operating software. Allow approximately 20 minutes for system warm-up.
3. Remove all filters from the light path. Move the selection knob on the front of the reflectance head to darkfield (DF) position.
4. Place the sample slide on the microscope stage.
5. Rotate the desired objective into place, and focus on the specimen. The 15X reflecting objective must be used for UV spectrum collection.
6. Adjust the transmission substage condenser to approximately 0.2 for the 4X and 20X objectives and approximately 0.5 for the 50X objective. Install the 15X reflecting condenser if UV spectrum collection is desired.
7. If the “Reflectance” and/or “Fluorescence” lamps are on, ensure shutters for these lamps are off.
8. Close the field iris diaphragm of the microscope, and adjust the substage condenser so that the iris diaphragm is focused.
9. Center the condenser if necessary.
10. Open the field iris diaphragm until the image is just outside of the field of view.
11. Move the sample of interest under the black box as viewed on the monitor, and focus on the area to be analyzed. Move to a clear area of slide near the specimen of interest.
12. Block light from the specimen, and collect “Dark Scan.”
13. Allow light through the path, and collect “Reference Scan.”
14. Optimize the sampling frequency—counts should be approximately 2,500-3,000 in the visible region and approximately 1,000 in the UV region. If the sampling frequency is changed, collect a new “Dark Scan” and “Reference Scan.”
15. Move the sample area to be analyzed under the black square on the monitor.
16. Press “Scan Sample” to begin data collection.

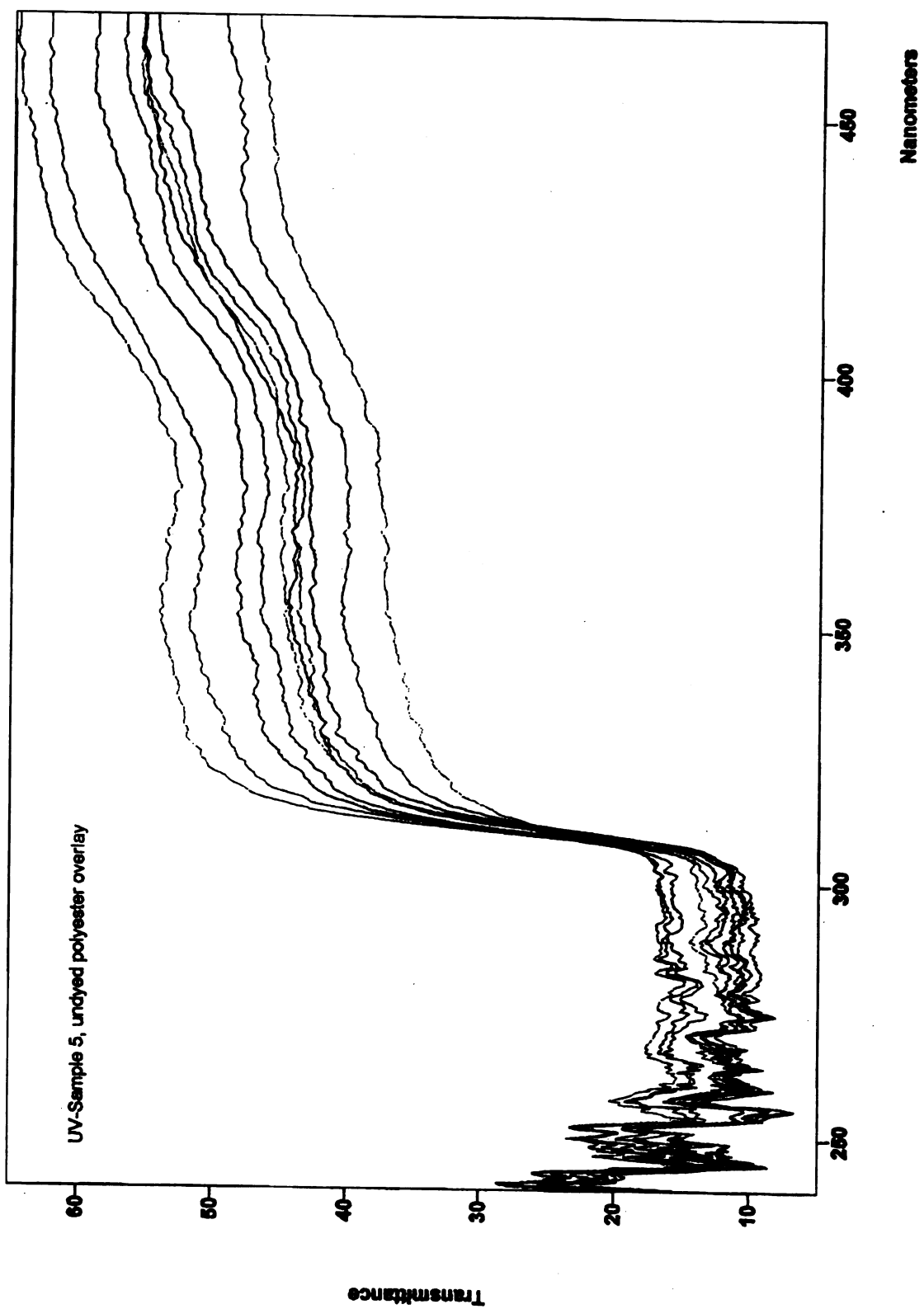
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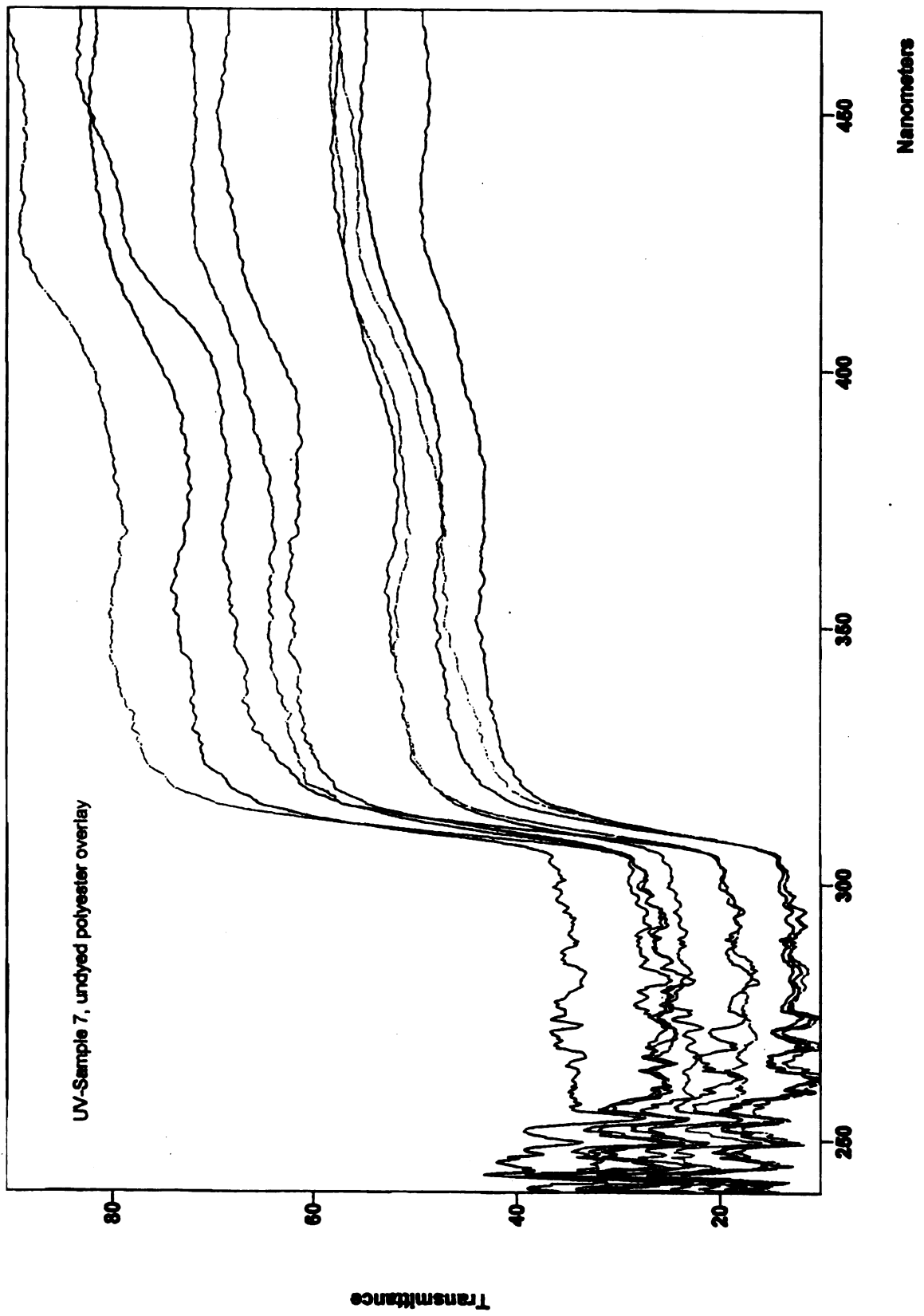
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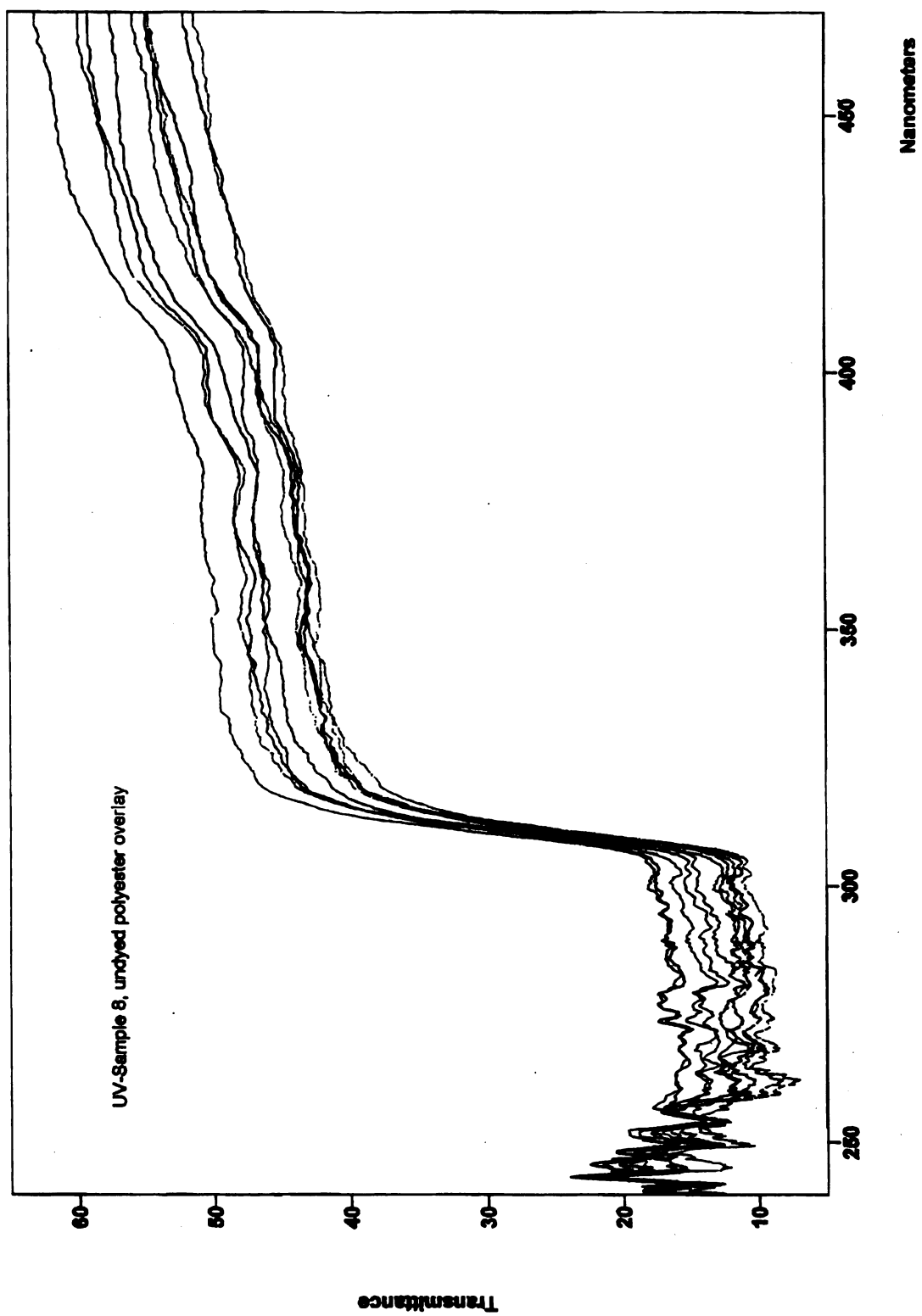


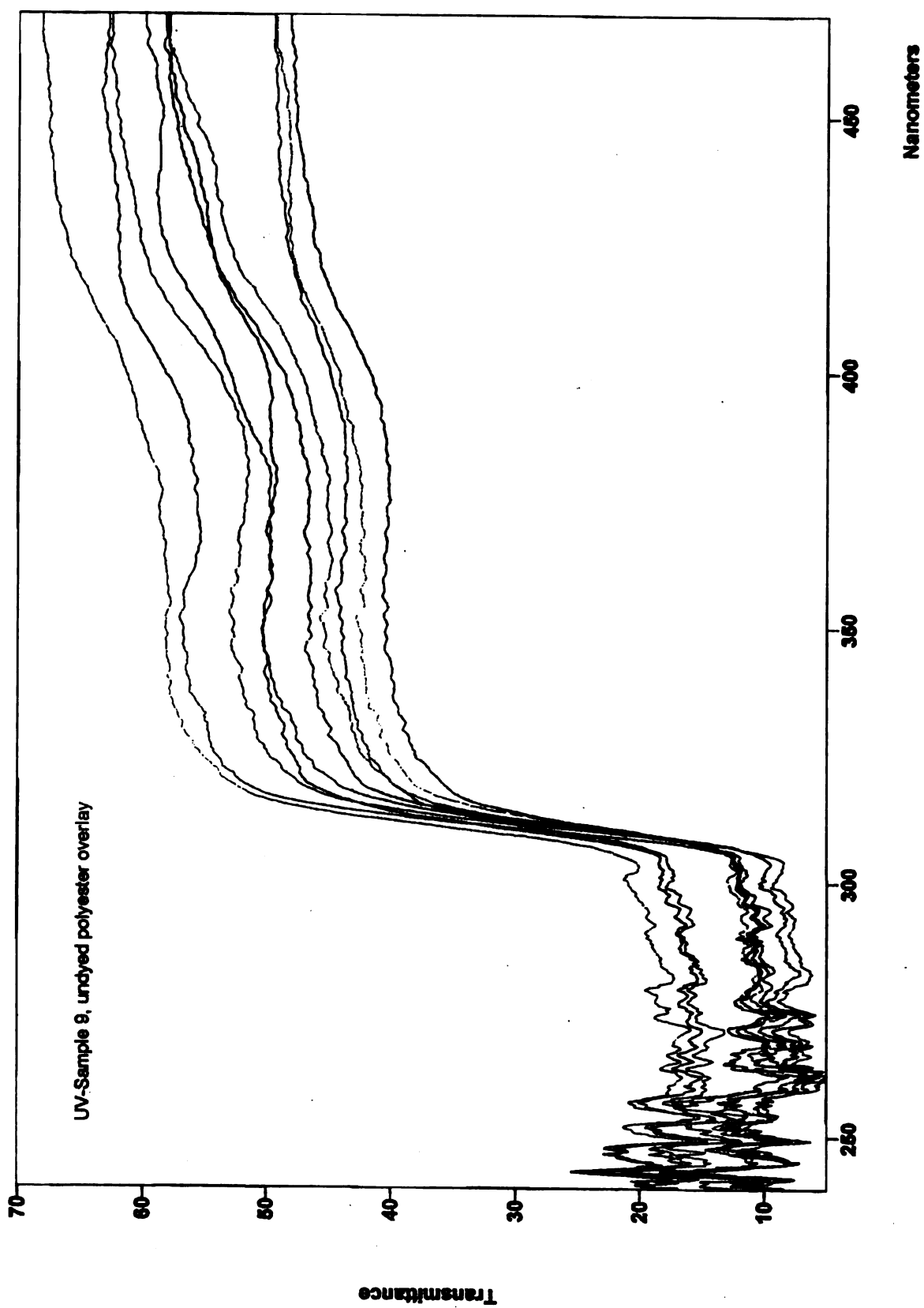


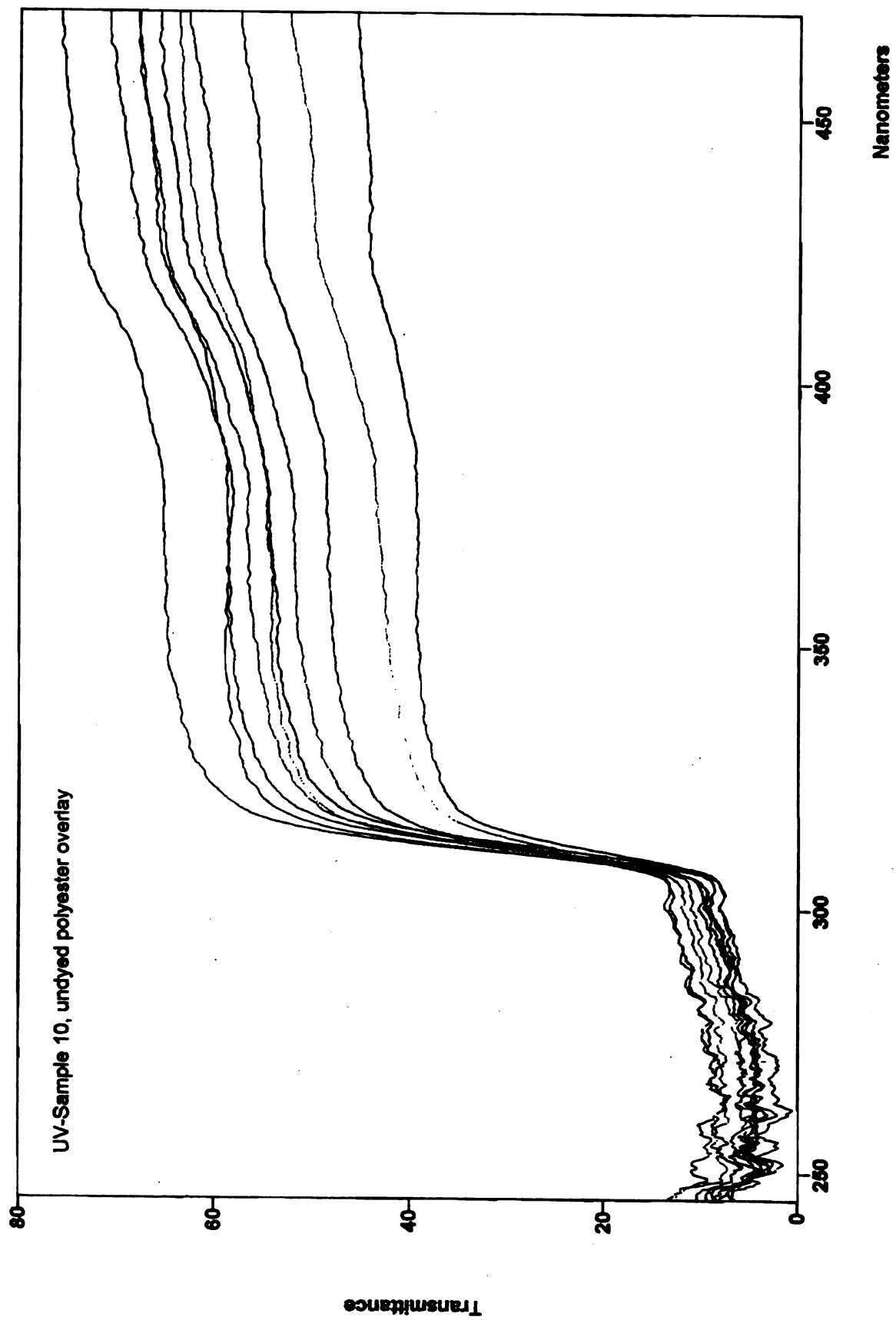


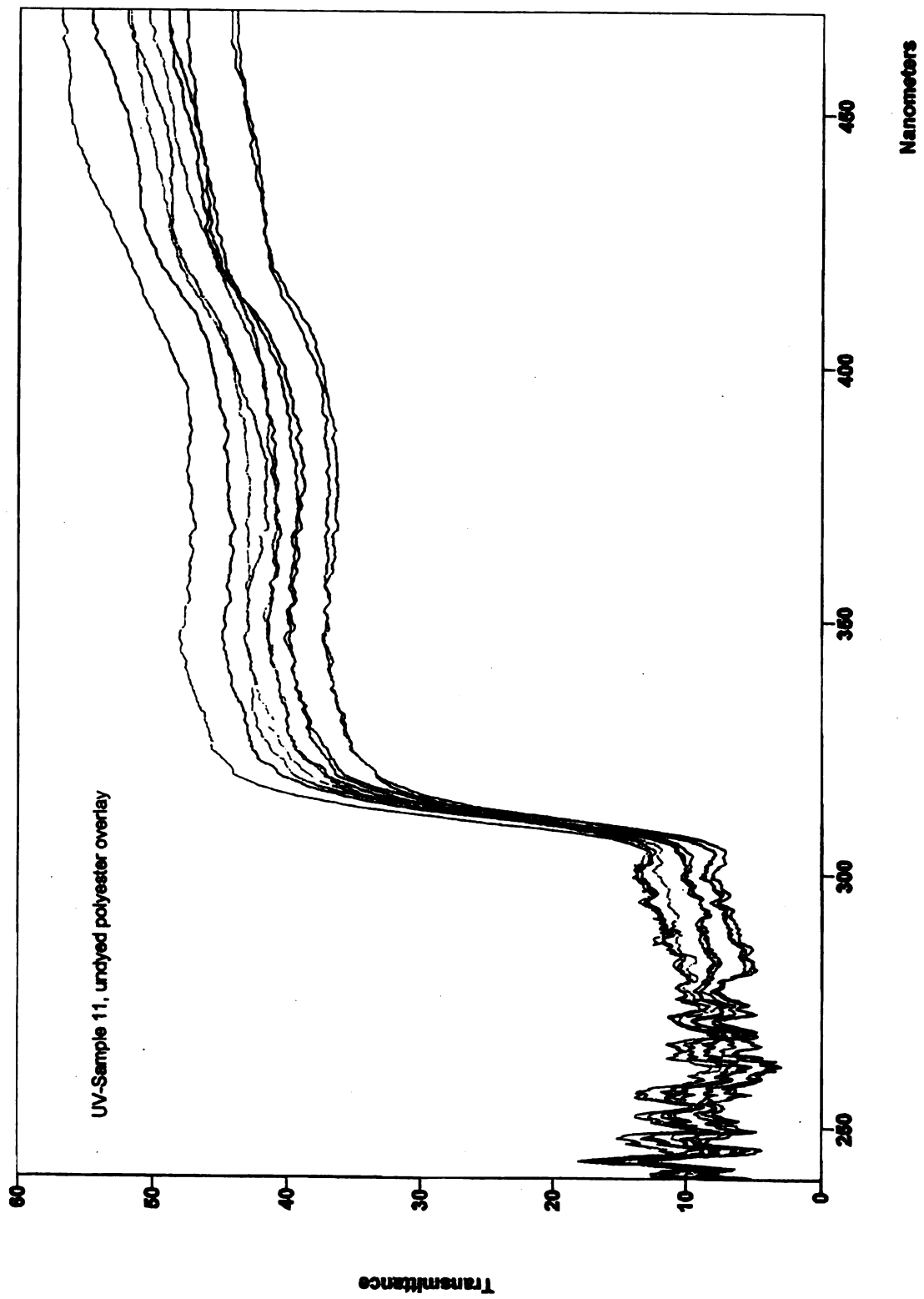


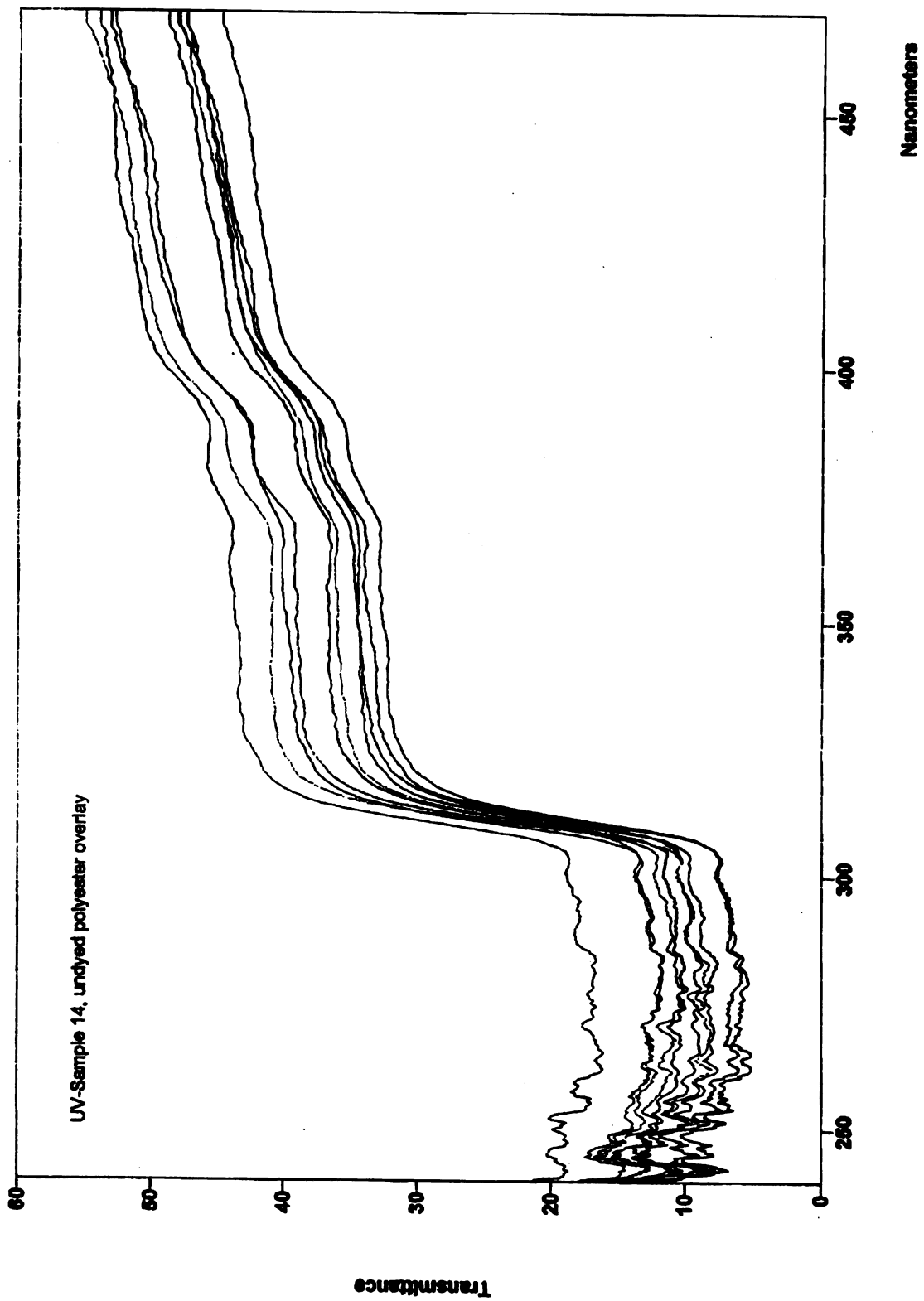


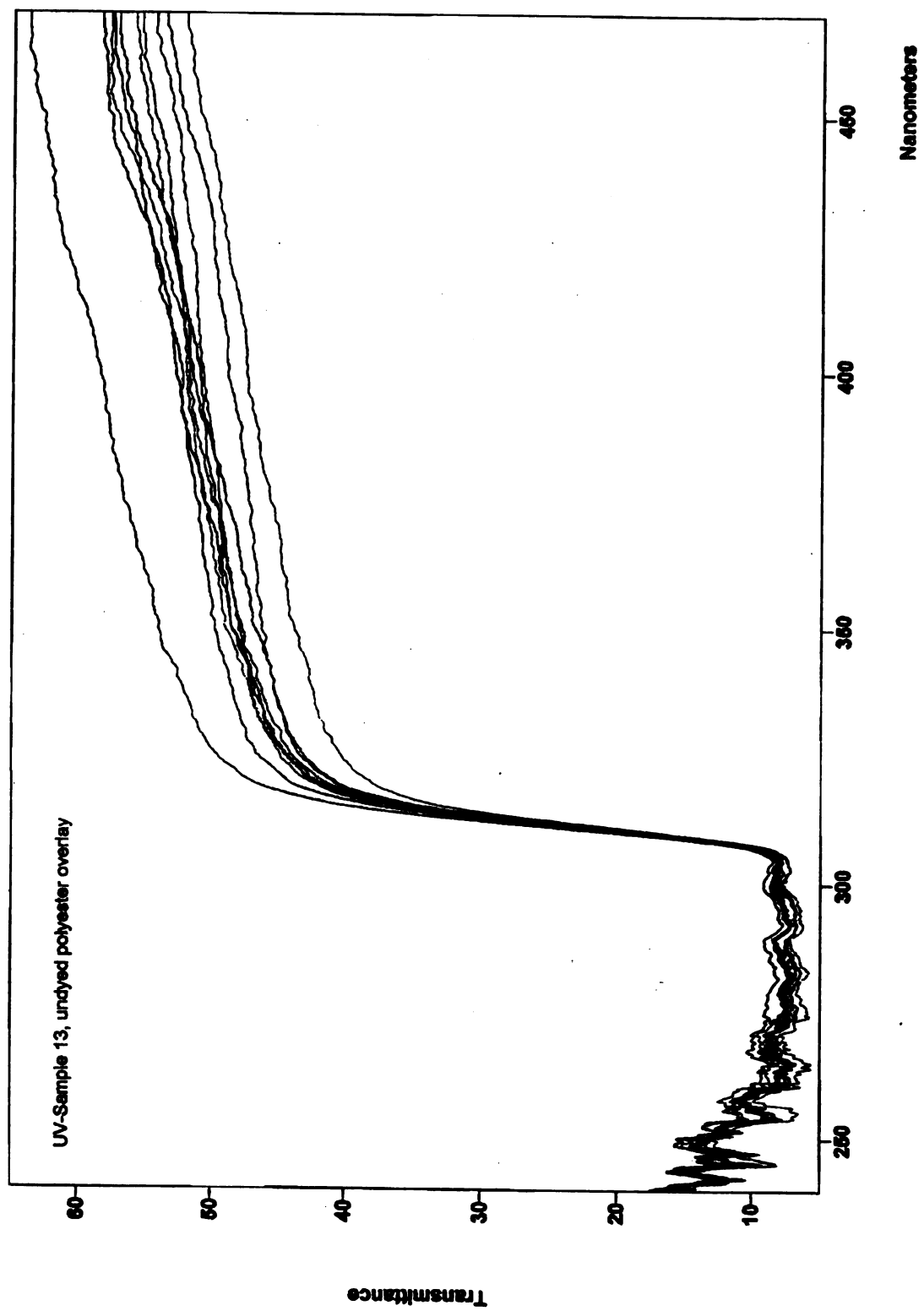


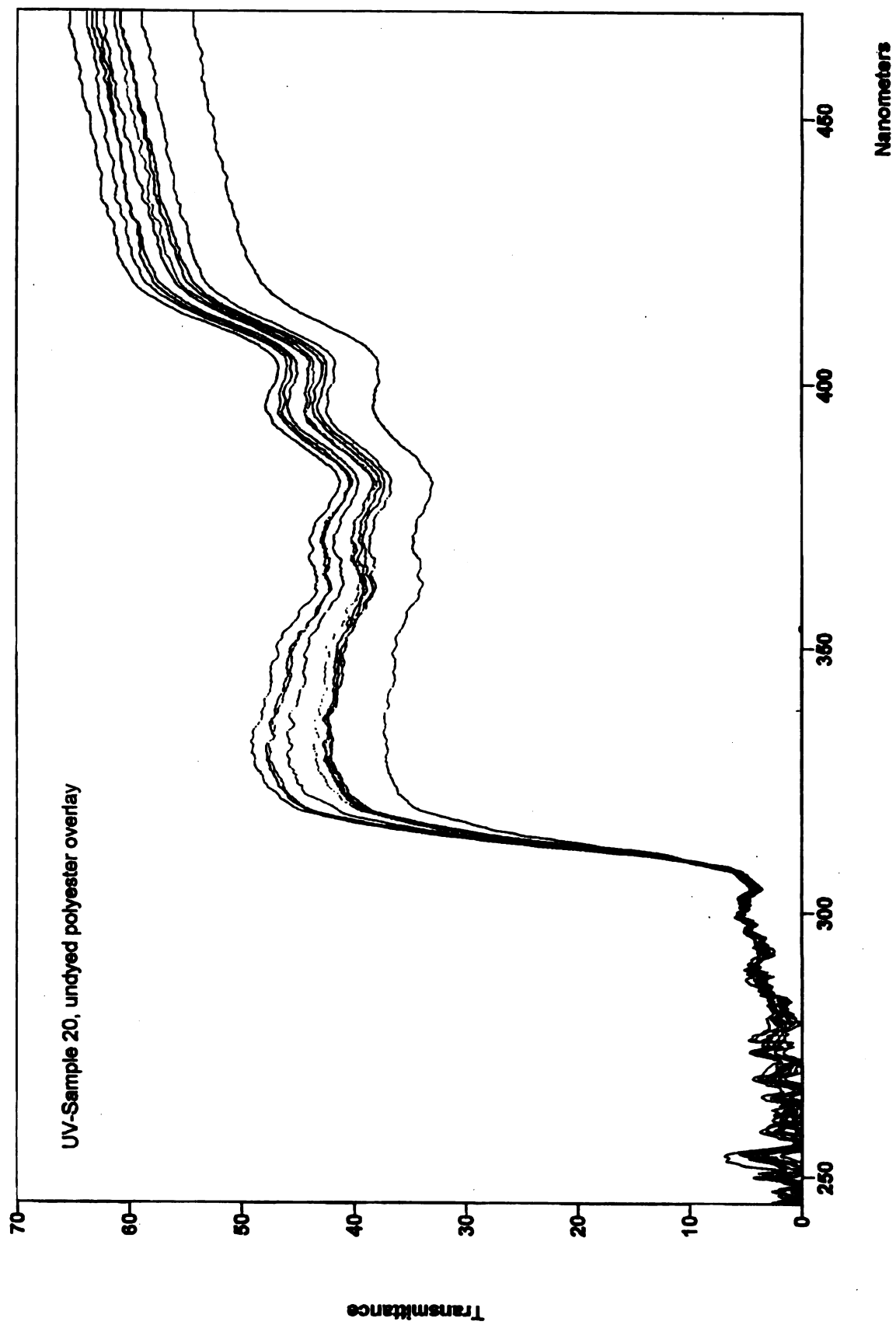


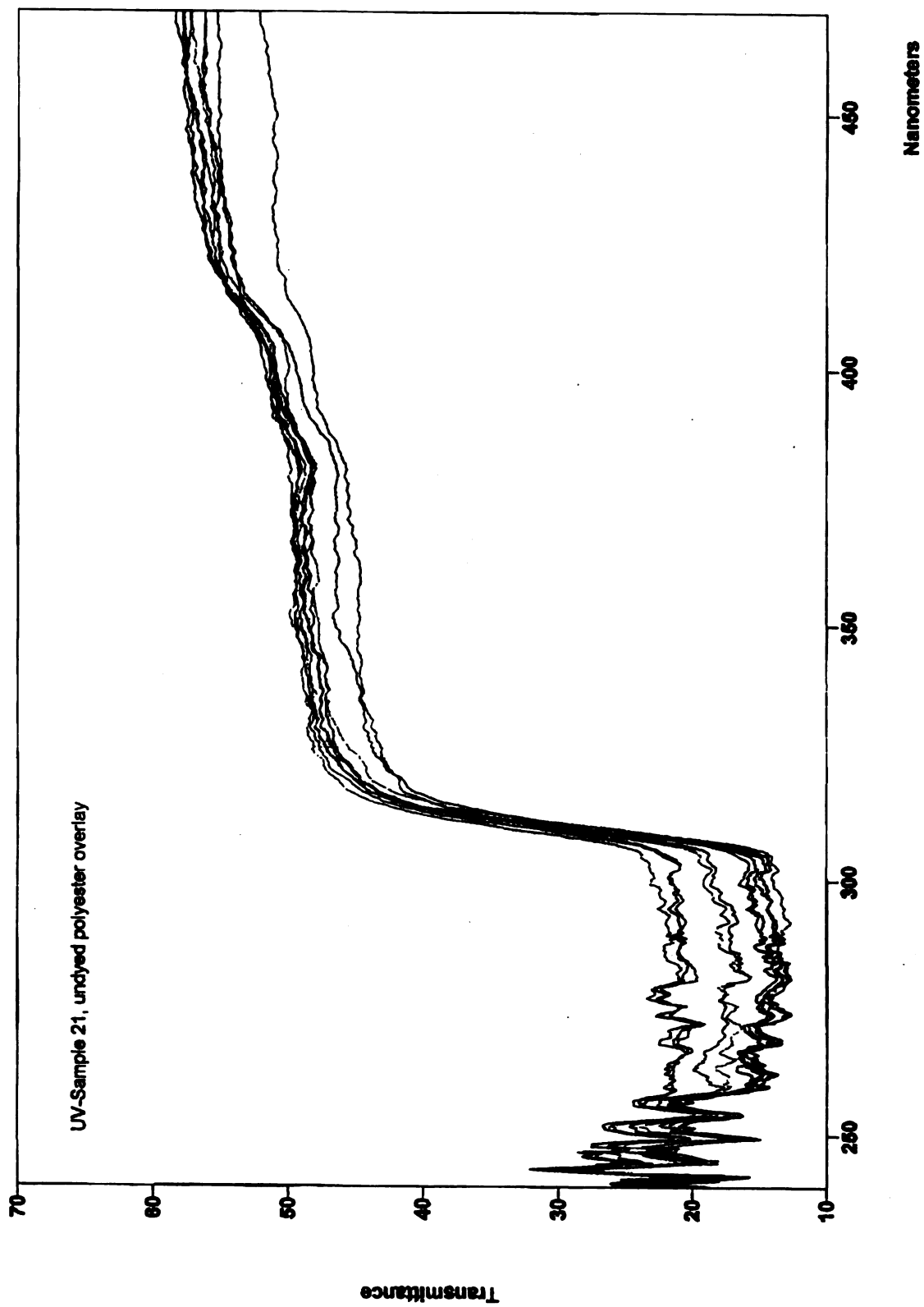


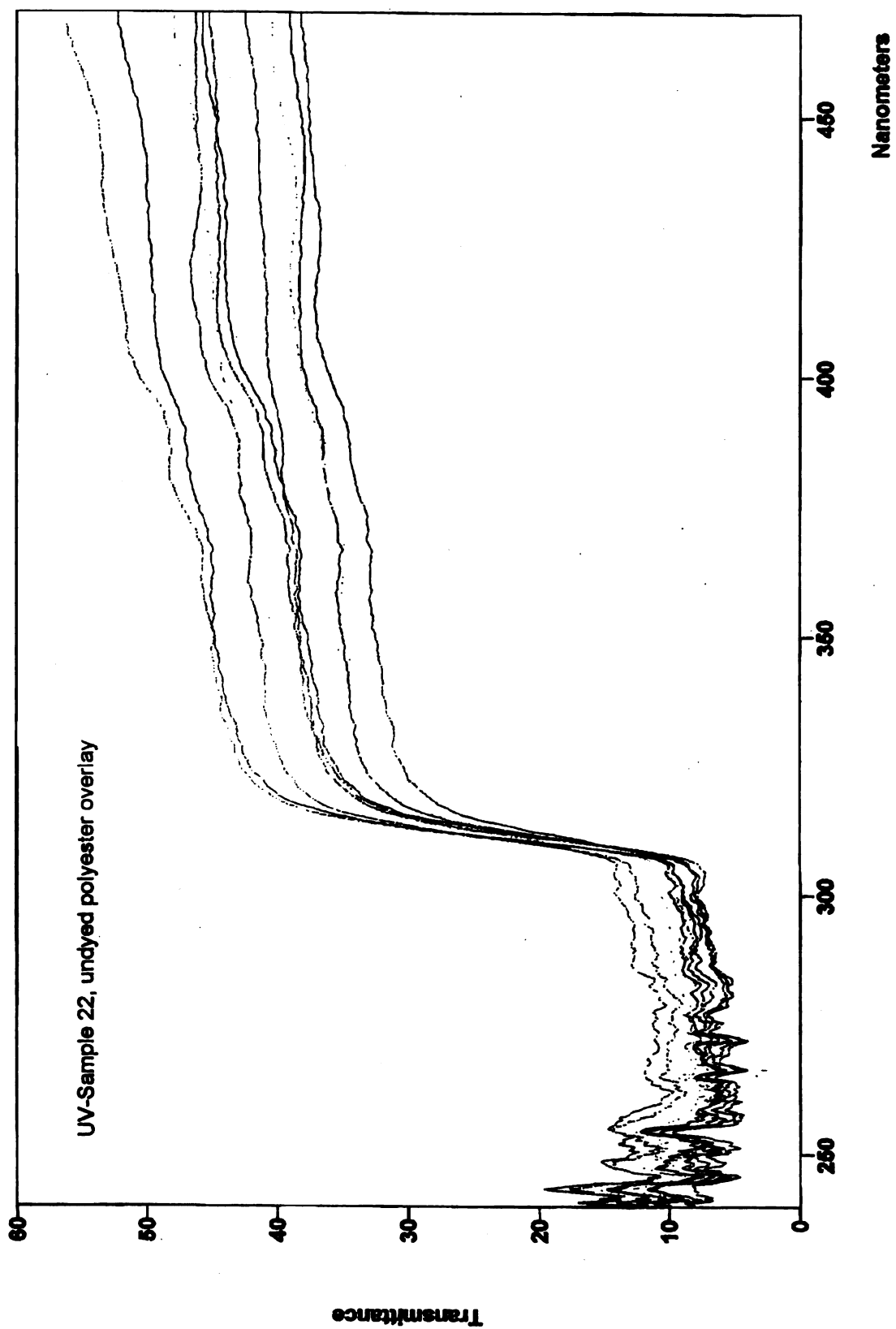


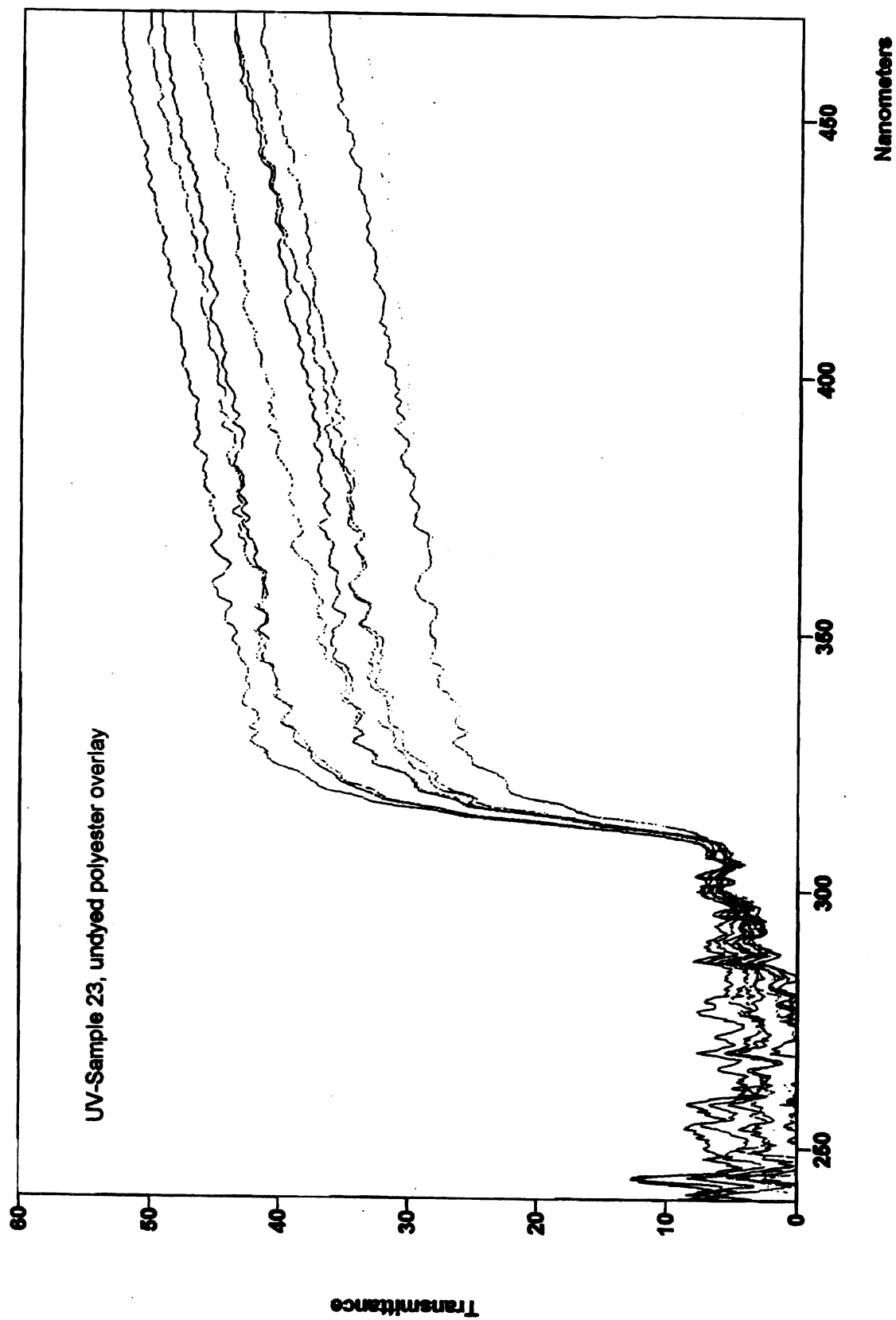


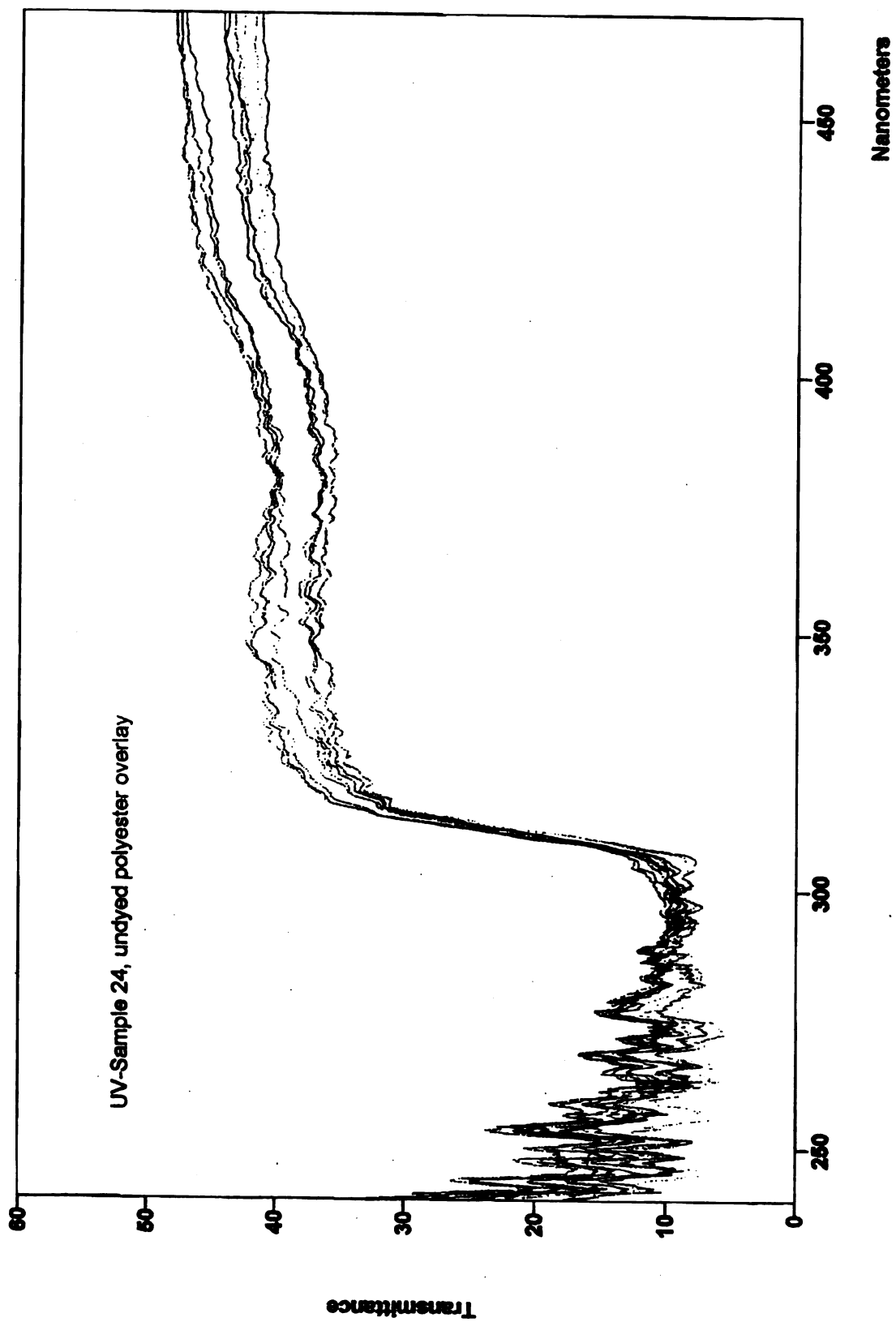


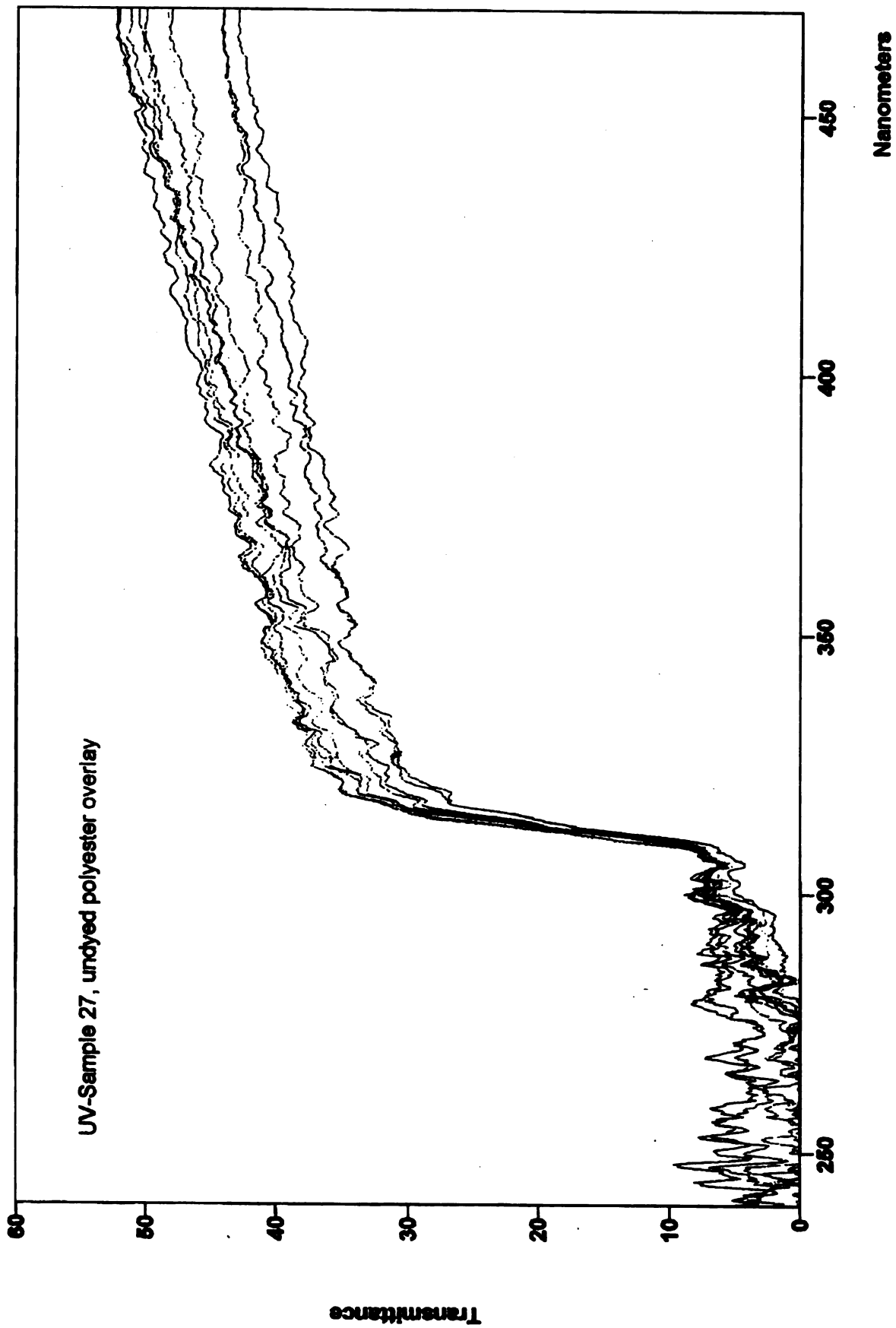


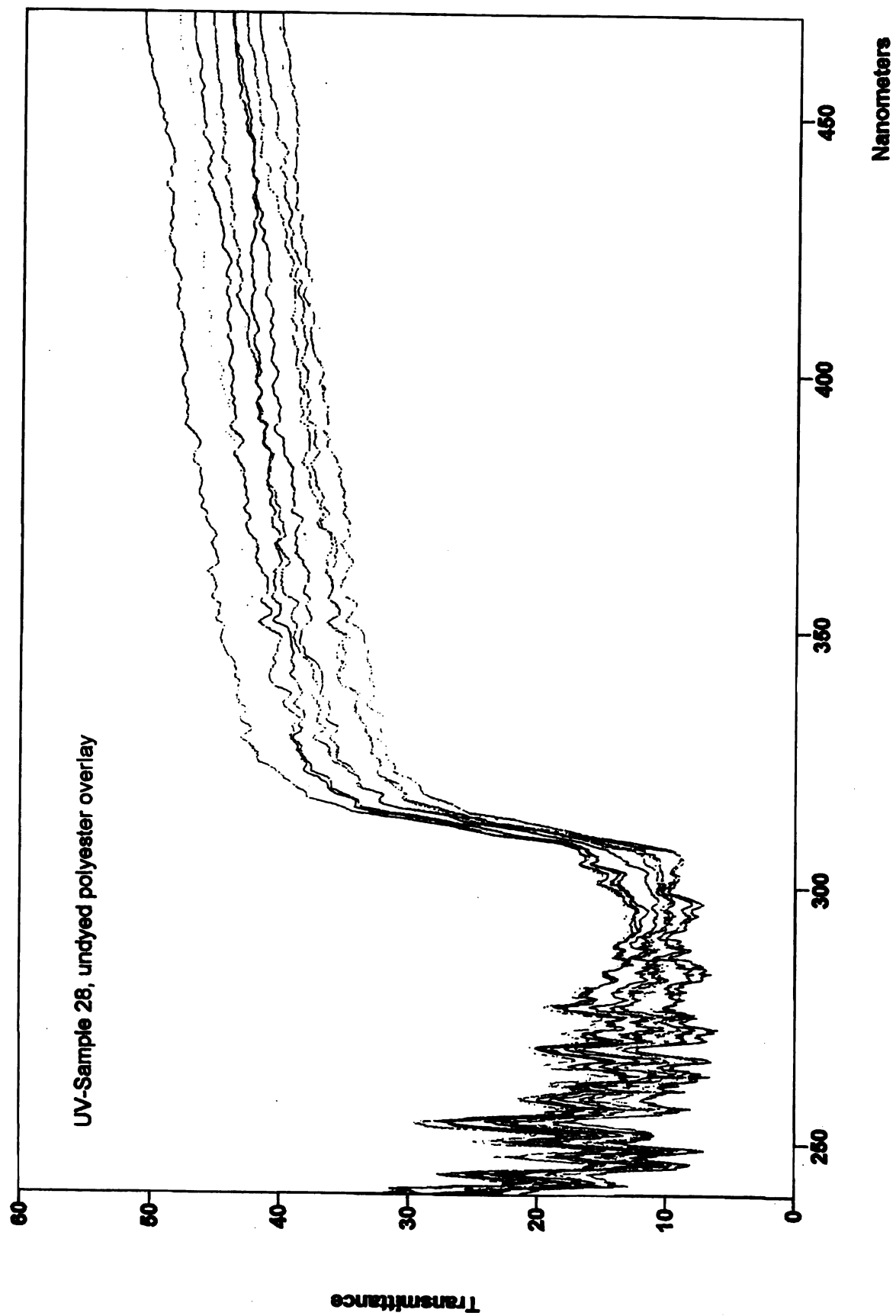


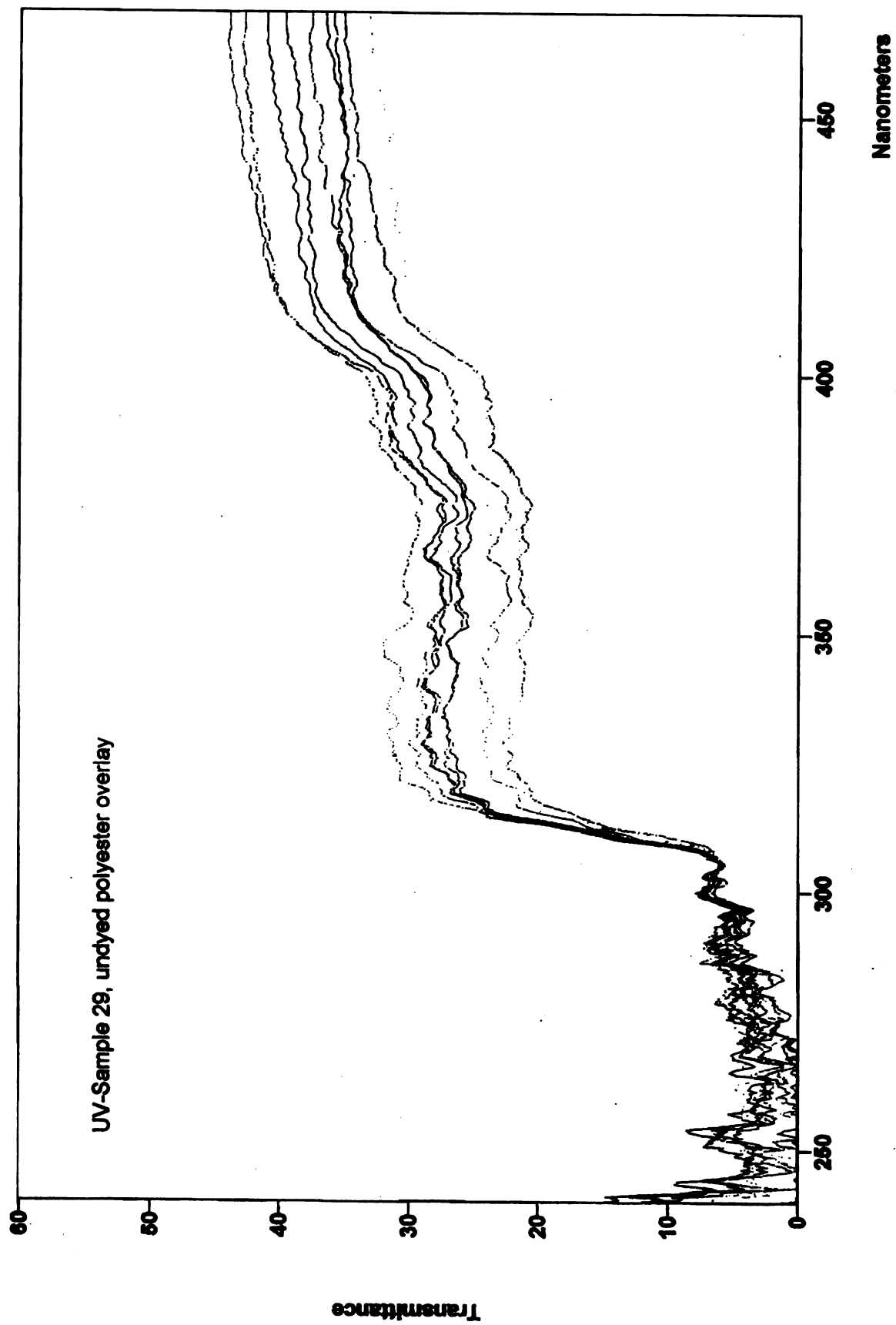


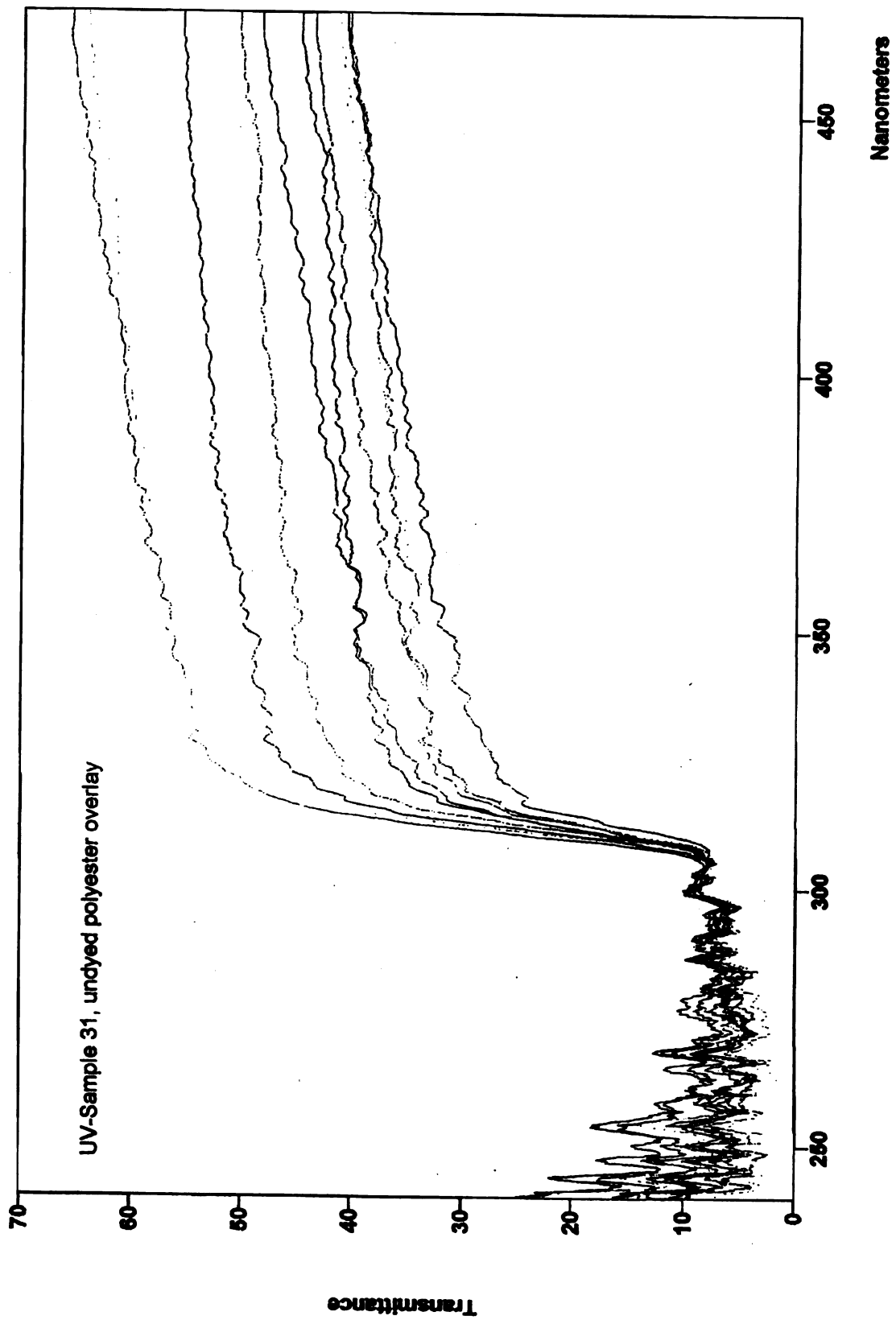


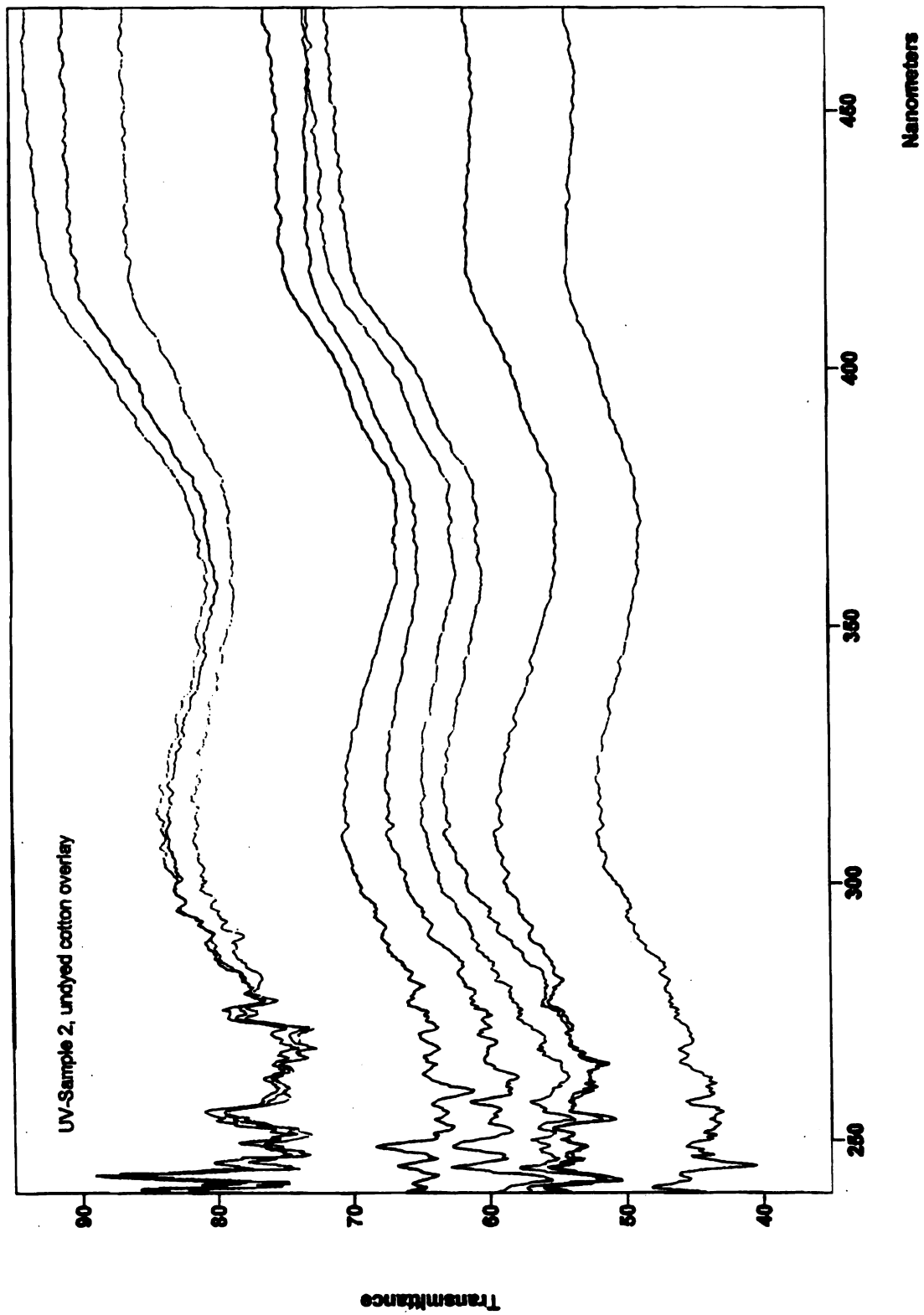


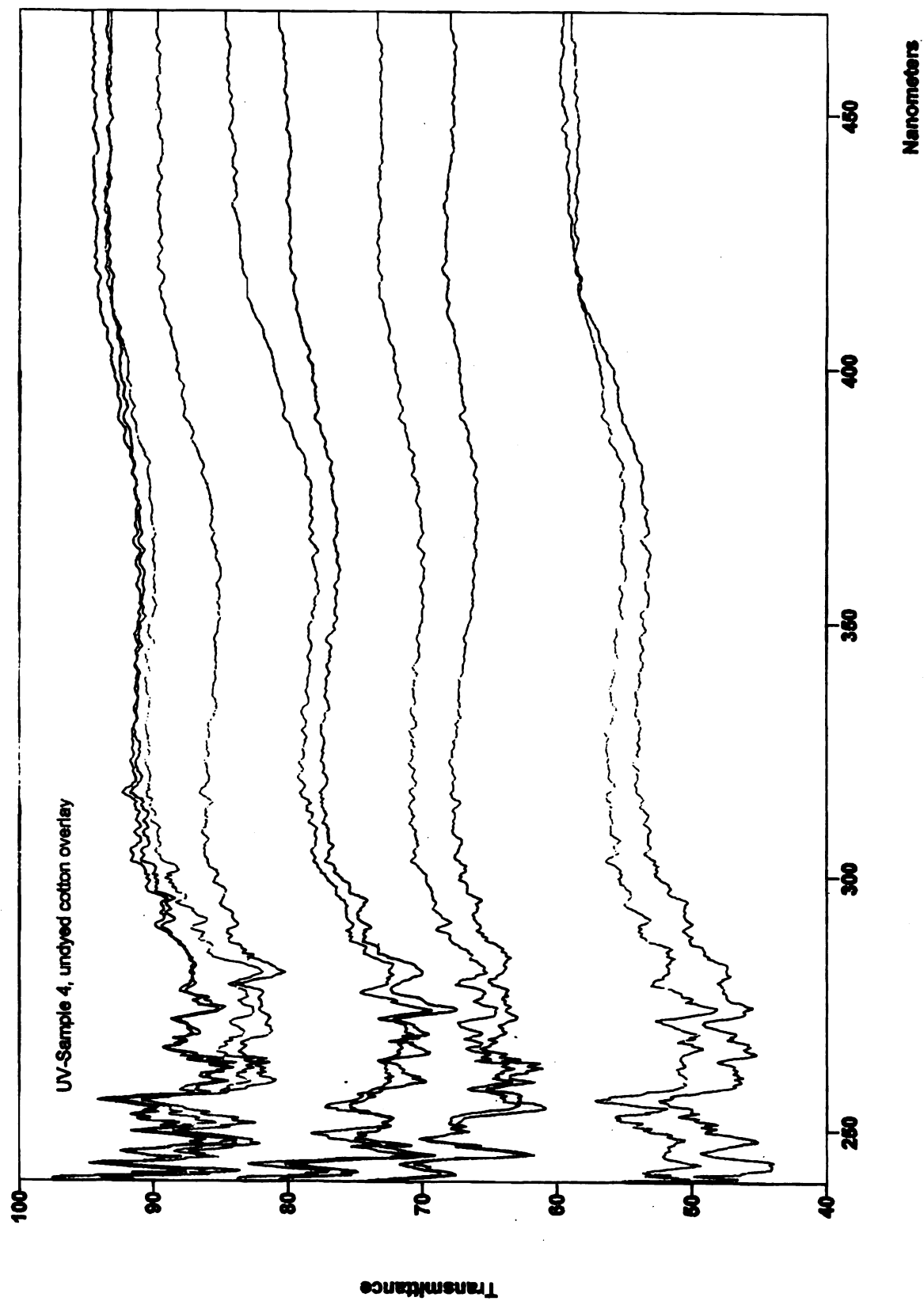


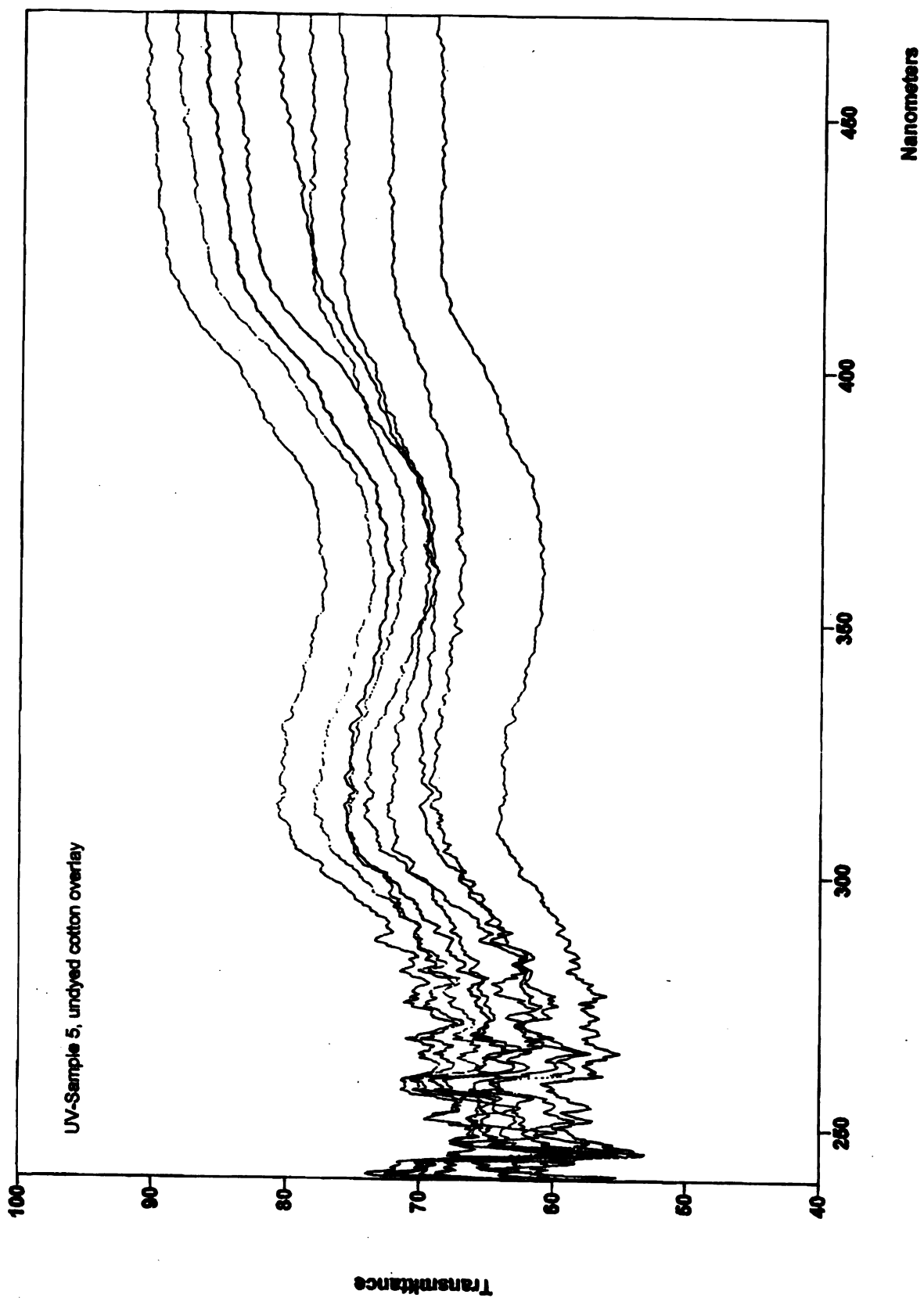


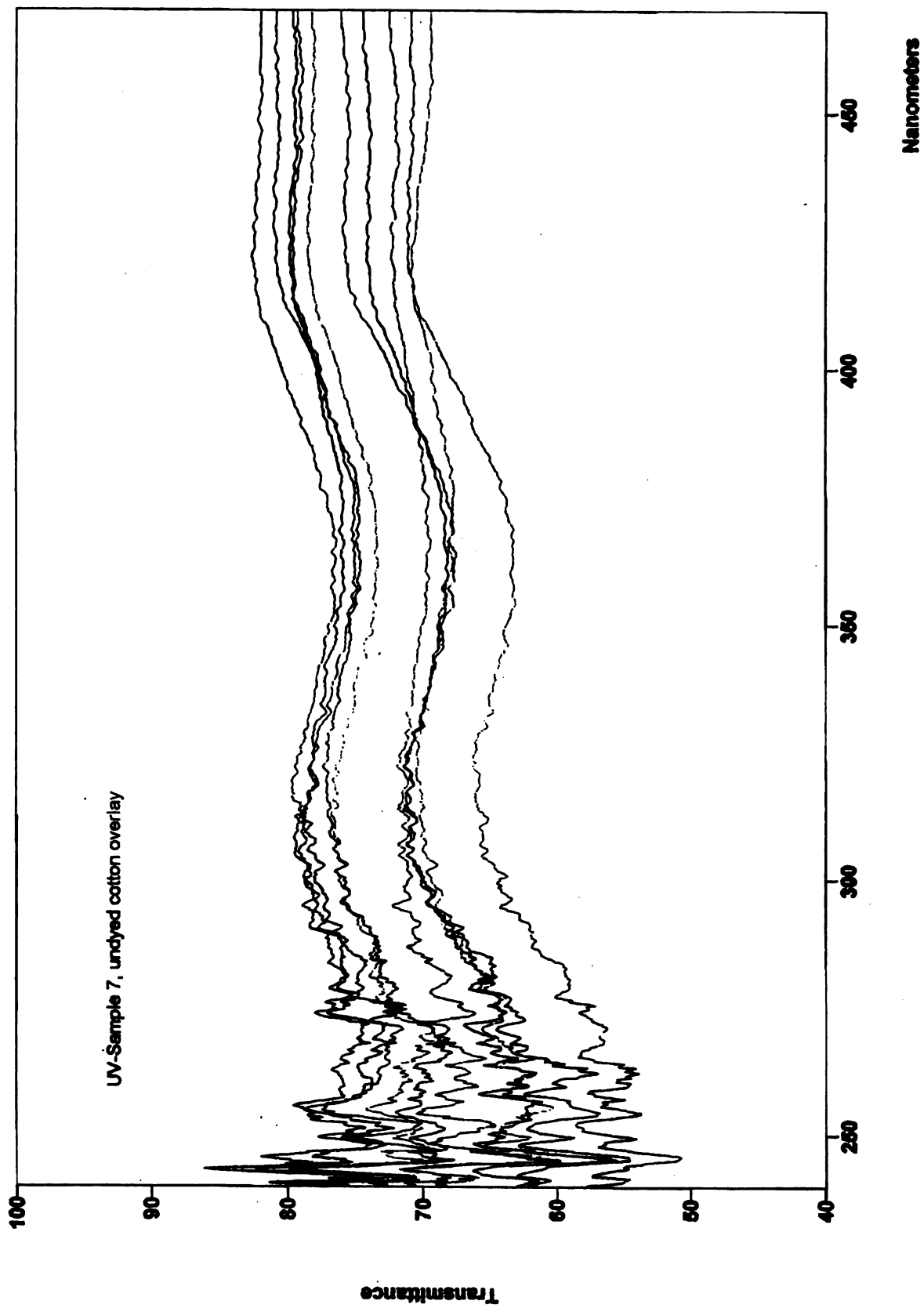


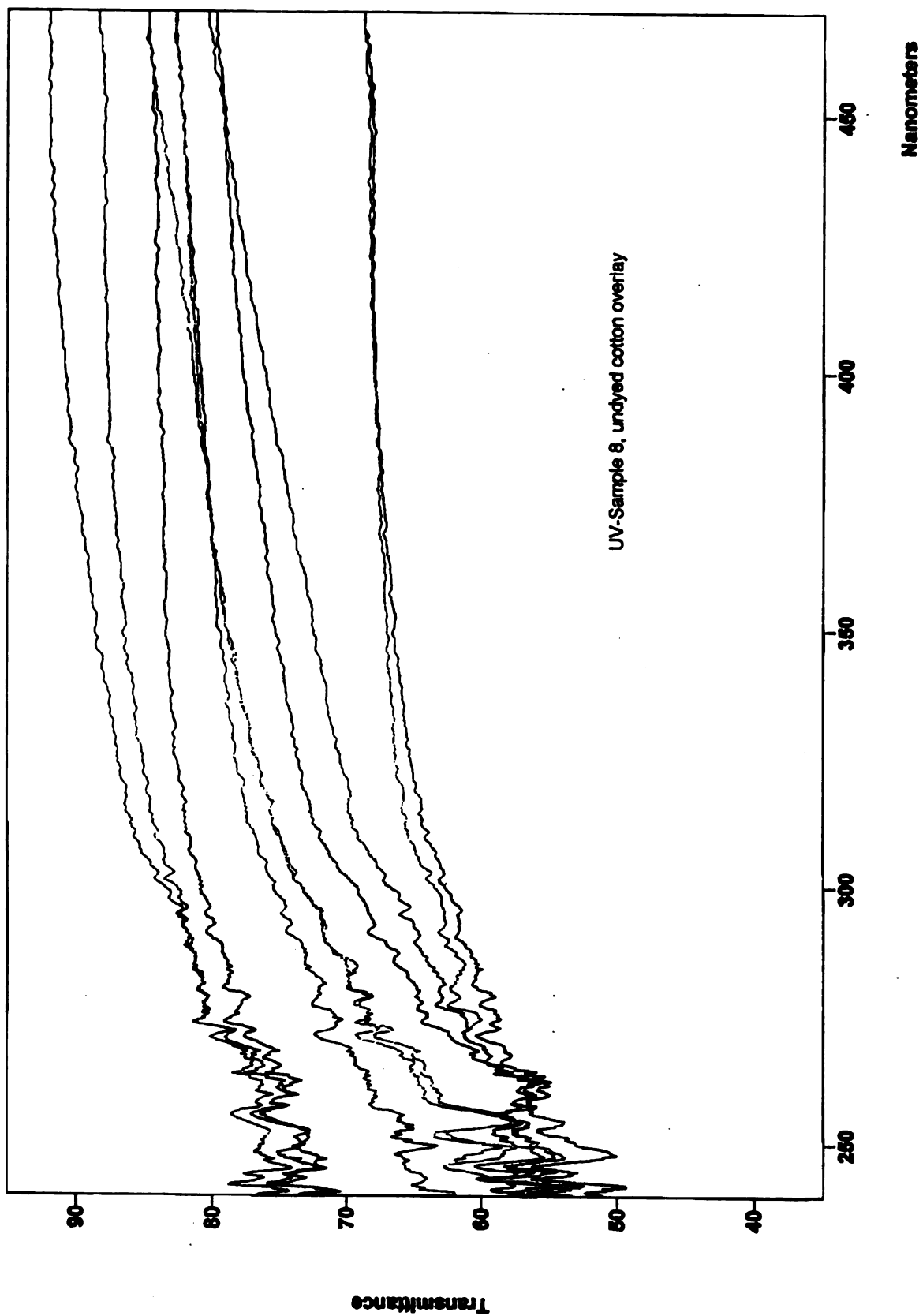


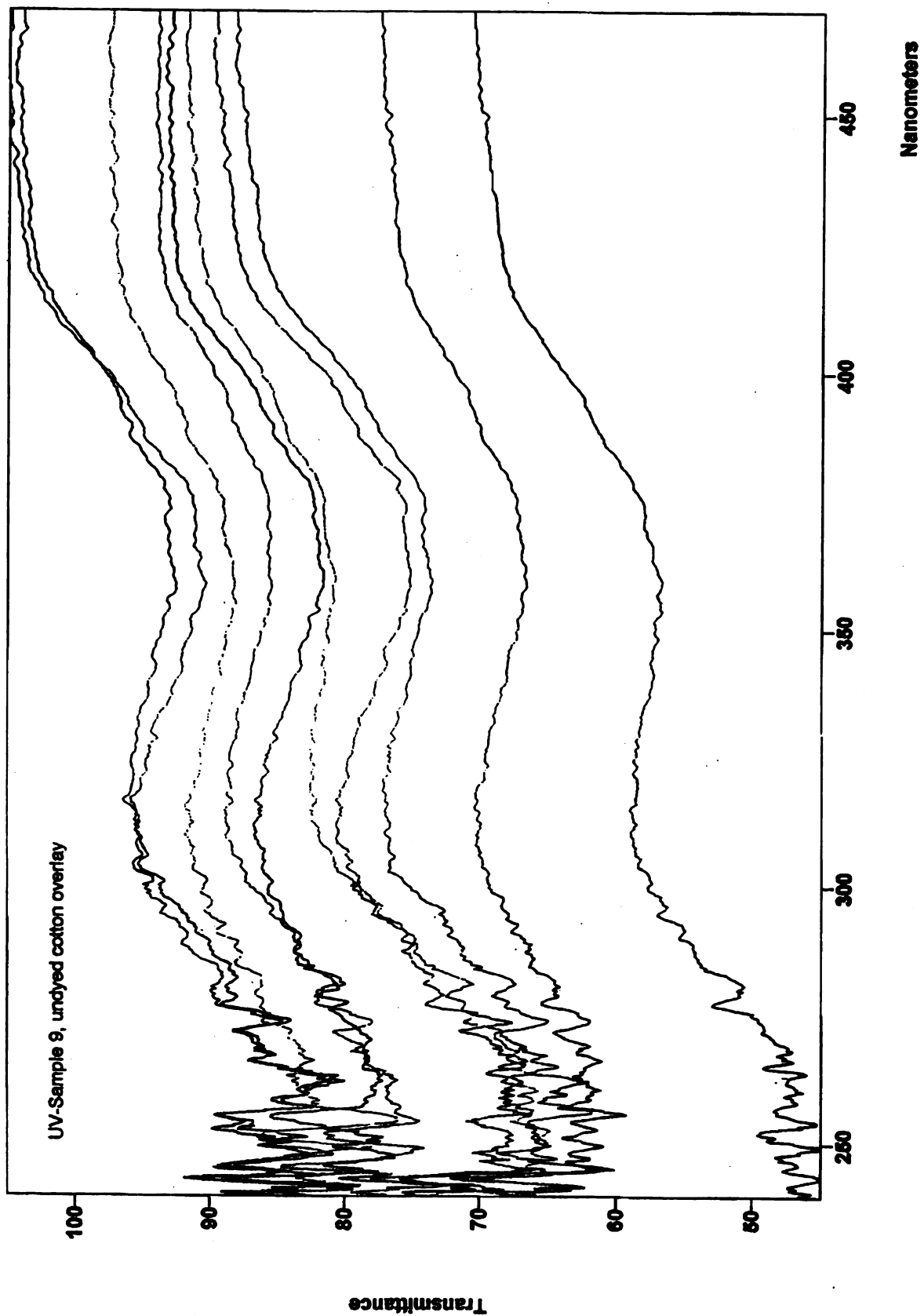


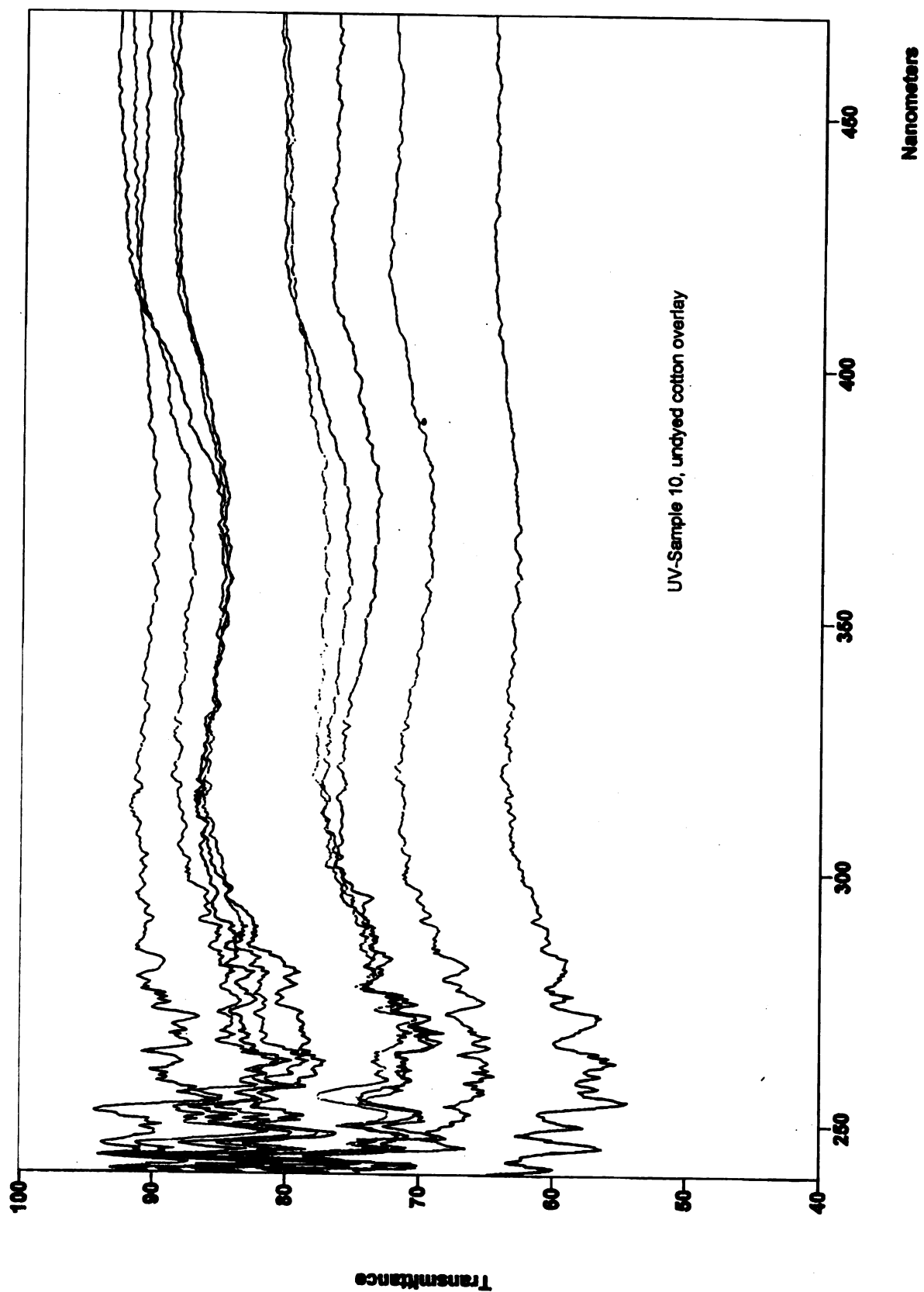


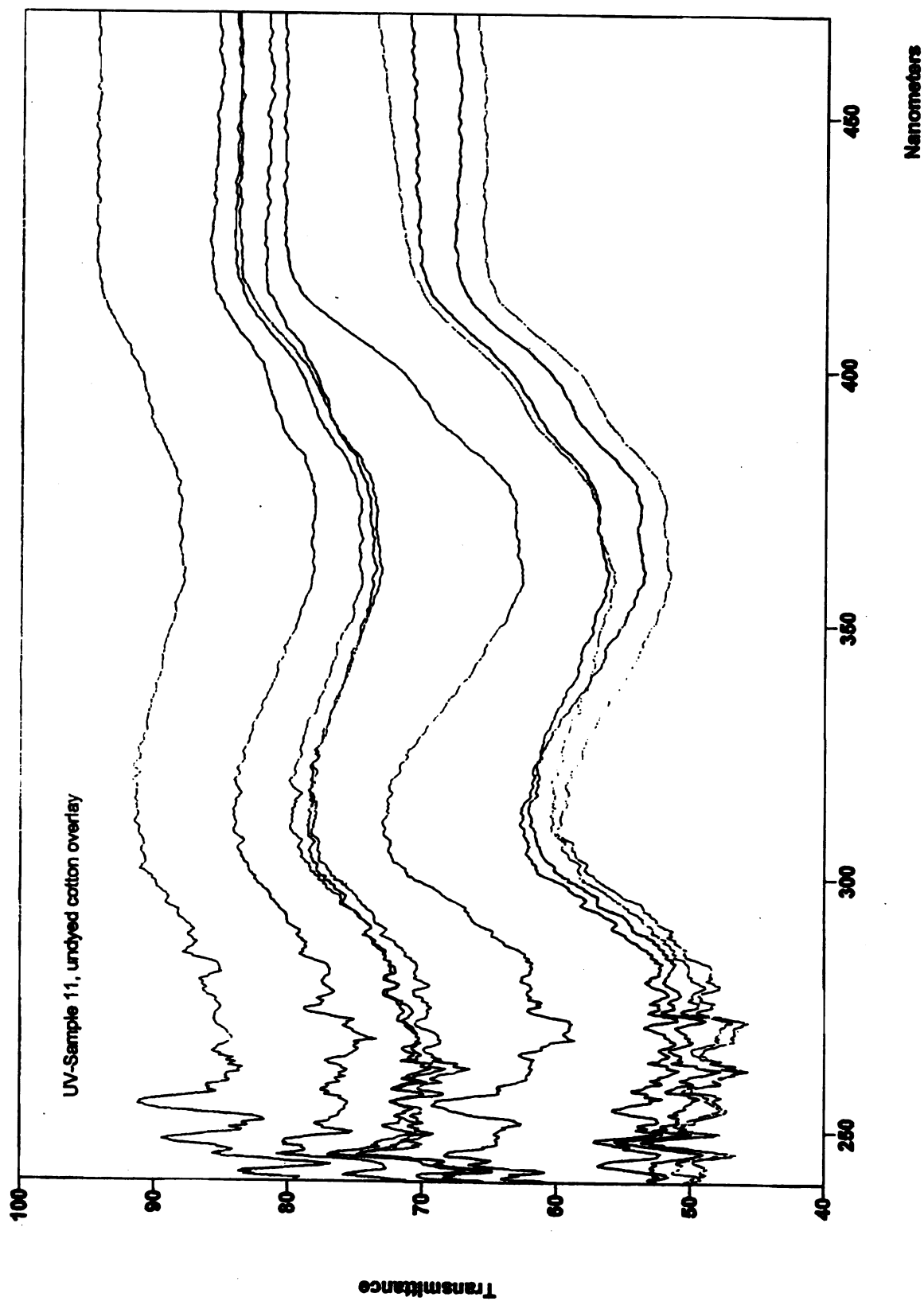


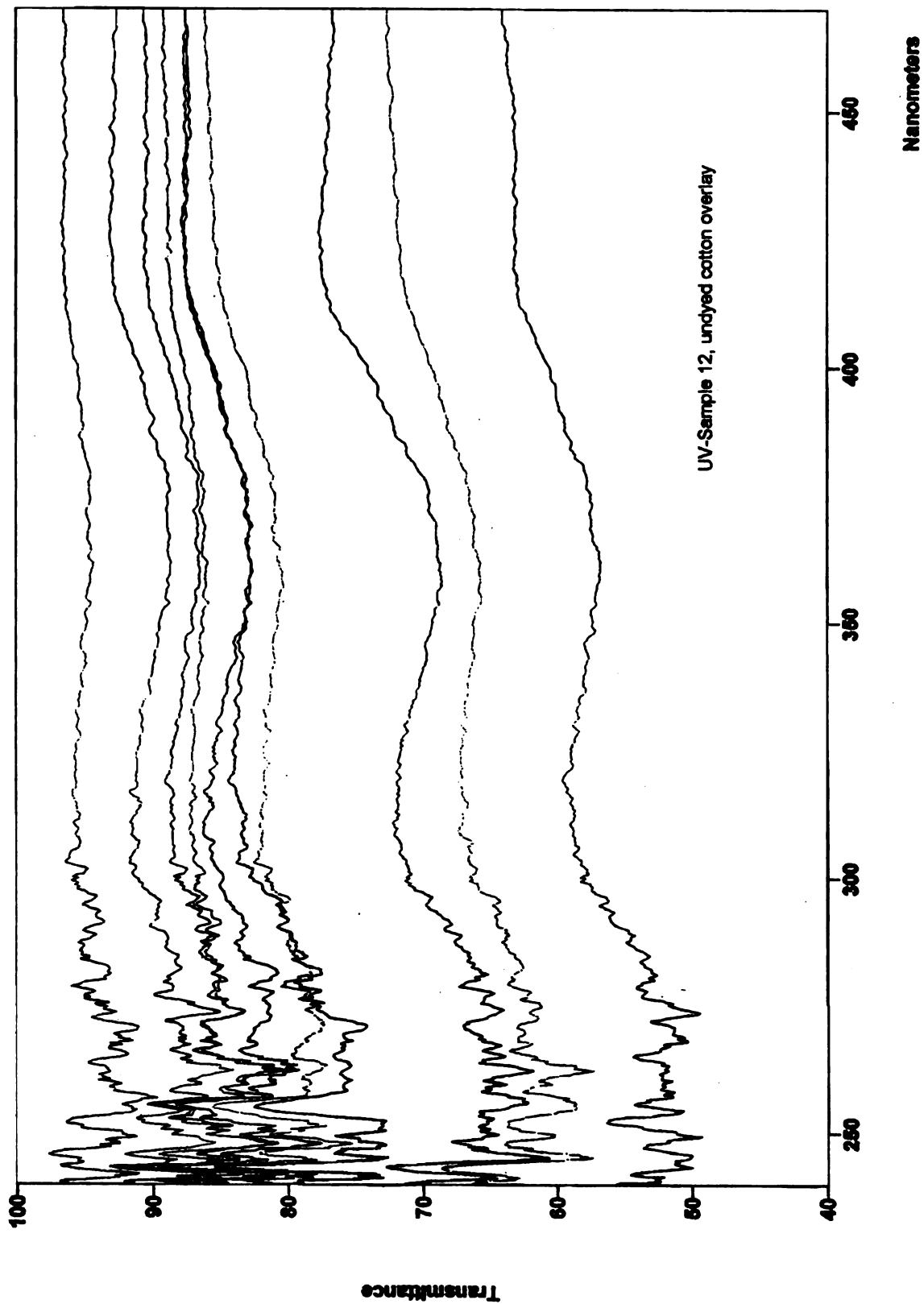


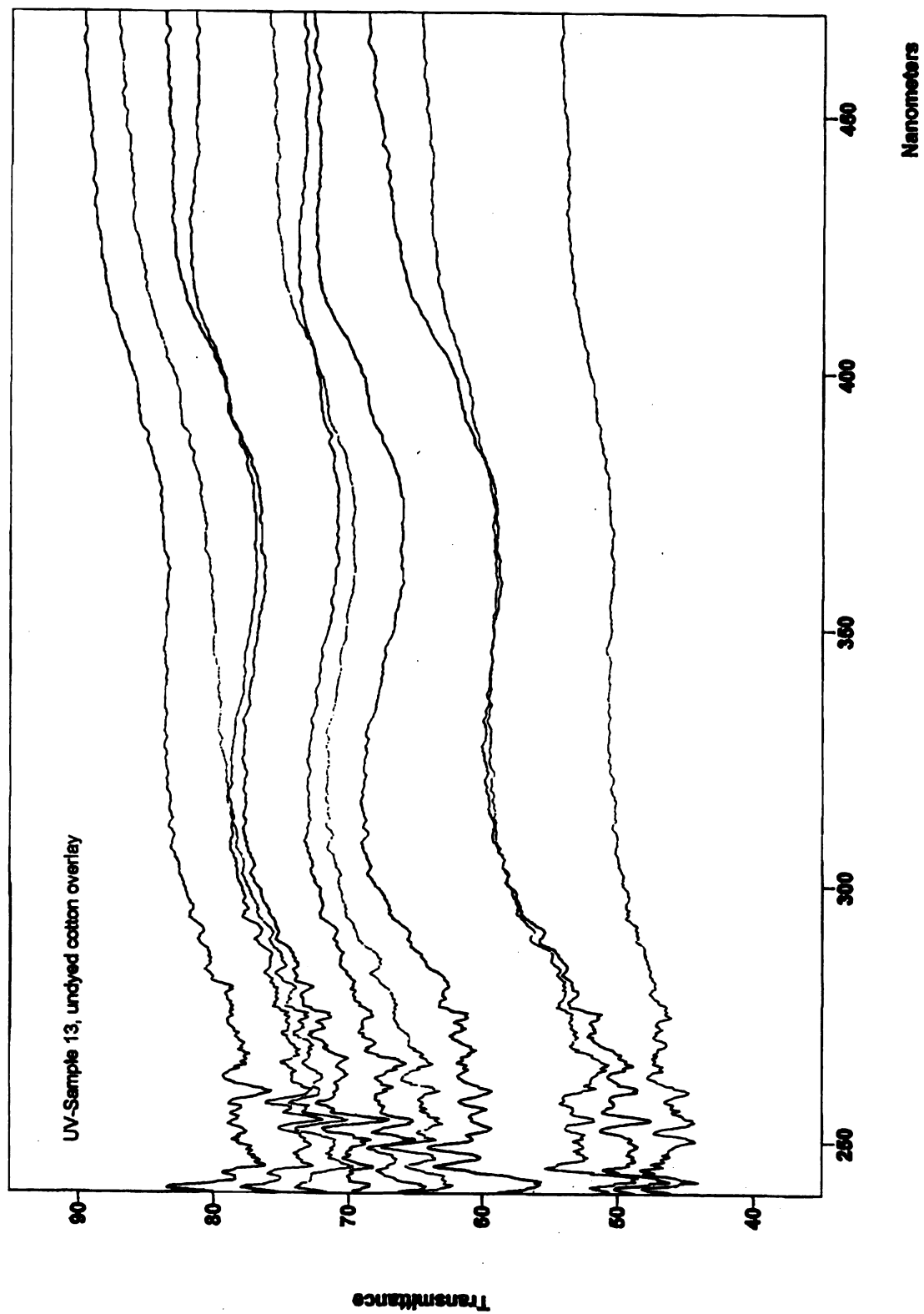


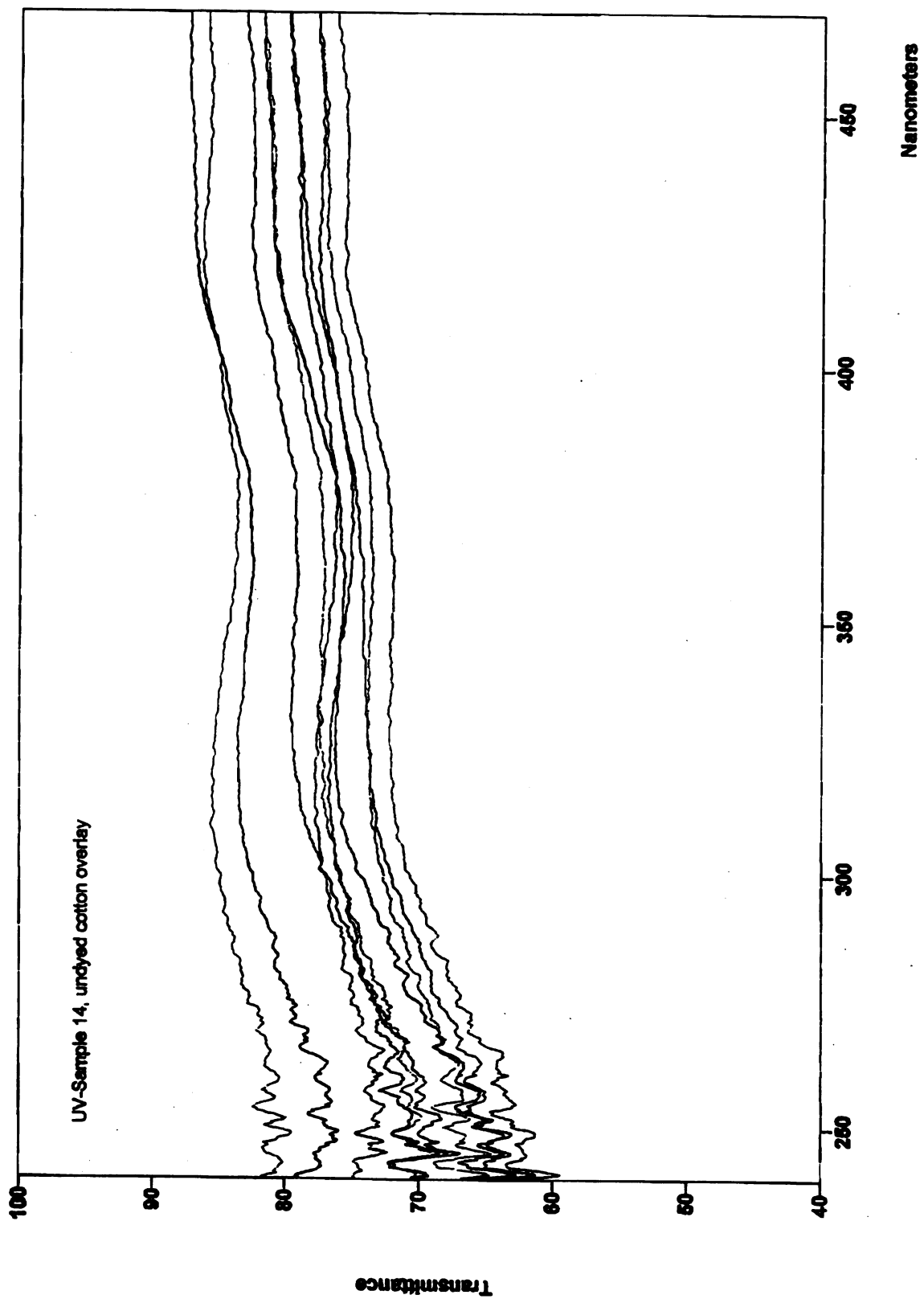


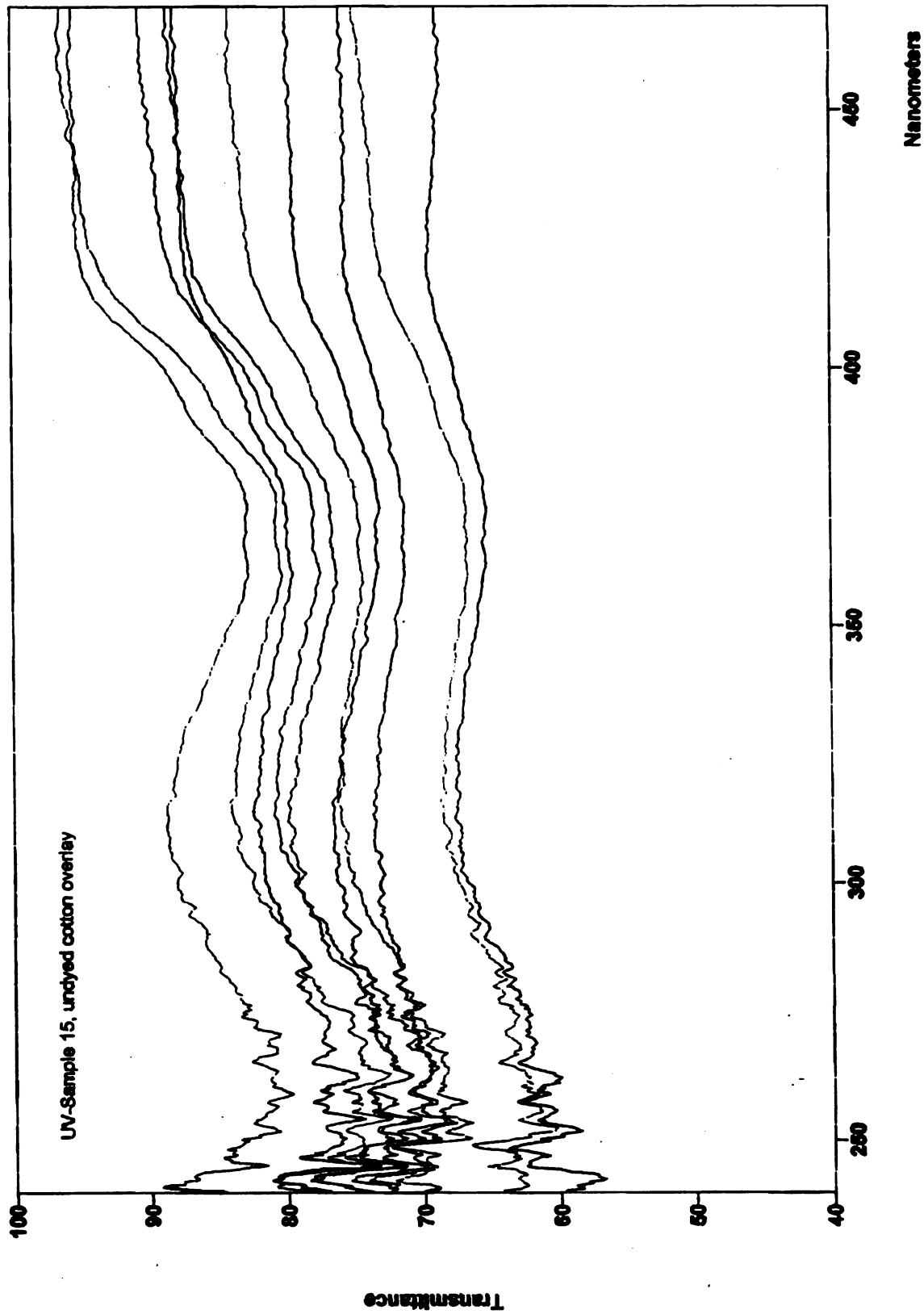


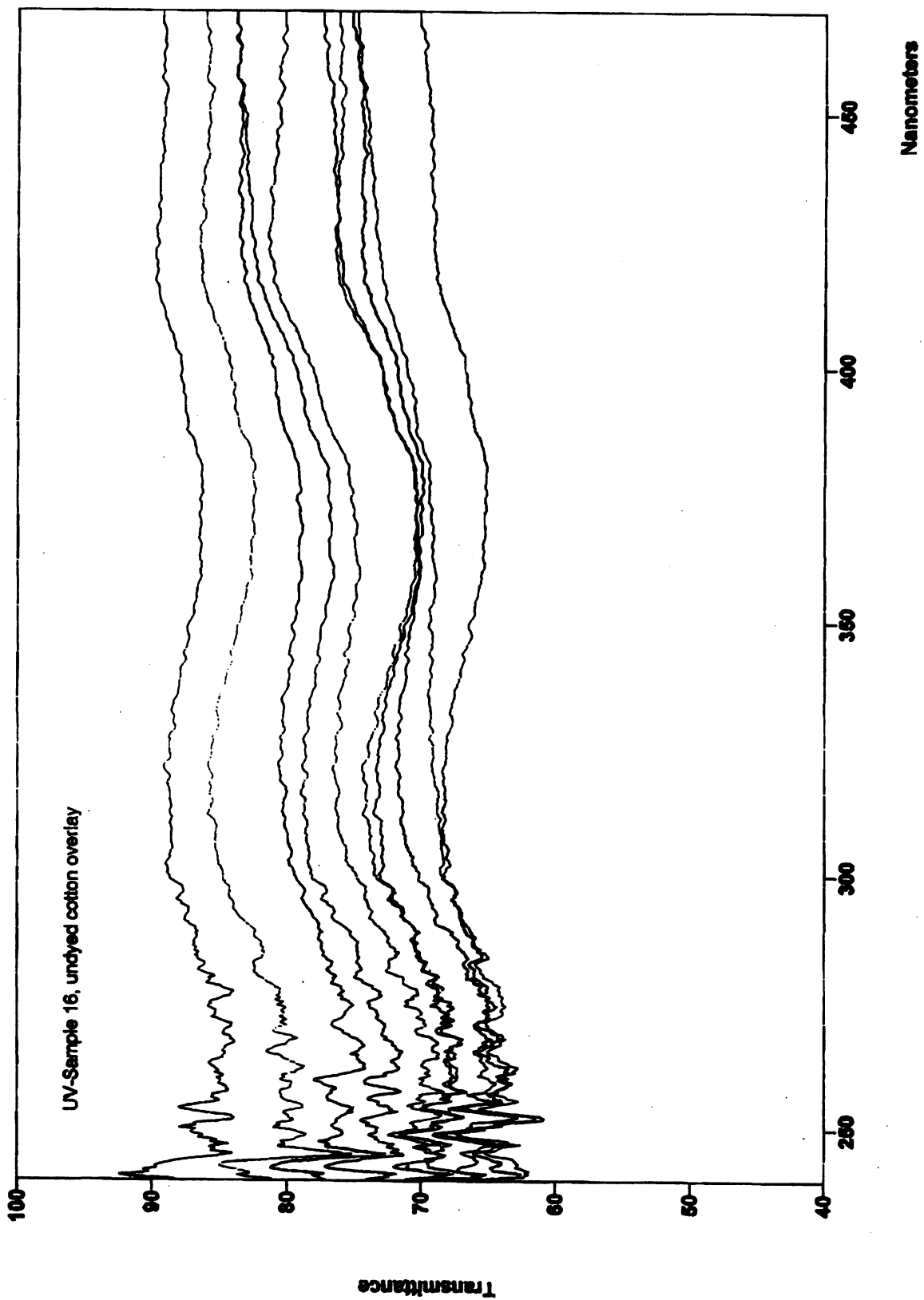


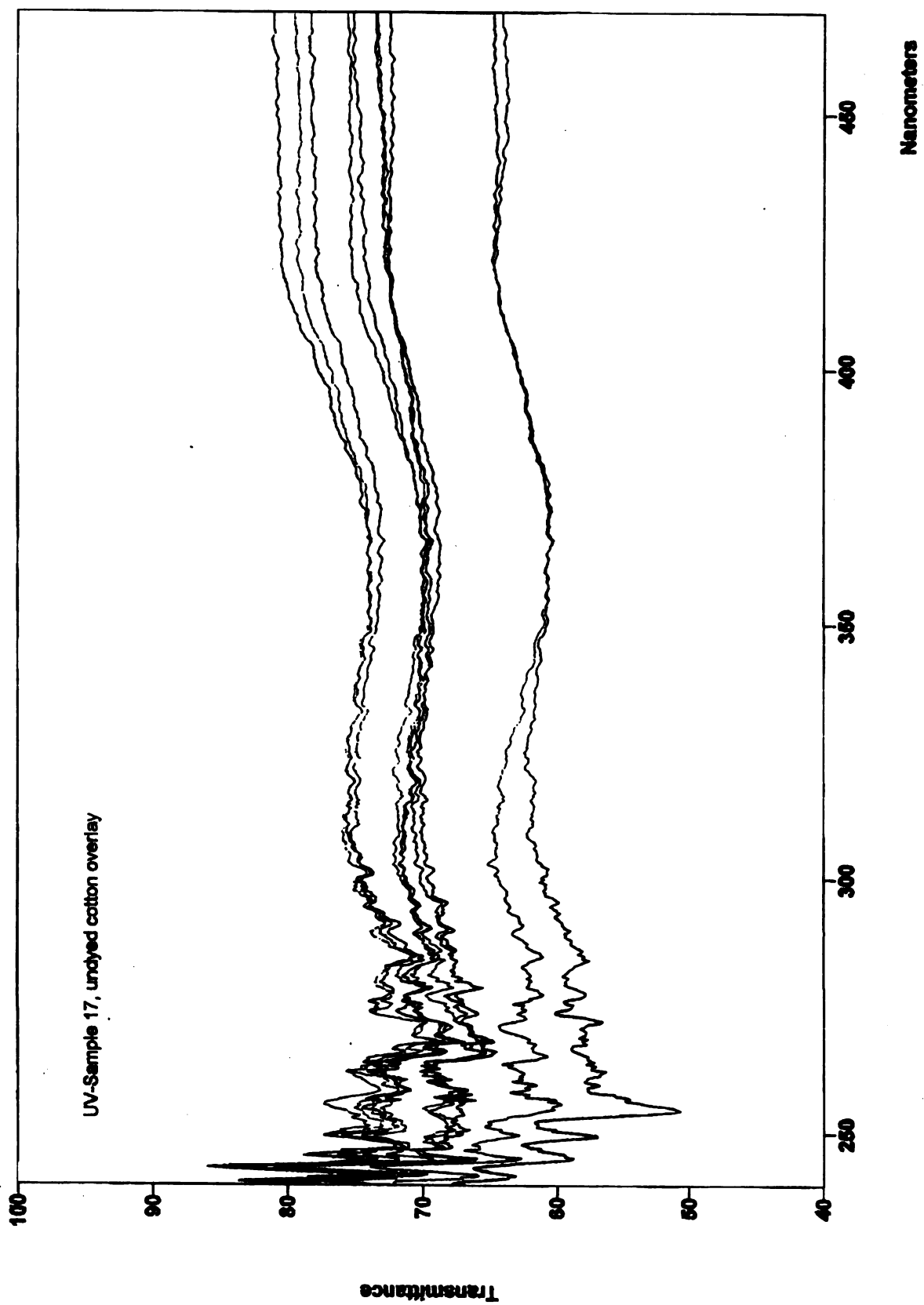


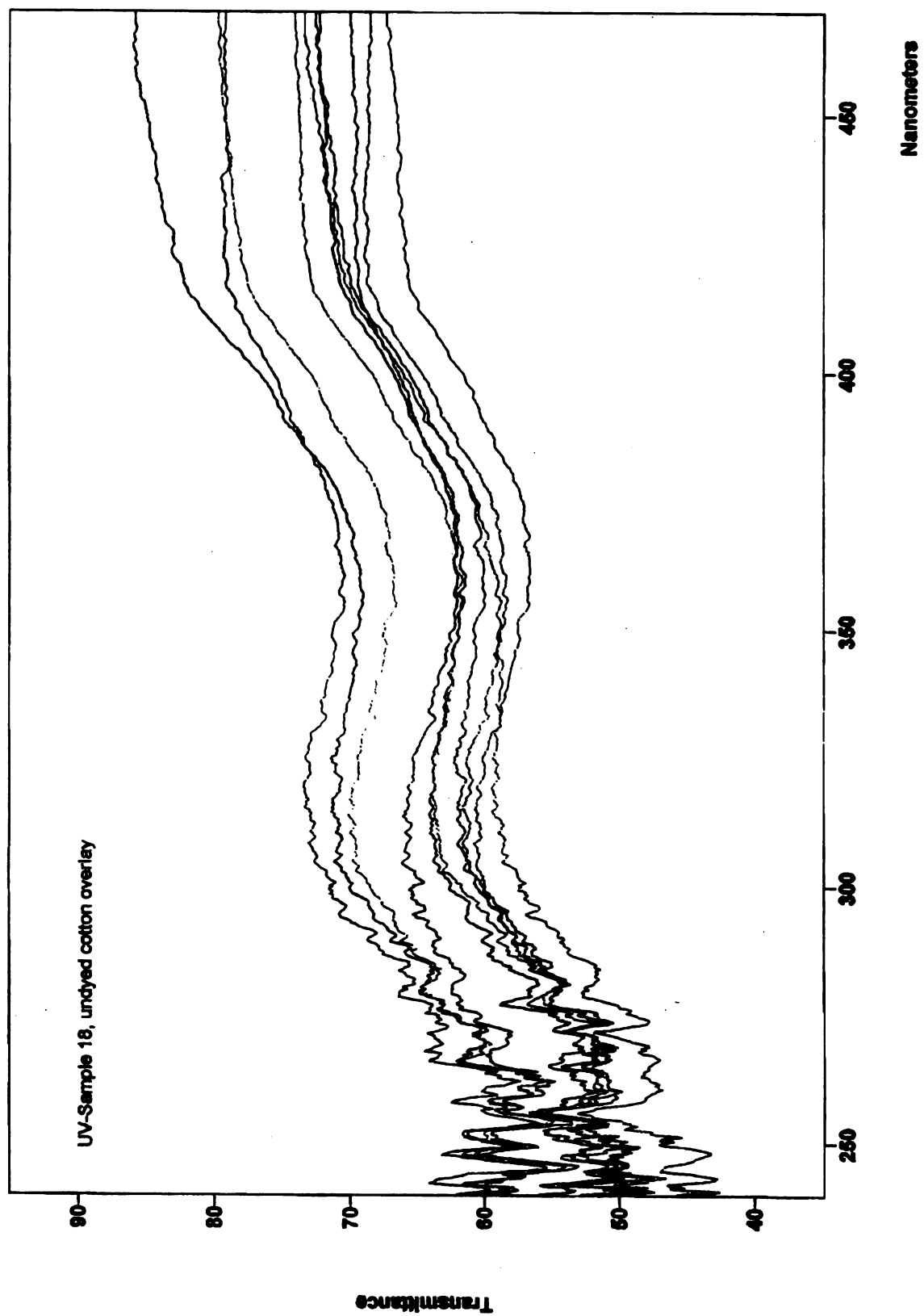


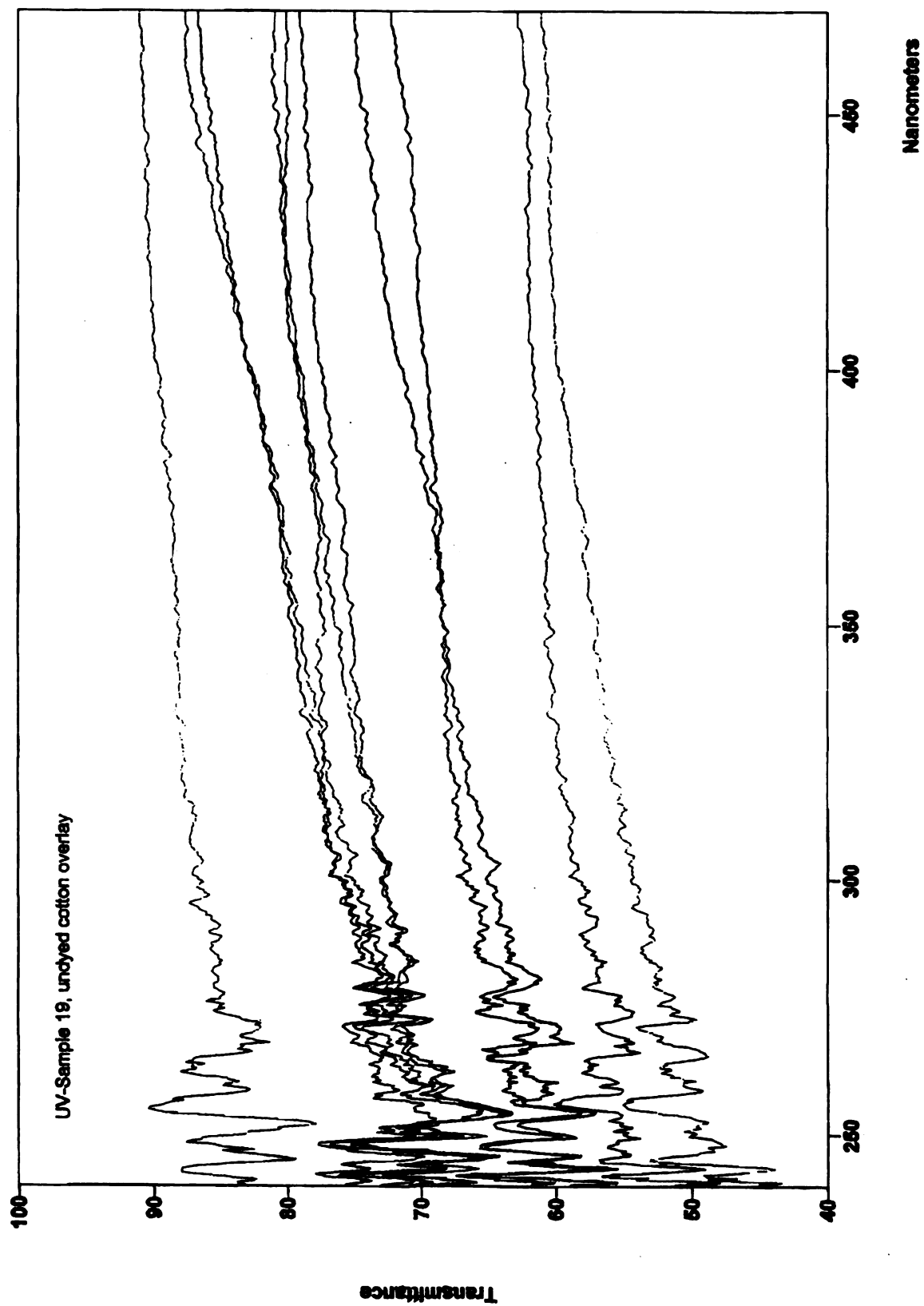


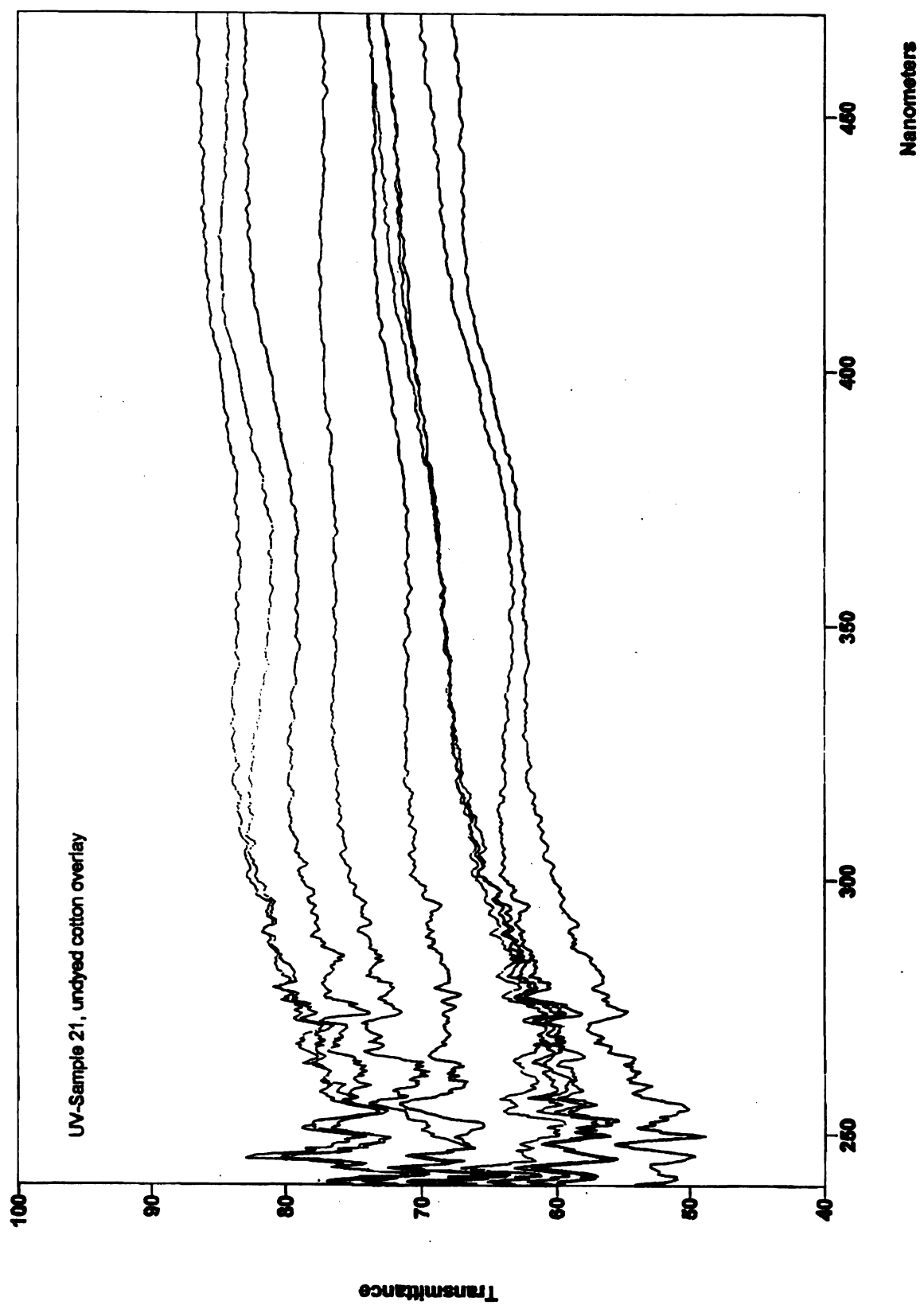


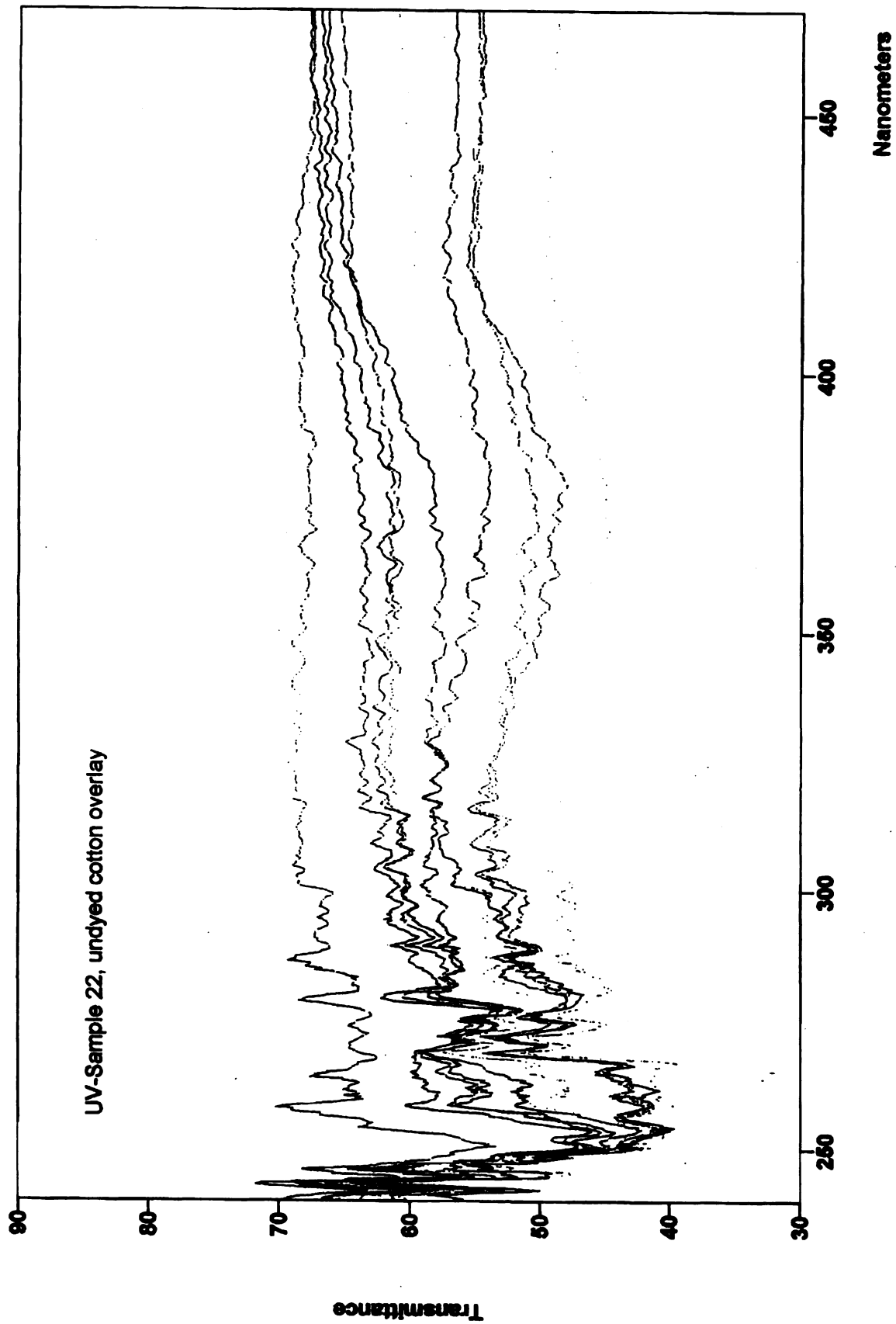


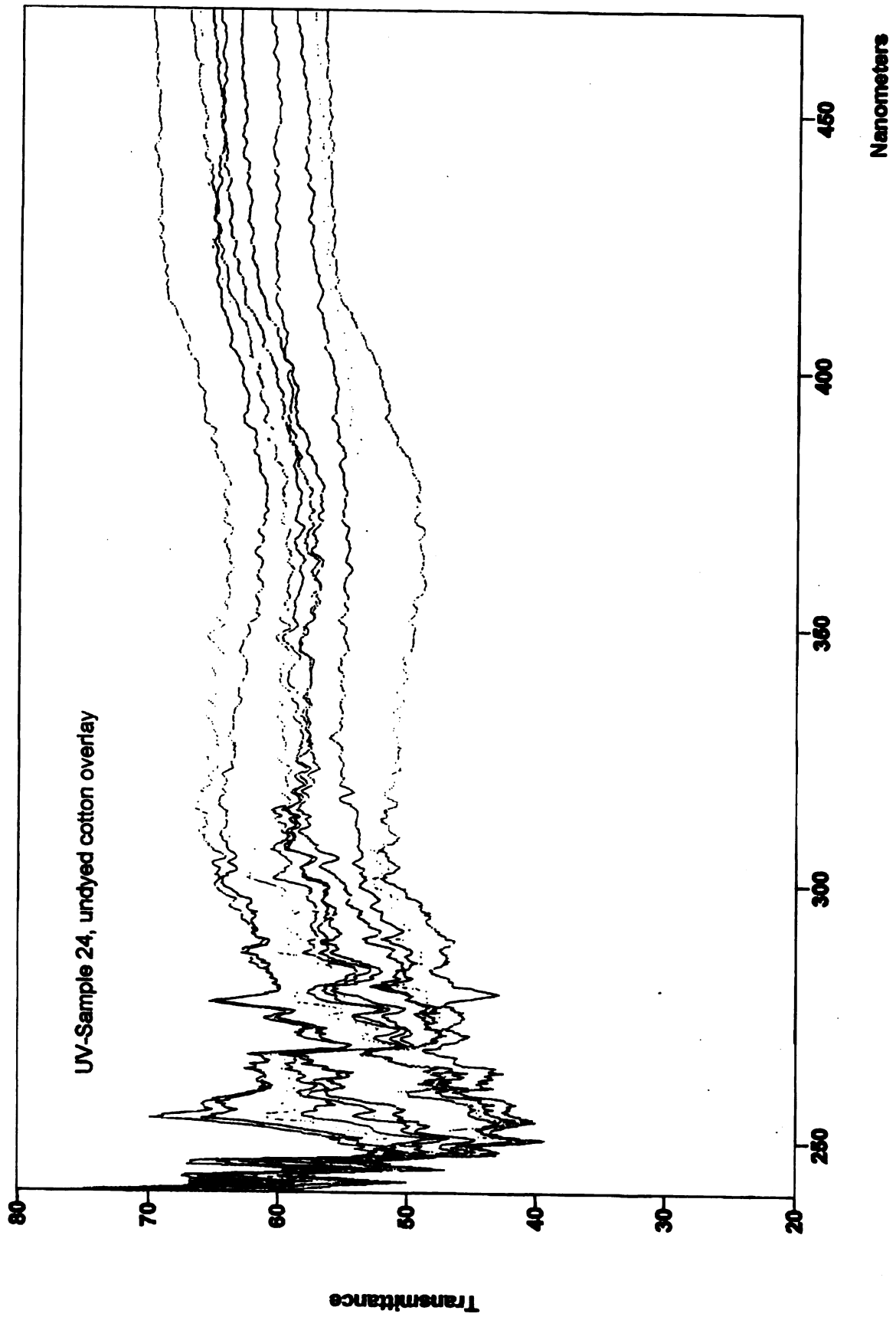


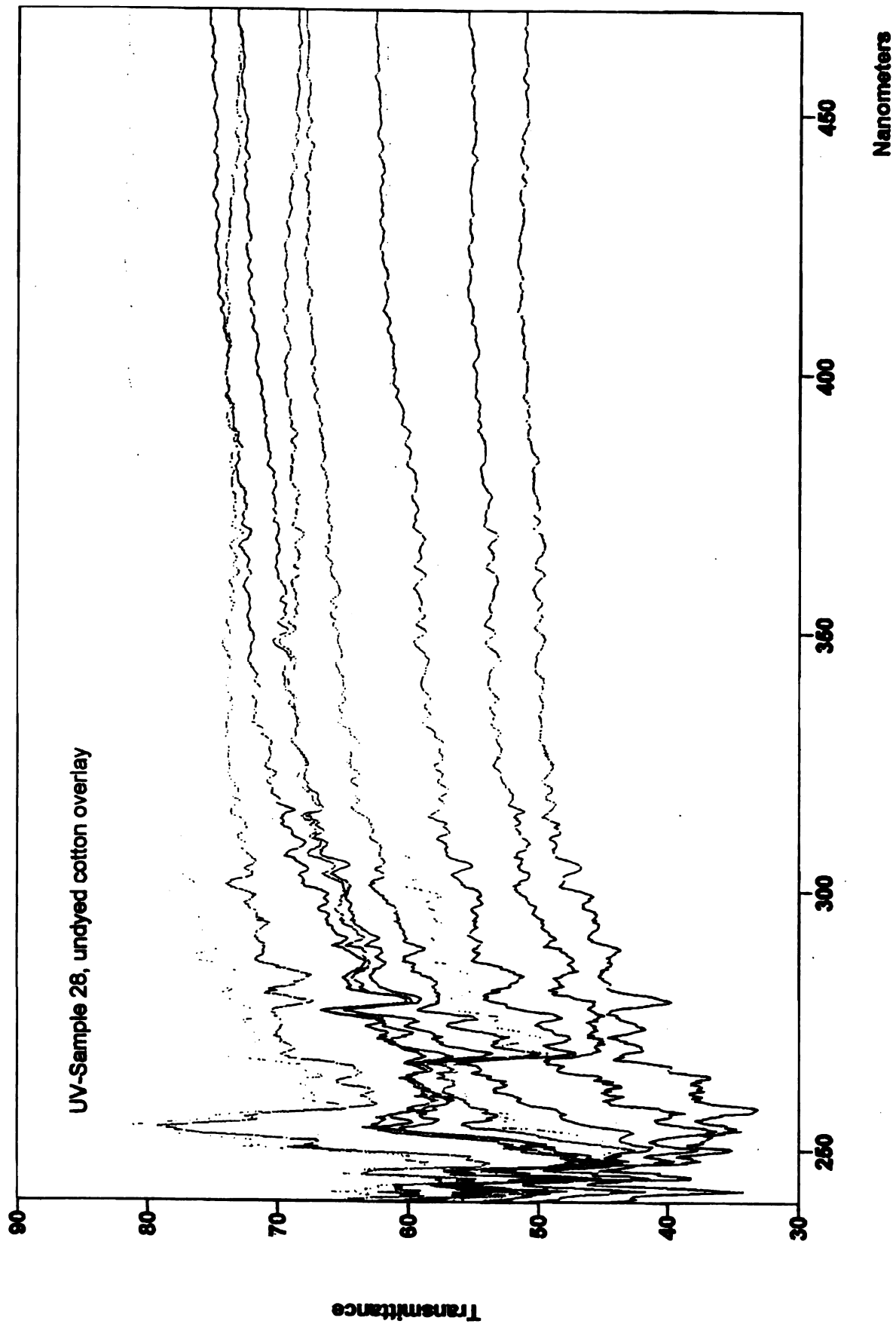


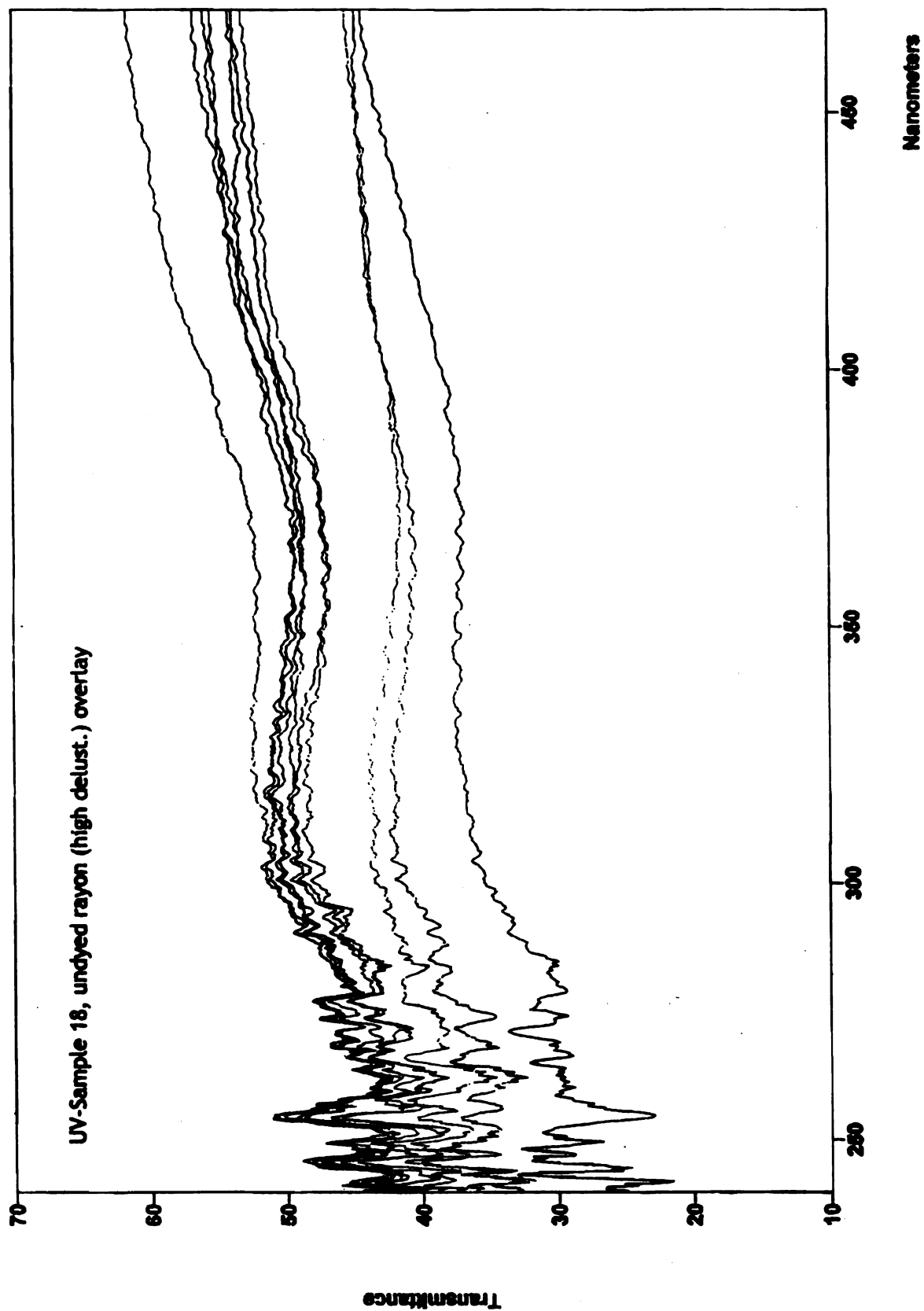


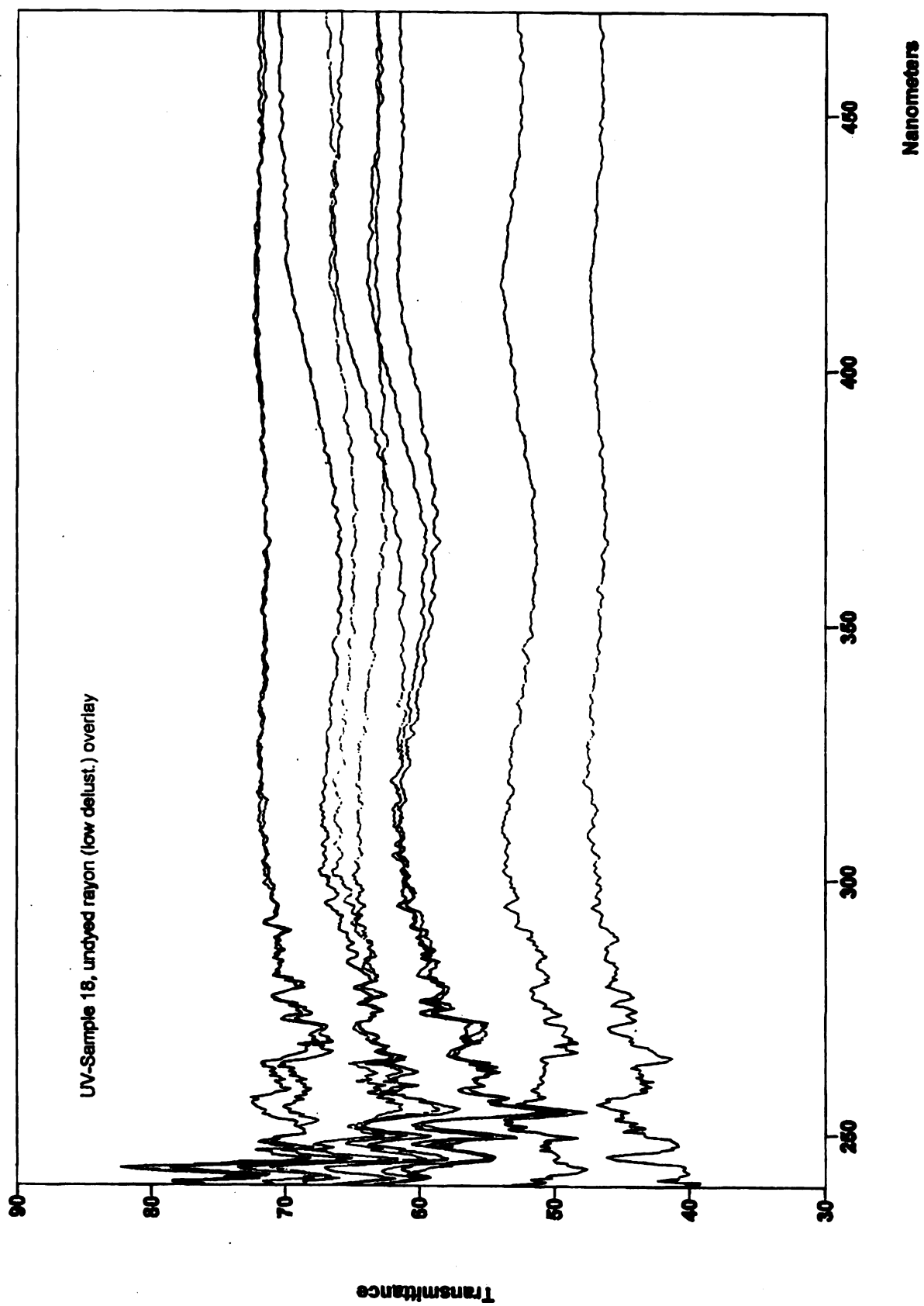


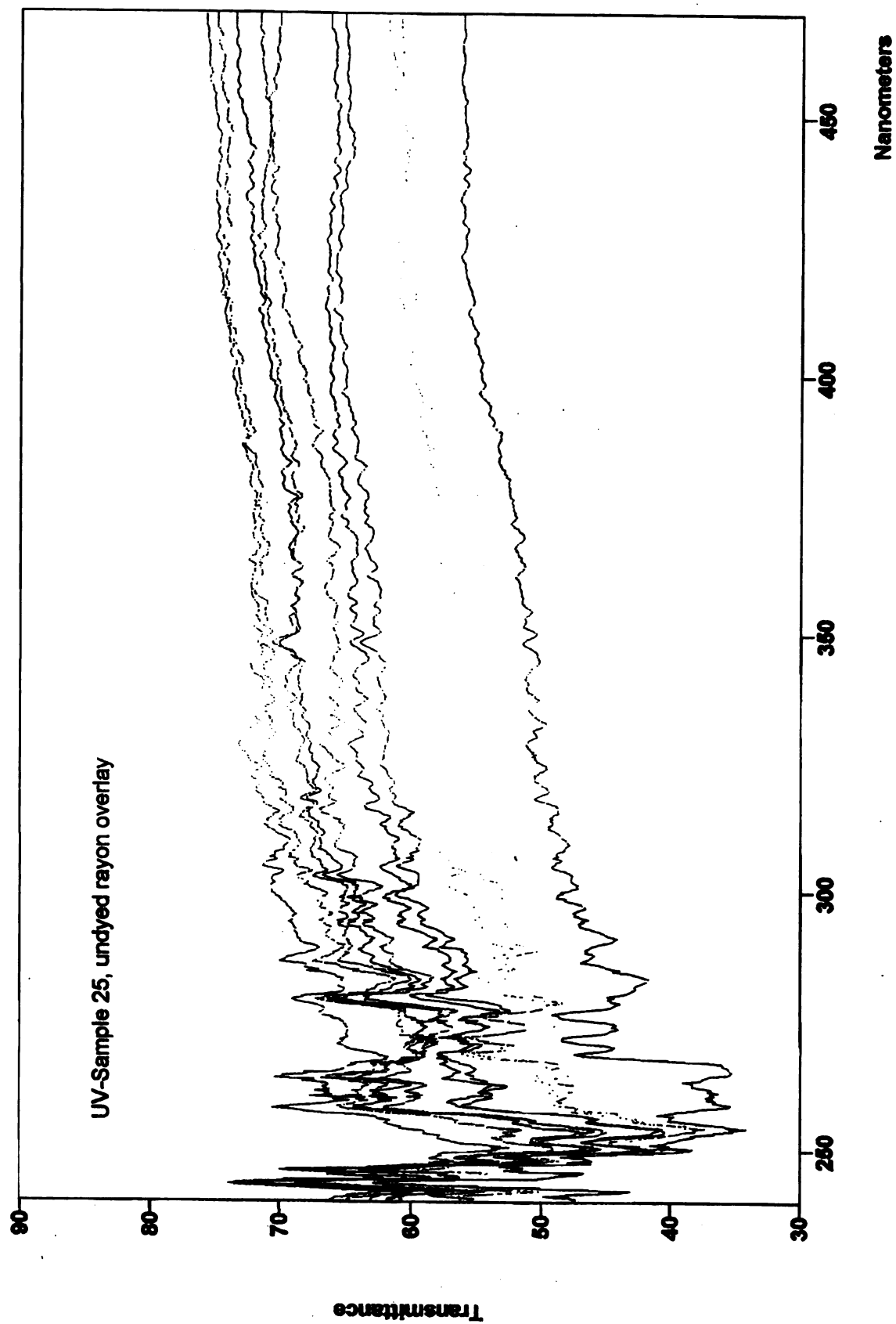


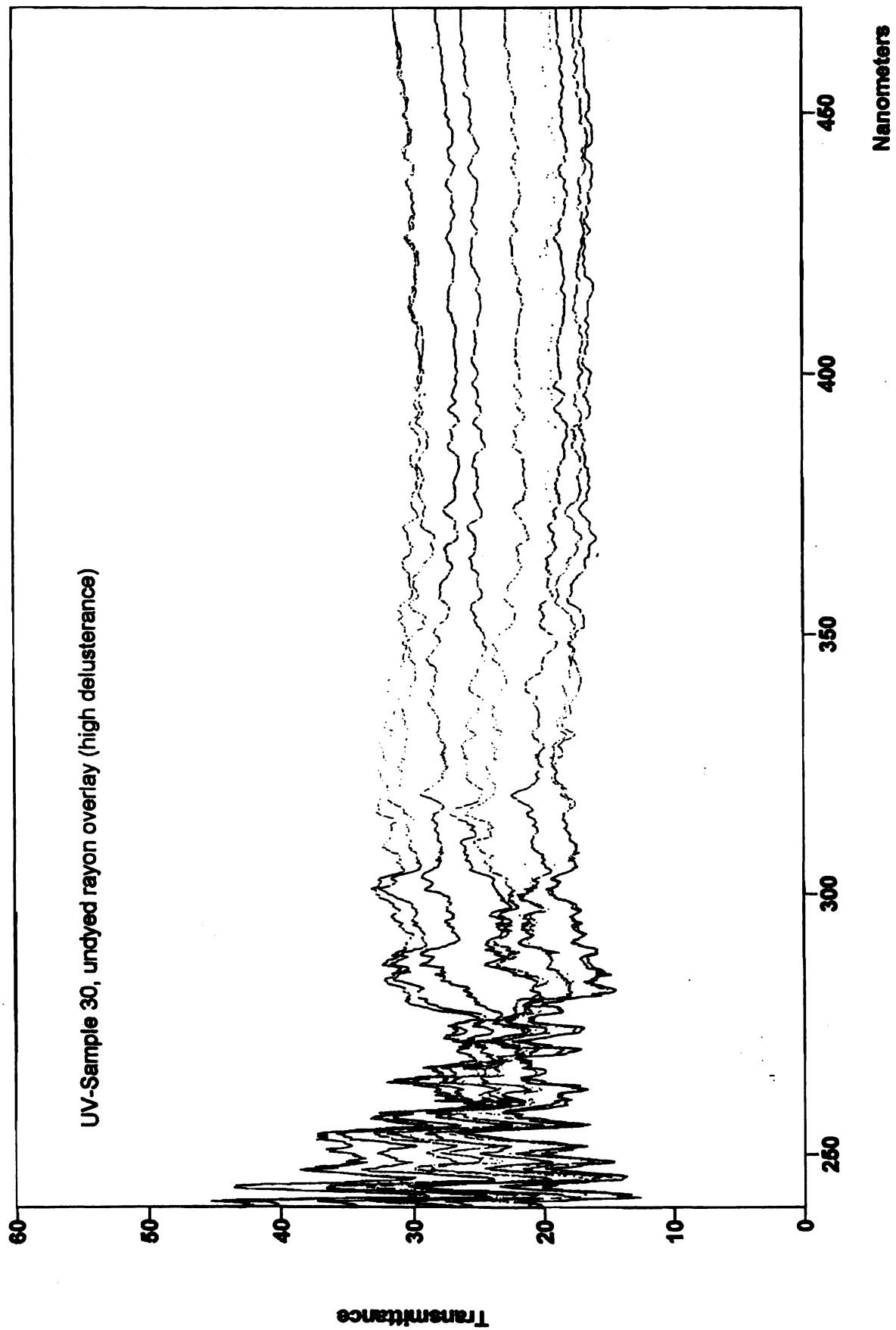


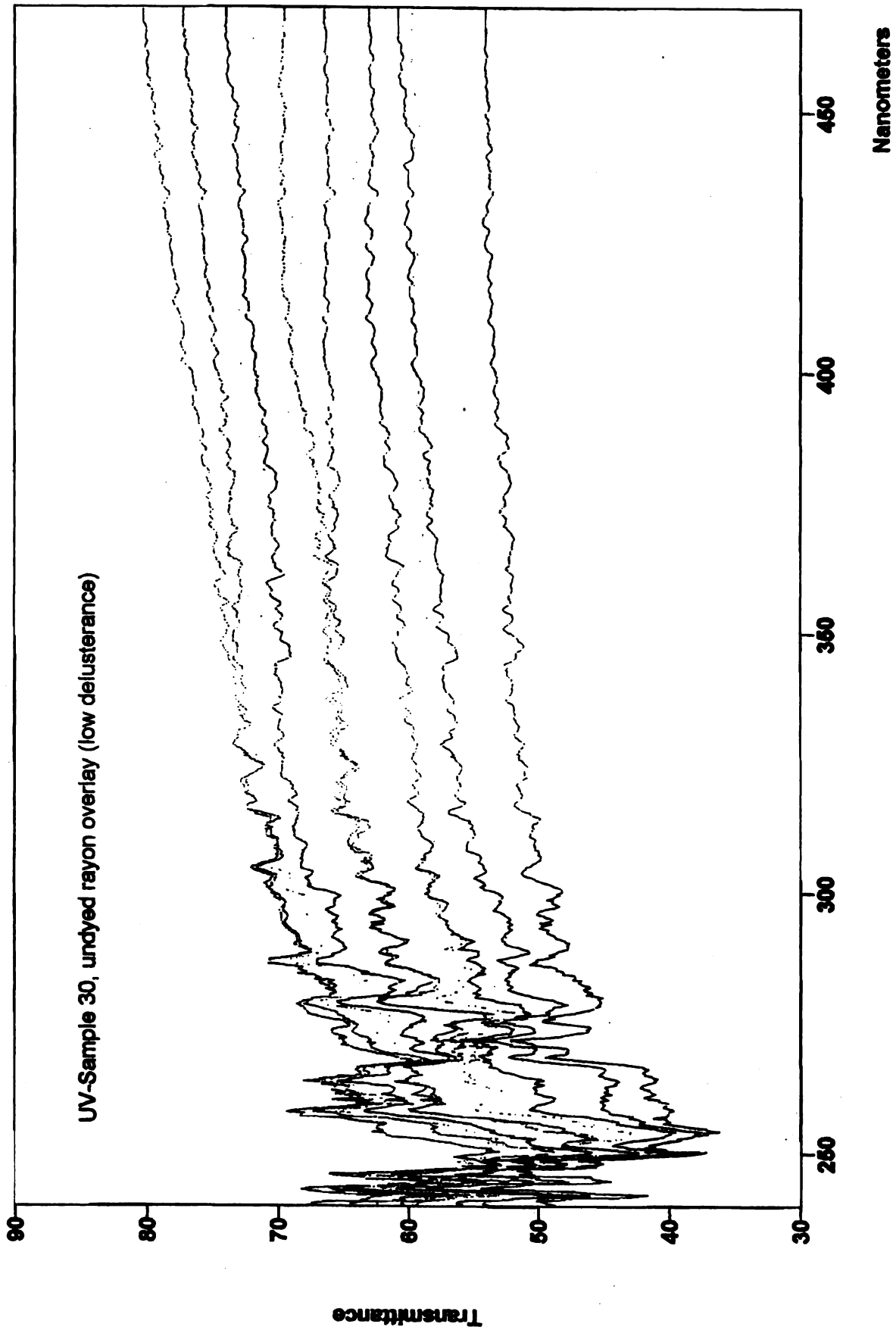


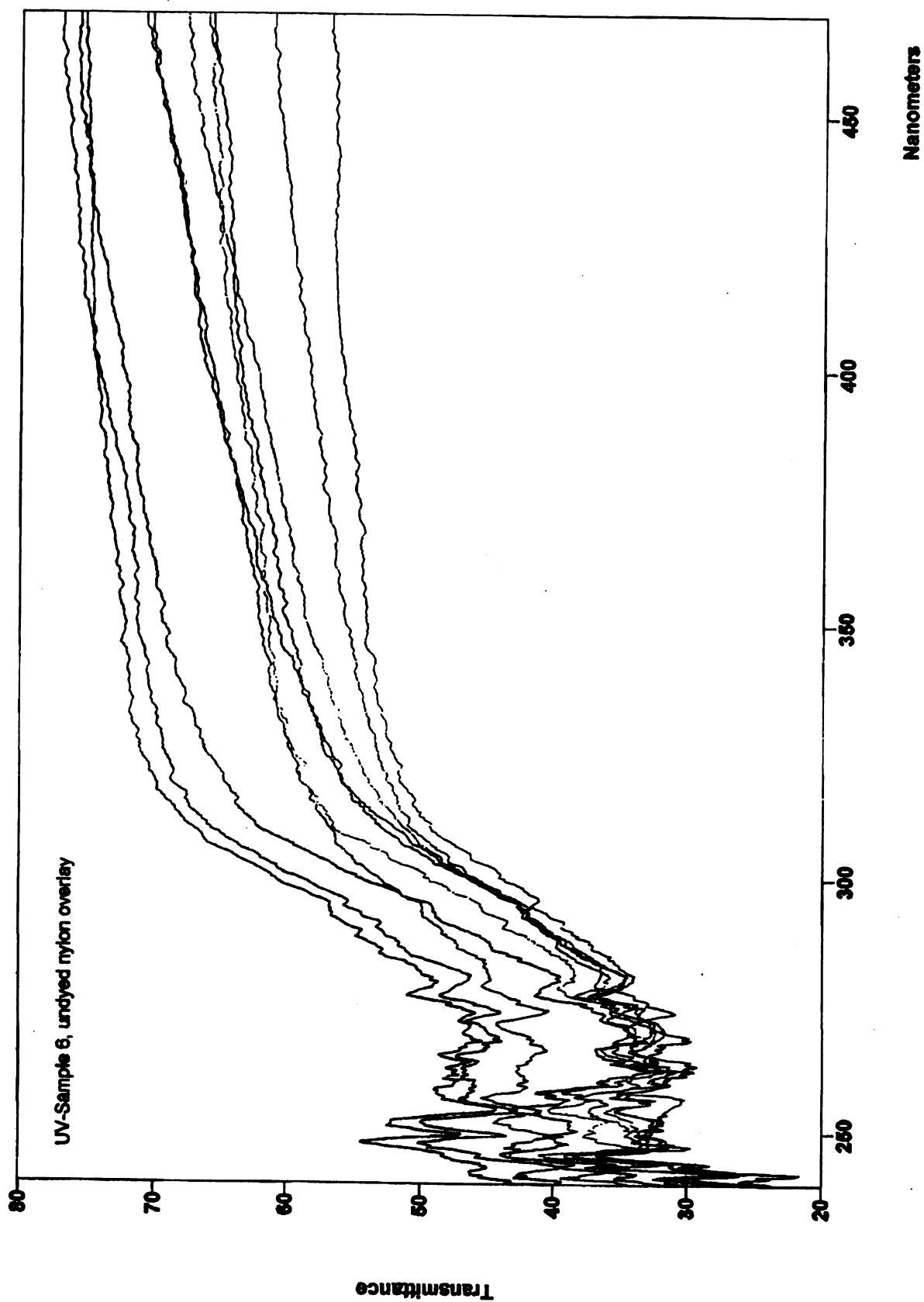


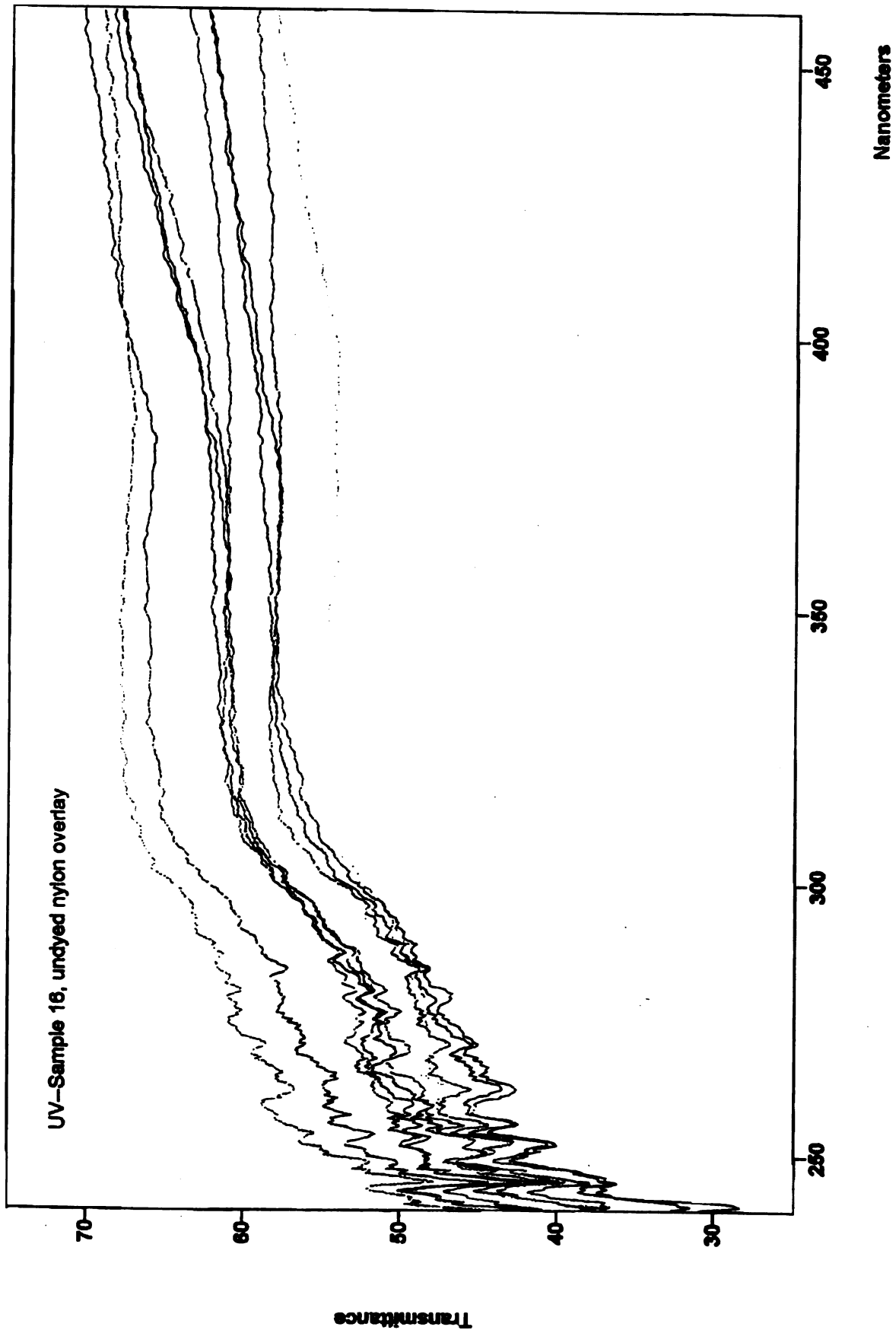


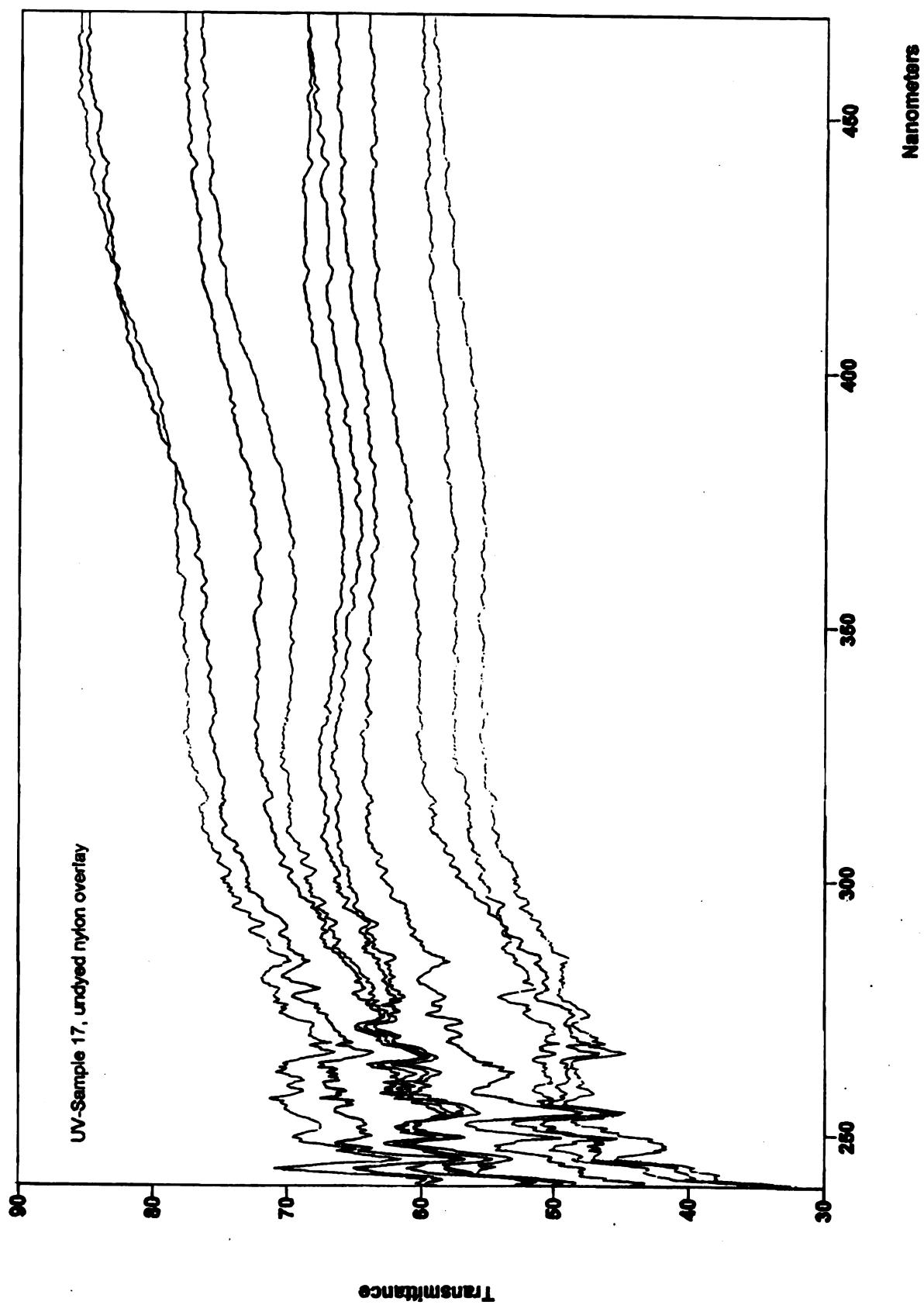


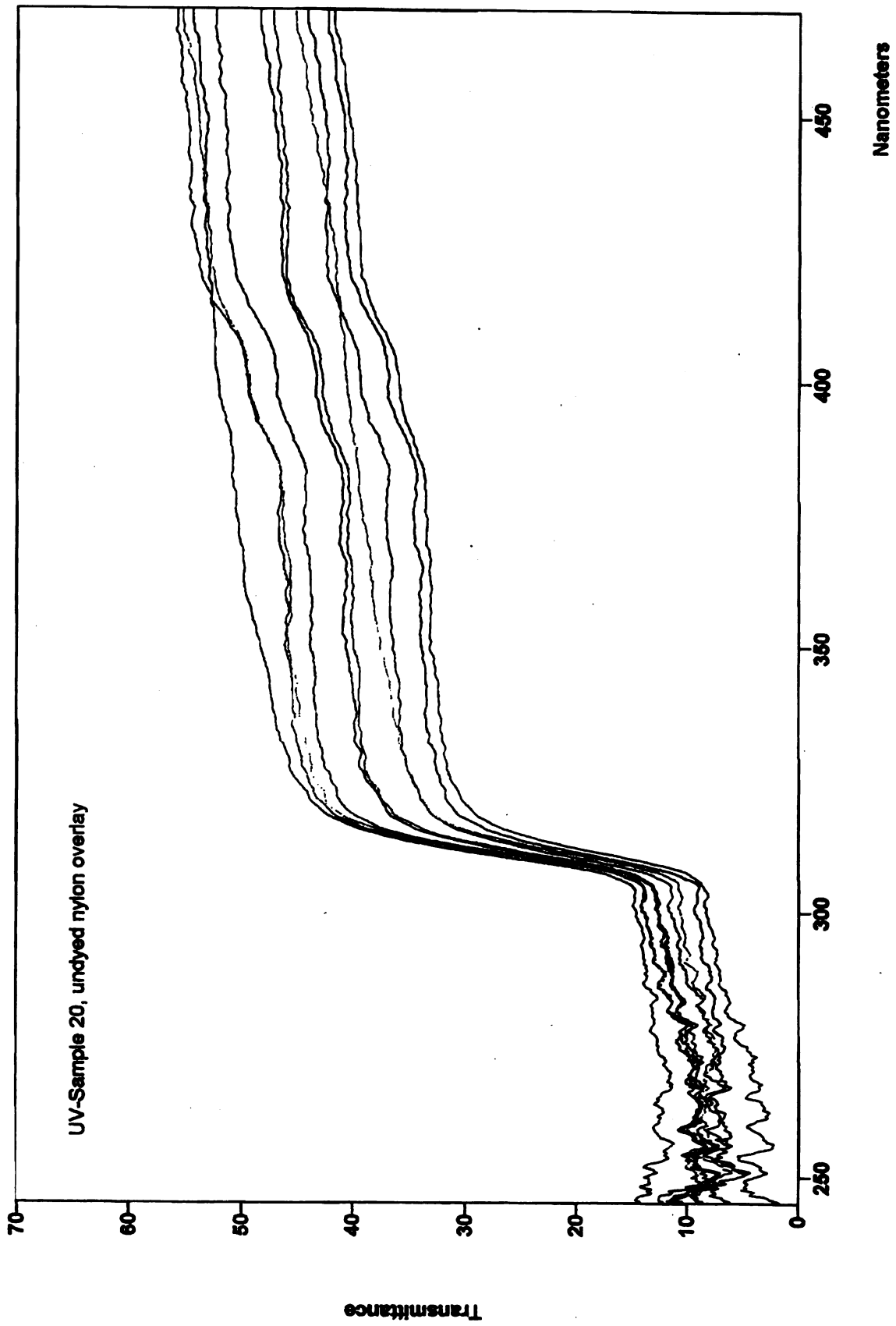


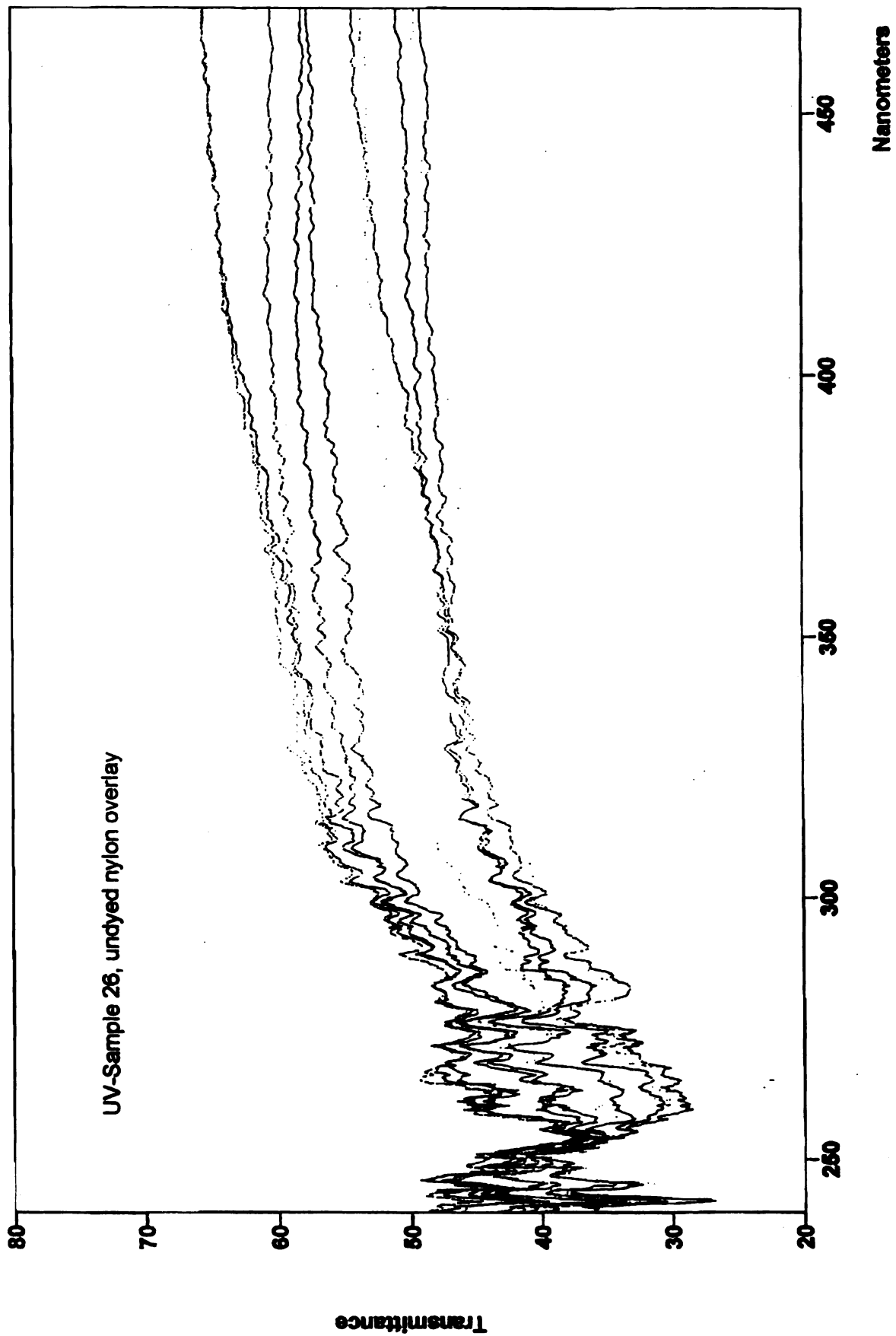


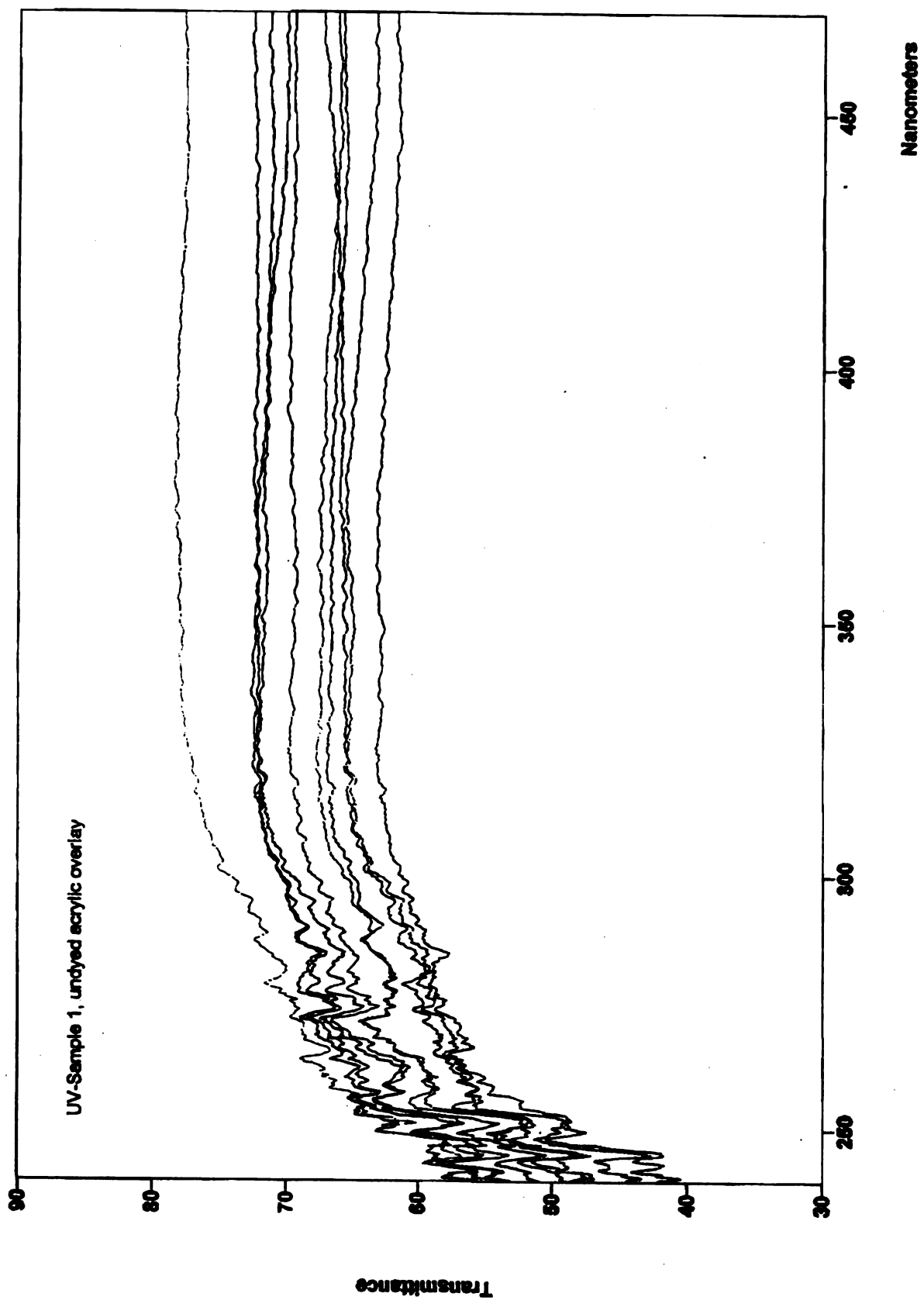


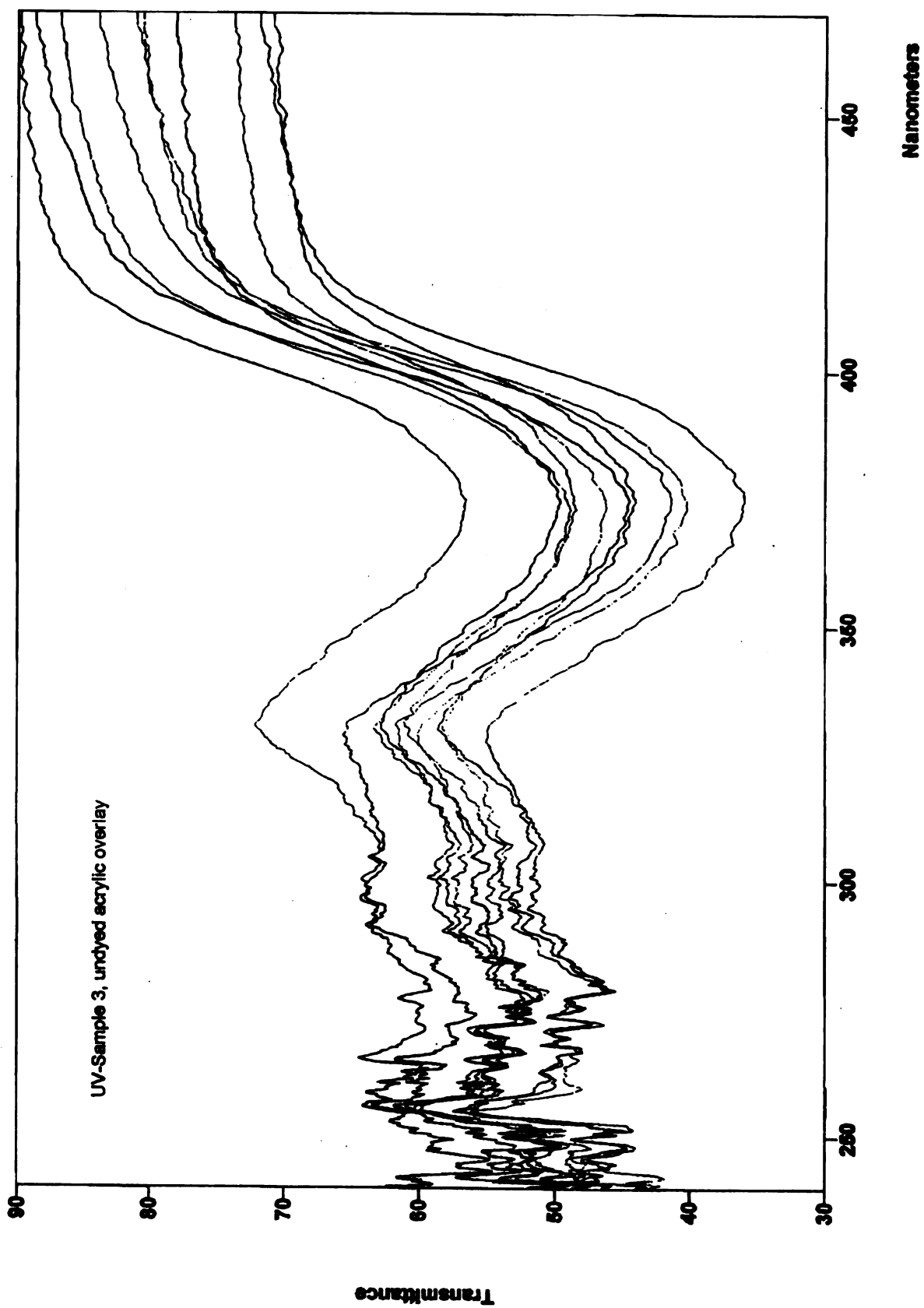


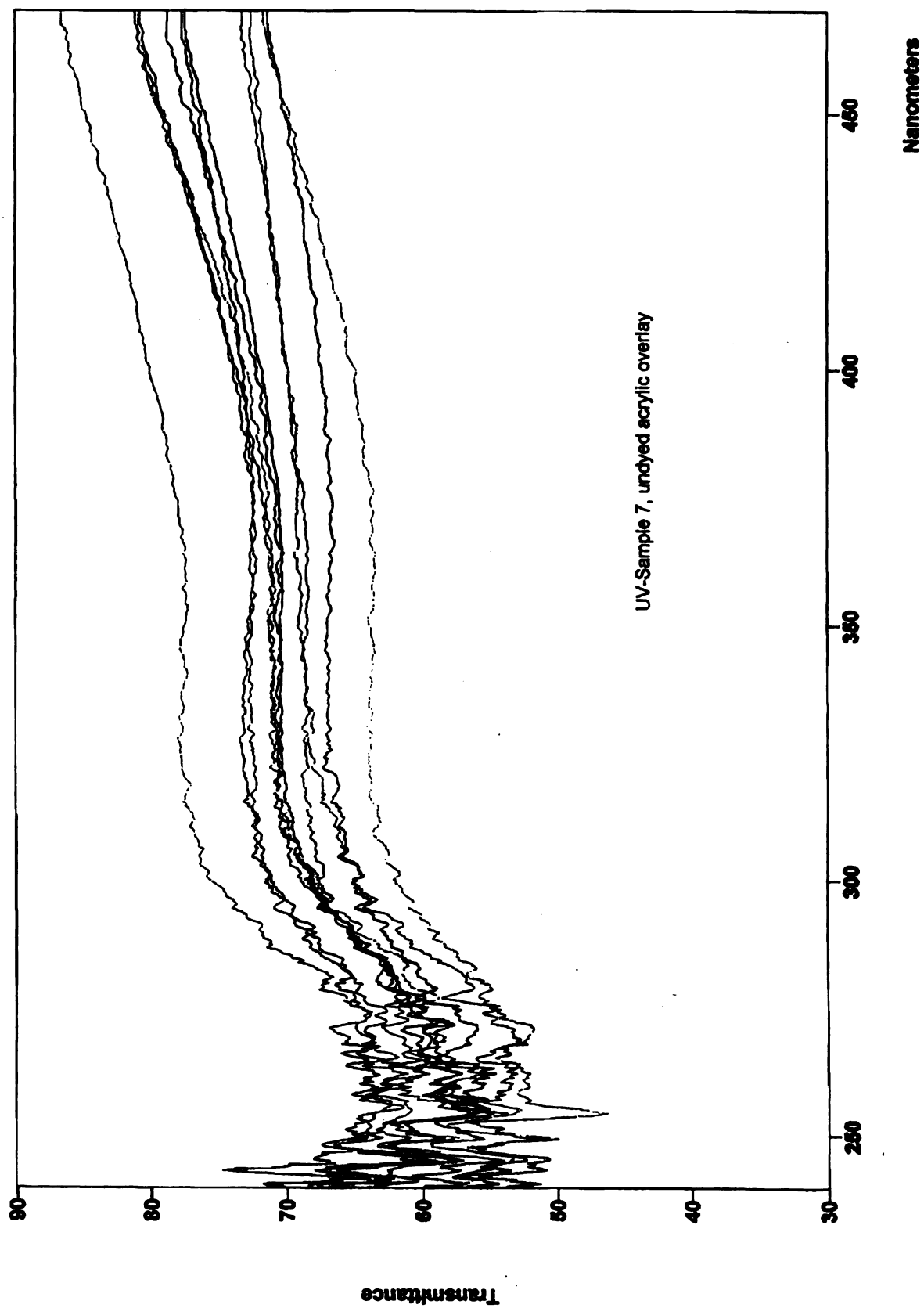


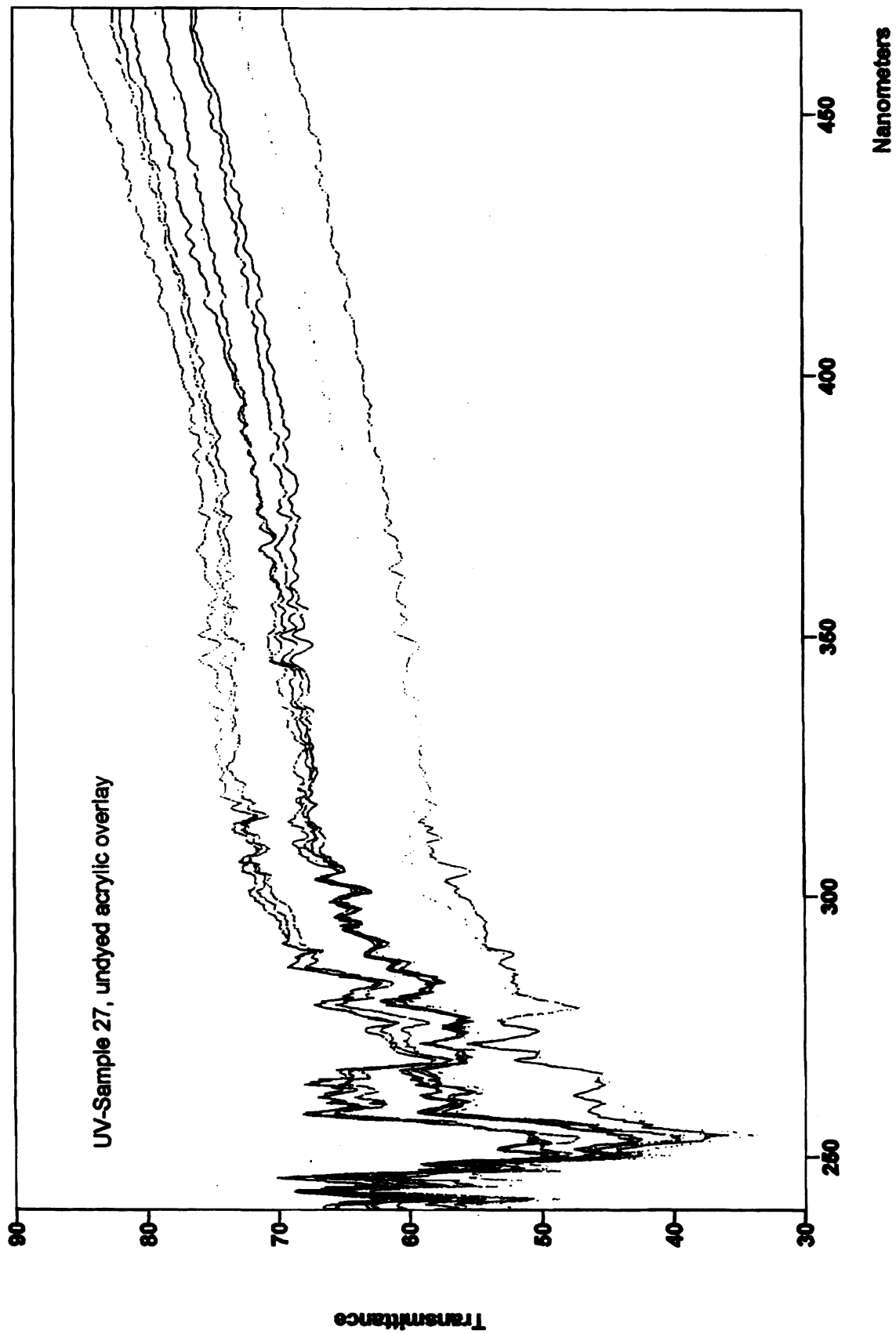


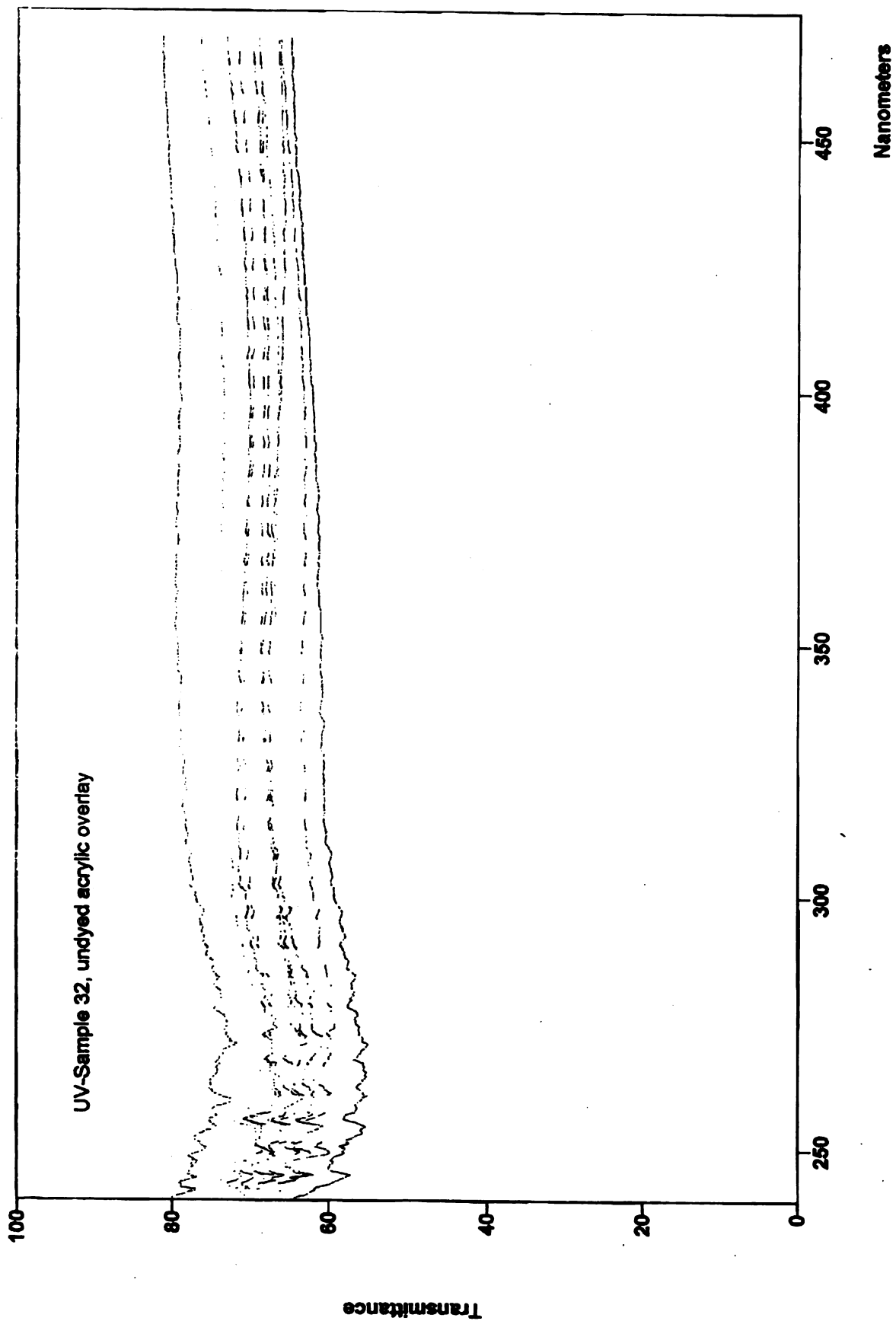






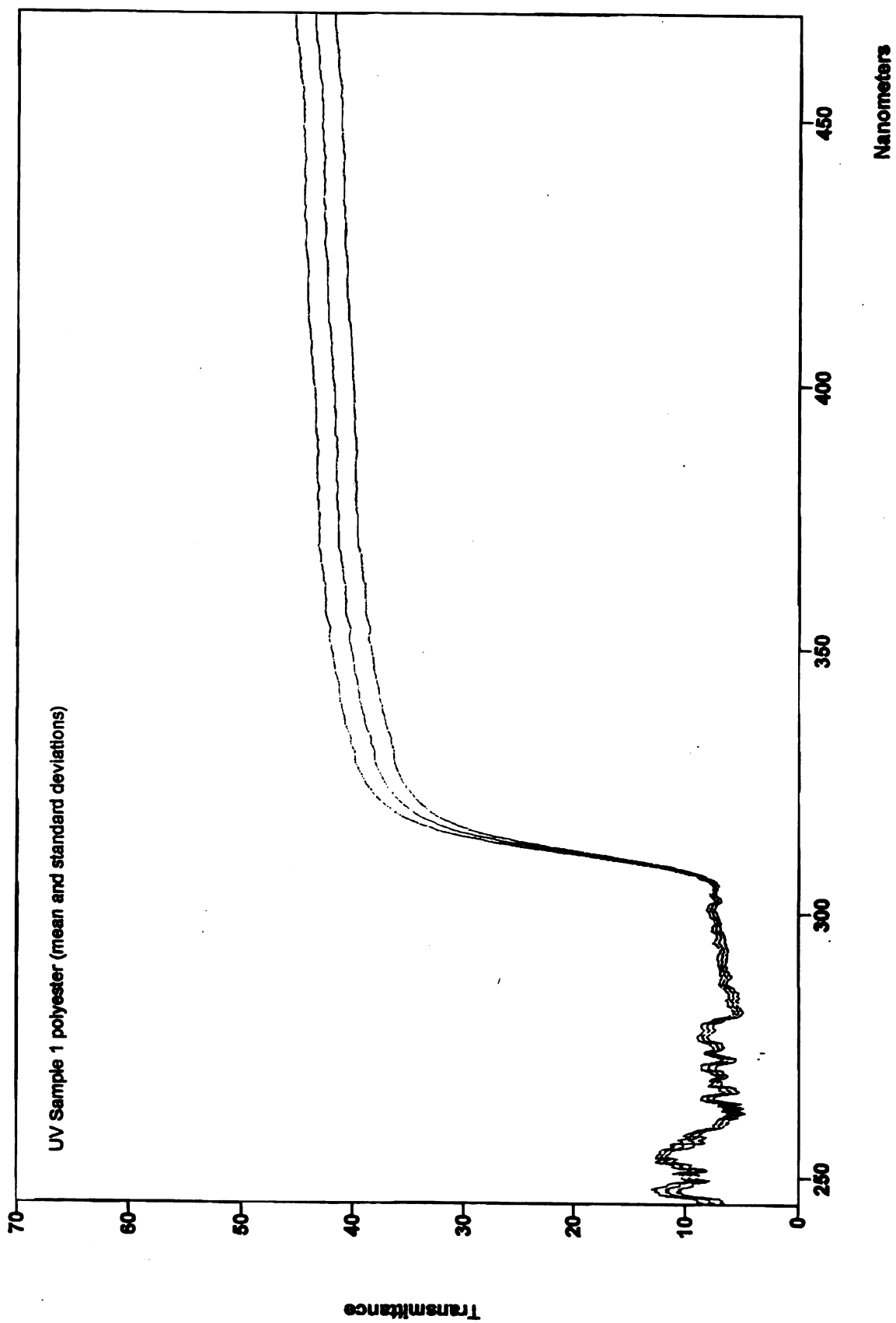


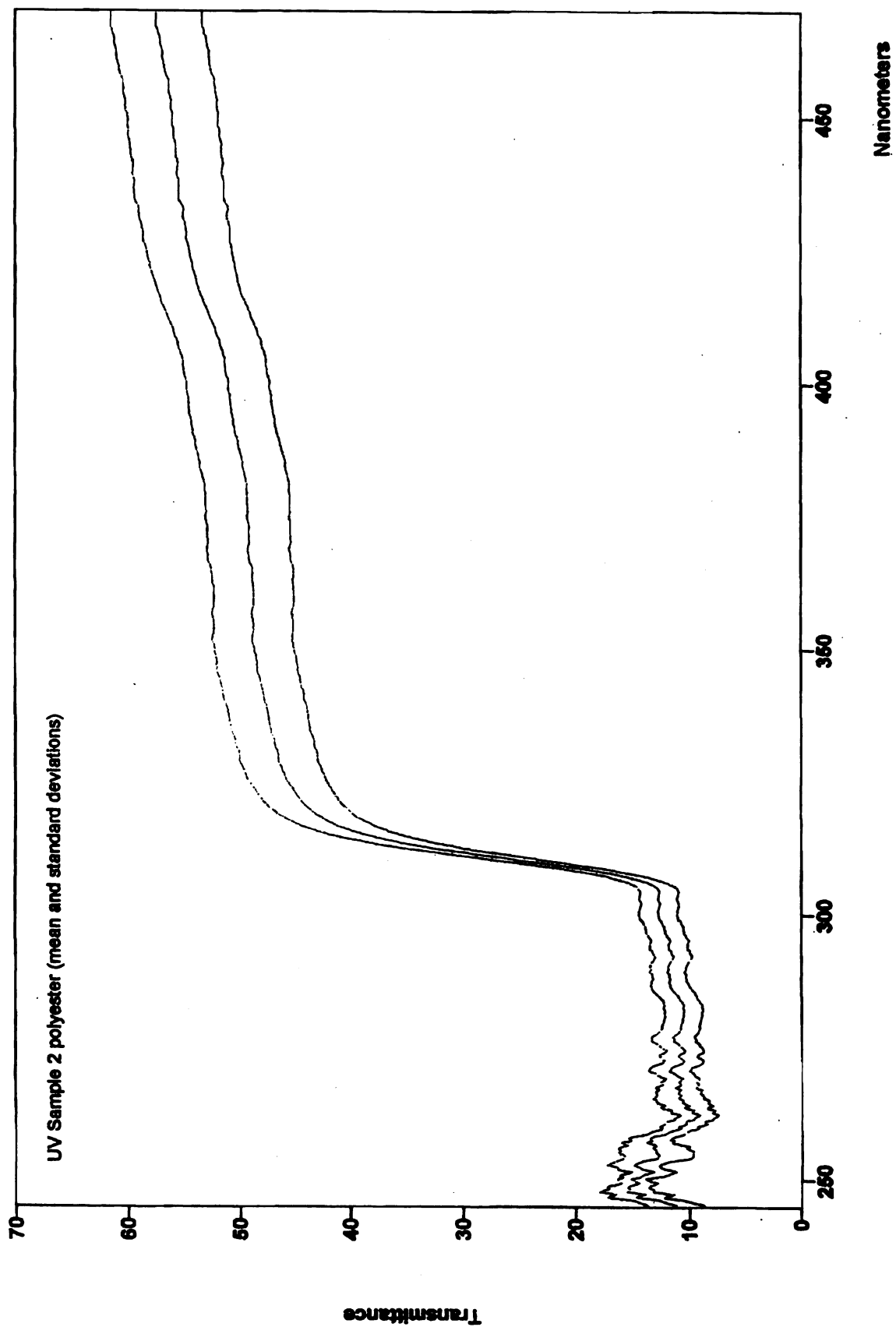


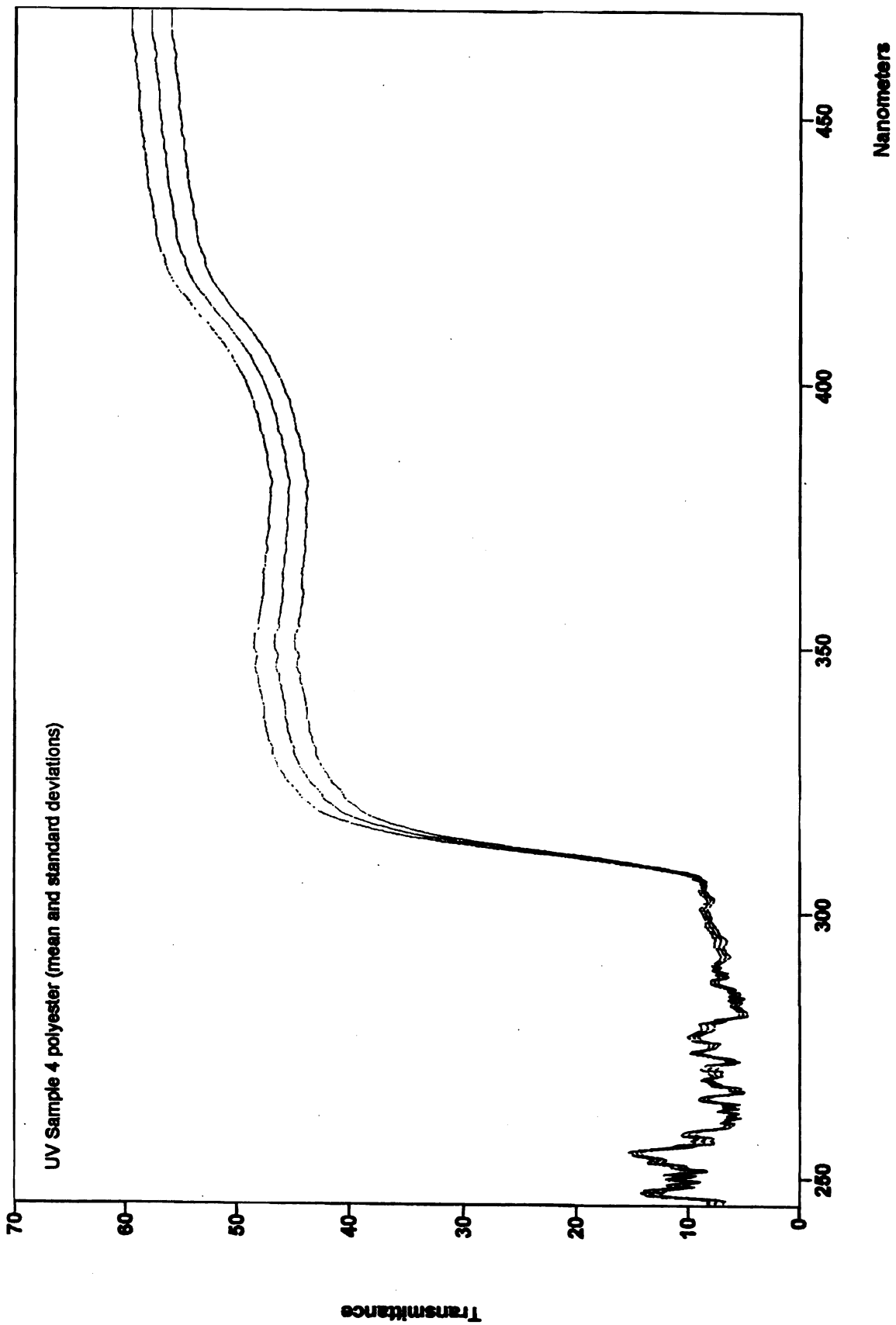


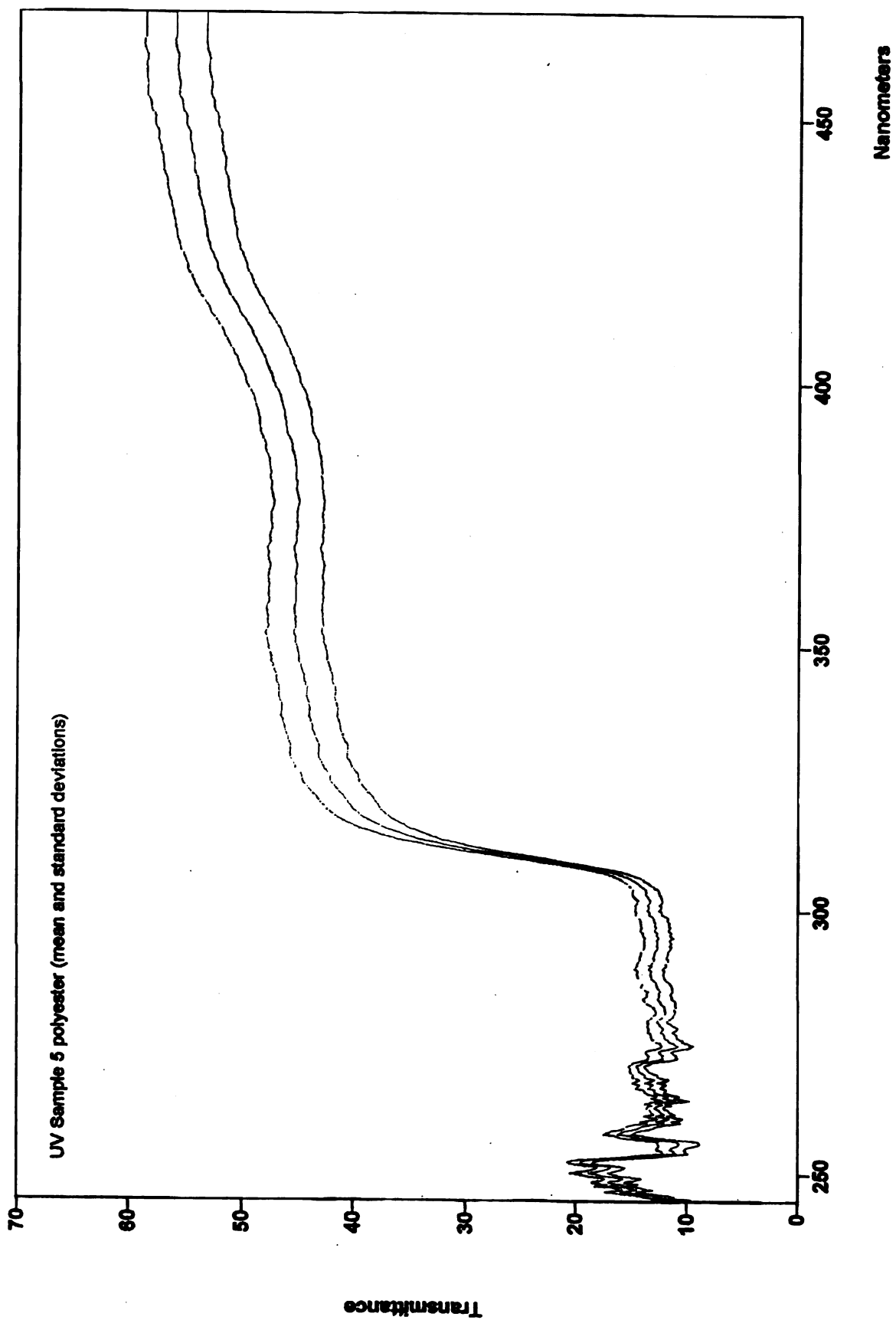
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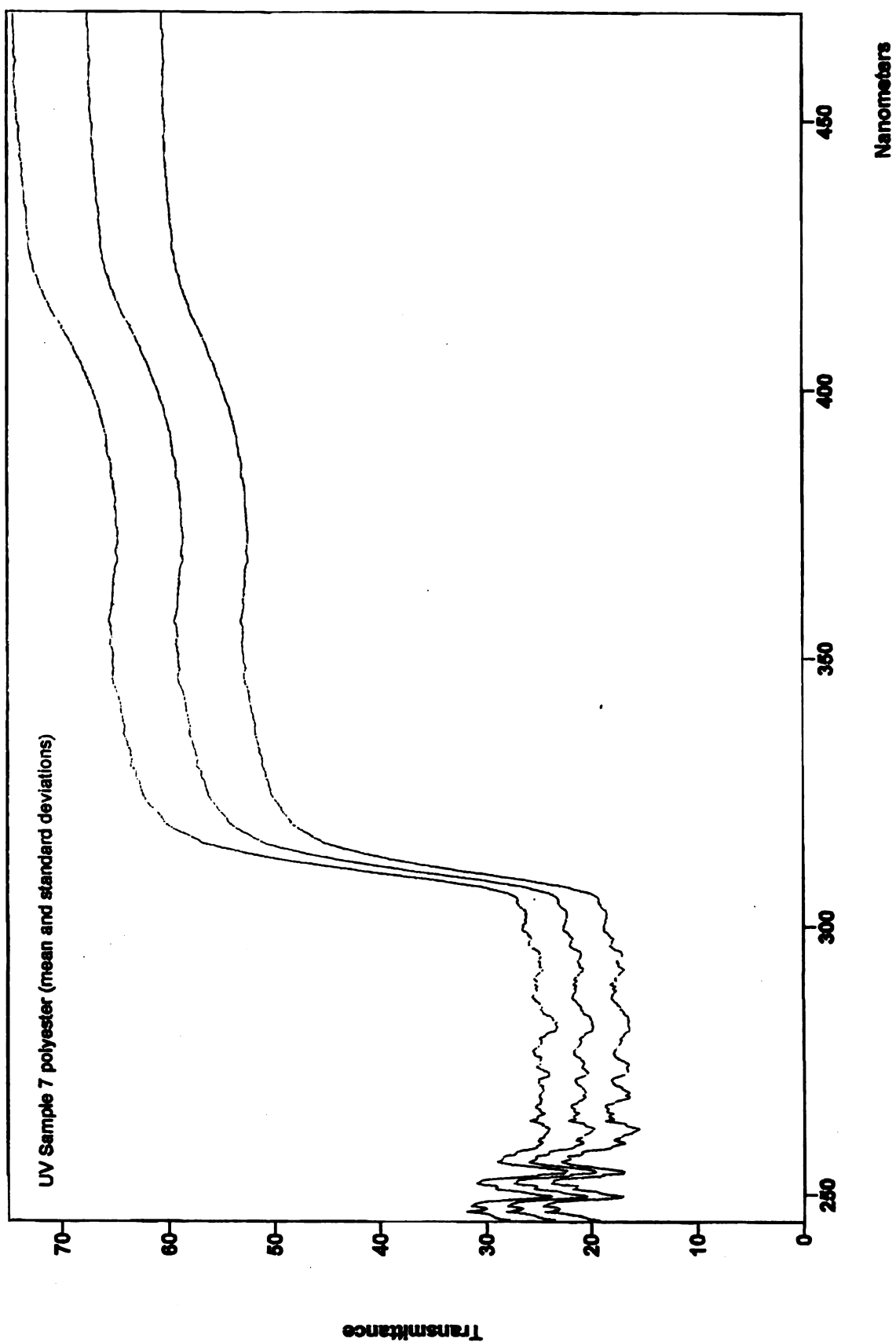
MEANS AND STANDARD DEVIATIONS

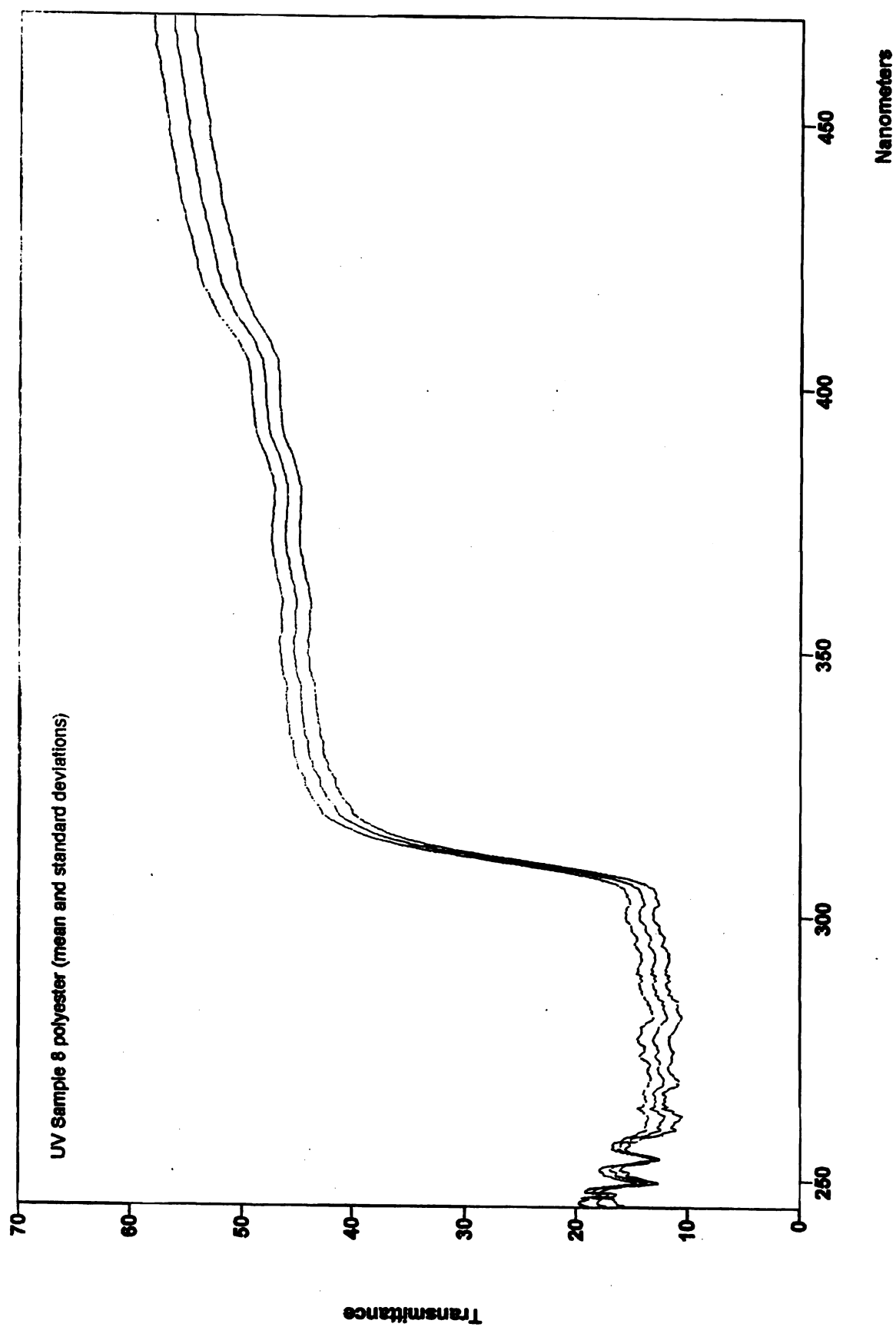


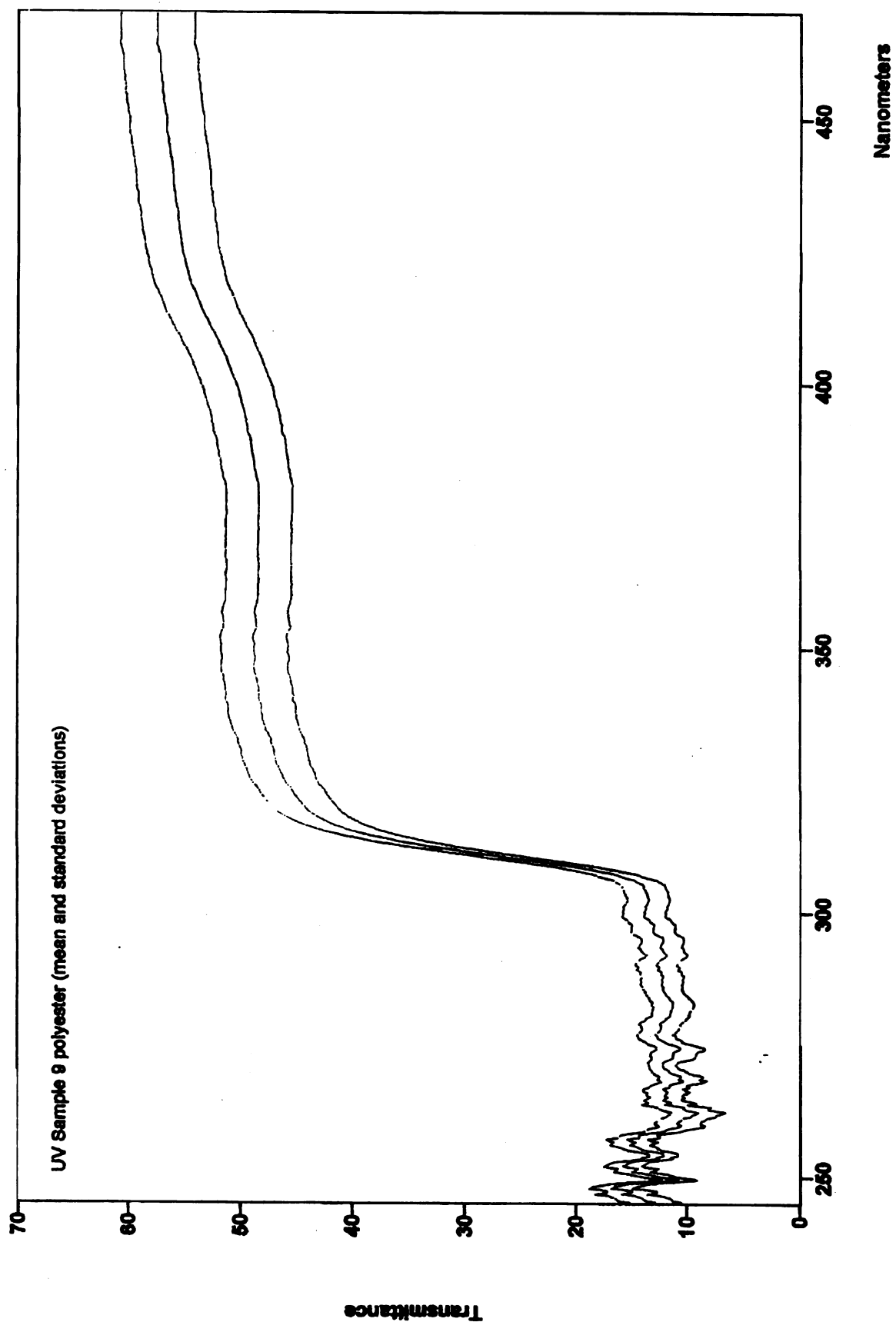


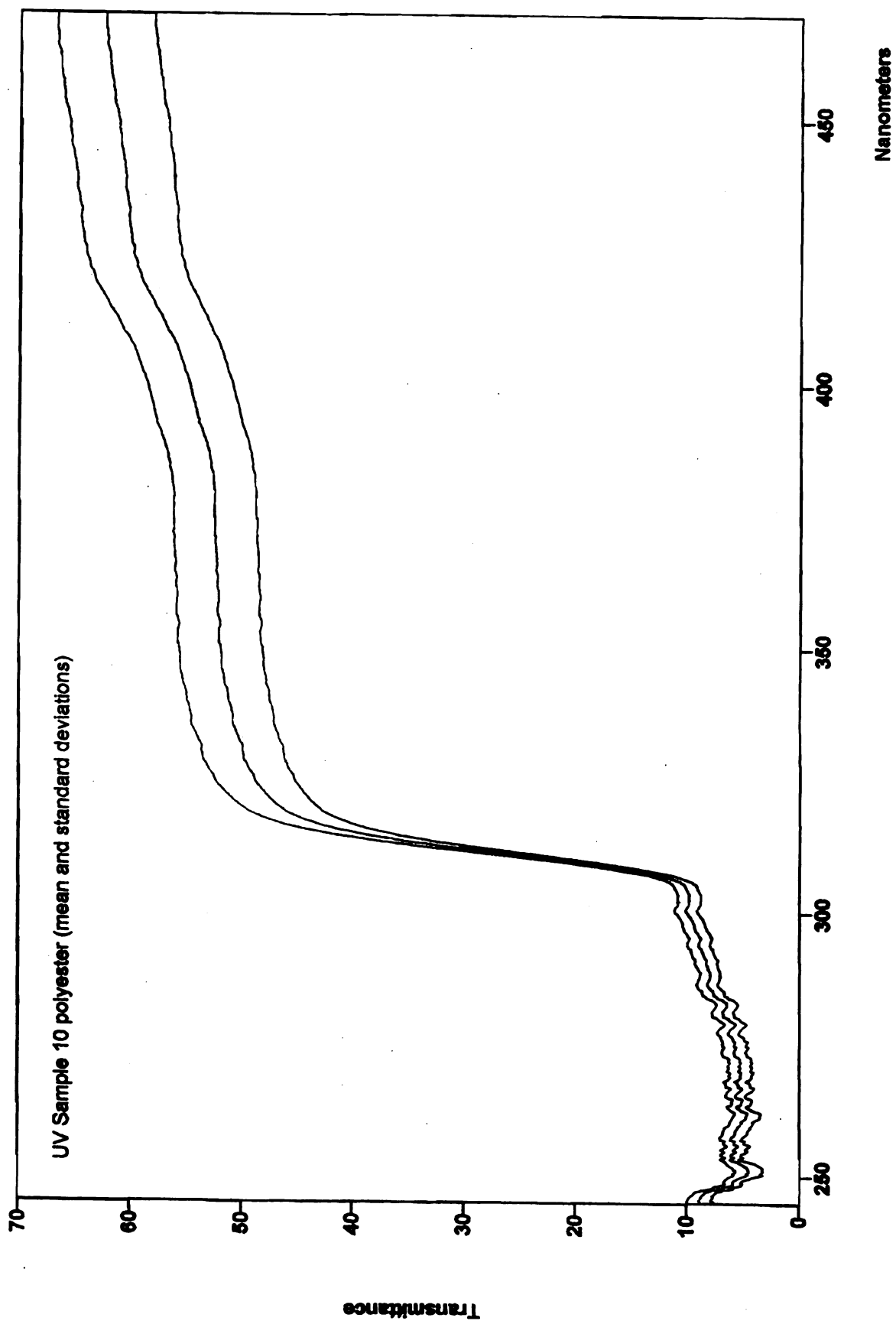


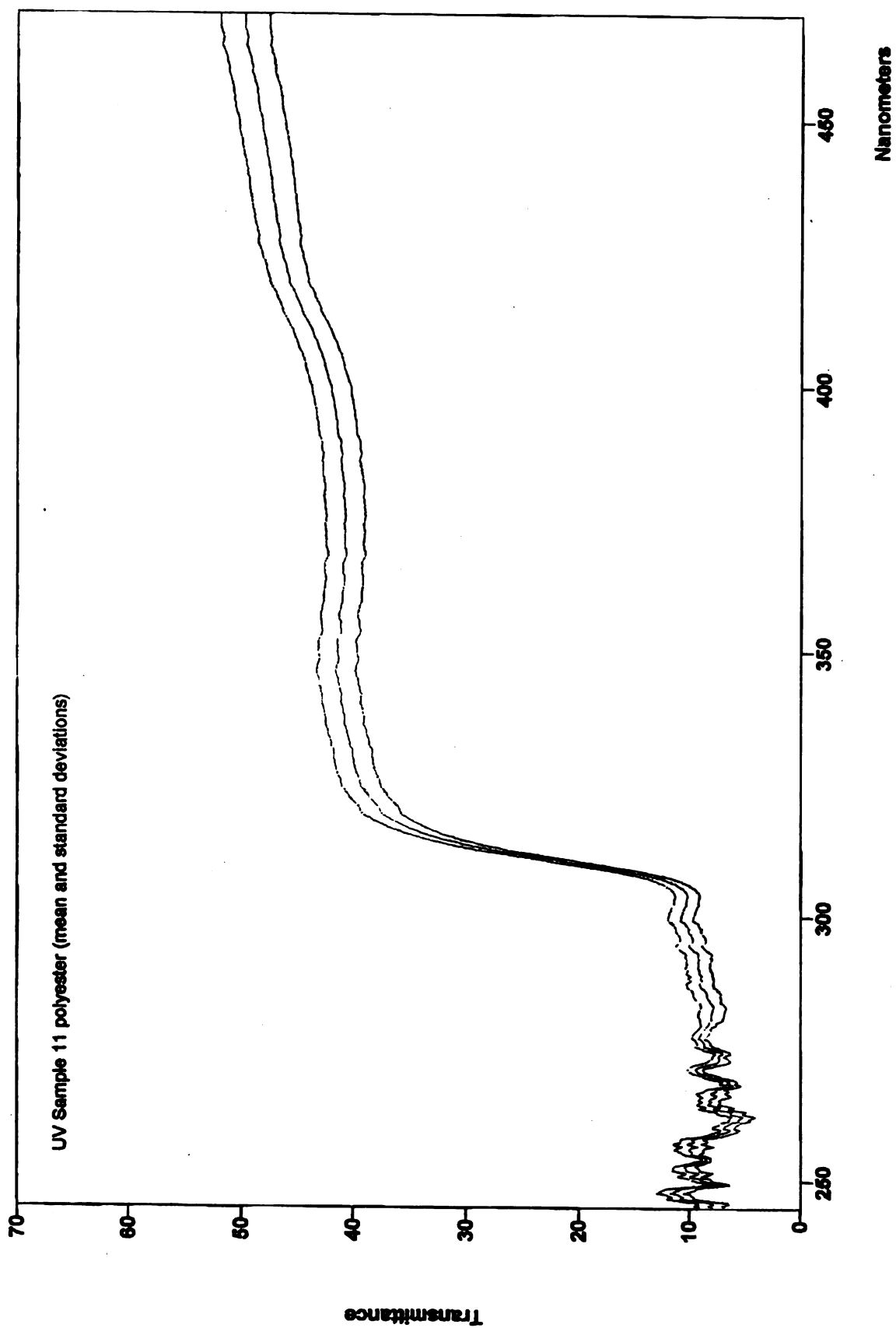


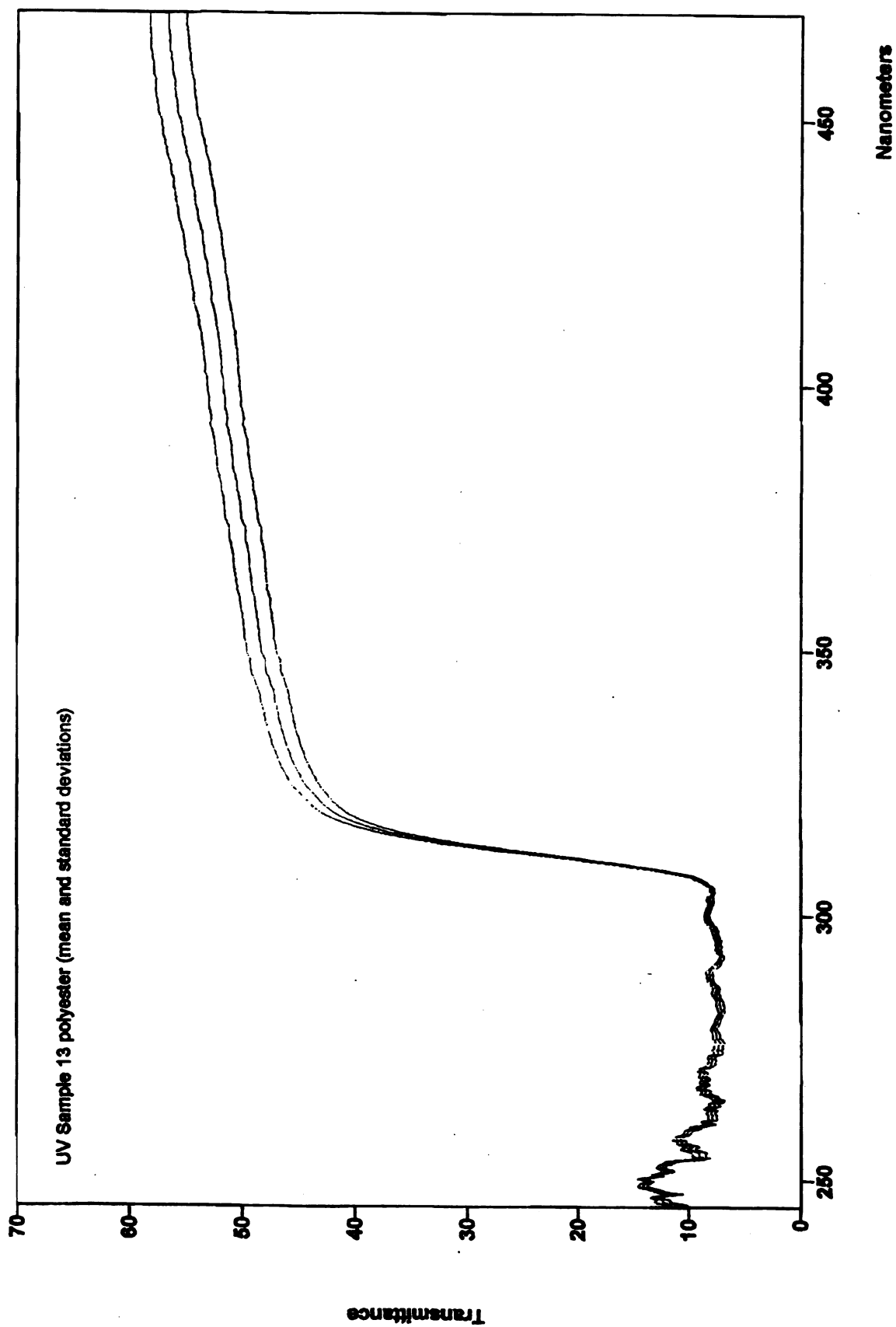


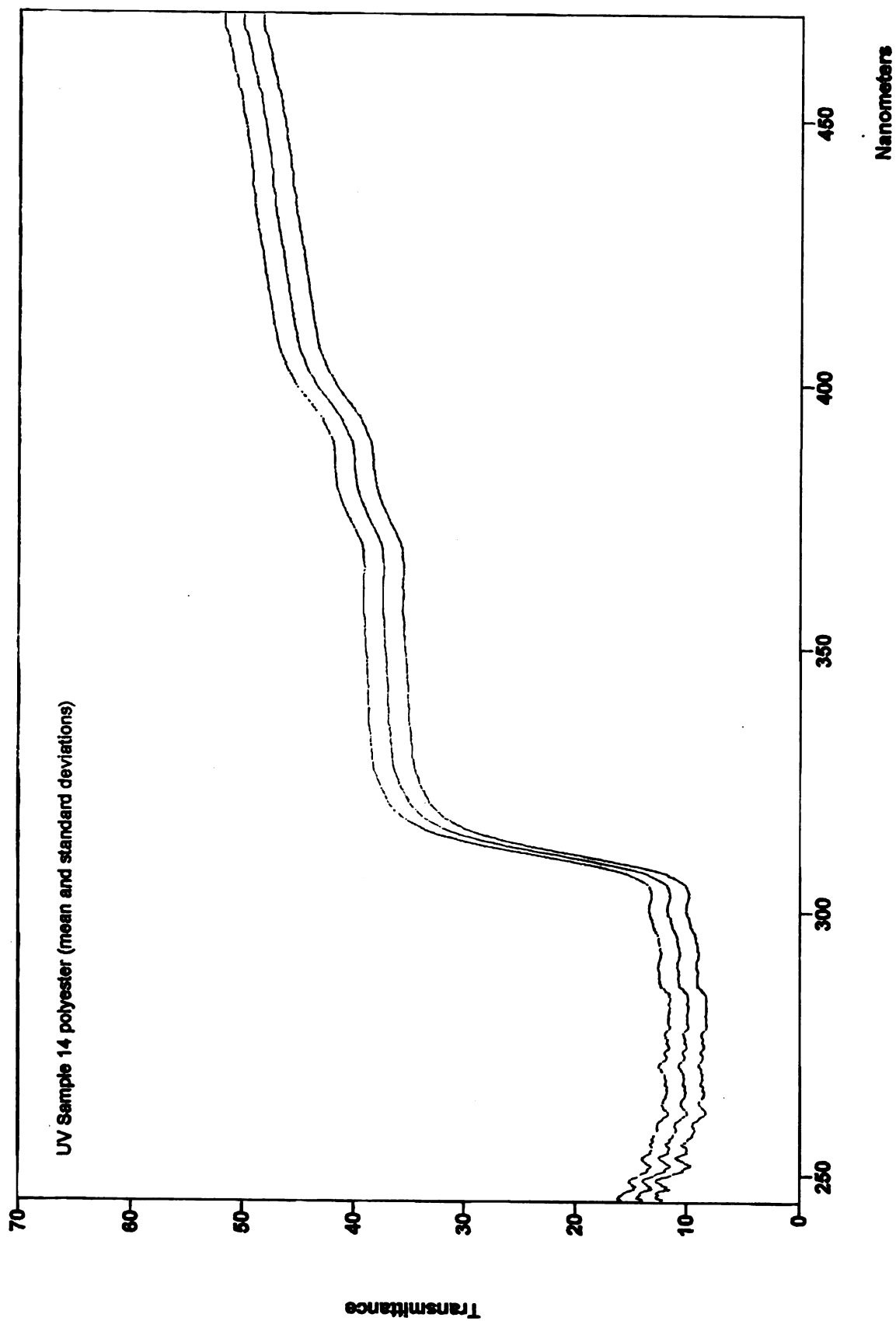


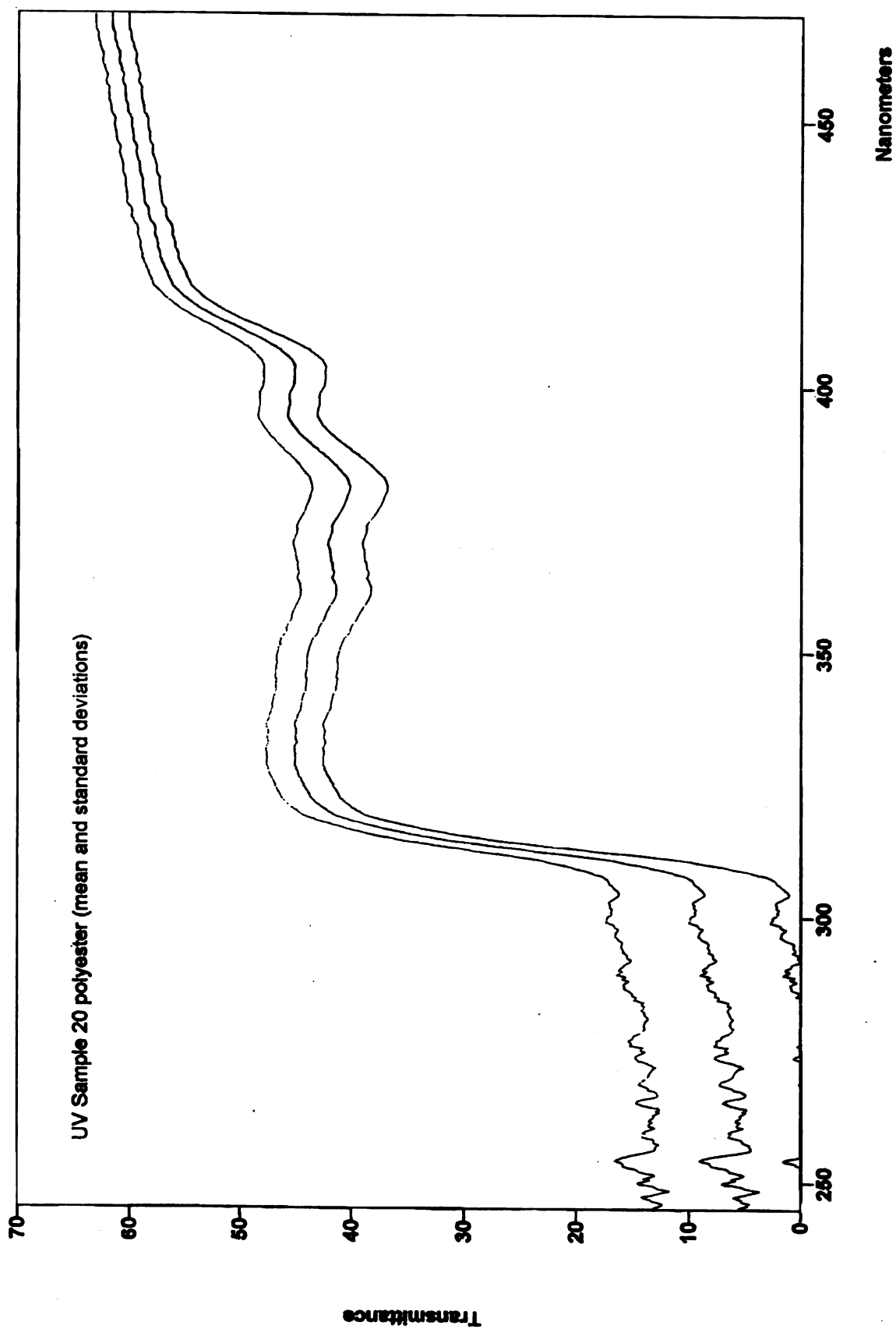


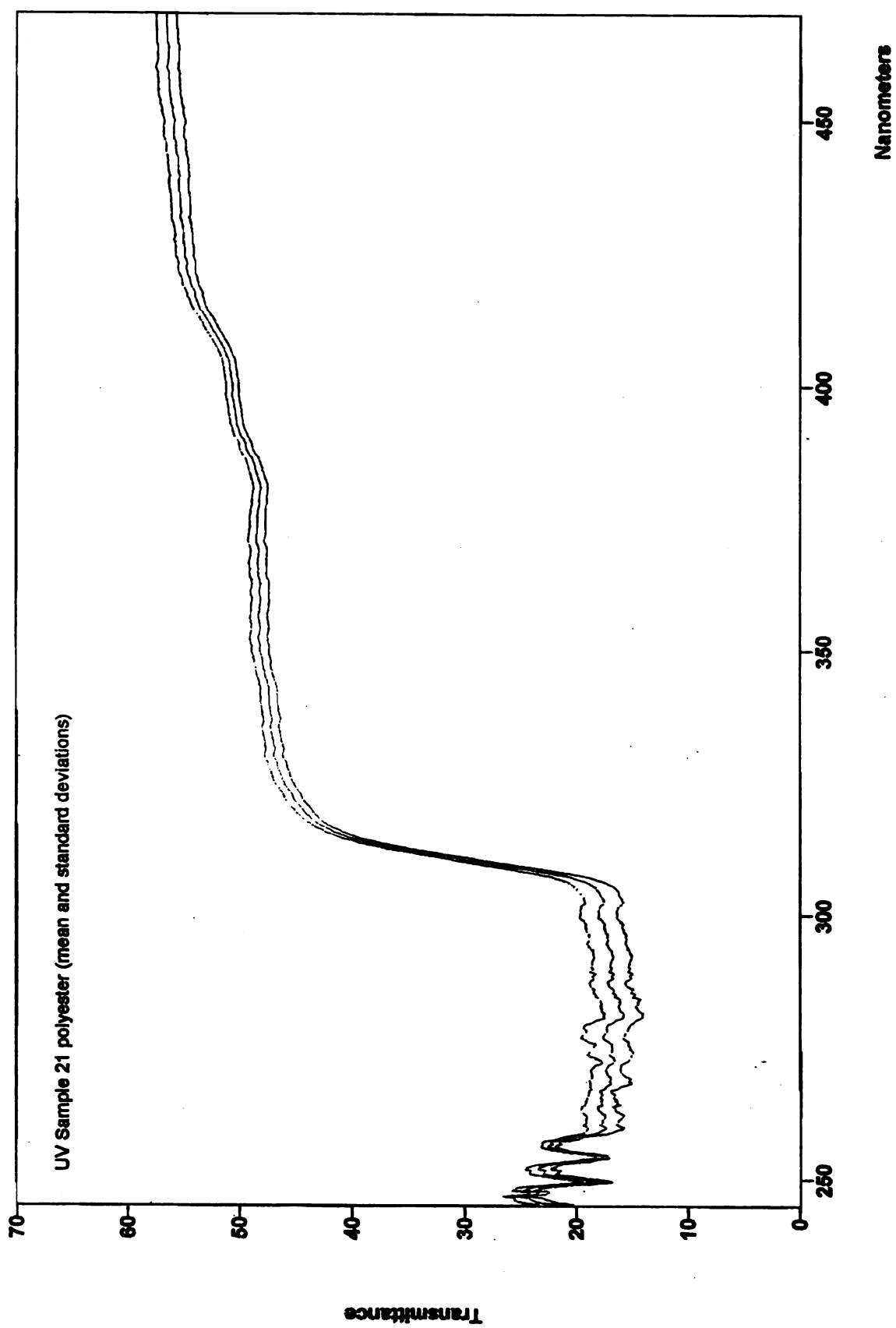


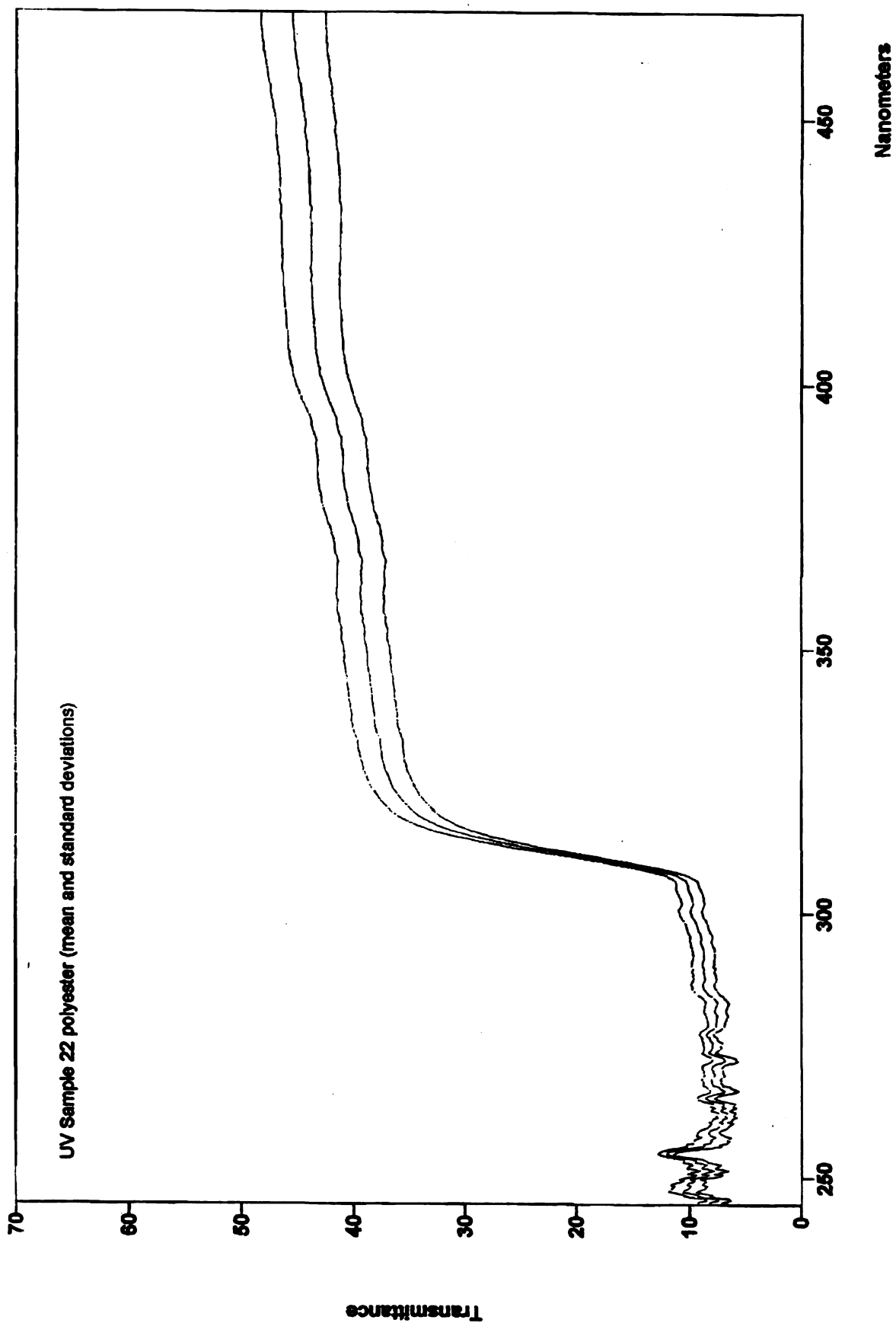


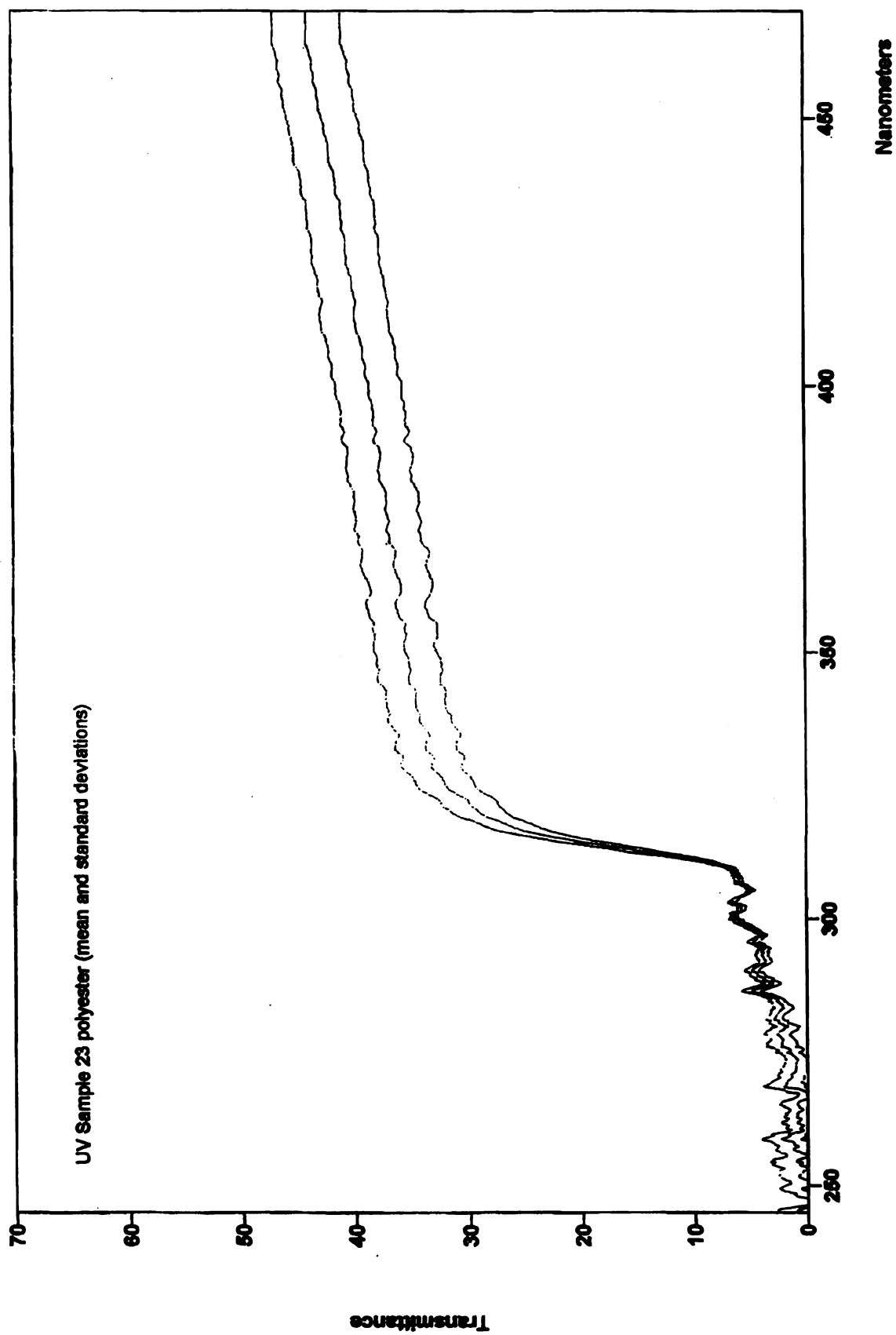


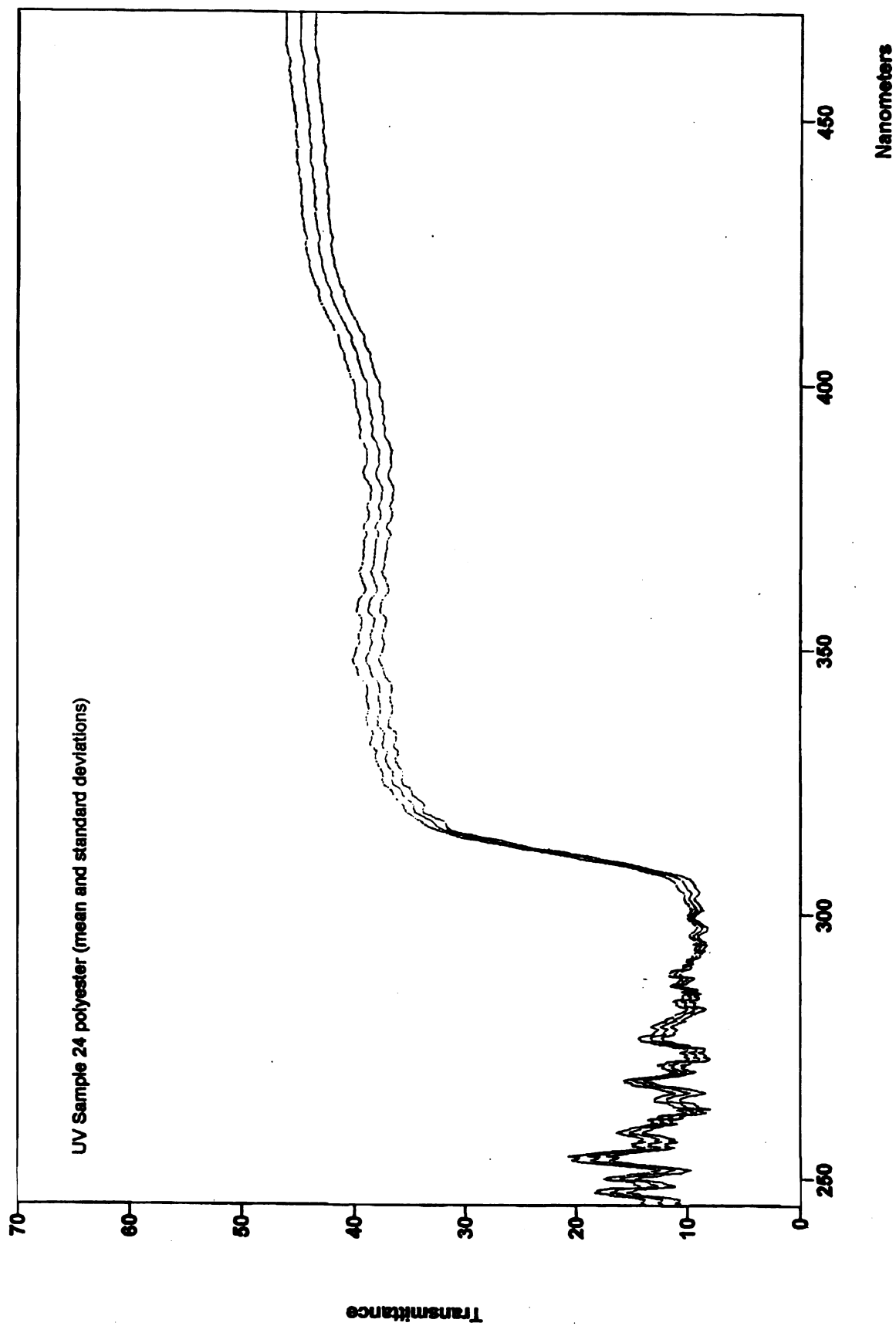


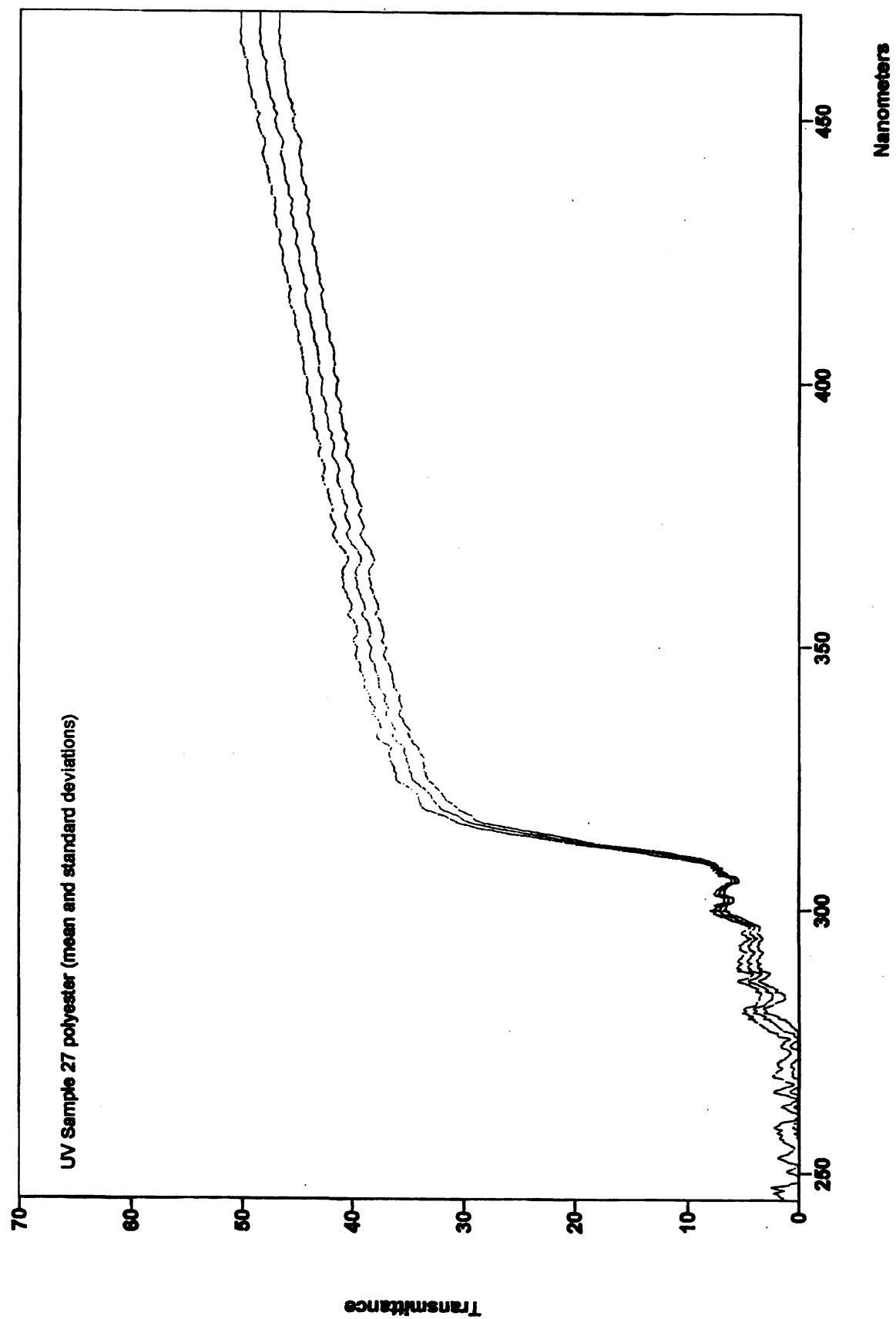


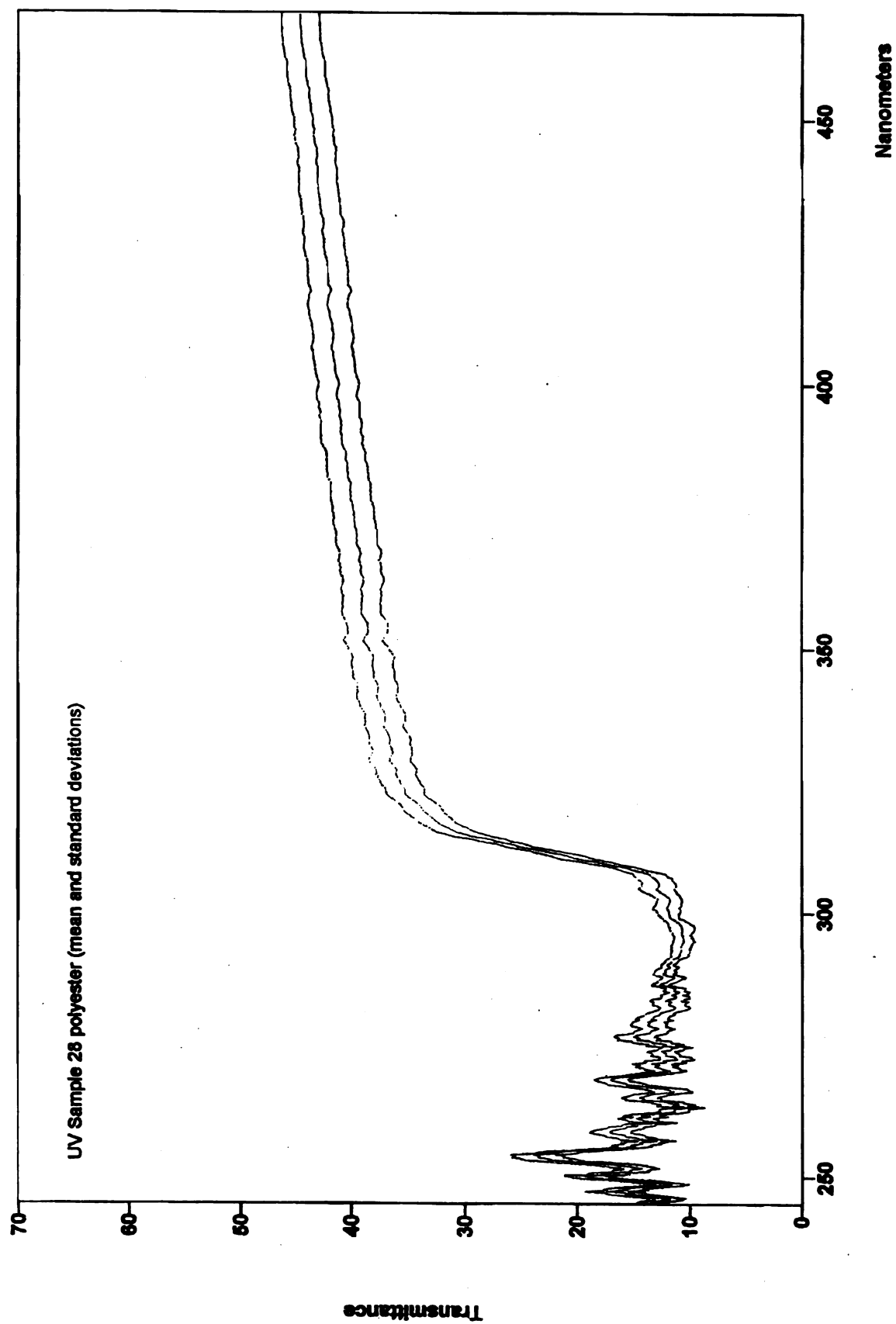


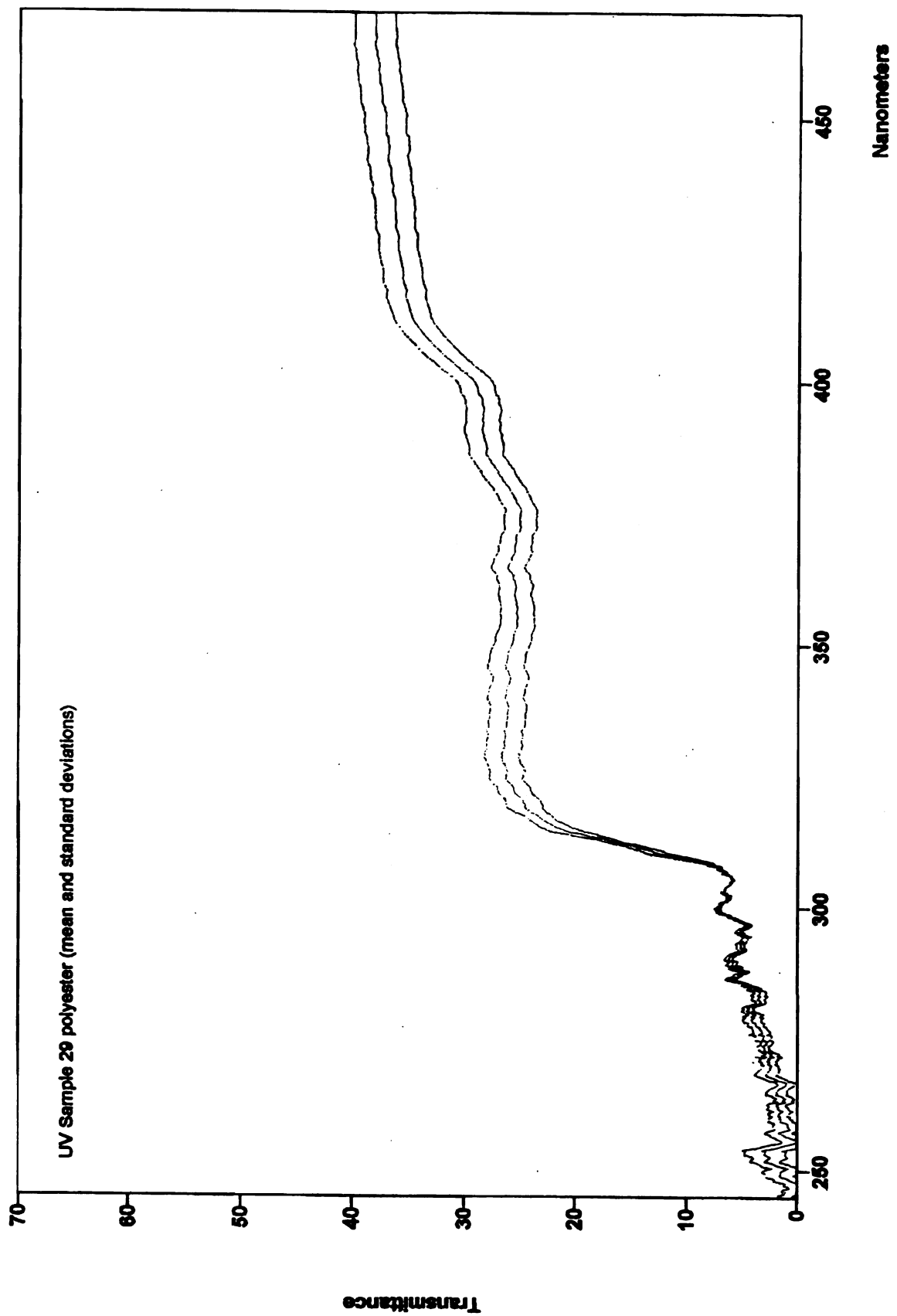


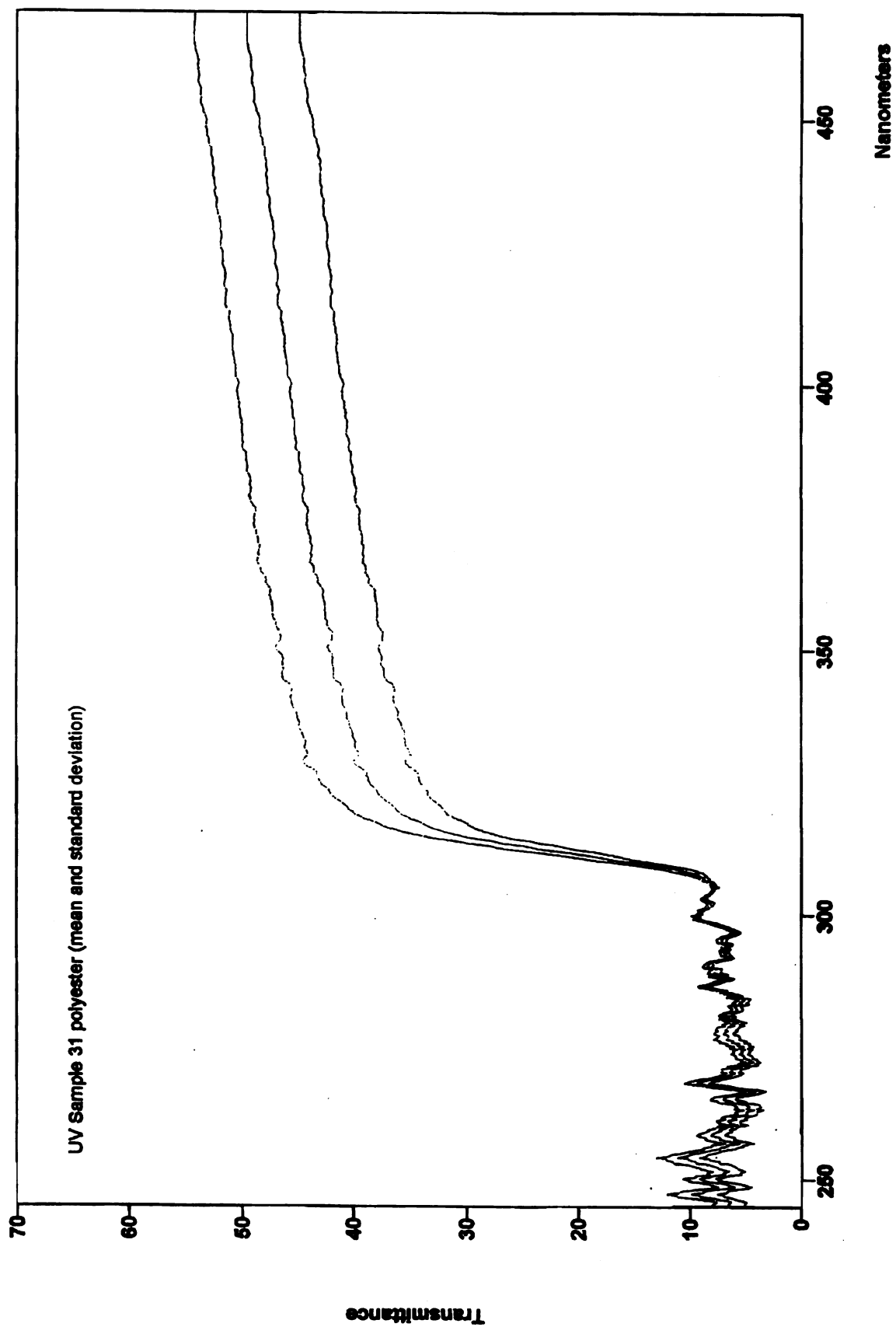


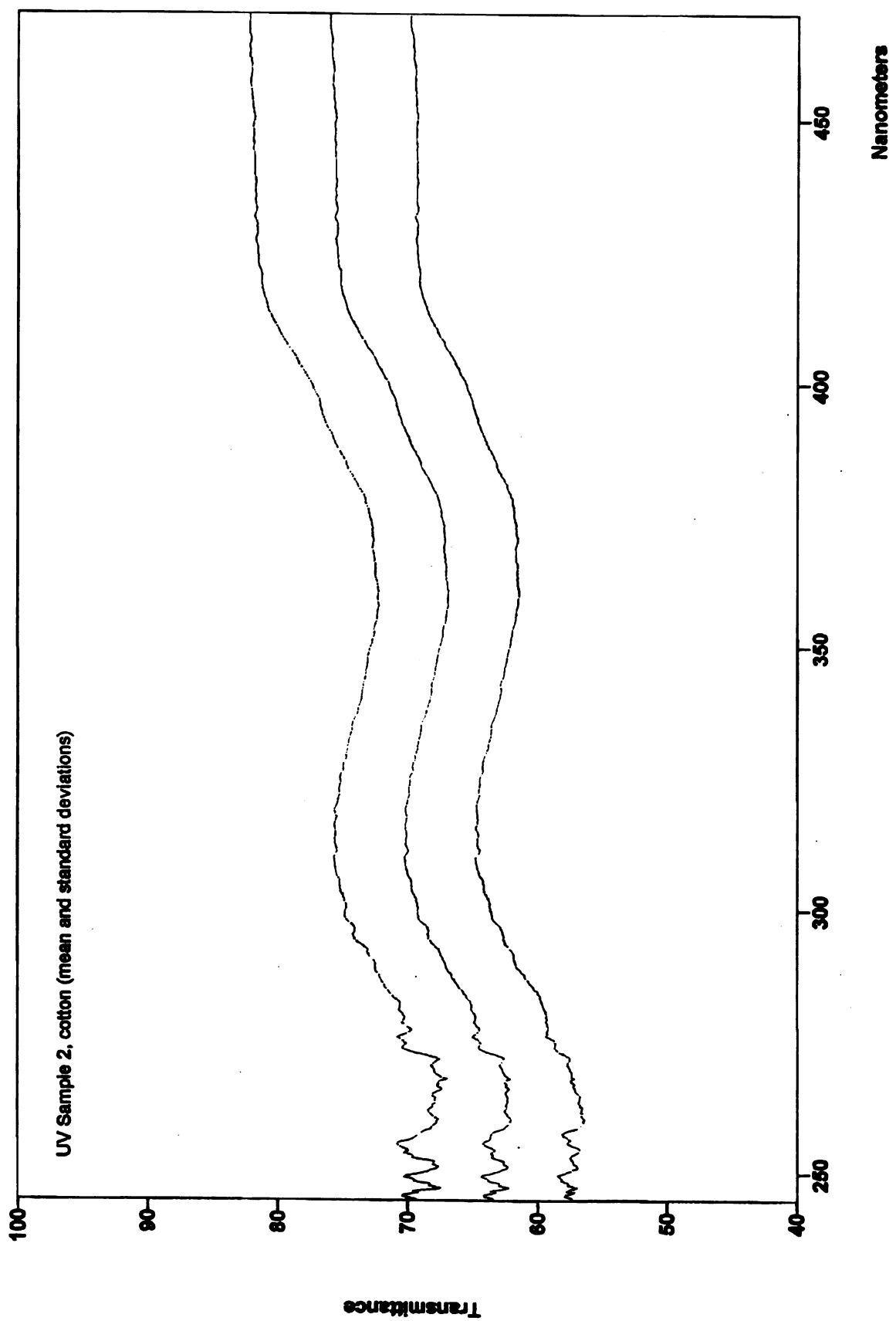


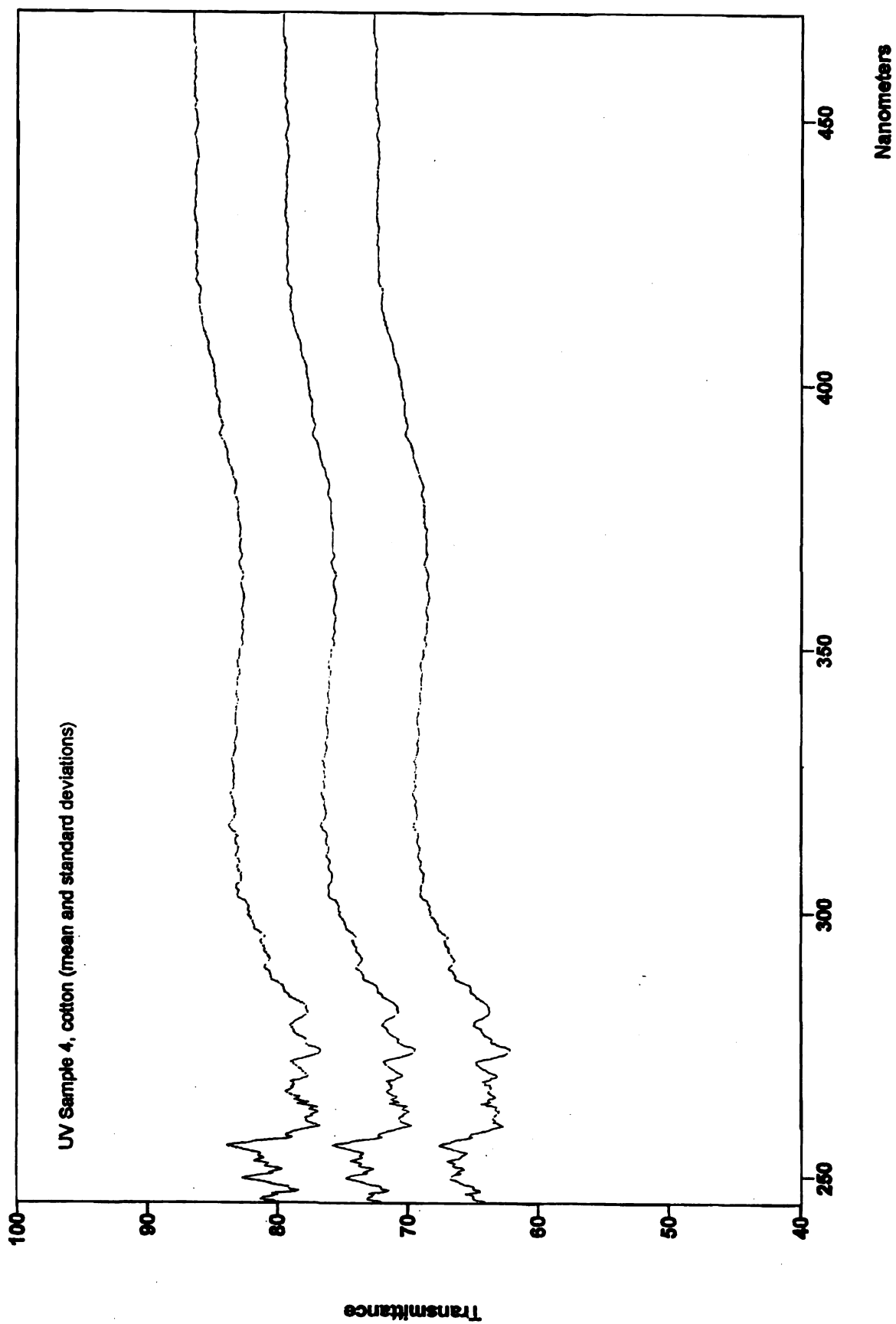


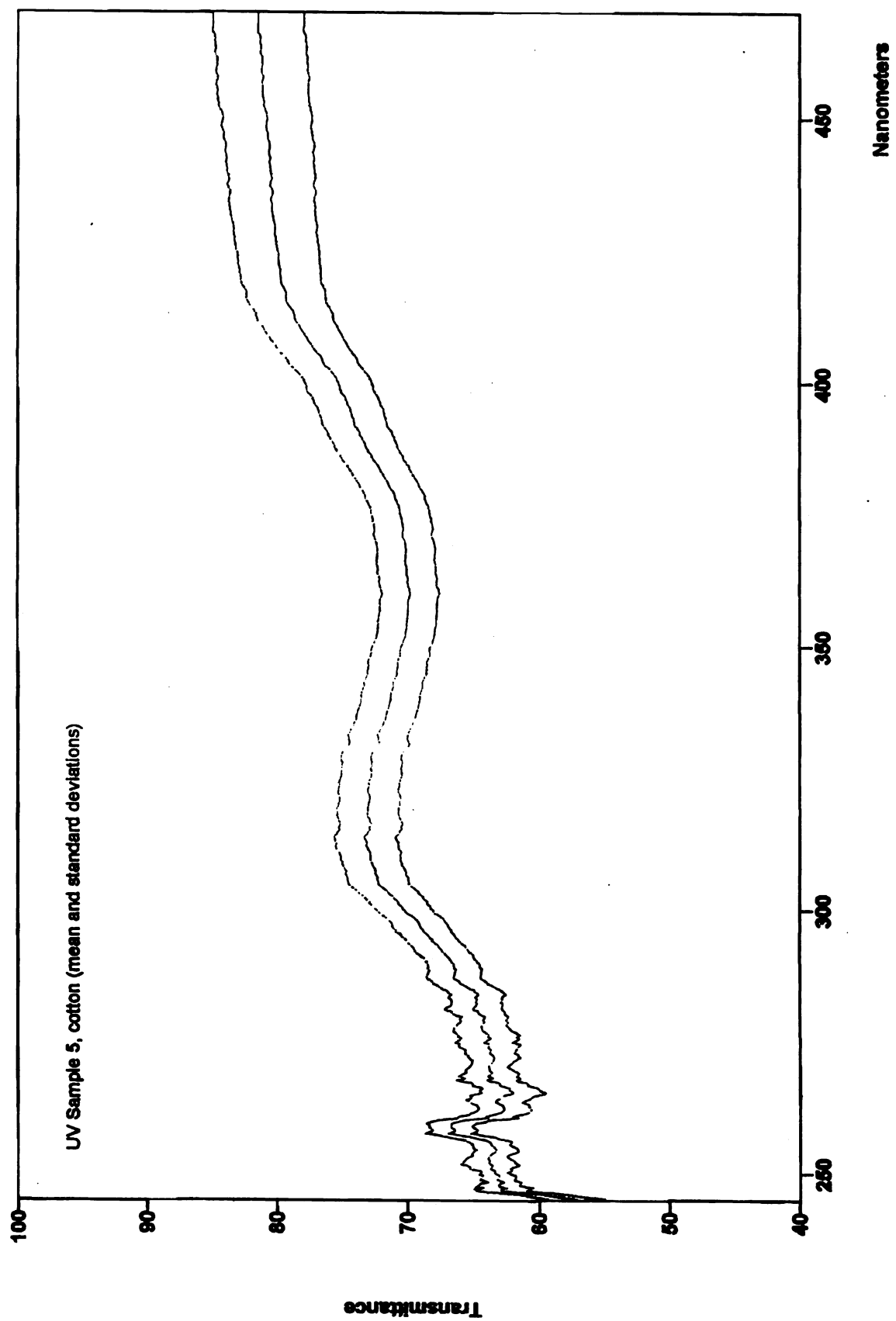


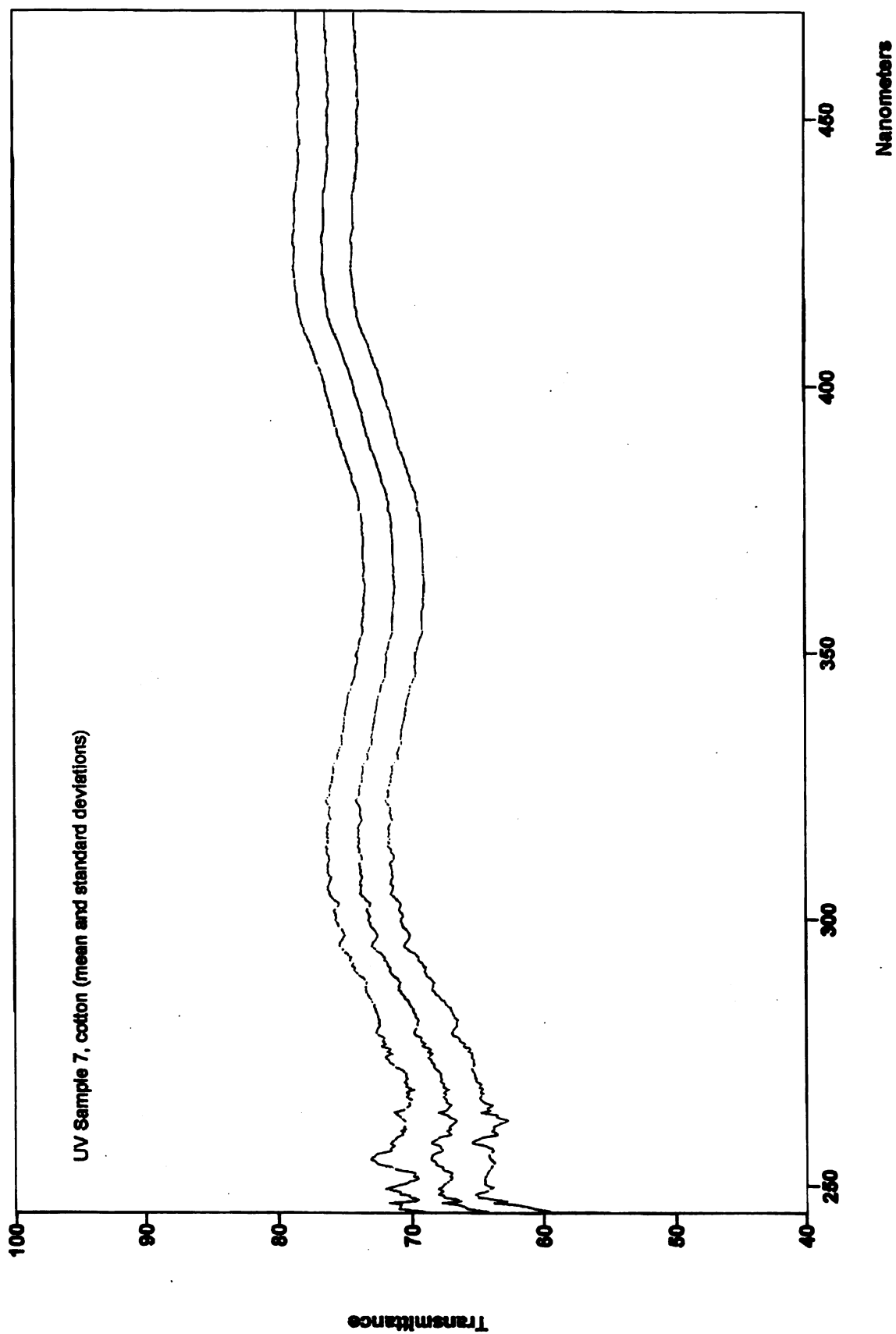


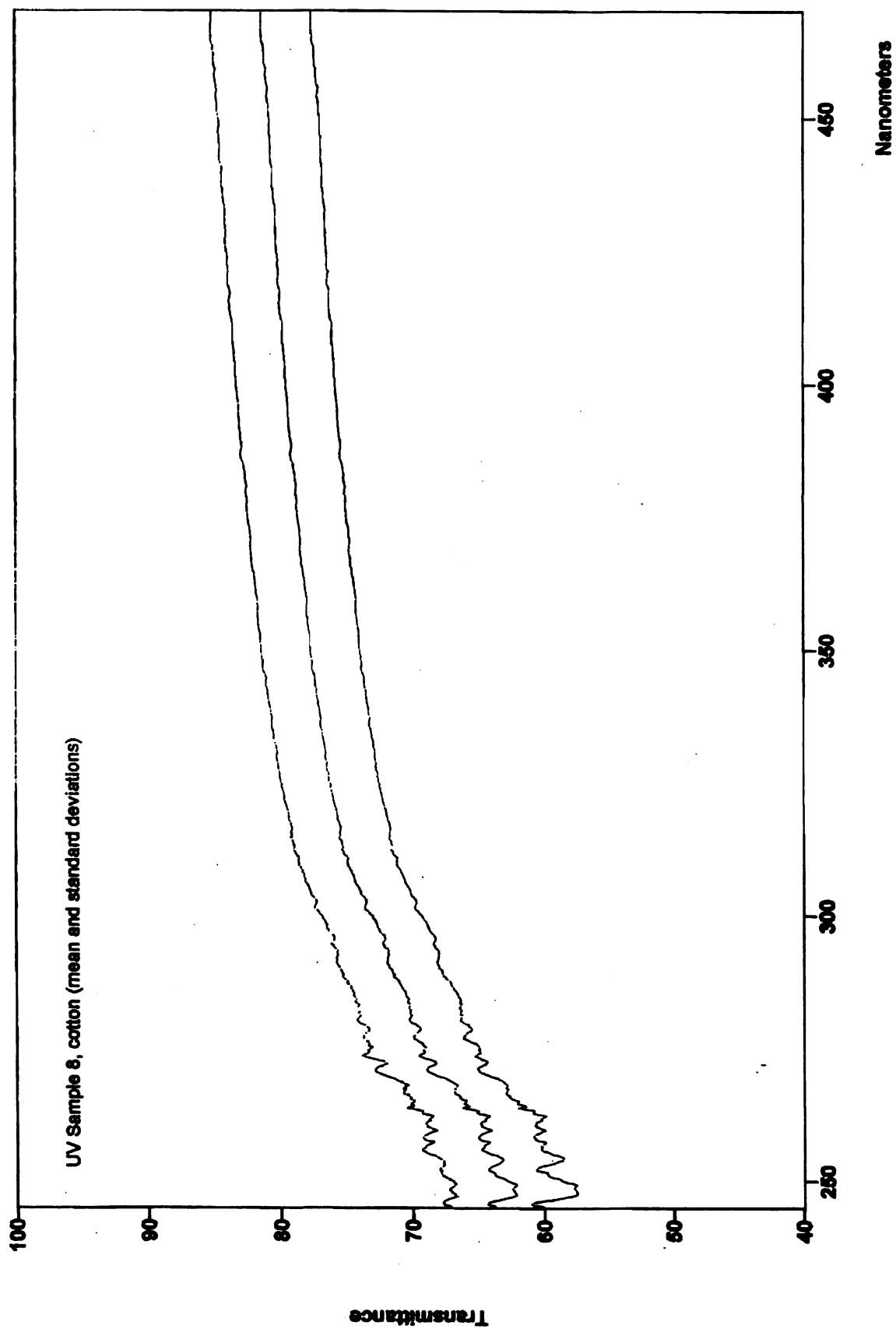


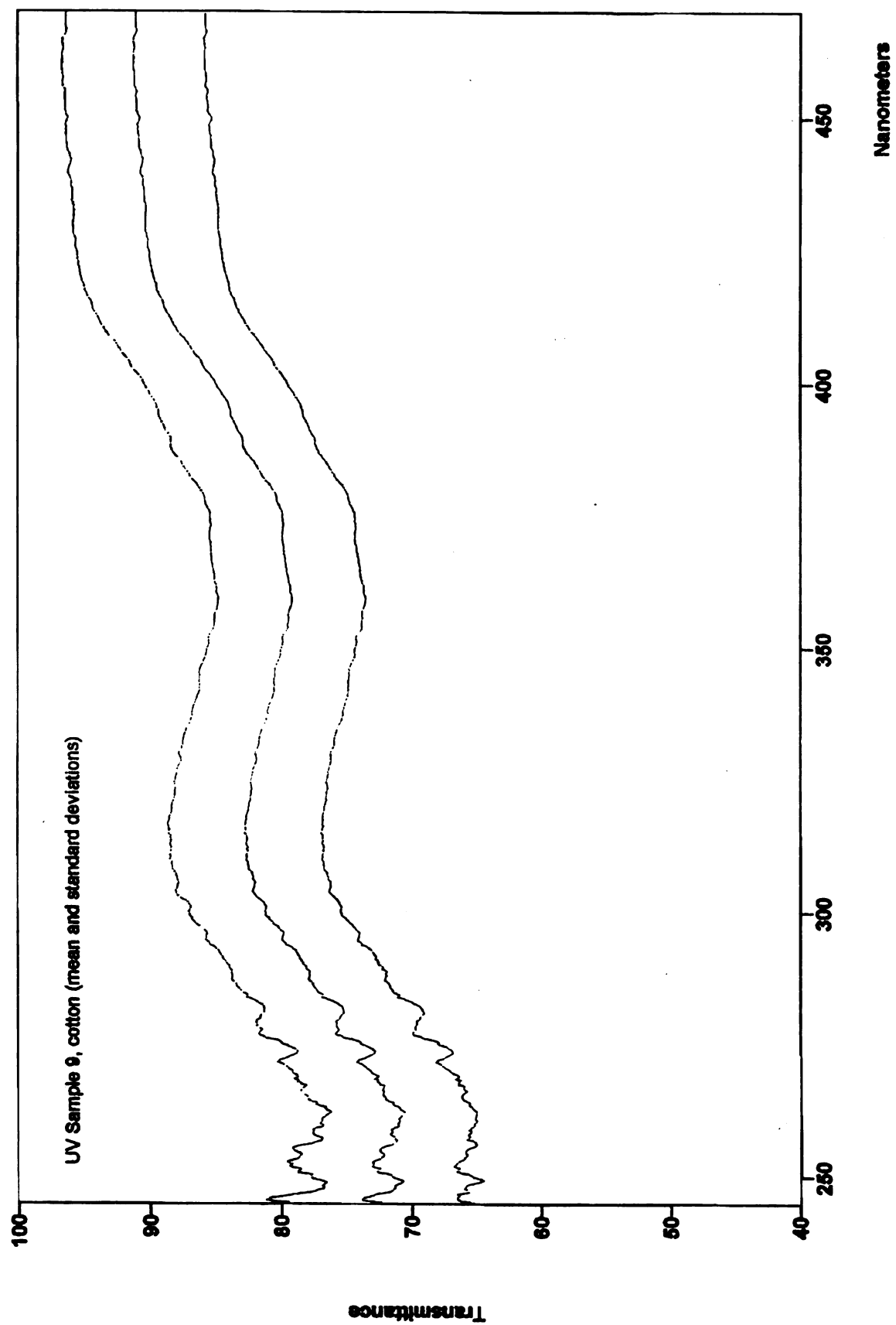


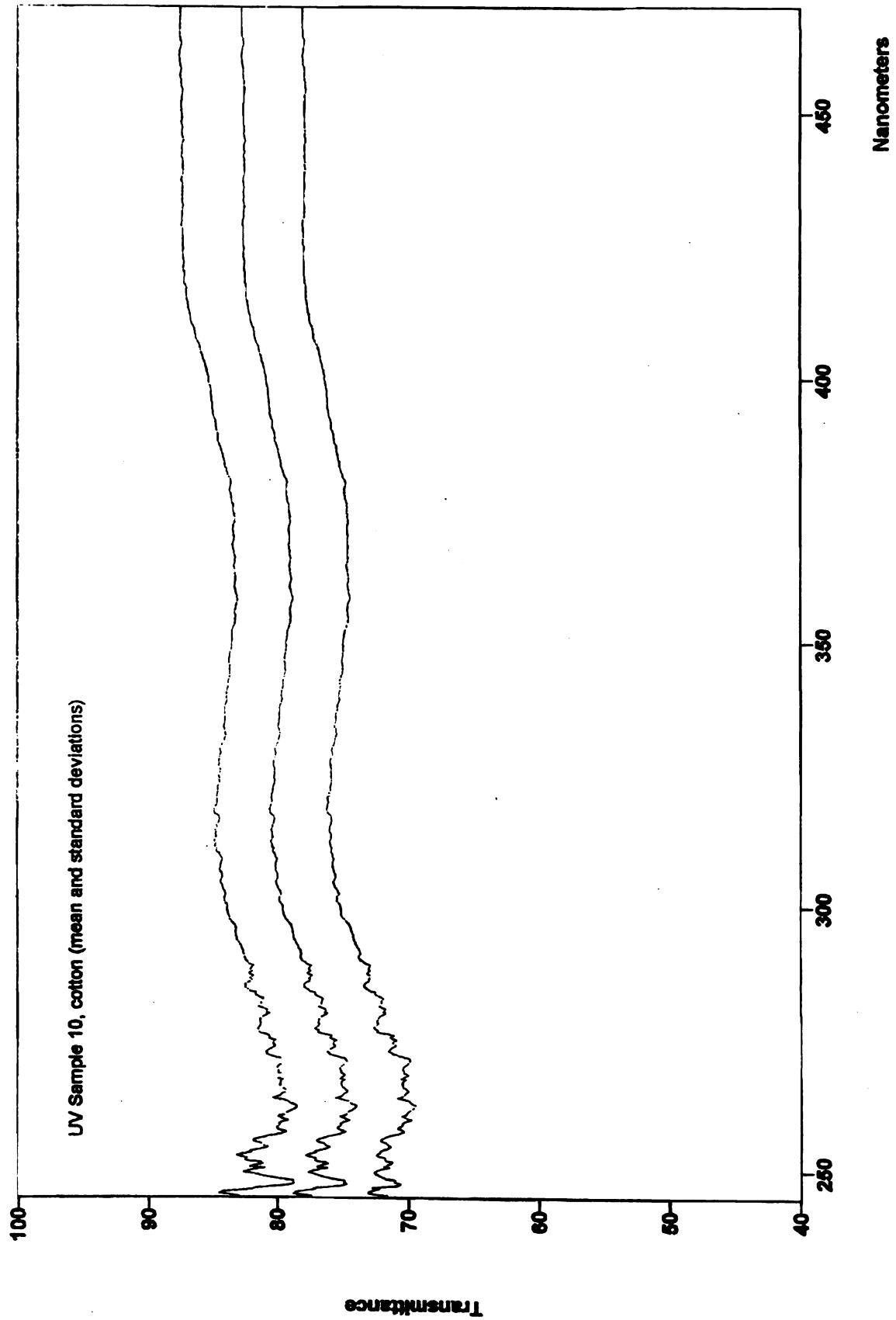


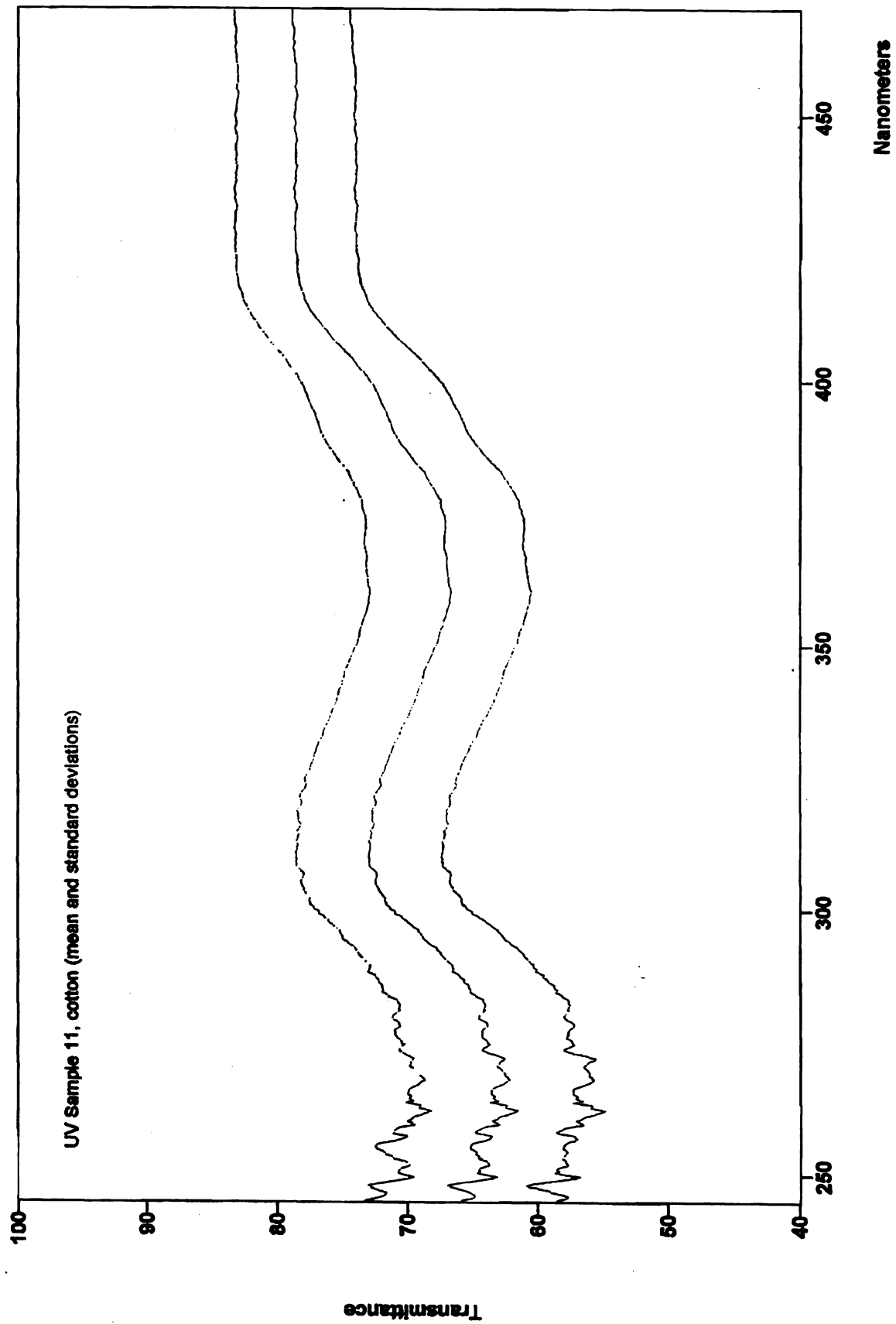


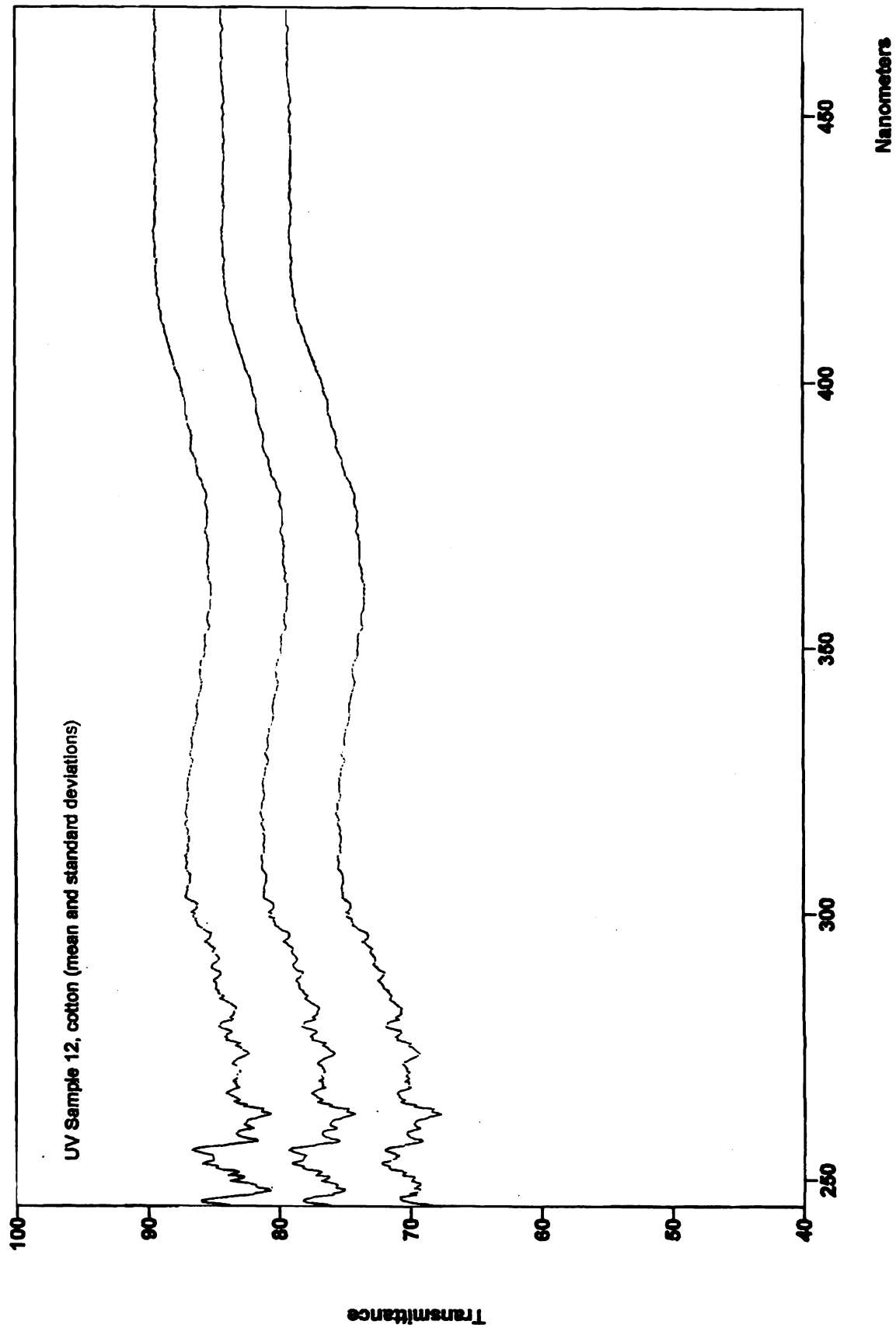


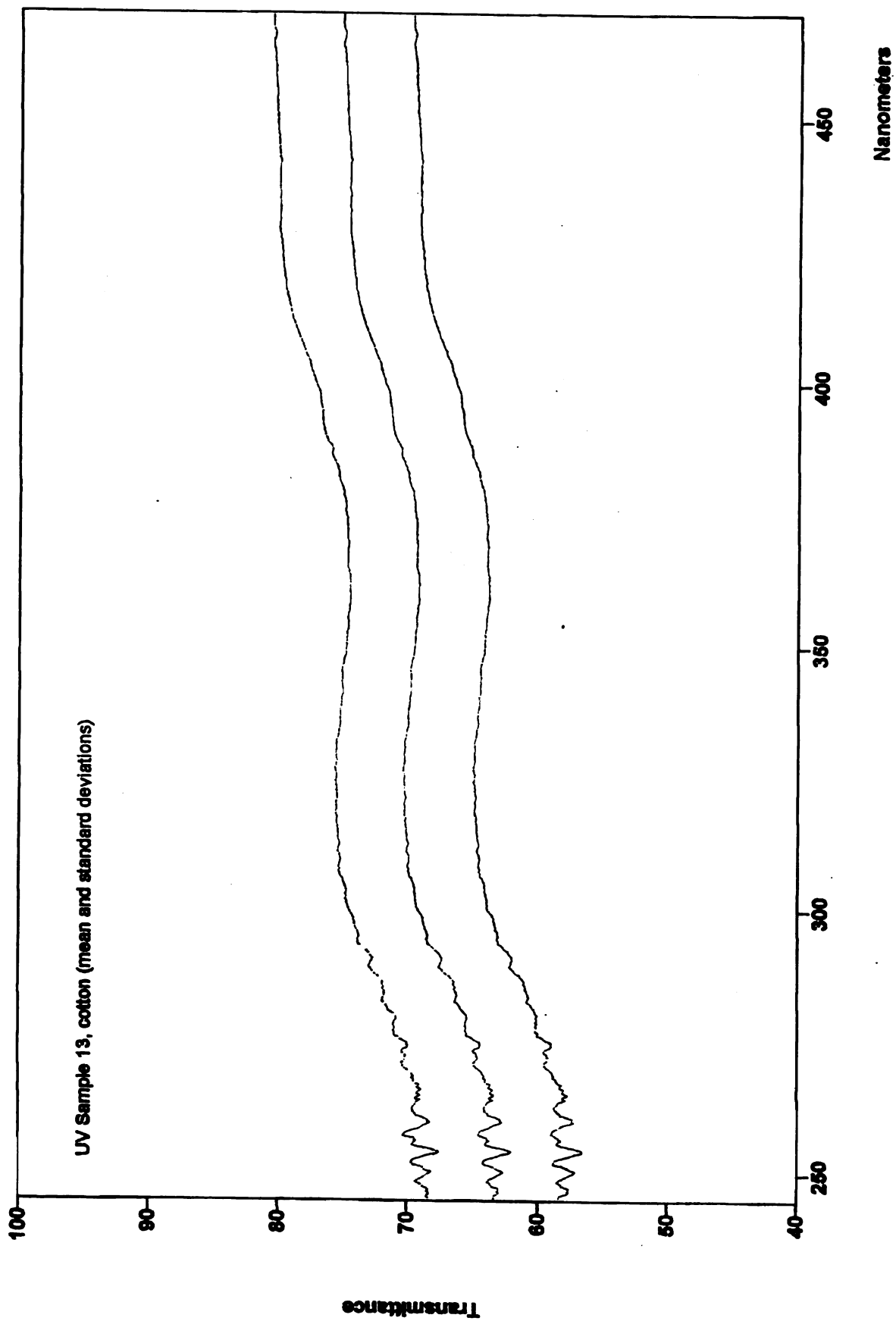


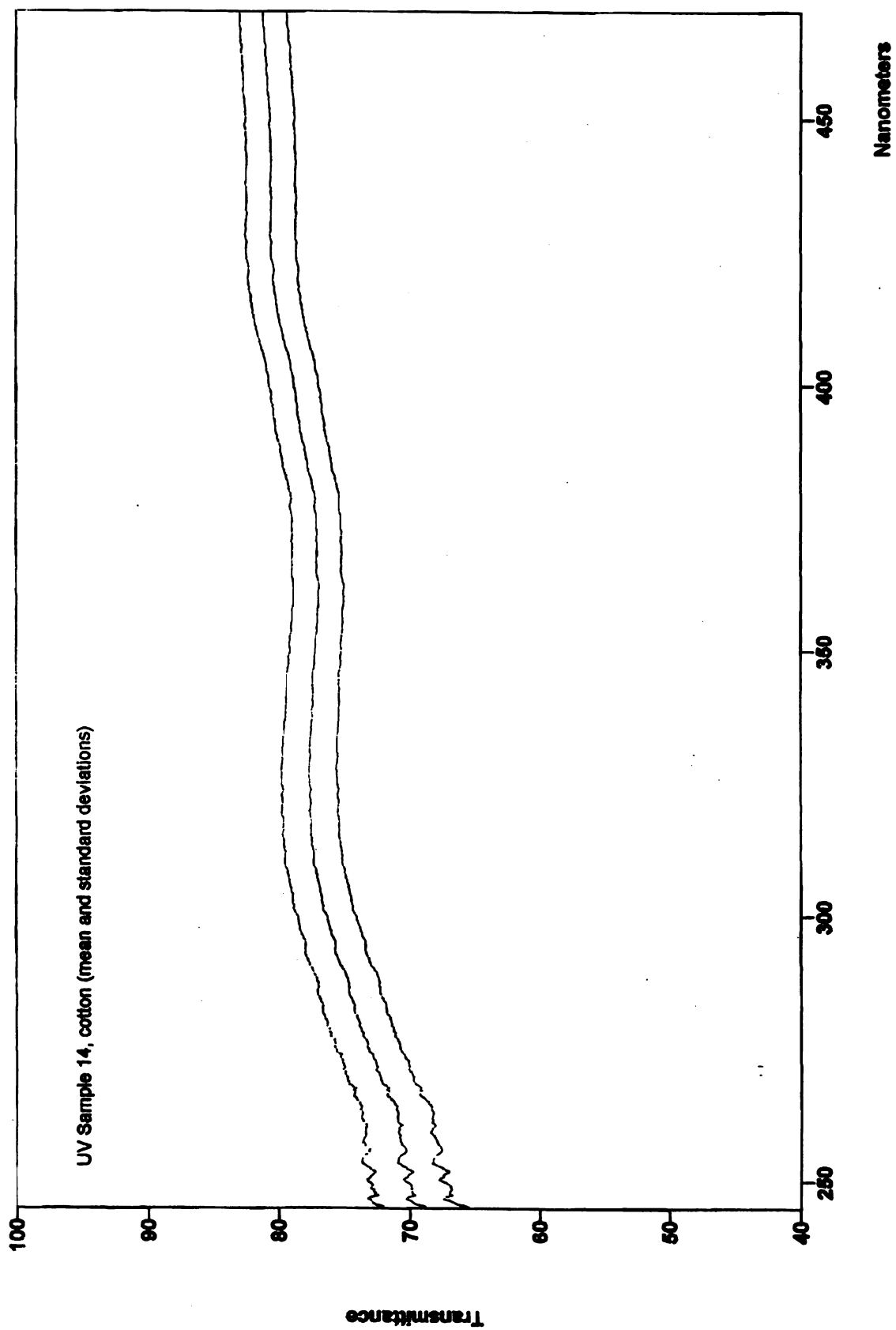


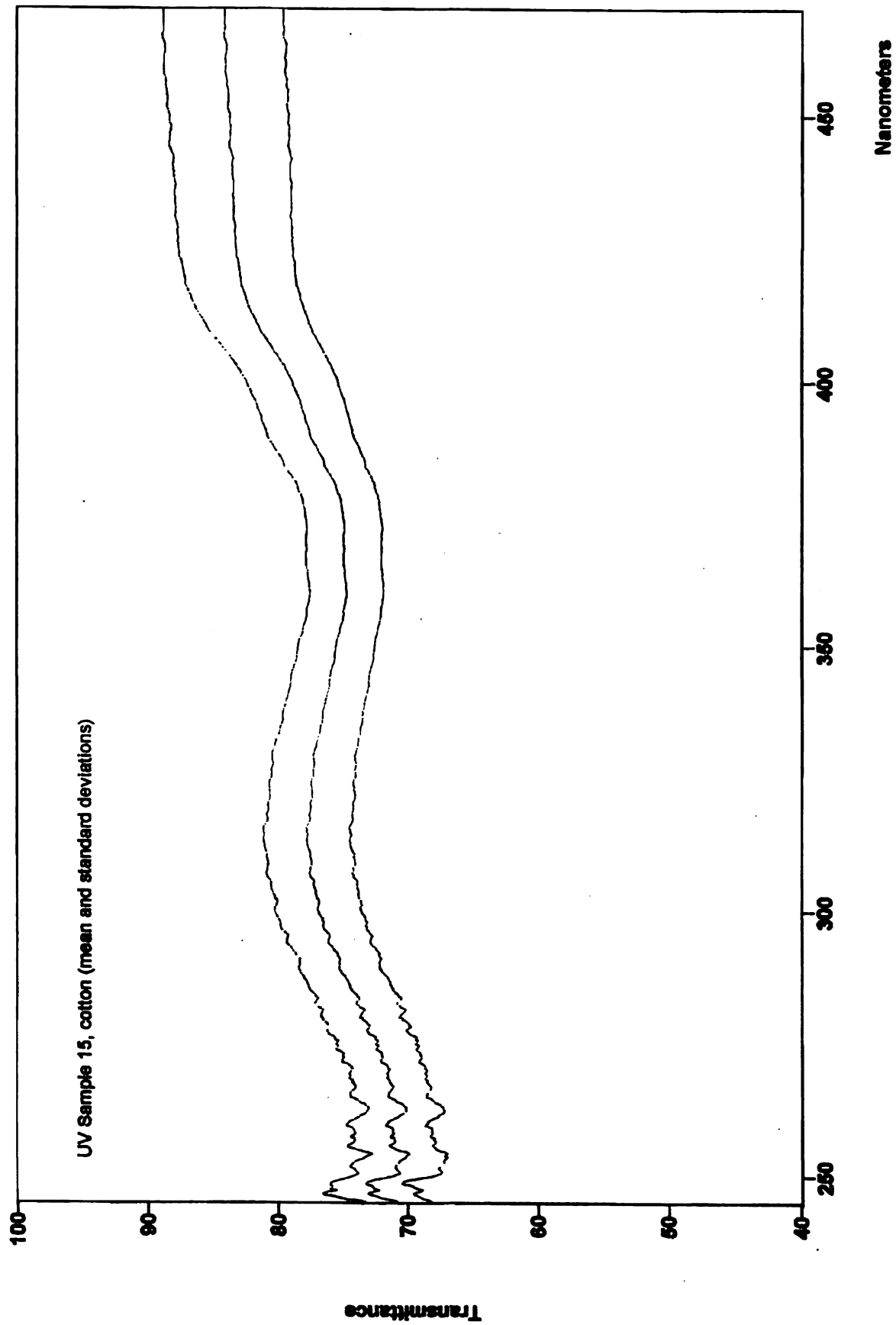


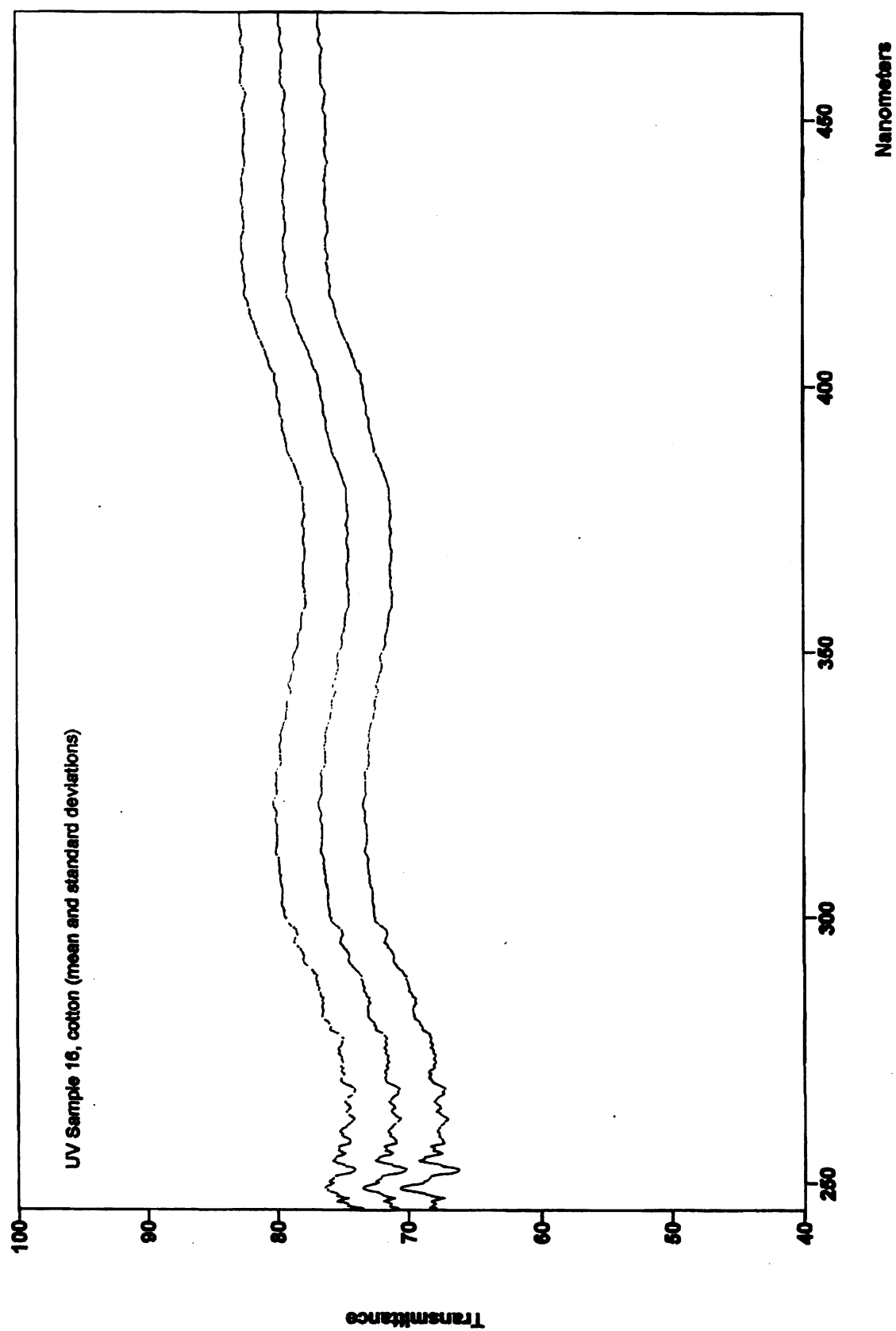


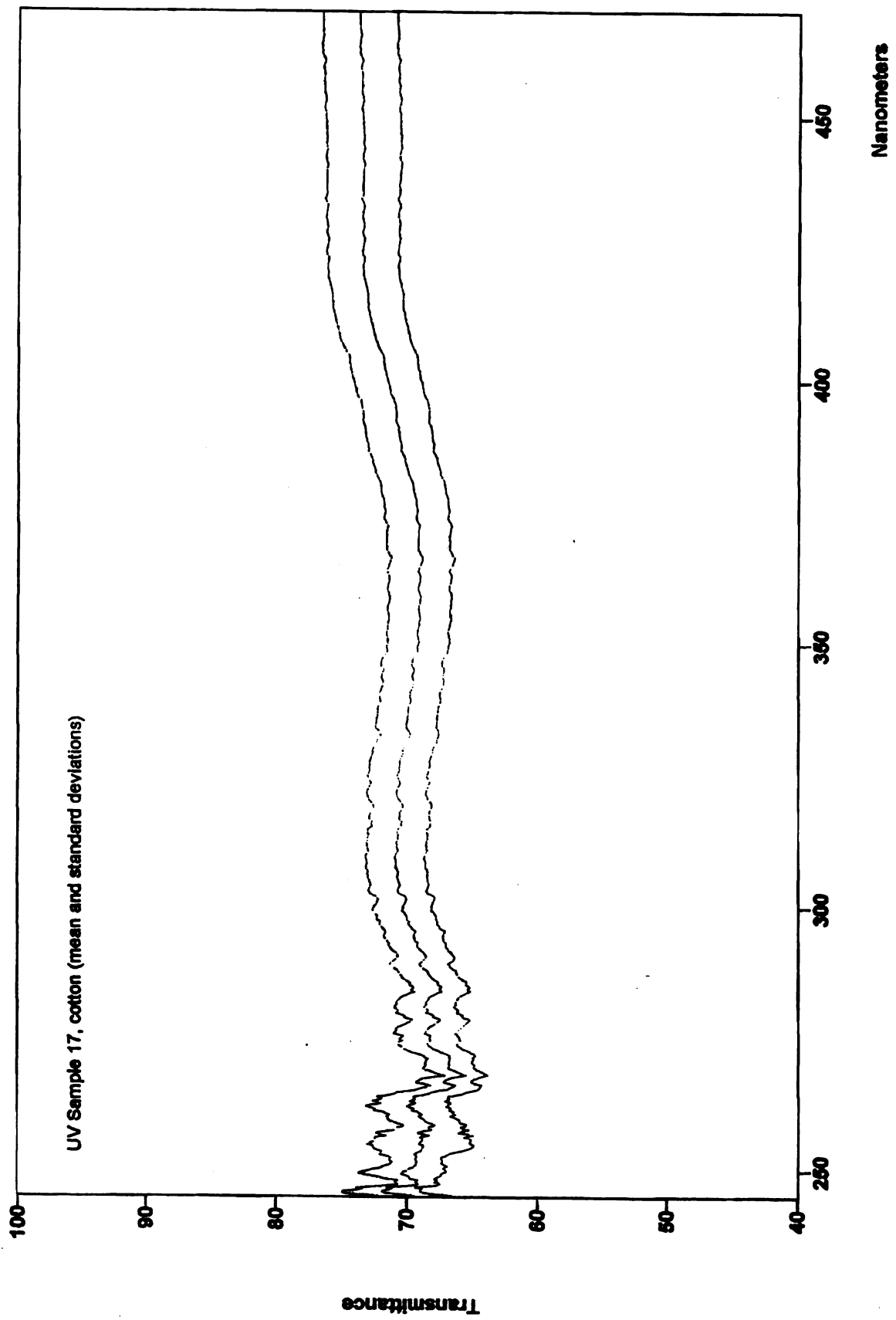


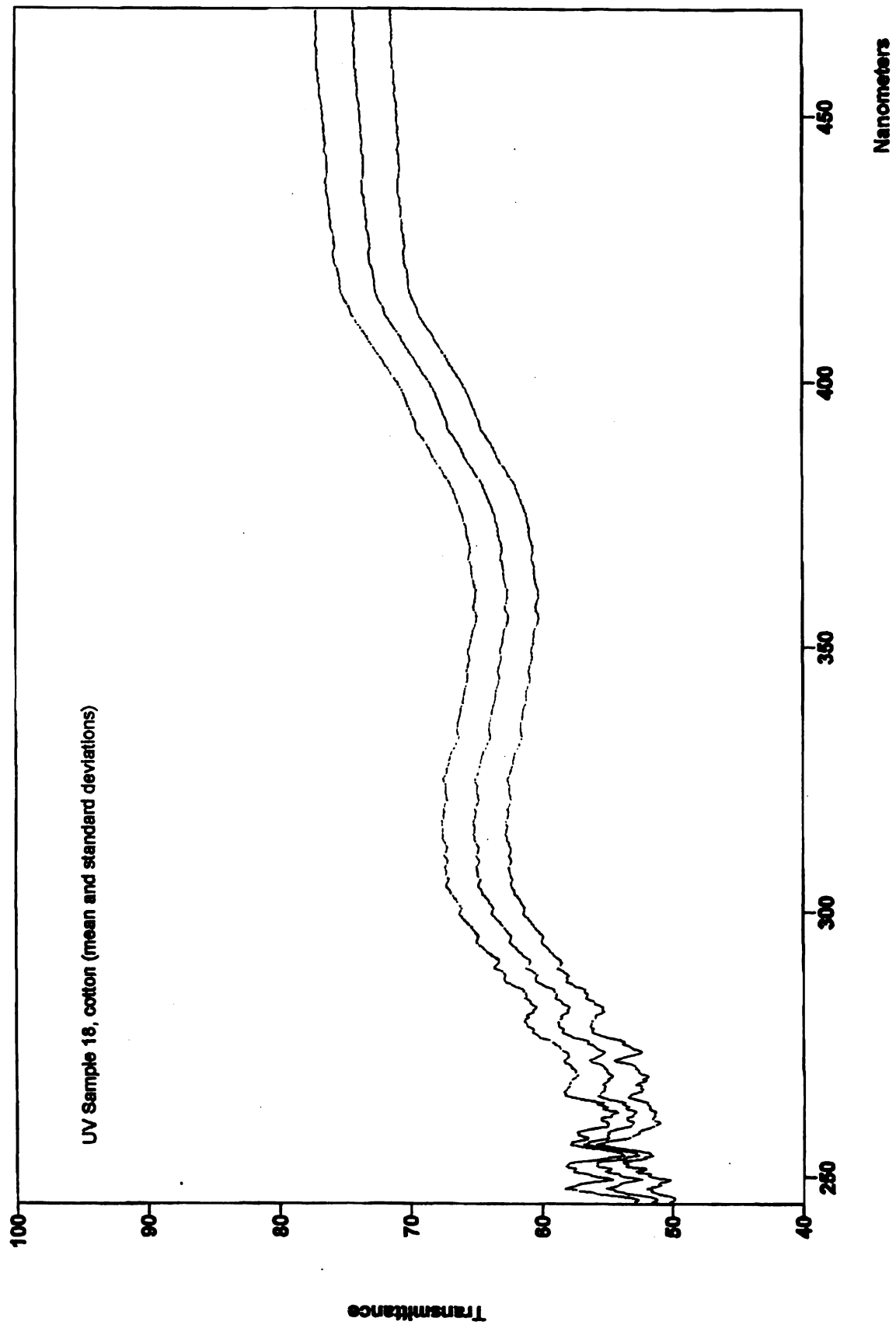


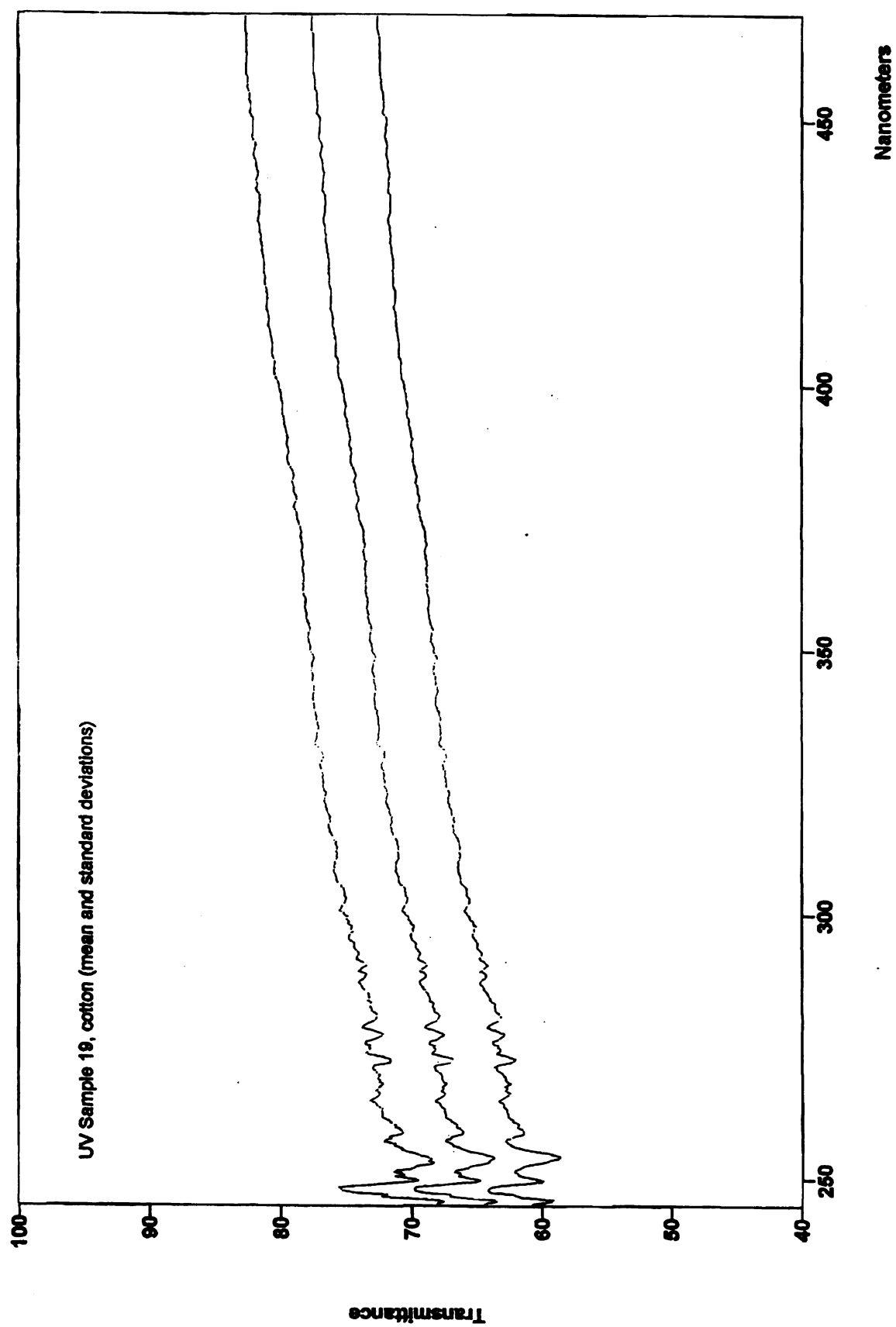


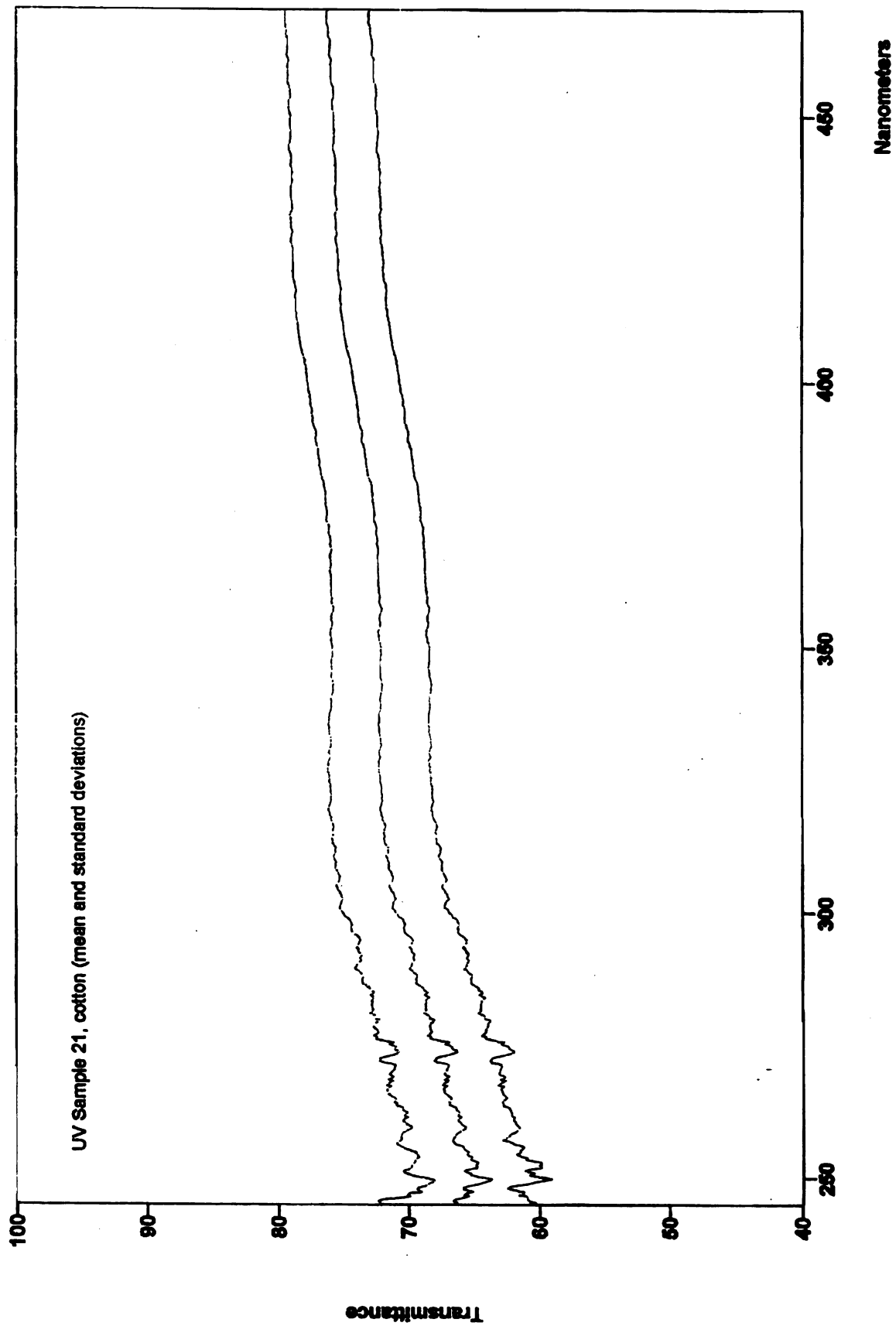


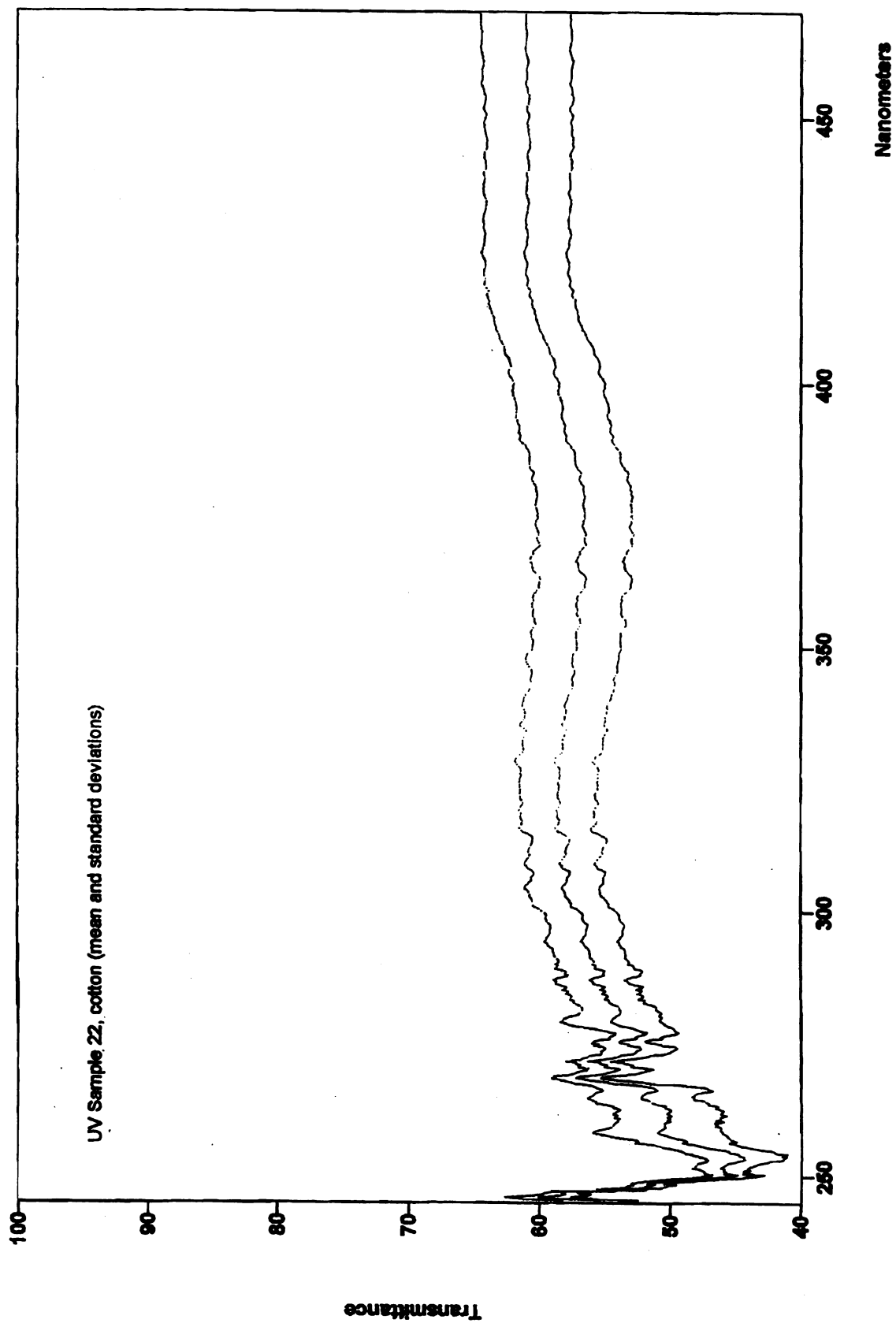


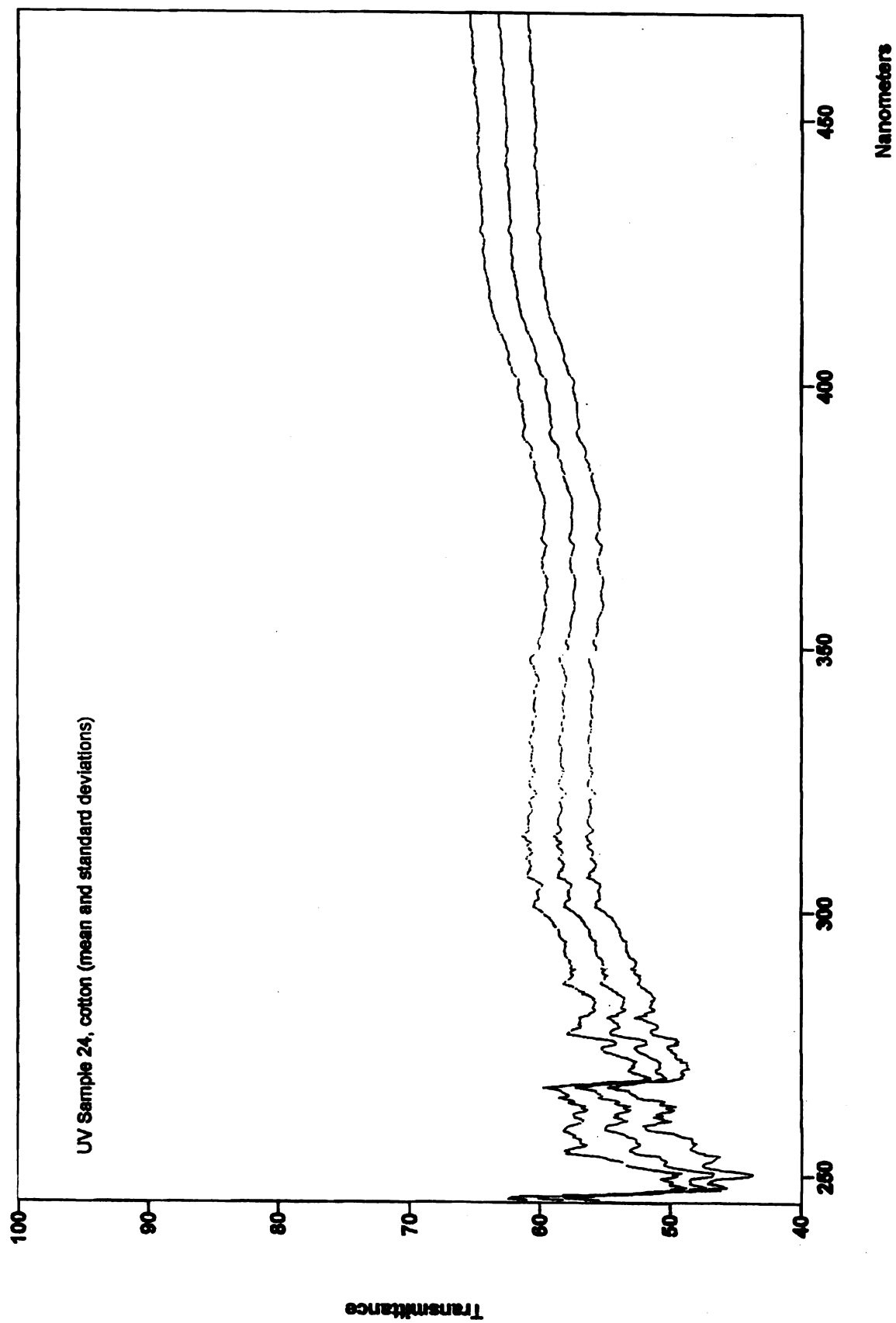


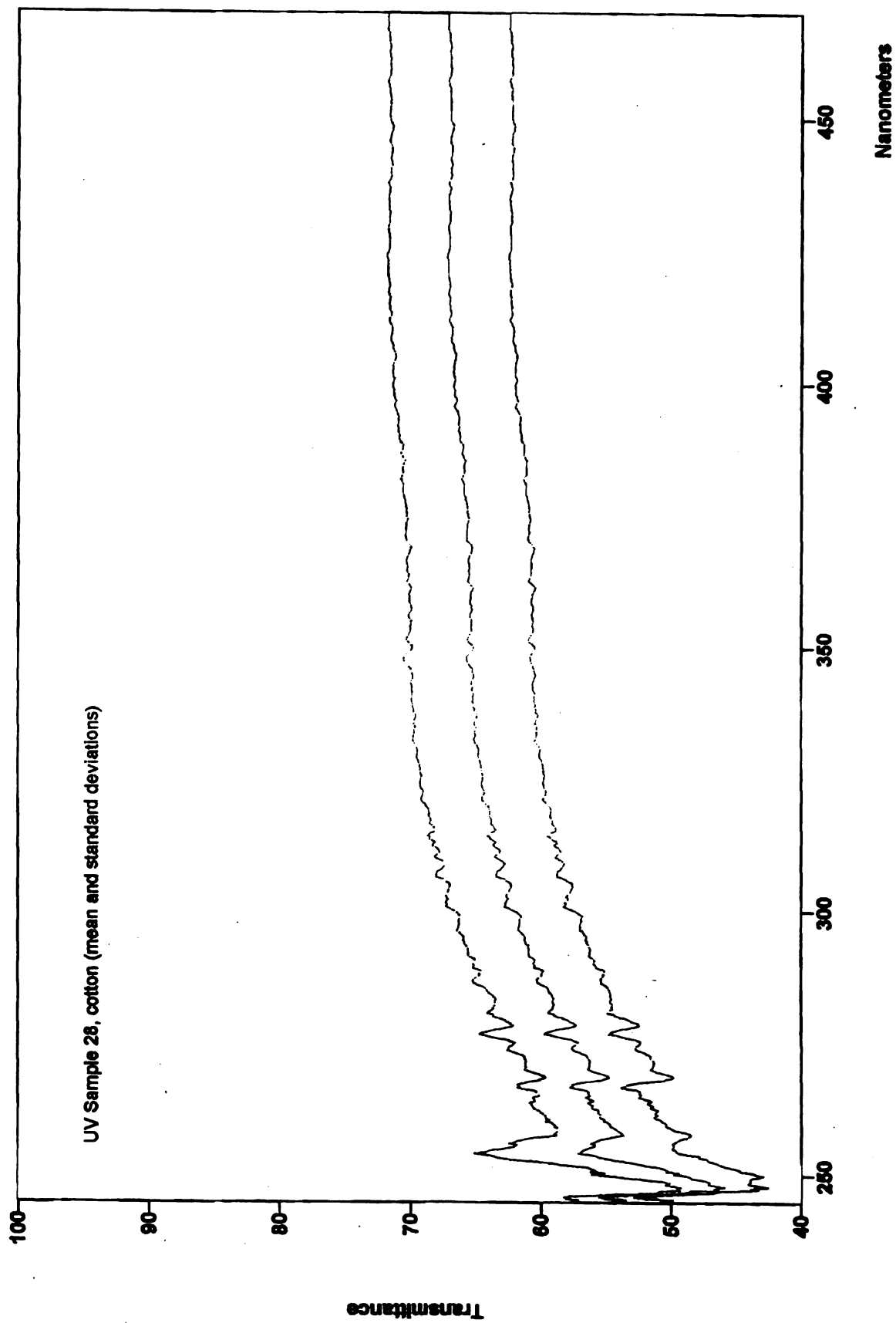


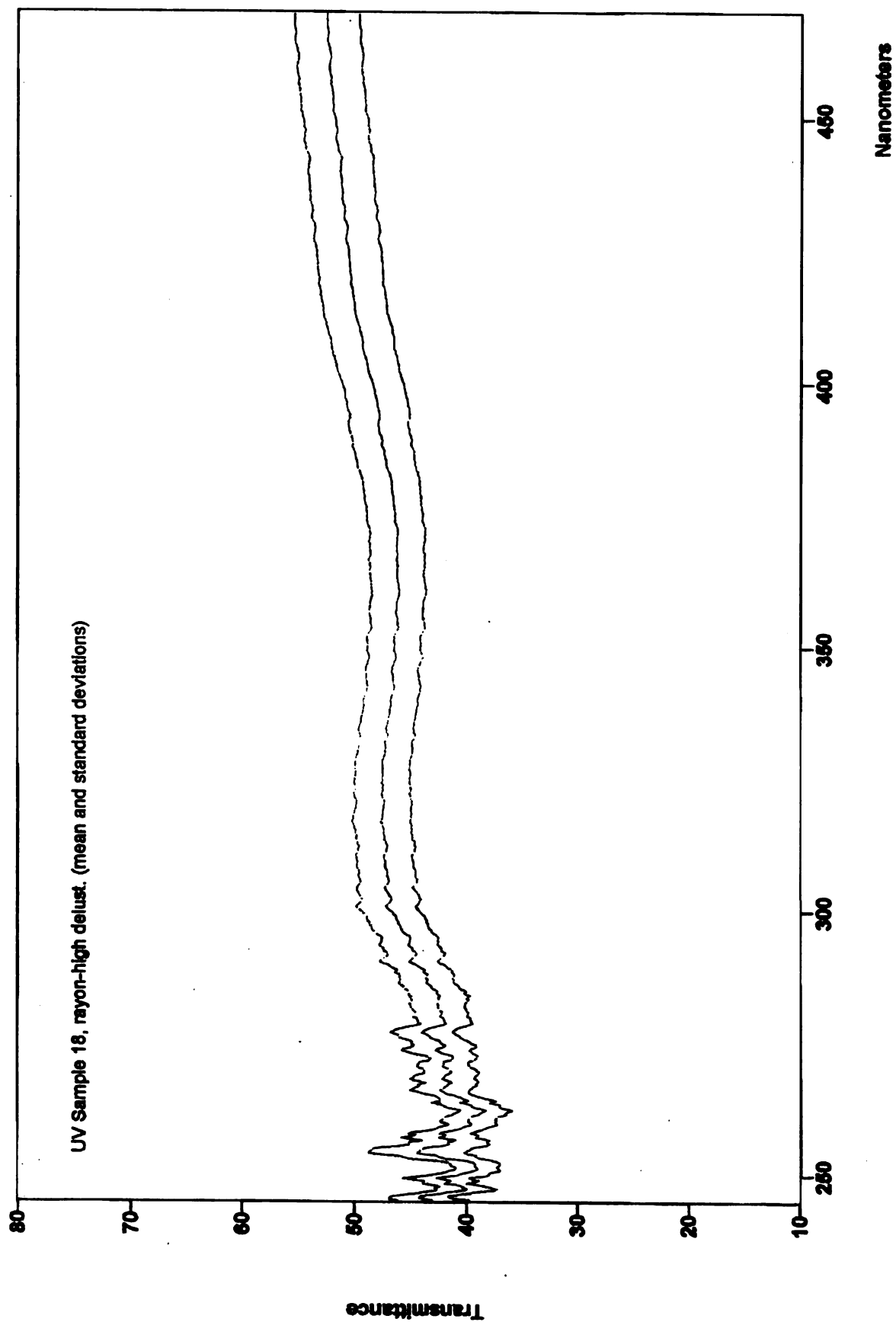


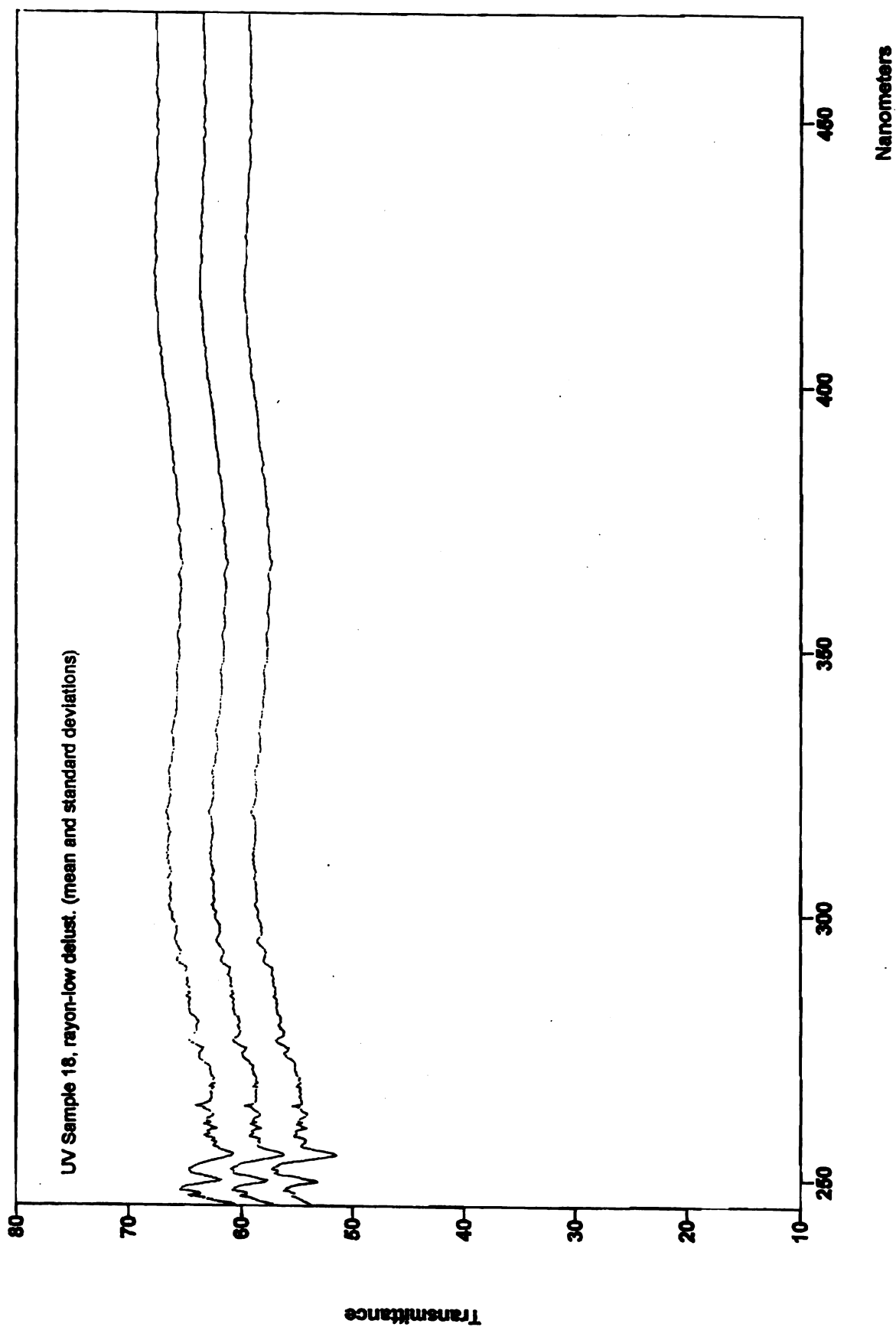


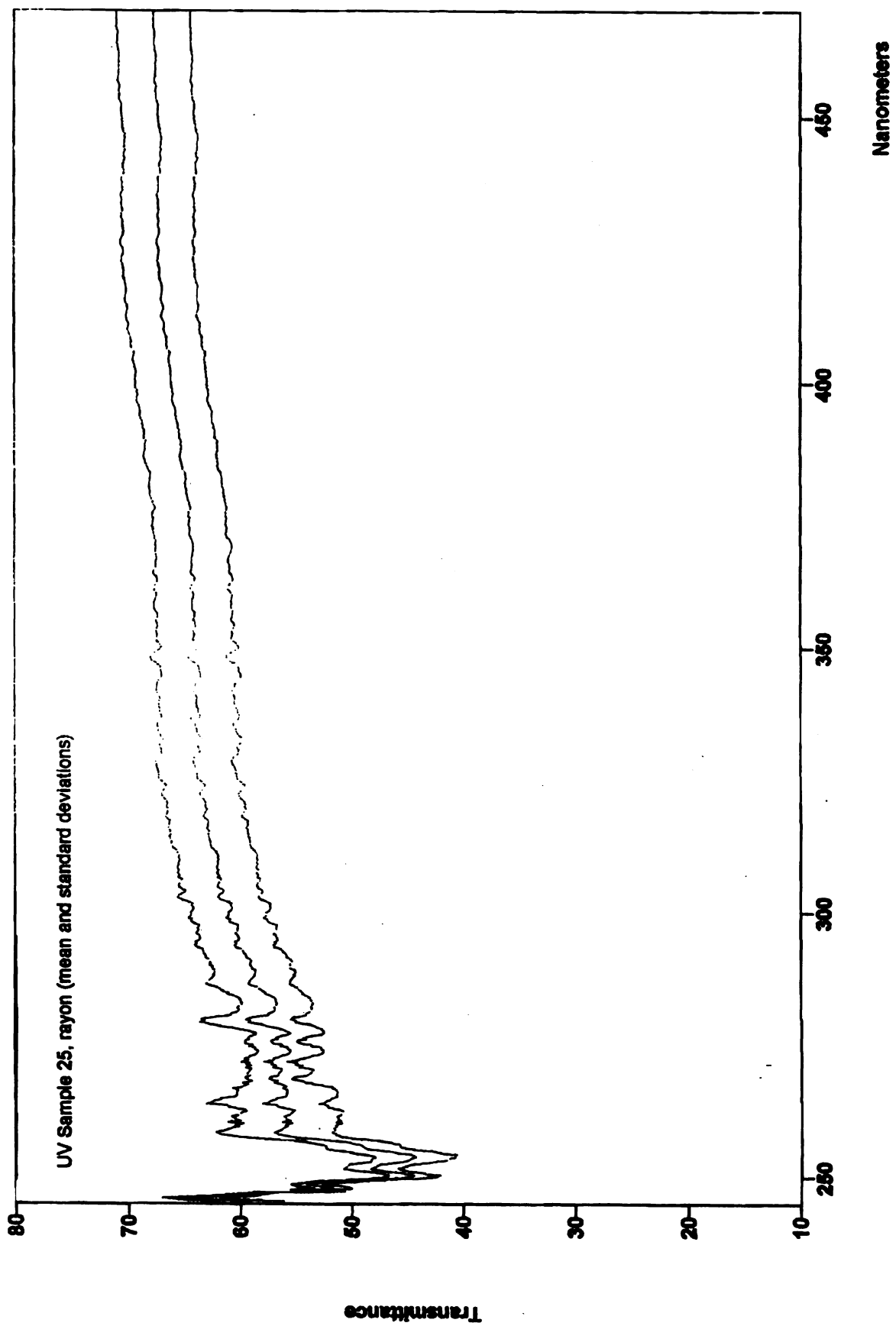


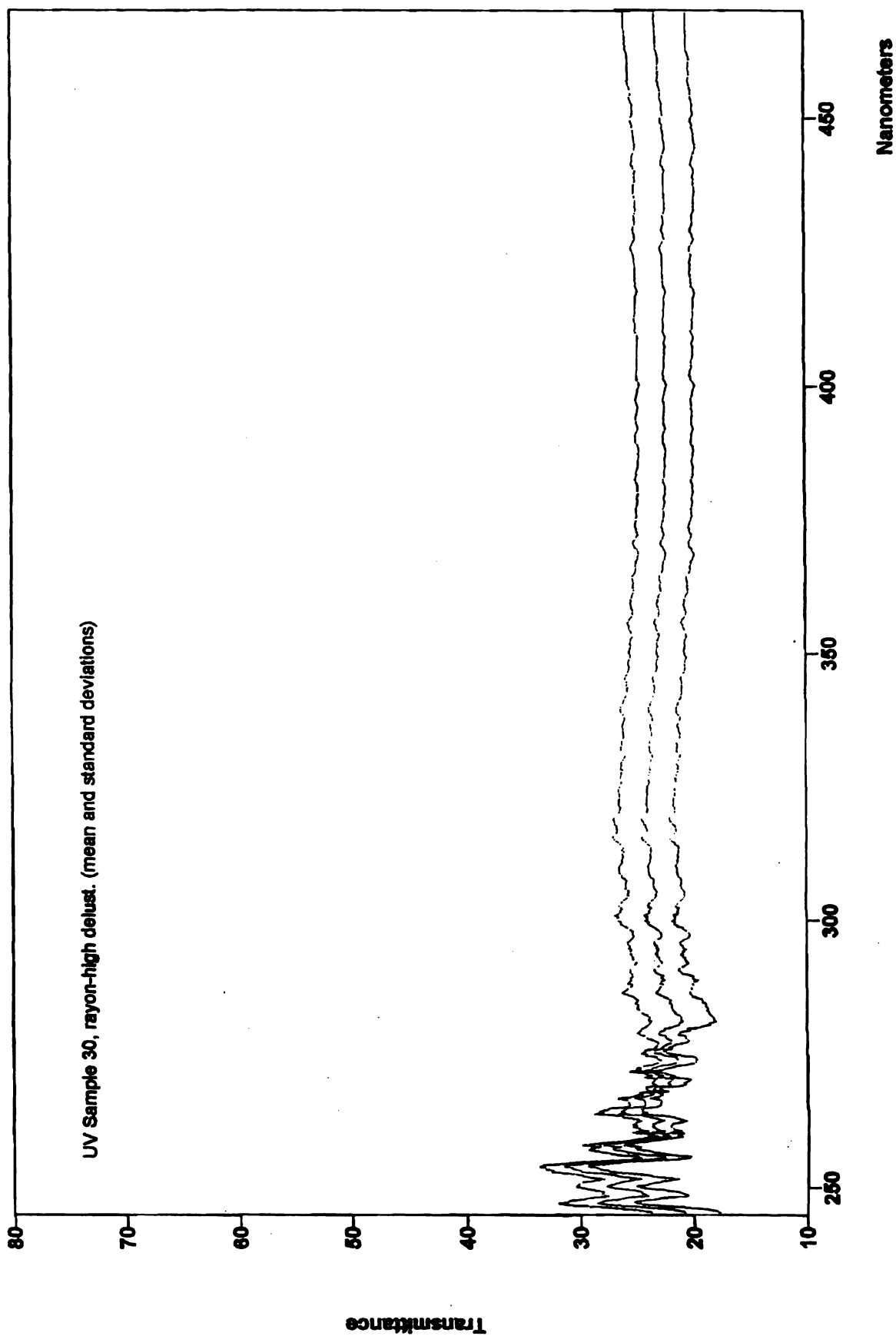


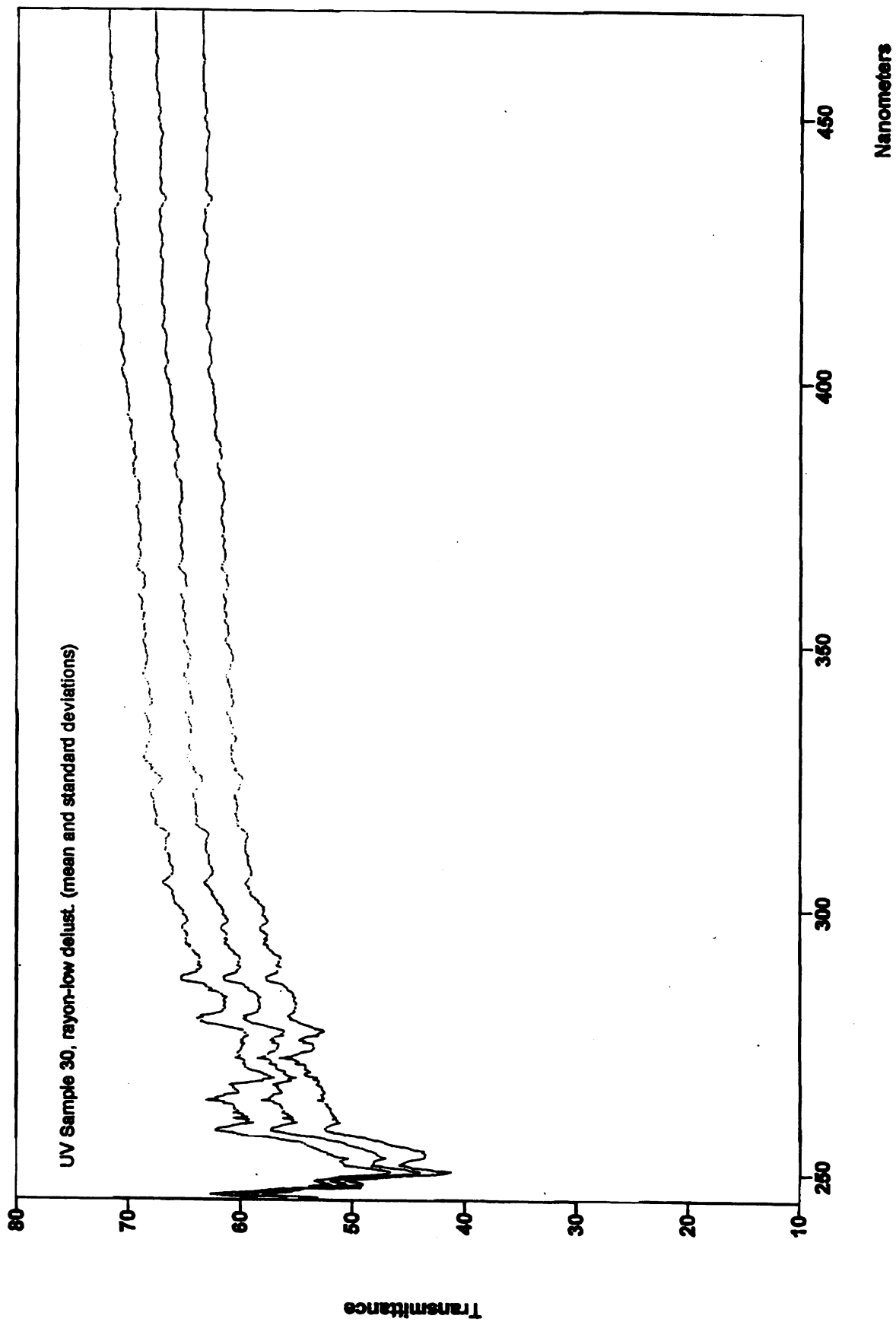


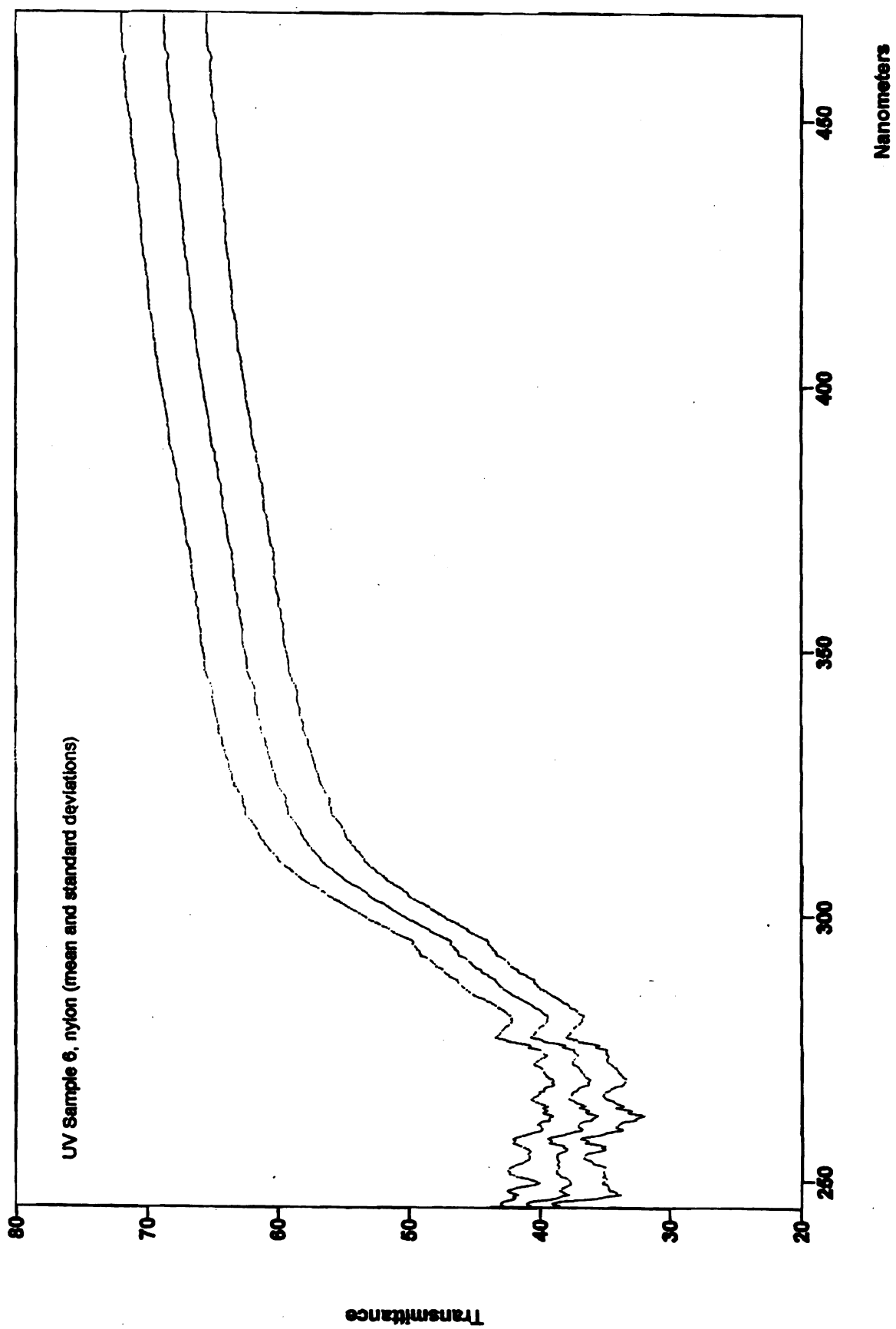


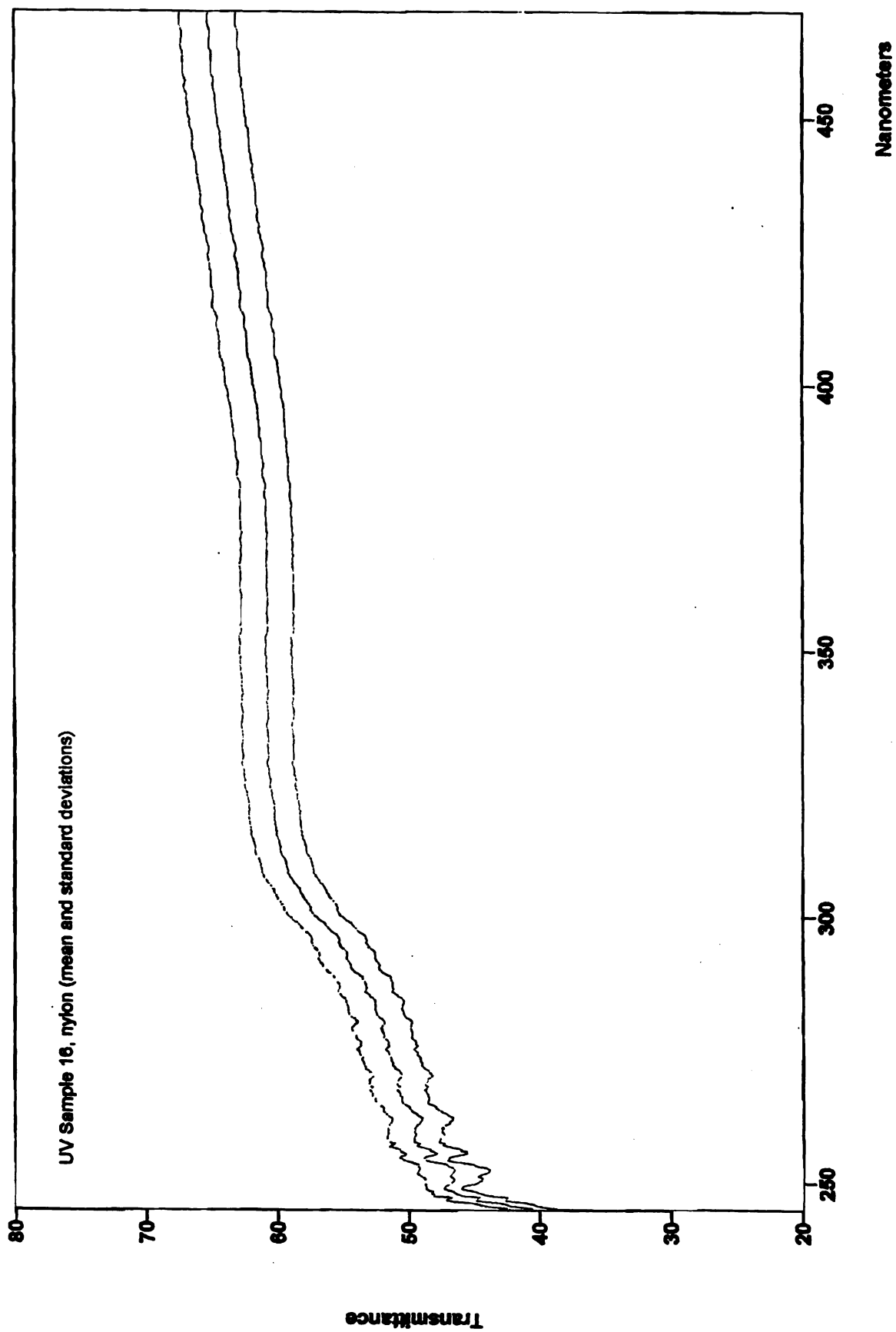


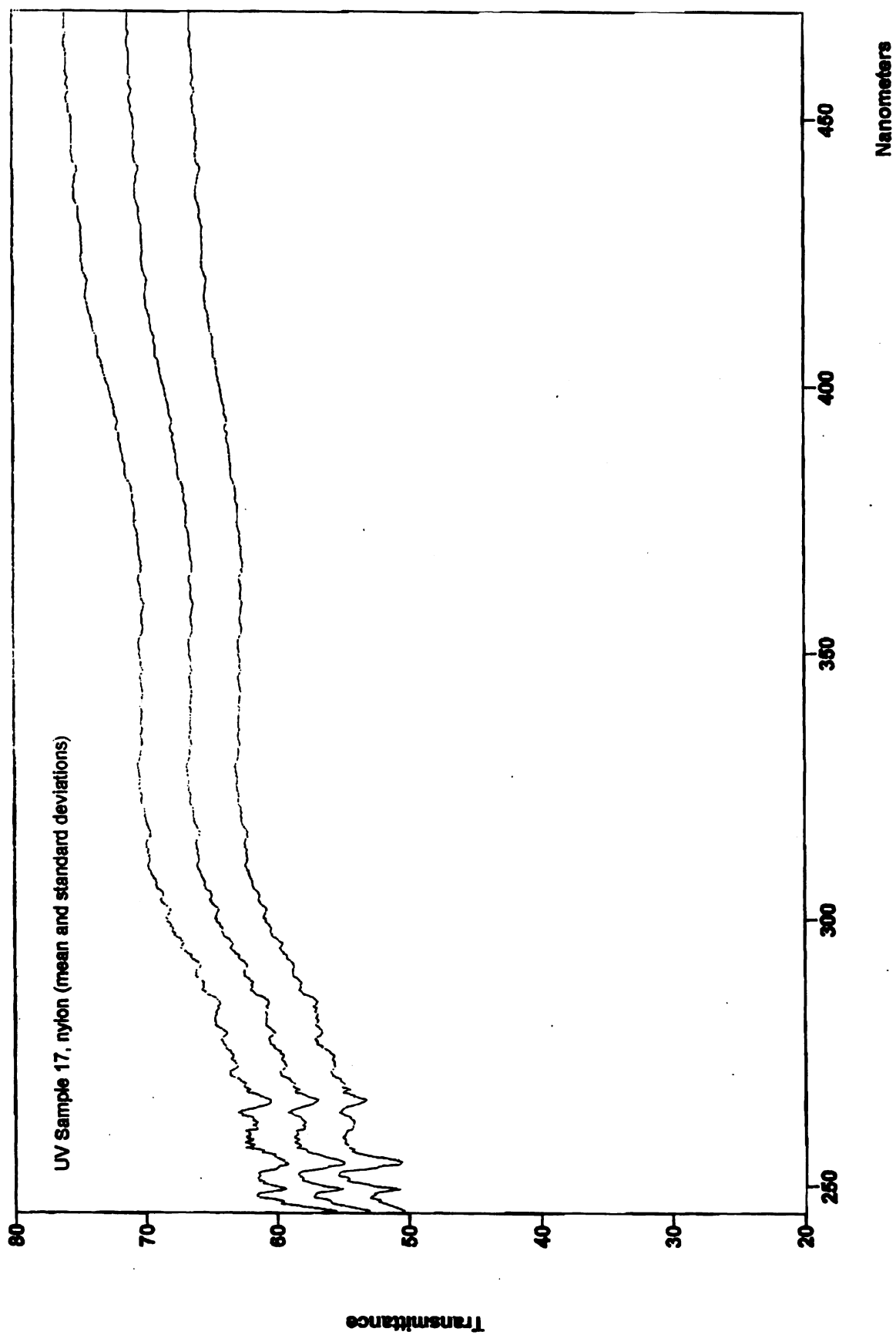


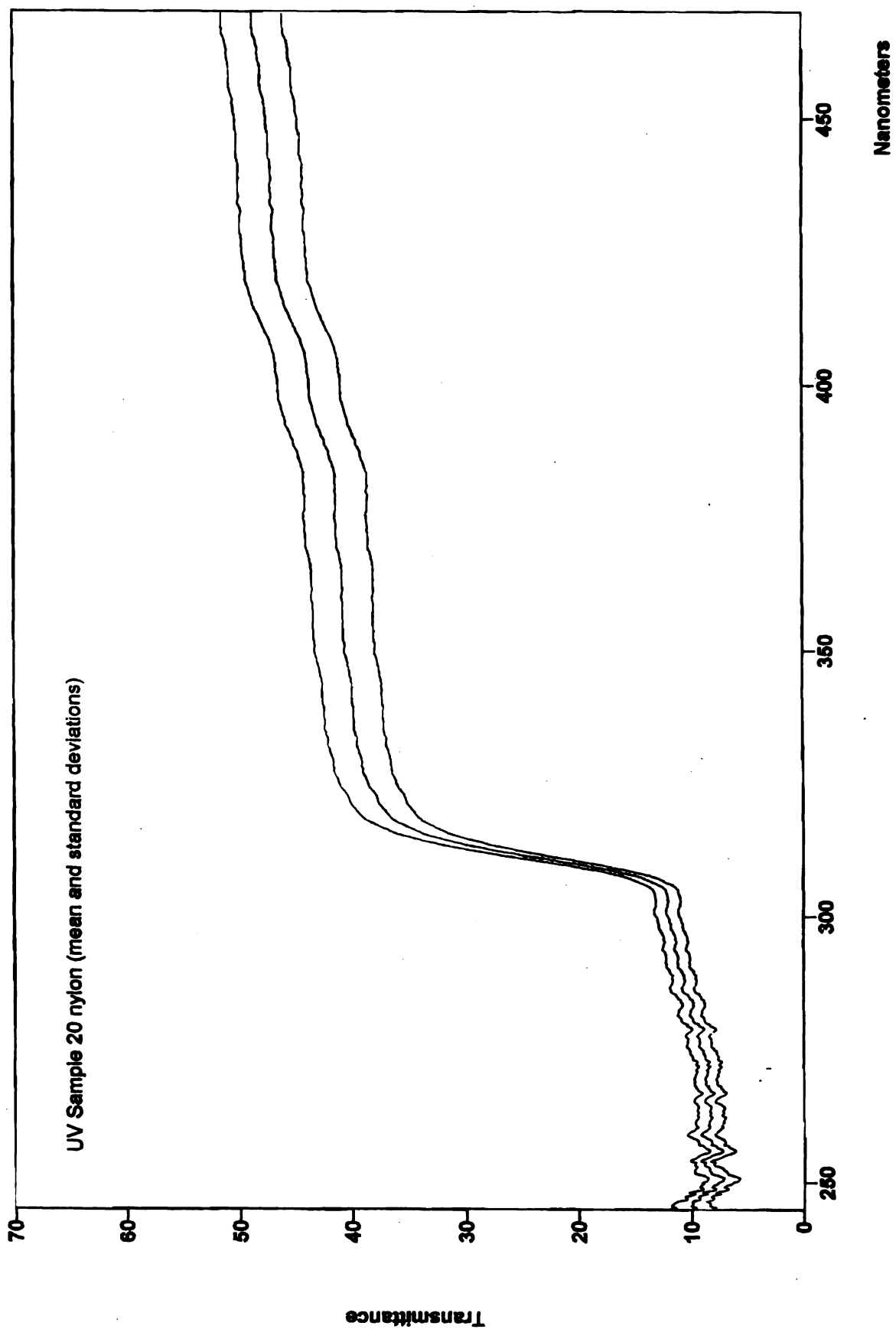


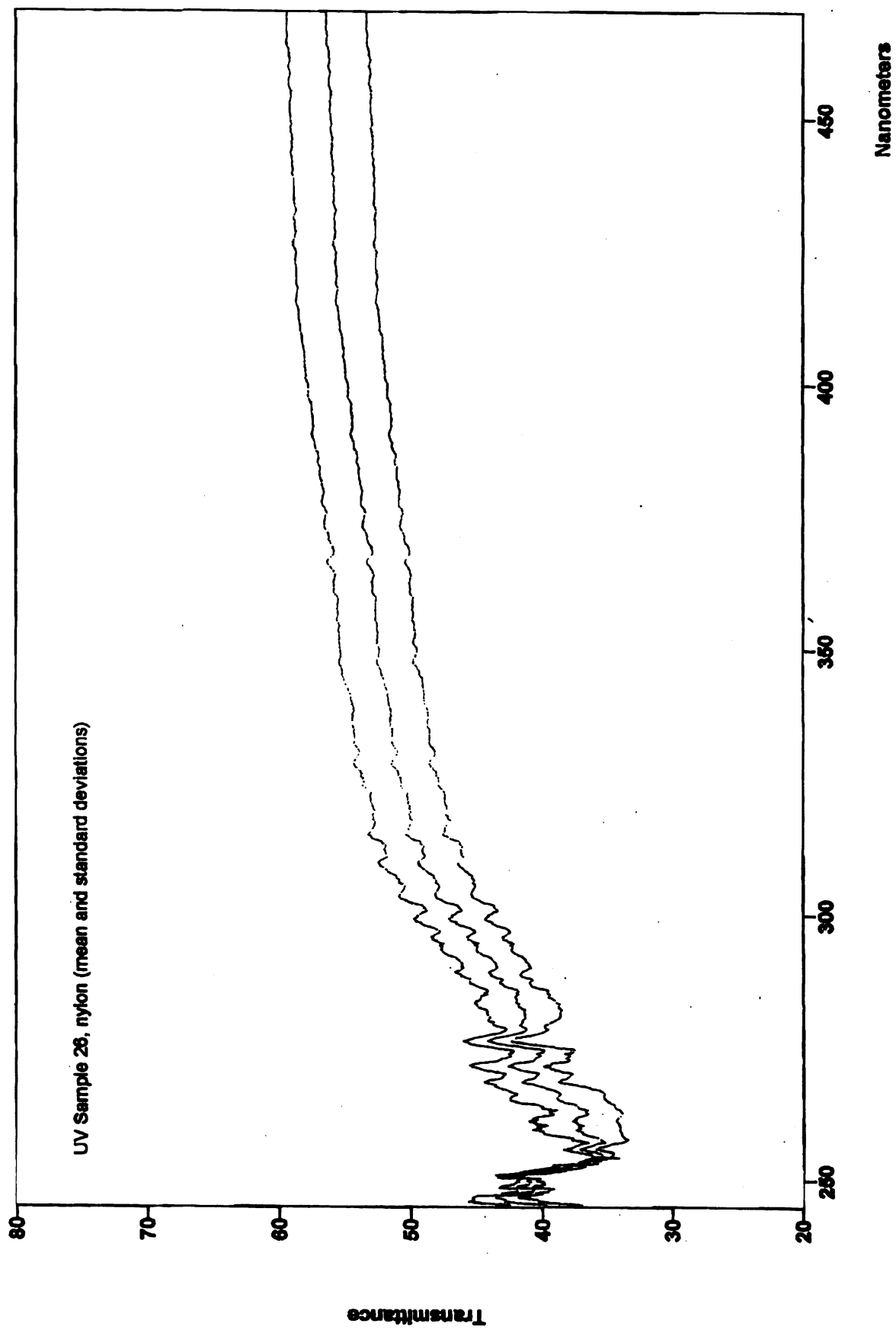


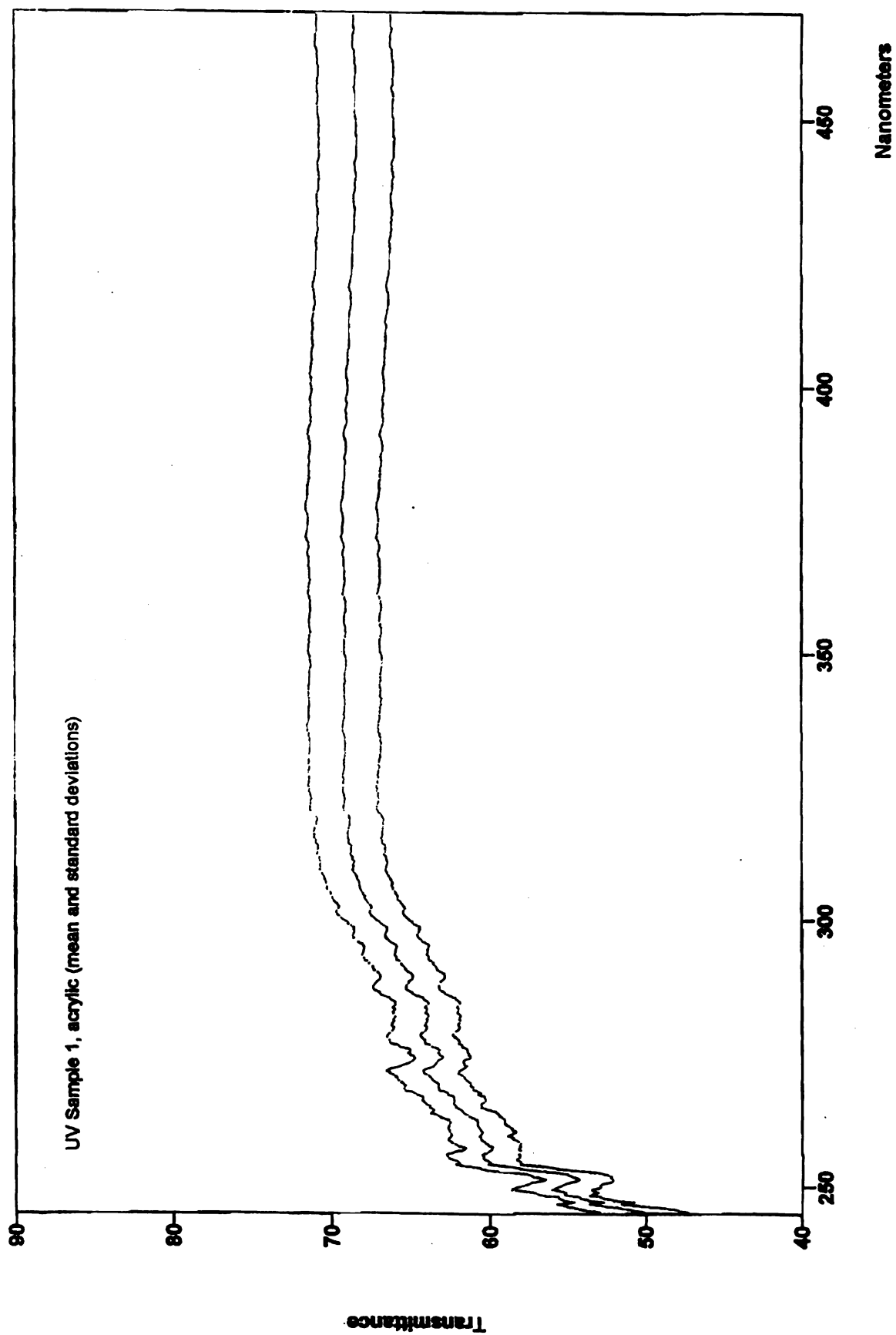


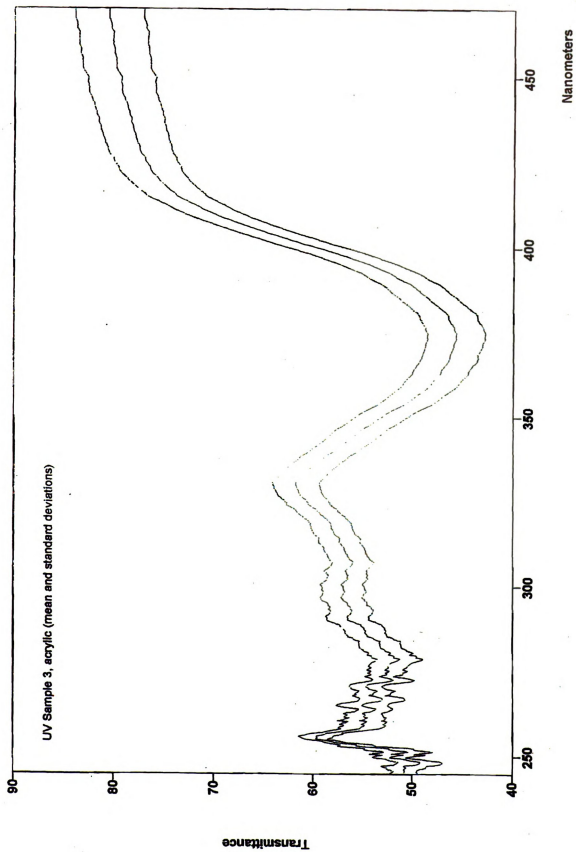


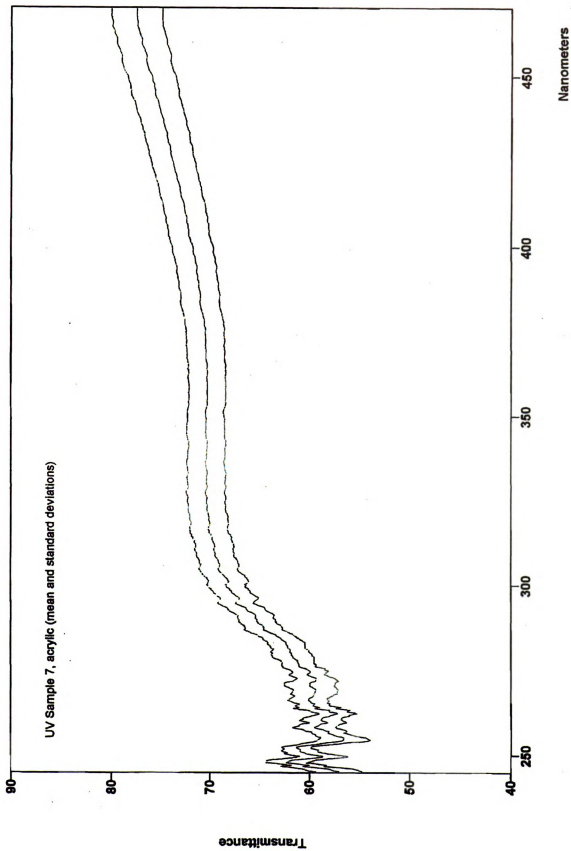


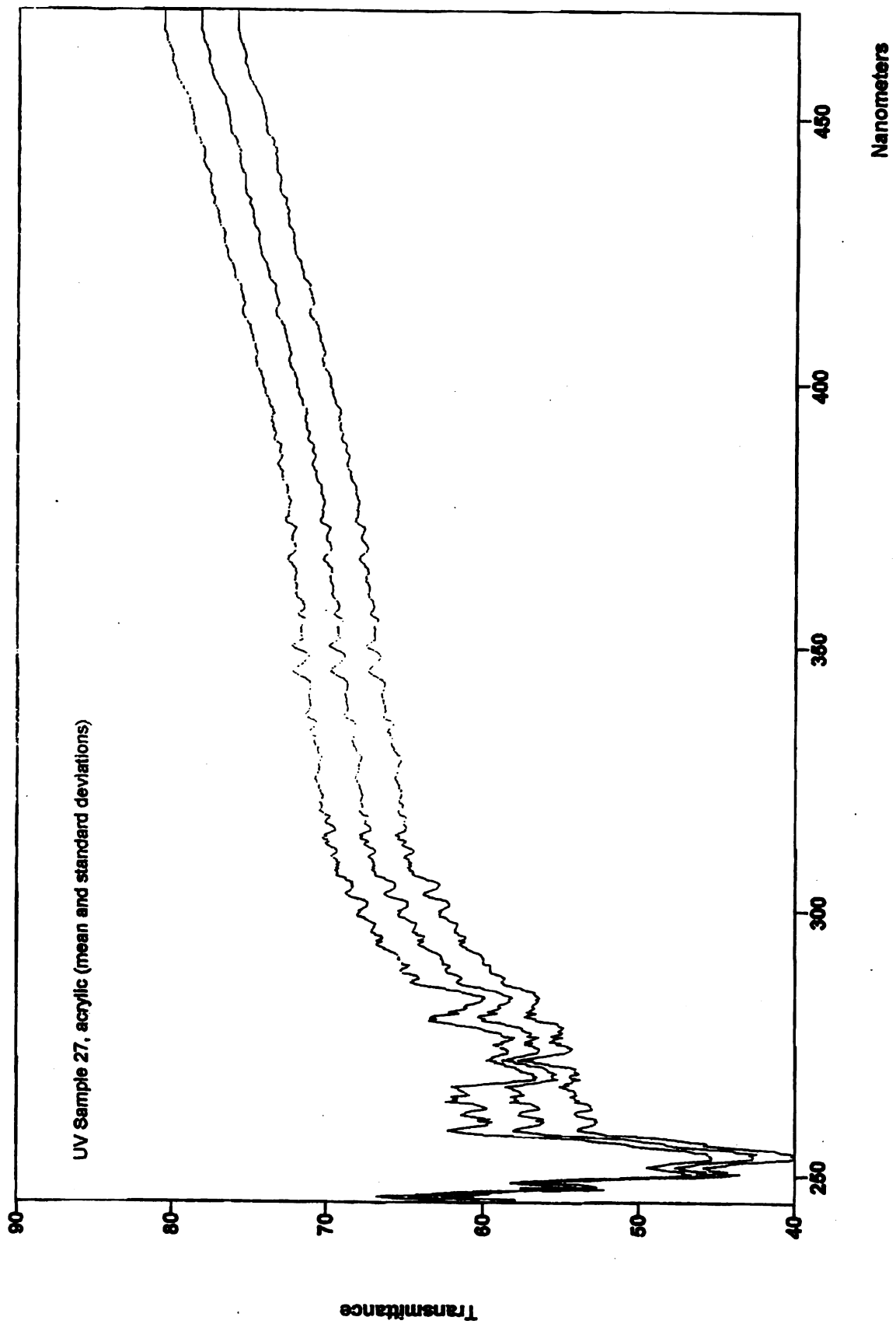


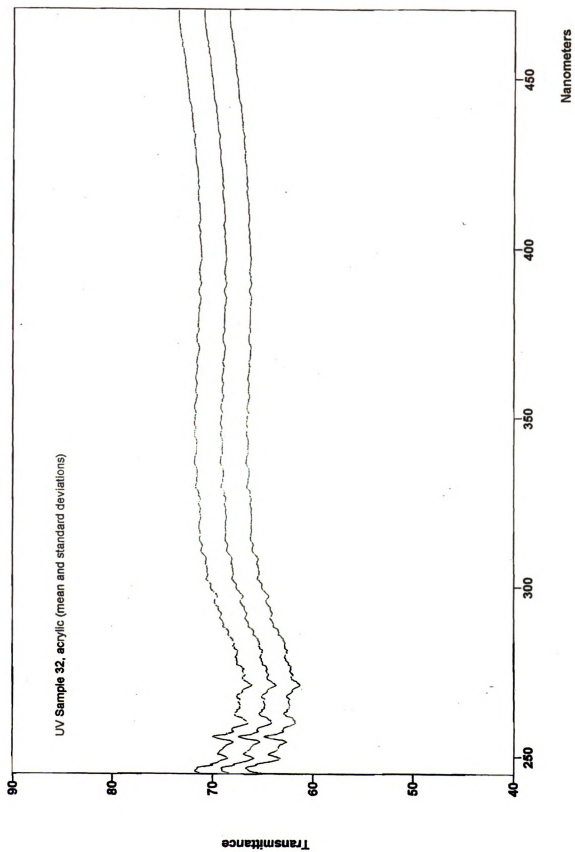












REFERENCES

1. U.S. Department of Justice, Federal Bureau of Investigation, "Microscopy of Textile Fibers," *Forensic Science Communications* 1, no. 1 (1999): <http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch2.htm>.
2. Samuel J. Palenik, "Microscopical Examination of Fibers," in *Forensic Examination of Fibers*, ed. James Robertson and Michael Grieve, 2nd ed. (Philadelphia: Taylor & Francis, 1999), 155-56.
3. Barry D. Gaudette, "The Forensic Aspects of Textile Fiber Examination," in *Forensic Science Handbook, Vol. 2*, ed. Richard Saferstein (Englewood Cliffs: Prentice Hall, 1988), 241.
4. James Dunlop, "Colour Analysis by Microspectrophotometry," in *Forensic Examination of Fibres*, ed. James Robertson (England: CRC Press, 1992), 127-28.
5. Franz-Peter Adolf, "Microscope Photometry and its Application in Forensic Science, Part 1," Presenter's notes from presentation in Helsinki, Finland, (1986), 8.
6. U.S. Department of Justice, Federal Bureau of Investigation, "Visible Spectroscopy of Textile Fibers," *Forensic Science Communications* 1, no. 1 (1999): <http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch3.htm>.
7. Franz-Peter Adolf and James Dunlop "Microspectrophotometry/Colour Measurement," in *Forensic Examination of Fibers*, ed. James Robertson and Michael Grieve, 2nd ed. (Philadelphia: Taylor & Francis, 1999), 251-52.
8. CRAIC Technologies, *UV-visible-NIR-microscope spectroscopy*, <http://www.microspectra.com>.
9. Dr. Paul Martin, "Ultraviolet Microspectral Analysis of Bleached Wool," *S.E.E. Incorporated Applications Report*, (1999), 1-8.
10. Andrea Desrosiers and Dr. Paul Martin, "Ultraviolet Microspectral Analysis of Nylon Fibers," *S.E.E. Incorporated Applications Report*, (1999), 1-4.
11. Mitchell Kupferman, letter from applications scientist at S.E.E. Incorporated, 9 September, 2002.
12. Charles R. Woodfall, *Microscope Micron Measuring*, <http://www.microscopy-uk.net/mag/artdec99/cwnano.html>.

BIBLIOGRAPHY

- Abramowitz, Mortimer. *Fluorescence Microscopy: The Essentials*. Melville: Olympus America, 1993.
- Adolf, Franz-Peter. "Microscope Photometry and its Application in Forensic Science, Part 1," Presenter's notes from presentation in Helsinki, Finland, (1986), 8.
- Adolf, Franz-Peter, and James Dunlop. "Microspectrophotometry/Colour Measurement." In *Forensic Examination of Fibers*, edited by James Robertson and Michael Grieve. 2nd ed., 251-90. Philadelphia: Taylor & Francis, 1999.
- CRAIC Technologies. *UV-visible-NIR-microscope spectroscopy*. <http://www.microspectra.com>.
- Desrosiers, Andrea, and Dr. Paul Martin. "Ultraviolet Microspectral Analysis of Nylon Fibers." *S.E.E. Incorporated Applications Report*. (1999): 1-4.
- Dunlop, James. "Colour Analysis by Microspectrophotometry." In *Forensic Examination of Fibres*, edited by James Robertson, 127-40. England: CRC Press, 1992.
- Gaudette, Barry D. "The Forensic Aspects of Textile Fiber Examination,," In *Forensic Science Handbook, Vol. 2*, edited by Richard Saferstein, 209-62. Englewood Cliffs: Prentice Hall, 1988.
- Martin, Paul. "Ultraviolet Microspectral Analysis of Bleached Wool." *S.E.E. Incorporated Applications Report*. (1999): 1-8.
- Palenik, Samuel J. "Microscopical Examination of Fibers." In *Forensic Examination of Fibers*, edited by James Robertson and Michael Grieve. 2nd ed., 153-78. Philadelphia: Taylor & Francis, 1999.
- U.S. Department of Justice, Federal Bureau of Investigation. "Introduction to Forensic Fiber Examination." In *Forensic Science Communications* 1, no. 1 (1999). <http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch1.htm>.
- U.S. Department of Justice, Federal Bureau of Investigation. "Microscopy of Textile Fibers." In *Forensic Science Communications* 1, no. 1 (1999). <http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch2.htm>.
- U.S. Department of Justice, Federal Bureau of Investigation. "Visible Spectroscopy of Textile Fibers." In *Forensic Science Communications* 1, no. 1 (1999). <http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houckch3.htm>.
- Woodfall, Charles R. *Microscope Micron Measuring*. <http://www.microscopy-uk.net/mag/artdec99/cwnano.html>.