

**Monitoring Dielectric Thin-Film Production on Product Wafers Using Infrared Emission
Spectroscopy***

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Abstract

Monitoring of dielectric thin-film production in the microelectronics industry is generally accomplished by depositing a representative film on a monitor wafer and determining the film properties off line. One of the most important dielectric thin films in the manufacture of integrated circuits is borophosphosilicate glass (BPSG). The critical properties of BPSG thin films are the boron content, phosphorus content and film thickness. We have completed an experimental study that demonstrates that infrared emission spectroscopy coupled with multivariate analysis can be used to simultaneously determine these properties directly from the spectra of product wafers, thus eliminating the need of producing monitor wafers. In addition, infrared emission data can be used to simultaneously determine the film temperature, which is an important film production parameter. The infrared data required to make these determinations can be collected on a time scale that is much faster than the film deposition time, hence infrared emission is an ideal candidate for an *in-situ* process monitor for dielectric thin-film production.

Key Words: IR emission, dielectric thin films, multivariate calibration, partial least squares (PLS), borophosphosilicate glass (BPSG)

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Introduction

Thin films produced using doped silicon oxide are widely used in the microelectronics industry as dielectrics between metallization layers, as passivation and planarization layers, and as traps (getters) for mobile alkali cations.¹ The most commonly used doped silica is borophosphosilicate glass (BPSG), with dopant concentrations of the films ranging from 1 to 6% for B and 2 to 8% for P. BPSG films are generally deposited using chemical vapor deposition (CVD). Quality control methods have traditionally consisted of periodically running a monitor wafer through the CVD apparatus and determining the B and P content using traditional laboratory methods after etching the film off the silicon substrate.² The time required to transport the monitor wafer to the laboratory, etch the film from the substrate, and perform the analysis is far less than ideal as a product monitor. Indeed, the actual product is not the subject of the monitoring process.

A number of analytical methods have been investigated as at-line quality control monitors for the production of these films. The most promising at-line technique has been infrared (IR) spectroscopy.¹⁻⁵ IR absorption spectroscopy⁶⁻⁸ when coupled with multivariate calibration, has been shown to be capable of meeting the requirements for an at-line quality control monitor of BPSG thin-film production. The monitoring of the thin-film properties using infrared spectroscopy has been carried out by depositing a film on an infrared transparent monitor wafer and collecting the IR data using transmission spectroscopy. IR reflection spectroscopy has been recently shown to be capable of generating data that when coupled with multivariate calibration produces property predictions equivalent in precision to those obtained using absorption spectroscopy.^{9,10} Reflection sampling does not require the beam to pass through the substrate. Indeed, we have recently shown that high quality IR reflection data can be

collected from product wafers and used as the basis for a rapid, accurate, precise, and nondestructive at-line quality monitor for BPSG film production on product wafers.^{11,12} Thus, IR reflection circumvents the need to periodically run a monitor wafer and makes it possible to access the quality of every product wafer. However, as constraints get ever tighter, the need to monitor and control the thin-film production *in-situ* becomes greater.

We have recently shown that non-destructive determination of thin-film dielectric properties on monitor wafers can be made using infrared emission spectroscopy.¹³⁻¹⁶ These experiments illustrated that although IR emission data collected from BPSG thin-film samples are complicated; it is possible to collect highly precise spectra on a time scale that is fast relative to the CVD process. Multivariate analysis methods applied to these spectra then provide rapid measurements of B content, P content, film temperature, and film thickness. Although these earlier studies were carried out on monitor wafers, IR emission sampling does not require an infrared transparent substrate. Further, IR emission does not require a probe beam. All that is required for *in-situ* monitoring is that there be optical access to the film being deposited, that data collection and processing be much faster than the deposition process, and that the sample temperature be different than that of the infrared detector. Infrared detectors are often cooled, and the substrate temperatures in CVD processes are elevated, hence the latter condition is readily achieved.

In this paper, we show that it is possible to simultaneously determine B content, P content, film thickness, and film temperature from IR emission spectra of BPSG films deposited on product wafers. *In-situ* monitoring of BPSG film production requires that sufficiently high signal-to-noise ratio spectra be collected on a time scale fast relative to the film deposition

process. We show that rapid collection of IR emission spectral data is possible, and we evaluate factors that influence the analysis precision.

Experimental

Two 21-sample calibration sets of BPSG thin films were produced at National Semiconductor Corp. (Santa Clara, CA) following a three-factor orthogonal experimental design with a randomized run order. The calibration sample properties spanned the following ranges: 2.8 to 5.2 wt.% for boron, 3.2 to 5.3 wt.% for phosphorus, and 0.76 to 0.92 μm for film thickness. The first set of films was deposited on 150-mm diameter product wafers with an underlying feature size of 0.65 μm . The majority of the product wafer surface contains logic device structures, but two regions on the wafers contain test devices. A second set of wafers (a sister set) of films was deposited on bare, 150-mm diameter monitor wafers using processing conditions identical to each of the individual product wafer films. All BPSG films were deposited at 430 $^{\circ}\text{C}$ using a tetraethoxysilane plasma-enhanced CVD process. Each of the wafers had been previously coated with a 0.1 μm layer of thermal oxide. After deposition, each BPSG sample was annealed at 800 $^{\circ}\text{C}$ in steam for 10 min followed by 20 min at 900 $^{\circ}\text{C}$ in nitrogen. The oxidative annealing removes stresses from the film and stabilizes the film against attack by moisture. Thus, the IR spectra of the annealed films are quite stable over the time frame of the experimental study.

The film thickness of each sample deposited on a monitor wafer was measured with a Nanospec film thickness analyzer. The monitor wafers were sent to Balazs Analytical Laboratories (Sunnyvale, CA) for quantitative determination of B and P content. The reference properties used for the product wafers were based on the determinations on the appropriate sister monitor wafer.

The product wafers were cleaved into pieces including a 32×32 mm square from the center of the wafer containing product structures (as opposed to the test structures) that were used in the emission studies. All the infrared emission spectral data were collected using a Nicolet Model 800 Fourier transform infrared (FTIR) spectrometer. The spectrometer's internal source was turned off, and a germanium-coated KBr beam splitter and a liquid-nitrogen-cooled (77 K) mercury-cadmium-telluride (MCT) detector were used for all emission measurements. The emission apparatus, the temperature controller, the data collection protocol, and the data processing are identical to those described in a prior communication.¹⁶

Infrared emission spectra of the 21 BPSG calibration samples were collected at six temperatures: 300, 320, 340, 360, 380, and 400 °C using a randomized run order. Spectra were also collected from a graphite flat at the same temperature as the corresponding sample spectrum. These graphite single-beam spectra serve as the "blackbody" reference. All samples and the blackbody were measured in the same apparatus in a nitrogen environment, and all infrared emission spectra were collected from the center of the sample or graphite flat. Interferograms that were the result of 6 and 128 signal-averaged scans were collected and stored. Each interferogram was transformed at 4 cm^{-1} , 8 cm^{-1} , 16 cm^{-1} , and 32 cm^{-1} resolution to evaluate the effect of resolution on prediction precision. Single-beam spectra of the BPSG samples at a given temperature were ratioed to the blackbody reference spectrum at the same temperature to yield infrared emittance spectra, which were used for data processing except where indicated.

Data analysis was carried out using the partial least squares (PLS) algorithm which is a part an Array Basic software package written at Sandia National Laboratories and operating within the GRAMS (Galactic Industries, Corp.) environment. Prior to the PLS analysis, the

spectral data were smoothed using a 15 point Savitsky-Golay routine. All calibrations were carried out using cross-validation. In the temperature determination using the single-beam spectral data, one spectrum was removed at a time. In the calibration using the emittance spectra measured from the 21 product wafer calibration set, a 6-out (a given BPSG sample at each of the six temperatures) cross-validation was employed.

Repeatability and reproducibility experiments were performed to identify the relative importance of the potential noise sources on the PLS prediction abilities for composition, film thickness, and sample temperature. Repeatability refers to repeated spectral measurements taken rapidly from a single sample held at constant temperature with no changes in the sample position or spectrometer conditions. Reproducibility experiments involve the collection of emittance data from a single sample over long times where each emittance spectrum is taken following the full procedure used when measuring multiple samples.

In the repeatability experiments, one of the product wafers was positioned in the IR emission apparatus, the apparatus heated to 360 °C, and 15 repeat data collects consisting of 6 co-added scans were collected over a period of one hour. Each single-beam was converted to emittance by ratioing to a "blackbody single-beam spectrum" separately collected from a graphite flat also maintained at 360 °C. The film properties of the test wafer were then predicted using a PLS calibration models built using the emittance data collected from the other 20 wafers in the calibration set across the 300 - 400 °C temperature range. By saving interferograms, the standard deviations of the 15 repeat predictions for each property could be determined as a function of resolution.

The reproducibility measurements of the same sample were also obtained at a constant temperature of 360 °C. In these experiments, 10 reproducibility emittance spectra were taken over a 16-hr. period since the time between data collects was 90 - 120 min.

Results and Discussion

The experimental results obtained when studying the IR emission spectra of monitor wafers indicated that a major source of error in the experiment was due to sample temperature uncertainties.¹⁶ The temperature variations were ascribed to lack of reproducible contact between the sample and the heater and/or the sample and the thermocouple when placing a sample in the emission apparatus. The temperature variations are potentially a significant source of error in the measurement of film properties on product wafers. The effects of temperature errors on the IR emission experiment were assessed by carrying out the experiments described below.

The first experiment consisted of collecting emission spectra from a single sample at 16 temperatures across the temperature range of 250 to 400 °C. The order in which the temperatures were run was randomized, but the sample was not moved between data collects (eliminating any variability in the sample-to-thermocouple or sample-to-heater contacts). The single-beam spectra collected in this experiment are shown in Figure 1. As expected the overall intensities of the spectra increase with temperature. The broad and overlapped spectral features due to the B-O and P=O stretching vibrations can be seen in the spectral region between 1250 and 1425 cm^{-1} . The derivative shaped feature at about 1075 cm^{-1} is due to the strong Si-O stretching vibration.¹⁶

A temperature calibration based on the data in Figure 1 could be carried out in a univariate fashion, i.e. the intensity at a selected frequency could be plotted against the

temperature. However, a multivariate calibration will signal average information across all the spectral data to effectively minimize the effect of random spectral noise at any specific frequency. The cross-validated PLS calibration based on the data covering the spectral region from 550 to 1600 cm^{-1} is shown in Figure 2. The cross-validated standard error of prediction (CVSEP) is 1.3 °C with a squared correlation coefficient (R^2) of 0.9993. In this experiment, noise associated with sample insertion has been eliminated. Thus, the CVSEP is affected by spectrometer noise, spectrometer drift, imprecision in the temperature controller, and calibration model error. Prior experimental results¹⁶ indicated that the temperature controller only maintained sample temperatures to a range of ± 1 °C over periods of time of two minutes and longer. Therefore, at least a portion of the imprecision in these repeatability experiments is due to the ability of the controller to maintain the temperature precisely.

Single-beam IR emission data could also be used to develop a calibration model for the other film parameters of interest: boron content, phosphorous content, and film thickness. However, all models based on single-beam spectra would be expected to be difficult to maintain. Long-term spectrometer drift would likely limit the utility of the calibration model that is not referenced to a background signal. Calibration transfer would also be expected to be very difficult with single-beam spectra due to small differences in spectrometer responses, e.g. differences in beamsplitter efficiency or detector response that are present even in spectrometers of the same model. Many of these differences in spectrometers as well as spectrometer drift can be eliminated to a first approximation by using spectra ratioed to an emission standard. In this case, the single-beam IR emission data can be ratioed to the single-beam emission spectrum of a blackbody reference at the same temperature to generate an emittance spectrum.

Figure 3 contains the emittance spectra collected from wafer 1 at the six temperatures used in experiment. Ratioing the single-beam emission spectrum collected from a sample to the single-beam emission spectrum collected from a reference sample, in this case a graphite flat, at the same temperature eliminates most of the monotonic increase in emission intensity with temperature that was present in the single-beam data. Figure 4 contains the emittance spectra measured for five different wafers at 360 °C. The five wafers in Figure 4 represent the extremes, or nearly so, in the BPSG film properties. Figure 4 demonstrates that there are significant differences in the spectra in the region near 1300 cm⁻¹, the region that contains the strongest B and P features. There are also significant spectral differences in the region between 1000 and 850 cm⁻¹ that correlate with film thickness.

Cross-validated PLS calibrations were carried out on the emittance spectra collected from the set of 21 product wafers. The calibration results are presented in Table I as a function of the signal averaging and spectral resolution for the determination of boron content, phosphorous content, film thickness, and temperature. Plots of the PLS cross-validated calibrations for boron content and sample temperature are shown in Figures 5 and 6, respectively. The calibration plots for phosphorous content ($R^2 = 0.94$) and film thickness ($R^2 = 0.98$) are similar in appearance to the boron calibration plot. The results obtained using 4 cm⁻¹ resolution and 128 signal averaged scans can be directly compared to the results obtained from a set of similarly designed BPSG films deposited on monitor wafers.¹⁶ In the monitor wafer experiment, the estimated cross-validated SEPs were 0.09 wt.% for boron content, 0.08 wt.% for phosphorous content, 36 Å for film thickness and 1.9 °C for temperature. As pointed out in the previous communication,¹⁶ these CVSEPs were limited by the reference determinations for boron and phosphorous content, the film thickness variation across a wafer in the case of film thickness, and the approximate ± 1

°C temperature range of the emission apparatus over the time required to collect 128 scans for the temperature determination. The only significant difference between the CVSEPs obtained from the monitor wafers and from the product wafers is for phosphorous. In the monitor wafer experiment, the reference determinations for boron content, phosphorous content, and film thickness were carried out on the identical wafers subjected to the IR emission measurement. In the product wafer experiment, the reference measurements were made on a set of sister wafers, a fact that adds an additional potential source of error to the reference values.

There is no significant degradation in the CVSEPs in Table I when the amount of signal averaging is decreased from 128 scans to 6 scans. Also, there is no degradation in the CVSEPs when the resolution is decreased from 4 cm^{-1} to 32 cm^{-1} . Collection of 6 scans at 32 cm^{-1} requires only about 2 s using the Nicolet model 800 FTIR spectrometer, a considerable speed increase when compared to the 2 min required to collect 128 scans at 4 cm^{-1} .

In order to identify the sources of noise limiting all property predictions in the IR emittance experiments, repeatability and reproducibility experiments were performed for a single product wafer at $360\text{ }^{\circ}\text{C}$ as discussed in the experimental section. The repeatability experiments included 15 emittance spectra collected rapidly without moving the sample. The PLS calibration models for each property were used to predict composition and film thickness and temperature for each of the 15 spectra. The standard deviations of the 15 repeats for the property predictions are shown in Table II as a function of spectral resolution. Reducing spectral resolution from 4 cm^{-1} to 32 cm^{-1} has no significant effect on the precision of the determinations. The precisions achieved in the repeat experiment for the boron content and phosphorous content are more than an order of magnitude better than the calibration CVSEPs for these determinations. The precision of the repeat thickness determination is about a factor of four better than the

corresponding calibration CVSEP, while the precision of the temperature determination is about a factor of three better than the corresponding calibration CVSEP. The time required for the collection of six scans at 4 cm^{-1} resolution is about six seconds, so temperature fluctuations during the data collection should be minimal. Spectrometer drift over the one-hour experiment is not thought to be significant in these experiments. Errors due to sample movement, contact with the heater, or the thermocouple contact should not affect this repeatability experiment since the sample is not moved between repeat spectra. Therefore, repeatability measurements should be a good indicator of the best precision that can be achieved with a given amount of signal averaging. These experiments demonstrate that the PLS calibration precisions are not limited by spectral noise or short-term system drift. The loss of precision due to long-term drift, the movement of the thermocouple and sample in the experiment, and reheating the sample was addressed by performing a reproducibility experiment.

The reproducibility experiments have the added variability between spectra that the emission apparatus was allowed to cool to room temperature, the sample was removed from the apparatus, the sample replaced in the apparatus, and the purge/heating cycle was reinitiated. The standard deviations obtained for the film property predictions from ten repeats of this cycle are shown in Table III as a function of spectral resolution. For boron and phosphorous determinations, the prediction precisions of the reproducibility experiments are about an order of magnitude worse than achieved in the repeatability experiments. The thickness standard deviation is about a factor of 2 worse than achieved in the repeatability experiment, and the temperature standard deviation is more than a factor of 4 worse. Clearly there are additional noise factors influencing the prediction results in the reproducibility experiments when compared to the repeatability experiments.

The time between data collects in the reproducibility experiment was 90 - 120 min. Since the entire reproducibility experiment took more than sixteen hours, spectrometer drift is expected to be a factor in the prediction precisions. Attempts were made to position the sample so that the same spot was sampled by the spectrometer each time. Positioning errors could be significant if the film was nonuniform or if the structure below the film affected the spectral data. BPSG films are not perfectly uniform, but near the center of the wafer the nonuniformity is slight and the differences in film properties due to sampling positional errors would be expected to be trivial.

The comparison of the repeatability and reproducibility experiments demonstrate that long-term drift, reinserting and reheating the sample, and making new thermal contacts have a detrimental effect on the prediction precision. One or more of these effects, therefore, dominates over the detrimental effects of short-term spectrometer noise.

If we compare the PLS CVSEP results in Table I with the reproducibility results in Table III, we find that the B and P reproducibility precision is lower than the corresponding CVSEP's. The reproducibility precision for film thickness is comparable to the CVSEP for film thickness and is approximately a factor of 2 worse for temperature. These results suggest that model error or reference error is dominant for B and P concentration predictions. Reproducibility factors of drift, sample insertion and repositioning, sample uniformity, etc. limit the precision for film thickness. The fact that the reproducibility precision for temperature is worse than in the calibration could be attributed to model instability over the months between the calibration and reproducibility experiments.

When comparing the calibration results achieved using the films deposited on the monitor wafers with the results achieved using the films deposited on the product wafers, it appears that the position of the beam on a given product structure underlying the film has little effect on the

information content of the spectral data.¹⁶ Indeed, the original calibration would include positioning errors, because each film sample had to be positioned in the IR emission apparatus for each sampling event. Thus, we conclude that changes in a given underlying structure due to positioning errors do not significantly affect the spectral data. This conclusion is further corroborated by our previous IR reflection spectroscopy experiments on product wafers.¹² In that reflection experiment, deliberate positional errors much larger than expected in this IR emission experiment showed little effect on the precision of property determinations as long as the IR beam was sampling identical product structures rather than test structures.

Conclusions

Dielectric thin films, such as BPSG films, used in the microelectronics industry are generally produced using chemical vapor deposition. Infrared emission spectroscopy is an ideal candidate for *in-situ* monitoring of the chemical vapor deposition process used to produce these dielectric films. A comparison of calibration results with repeatability and reproducibility experiments have allowed us to identify potential factors limiting prediction ability. The ideal process monitor would be capable of measuring film temperature, film thickness, and dopant concentrations on actual products. Provisions would need to be made in the CVD reactor to assure that the IR window was free of significant contamination. We have shown that IR emission can be used to make simultaneous and precise determinations of all of these critical parameters on a time scale that is fast relative to the CVD process time. The requirements on the spectrometer are not severe. A system capable of operating at 32 cm^{-1} resolution with little or no signal averaging has been demonstrated to produce spectral data that can achieve adequate prediction ability for *in situ* monitoring of BPSG films on product wafers. The ability to monitor film production on all product wafers, as opposed to periodically running a monitor wafer

through the CVD apparatus, is a significant advantage of IR emission for *in-situ* process monitoring. The precisions of the IR emission determinations on product wafers have been shown to be equivalent to the precision of the methods applied to monitor wafers. Avoiding the expense associated with running monitor wafers through the CVD process on a periodic basis combined with the ability to monitor the film quality on every product wafer that passes through the process are significant advantages of the IR emission monitoring method. Ultimately, the information provided by the IR emission monitor could be used in a real-time feedback loop to control the film deposition *in-situ*.

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Table I. Cross-validated calibration results as a function of resolution and amount of signal averaging obtained from IR emittance spectra from the 21 BPSG samples on product wafers.

Resolution (cm ⁻¹)	4		8		16		32	
No. Scans Averaged	128	6	128	6	128	6	128	6
CVSEP B Content (wt.%)	0.09	0.10	0.10	0.10	0.09	0.10	0.09	0.10
CVSEP P Content (wt.%)	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.15
CVSEP Thickness (Å)	50	57	43	43	41	52	50	53
CVSEP Temperature (°C)	2.4	2.5	2.4	2.4	2.8	2.6	2.8	2.8

Table II. Standard deviations (SD) as a function of resolution obtained from PLS property predictions during the emittance repeatability experiments using six averaged scans.

Resolution (cm ⁻¹)	4	8	16	32
SD B Content (wt.%)	0.008	0.005	0.004	0.006
SD P Content (wt.%)	0.005	0.006	0.005	0.005
SD Thickness (Å)	16	14	14	15
SD Temperature (°C)	0.9	0.8	0.9	0.8

Table III. Standard deviations (SD) as a function of resolution obtained from PLS property predictions during the emittance reproducibility experiment using six averaged scans.

Resolution (cm ⁻¹)	4	8	16	32
SD B Content (wt.%)	0.061	0.047	0.034	0.049
SD P Content (wt.%)	0.046	0.070	0.041	0.041
SD Thickness (Å)	35	57	50	38
SD Temperature (°C)	4.2	4.9	5.1	3.2

Figure Captions

Figure 1 Single-beam IR emission spectra collected from a BPSG thin film on a product wafer at 10 °C increments over the temperature range of 250 °C to 400 °C.

Figure 2. Cross-validated calibration results for temperature based on the single-beam IR emission data collected from the BPSG film deposited on a patterned wafer.
 $CVSEP = 1.3 \text{ } ^\circ\text{C}$, $R^2 = 0.99$.

Figure 3 Emittance spectra collected from one of the BPSG samples at six temperatures from 300 to 400 °C.

Figure 4 Emittance spectra collected from five BPSG thin-film samples at 360 °C. These five samples represent the extremes, or nearly so, in the reference film properties.

	<u>B (wt %)</u>	<u>P (wt %)</u>	<u>Thickness (μm)</u>
-----	5.32	2.89	0.7589
- - - - -	4.13	2.85	0.7658
_____	4.77	4.66	0.7849
- - - - - -	3.57	4.16	0.8021
.....	5.09	3.73	0.6574

Figure 5 Cross-validated (6-out) PLS predicted B content vs. reference B content based on the emittance spectra collected over the temperature range of 300 to 400 °C from the 21 BPSG samples deposited on product wafers ($R^2 = 0.98$). The solid line is the line of identity.

Figure 6 Cross-validated (6-out) PLS predicted temperature vs. reference temperature based on the emittance spectra collected over the temperature range of 300 to 400 °C from the 21 BPSG samples deposited on product wafers ($R^2 = 0.99$). The solid line is the line of identity.

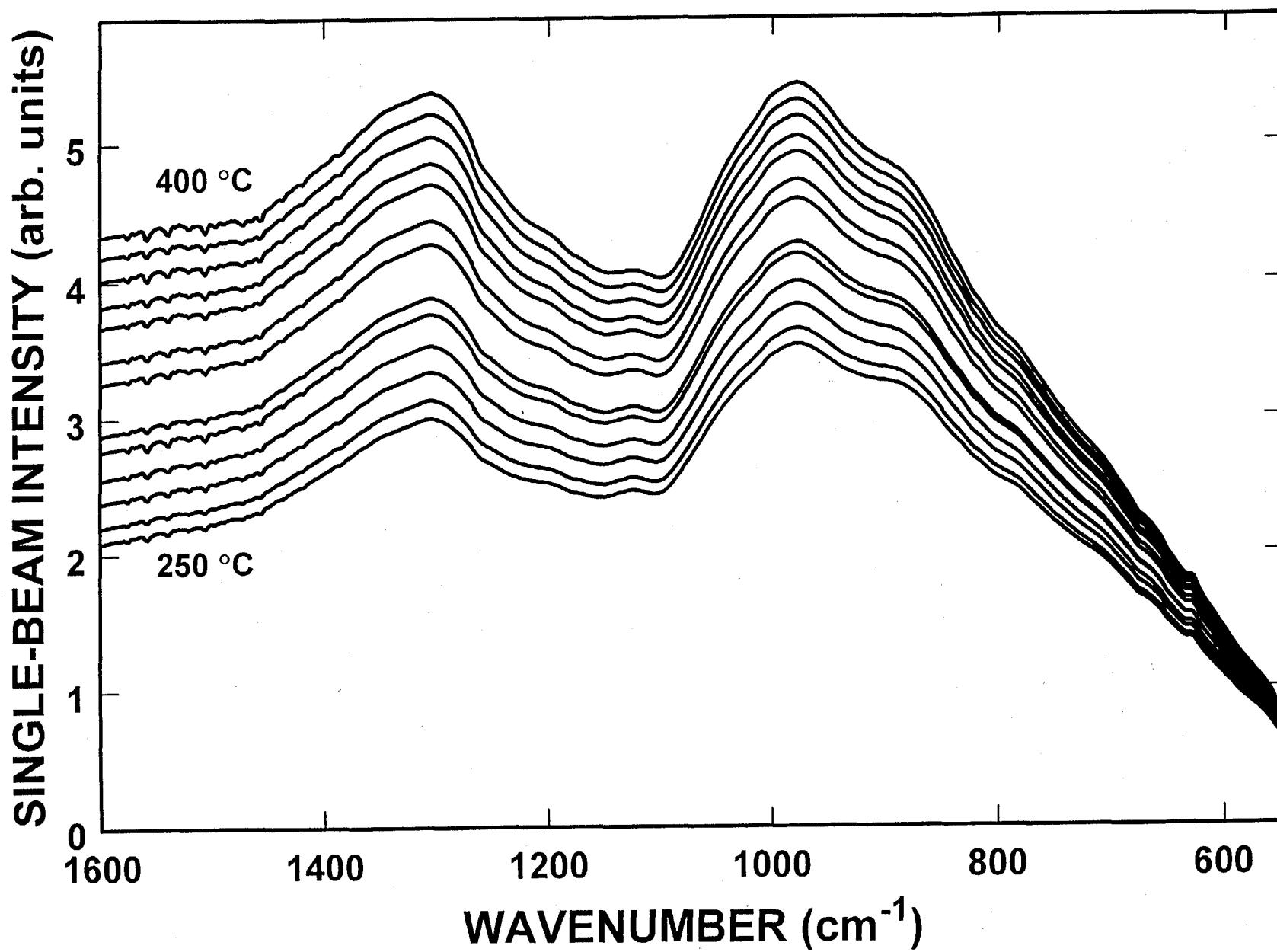


Figure 1

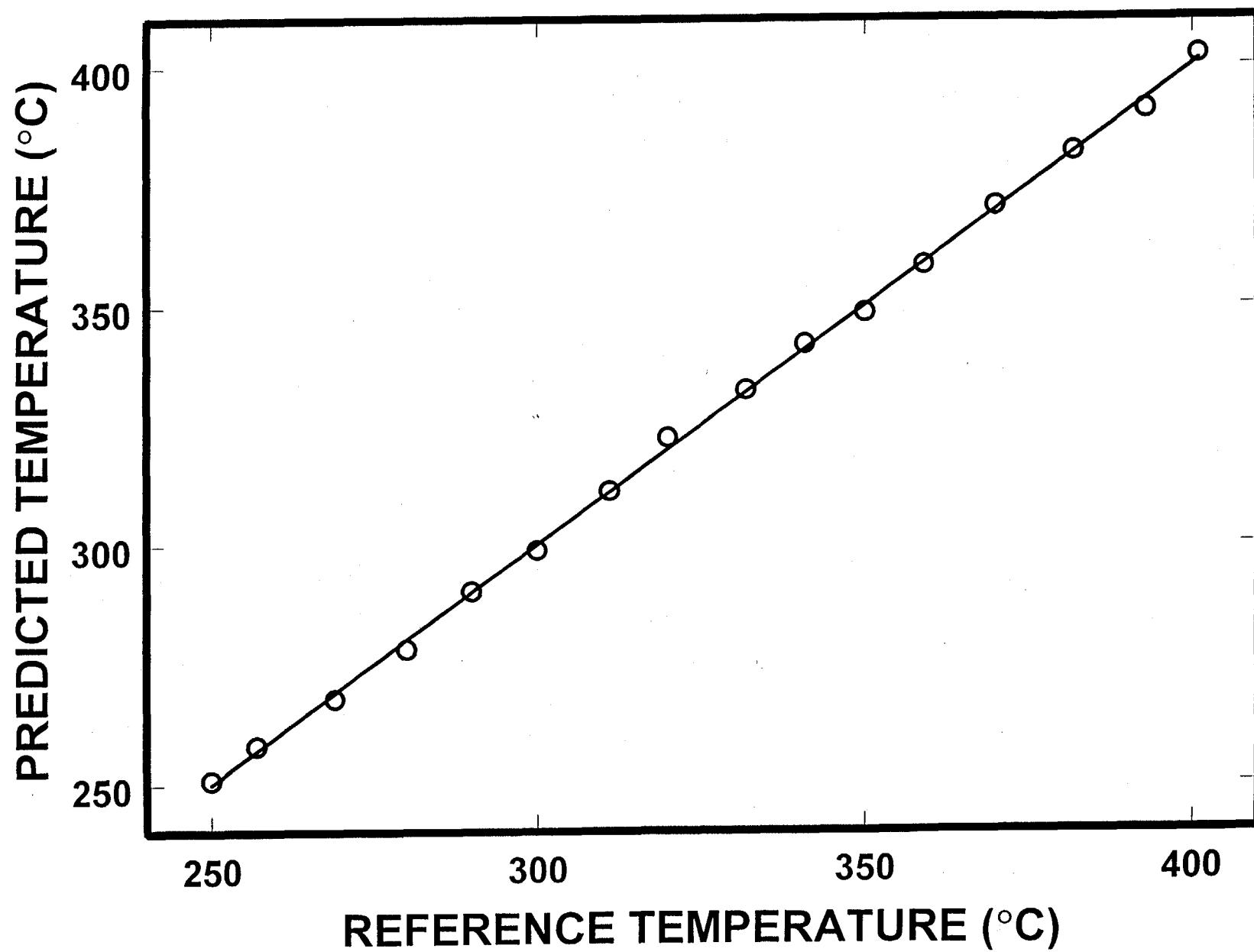


Figure 2

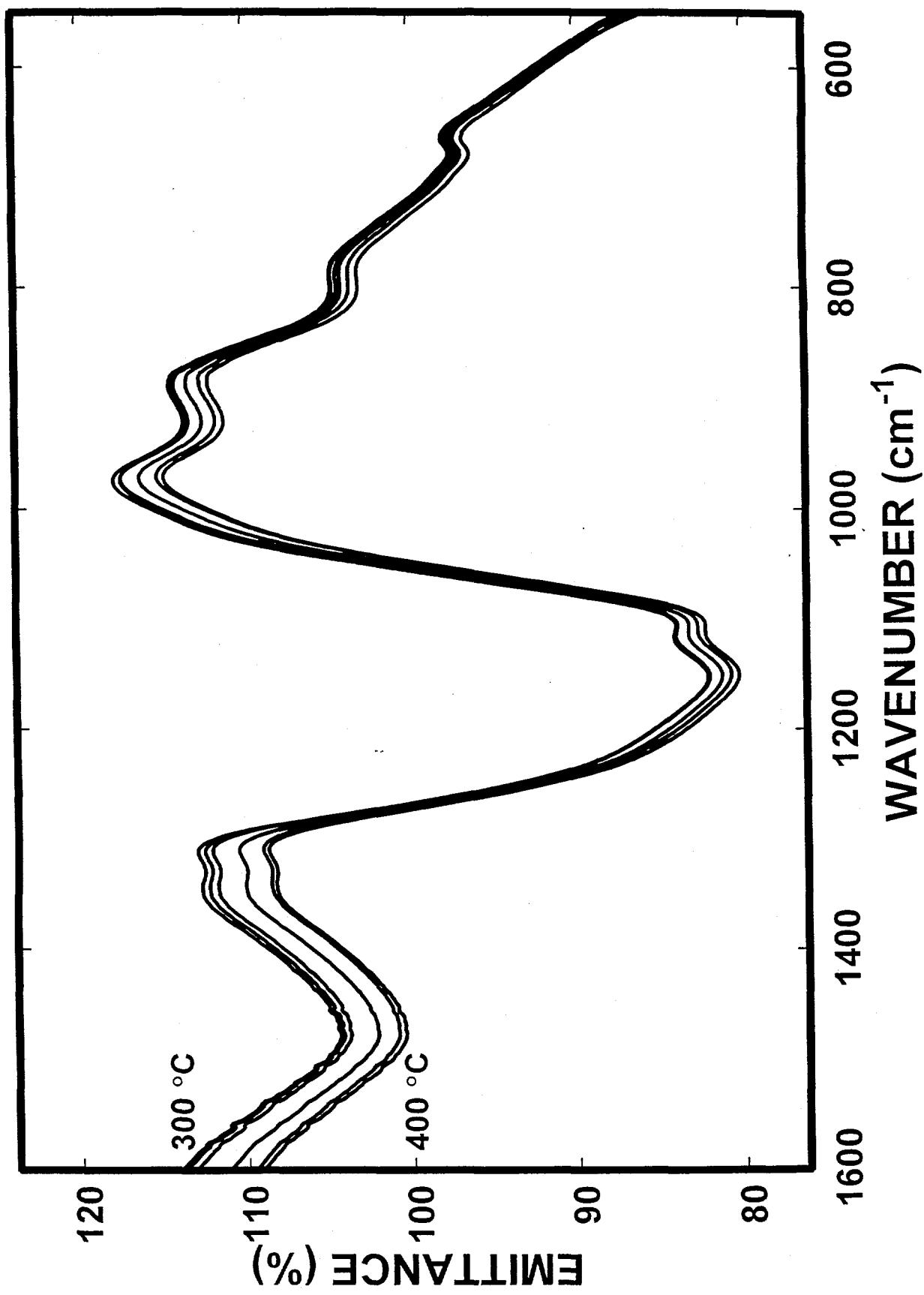


Figure 3

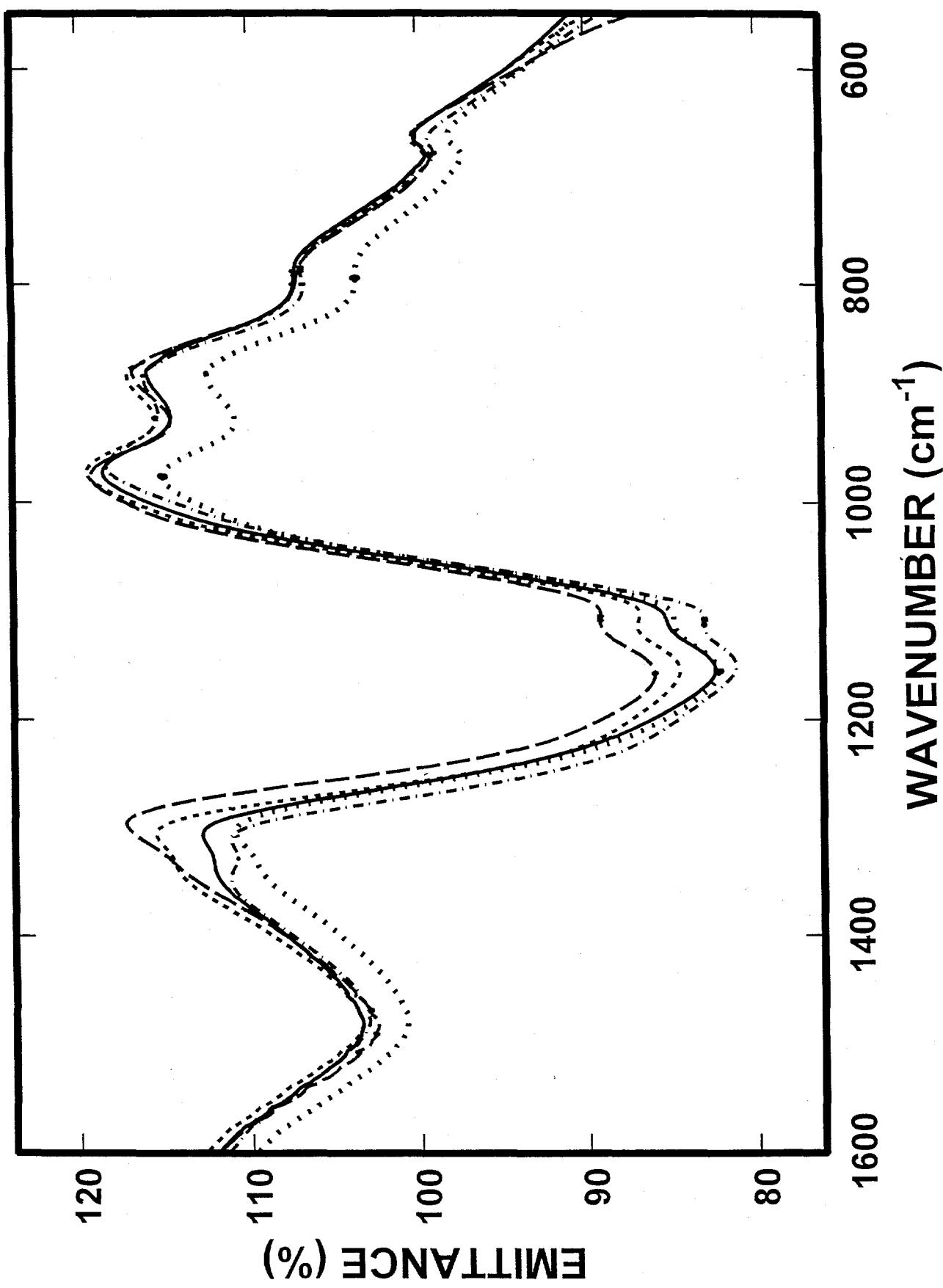
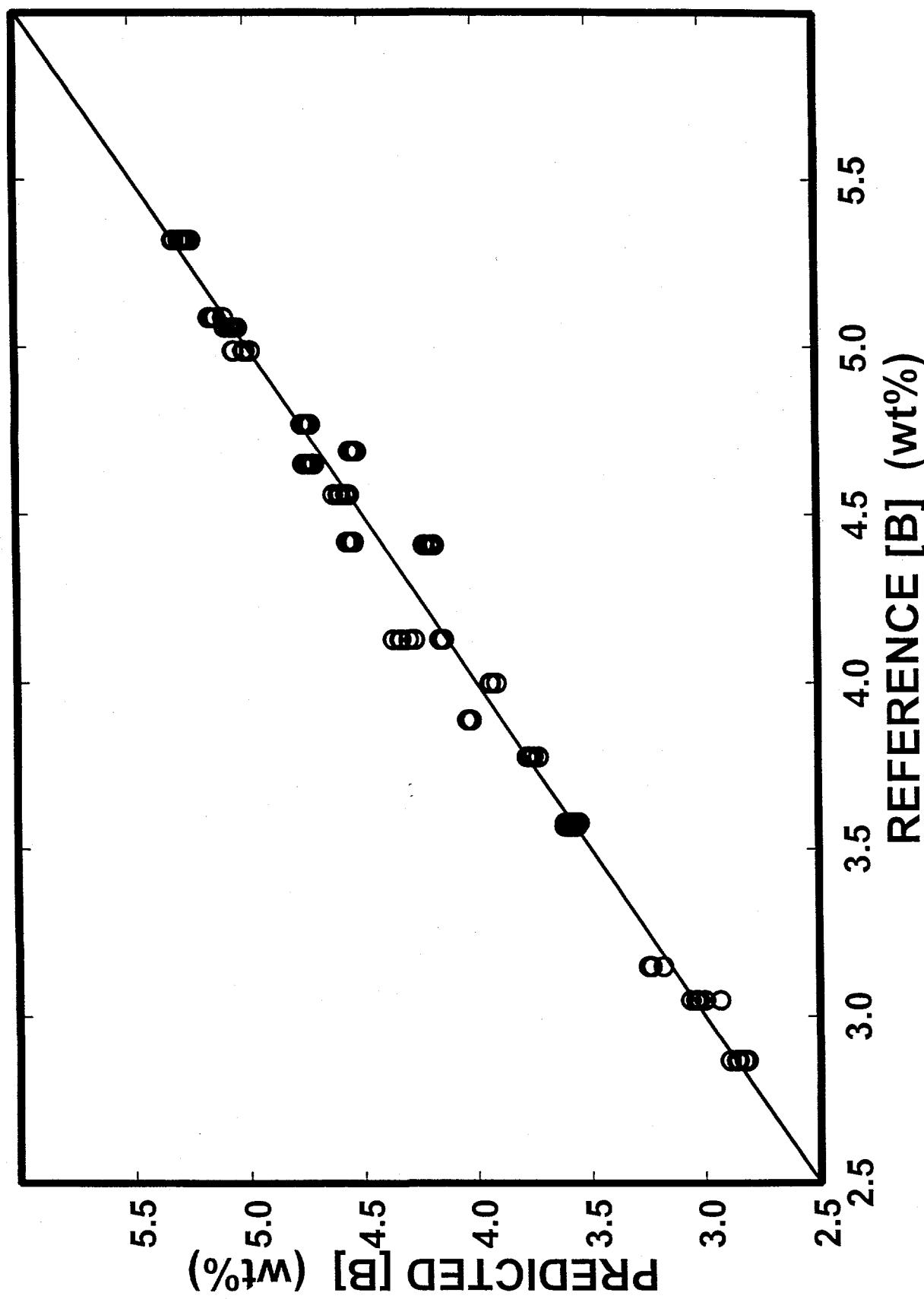


Figure 4



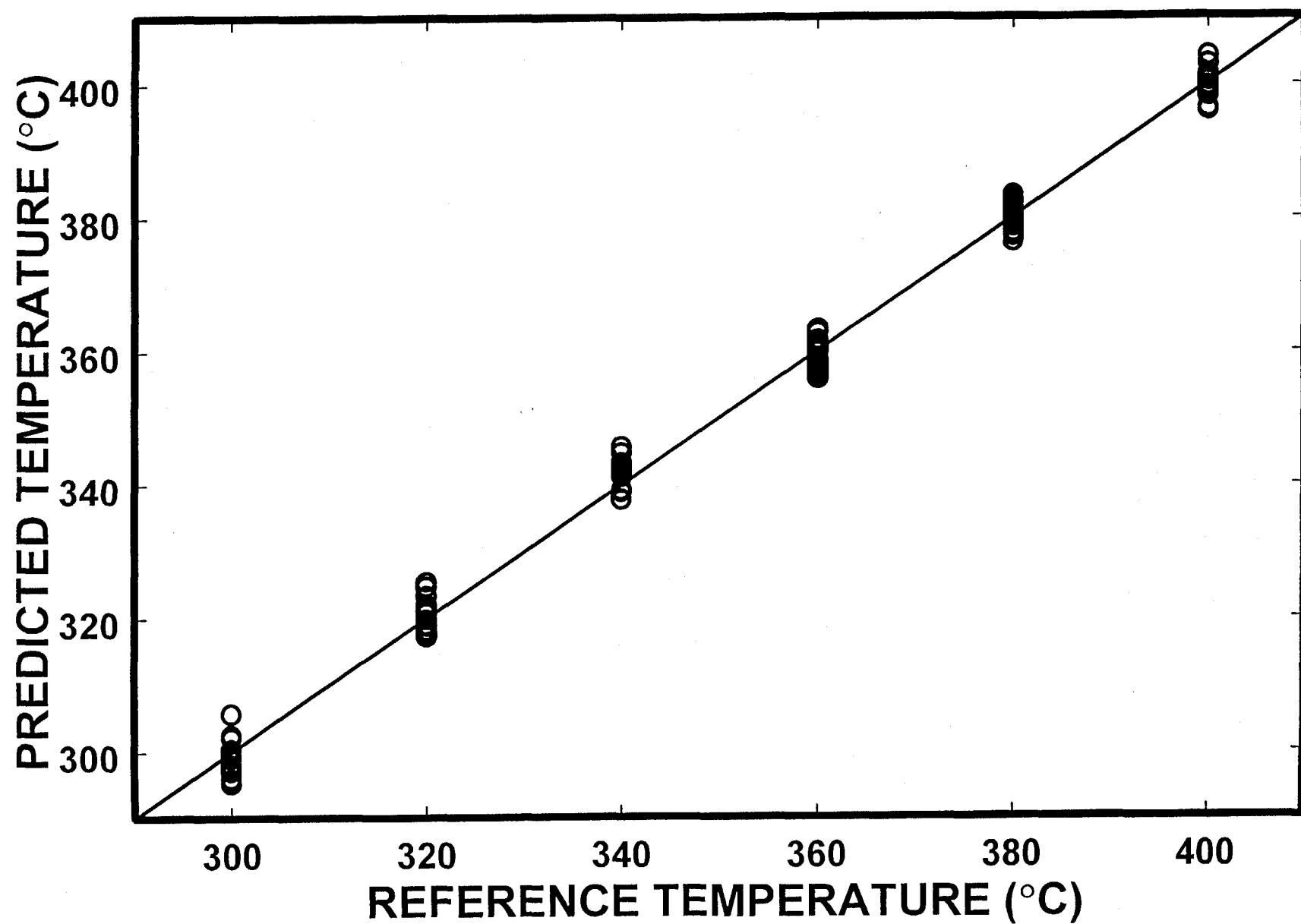


Figure 6