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Low Severity Coal Liquefaction Promoted by Cyclic Olefins

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Quarterly Report
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DISCLAIMER

Acknowledgments

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FTIR at Combined Elevated Temperatures and Pressures

Introduction

The goal of this research is to develop a methodology for analyzing the reactivity of cyclic olefins in situ in a high temperature and high pressure infrared cell. Cyclic olefins, such as 1,4,5,8-tetrahydronaphthalene (isotetralin) and 1,4,5,8,9,10-hexahydroanthracene (HHA), are highly reactive donor compounds that readily donate their hydrogen to coal and model acceptors when heated to temperatures of 200 °C and above. These donors are active donors in the low severity liquefaction of coal at 350 °C as shown in the research performed in this project. The infrared studies are being performed in a high temperature infrared cell that was obtained from AABSPEC. Modifications to that cell have been made and have been reported in previous progress reports.

The useful temperature range of the high temperature infrared cell has been extended to 230 °C through the use of a high-boiling perfluorocarbon solvent. The solvent used was an Air Products and Chemicals Company proprietary product, trade named Multifluor APF-240. Solubilities of aromatics and cyclic olefins were quite low in APF-240, usually less than 0.1 wt% at room temperature, but were found to be a strong function of temperature, increasing markedly when the mixture were heated to 65 °C. High temperature infrared analyses have been performed using isotetralin, tetralin, naphthalene, 1,4-dihydronaphthalene and 1,2-dihydronaphthalene. Stability studies have shown that naphthalene was quite stable at temperatures up to 230 °C, as were tetralin, decalin and 1,4-dihydronaphthalene. High temperature FTIR analysis of isotetralin and 1,2-dihydronaphthalene reacted at elevated temperatures forming tetralin and 1,4-dihydronaphthalene, respectively. High temperature analyses were performed under 500 psi of N_2 gas. Although several

successful analyses were performed, severe problems occurred that resulted in loss of the FTIR signal. Since the spectrometer is being successfully used for another project, the problems encounter must be caused by the Aabspec cell. The difficulties are described in the report.

Current Work

During this quarter, stability studies were conducted at both ambient pressure and under a 500 psig nitrogen blanket. The first stability test performed this quarter was a repetition of an earlier stability test. The work with 1,4-dihydronaphthalene was repeated with the hope of reducing the noise level. The details of the procedure for the stability test have been reported previously. On July 24th, the backgrounds were collected at the various temperatures using APF-240 as solvent. When the cell was filled with 1,4-dihydronaphthalene solution and placed in the spectrometer, the interferometer signal dropped to 0.13 volts. An interferometer signal of six volts or more is required for collecting good FTIR data. The study was postponed and the cell placed in an oven at 65 °C overnight. The following morning the signal strength had returned to 6 volts so the stability test was conducted using the backgrounds taken the previous day. Figures 1 through 5 present the initial scans at 65, 100 150, 200 and 230 °C, respectively. The scans became progressively noisier as the temperature was increased. Also, the baseline level dropped as the temperature was increased. The baseline began to have a slope at 230 °C and the noise had reached the level that it began to significantly obscure the data. The test was aborted and the cell placed in the oven at 65 °C overnight. It was decided at this point to determine whether the backgrounds could be suspect. The cell temperature was raised directly to 230 °C and the sample scanned against a background taken on June 13th which had been stored on the computer hard disk. This resulted in a significant

absorbances of the backgrounds taken on July 24th were low compared to those taken on June 13th under identical conditions. This was not noticed at the time because the display autoscales to fill the screen. In the future, the scale of the absorbance axis on background scans will be monitored to ensure that the background absorbances are sufficiently strong. It was suspected that the low interferometer signal contributed to the weak background absorbances and ultimately to the noisy scans. The focus now shifted to determining the cause of the deterioration in the interferometer signal.

Since other work which was being concurrently performed on the FTIR was not being affected by a loss of signal or increase in the noise level, the Aabspec cell or the way in which the cell was being used became suspect. The first conjecture to be tested was that the heat being generated by the cell in holding the sample at high temperatures was affecting the FTIR system components. To test this, a thermometer was installed in the box near the detector and a thermocouple probe was inserted between the two electronic system control cards. During 19 hours of operation with a sample temperature of 230 °C, the temperature in the box never rose above 32 °C at either port. Since the ambient temperature was 30 to 32 °C, heat accumulation in the interferometer box as a source of the problem was discredited.

A second possibility was that elevated temperatures caused increased background radiation from the cell which contributed to the noise. To test this, neat APF-240 was scanned againt itself as background at both 65 and 230 °C. This procedure should generate a straight line. Figures 7 and 8 present the results of these two scans. Both show straight lines with quite acceptable noise levels so background radiation was ruled out as a source of the noise.

Ambient Pressure 1,4-Dihydronaphthalene Stability Study. Even though the diagnostic work was incomplete, progress still needed to be made. A stability test of 1,4-dihydronaphthalene was repeated because of the presence of noise in the previously obtained spectra. A 0.26 wt% solution of 1,4-dihydronaphthalene in APF-240 was placed in the cell and an ambient pressure stability test was conducted. Figure 9 presents the initial scan at 65 °C. Figures 10 and 11 present the initial and final scans at 230 °C. Figure 12 presents the initial scan at 65 °C with the final scan at 230 °C on the same plot. This confirms the previously published result that some reaction is taking place. Figure 13 presents the previously reported data. Note in Figure 12 that the fine noise which is apparent in Figure 13 has been eliminated, which was the goal of repeating this stability study. This fine noise was eliminated by the realignment necessitated by extending the Aabspec's cell holder to accomodate high pressure operation as reported previously. The noise which plauges operation now shows a much brodaer peak-to-peak distance, as shown in Figure 14. Our electronics support engineer has suggested that this periodic waveform could be due to AC leakage into the system.

Ambient Pressure 1,2-Dihydronaphthalene Stability Study. Encouraged by the promising results of the 1,4-dihydronaphthalene stability study, the ambient pressure 1,2-dihydronaphthalene stability study was repeated, also with the hope of reducing the fine noise. A 0.30 wt% solution of 1,2-dihydronaphthalene in APF-240 was placed in the Aabspec cell and an ambient pressure stability study was conducted. Figures 15 and 16 present the initial scans at 65 and 230 °C, respectively. Figures 17 through 20 present scans at one-half hour intervals at 230 °C. Note the gradual degradation of the absorbances with time. One hour into the 230 °C portion, the machine generated "too many bad scans". Persisitent rescanning resulted in the figures shown. By the second hour, 4 or 5 attempts to rescan still resulted in "too many bad scans". The cover to the interferometer was

removed and the sample scanned. Figure 21 presents this scan. Although the peak heights improved somewhat, the noise level was also higher. The test was aborted at this time. It should be noted that the interferometer signal was at 7.5 volts, so there is something occuring here other than loss of signal.

High Pressure Naphthalene Stability Study. In a previous high pressure stability study of naphthalene, a peak emerged around 2950 cm^{-1} (see Figure 22). Since no such peak was apparent in the ambient pressure stability study, an impurity, perhaps a residue, was suspected. Furthermore, had a reaction taken place, a corresponding decrease in a naphthalene peak would be expected. To test the theory, the cell was flushed with 30 mL of trichloroethane, which is 100 times the sample cavity's volume, and dried with nitrogen. Scans of the clean cell for residual hydrocarbons proved negative. Neat APF-240 was placed in the clean Aabspec cell and a 500 psig nitrogen blanket applied. The routine stability test was conducted in the hope of "baking out" any impurity. In the case of neat solvent, each scan should have resulted in a straight line, any abberations being indicative of the noise level, while any peaks would indicate the presence of an impurity. Figures 23 and 24 present the initial scans at 65 and 230 °C, respectively. The baseline gradually drifted to higher absorbance values. Figure 25 presents the final scan at 230 °C. The noise level increased dramatically and the base line began to slope as the sample was held at elevated temperature. It was postulated that length of time at elevated temperature played some role in data degradation, or that the strain introduced on the cell by the pressurized tubing moved the cell out of alignment over a period of time.

Satisfied that the cell was free of any residual impurities, the high pressure naphthalene stability study which was performed last quarter was now repeated. Figures 26 and 27 present the initial scans at 65 and 230 °C, respectively. Figure 28 presents the final scan at 230 °C. Note the absence of the large

broad peak around 2950 cm^{-1}, which emerged during last quarter's study (see Figure 22). It was thus concluded that some impurity was responsible for the emergence of this peak.

A Tripplite Model LC1800 voltage conditioner was installed with the FTIR in the hope of stabilizing the interferometer signal and reducing the noise, as well as to prevent computer malfunctions which frequently accompany power supply voltage fluctuations. The optical bench, CPU, monitor, service module and plotter were all plugged into the voltage conditioner. This caused the monitor display to waver. Relocating the voltage conditioner farther from the monitor corrected this problem.

High Pressure Decalin Stability Test. A 0.11 wt% solution of cis-decalin in APF-240 was subjected to the routine stability test under a 500 psig nitrogen blanket. Figures 29 and 30 present the initial scans at 65 and 230 °C, respectively. Figure 31 presents the final scan at 230 °C. Note the emergence of a peak at about 3070 cm^{-1}, which becomes quite pronounced by the end of the test. Upon cooling to 65 °C and letting the sample sit over the weekend, this peak disappeared (see Figure 32). It was thus thought to be a temperature effect rather than a reaction product. The reaction product was later saved and this could be confirmed by GC analysis. It could also be a pressure effect, as the ambient pressure decalin stability study does not show the emergence of a peak around 3070 cm^{-1} (see Figure 33). The cell was returned to 230 °C under a 500 psig nitrogen blanket to see if the peak around 3070 cm^{-1} would reappear, but the interferometer signal was lost. The interferometer signal could not be recovered, even at 65 °C. As data acquisition using the Aabspec cell was now quite impossible, the focus shifted to diagnostic work.

Diagnostic Work, Plumbing Modifications, and Cell Alignment. Three different corrections were suggested:

(1) Open the aperture in the extension which was built to rotate the cell 90 degrees to accomodate high pressure usage to the same size as the aperture in the cell holder provided by Nicolet. The small size of the aperture in the extension may have been limiting the amount of radiation passing through the sample cell and reaching the detector.

(2) Allow for movement in the positioning of the cell in the front-to-back direction.

(3) Provide a way to reproducibly anchor the cell to guard against movement caused by thermal expansion and pressure effects. This could be accomplished through the use of a dual axis micrometer adjustor.

The following observations were made:

(1) The cell is sensitive to the way in which it is heated. It has some elastic memory because the signal is recovered after the cell has been in the oven overnight.

(2) The different cell materials (stainless steel body and high pressure tubing, salt windows, Viton O-rings, brass fittings, copper coolant tubing), all have different rates of thermal expansion and coefficients of thermal expansion, which could alter the alignment of the cell upon heating.

Our departmental mechanician completed suggestions #1 and #2 above. An attempt was made to align the cell but a sufficiently strong interferometer signal was not obtained. At this point, modifications to the cell's plumbing were made. The rigid connections for the coolant lines were placing the cell under tension and rendering precision adjustments impossible. With these modifications and a realignment, the signal rebounded. However, within one hour at 65 °C the signal decayed to less than half of its original value. A good deal more work is indicated in this area.

GC Analysis of Naphthalene and Cyclic Olefins. Methods development for the determination of reaction products by GC analysis proceeded during Summer quarter, 1996. This work was performed

on a Varian 3400 Gas Chromatograph using a J&W Scientific 30m x 32mm ID capillary with a DB5 phase of film thickness 0.25 microns.

The first problem encountered with separating naphthalene and cyclic olefins was that naphthalene and 1,4-dihydronaphthalene had the same elution time. Three standard methods for separating peaks with very similar elution times are:

(1) reduce the initial column temperature,

(2) reduce the ramp rate and

(3) increase the initial hold time.

It was decided not to reduce the initial column temperature below 65 °C because of the low solubilities of naphthalene and cyclic olefins in APF-240 below this temperature. Neither reducing the ramp rate from 5 to 3 °C per minute or increasing the initial hold time from zero to ten minutes resolved the two peaks. Neither did using these two effects in combination. A 60 meter column has been procured and it is hoped that it will be able to separate naphthalene from 1,4-dihydronaphthalene.

Internal Standard Methods Development

Because of the low volume of reaction products, typically less than 0.3 mL, the autosampler could not be used for injecting samples onto the GC. The samples had to be injected manually. So that the size of the sample injected could be known precisely, it was decided to develop an internal standard method for this work. One problem encountered was that APF-240 elutes as a broad smear of peaks in the range from about 2 minutes to about 5.25 minutes. This was in the range where typical internal standards like toluene and para-xylene eluted, rendering quantitation using these compounds imposssible. Biphenyl was tried, and although it eluted well after the APF-240, being a solid its solubility in APF-240 was quite low. It was rejected in favor of diphenylmethane, which also elutes well after the APF-240, but being a liquid,

shows a greater solubility in APF-240. Diphenylmethane shows promise for use as an internal standard in this work.

Future Work

The diagnosis of the problem with the Aabspec cell is of paramount importance to the continuation of this work. Our electronics support engineer has suggested a number of experiments which will be tried during Fall Quarter, 1996. It may be necessary to contact Aabspec in connection with this diagnostic work. When this problem is corrected, the nitrogen blanket stability studies will be extended to tetralin, isotetralin, 1,2-dihydronaphthalene and 1,4-dihydronaphthalene. The question of the decalin peak which emerged around 3070 cm^{-1} in the high pressure study remains to be resolved. The stability studies will also be conducted under a 500 psig hydrogen blanket. The existing 30 meter column in the GC will be replaced by a 60 meter column. The internal standard method for determining the concentrations of naphthalene and cyclic olefins will be developed into a routine. In future research, the stability studies will be extended to 350 °C and 500 psig using a perfluoroether obtained from DuPont. The linear region of the absorbance versus concentration curves will be determined. It may be necessary to use the least squares and/or deconvolution software packages in connection with this determination. The kinetics of the reaction of isotetralin at elevated temperatures and pressures will be determined.

FIGURE 1

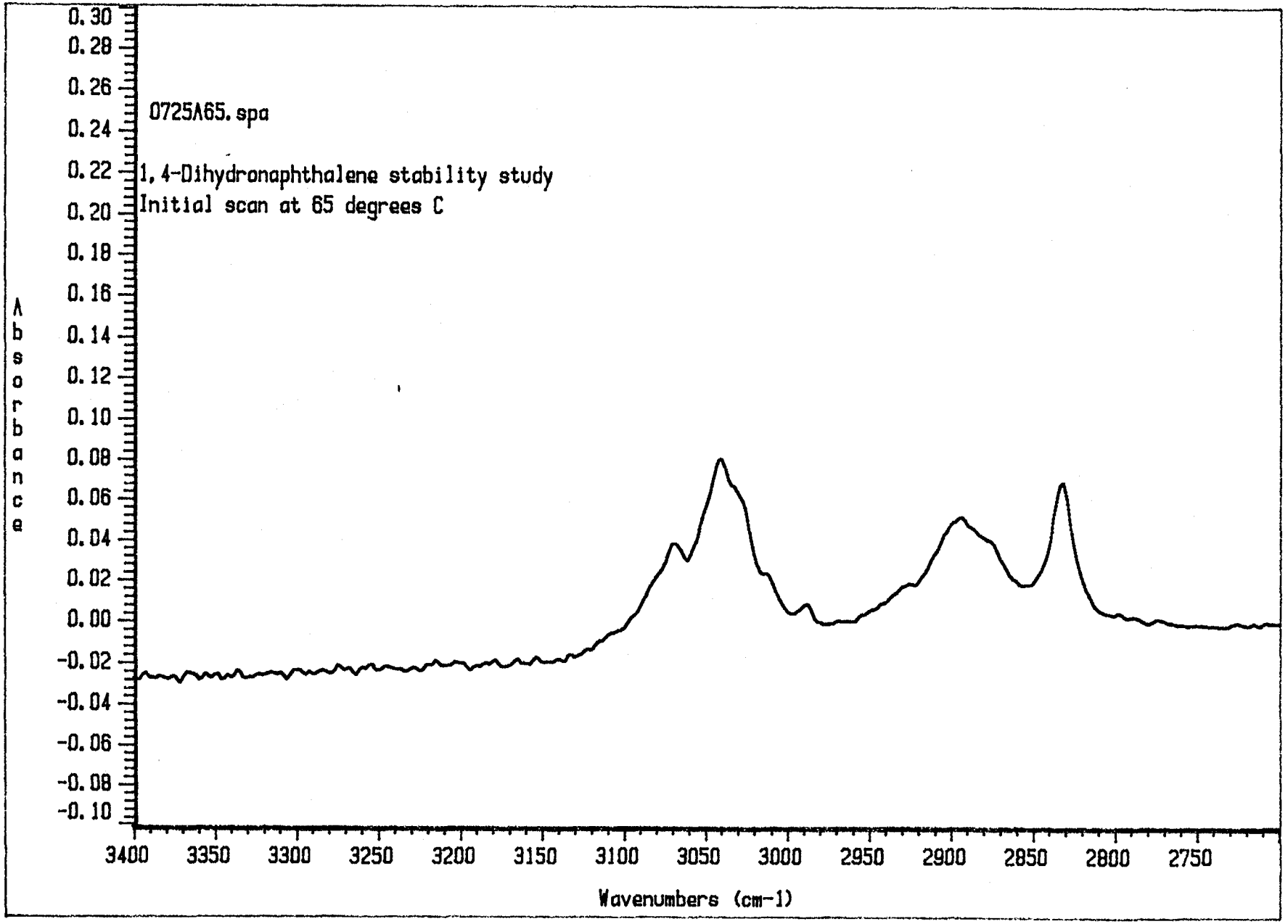

0725A65.spa
1,4-Dihydronaphthalene stability study
Initial scan at 65 degrees C
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 2

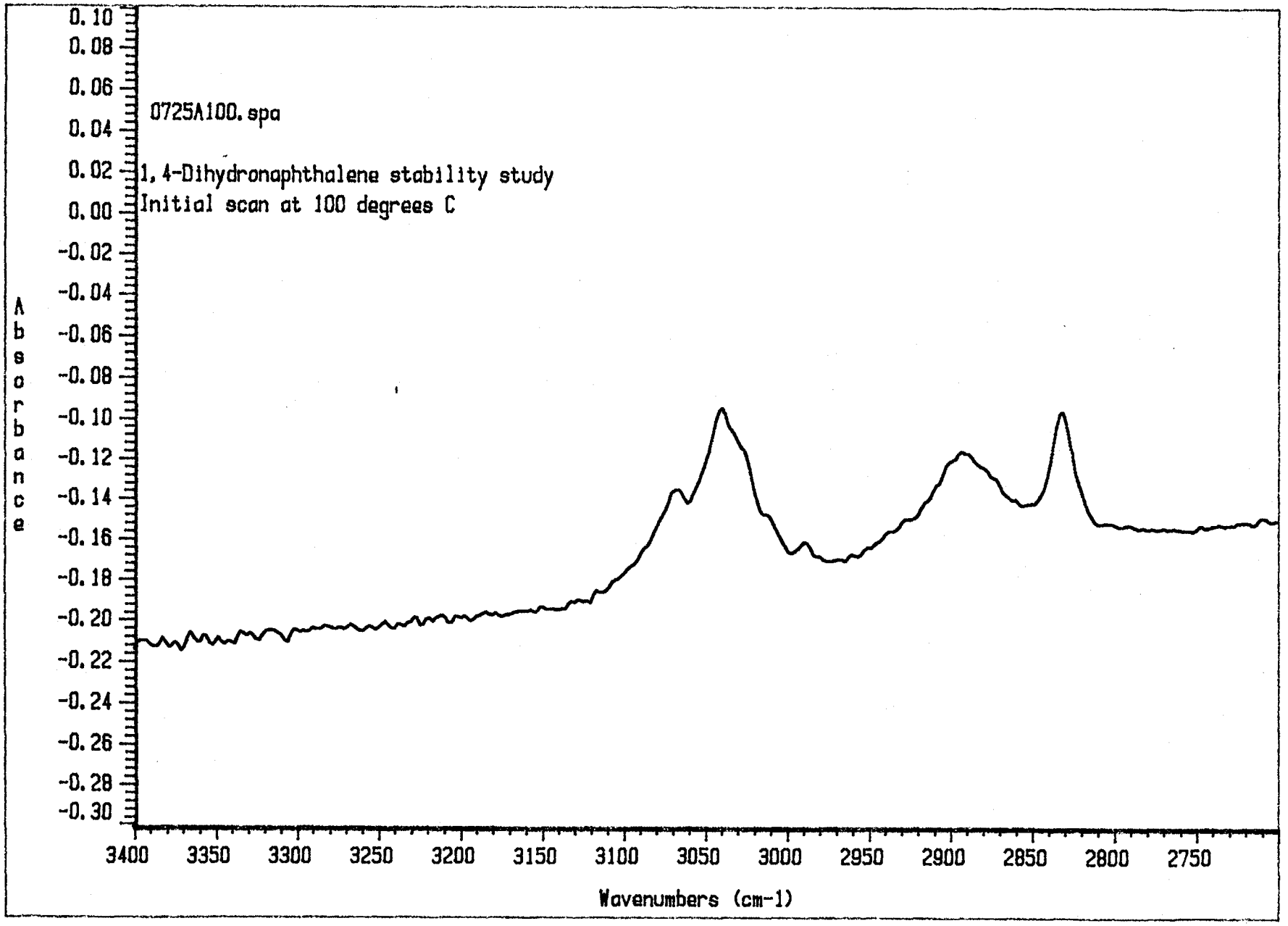

0725A100.spa
1,4-Dihydronaphthalene stability study
Initial scan at 100 degrees C
Absorbance
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
-0.12
-0.14
-0.16
-0.18
-0.20
-0.22
-0.24
-0.26
-0.28
-0.30
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 3

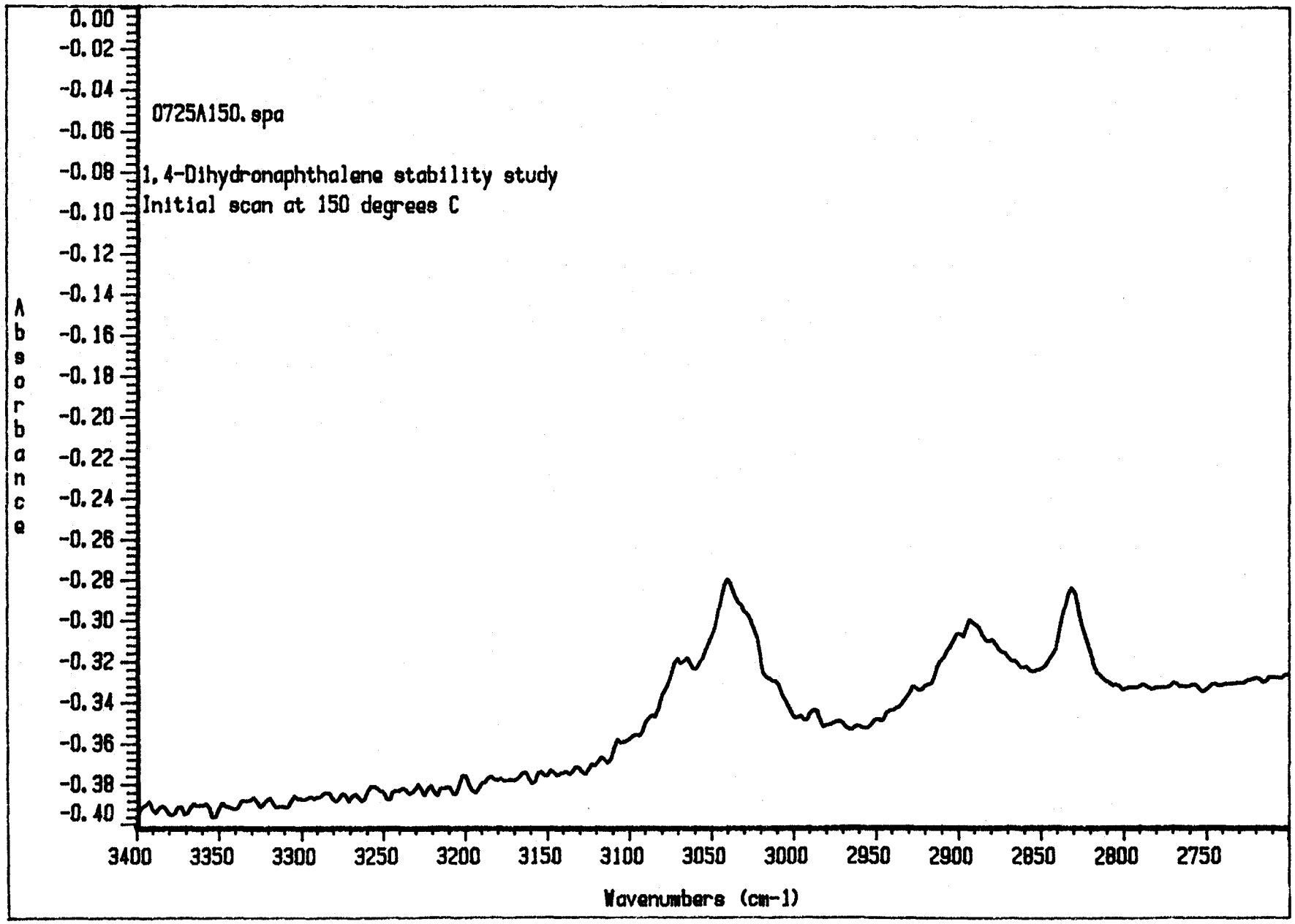

0725A150.spa
1,4-Dihydronaphthalene stability study
Initial scan at 150 degrees C
Absorbance
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
-0.12
-0.14
-0.16
-0.18
-0.20
-0.22
-0.24
-0.26
-0.28
-0.30
-0.32
-0.34
-0.36
-0.38
-0.40
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 4

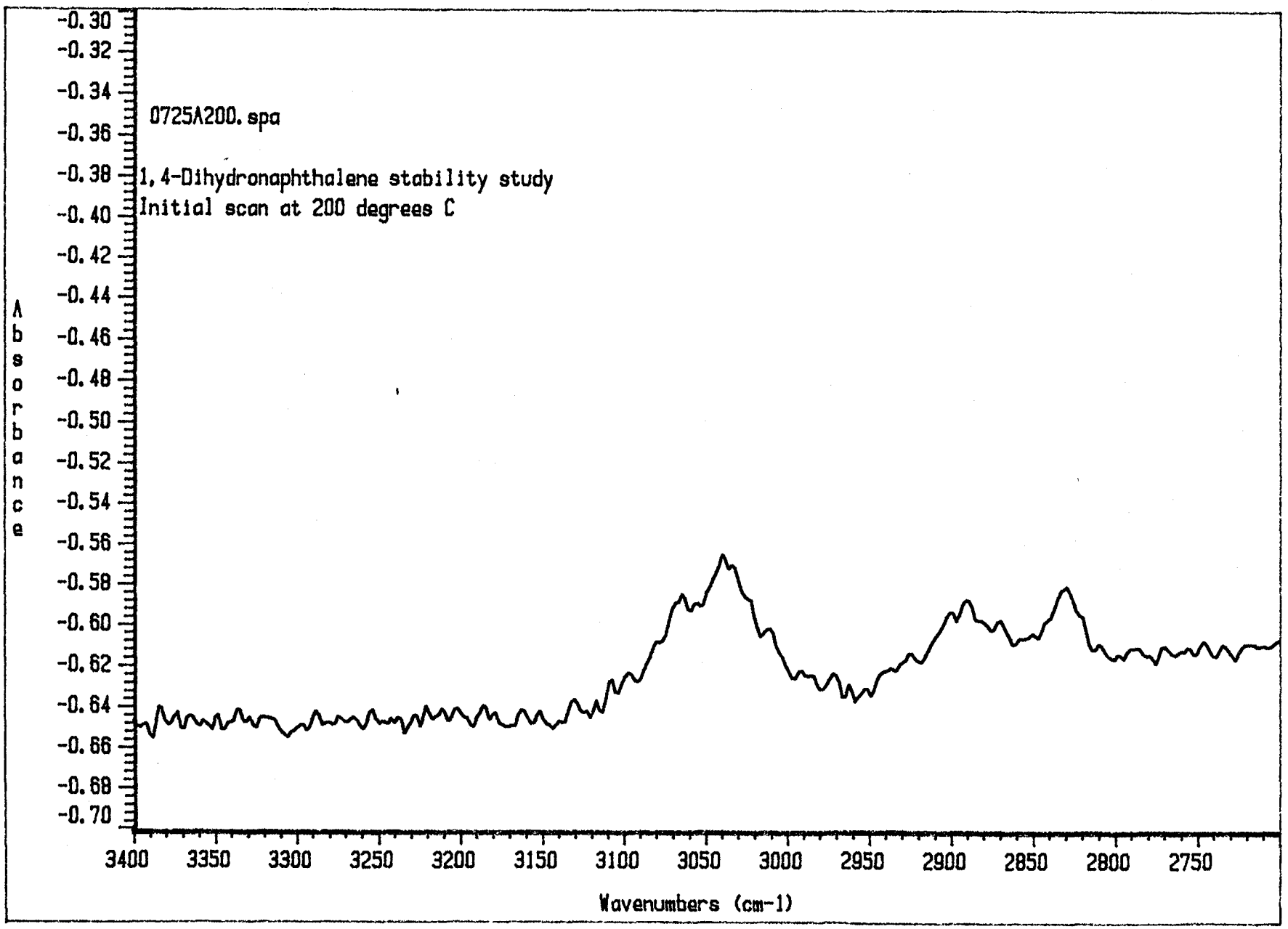

0725A200.spa
1,4-Dihydronaphthalene stability study
Initial scan at 200 degrees C
Absorbance
-0.30
-0.32
-0.34
-0.36
-0.38
-0.40
-0.42
-0.44
-0.46
-0.48
-0.50
-0.52
-0.54
-0.56
-0.58
-0.60
-0.62
-0.64
-0.66
-0.68
-0.70
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 5

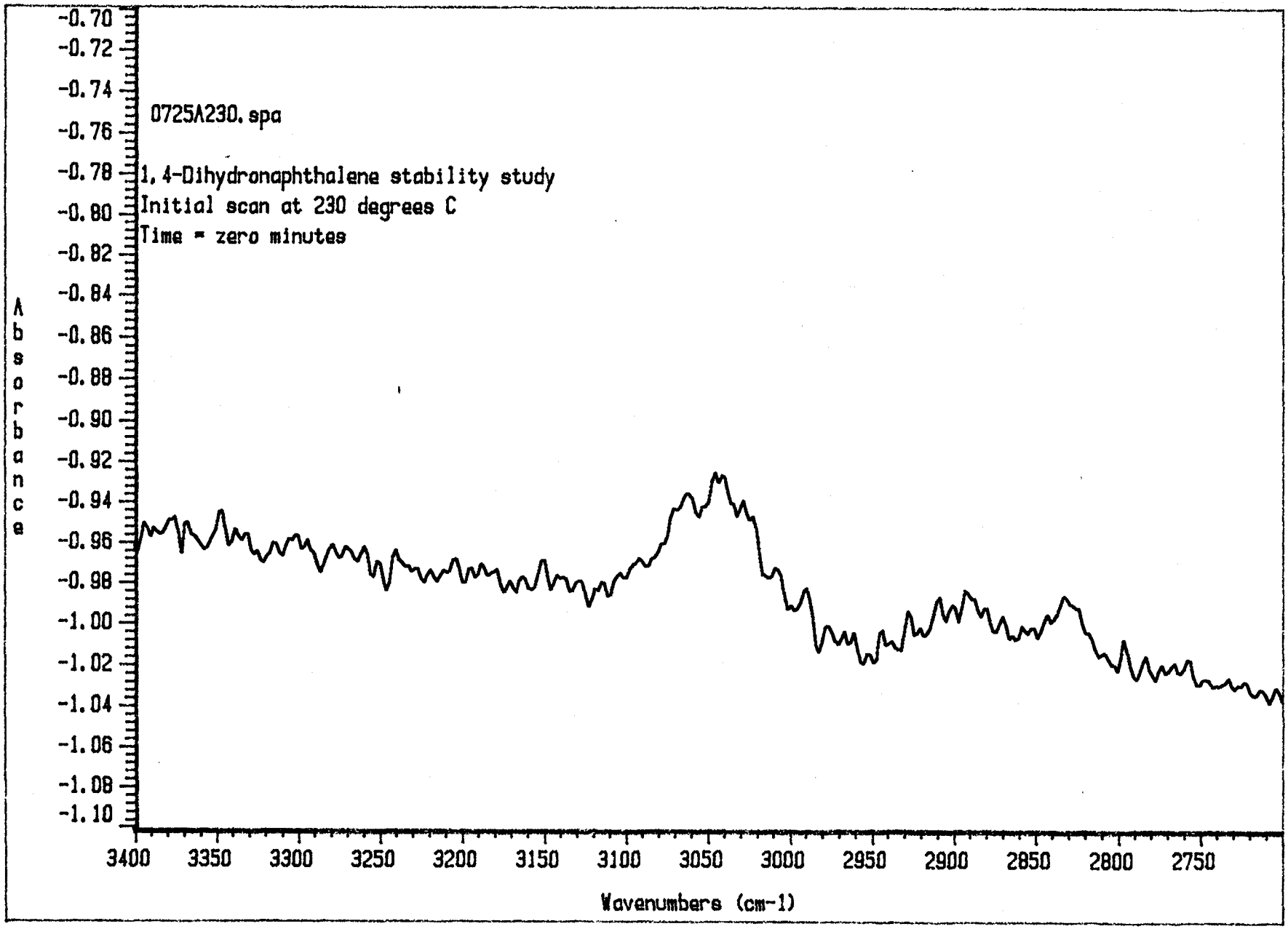

0725A230.spa
1,4-Dihydronaphthalene stability study
Initial scan at 230 degrees C
Time = zero minutes
Absorbance
-0.70
-0.72
-0.74
-0.76
-0.78
-0.80
-0.82
-0.84
-0.86
-0.88
-0.90
-0.92
-0.94
-0.96
-0.98
-1.00
-1.02
-1.04
-1.06
-1.08
-1.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 6

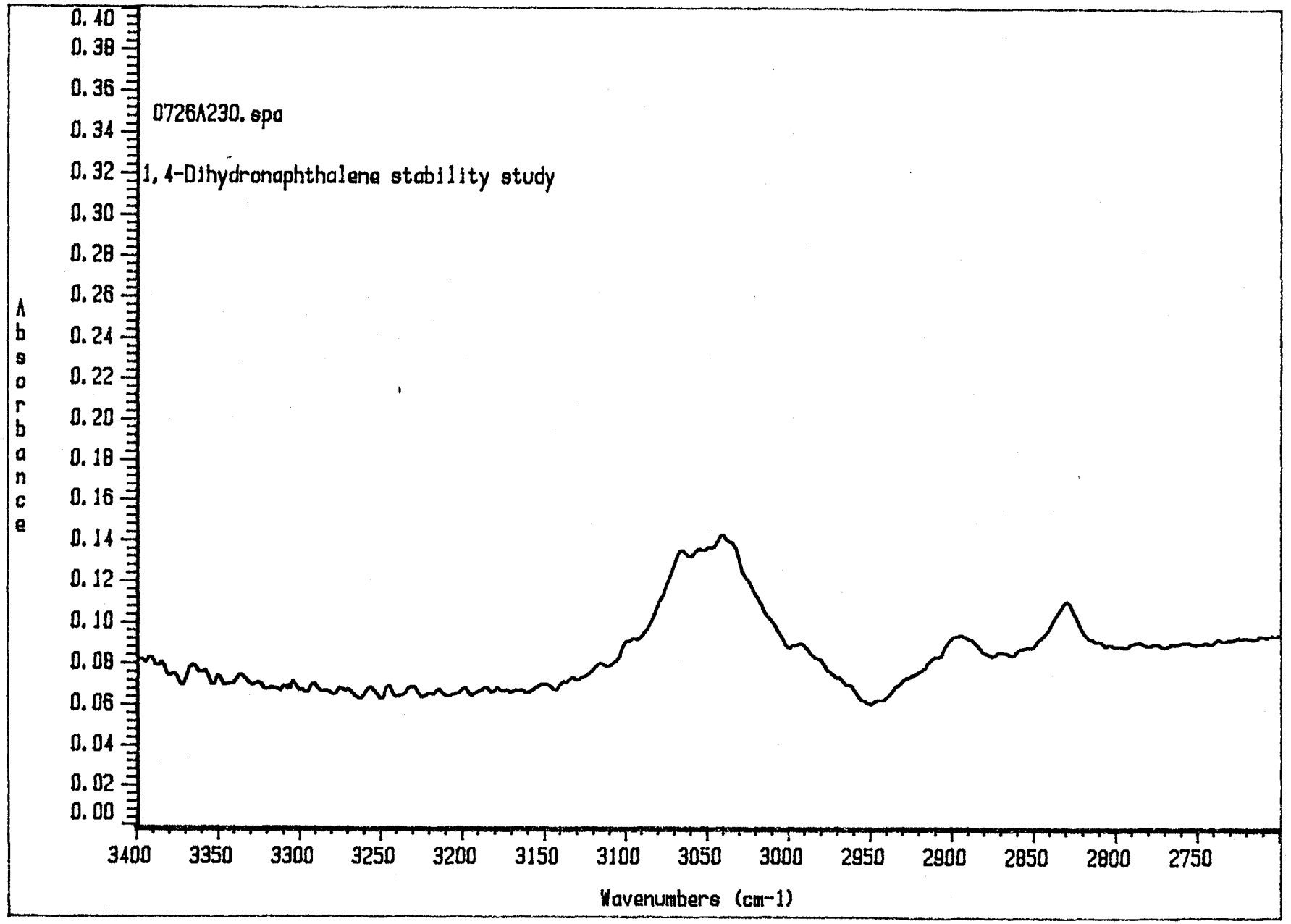

0726A230.spa
1,4-Dihydronaphthalene stability study
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 7

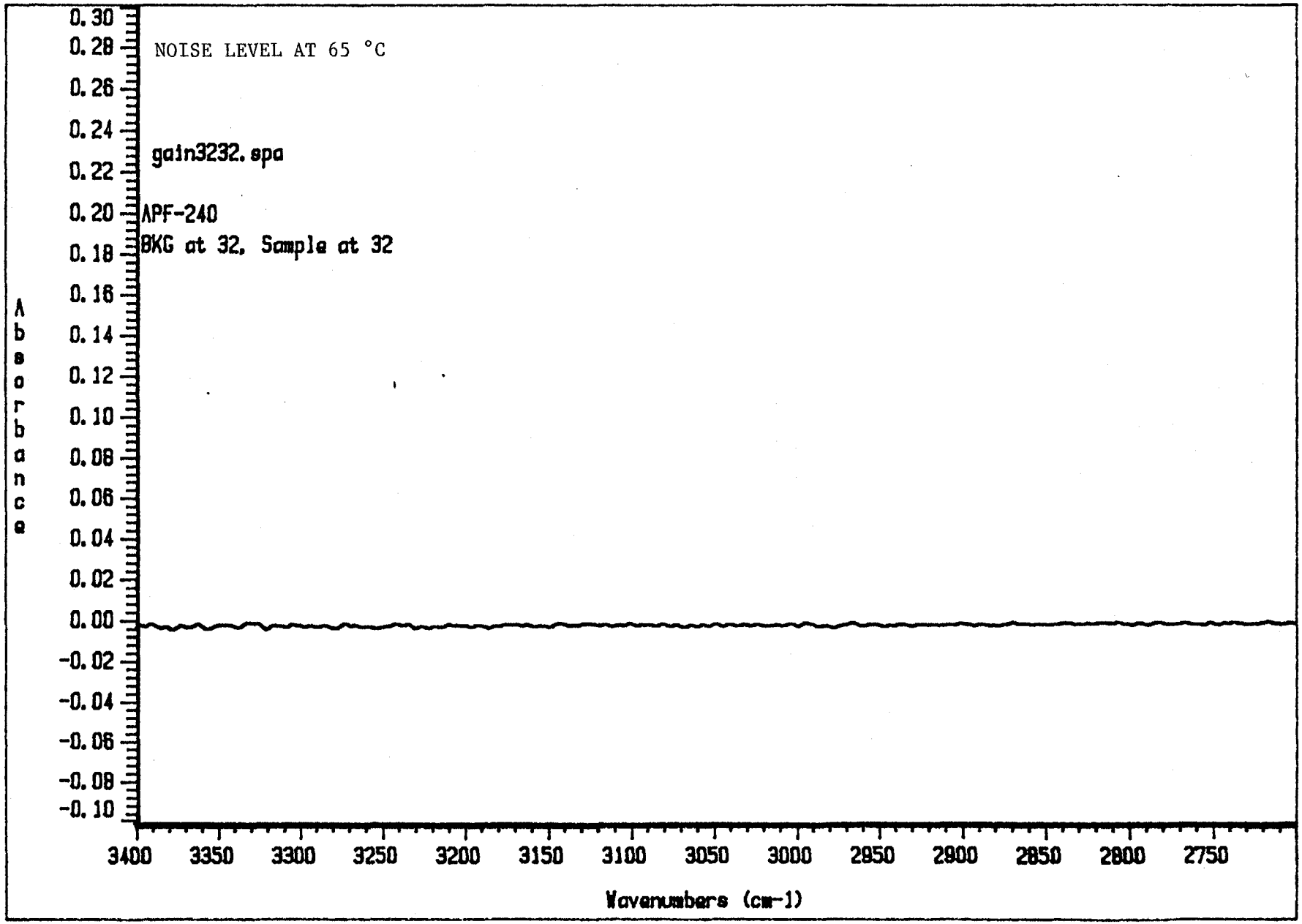

NOISE LEVEL AT 65 °C
gain3232.spa
APF-240
BKG at 32, Sample at 32
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 8

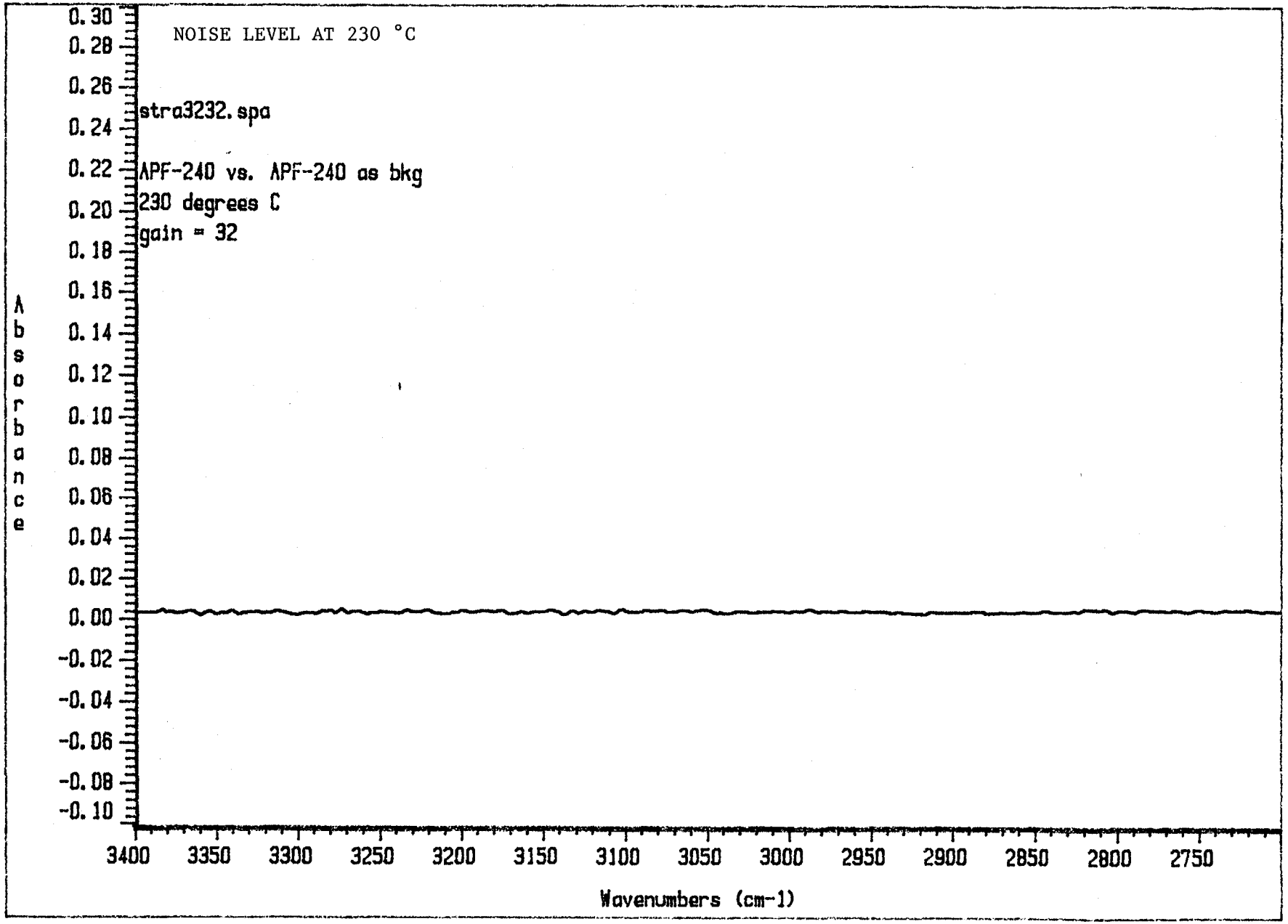

NOISE LEVEL AT 230 °C
stra3232.spa
APF-240 vs. APF-240 as bkg
230 degrees C
gain = 32
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 9

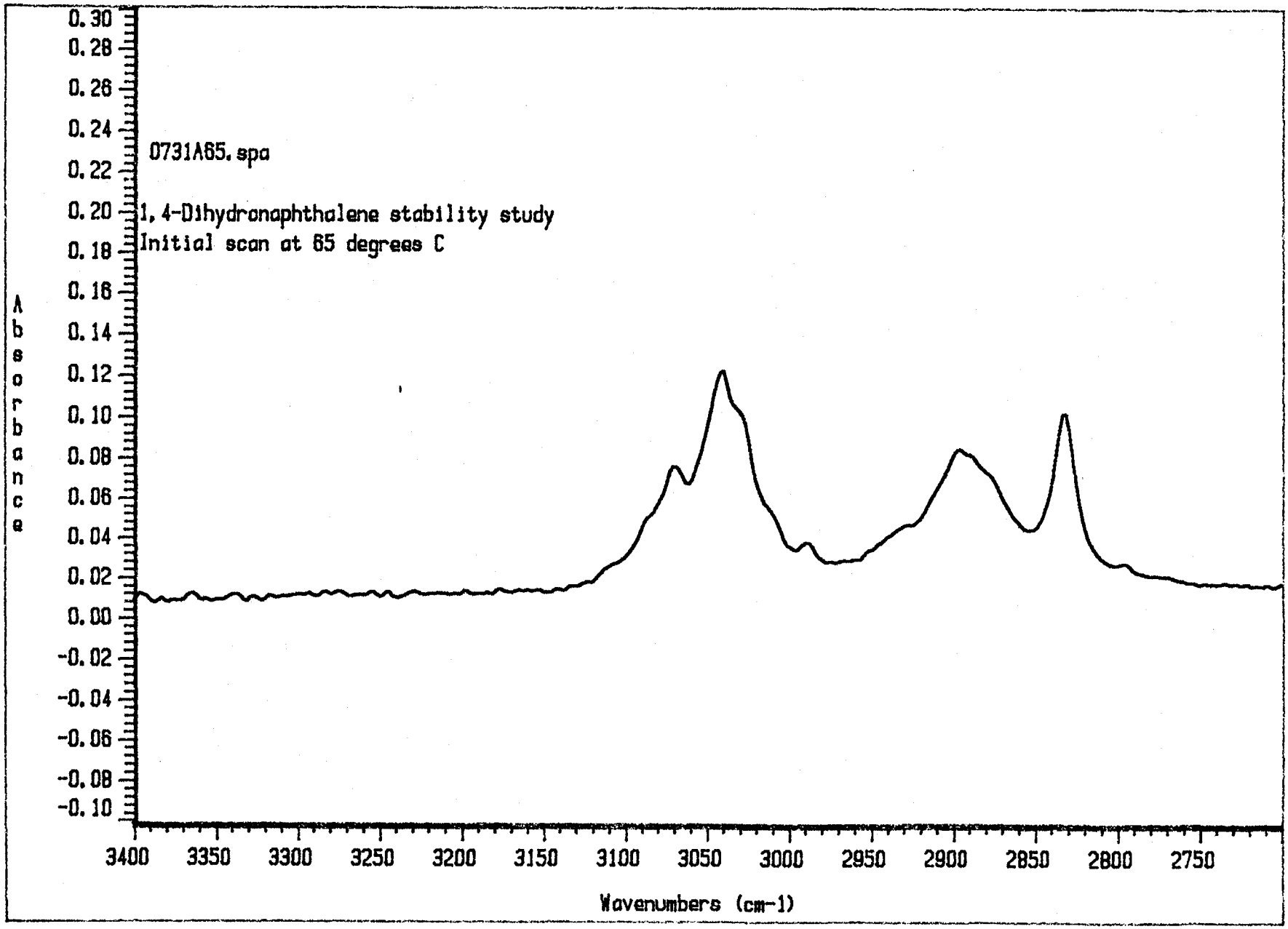

0731A65.spa
1,4-Dihydronaphthalene stability study
Initial scan at 65 degrees C
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 10

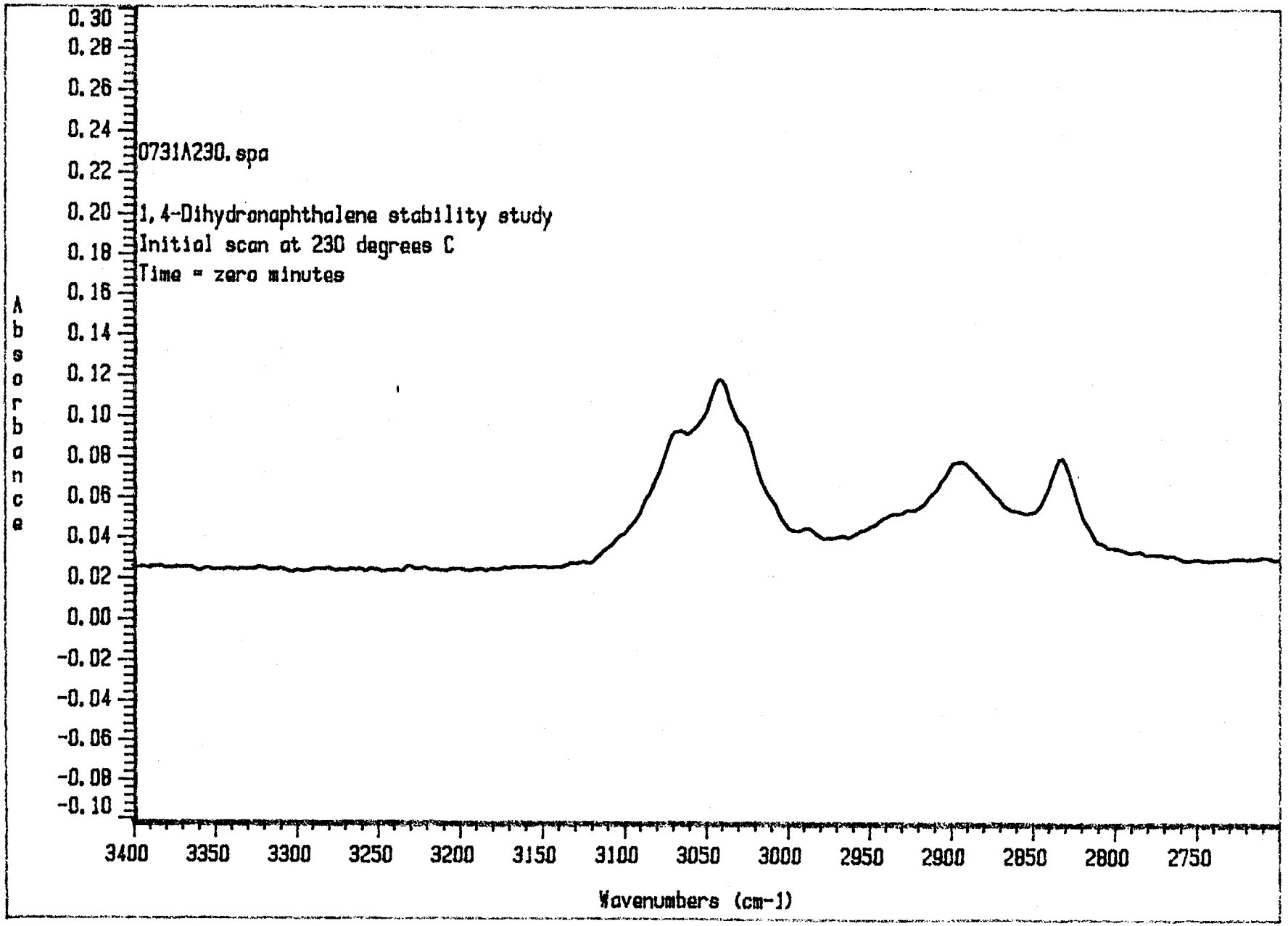

0731A230.spa
1,4-Dihydronaphthalene stability study
Initial scan at 230 degrees C
Time = zero minutes
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 11

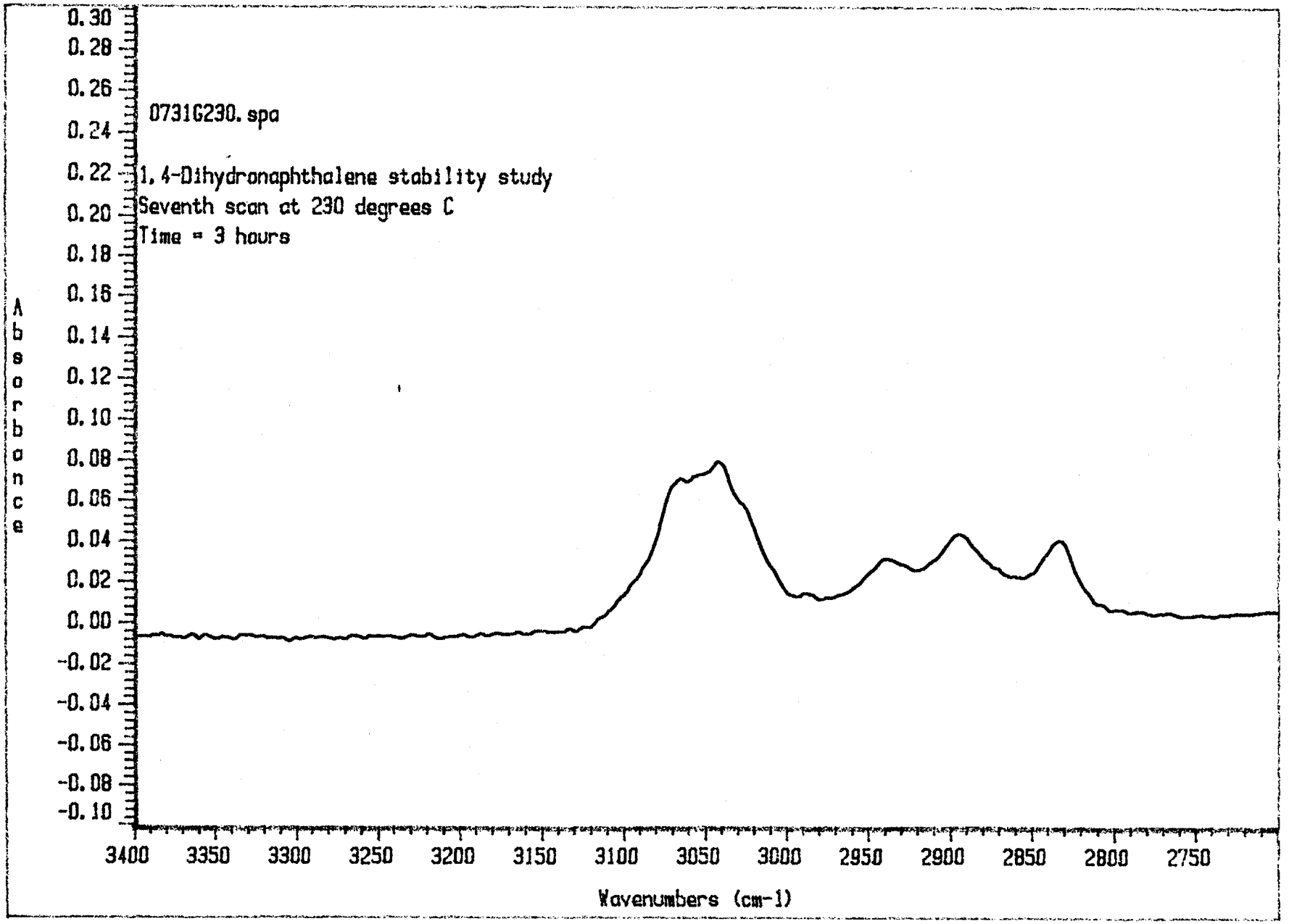

0731G230.spa
1,4-Dihydronaphthalene stability study
Seventh scan at 230 degrees C
Time = 3 hours
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 12

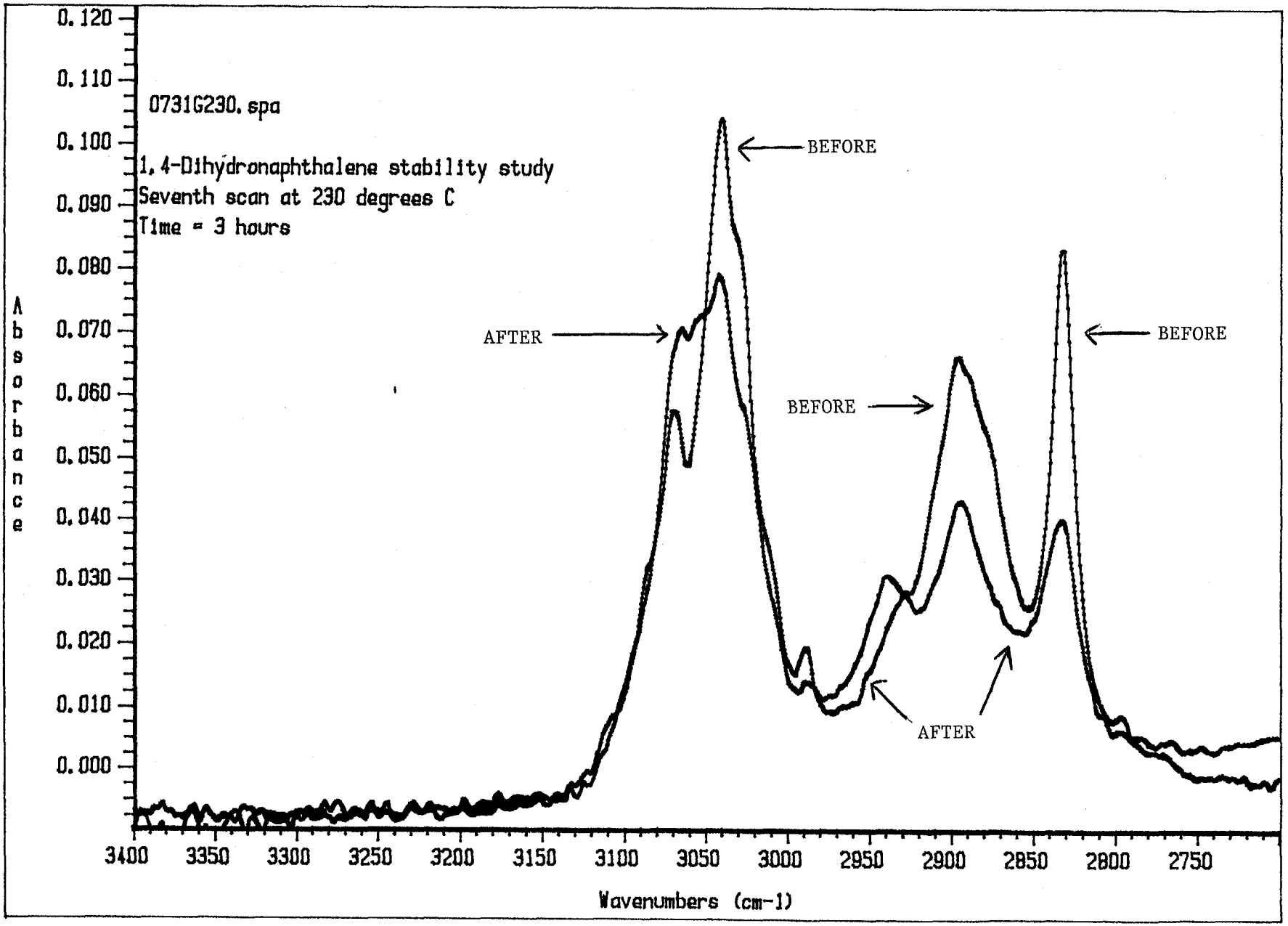

0731G230.spa
1,4-Dihydronaphthalene stability study
Seventh scan at 230 degrees C
Time = 3 hours
BEFORE
AFTER
BEFORE
BEFORE
AFTER
Absorbance
0.120
0.110
0.100
0.090
0.080
0.070
0.060
0.050
0.040
0.030
0.020
0.010
0.000
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 13

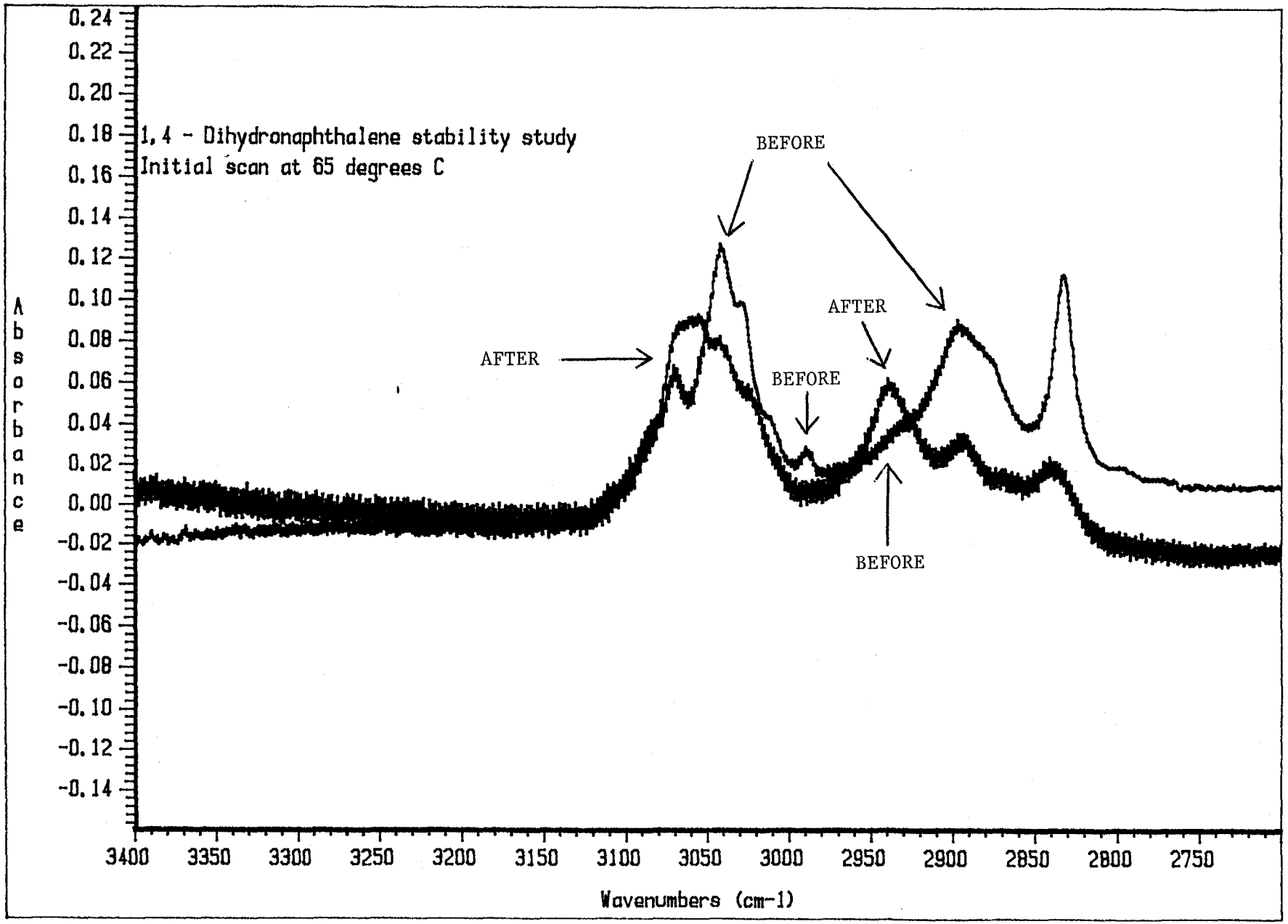

1,4 - Dihydronaphthalene stability study
Initial scan at 65 degrees C
BEFORE
AFTER
AFTER
BEFORE
BEFORE
Absorbance
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
-0.12
-0.14
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 14

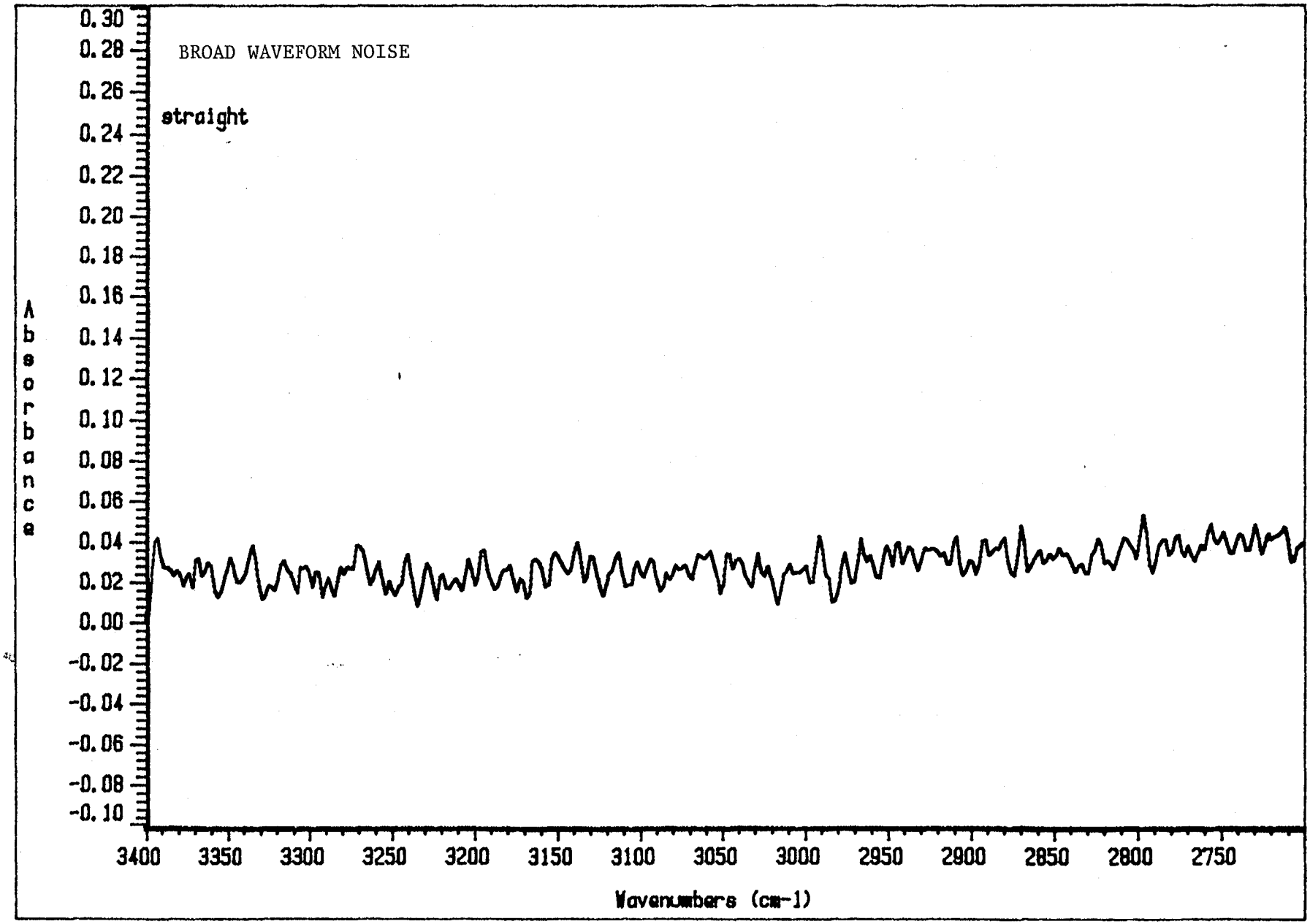

BROAD WAVEFORM NOISE
straight
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 15

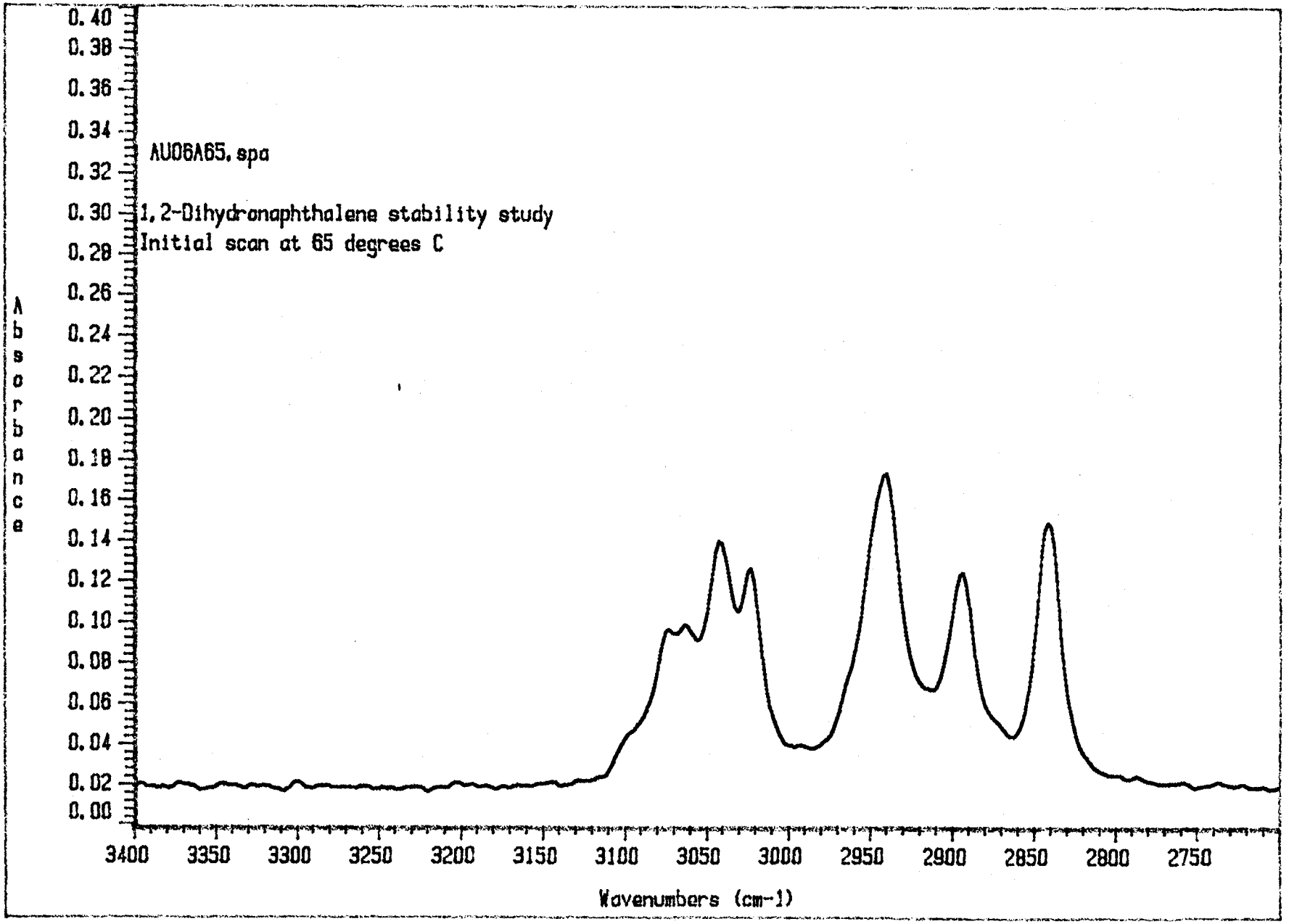

AU06A65.spa
1,2-Dihydronaphthalene stability study
Initial scan at 65 degrees C
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 16

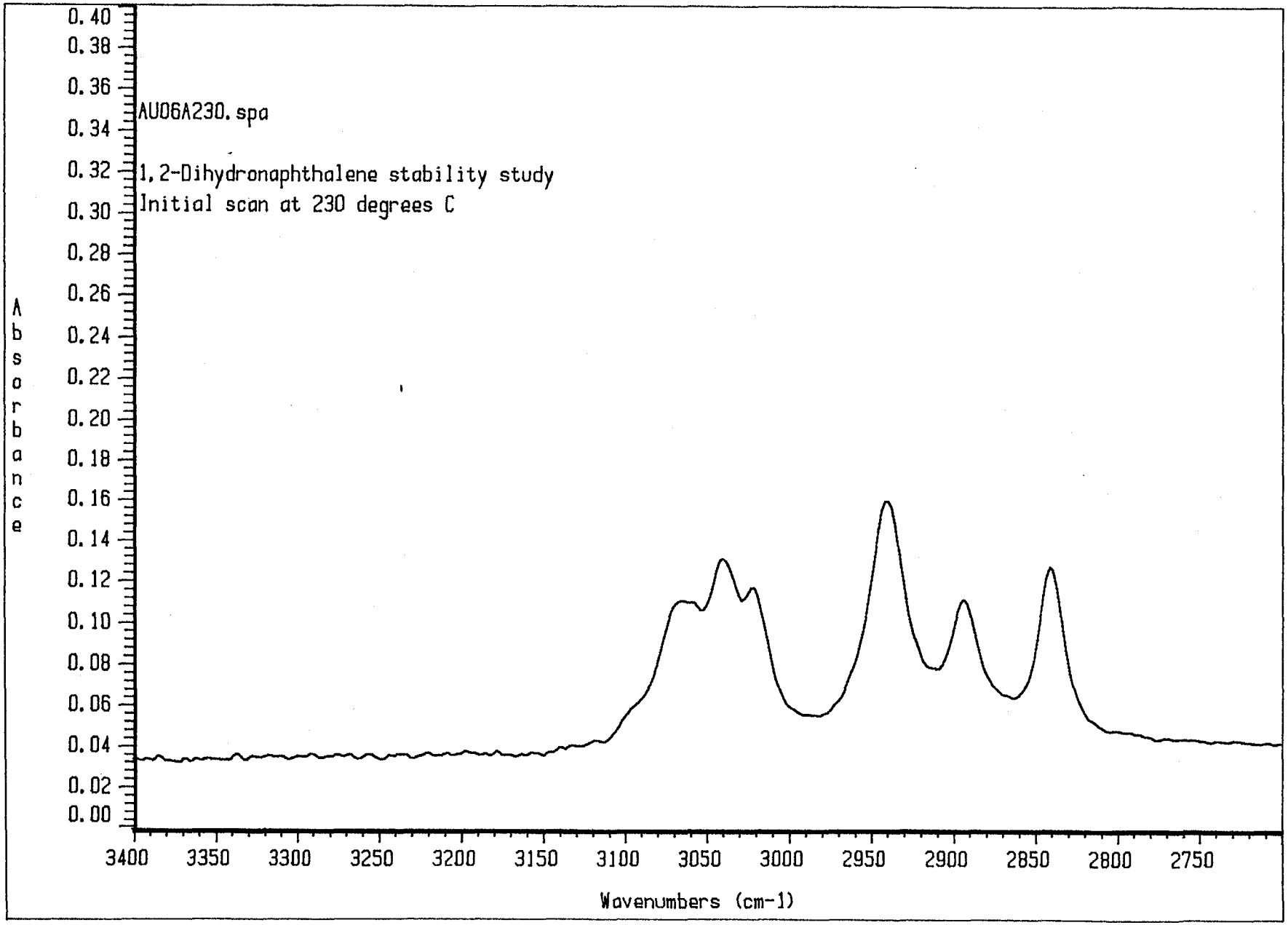

AU06A230.spa
1,2-Dihydronaphthalene stability study
Initial scan at 230 degrees C
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 17

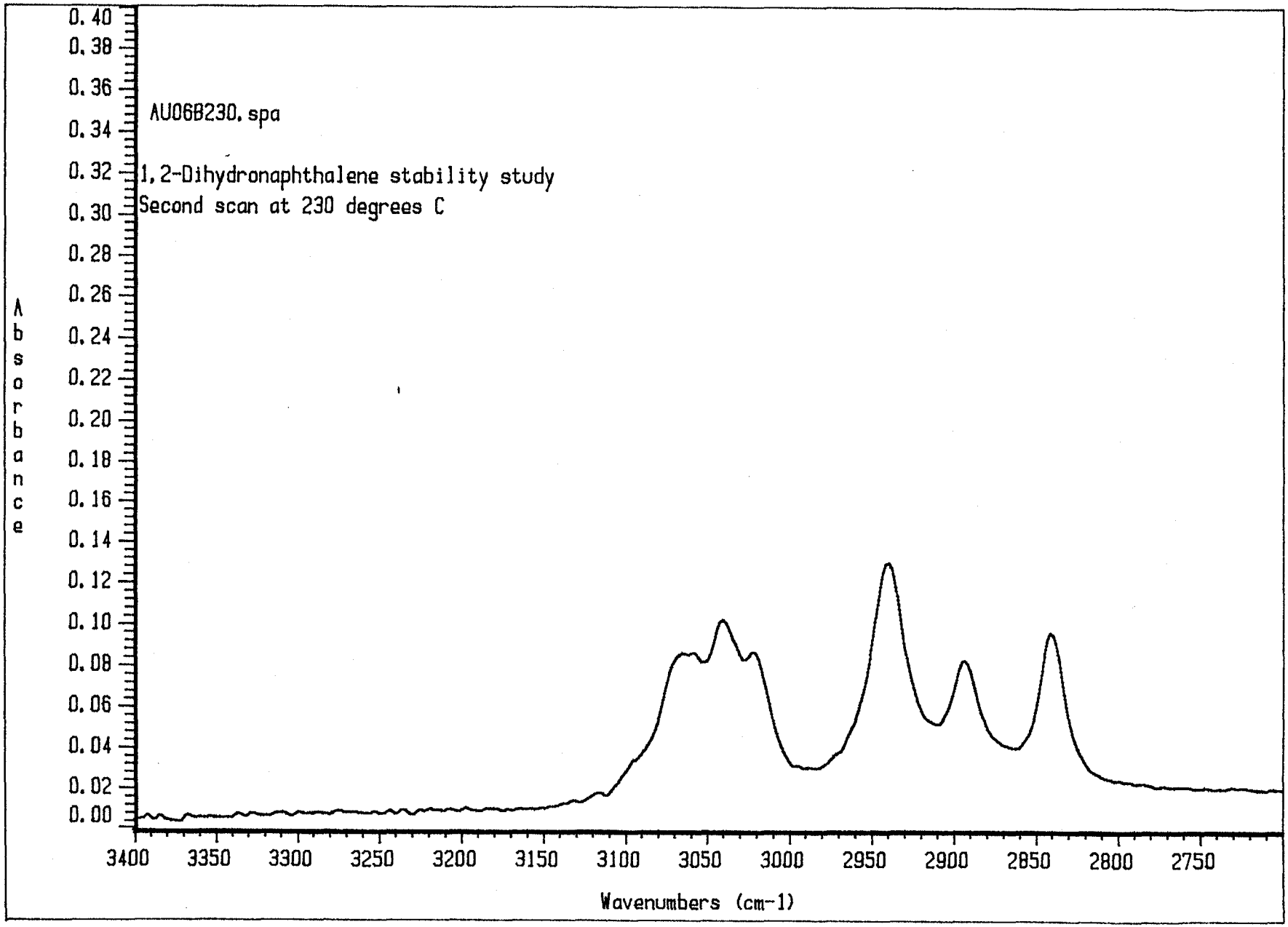

AU06B230.spa
1,2-Dihydronaphthalene stability study
Second scan at 230 degrees C
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 18

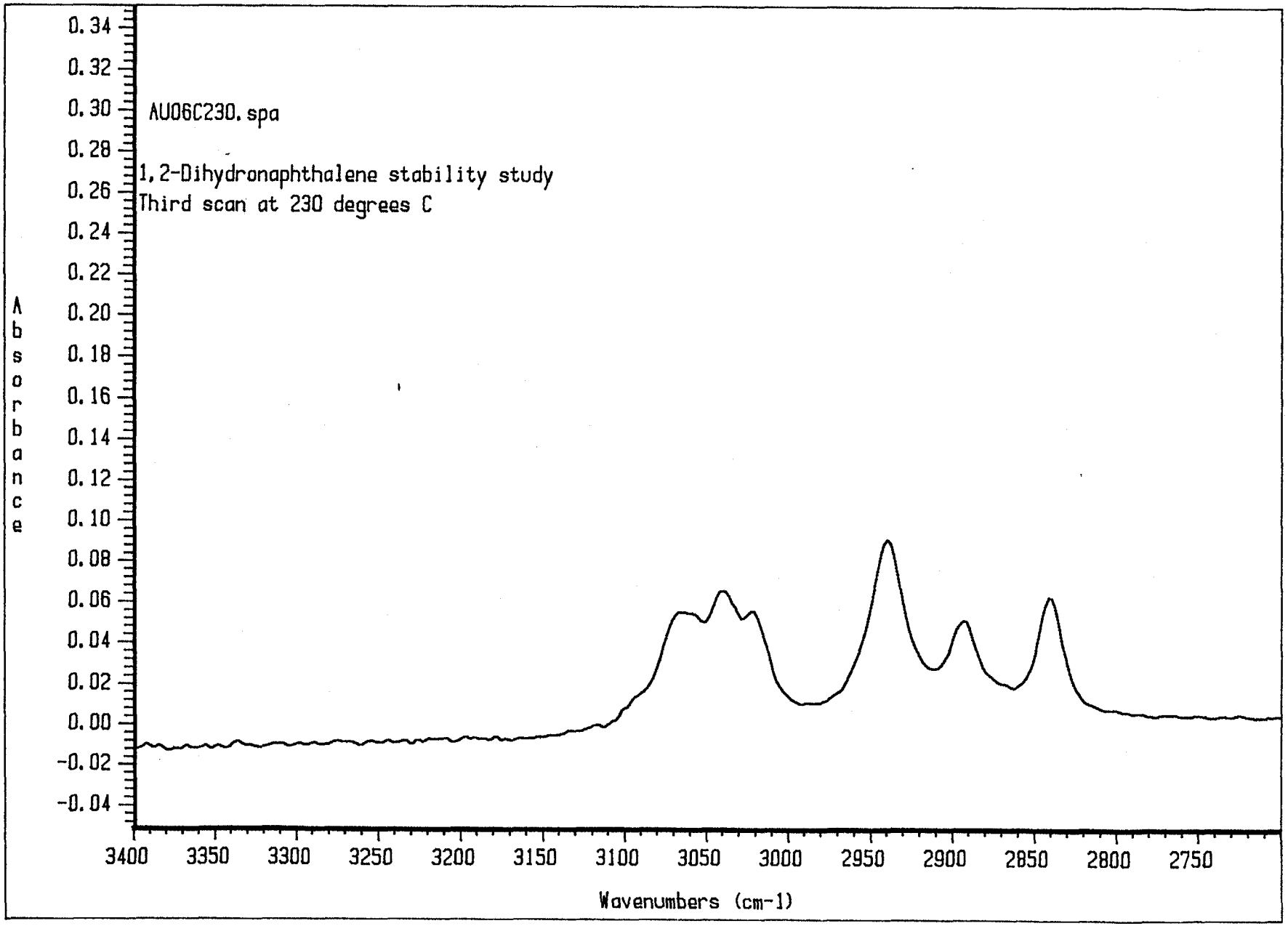

AU06C230.spa
1,2-Dihydronaphthalene stability study
Third scan at 230 degrees C
Absorbance
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 19

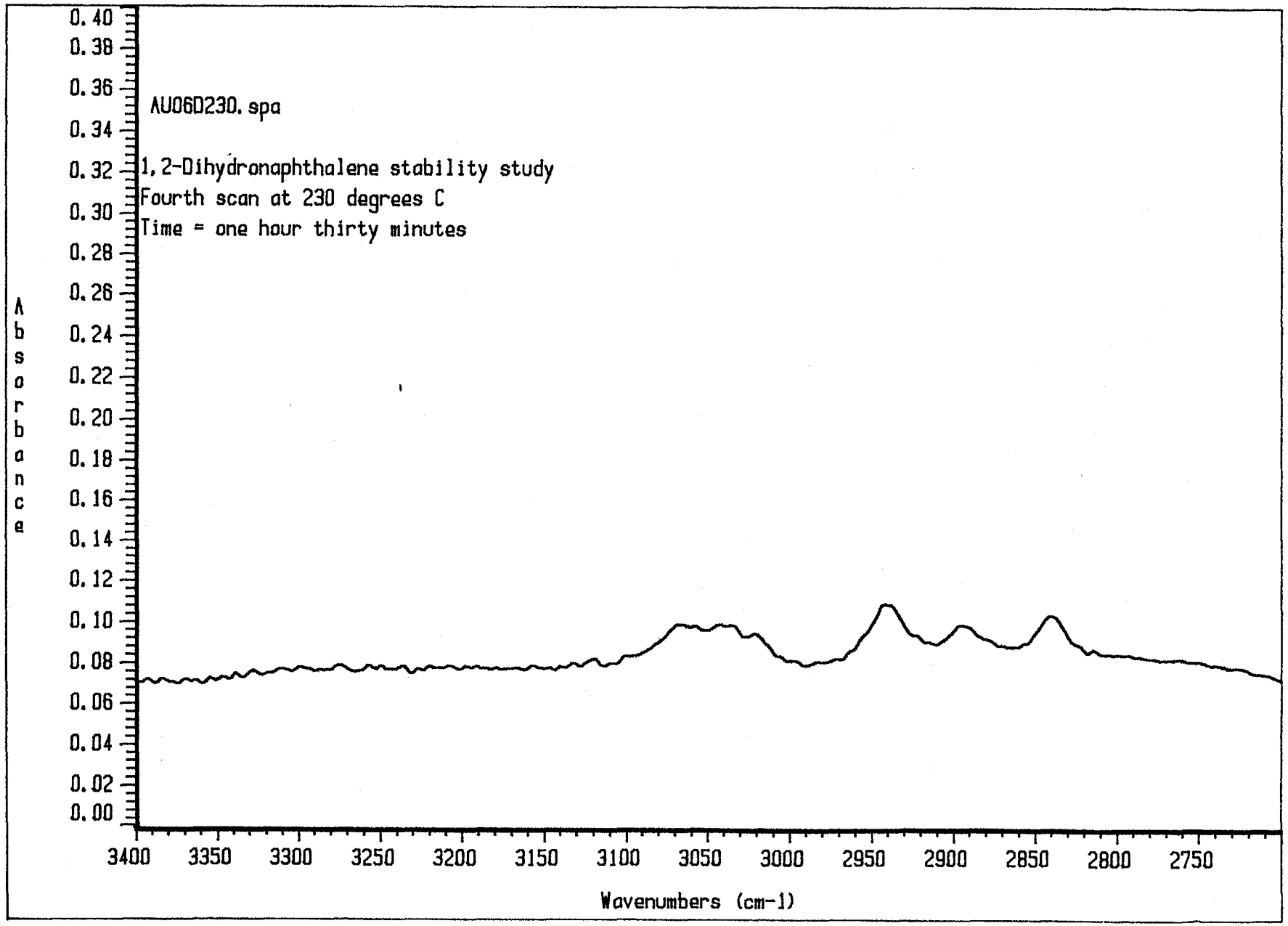

AU06D230.spa
1,2-Dihydronaphthalene stability study
Fourth scan at 230 degrees C
Time = one hour thirty minutes
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 20

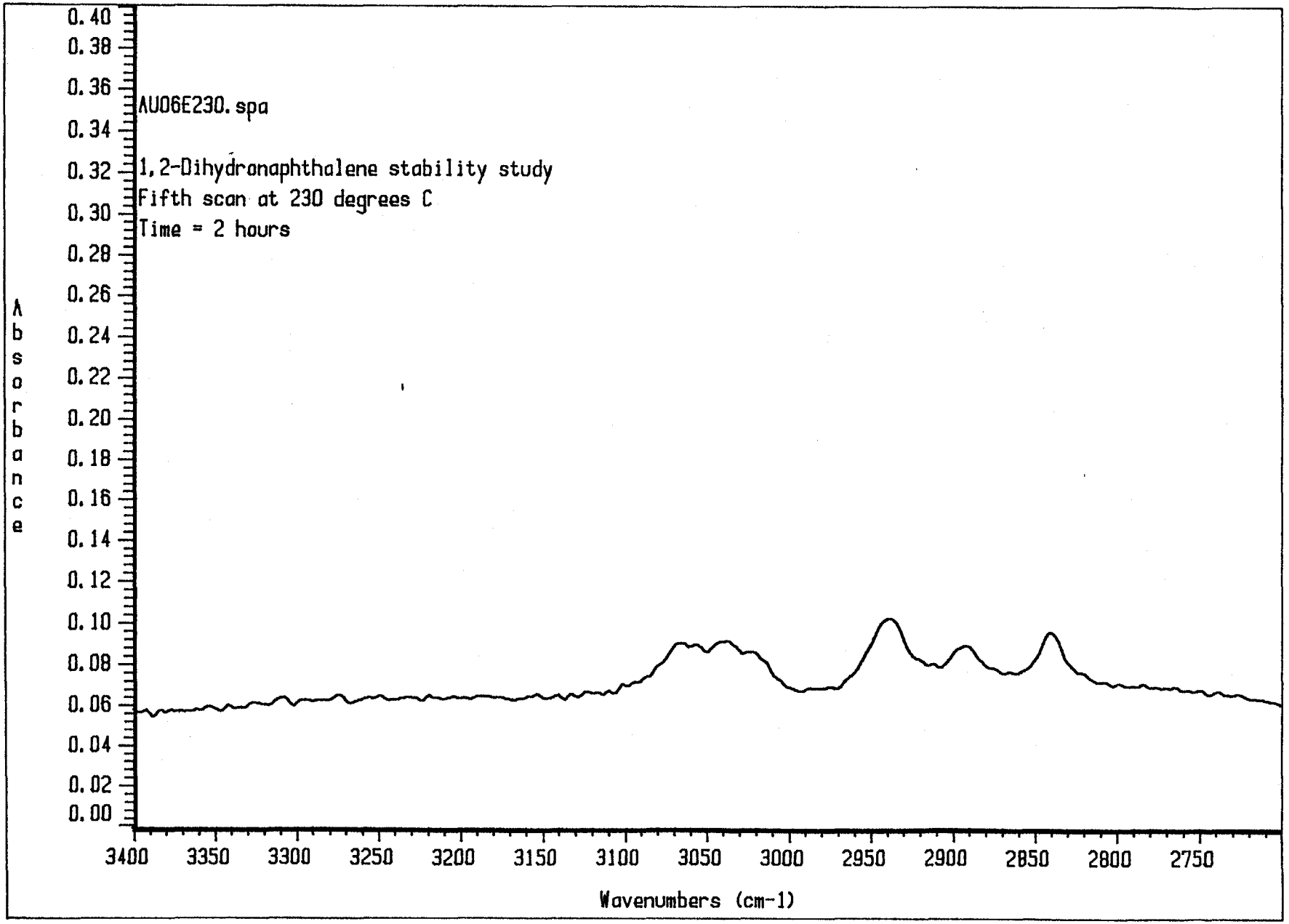

AU06E230.spa
1,2-Dihydronaphthalene stability study
Fifth scan at 230 degrees C
Time = 2 hours
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 21

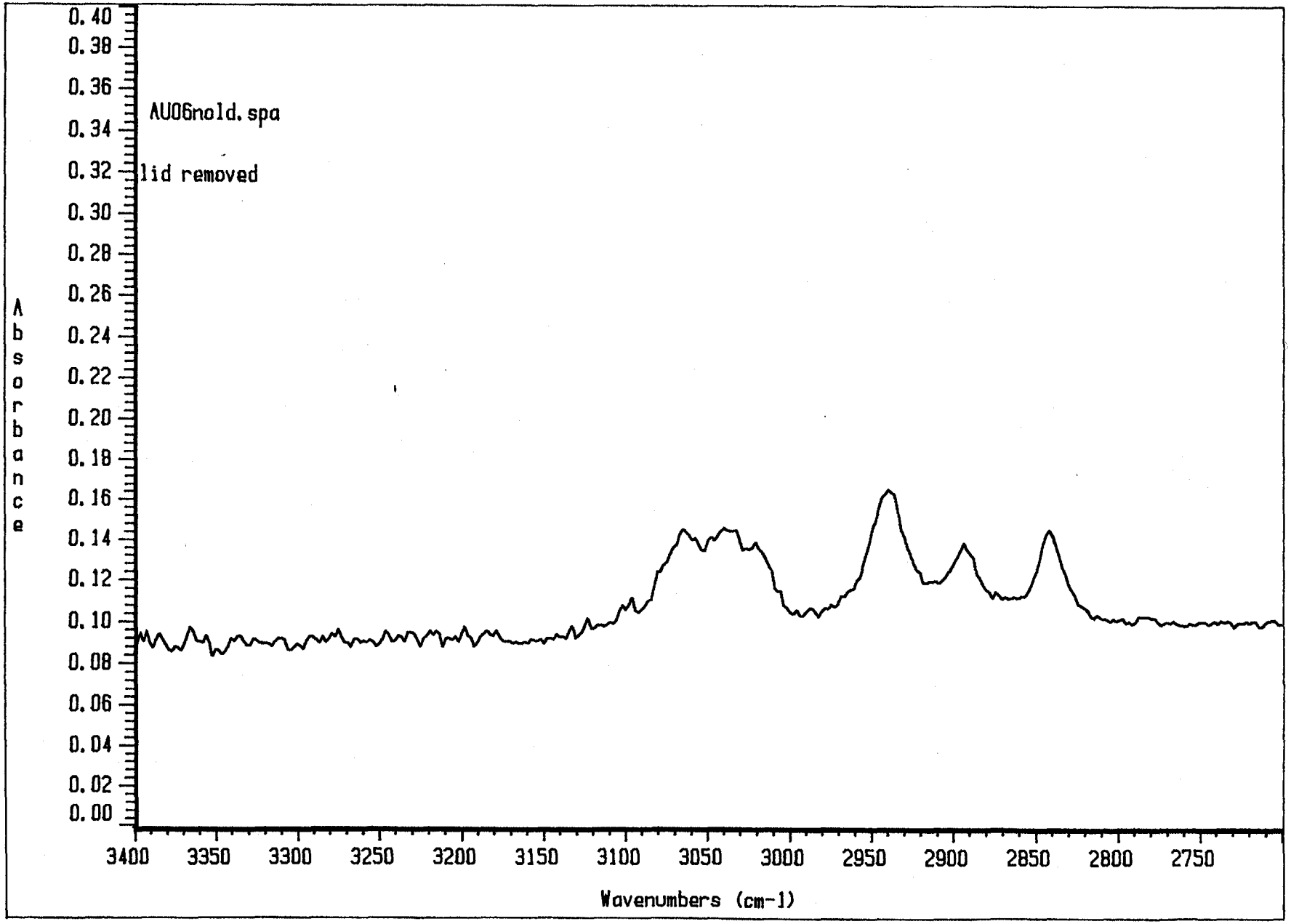

AU06nold.spa
lid removed
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 22

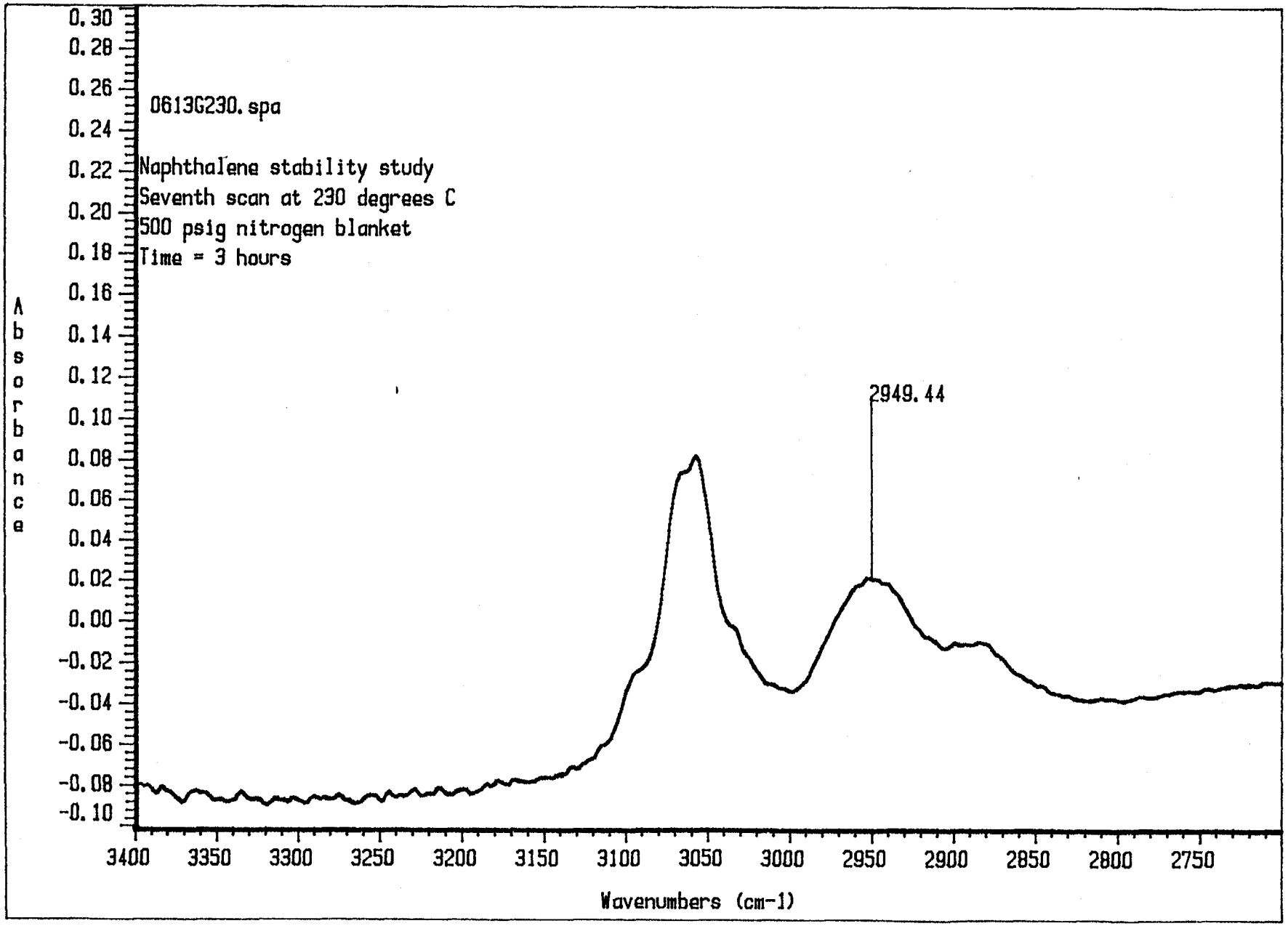

0613G230.spa
Naphthalene stability study
Seventh scan at 230 degrees C
500 psig nitrogen blanket
Time = 3 hours
2949.44
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 23

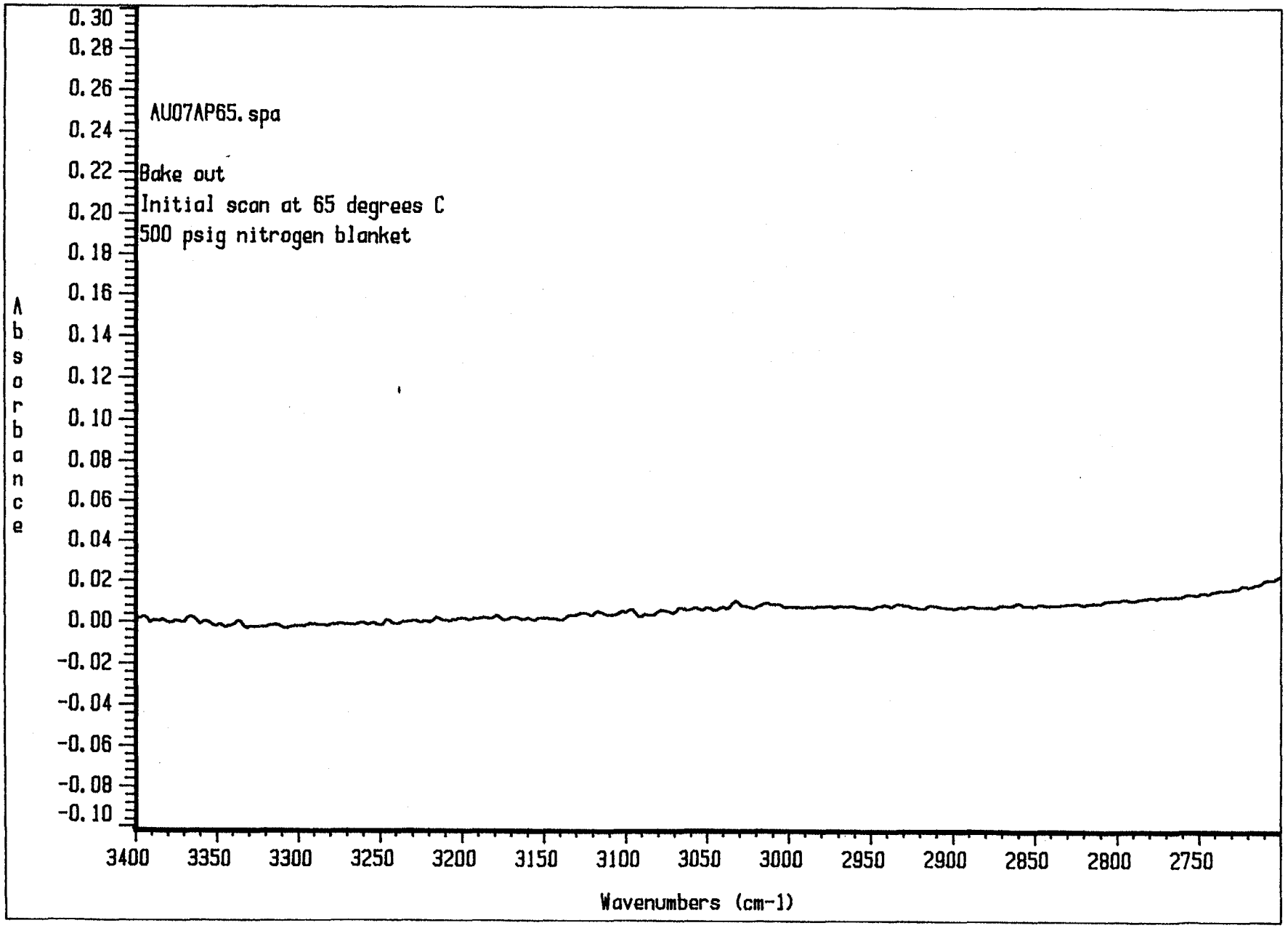

AU07AP65.spa
Bake out
Initial scan at 65 degrees C
500 psig nitrogen blanket
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 24

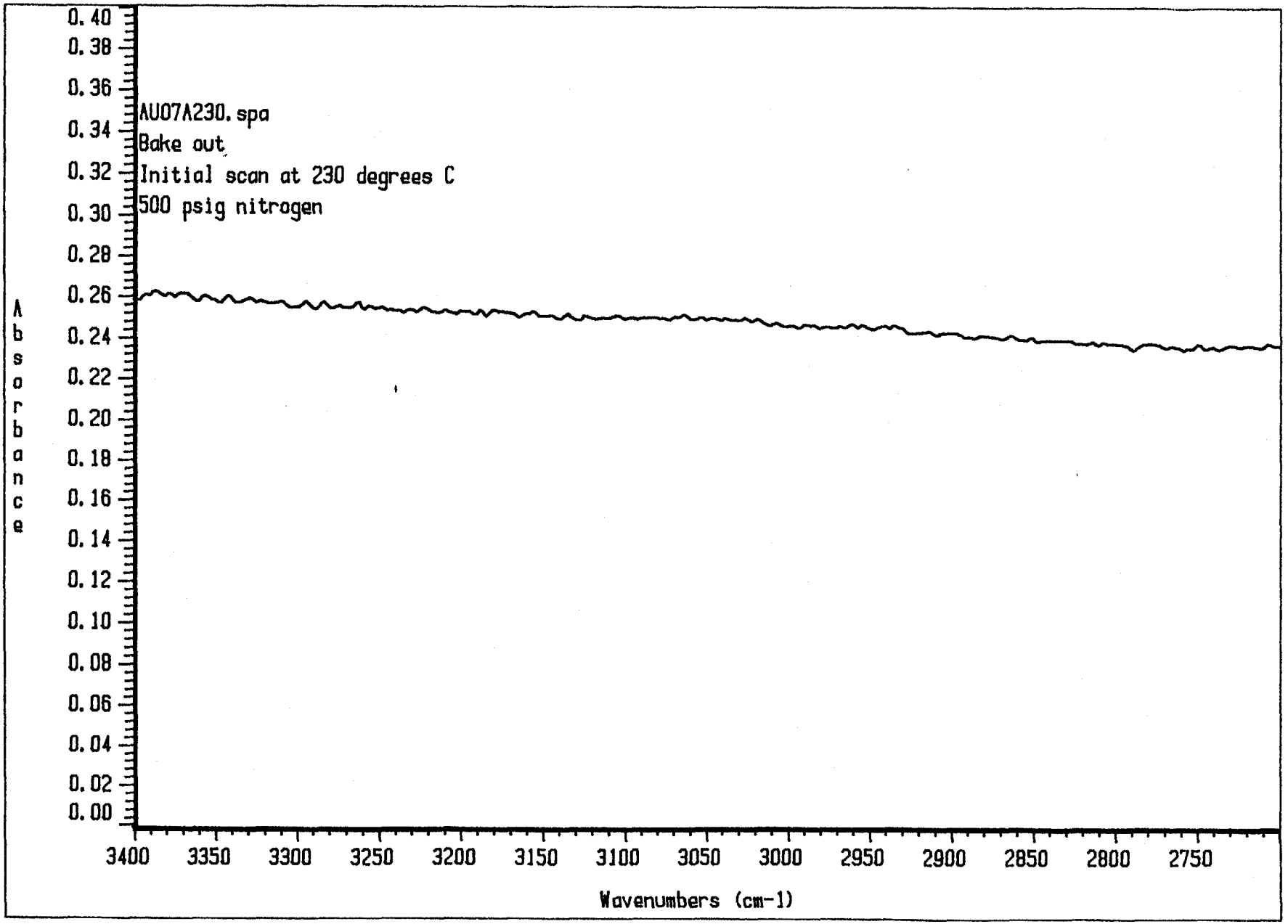

AU07A230.spa
Bake out
Initial scan at 230 degrees C
500 psig nitrogen
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 25

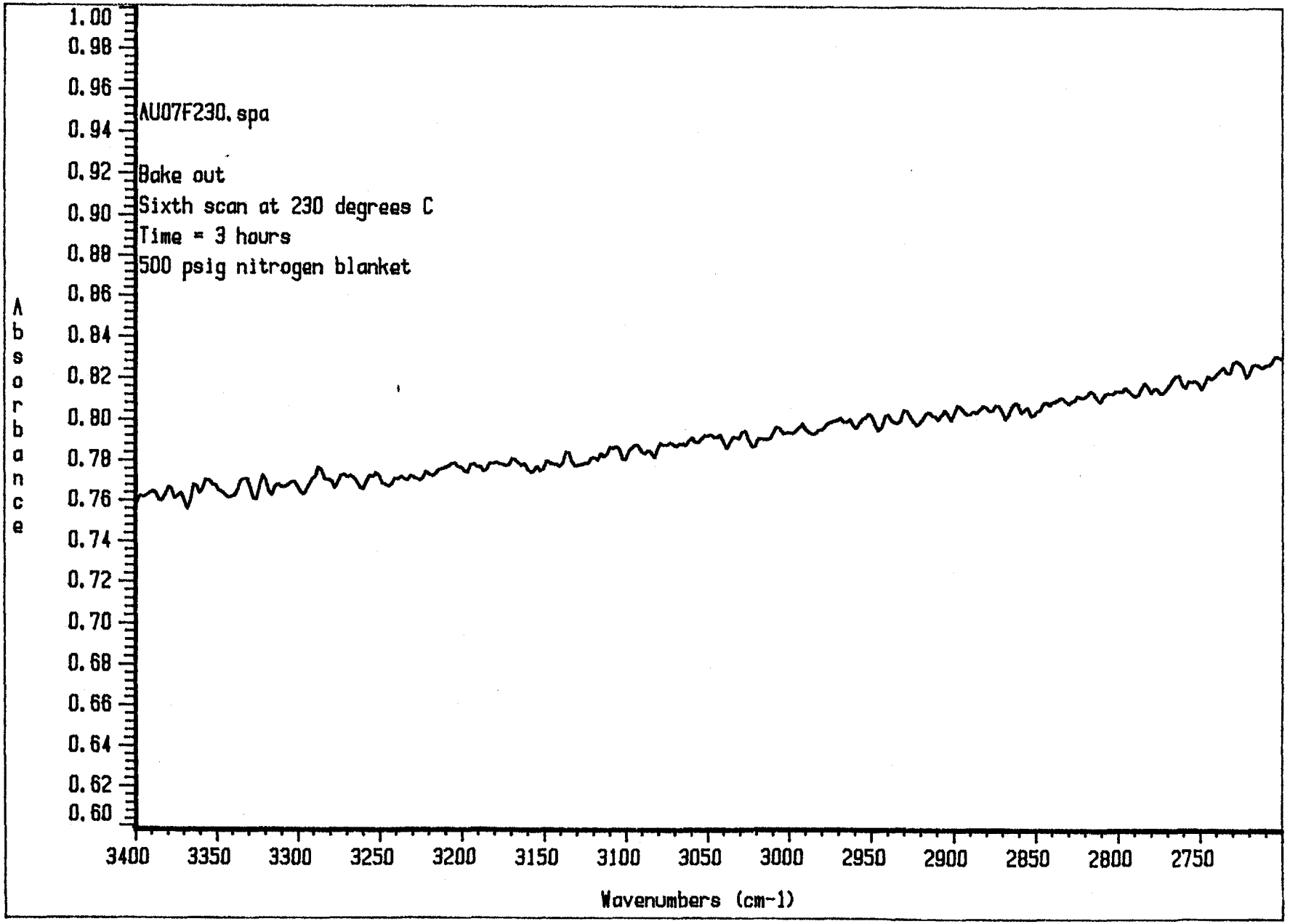

AU07F230.spa
Bake out
Sixth scan at 230 degrees C
Time = 3 hours
500 psig nitrogen blanket
Absorbance
1.00
0.98
0.96
0.94
0.92
0.90
0.88
0.86
0.84
0.82
0.80
0.78
0.76
0.74
0.72
0.70
0.68
0.66
0.64
0.62
0.60
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 26

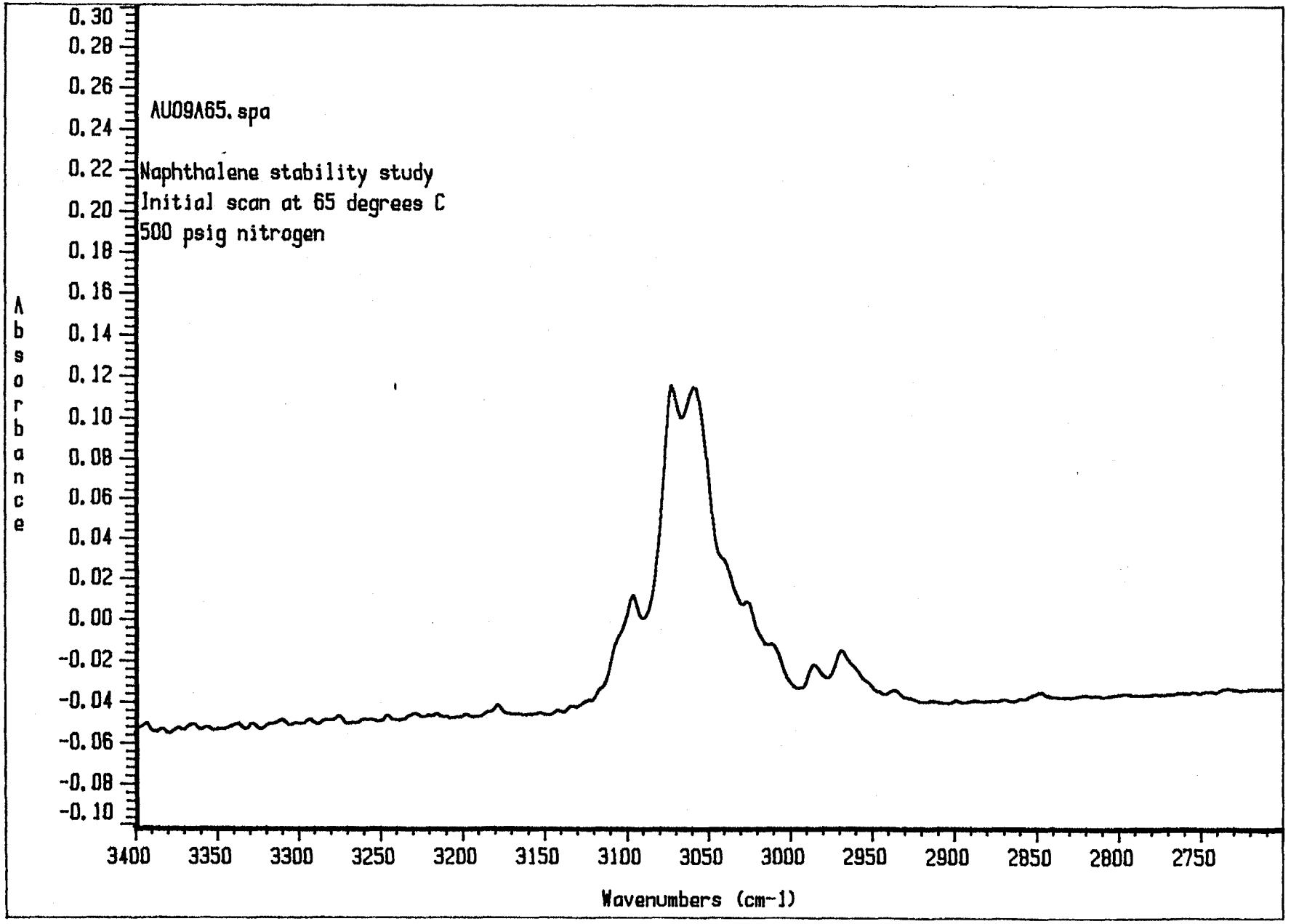

AU09A65.spa
Naphthalene stability study
Initial scan at 65 degrees C
500 psig nitrogen
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 27

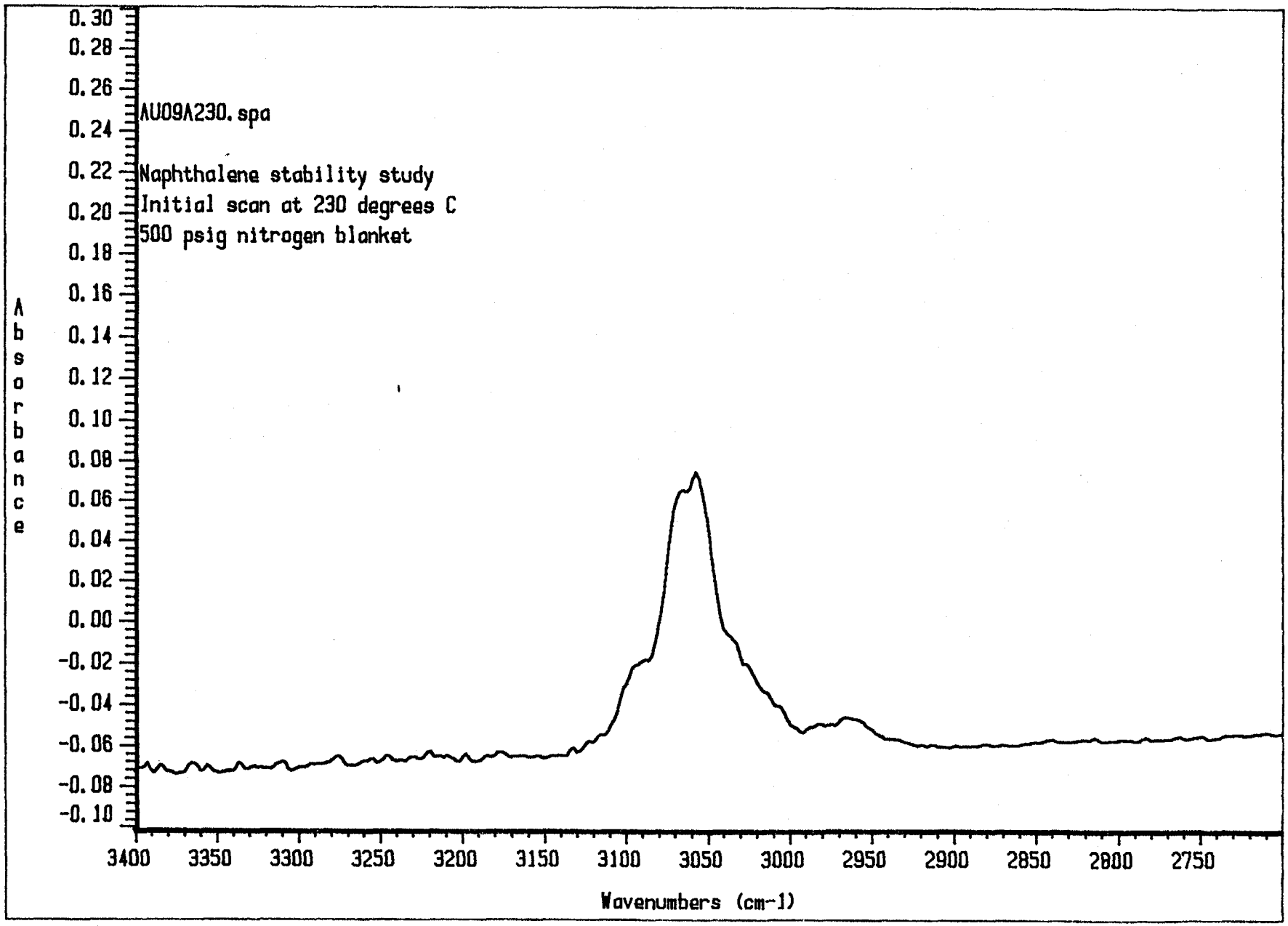

AU09A230.spa
Naphthalene stability study
Initial scan at 230 degrees C
500 psig nitrogen blanket
Absorbance
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
-0.06
-0.08
-0.10
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 28

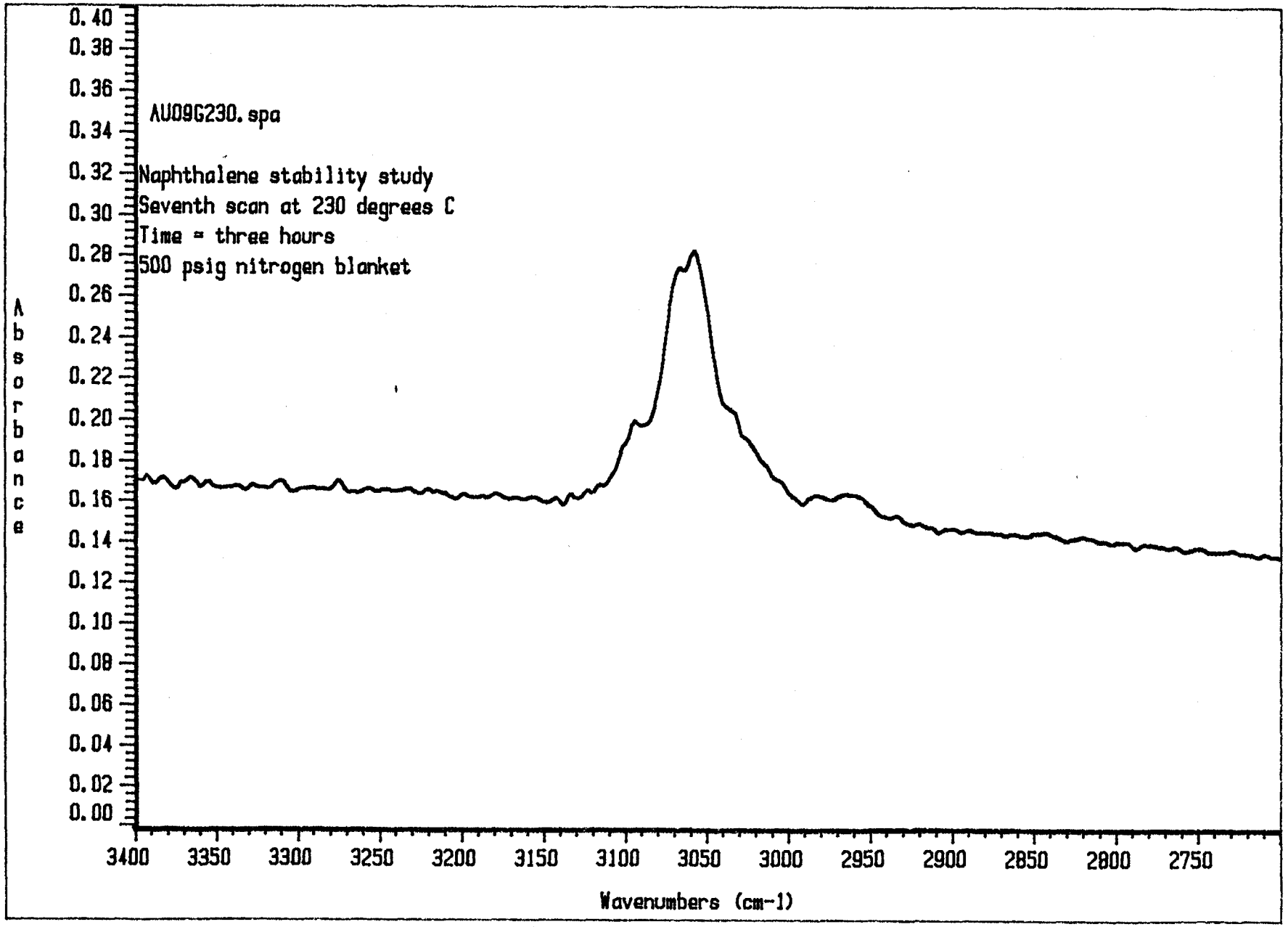

AU09G230.spa
Naphthalene stability study
Seventh scan at 230 degrees C
Time = three hours
500 psig nitrogen blanket
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 29

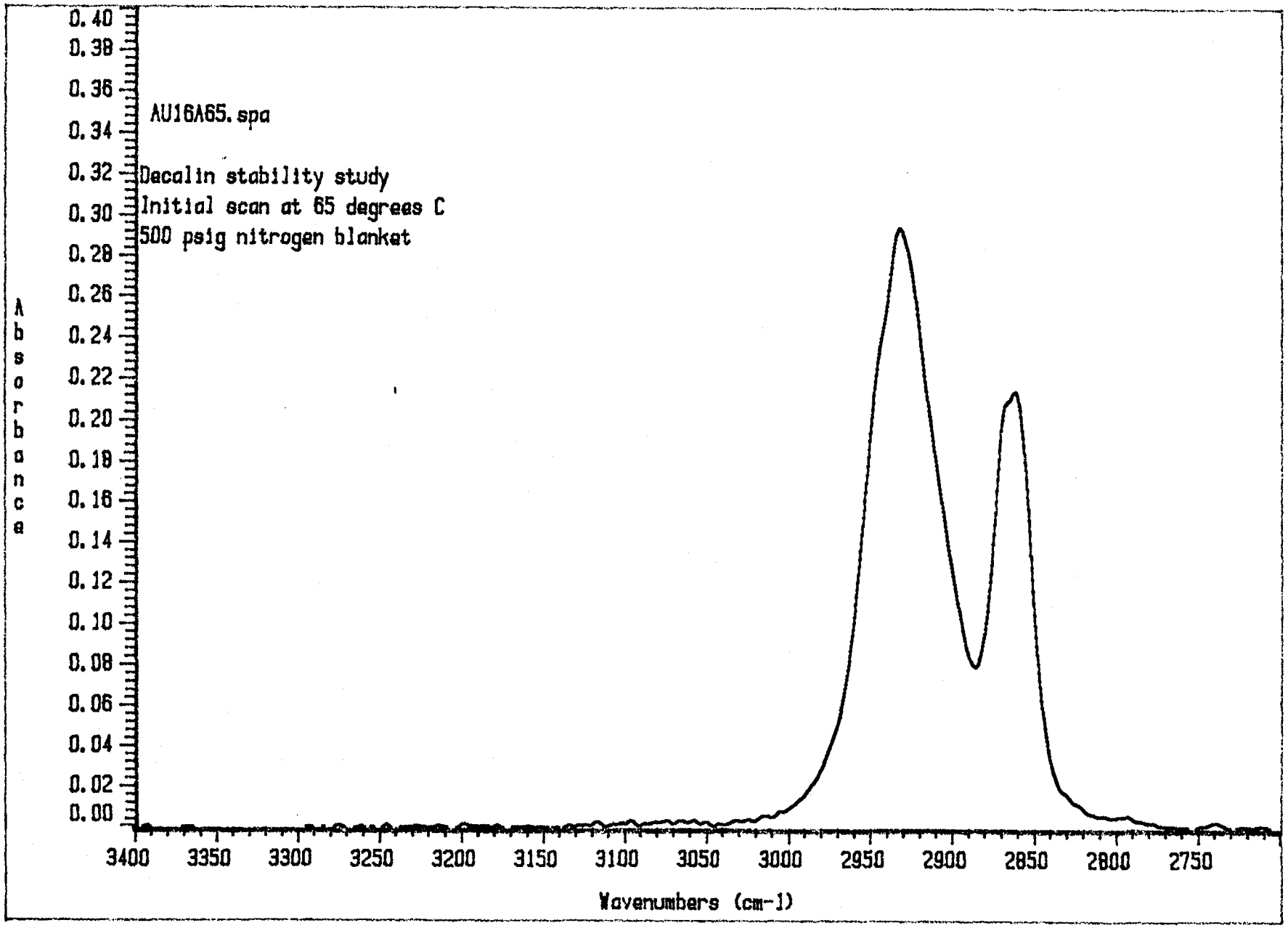

AU16A65.spa
Decalin stability study
Initial scan at 65 degrees C
500 psig nitrogen blanket
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 30

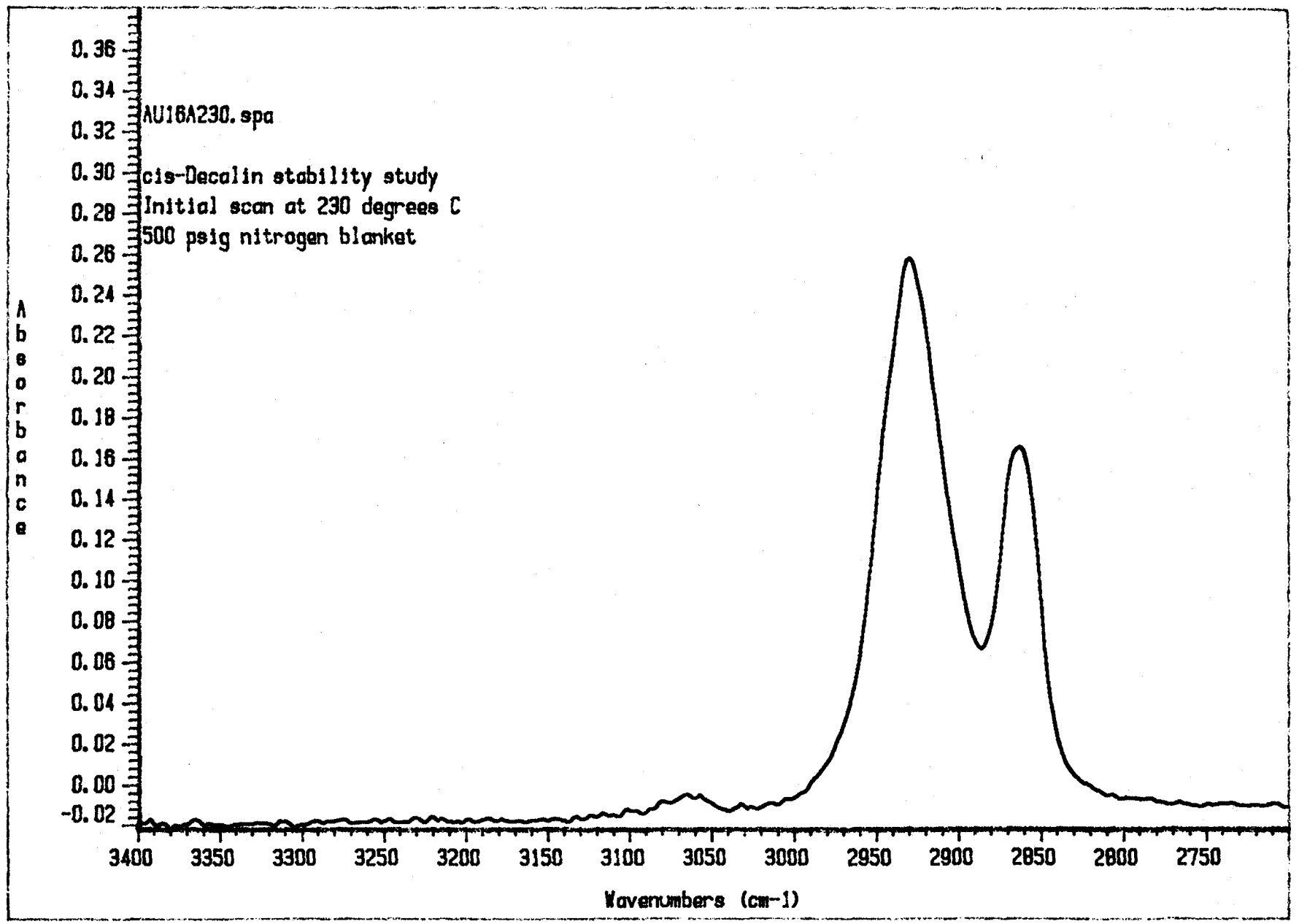

AU16A230.spa
cis-Decalin stability study
Initial scan at 230 degrees C
500 psig nitrogen blanket
Absorbance
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 31

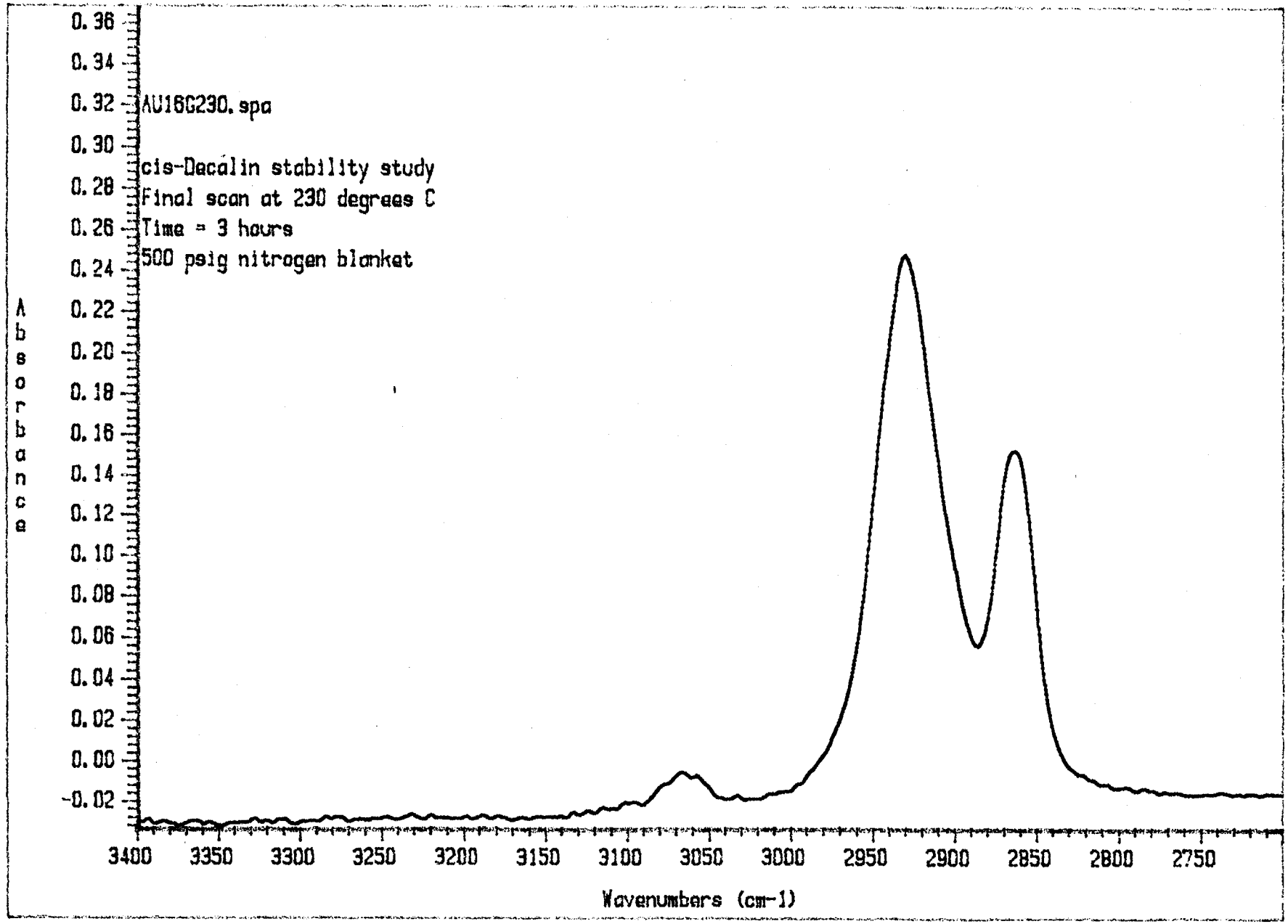

AU16G230.spa
cis-Decalin stability study
Final scan at 230 degrees C
Time = 3 hours
500 psig nitrogen blanket
Absorbance
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 32

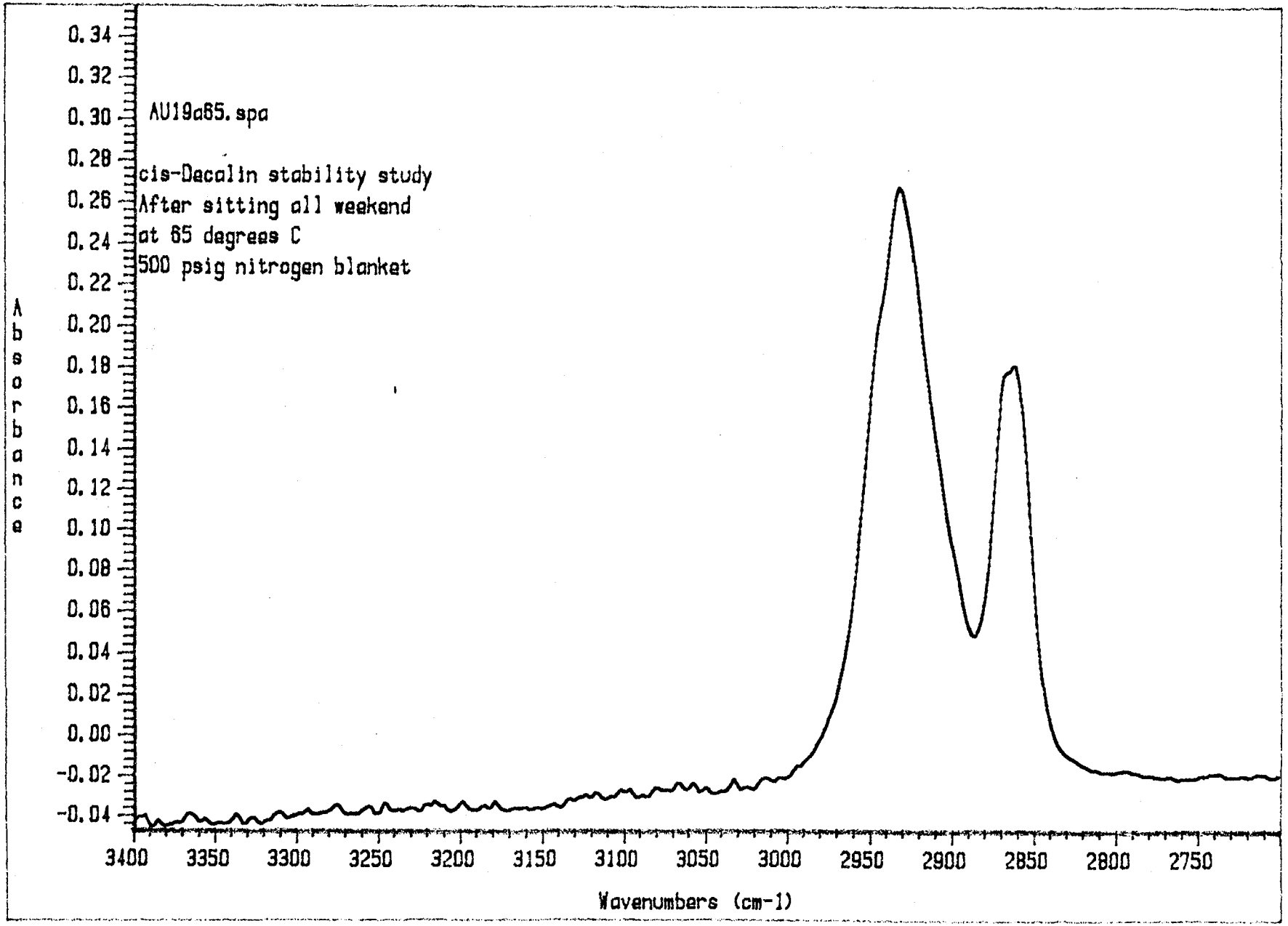
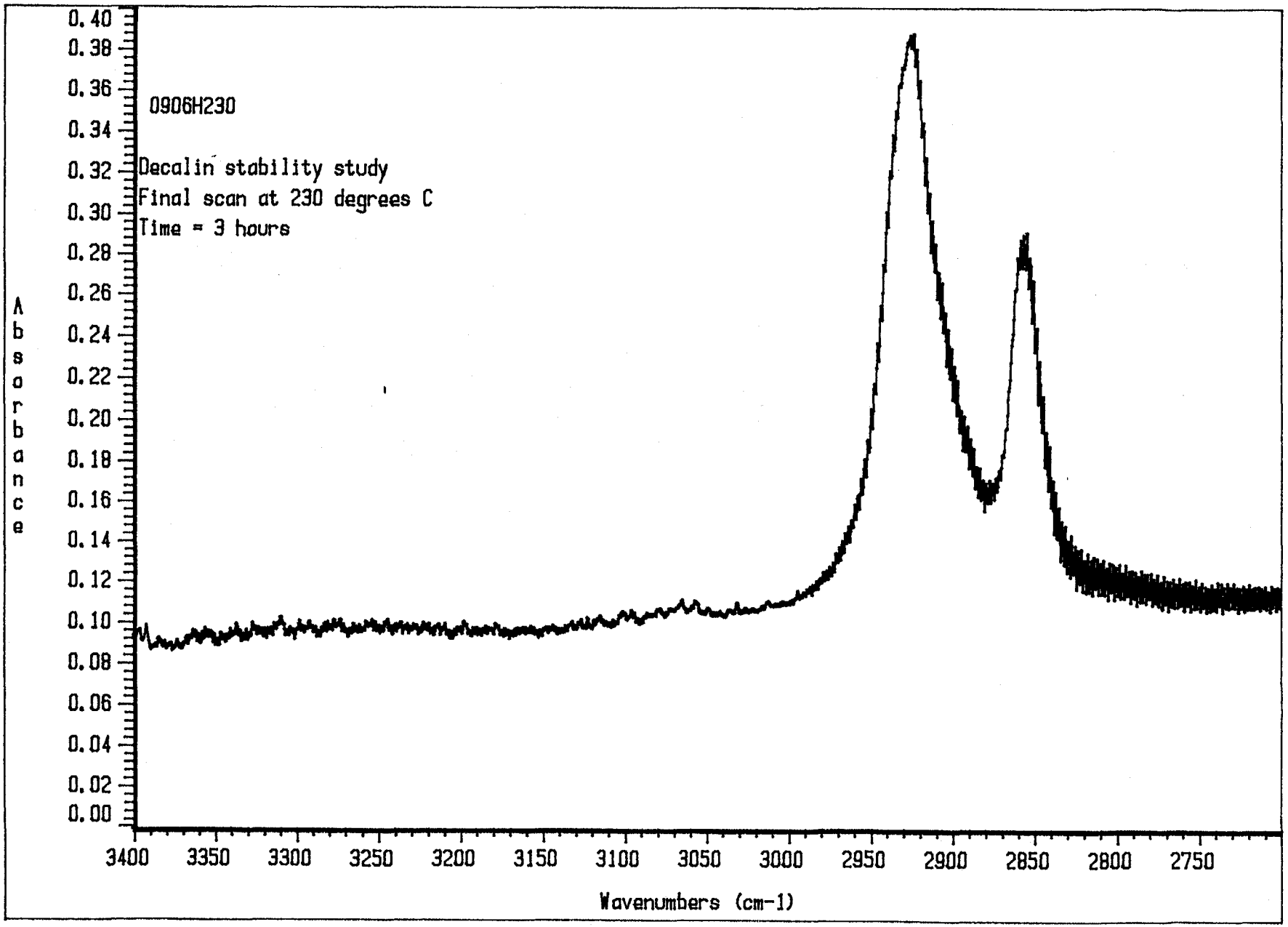

AU19a65.spa
cis-Decalin stability study
After sitting all weekend
at 65 degrees C
500 psig nitrogen blanket
Absorbance
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-0.04
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)

FIGURE 33


0906H230
Decalin stability study
Final scan at 230 degrees C
Time = 3 hours
Absorbance
0.40
0.38
0.36
0.34
0.32
0.30
0.28
0.26
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0.00
3400
3350
3300
3250
3200
3150
3100
3050
3000
2950
2900
2850
2800
2750
Wavenumbers (cm-1)