

**ON-LINE EMISSIONS MONITORING OF CHLOROBENZENE INCINERATION
USING FOURIER TRANSFORM INFRARED SPECTROSCOPY***

by

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ABSTRACT

Incineration of chlorobenzene in a small laboratory incinerator was monitored by using Fourier transform infrared spectroscopy (FTIR) coupled with a heated long-path cell (LPC) to analyze and quantify flue gas emissions in near real time. The effects of operating conditions under stable and decreasing incineration temperatures on the destruction of chlorobenzene were studied. The results from the decreasing temperature experiments were found to be consistent with those from experiments at stable temperatures. This finding demonstrates that the FTIR/LPC, as a continuous emissions monitor, can effectively detect dynamic changes in the incinerator emissions and can contribute significantly to the safety of incinerators.

INTRODUCTION

Incineration has generally become recognized as a preferred treatment technology in the management of hazardous wastes. However, public concern related to incinerator stack emissions has greatly impeded the needed buildup of incinerator capacity in the United States. Currently, the monitoring of incinerator emissions, if done at all, is done indirectly through the measurement of surrogates, such as carbon monoxide and total hydrocarbons. There is no on-stream method that can detect specific hazardous components in the emissions. Therefore, development of an on-stream continuous emissions monitor (CEM) that can differentiate species in the parts per million (ppm) and parts per billion (ppb) ranges would help to demonstrate the safety and reliability of incinerators.

Infrared absorption spectroscopy has long been used for molecular identification and quantification in a variety of chemical systems. Recent development of Fourier transform infrared spectroscopy (FTIR), combined with extensive use of computers, greatly expands its potential as a continuous monitoring system for incinerators. For example, Demirgian and Erickson (1) analyzed mixtures of simulated incinerator gases and actual stack gas samples with FTIR; they demonstrated the ability of the instrumentation to detect the principal organic hazardous constituent (POHC), chloroform, in a real stack gas sample. Hall et al. (2) reported that ethylene and vinyl chloride were detected by FTIR in the incineration of ethyl chloride in a turbulent-flow reactor, and problems related to the quantitative analysis with FTIR were discussed. Although these studies have shown that FTIR could be a good candidate for emissions monitoring, additional work is required to apply this technique to on-site continuous monitoring.

As a first stage in the development of such a CEM system, we have used FTIR to monitor the emissions from a small laboratory incinerator. This paper reports results of incineration of chlorobenzene. Methane, toluene, benzene, chlorobenzene, hydrogen chloride, and carbon monoxide in the flue gas were simultaneously analyzed. The temperature of the incinerator, the oxygen concentration in the secondary air, and the mean residence time were varied to simulate both normal and abnormal conditions in the incineration and to test the ability of the system to detect concentration changes in the emissions.

DESCRIPTION OF EXPERIMENT

LAB INCINERATOR

Figure 1 shows the experimental equipment setup for the laboratory incinerator and the FTIR/long-path cell (LPC) emissions monitor. The alumina-walled incinerator was

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3.81 cm in diameter and 64 cm in height. Fuel (usually methane) and primary air were premixed and injected through eight holes (0.12 cm in diameter) in the base plate. Secondary air was supplied through a 0.2-cm circumferential slot, 2.5 cm above the premixed fuel inlet holes. Chlorobenzene was injected with a micrometering pump through an injection probe located 2.5 cm above the burner holes. A tube evaporator heated to 250°C was used to evaporate chlorobenzene before it entered the probe. Electric heaters outside the incinerator were necessary to compensate for conduction heat loss. A thermocouple located at the secondary air inlet was used for monitoring flame temperature. Another thermocouple, 23 cm above the burner holes, was used to indicate the average incinerator temperature. The flue gas entered a quench section, where it was quenched to 250°C by Hastalloy coils cooled to 160°C. The injection probe and the burner base plate were cooled with Dowtherm-A, a heat-transfer fluid, which was circulated at 130°C. Flue gas filtered with glass wool passed through a length of glass tubing, and entered the LPC. The filter, tubing and LPC were

encased in metal sheathing (copper or aluminum), which was maintained at 150 to 180°C by electrical heating to prevent water from being condensed.

ANALYTICAL MEASUREMENTS

A Nicolet 8220 FTIR spectrometer coupled with Nicolet DX software was used for all analytical measurements. An MCT-A detector cooled with liquid nitrogen was used, and the resolution of the FTIR was 2 cm^{-1} . The FTIR was linked to a personal computer (PC), which used Nicolet PCIR version 3.0 software to perform a classical least-squares (CLS) analysis.

A custom-designed, heated (150°C) Hanst 4-22 long-path cell (Infrared Analysis, Anaheim, Calif.) was set at pathlength 15.4 m. The sample compartment of the FTIR spectrometer was modified to accept the heated cell. The cell was connected to a vacuum pump so that it could be evacuated and subsequently filled with calibration gases when the system was off-line.

Calibration gases were purchased from Scott Specialty Gases and were certified to be within 2% of the reported concentration. Data precision was determined by duplicating runs for each standard. The percent deviation was less than 1%, as was reported in a previous paper (3).

Linearity for chlorobenzene was determined by sequentially injecting liquid samples into the heated, evacuated cell. Then the cell pressure was raised to one atmosphere by adding nitrogen gas, and data were collected. The amount injected was converted to molar ppm based upon a 5-L cell volume. There are two major peaks in the infrared spectrum of chlorobenzene: 3123-2979 cm^{-1} and 1143-987 cm^{-1} . Peak areas for each were integrated and plotted versus the concentrations, as shown in Fig. 2. The datum at 45.7 ppm was obtained

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using a Scott-calibrated gas standard. A good linear relation between the absorbance peak area and the concentration for chlorobenzene in the concentration range of 0-420 ppm was observed.

Hydrogen chloride concentration was obtained using a 75-ppm gas standard. However, a quantitatively accurate reference spectrum could not be obtained. The HCl gas was both reacted with and adsorbed onto the cell and the transfer line. This became evident when spectral data were collected at 2 to 3 min intervals after the cell was filled with an HCl standard gas, and inlet and outlet stopcocks were closed. The spectral absorption of HCl continuously decreased after each data-collection interval, due to adsorption. Hence, the HCl concentrations presented in this paper, while internally consistent, cannot be considered quantitatively accurate.

Table I lists the detection limit for each target analyte in this study. The absorbance peak height for each compound with known concentration was determined from its standard,

and the peak-to-peak noise in the corresponding wave number region was determined by expanding a blank spectrum in that region. The detection limit was then calculated by assuming an acceptable signal-to-noise ratio of 4:1. The detection level for a given analyte is a function of its molar absorptivity and is limited by the noise and the pathlength of the system. Methane is a strong infrared absorber and can be detected at concentrations as low as 70 ppb. Other compounds, which have lower absorptivity, are detected in the ppm range. The detection limit can be further improved by either increasing the pathlength or reducing the noise.

RESULTS AND DISCUSSION

HOT CELL EFFECT

Most infrared spectra, especially commercially available spectra library, are taken at room temperature. Temperature effects on the spectra were examined, as shown in Fig. 3.

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Small molecules, such as carbon monoxide and hydrogen chloride, have a single major infrared absorption band that is an array of individual lines. As temperature increases from room temperature to 150°C, the major band range and the positions of individual lines are not changed. However, the height of side band lines relative to the central band lines increases as temperature increases, resulting in a slight change of the band shape, as shown in Figs. 3A and 3B. For large molecules, such as benzene and chlorobenzene, so many lines overlap each other that the spectral features are broad and smooth, except for occasional "spikes." Temperature change does not alter the global band range, but it affects details of the band shape, as shown in Figs. 3C and 3D. The band shape change for benzene was found to be quite significant.

This spectral change affects quantitative analysis, wherein a classical least-squares fit of the sample spectrum to the standard spectra of the target compounds is performed. If a sample spectrum taken at a 150°C cell temperature were analyzed with the standard spectra obtained at room temperature, the temperature effect on the spectra would lead to large fitting errors. Therefore, all standard spectra used for quantitative analysis were collected at 150°C.

WATER AND CARBON DIOXIDE INTERFERENCE

It is estimated that under the present experimental conditions, concentrations of water and carbon dioxide in the flue gas are about 11% and 6%, respectively. Figure 4 shows the infrared spectrum of the effluent gas during a clean burn of methane.

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The primary wave number regions for analysis in this study were 3300-2600 cm^{-1} , 2200-2000 cm^{-1} , and 1300-800 cm^{-1} , where water and carbon dioxide interferences are relatively minor. Two methods were used to reduce these interferences. In the first method, the sample spectrum was taken ratio to the background spectrum, which was collected when only air was flowing through the system. In the quantitative analysis, the spectra of water and CO_2 were added into the standards, so that water and CO_2 absorbance in these regions can be taken into account. In the second method, the background spectrum was taken during a clean burn of methane before injection of chlorobenzene. This background spectrum contained absorbance of water and CO_2 . The sample spectrum obtained after injection of chlorobenzene was taken ratio to the clean burn spectrum, so that most water and CO_2 bands were removed. A comparison of the two methods is given in Table II. The two methods gave consistent results. In the following experiments, method 1 was used for quantitative analysis of the selected target compounds.

MONITORING AT STABLE INCINERATION TEMPERATURES

The FTIR/LPC system was used to continuously monitor emissions from the laboratory incinerator when chlorobenzene was incinerated at various stable incineration temperatures. Four temperature levels (from 800 to 1100°C) and two excess oxygen ratios for each temperature level were selected for study.

Table III shows a continuous monitoring result for one set of conditions, in which chlorobenzene was completely destroyed. Before the injection of chlorobenzene, the incinerator was brought to the desired stable condition, and emissions from methane combustion were analyzed to ensure that combustion was complete. Then, chlorobenzene was injected at a constant rate of 0.0708 g/min by a micrometering pump. The injection of chlorobenzene caused only a slight disturbance of temperature, since the injection rate was low.

The first sample was taken 10 min after starting injection. Only benzene and hydrogen chloride were detected, at concentrations of 7.78 ± 3.0 ppm and 646.8 ± 17.1 ppm, respectively. Other target analytes were not detected. The following samples were taken at 10 min intervals and gave similar results, indicating a stable and complete destruction of chlorobenzene. The FTIR/LPC system was able to detect the emissions concentration changes after the injection of chlorobenzene.

Although chlorobenzene was completely destroyed at the stated conditions, another toxic component, benzene, was emitted at a significant concentration, 7.5-8.5 ppm. Hydrogen chloride, as a combustion product of chlorobenzene, was detected at concentrations of 580-850 ppm in the emissions. These values were significantly lower than those calculated when chlorobenzene was assumed to be totally converted to water, CO_2 , and HCl, as discussed earlier.

The monitoring was continued after the injection of chlorobenzene was stopped. Benzene and hydrogen chloride did not immediately disappear from the emissions, but their

concentrations gradually decreased. This could not be explained as a slow response of the FTIR/LPC system, since the response was proven to be fast, as shown in the beginning of the injection of chlorobenzene. An explanation could be that benzene and HCl, especially HCl, were adsorbed on the wall of the system (including incinerator, filter, transfer lines, and long-path cell). The adsorption of HCl caused HCl emissions concentrations to be systematically low in the incineration of chlorobenzene, and the desorption of HCl was responsible for the continuous HCl emissions after the injection of chlorobenzene was discontinued.

A similar phenomenon was reported by Mason et al. (4) during a boiler trial burn, in which stack concentrations of waste species continued for several hours after waste firing was completed. The mechanism of hysteresis has not been fully understood, and the FTIR/LPC system provides a useful tool to look into this problem.

EFFECT OF OPERATING CONDITIONS ON CHLOROBENZENE INCINERATION

Table IV summarizes results of the incineration of chlorobenzene in the lab incinerator at temperatures of 800-1100°C. As shown in Table IV, when the excess oxygen ratio was 1.63, chlorobenzene was detected in the flue gas at all temperature levels except 1100°C, indicating the incomplete destruction of chlorobenzene under these conditions. Although chlorobenzene was not found at 1100°C, benzene and carbon monoxide were still detected in the flue gas. The benzene concentration reached a maximum at around 900°C. This could correlate with a mechanism change in the destruction of chlorobenzene. As the temperature decreased, the destruction removal efficiency (DRE) of chlorobenzene decreased as well, resulting in more toxic emissions. Hydrogen chloride concentration was found in the range of 650-750 ppm for all temperatures, which was consistent with the fact that the chlorobenzene injection rate was similar in all runs. However, the measured value was significantly lower than expected, as discussed previously.

The mean residence time was kept unchanged when the excess oxygen ratio was increased from 1.63 to 2.65. This was accomplished by adding auxiliary pure oxygen into the secondary air stream while the total secondary air flow rate was maintained constant. The oxygen concentration in the inlet was increased from 19.8% to 32.1%. The increase of the excess oxygen ratio greatly improved the DRE of chlorobenzene. Even at temperatures as low as 900°C, chlorobenzene was still completely destroyed. Benzene was detected at all temperature levels, but at concentrations much lower than that in the previous set. The increase of the excess oxygen ratio also greatly reduced CO emissions.

Another experiment at 1100°C was conducted with an excess oxygen ratio of 1.54 and an inlet oxygen concentration of 30.5%. This condition was obtained by introducing pure oxygen into the secondary air stream while reducing the flow rate of secondary air by 60% to keep the excess oxygen ratio close to 1.63. The mean residence time was then increased from 1.06 to 1.70 s. No significant change in emission concentrations was found, except for CO and HCl. It was found, by comparing run 11-2 and run 11-3, that a decrease of excess air ratio caused a slight increase of CO emission. Oxygen concentration in the inlet was found to have more impact on the CO emission, comparing run 11-1 with run 11-3. The high

concentration of HCl in run 11-3 was believed to be caused by the decrease in total volumetric flow rate and the constant chlorobenzene injection rate.

Toluene concentration was reported as zero in all runs. Since the spectral absorbance of toluene overlaps that of benzene and chlorobenzene, toluene provided a test to see if the method would report false positive results. Relative to toluene, the method appears, in this sense, to be accurate.

Soot was found in the glass wool filter for all cases, except in run 11-2. Soot accumulated in the filter gradually increases the pressure drop and affects the pressure and the flow rate of the system. Therefore, continuous monitoring was limited to 1-2 h to avoid this problem. After each run, the filter was cleaned and the glass wool was replaced.

MONITORING UNDER DECREASING TEMPERATURE CONDITIONS

Table V shows the results of data collected every 10 min in a continuous run; the incinerator was first brought to its stable condition at 1100°C, and then the temperature was continuously decreased to 830°C over a period of 80 min while feed rates of air, methane, and chlorobenzene were maintained at constant values.

At temperatures above 1000°C, chlorobenzene was not detected in the emissions, but benzene and carbon monoxide were found at significant concentration levels. When the temperature decreased to below 1000°C, incomplete destruction of chlorobenzene was observed. A maximum of benzene concentration in the flue gas occurred around 950°C, showing that benzene is a very stable combustion by-product. Hydrogen chloride concentration in the emissions was found to be in the same range as that in the stable temperature case.

Figure 5 shows actual infrared spectra taken at different temperatures, along with the reference standards for methane, benzene, chlorobenzene, and hydrogen chloride in the

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wave number regions 3200-2600 cm^{-1} and 1220-850 cm^{-1} . By visual comparison of the sample spectra with the reference standards, hydrogen chloride bands can be seen in all cases. Also seen are chlorobenzene bands in the wave number region 1140-1000 cm^{-1} at temperatures of 913 and 833°C, respectively. Methane bands overlap with bands of hydrogen chloride, benzene, and chlorobenzene, but the major peak of methane can be found at 833°C. Benzene and chlorobenzene bands are highly overlapped with each other in the region 3140-3000 cm^{-1} and cannot be visually distinguished. Besides these bands, there are many other peaks, which are believed to be water bands. Nicolet software, which performs a least-squares fit of the sample spectrum to the reference spectra, provides quantitative analytical results, as shown in Table V.

The data obtained under decreasing temperatures were qualitatively consistent with the data at the corresponding stable temperatures. If the hysteresis of the system and uncontrolled factors in the incineration are considered, the consistency of the data is quite satisfactory. This demonstrates that the FTIR/LPC system as a continuous emissions monitor can effectively detect dynamic changes in the incinerator emissions.

BENZENE — AN IMPORTANT TARGET ANALYTE

Benzene was not present in the feedstock but was detected in all runs. Even when chlorobenzene was completely destroyed, benzene concentration in the flue gas was found to be as high as 10 ppm.

As a product of incomplete combustion (PIC), benzene could be formed from chlorobenzene under local oxygen-starved conditions through C-Cl bond rupture or displacement of a chlorine atom by a hydrogen atom. Once benzene is formed, it is more difficult to destroy than chlorobenzene, according to a study on thermal stability ranking of hazardous organic compounds (5). Therefore, the condition for complete destruction of chlorobenzene is not expected to completely destroy benzene.

Similar results were found in our previous study on toluene incineration (3) and in other studies. For example, Trenholm and Lee (6) listed benzene as one of the most frequently identified PICs in air emissions of hazardous waste incinerators. Tirey et al. (7) reported that benzene was identified as a good PIC surrogate when a mixture of six compounds, which were expected to be very difficult to destroy, was thermo-decomposed in the absence of oxygen.

Selection of target analytes for on-line emissions monitoring is an important but challenging task. The current work, along with other studies, suggests that benzene is an important analyte to be considered for continuous monitoring in full-scale incinerators.

CONCLUSIONS

The FTIR/LPC system has been successfully used to continuously monitor selected target analytes in emissions generated by the incineration of chlorobenzene. Benzene was found to be a major PIC and is suggested as a target analyte to be monitored in full-scale incinerators. Hysteresis of incineration was observed in this study. Increasing the oxygen concentration in the inlet was found to greatly reduce the temperature required for complete destruction of chlorobenzene.

Future work will be considered to increase the sensitivity of the system, to solve problems related to the measurement of hydrogen chloride, to incinerate more hazardous materials to determine target analytes for on-site continuous monitoring, and to demonstrate FTIR continuous monitoring in the field.

REFERENCES

1. J.C. DEMIRGIAN and M.D. ERICKSON, Waste Management, 10:227 (1990).
2. M.J. HALL, D. LUCAS, and C.P. KOSHLAND, Environ. Sci. Technol., 25:260 (1991).
3. J.C. DEMIRGIAN, Z. MAO, M.J. MCINTOSH, and C.A. WENTZ, Proceedings of the 1991 Incineration Conference, Knoxville, Tenn., May 1991.
4. H. MASON, J. NICHOLSON, R. DEROSIE, C. WOLBACH, and I. LICIS, Proceedings of the Fourteenth Annual Research Symposium, Cincinnati, Ohio: U.S. EPA Hazardous Waste Engineering Research Laboratory, EPA 600/9-88/021, July 1988.
5. P.H. TAYLOR, B. DELLINGER, and C.C. LEE, Environ. Sci. Technol., 24:316-328 (1990).
6. A. TRENHOLM and C.C. LEE, Proceedings of the Twelfth Annual Research Symposium, Cincinnati, Ohio: U.S. EPA Hazardous Waste Engineering Research Laboratory, EPA 600/9-86/022, August 1986.
7. D.A. TIREY, B. DELLINGER, P.H. TAYLOR, and C.C. LEE, Fifteenth Annual EPA Research Symposium on Land Disposal, Remedial Action, Incineration and Treatment of Hazardous Waste, Cincinnati, Ohio, April 1989.

TABLE I Detection Limit for Target Compounds (Cell: 150°C, 1 atm)

Compound	Concentration (ppm)	Absorbance (unit of peak height)	Peak Position (1/cm)	Noise Level	Detection Limit (ppm)
CO	42.8	0.0524	2176	0.0007	2.4
CH ₄	0.907	0.0625	3017	0.0012	0.07
HCl	75	0.0769	2963	0.0012	4.7
Benzene	47.6	0.0914	3057	0.0012	2.5
Toluene	44.2	0.0906	3042	0.0012	2.3
Chlorobenzene	49.8	0.1212	1091	0.0005	0.8

TABLE II Comparison of Two Methods for Reduction of Water Interference

Compound	Sample 1		Sample 2	
	Method 1	Method 2	Method 1	Method 2
Methane	3.07 ± 0.78	5.54 ± 0.74	6.95 ± 1.04	9.04 ± 1.09
Toluene	0	0	0	0
Benzene	167.9 ± 12	167.9 ± 10	156.4 ± 15	155.4 ± 15
Chlorobenzene	284.9 ± 13	289.7 ± 13	462.2 ± 22	466.6 ± 23
CO	516.7 ± 20	501.9 ± 18	612.7 ± 25	593.3 ± 23
HCl	706.3 ± 24	676.8 ± 25	699.5 ± 25	674.0 ± 29

TABLE III Continuous Monitoring Results in Steady State Experiment
(1000°C, excess oxygen ratio 2.64, mean residence time 1.13 s)

Time (min)	CH ₄	Toluene	Benzene	Chloro- benzene	CO	HCl
10	0	0	7.78 ± 3.0	0	0	646.8 ± 17
20	0	0	8.25 ± 3.3	0	0	862.9 ± 23
30	0	0	7.59 ± 3.0	0	0	651.2 ± 17
40	0	0	8.38 ± 3.0	0	21.7 ± 6.9	585.6 ± 16
50	0	0	7.81 ± 3.0	0	0	588.7 ± 16
60	0	0	7.78 ± 3.1	0	0	648.0 ± 17
65			(Stop injection of chlorobenzene)			
75	0	0	4.20 ± 2.7	0	0	132.2 ± 8
100	0	0	2.92 ± 2.7	0	0	35.4 ± 7

TABLE IV Data for Destruction of Chlorobenzene at Various Conditions

Item	Value at 1100°C			Value at 1000°C	
	Run 11-1	Run 11-2	Run 11-3	Run 10-1	Run 10-2
Gaseous feeds (cm³/min)					
Primary air	1200	1200	1200	1200	1200
Secondary air	6324	5162	2600	6324	5162
Auxiliary oxygen	0	1249	718	0	1249
Methane	436	436	436	436	436
Liquid feed, chloro- benzene (g/min)	0.0631	0.0692	0.0740	0.0680	0.0708
Excess oxygen ratio	1.64	2.65	1.54	1.62	2.64
Residence time (s)	1.06	1.05	1.70	1.14	1.13
Inlet concentration					
Oxygen (%)	19.8	32.1	30.5	19.8	32.1
Methane (%)	5.47	5.41	8.7	5.41	5.41
Chlorobenzene (ppm)	1689	1833	3180	1821	1875
Outlet concentration (ppm)					
Methane	0	0	0	0	0
Toluene	0	0	0	0	0
Benzene	10.2 ± 3.1	7.2 ± 3.0	11.7 ± 4.4	50.9 ± 3.6	7.9 ± 3.0
Chlorobenzene	0	0	0	2.5 ± 1.0	0
Carbon monoxide	87.5 ± 7.8	0	12.8 ± 3.3	215 ± 9.5	3.6 ± 1.2
Hydrogen chloride	674 ± 18	695 ± 8	988 ± 33	691 ± 18	664 ± 18
Soot observed	Yes	No	Little	Yes	Yes
DRE of chlorobenzene (%)	>99.95	>99.95	>99.97	99.86	>99.95

TABLE IV (Cont.)

Item	Value at 900°C		Value at 800°C	
	Run 9-1	Run 9-2	Run 8-1	Run 8-2
Gaseous feeds (cm ³ /min)				
Primary air	1200	1200	1200	1200
Secondary air	6324	5162	6324	5162
Auxiliary oxygen	0	1249	0	1249
Methane	436	436	436	436
Liquid feed, chloro- benzene (g/min)	0.0659	0.0723	0.0669	0.0680
Excess oxygen ratio	1.63	2.64	1.63	2.66
Residence time (s)	1.24	1.23	1.36	1.34
Inlet concentration				
Oxygen (%)	19.8	32.1	19.8	32.1
Methane (%)	5.41	5.41	5.41	5.41
Chlorobenzene (ppm)	1764	1915	1792	1801
Outlet concentration (ppm)				
Methane	2.43 ± 0.7	0	1.0 ± 0.3	0
Toluene	0	0	0	0
Benzene	125.2 ± 9.0	7.11 ± 2.9	41.1 ± 5.0	15.3 ± 3.6
Chlorobenzene	194.6 ± 9	0	135.1 ± 5.8	18.7 ± 2.2
Carbon monoxide	482.9 ± 1	33.1 ± 6.8	393.1 ± 14	216.7 ± 11
Hydrogen chloride	765 ± 25	658 ± 17	665 ± 2	755 ± 21
Soot observed	Yes	Yes	Yes	Yes
DRE of chlorobenzene (%)	88.97	>99.95	92.46	98.96

TABLE V Continuous Monitoring Results in Nonsteady-State Experiment (Excess oxygen ratio 1.62, chlorobenzene injection rate 0.0695 g/min)

Time (min)	T (°C)	Outlet Concentration (ppm)					DRE of Chloro- Benzene (%)	
		CH ₄	Benzene	Chloro- benzene	CO	HCl		
10	1101	0	9.08 ± 2.8	0	46.4 ± 6.6	573.2 ± 15	>99.95	
20	1104	0	10.1 ± 2.9	0	77.0 ± 7.3	708.6 ± 18	>99.95	
30	1069	0	10.0 ± 2.9	0	15.0 ± 6.5	623.0 ± 16	>99.95	
40	1008	0	27.9 ± 3.0	0	162.6 ± 8	611.2 ± 16	>99.95	
50	958	3.2 ± 0.7	196.3 ± 8.2	63.5 ± 5.2	438.2 ± 17	774.6 ± 25	96.6	
60	913	1.4 ± 0.5	101.7 ± 5.8	93.0 ± 5.3	372.9 ± 14	641.0 ± 20	95.0	
70	862	2.0 ± 0.6	71.5 ± 5.6	111.5 ± 6	400.1 ± 15	614.4 ± 19	94.0	
80	883	7.0 ± 1.0	156.4 ± 15	462.2 ± 22	612.7 ± 25	699.5 ± 26	75.2	
82			(Stop injection of chlorobenzene)					
97	776	0	8.4 ± 2.9	0	12.5 ± 6.6	64.8 ± 7.8		

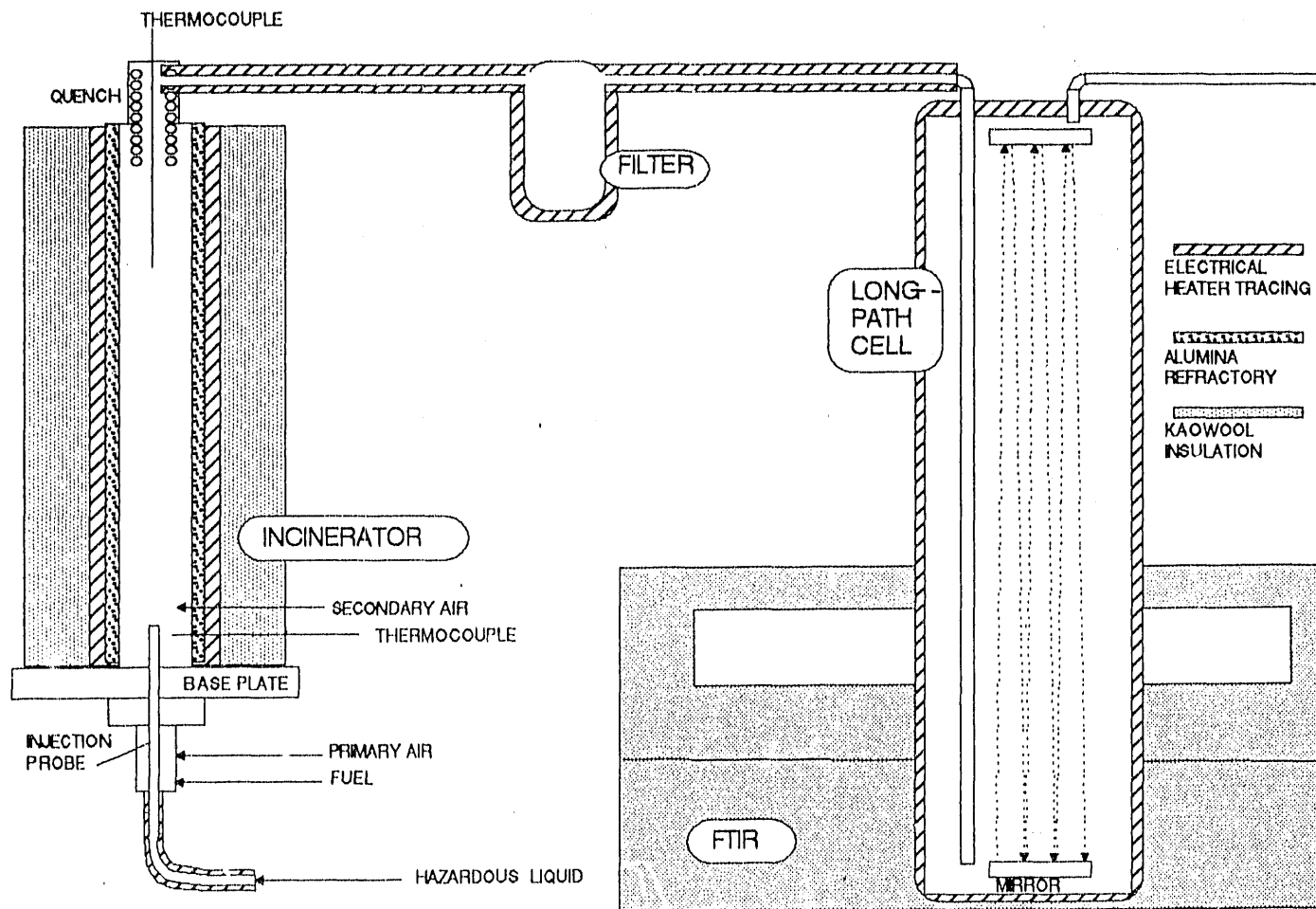


Figure 1. Laboratory incinerator with the FTIR/LPC emissions monitor

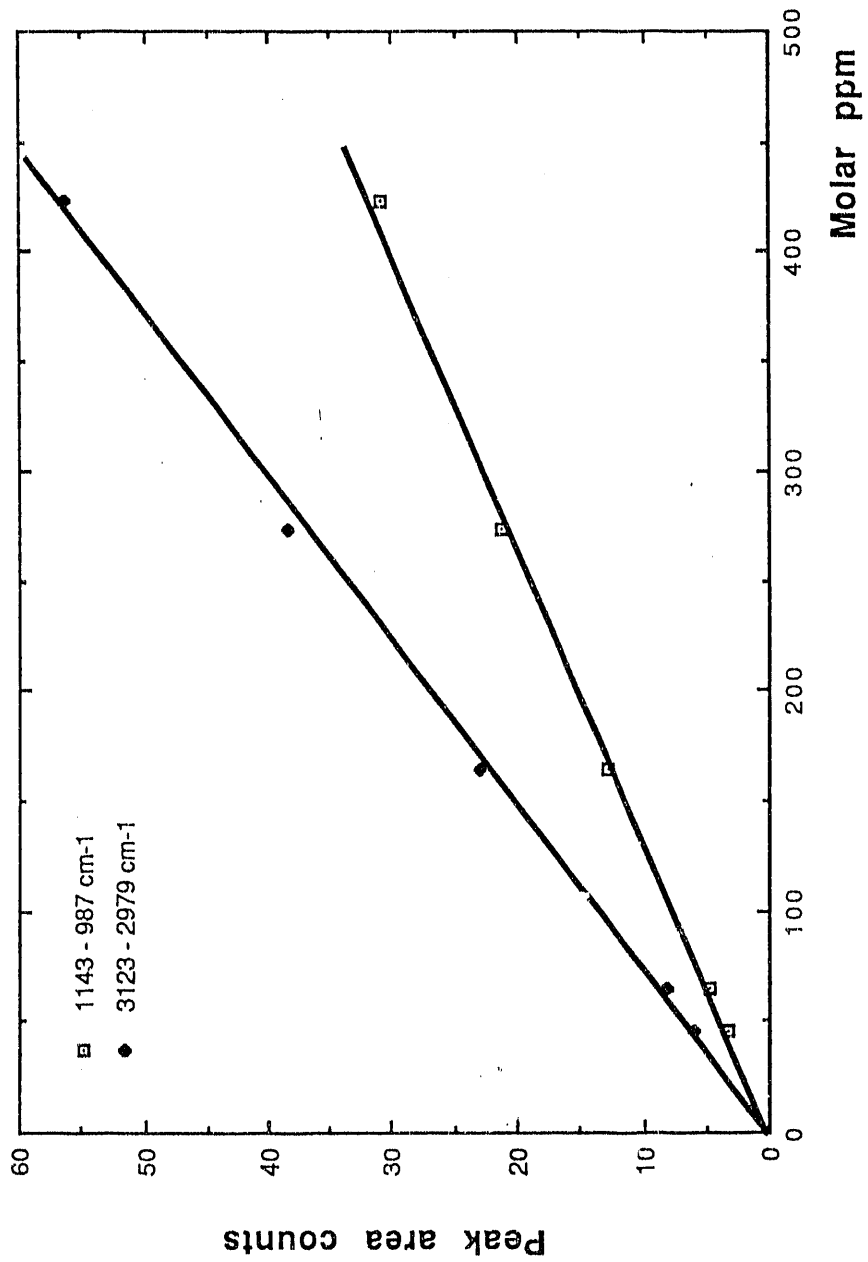


Figure 2 Calibration for Chlorobenzene

Figure 3A. Temperature Effect on CO Spectrum

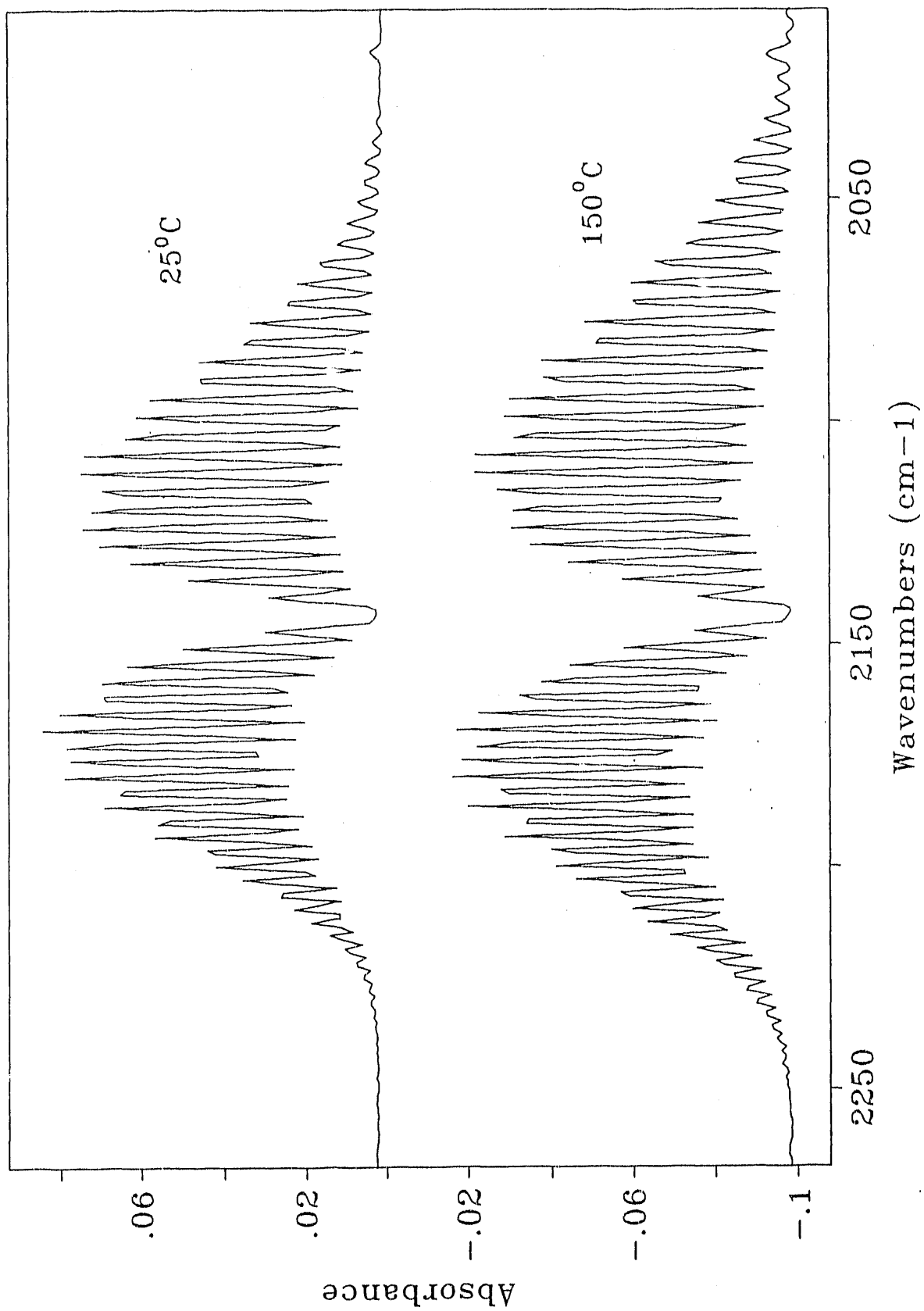


Figure 3B. Temperature Effect on HCl Spectrum

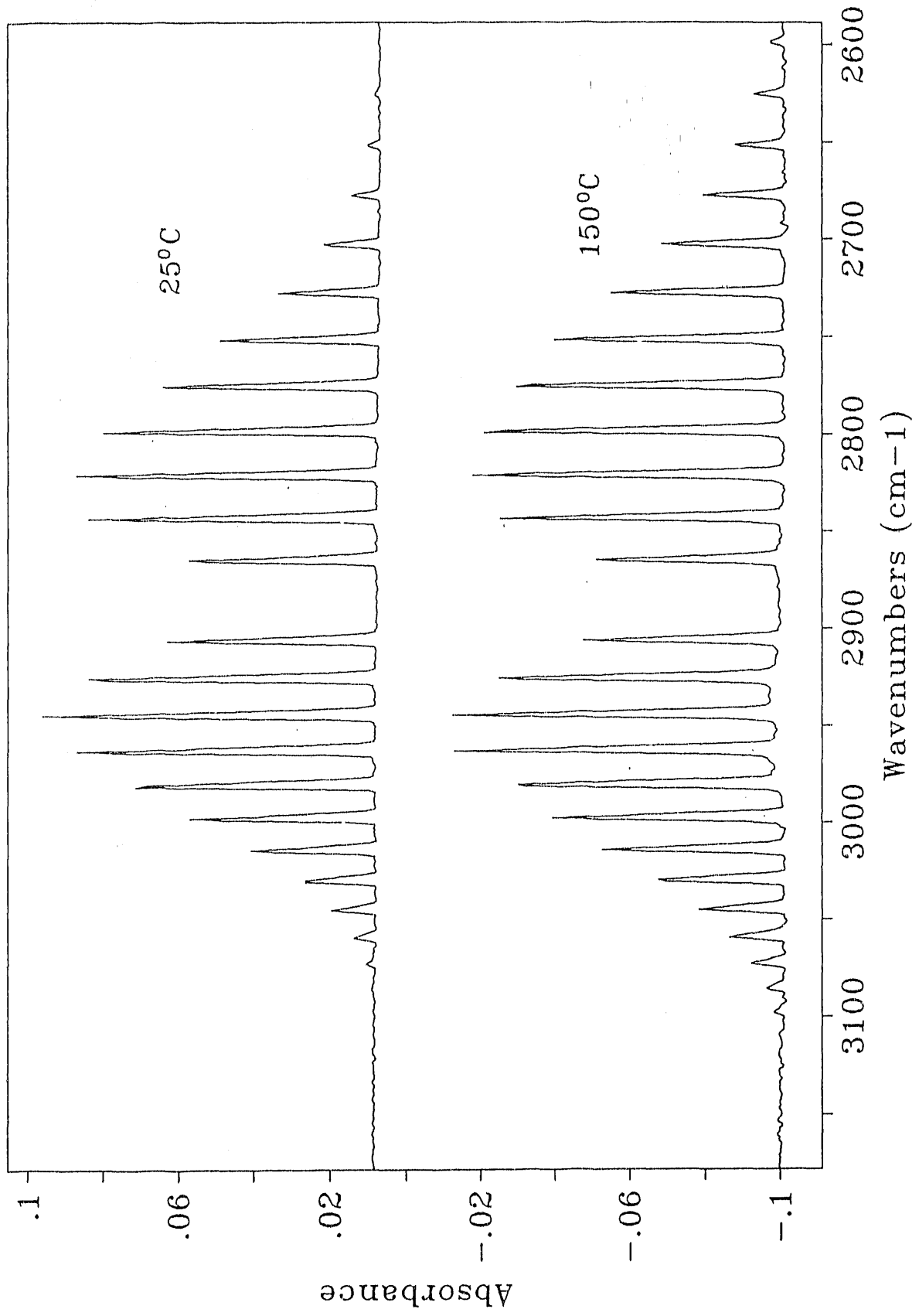


Figure 3C. Temperature Effect on Benzene Spectrum

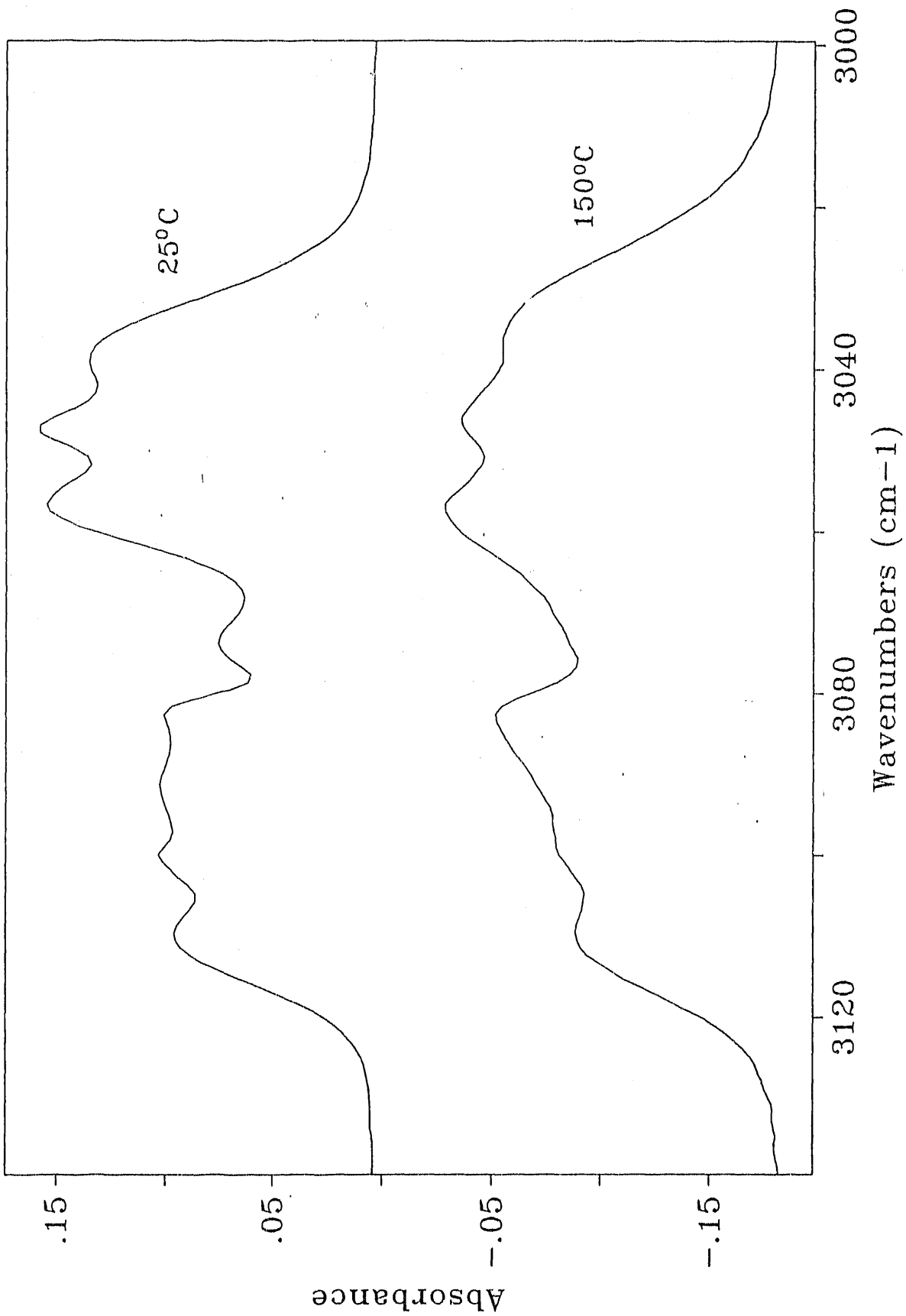


Figure 3D. Temperature Effect on Chlorobenzene Spectrum

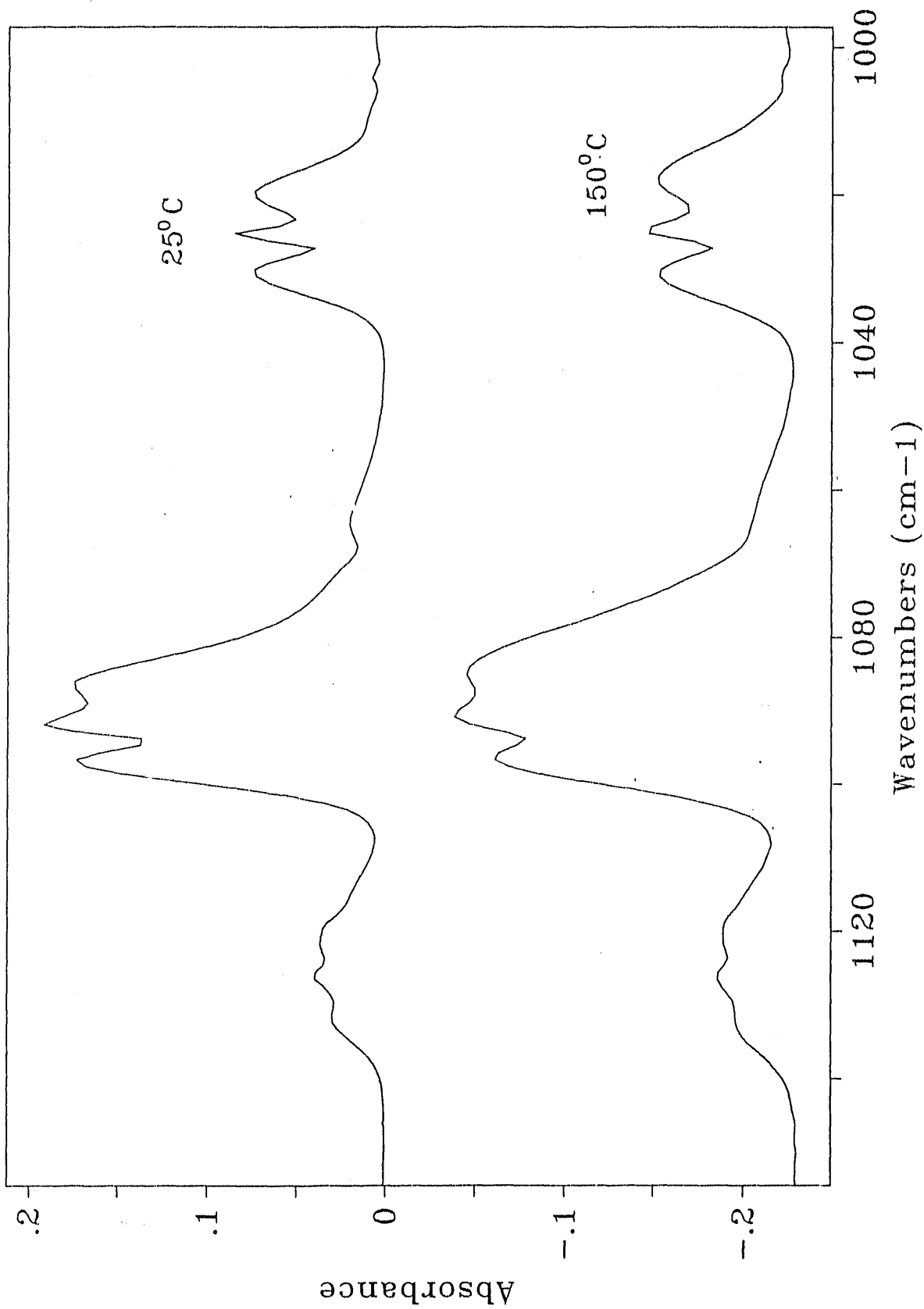


Figure 4. Background Infrared Spectrum of Clean Methane Burn

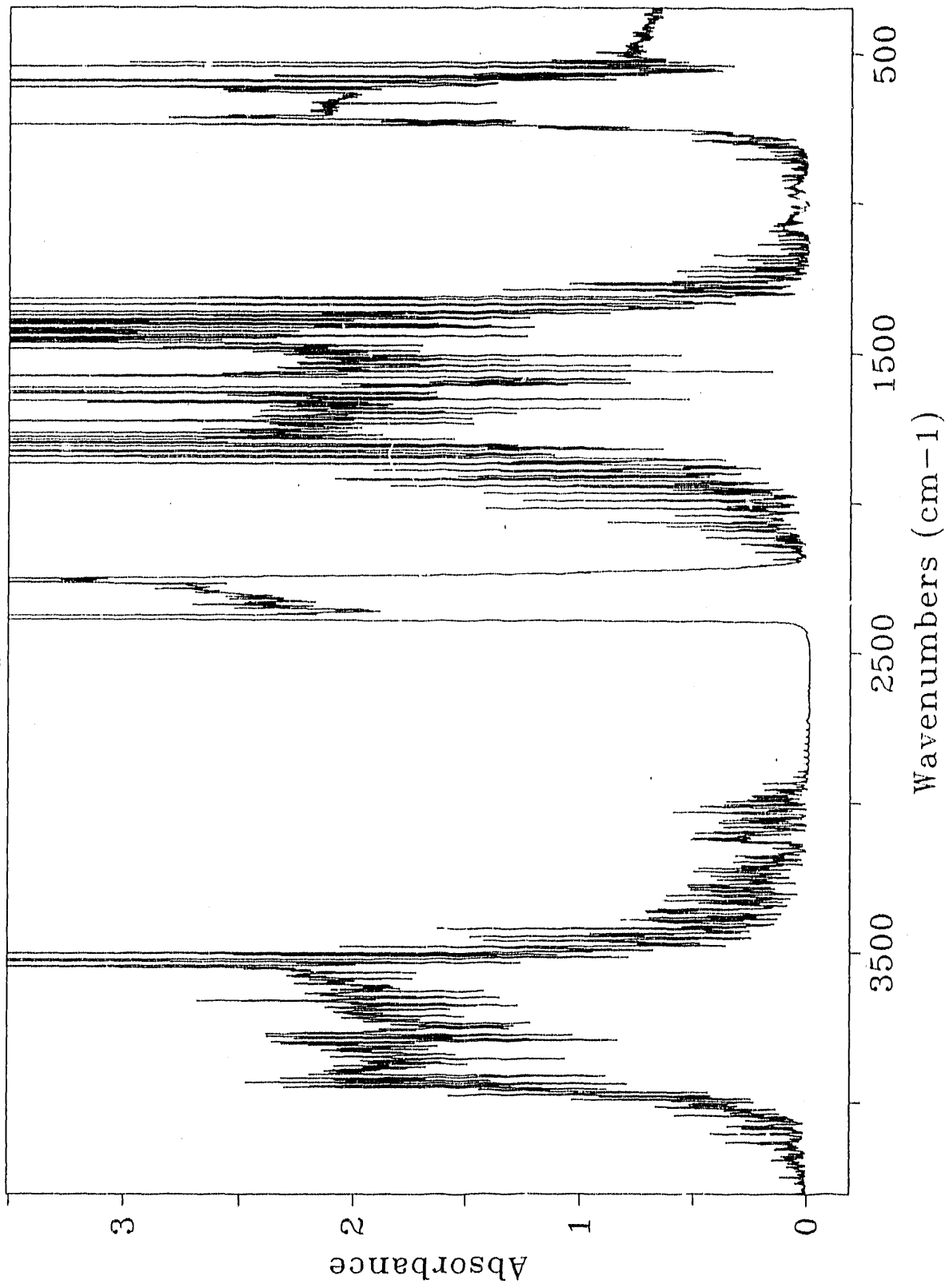


Figure 5a Infrared Spectra Taken During a Period of Temperature Decrease

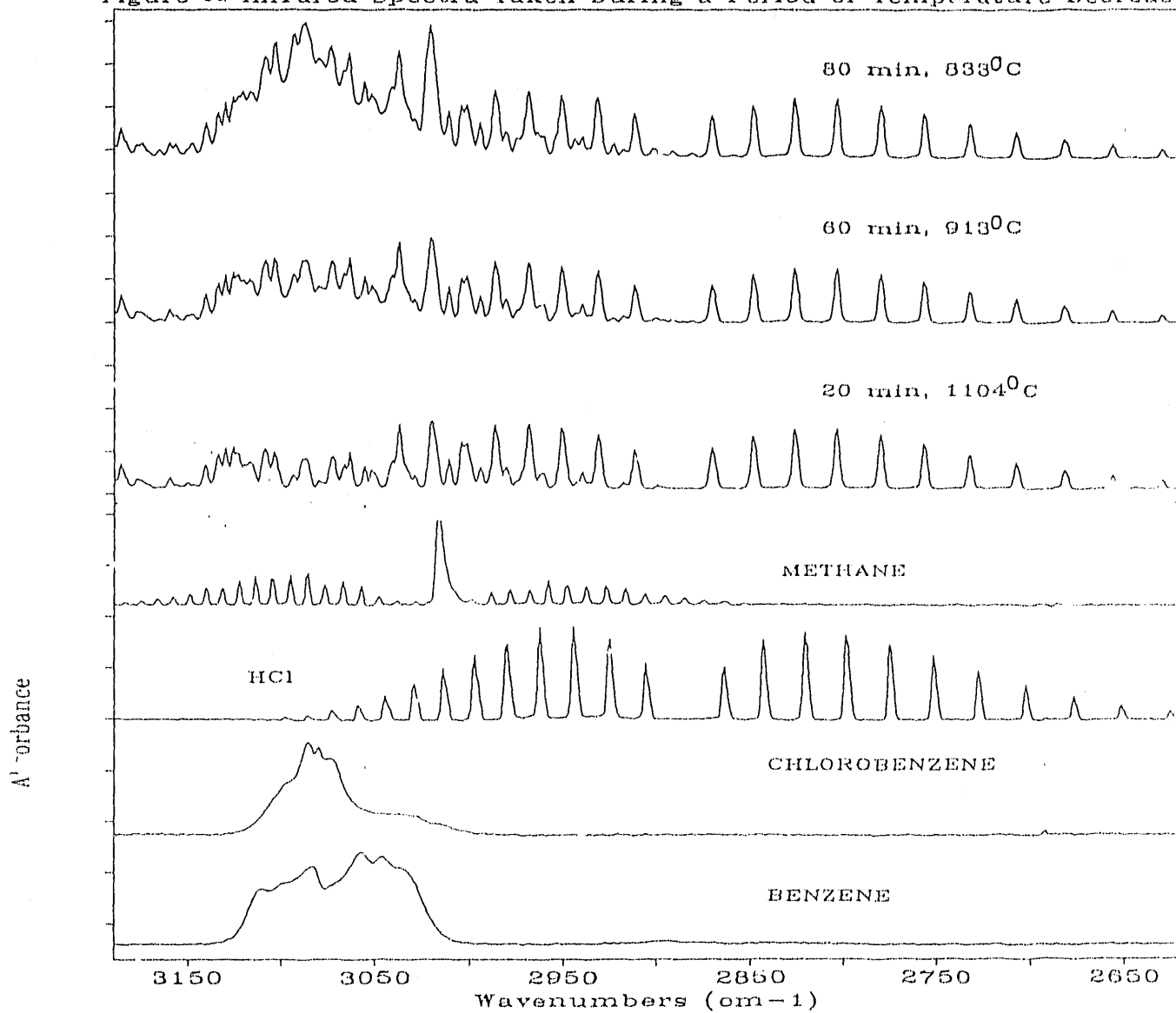
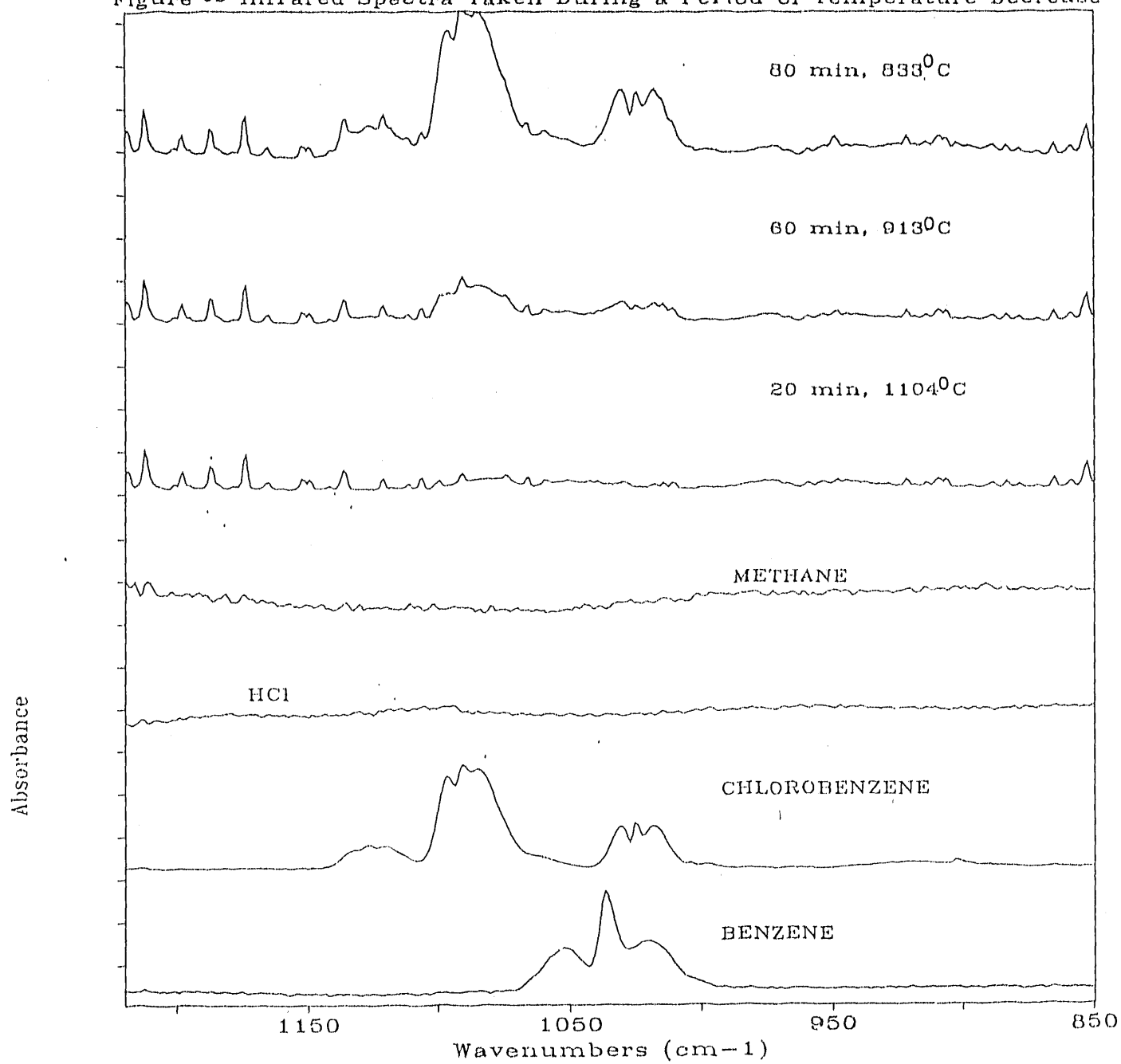


Figure 5b Infrared Spectra Taken During a Period of Temperature Decrease



END

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6/16/92