



**EDGEWOOD**

**CHEMICAL BIOLOGICAL CENTER**

**U.S. ARMY SOLDIER AND BIOLOGICAL CHEMICAL COMMAND**

**ECBC-TR-297**

**QUANTITATIVE INFRARED REFERENCE LIBRARY  
VOLUME I**

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**EAI CORPORATION**  
*A Registered ISO-9001 Company*

**July 2003**

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13. ABSTRACT (Maximum 200 words) The U. S. Army's Chemical Biological Applications and Risk Reduction Business Unit (CBARR) at Aberdeen Proving Ground, MD, is actively involved in Chemical Warfare Materiel (CWM) air monitoring and sample analysis for a variety of missions. These include CWM investigations, remediation of chemical disposal sites, and environmental assessments. To enhance CBARR's ability to accomplish these tasks, the organization is continually examining and evaluating new technologies in this arena. The instrument of interest for this study was the TravellIR by Sensir, Inc. It is a lightweight, portable, closed cell Fourier Transform Infrared (FTIR) Spectrometer designed to measure and collect radiation in the mid-infrared band. Infrared detection is performed with a DTGS detector that operates at room temperature. The gas cell used has a 2.4-m path length, L. Data was acquired by co-adding 64 spectra at 2 cm <sup>-1</sup> resolution. The experiment was run with a series of quantitative concentrations of analytes, to include sarin (GB), distilled mustard (HD), tabun (GA), and soman (GD).				
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## PREFACE

The work described in this report was authorized under Project No. 2VJRZZ. The work was started in February 2002 and completed in June 2002.

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# QUANTITATIVE INFRARED REFERENCE LIBRARY VOLUME I

## 1. INTRODUCTION

Recent technology developments in Fourier Transform Infrared (FTIR) spectroscopy, such as stand-off infrared detection of chemical agents and related compounds for monitoring applications, has created a need for condensed and vapor phase FTIR databases which utilize current technology. Old infrared databases are obsolete as they were collected on first generation instruments with poor signal-to-noise characteristics arising from a variety of instrumental variations (such as noisy A/D converters, wavelength calibration problems, etc.) relative to the capabilities of present day FTIR instrumentation. In addition, commercial infrared spectra compilations do not adequately address militarily significant, or directly related, compounds.

## 2. OBJECTIVE

The objective of this effort is to create, validate, and maintain a vapor phase FTIR database, with a focus on compounds of military significance or potential terrorist threat, using instrumentation that reflects current analytical capabilities.

## 3. DESCRIPTION

A series of quantitative concentrations of the analytes were established with a Kin-Tek model 491-M gas/vapor generation system. The Kin-Tek generator uses chemical-filled ¼" o.d. polytetrafluoroethylene permeation tubes contained in a glass holder to establish continuous streams of chemical compounds. The glass permeation tube holder sits in a heated oven block, regulated by a digital temperature controller. Balance gas, regulated by digital mass flow controllers, is supplied to the vapor stream at two points: the permeation tube holder and downstream of the permeation tubes to provide additional diluent. The output concentration of the device can be varied by either changing the temperature of the oven block or the flow rate of the diluent gas. For the series of experiments in this paper, dry nitrogen, supplied by boiloff from a 230-L liquid nitrogen Dewar, was used.

The rate of mass flow from permeation tubes can be determined gravimetrically, by weighing the tubes before and after an operation. Because of the low volatilities of the compounds in these tests, this would have required potentially several weeks for each agent in order to accurately measure the mass loss. Furthermore, chemical agents tend to be chemically "sticky" and can adsorb to tubing walls and fittings. For this reason, the mass rate was determined with a secondary method. Using a mass flow controller, a measured volume of the effluent was drawn onto a sorbent tube filled with Tenax-TA for later desorption and analysis by gas chromatography. Analysis of the tubes was performed on an Agilent 5890A gas

chromatograph (GC) equipped with a Dynatherm thermal desorption apparatus and a flame ionization detector. The instrument was calibrated prior to each operation with external standards, prepared by serial dilution from the neat material in hexane, which were injected onto a sorbent tube and then desorbed into the GC. Six tubes were collected for each operation (agent). A statistical analysis of the data from the sorbent tubes showed typical RSD's of better than 5%.

Transfer lines between the generator and the FTIR gas cell were plumbed with ¼" o.d. Silicosteel® from Restek Corporation. This material minimizes adsorption by a wide variety of chemical compounds, enabling a more rapid stabilization of the concentration of the analyte in the gas cell.

Reference spectra of the effluent from the generator were acquired with a Protégé model Fourier transform infrared (FTIR) spectrometer from Thermo-Nicolet. The spectrometer is equipped with a mercury-cadmium-telluride (MCT) detector operating at cryogenic temperatures (achieved by filling the detector with liquid nitrogen prior to operation). The spectrometer is also outfitted with a 10-meter fixed pathlength gas cell from Thermo-Spectratech. Spectral conditions were 0.5 cm<sup>-1</sup> resolution, 3-term Blackman-Harris apodization, and co-adding of 64 scans to achieve the final spectra. Prior to any change in concentration, a fresh background (single beam) spectrum of the dry nitrogen was collected. As a quality check, a statistical analysis of the data was made, using peak heights to fit several representative bands at the vapor concentrations analyzed to a least squares fit, in order to determine compliance with Beer's Law.

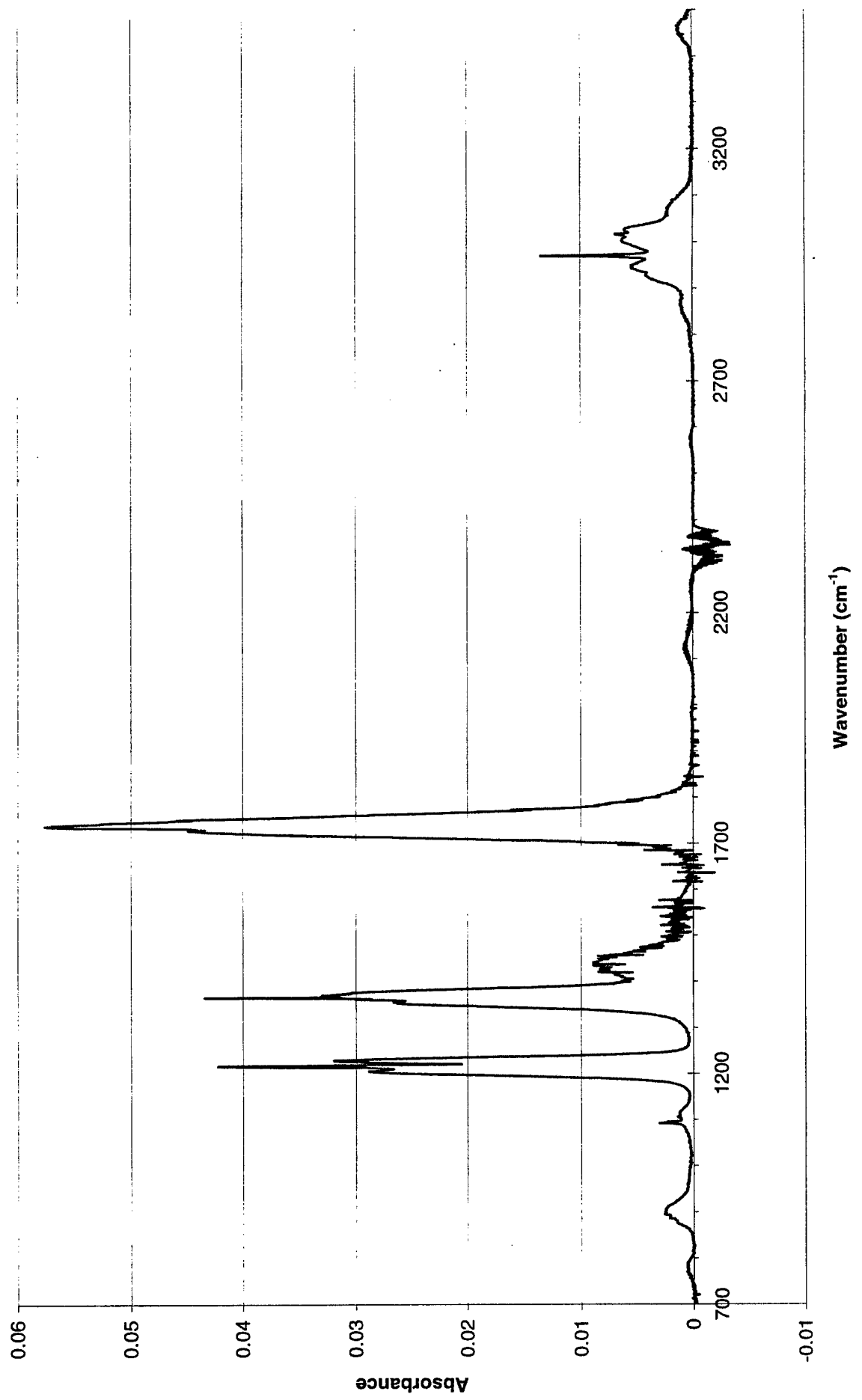
APPENDIX  
EDGEWOOD CHEMICAL BIOLOGICAL CENTER  
QIRL DATABASE

**Edgewood Chemical Biological Center  
QIRL Database**

<b>QIRL Filename</b>	<b>Compound Name</b>	<b>CAS</b>	<b>Molecular Formula</b>	<b>CL (ppm-m)</b>
QIRL0010	Acetone	67-64-1	C <sub>3</sub> H <sub>6</sub> O	100
QIRL0020	2-Propanol (isopropyl alcohol)	67-63-0	C <sub>3</sub> H <sub>8</sub> O	100
QIRL0030	Dimethyl methylphosphonate (DMMP)	756-79-6	C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P	49
QIRL0040	Diethyl methylphosphonate (DEMP)	683-08-9	C <sub>5</sub> H <sub>13</sub> O <sub>3</sub> P	38
QIRL0050	Triethyl phosphate (TEP)	78-40-0	C <sub>6</sub> H <sub>15</sub> O <sub>4</sub> P	18
QIRL0060	Diisopropyl methylphosphonate (DIMP)	1445-75-6	C <sub>7</sub> H <sub>17</sub> O <sub>3</sub> P	24
QIRL0070	Methyl salicylate	119-36-8	C <sub>8</sub> H <sub>8</sub> O <sub>3</sub>	171
QIRL0080	Triethyl phosphite	122-52-1	C <sub>6</sub> H <sub>15</sub> O <sub>3</sub> P	84
QIRL0090	Diethyl phosphite	762-04-9	C <sub>4</sub> H <sub>11</sub> O <sub>3</sub> P	72
QIRL0100	Monoethanolamine (MEA)	141-43-5	C <sub>2</sub> H <sub>7</sub> NO	92
QIRL0110	Trimethyl phosphite	121-45-9	C <sub>3</sub> H <sub>9</sub> O <sub>3</sub> P	93
QIRL0120	Dimethyl phosphite	868-85-9	C <sub>2</sub> H <sub>7</sub> O <sub>3</sub> P	80
QIRL0130	Diethyl malonate (DEM)	105-53-3	C <sub>7</sub> H <sub>12</sub> O <sub>4</sub>	103
QIRL0140	Trimethyl phosphate (TMP)	512-56-1	C <sub>3</sub> H <sub>9</sub> O <sub>4</sub> P	97
QIRL0150	1,4 Thioxane	15980-15-1	C <sub>4</sub> H <sub>8</sub> OS	147
QIRL0160	2-Chloroethyl ethyl sulfide (CEES)	693-07-2	C <sub>4</sub> H <sub>9</sub> ClS	98
QIRL0170	n-Butyl mercaptan (BUSH)	109-79-5	C <sub>4</sub> H <sub>10</sub> S	156
QIRL0180	2-(Diisopropylamino) ethanol	96-80-0	[(CH <sub>3</sub> ) <sub>2</sub> CH] <sub>2</sub> NCH <sub>2</sub> CH <sub>2</sub> OH	97
QIRL0190	O,O-Diethyl methylphosphonothionate	6996-81-2	(CH <sub>3</sub> CH <sub>2</sub> O) <sub>2</sub> P(S)CH <sub>3</sub>	25
QIRL0200	O,S-Diethyl methylphosphonothionate	2511-10-6	(CH <sub>3</sub> CH <sub>2</sub> O)(CH <sub>3</sub> CH <sub>2</sub> S) <sub>2</sub> P(O)CH <sub>3</sub>	74

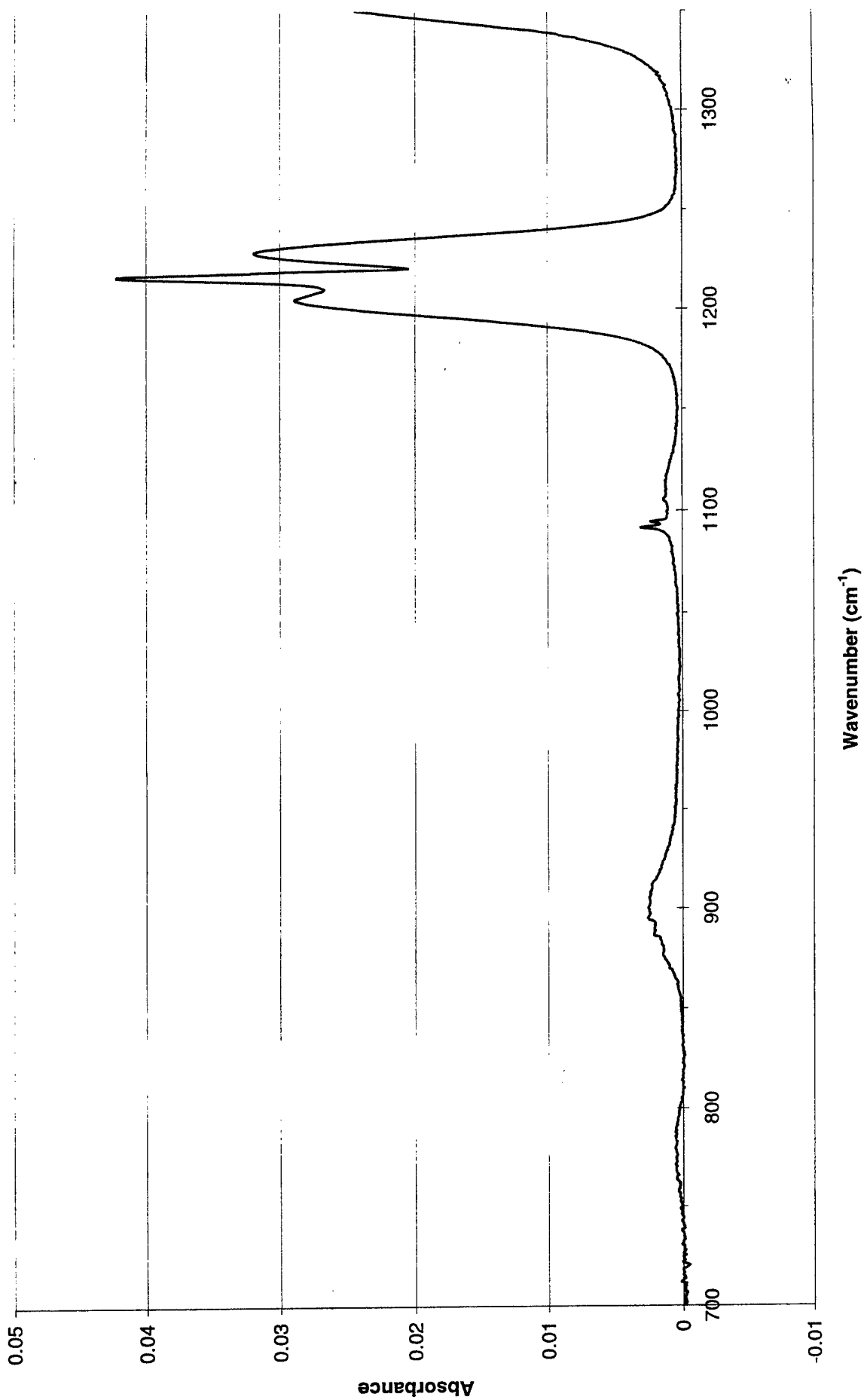


**QIRL0010 - Acetone**  
100 ppm- $\mu$ m



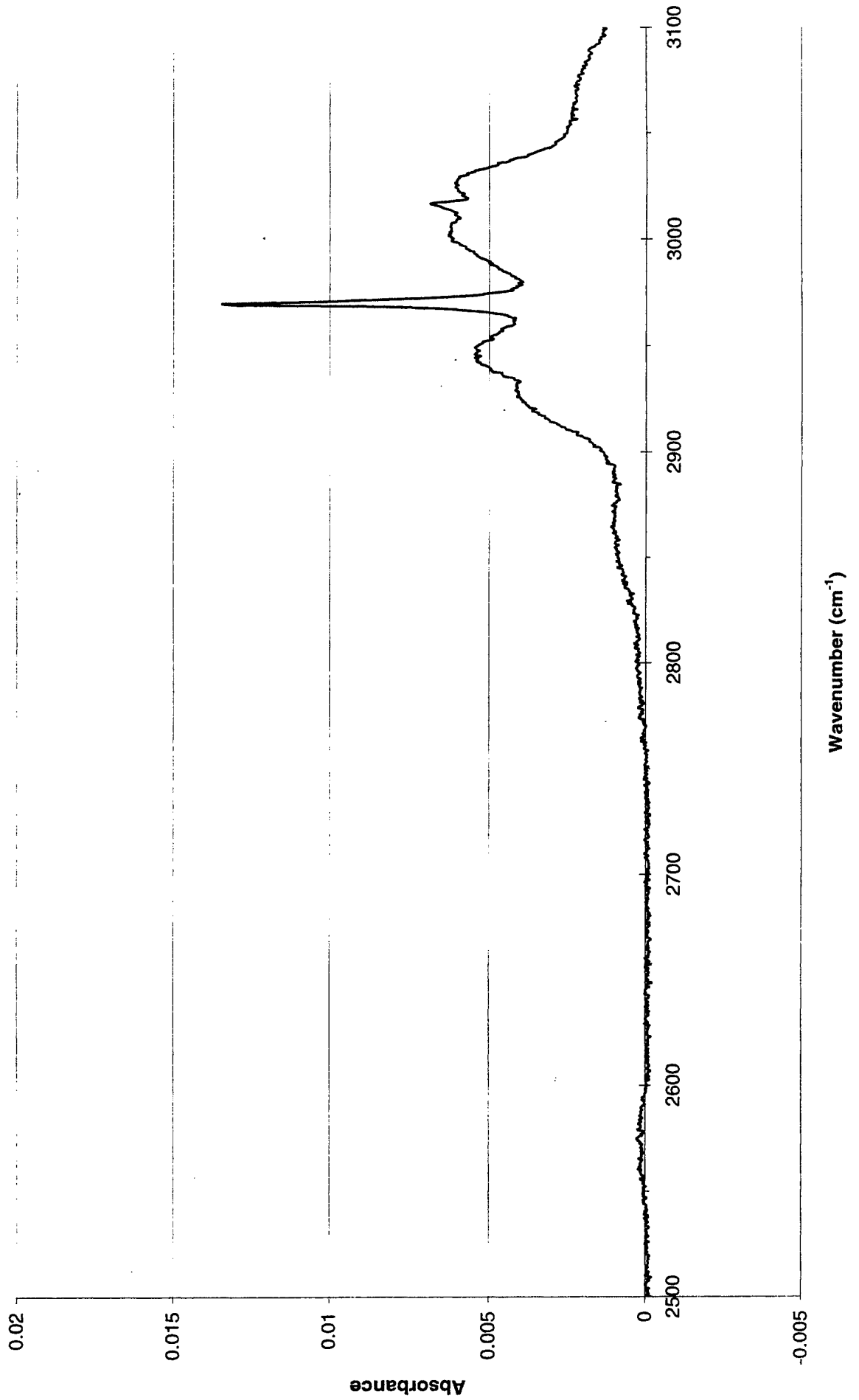


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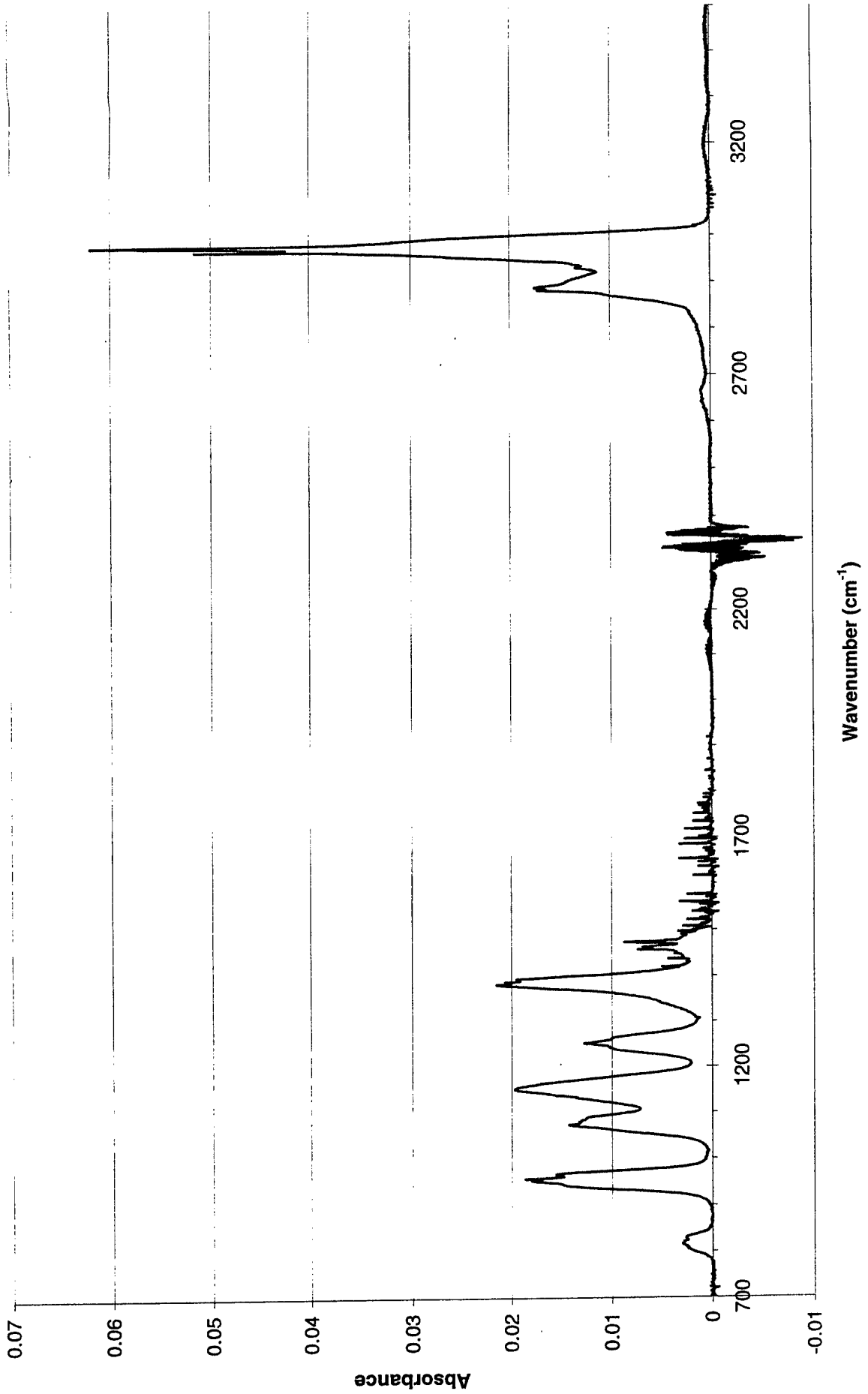


**QIRL0010 - Acetone**  
100 ppm-m



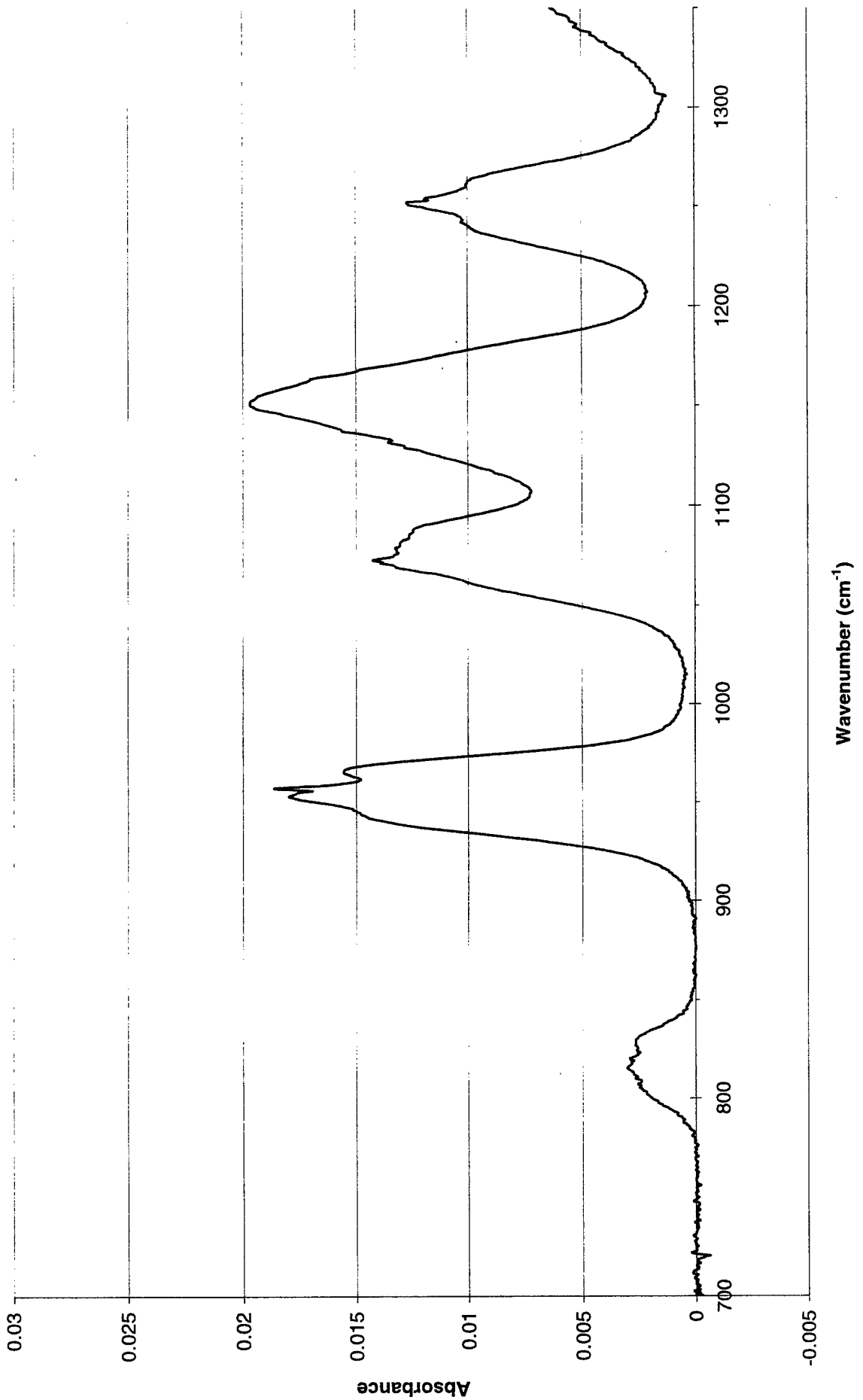


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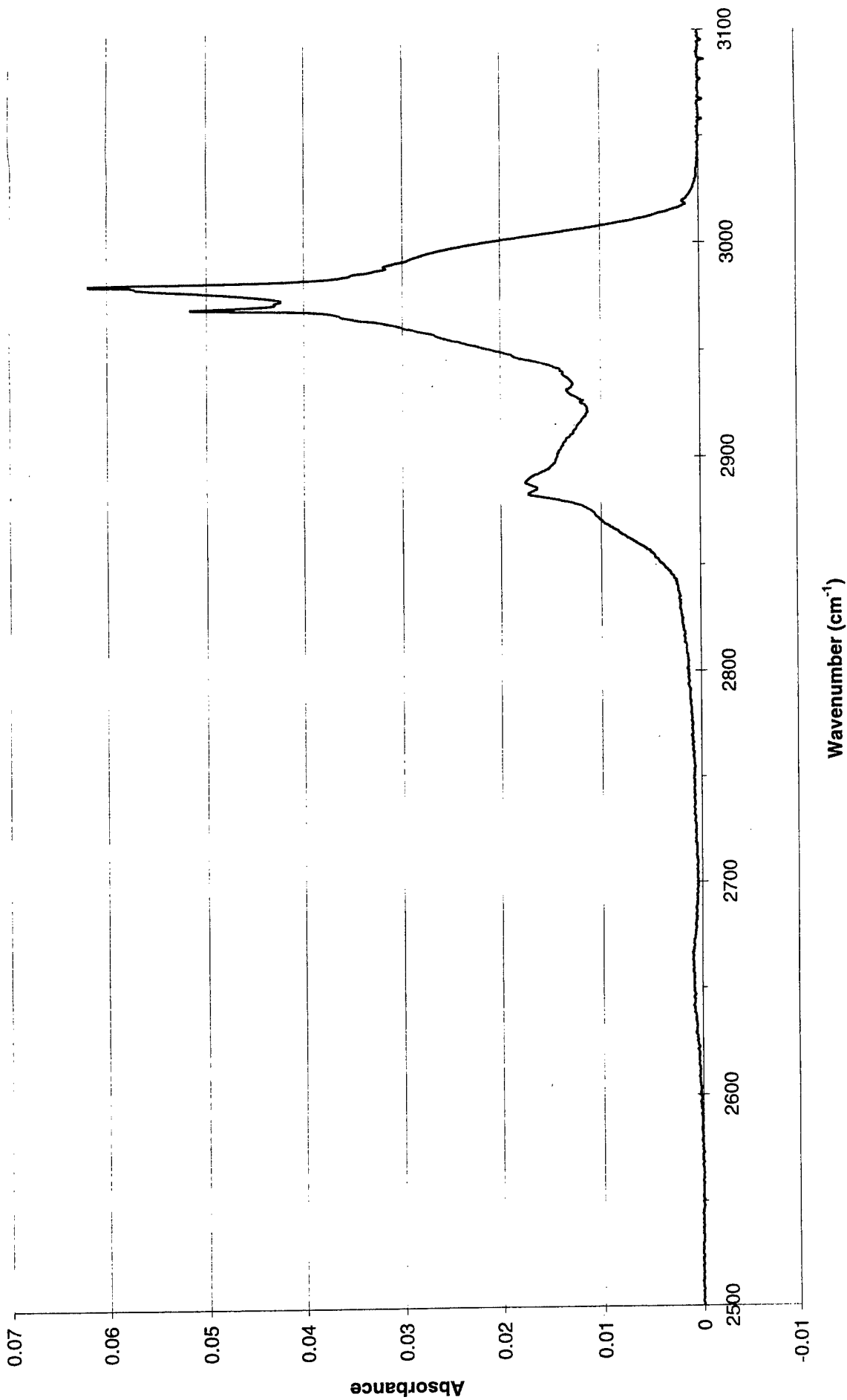


**QIRL0020 - Isopropyl alcohol**  
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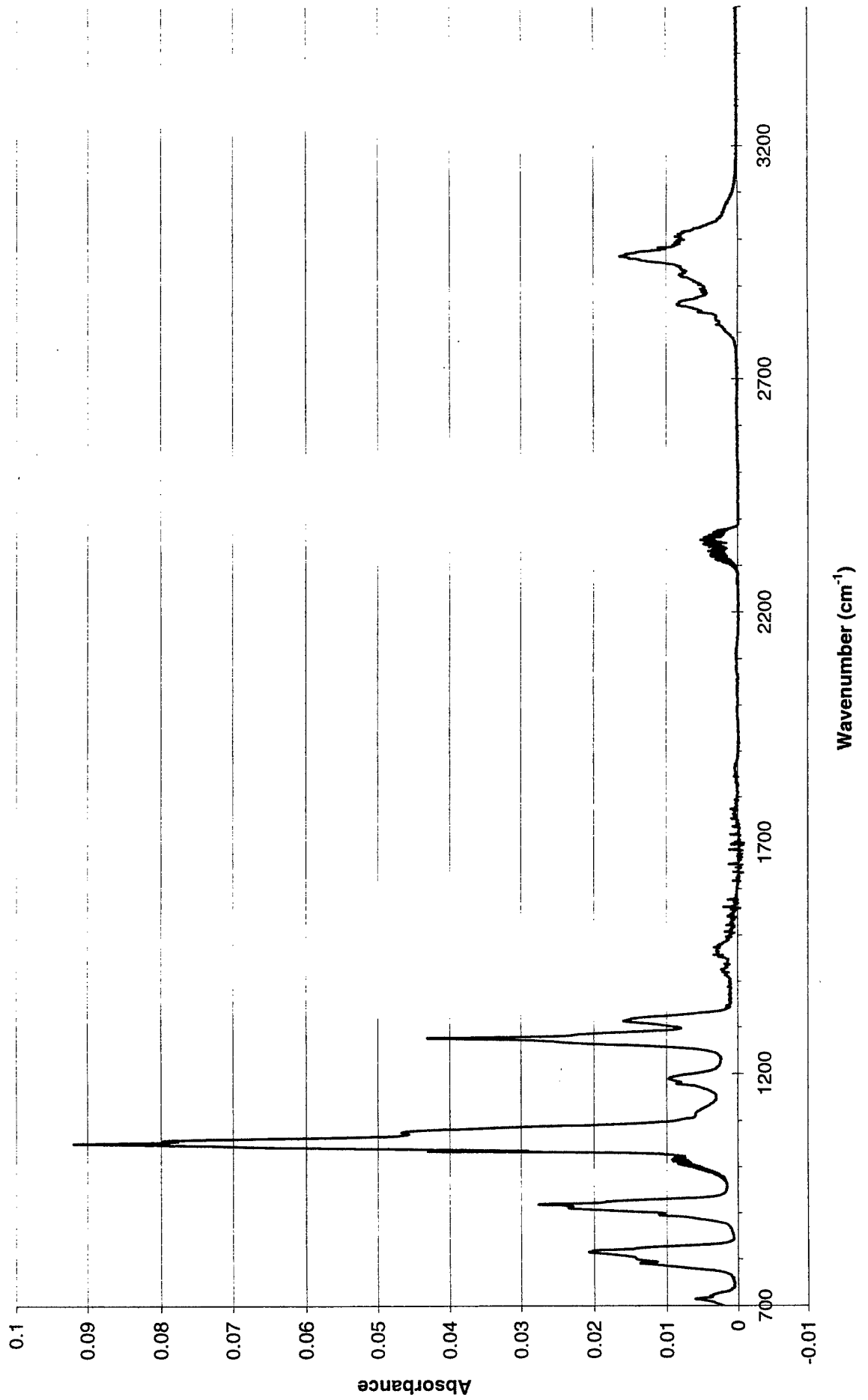


**QIRL0020 - Isopropyl alcohol**  
100 ppm-m





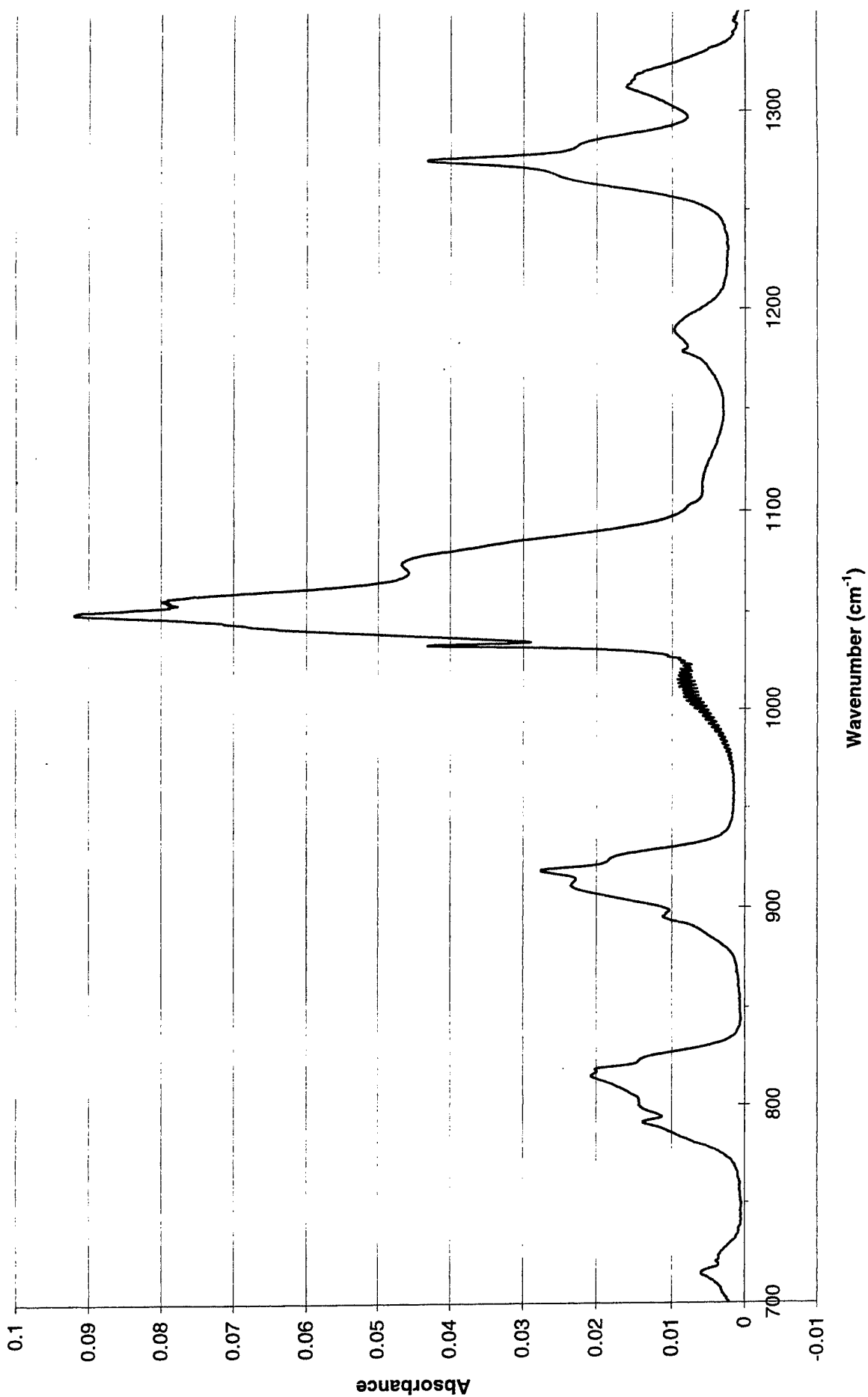
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49 ppm-m





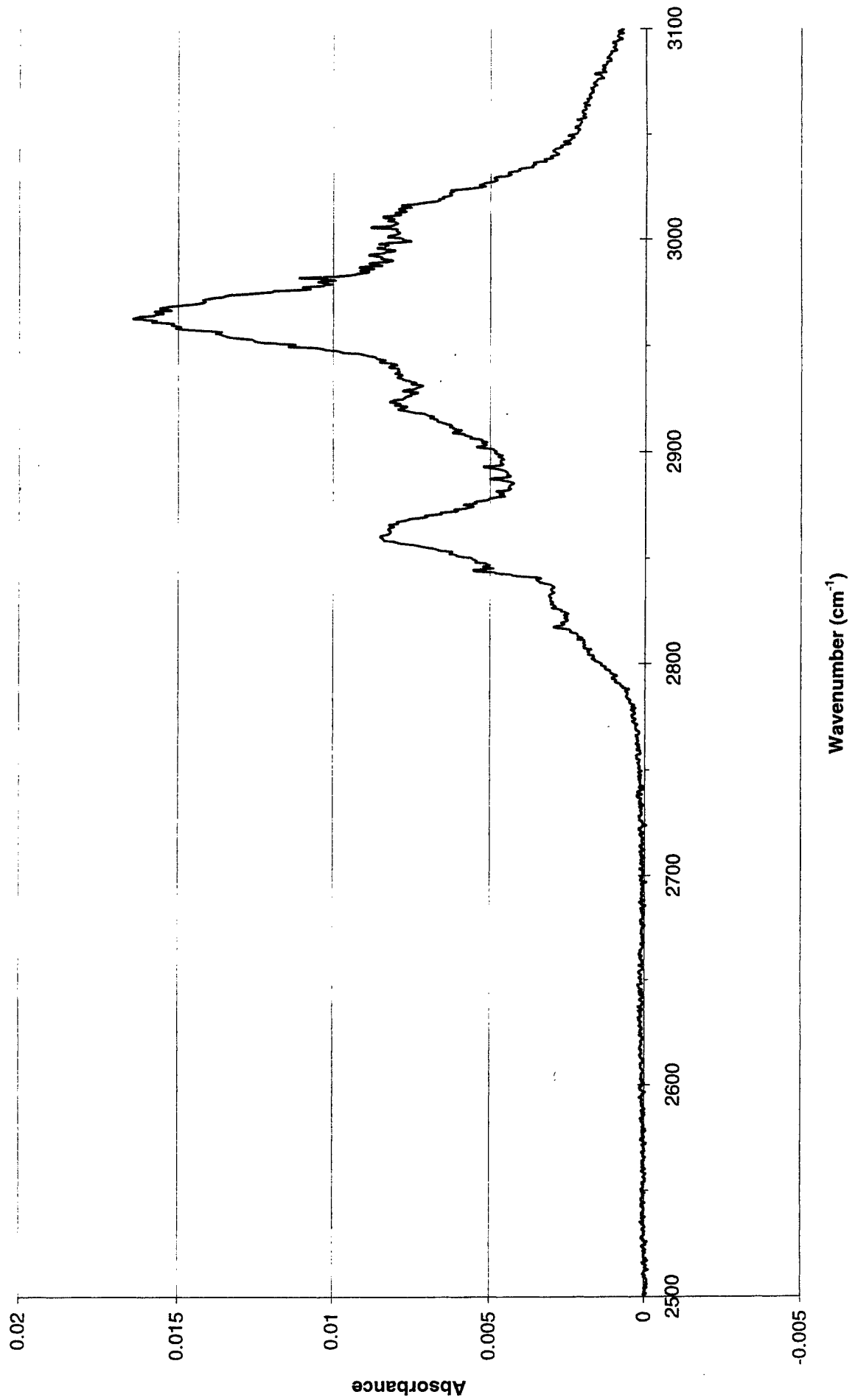
# QIRL0030 - Dimethyl methylphosphonate (DMMP)

49 ppm-m





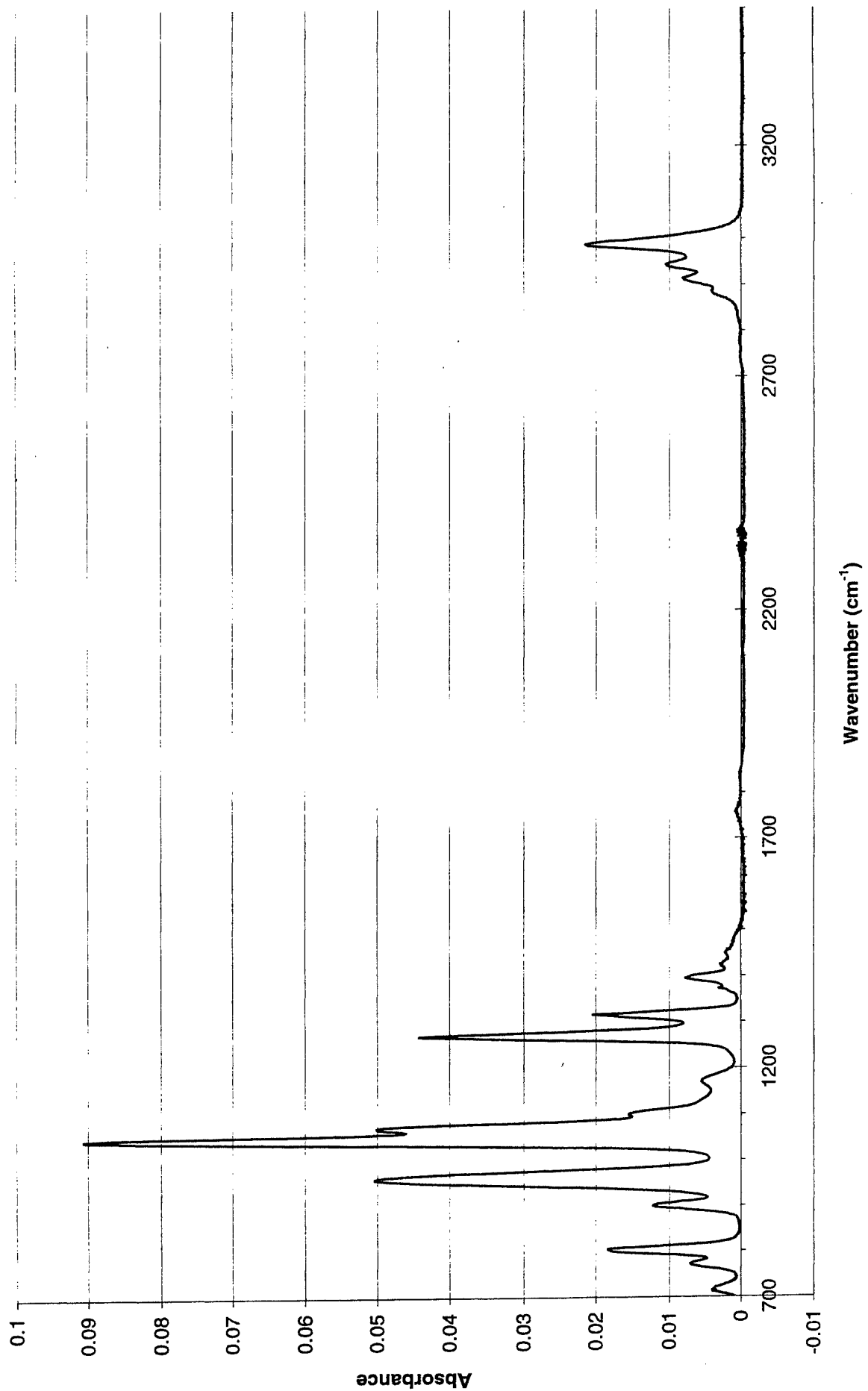
**QIRL0030 - Dimethyl methylphosphonate (DMMP)**  
49 ppm-m





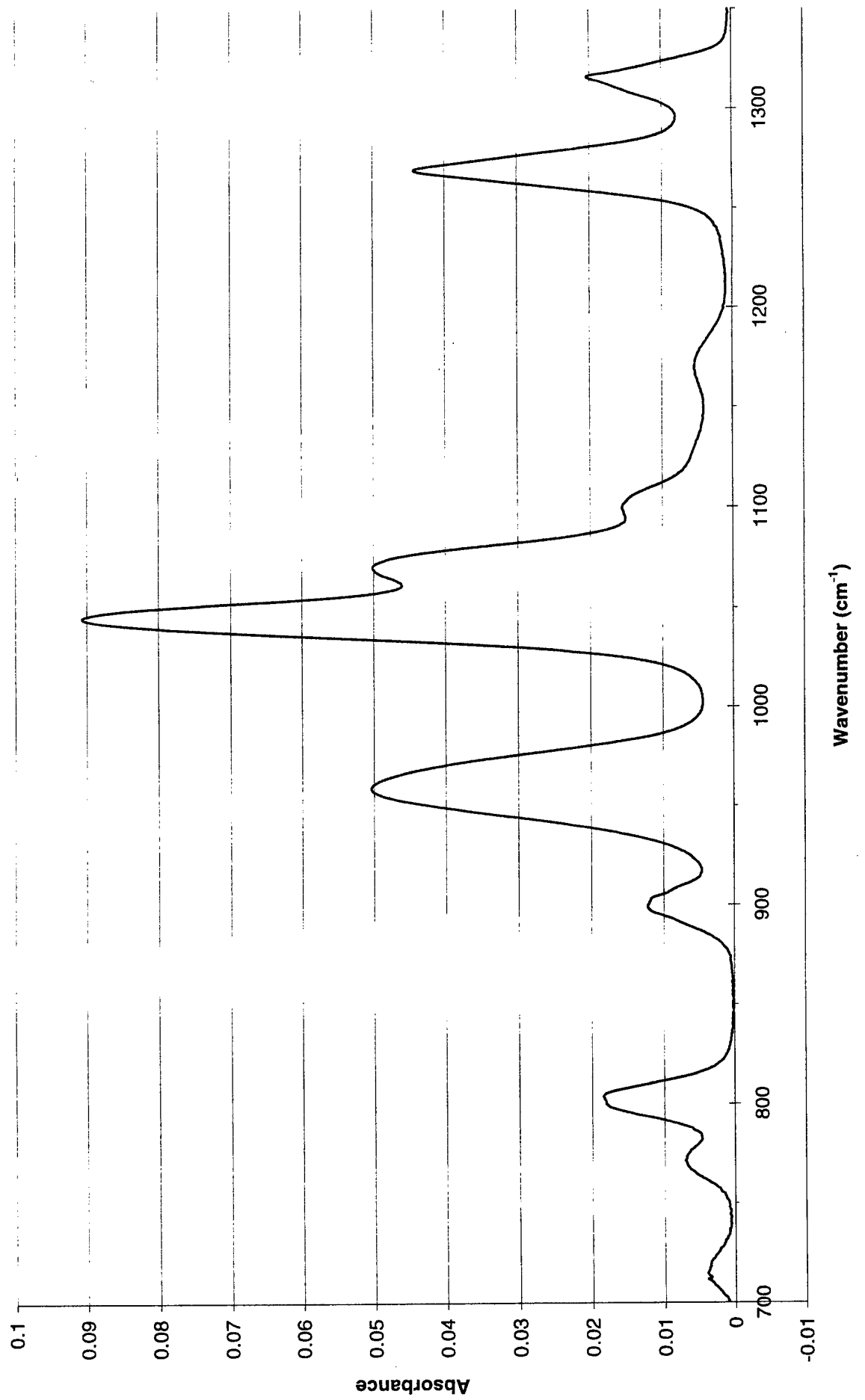
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38 ppm-m





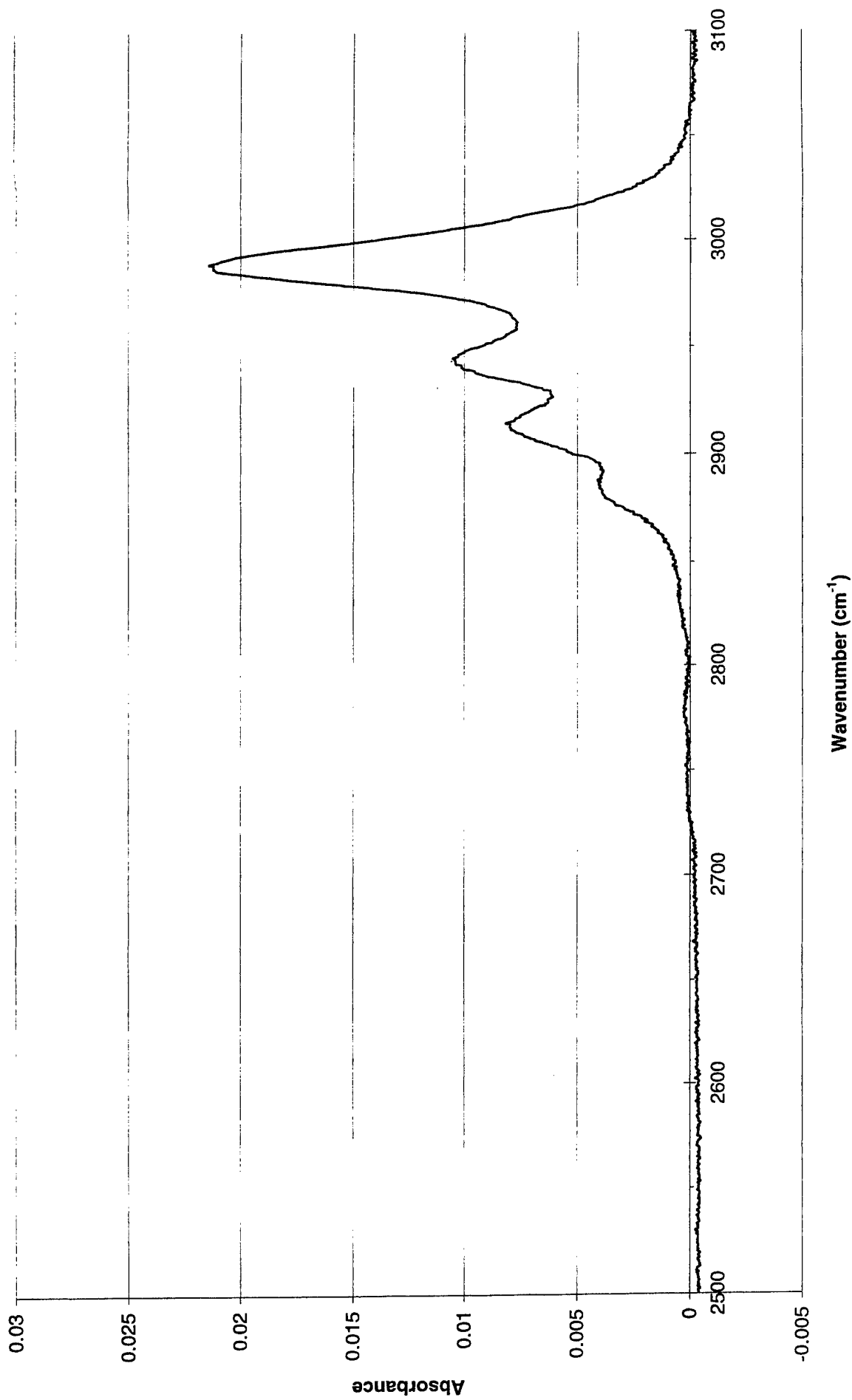
**QIRL0040 - Diethyl methylphosphonate (DEMP)**  
38 ppm-m





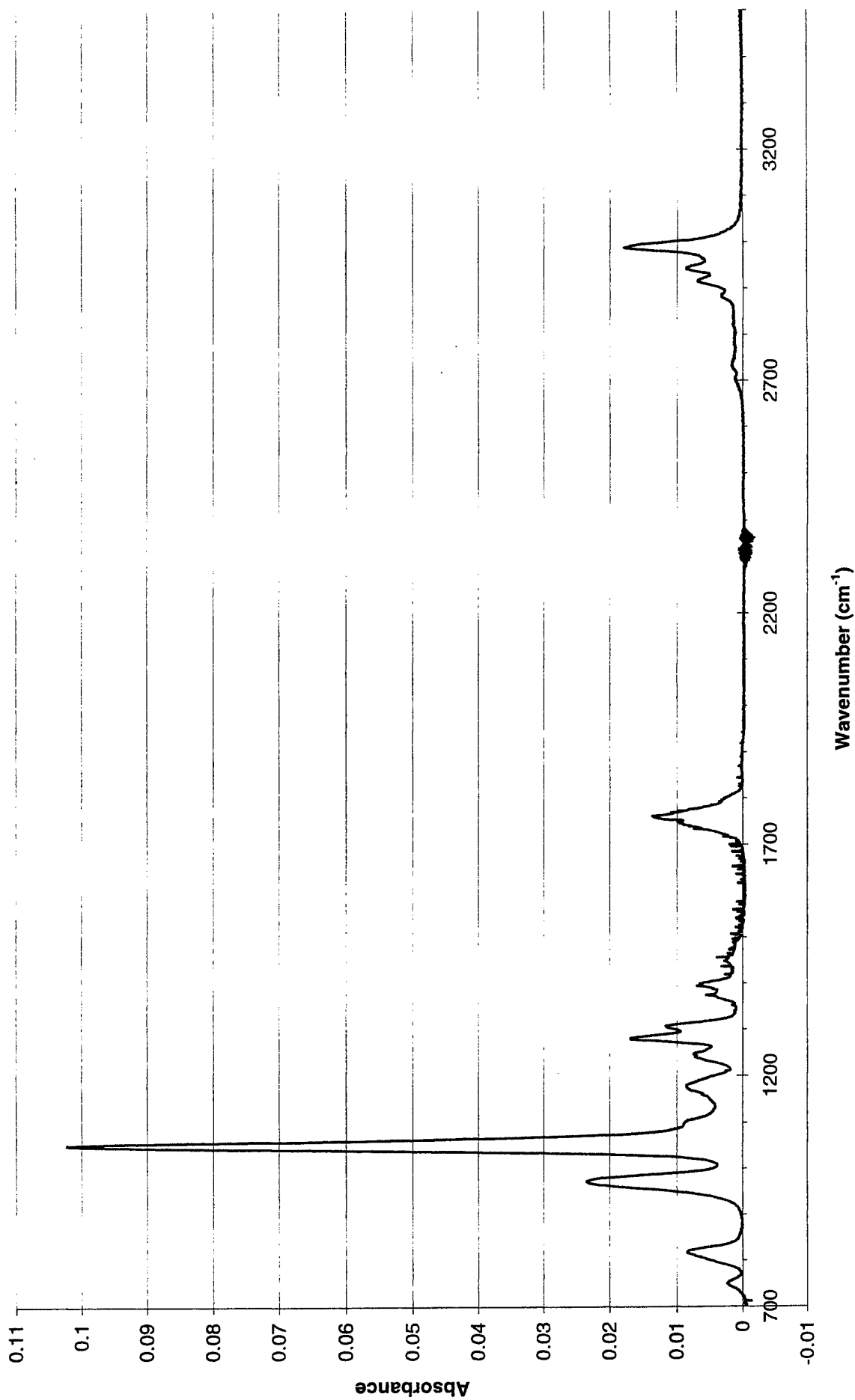
# QIRL0040 - Diethyl methylphosphonate (DEMP)

38 ppm-m



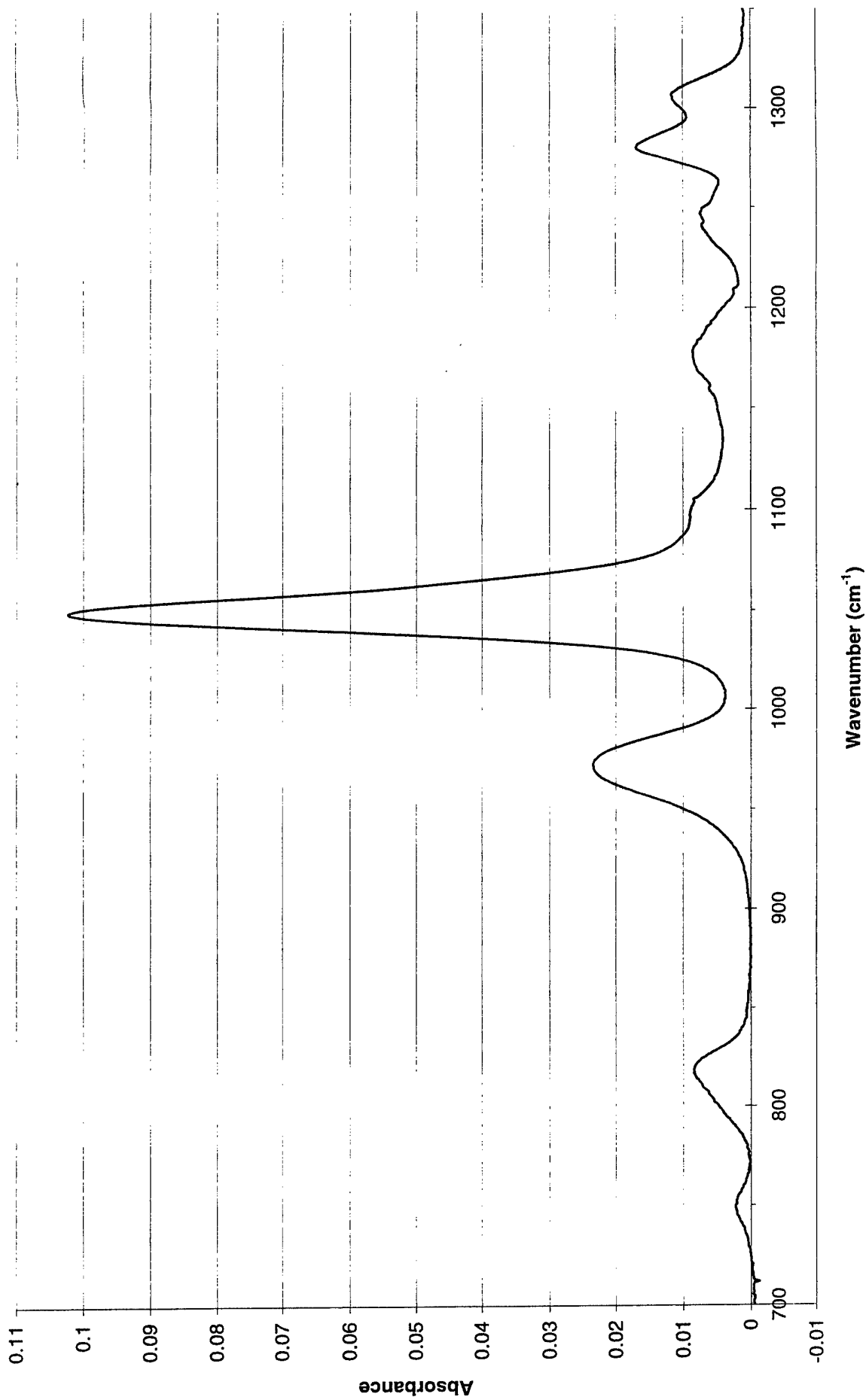


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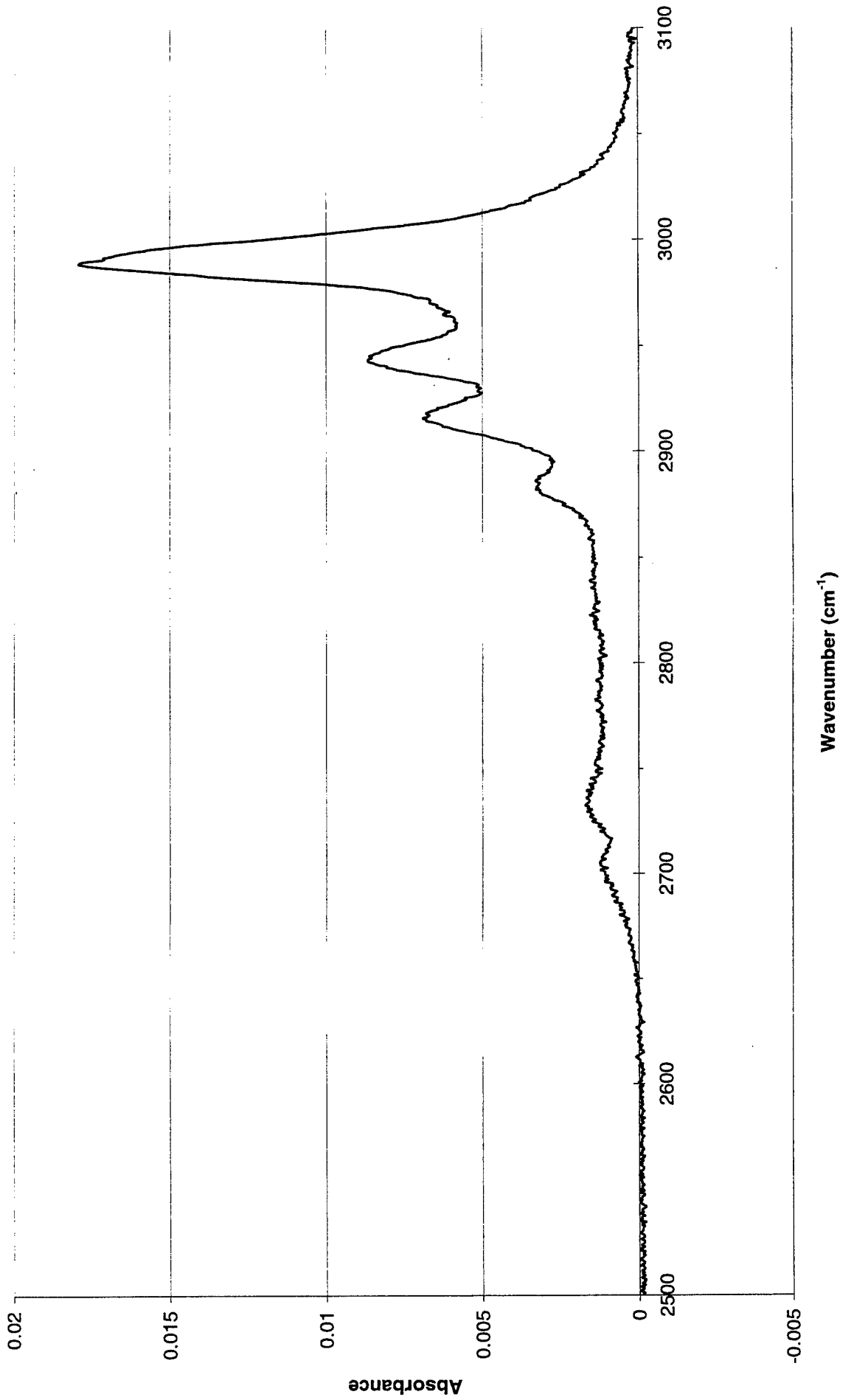


**QIRL0050 - Triethyl phosphate (TEP)**  
18 ppm-m



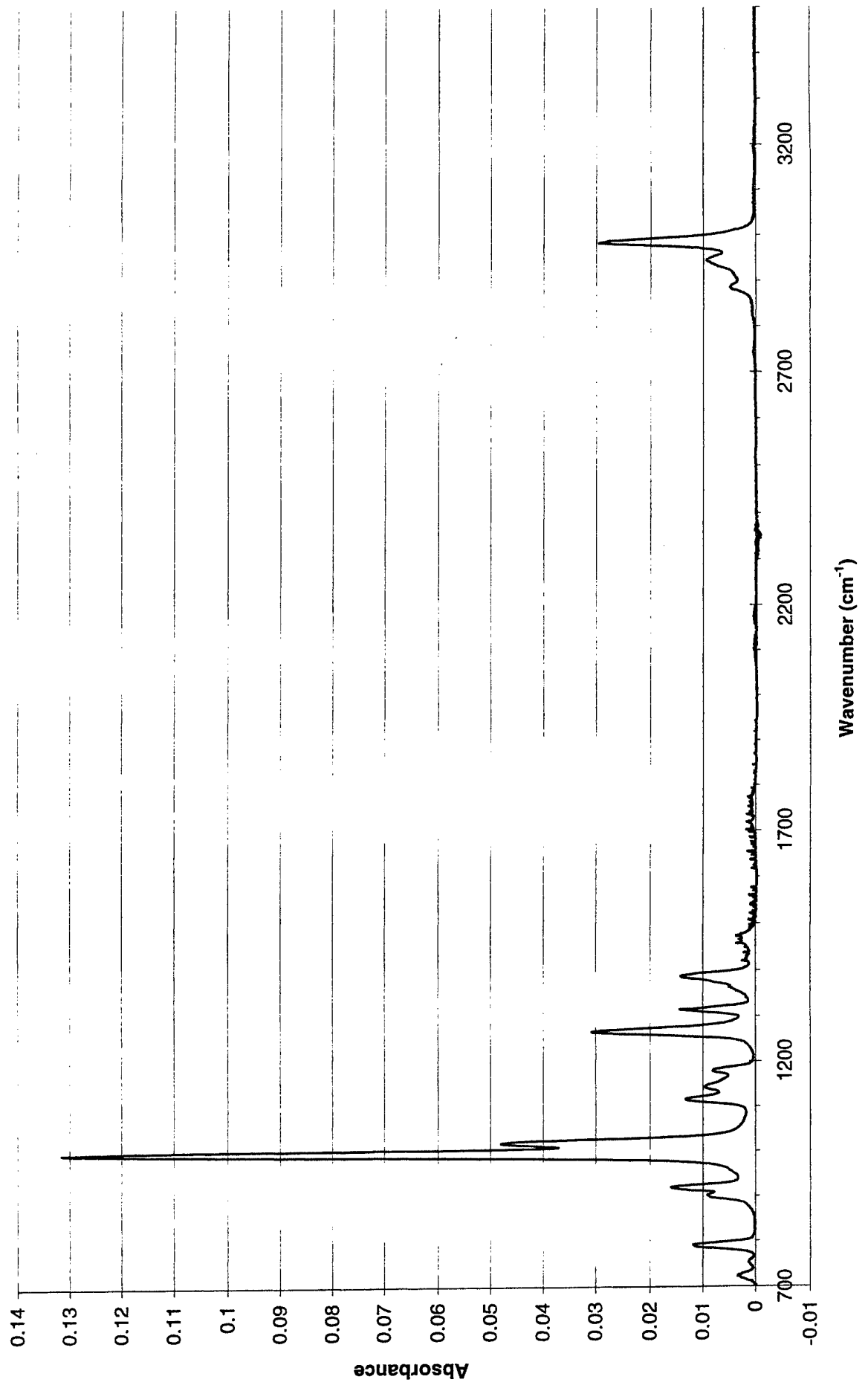


**QIRL0050 - Triethyl phosphate (TEP)**  
18 ppm-m





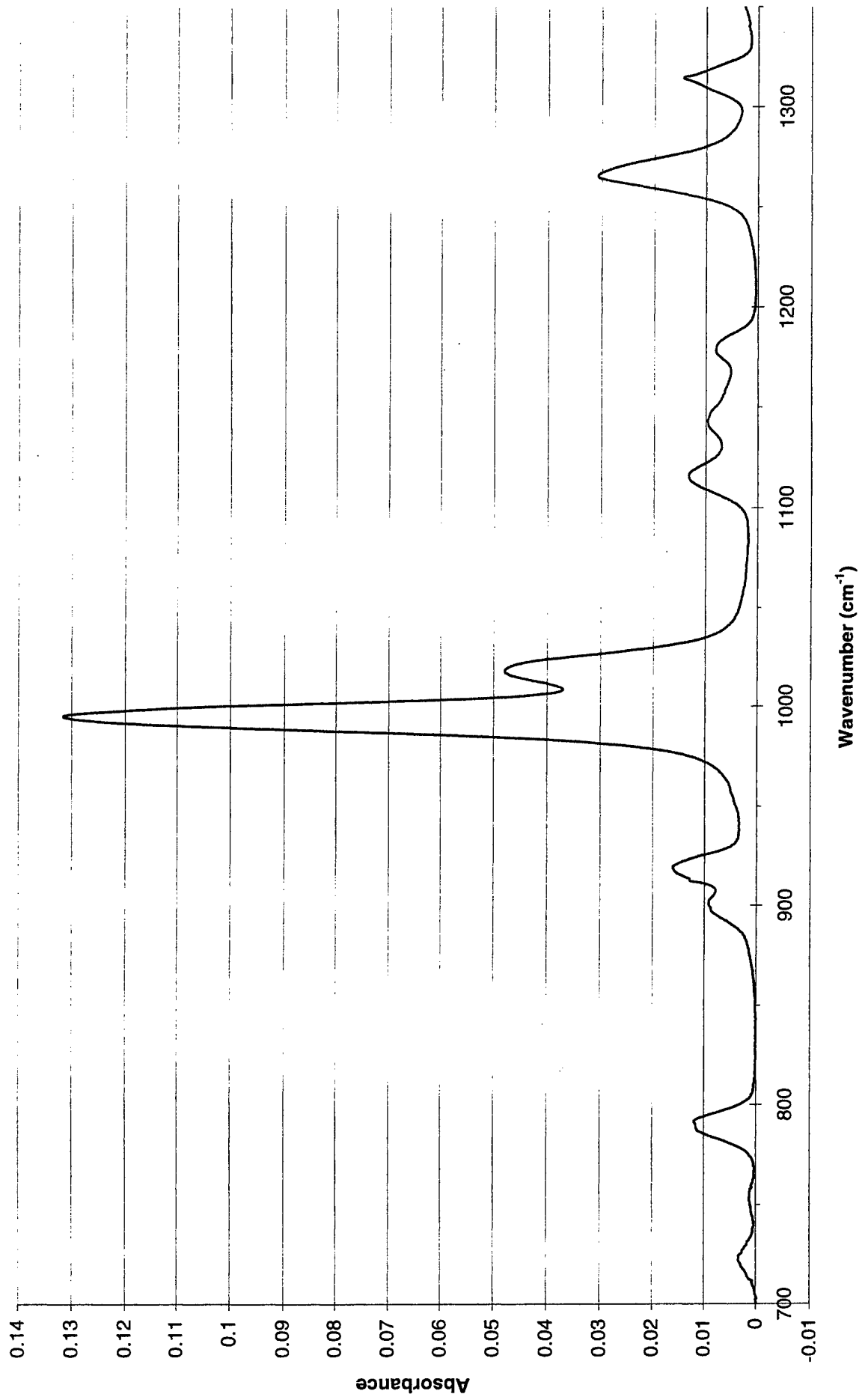
# QIRL0060 - Diisopropyl methylphosphonate (DIMP) 24 ppm-m





# QIRL0060 - Diisopropyl methylphosphonate (DIMP)

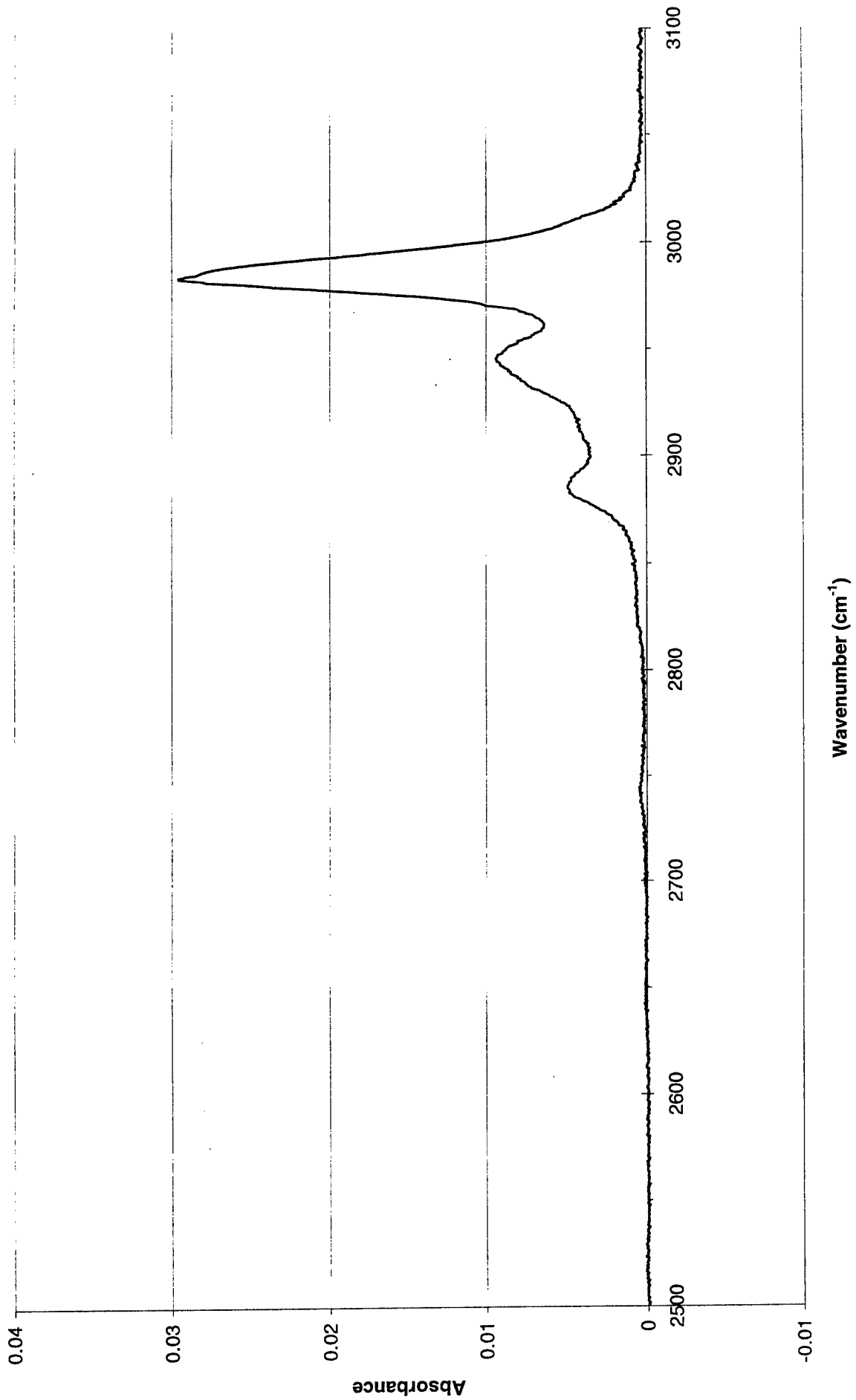
24 ppm-m





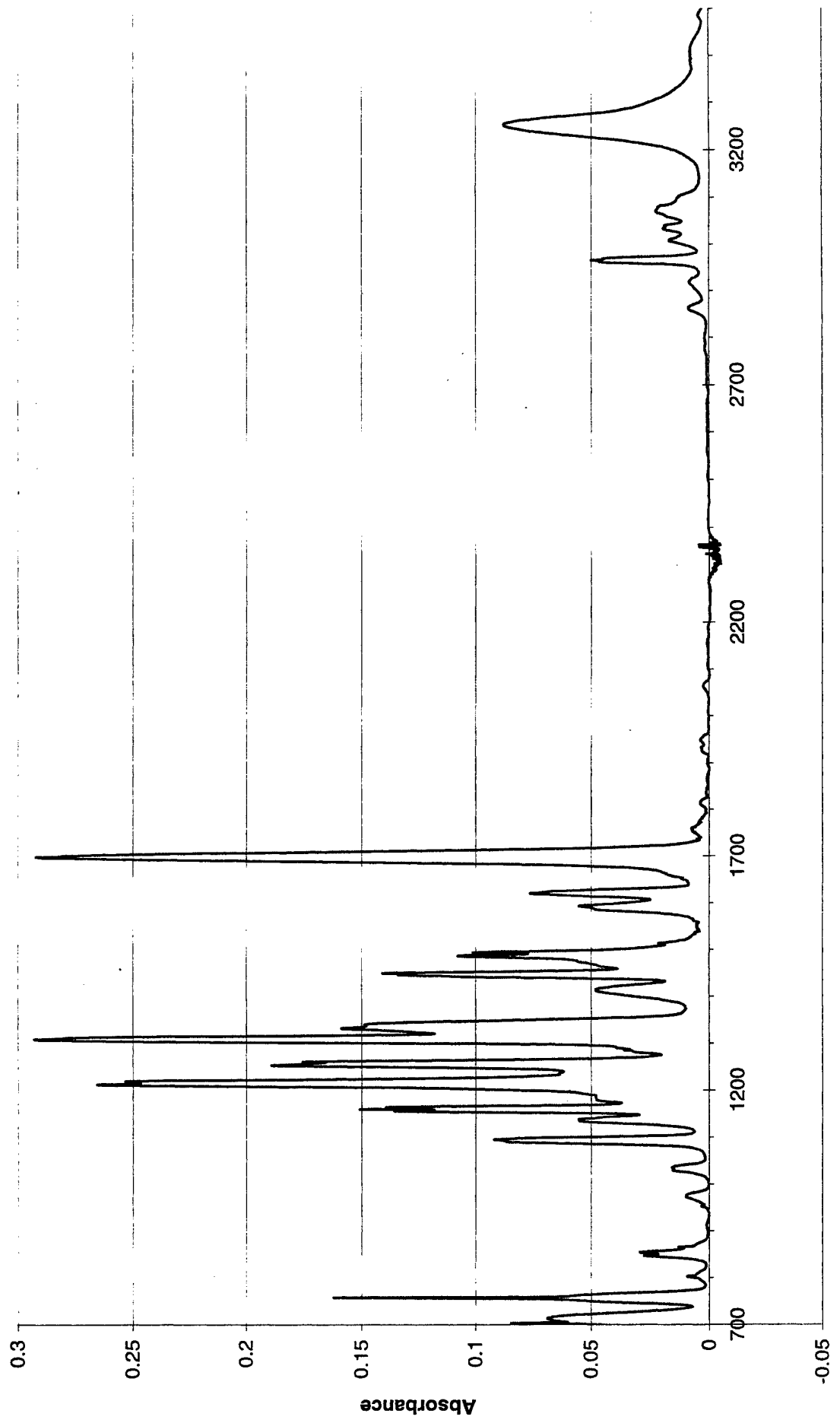
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24 ppm-m



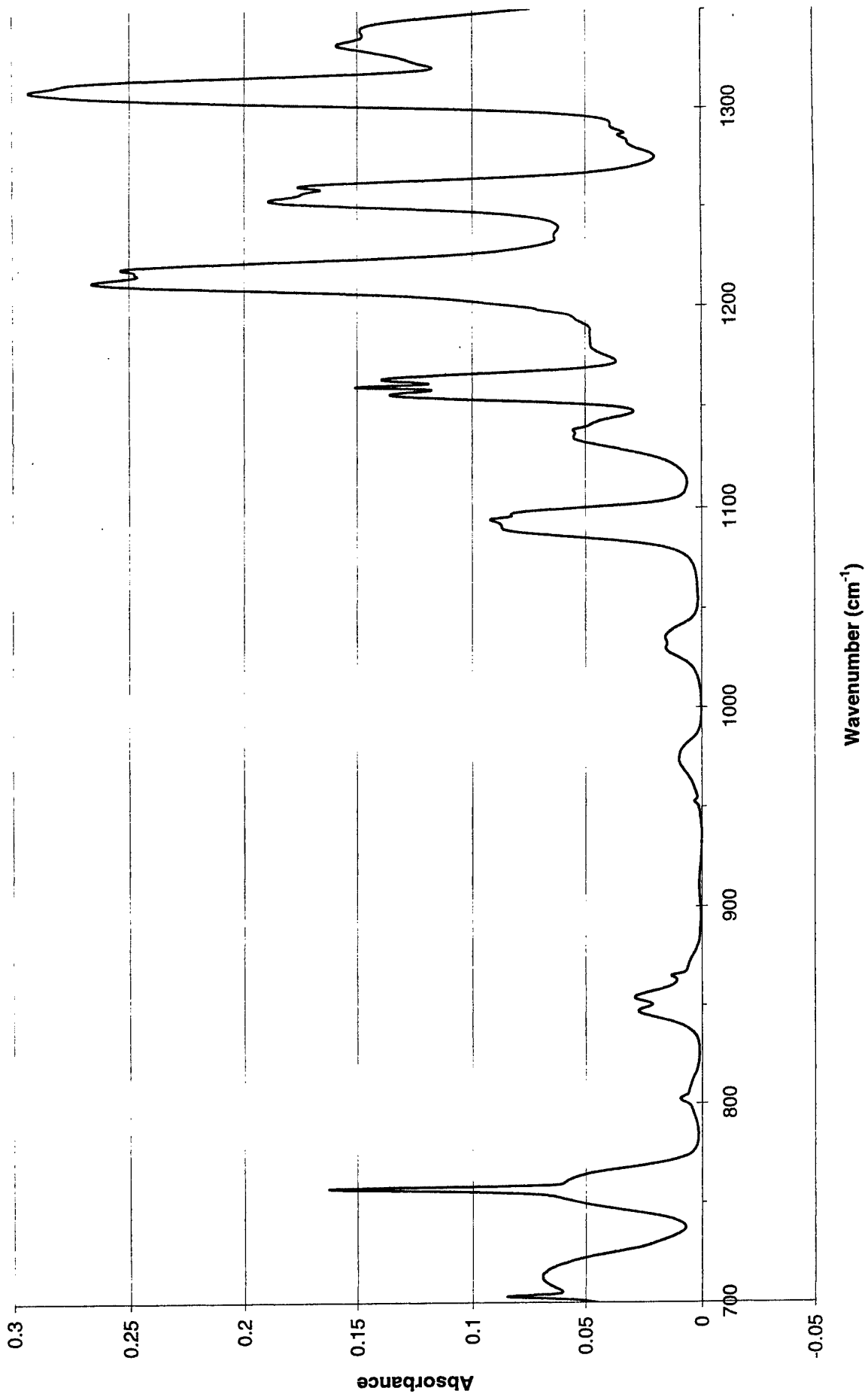


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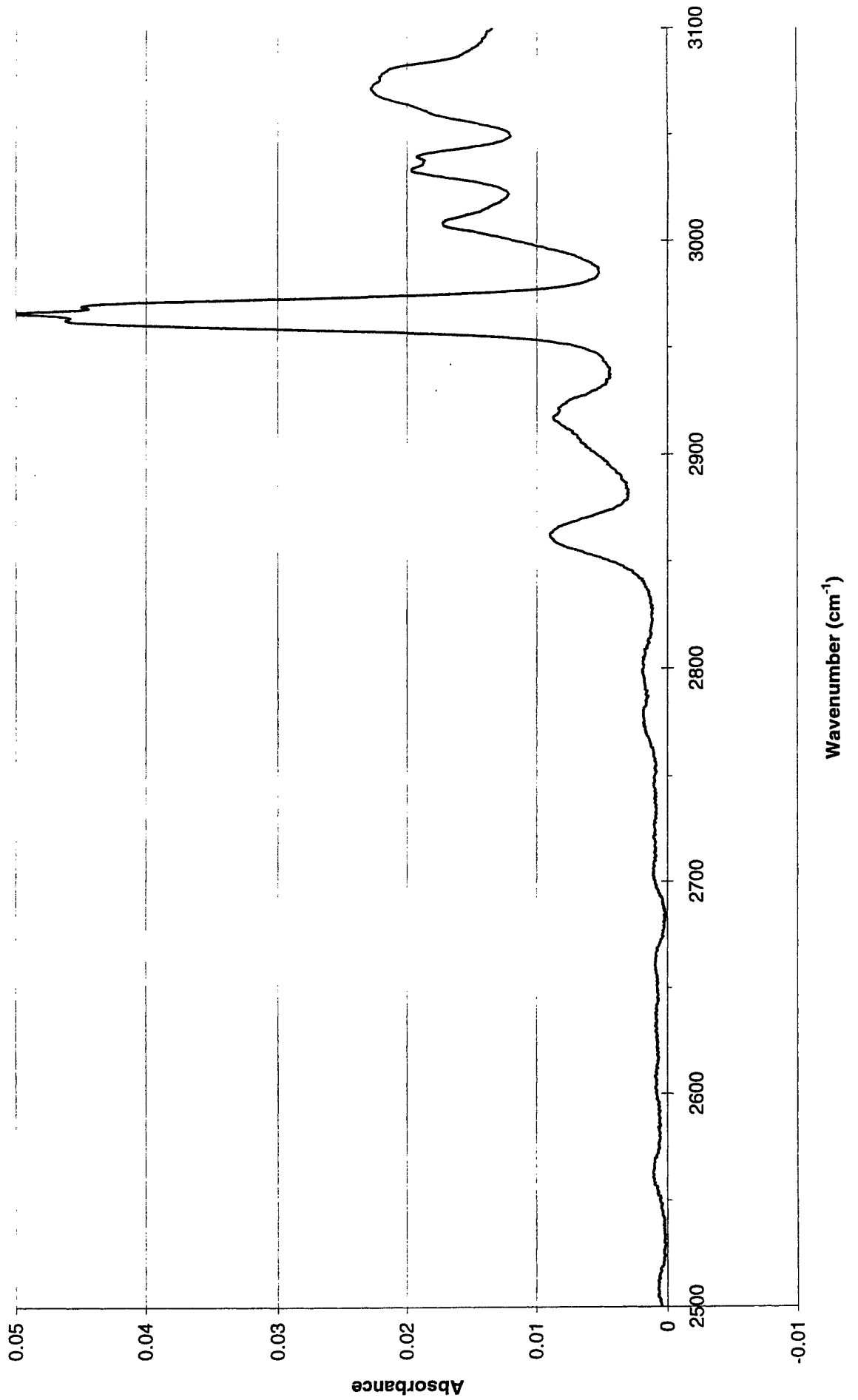


**QIRL0070 - Methyl salicylate**  
171 ppm-m





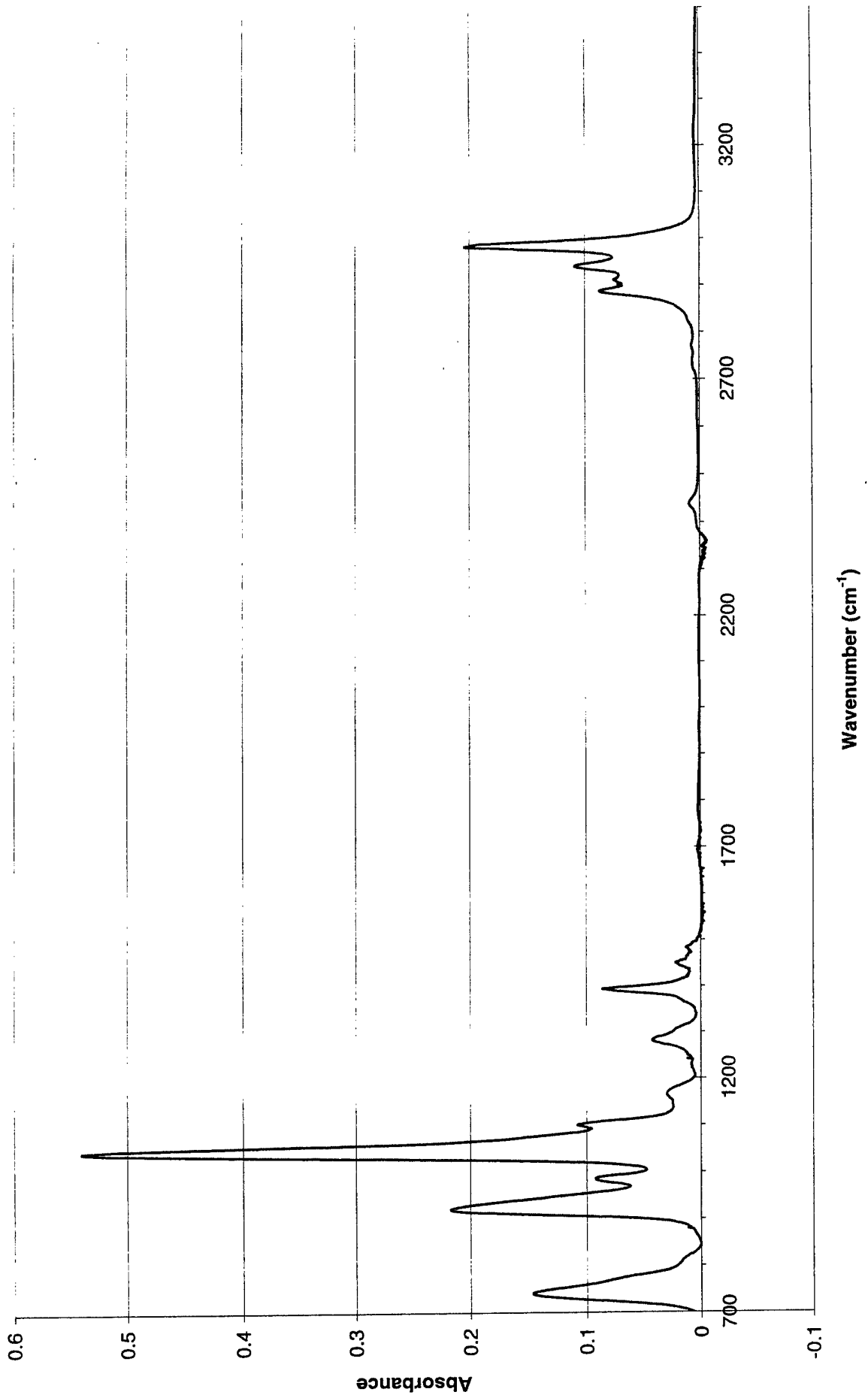
**QIRL0070 - Methyl salicylate**  
171 ppm-m





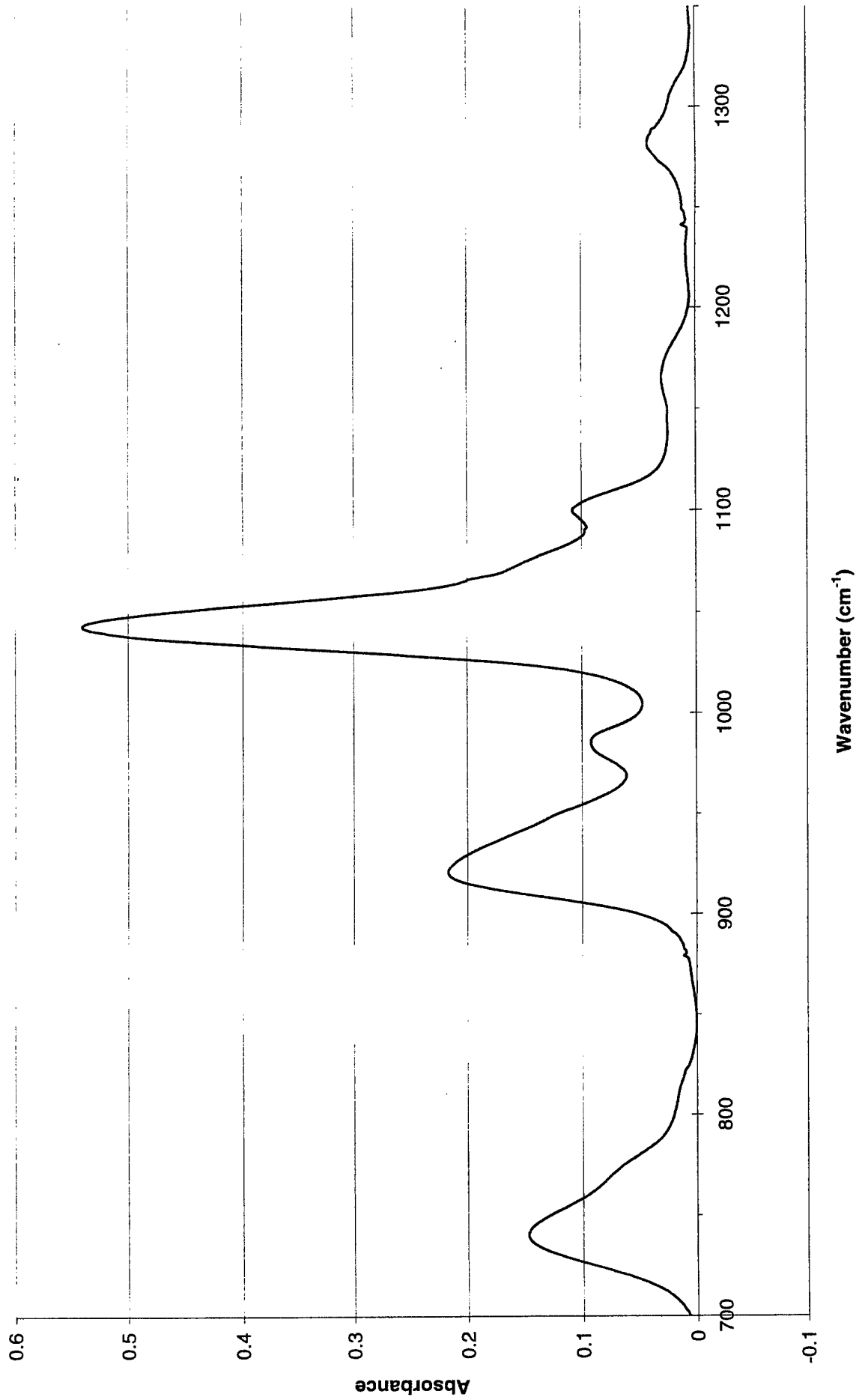
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84 ppm-m



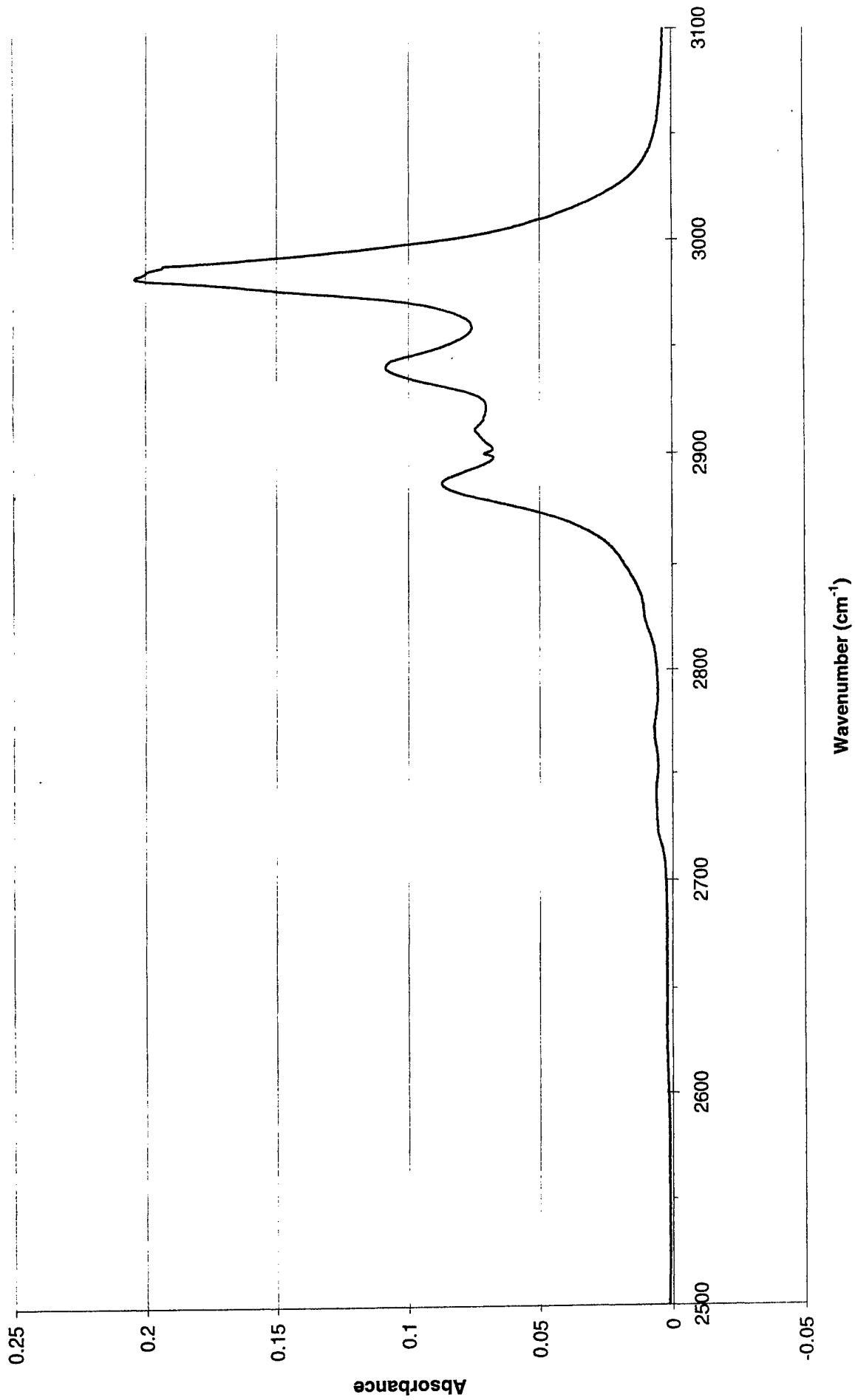


**QIRL0080 - Triethyl phosphite**  
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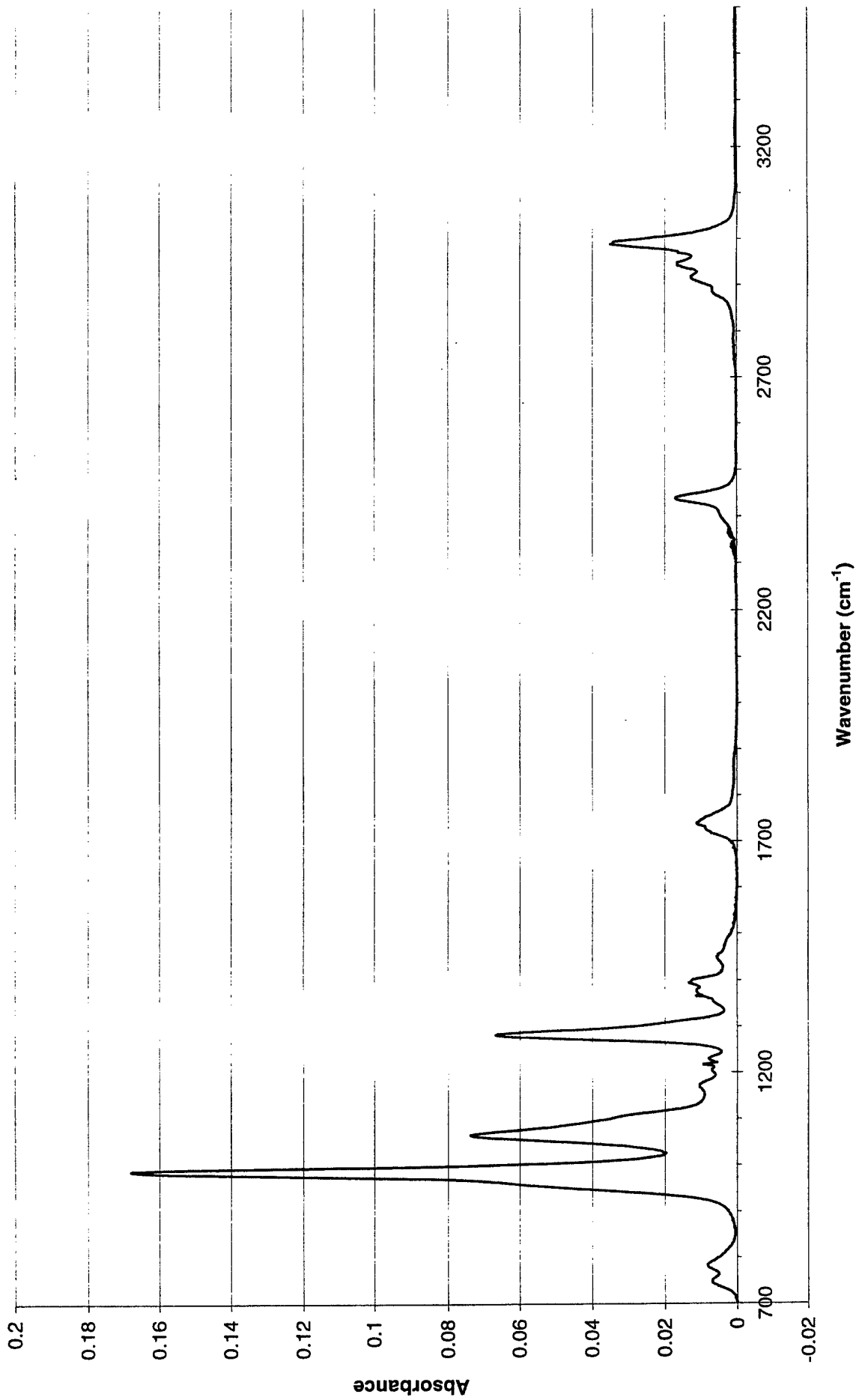


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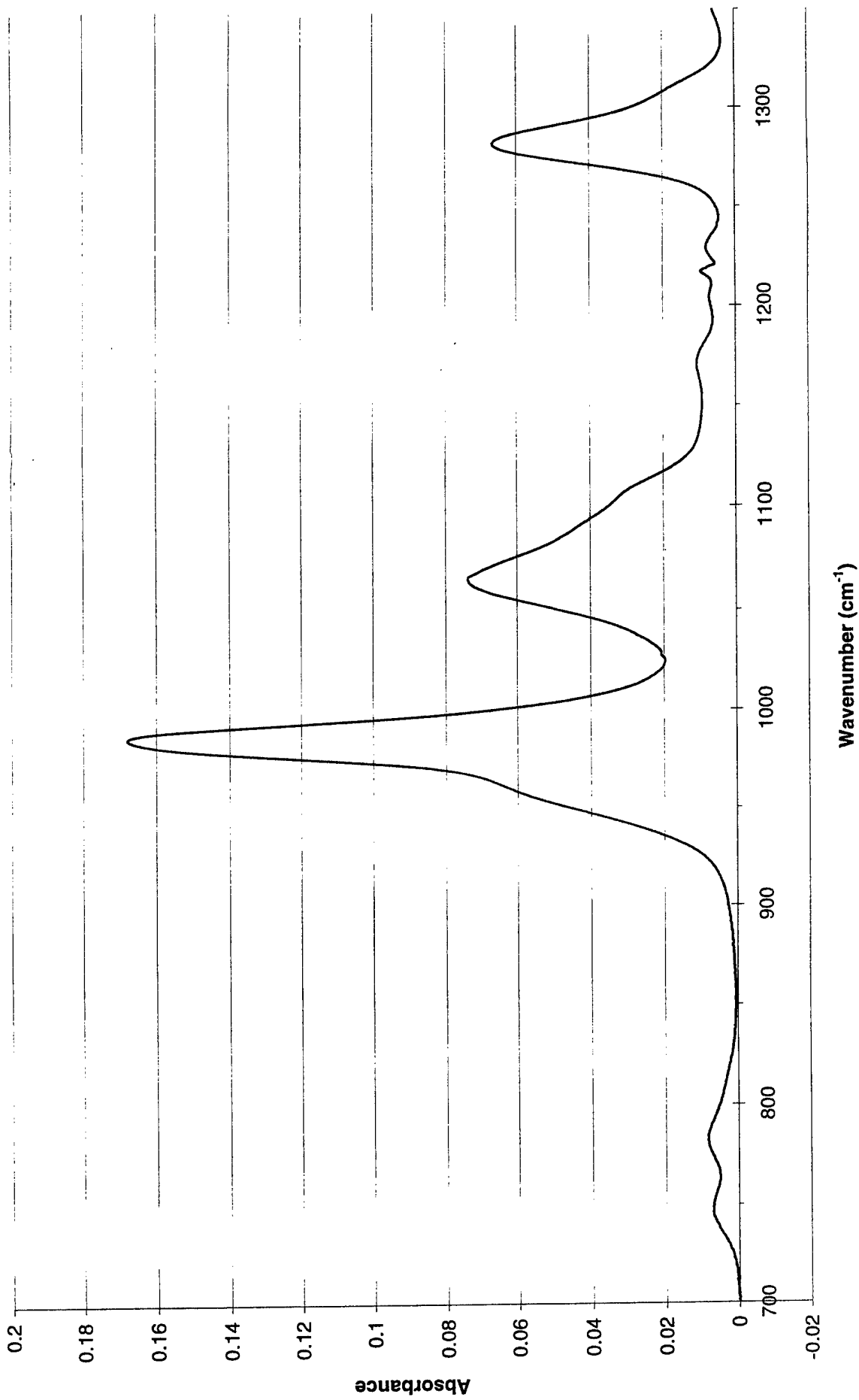
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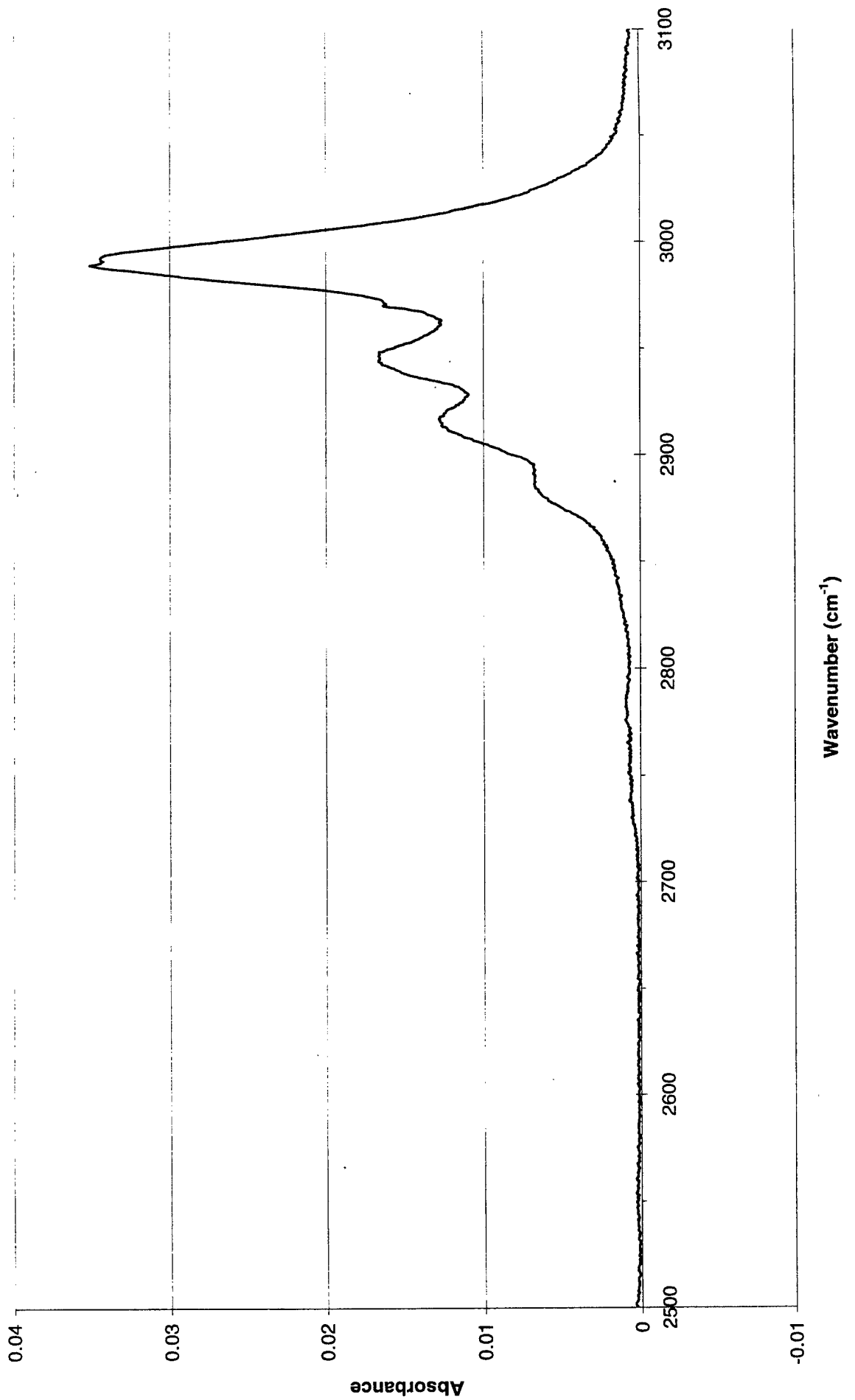
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72 ppm-m





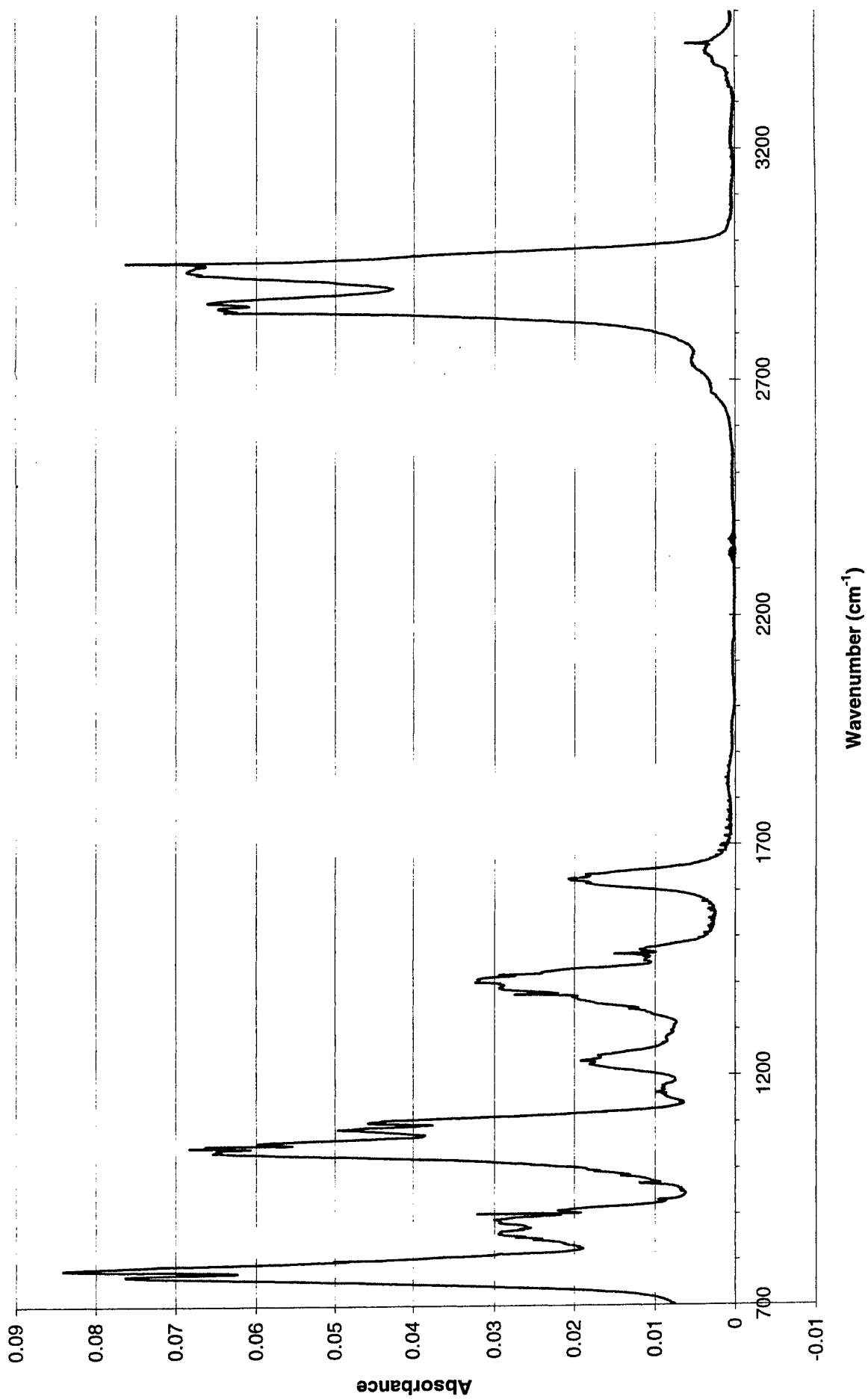
**QIRL0090 - Diethyl phosphite**  
72 ppm-m





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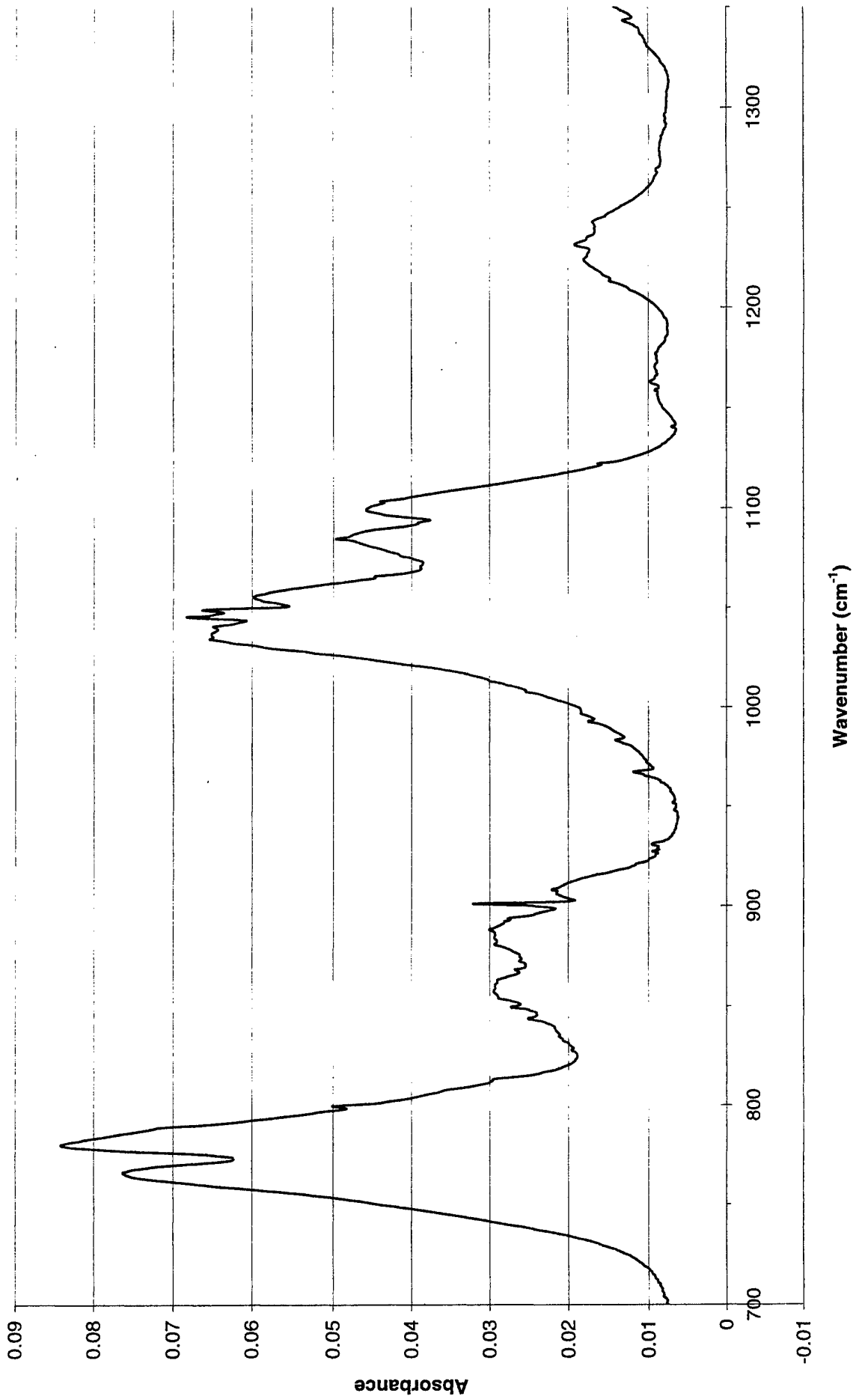
92 ppm-m





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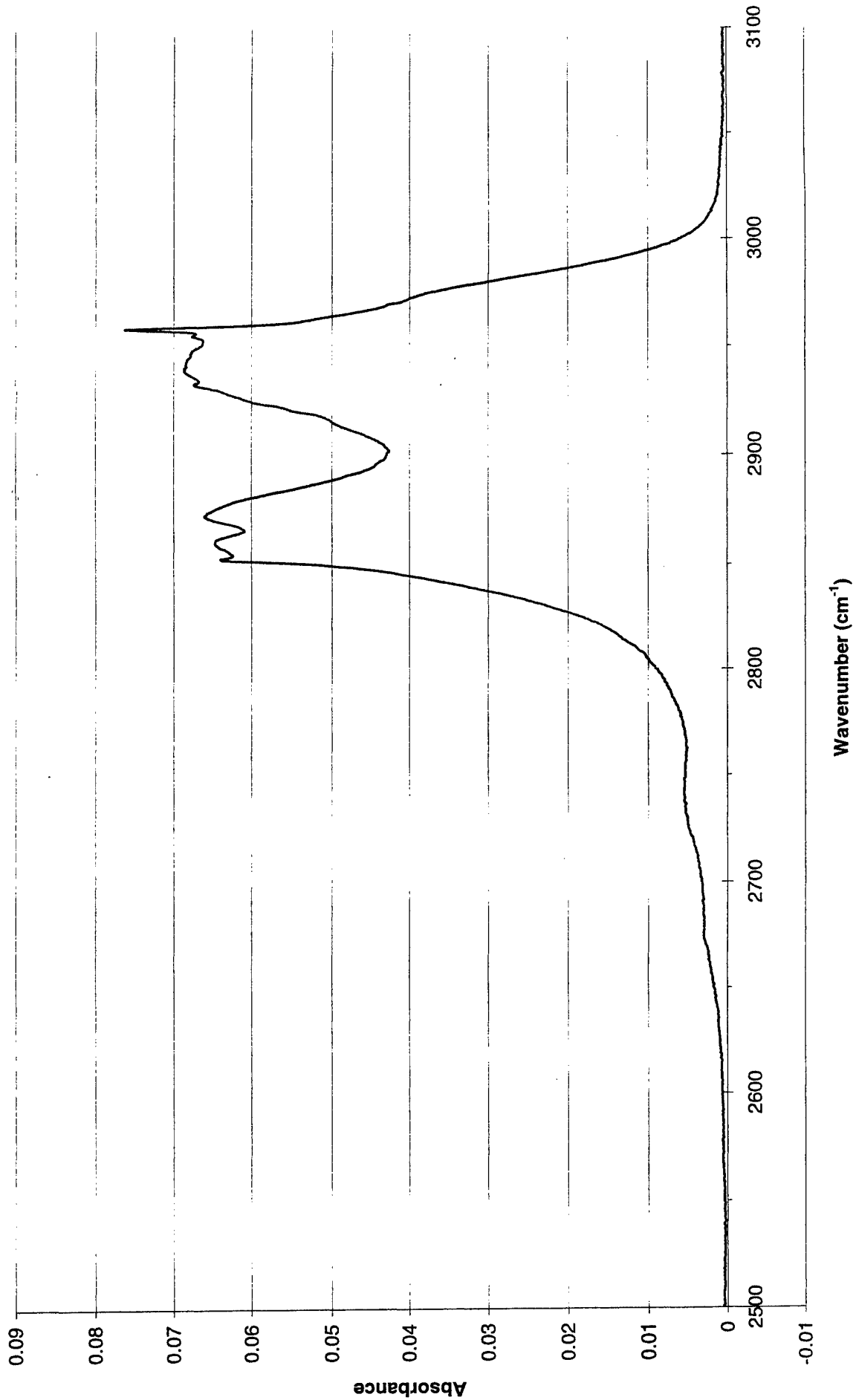
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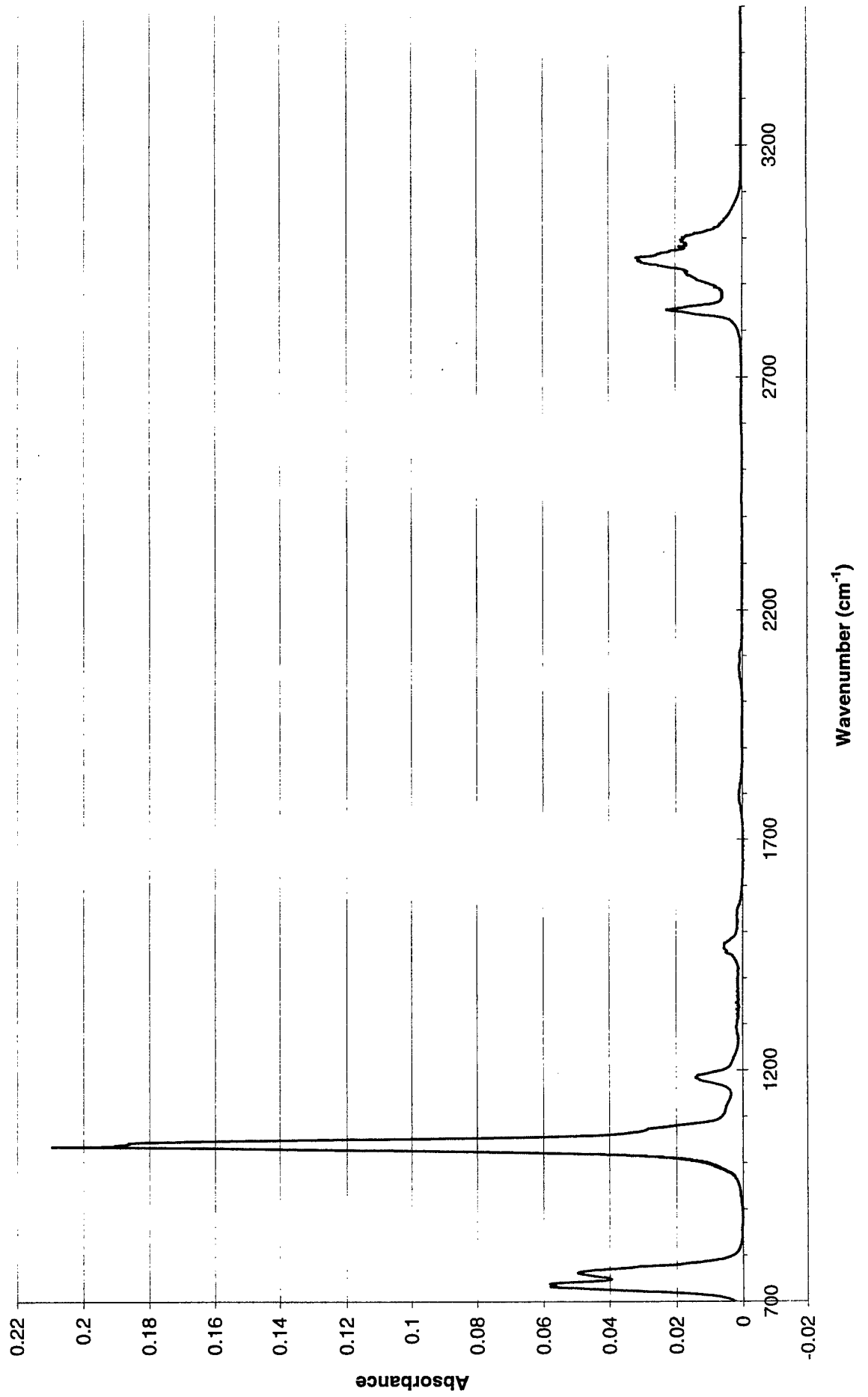
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92 ppm-m





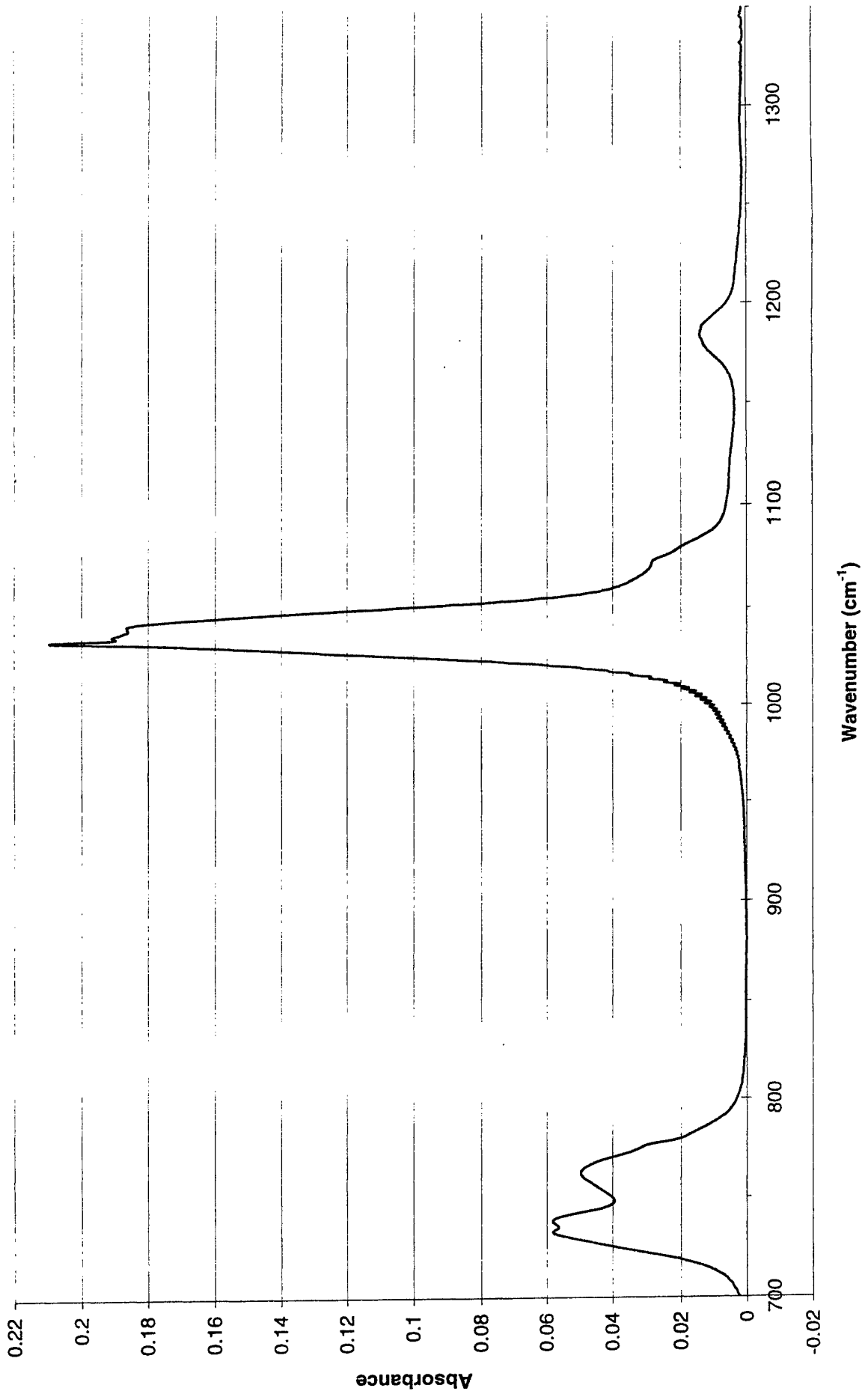
**QIRL0110 - Trimethylphosphite**  
93 ppm-m





# QIRL0110 - Trimethylphosphite

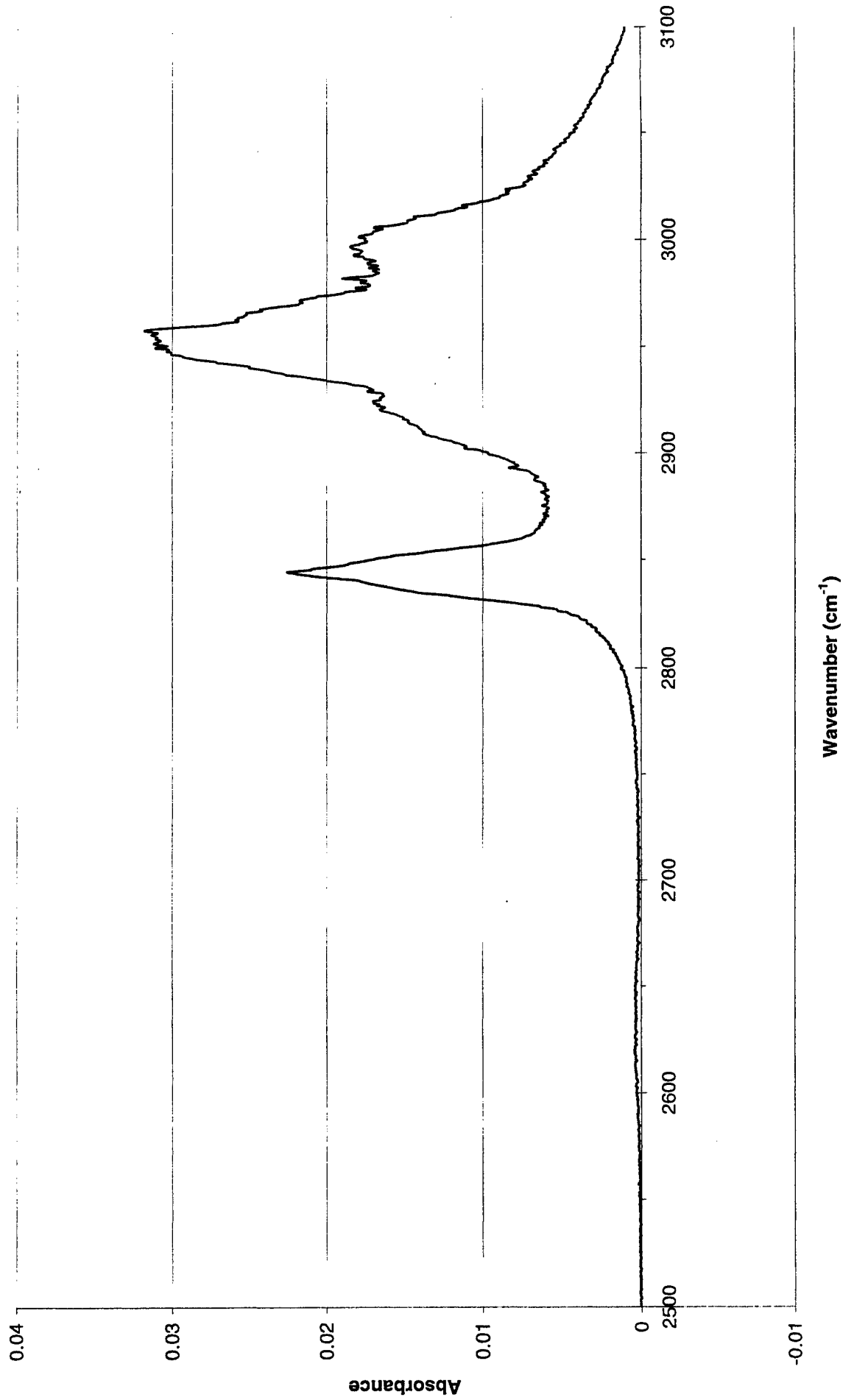
93 ppm-m





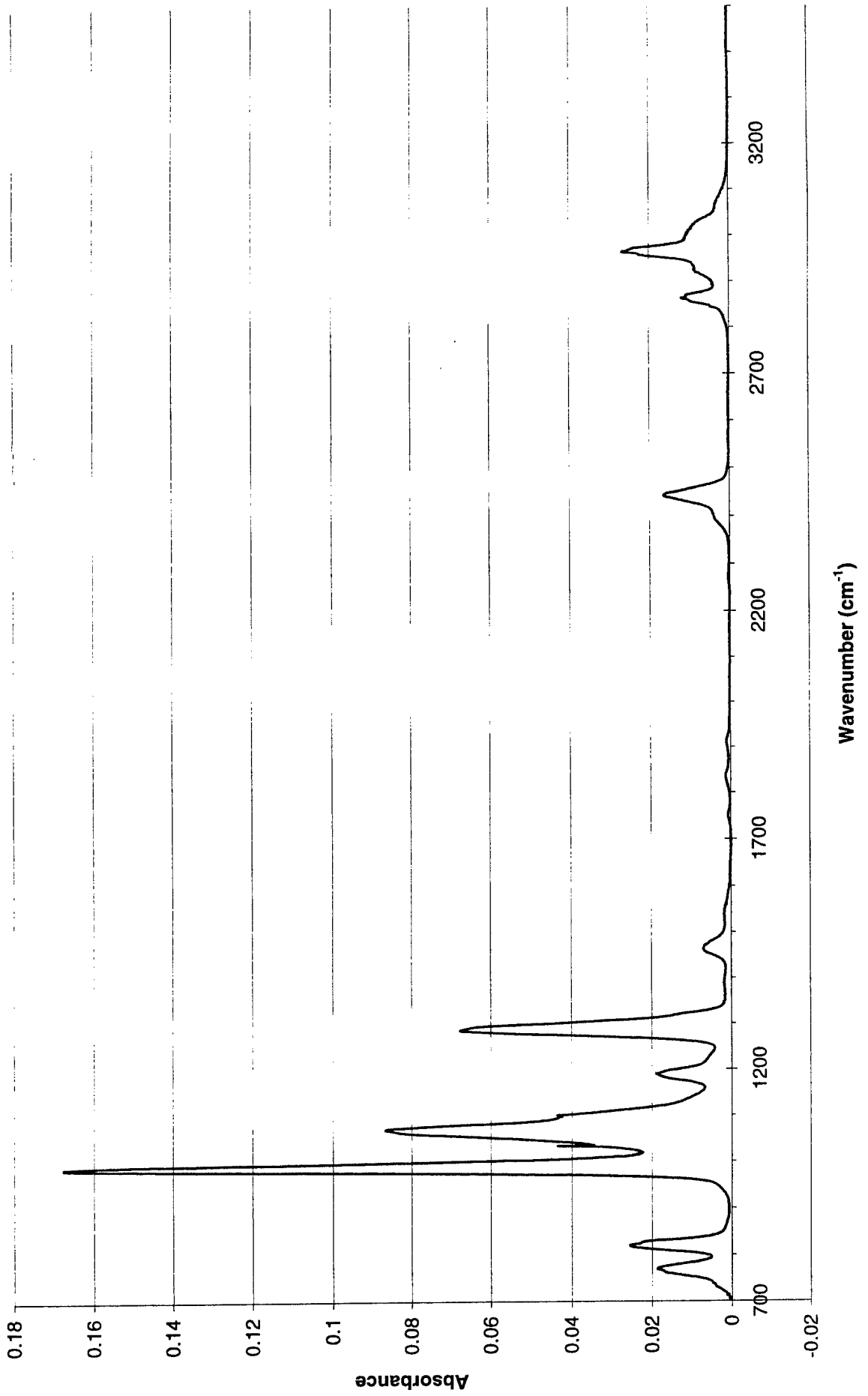
# QIRL0110 - Trimethylphosphite

93 ppm-m



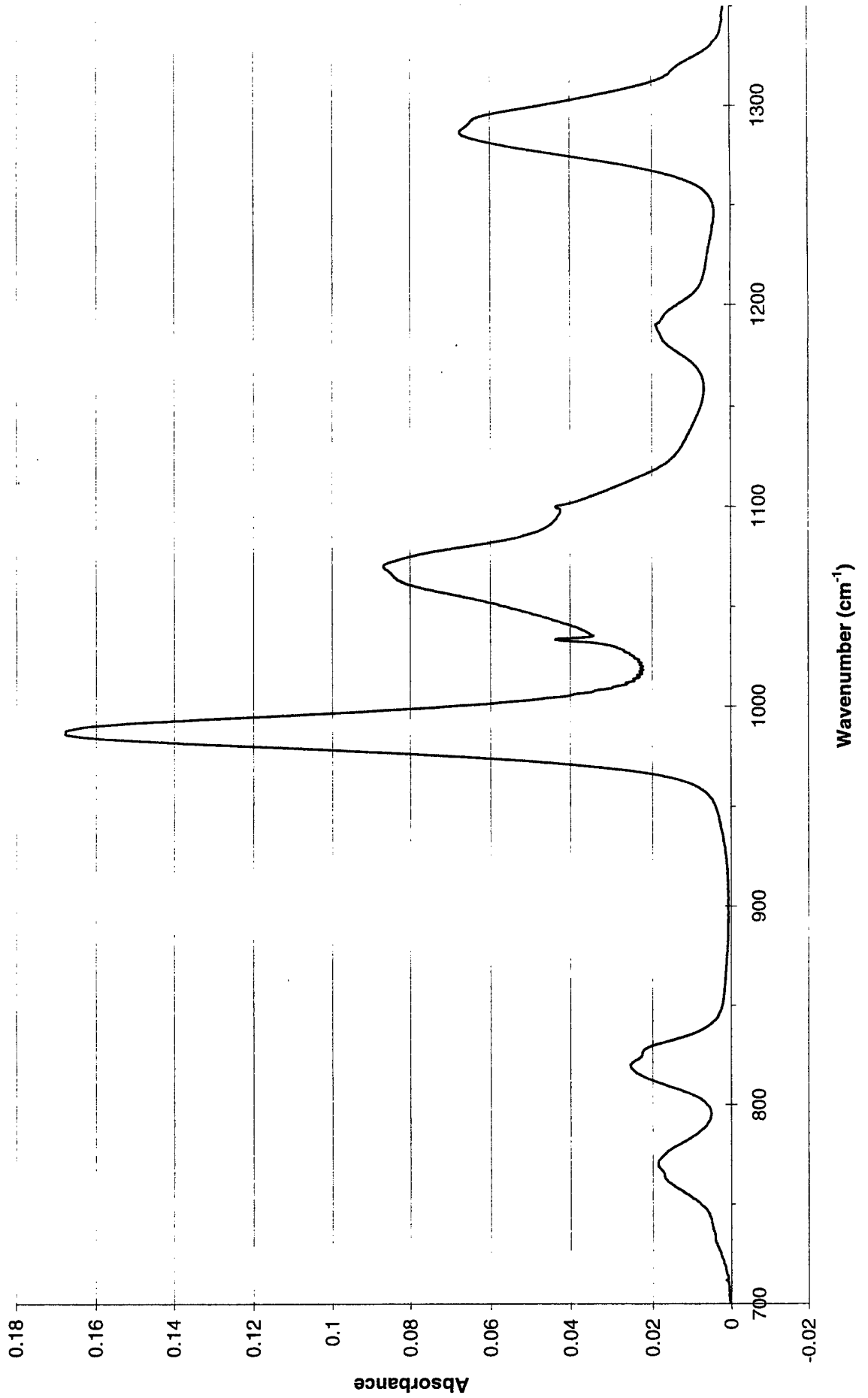


**QIRL0120 - Dimethyl phosphite**  
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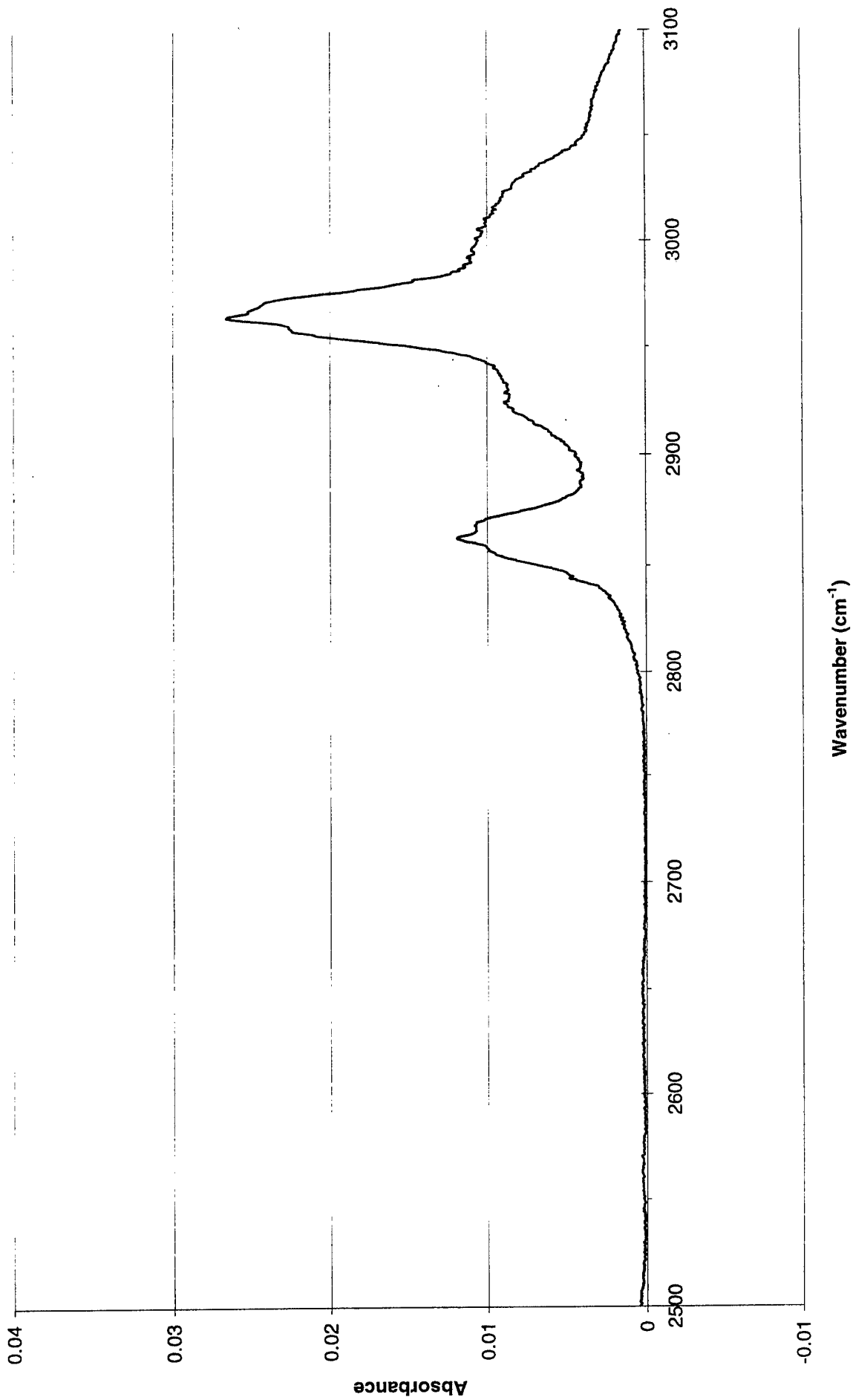


**QIRL0120 - Dimethyl phosphite**  
80 ppm-m



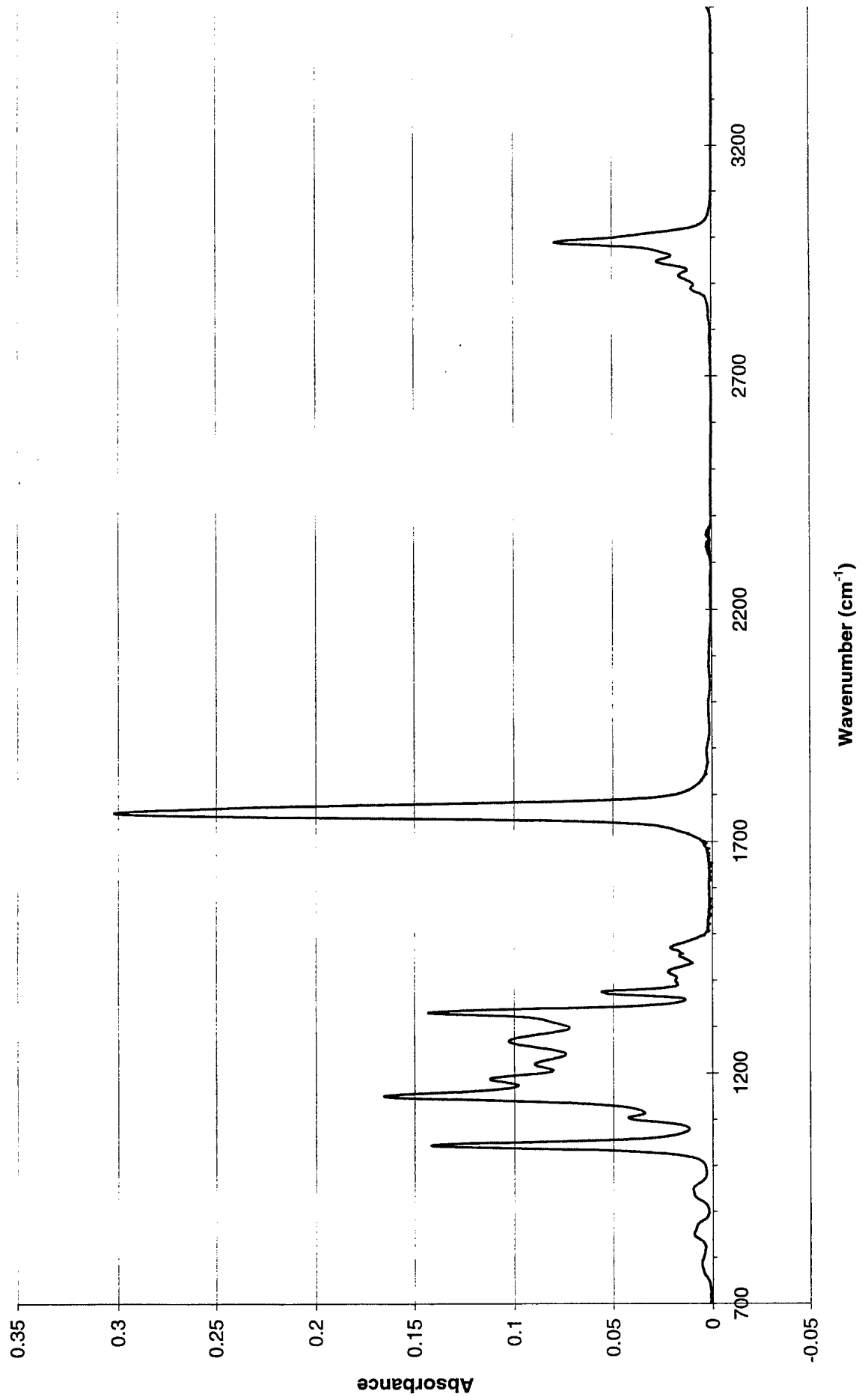


**QIRL0120 - Dimethyl phosphite**  
80 ppm-m





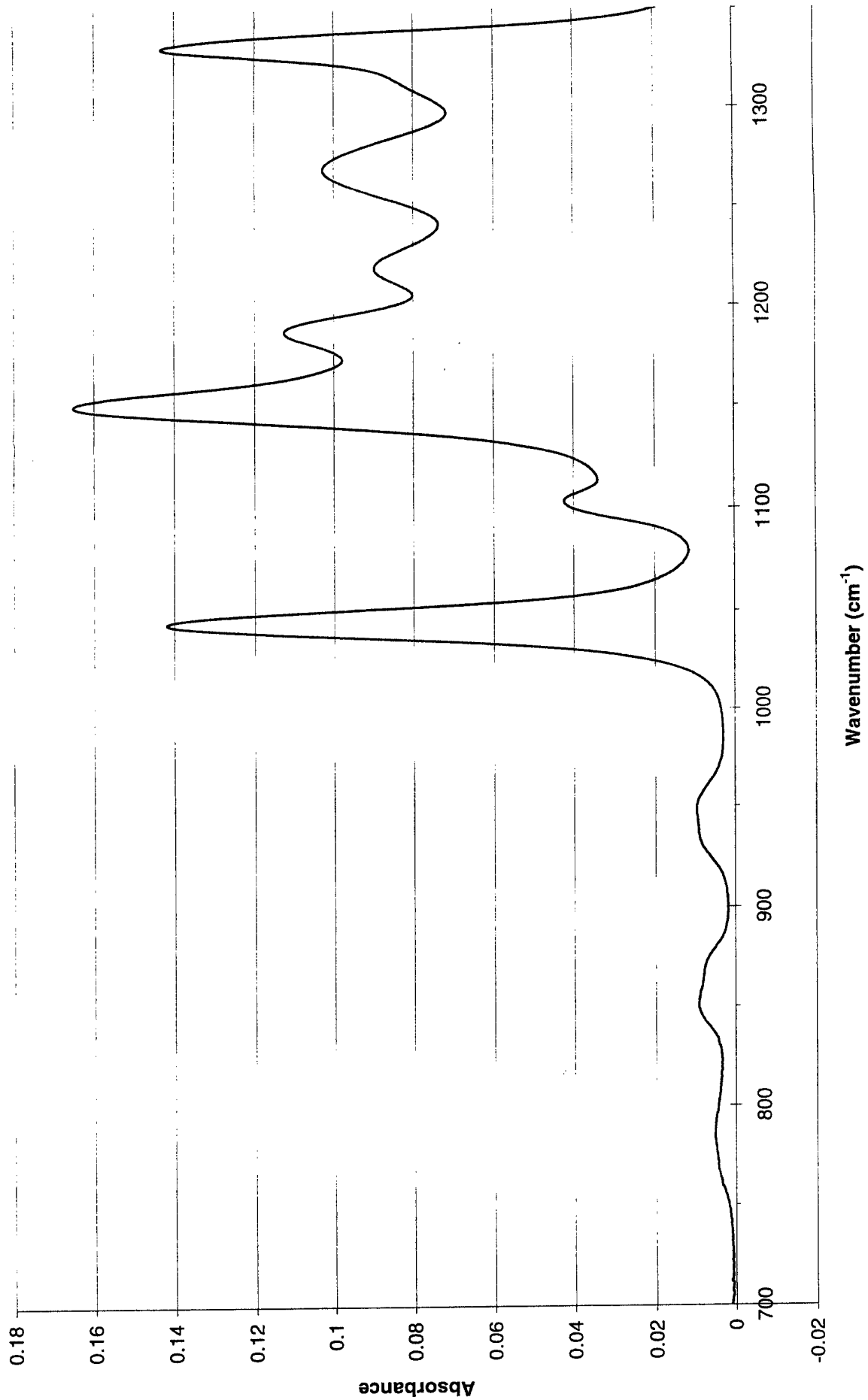
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103 ppm-m





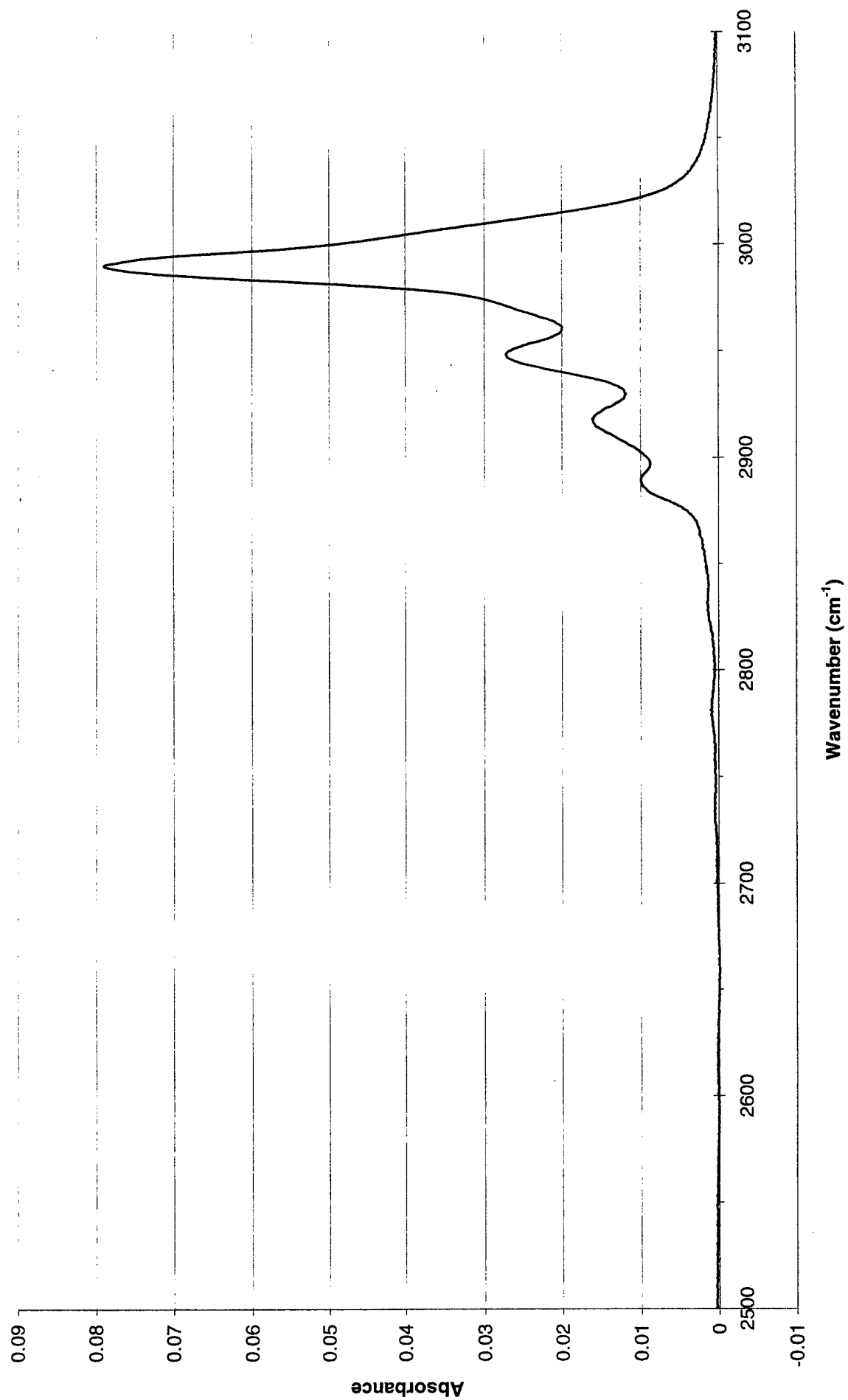
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103 ppm-m



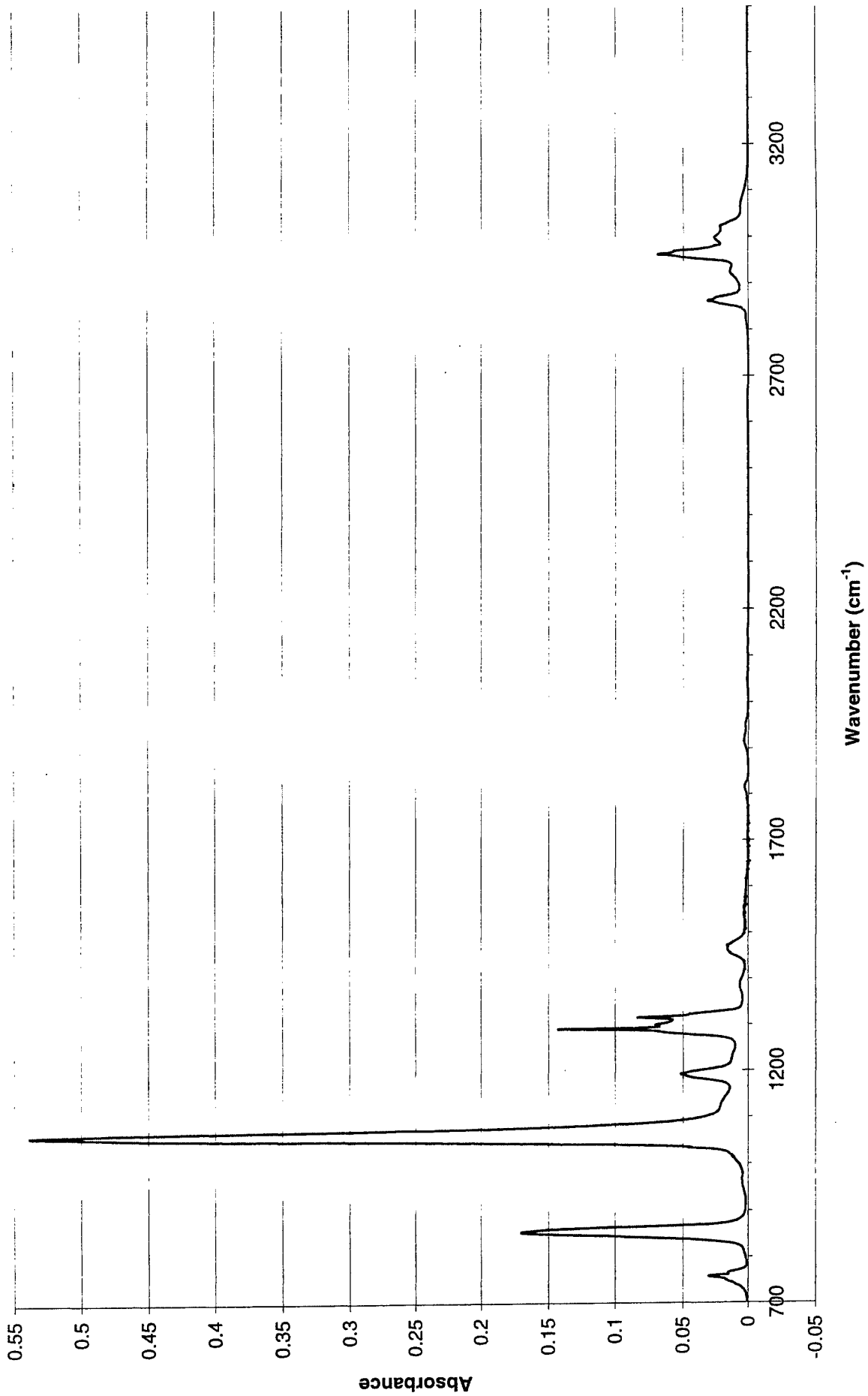


**QIRL0130 - Diethyl malonate (DEM)**  
103 ppm-m





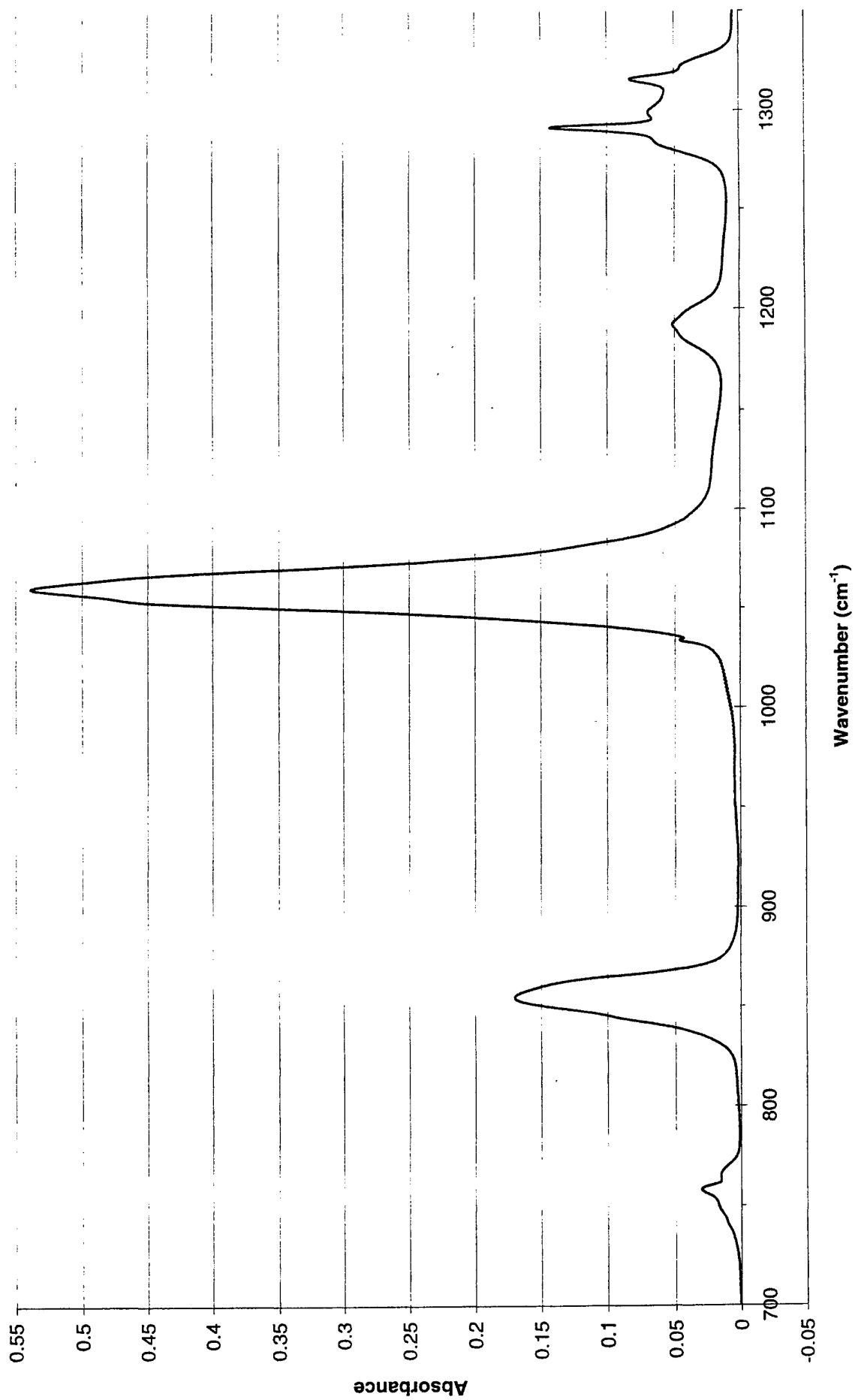
**QIRL0140 - Trimethyl phosphate (TMP)**  
97 ppm-m





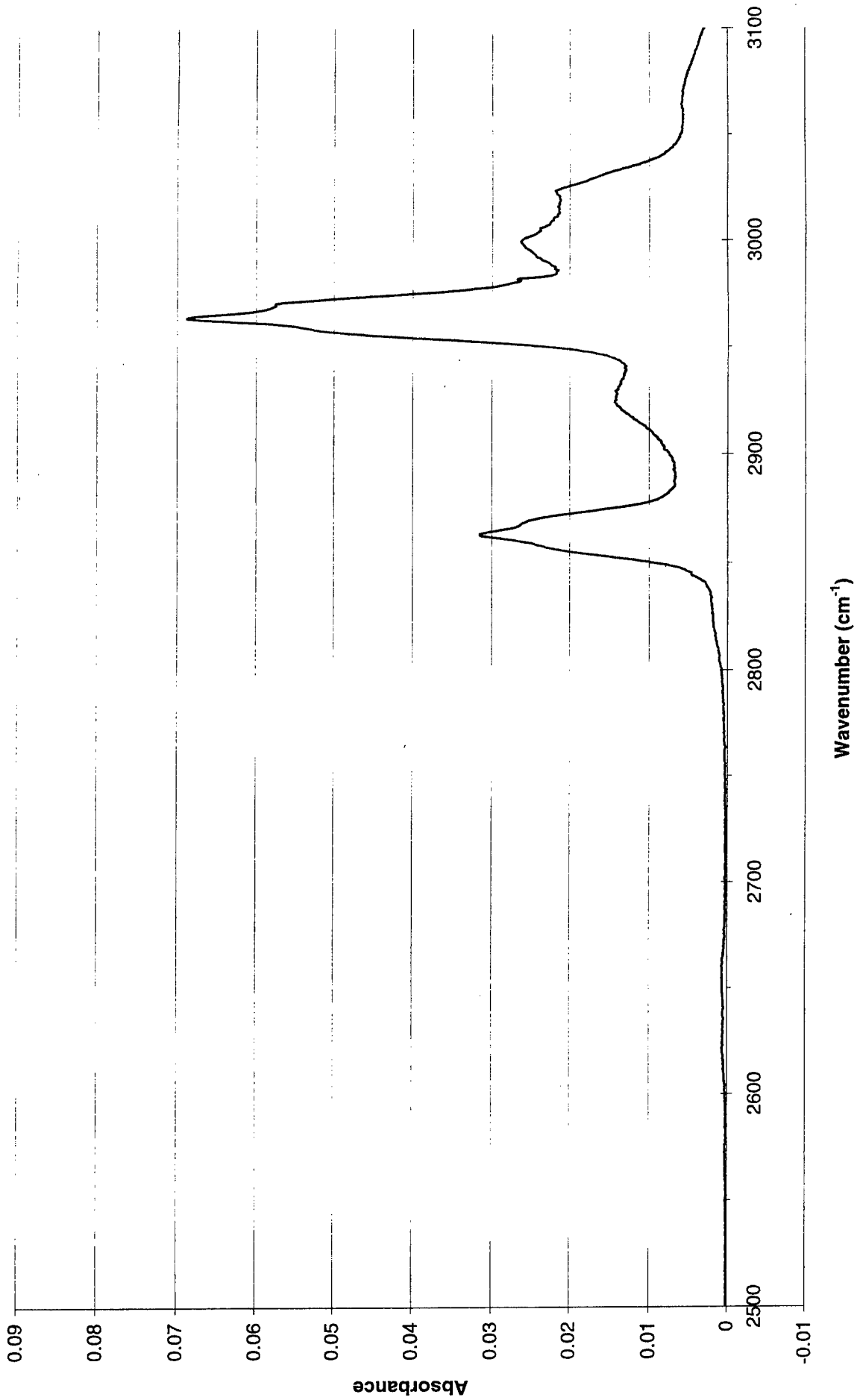
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97 ppm-m



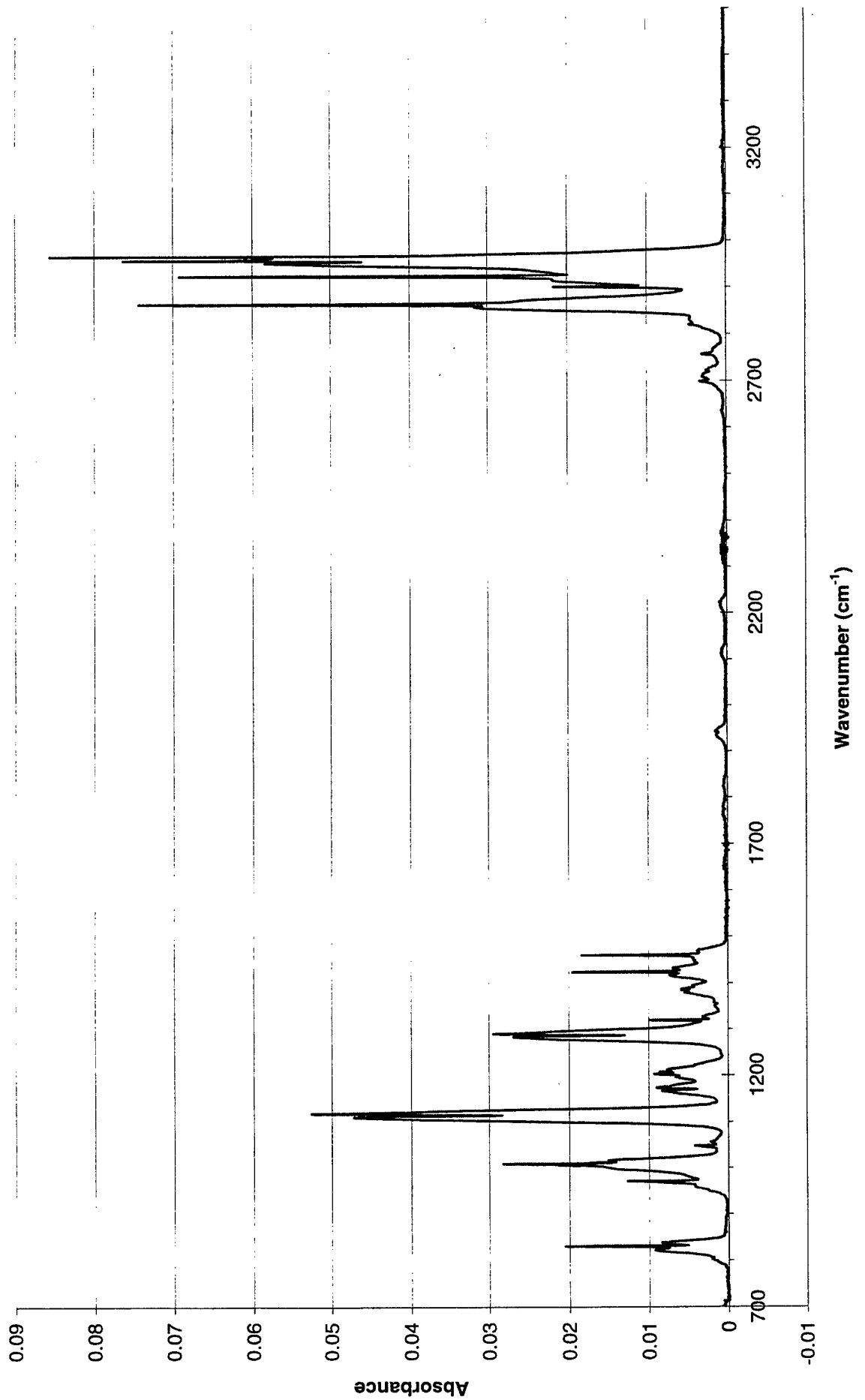


**QIRL0140 - Trimethyl phosphate (TMP)**  
97 ppm-m



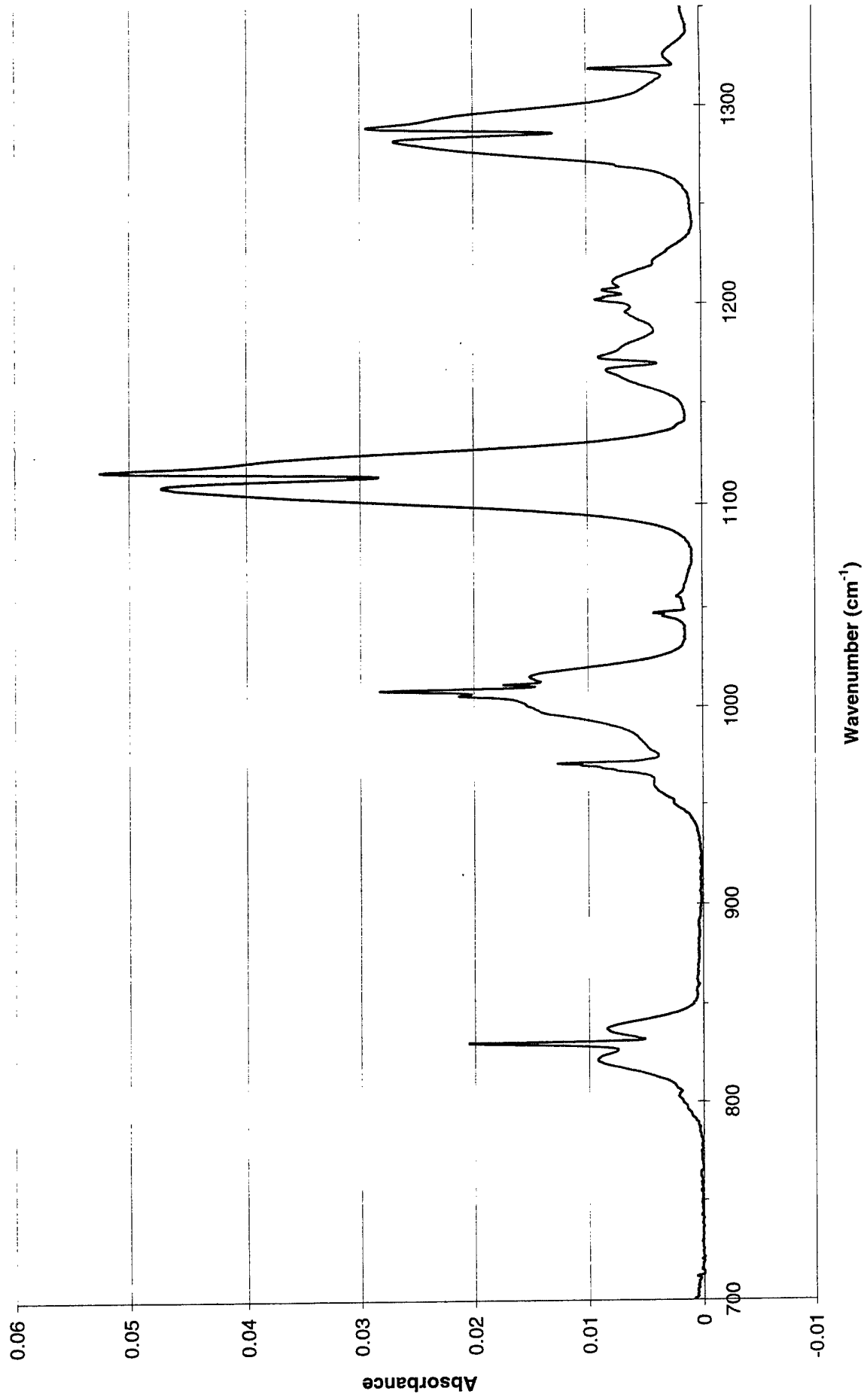


**QIRL0150 - 1,4 Thioxane**  
147 ppm-m



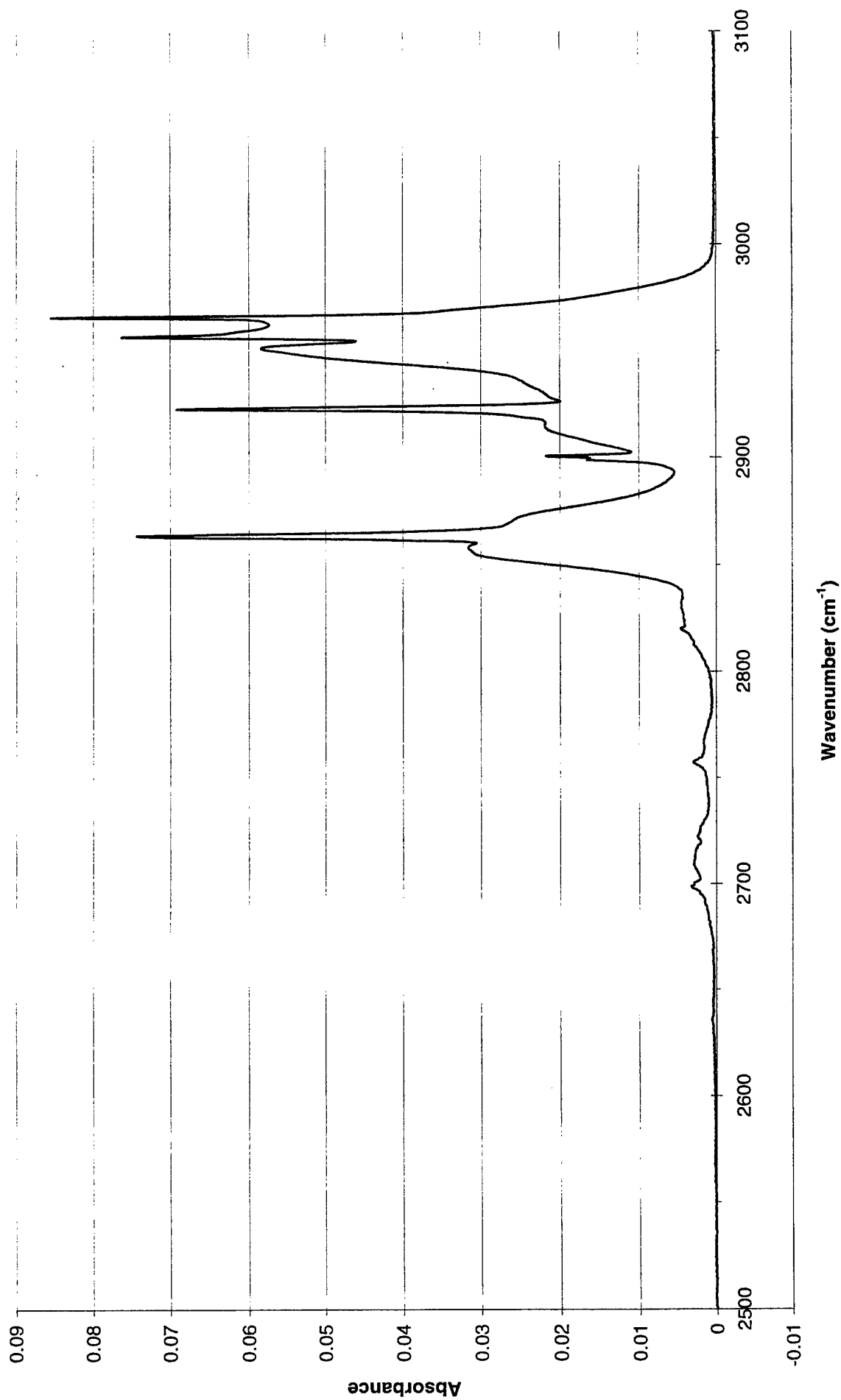


**QIRL0150 - 1,4 Thioxane**  
147 ppm-m





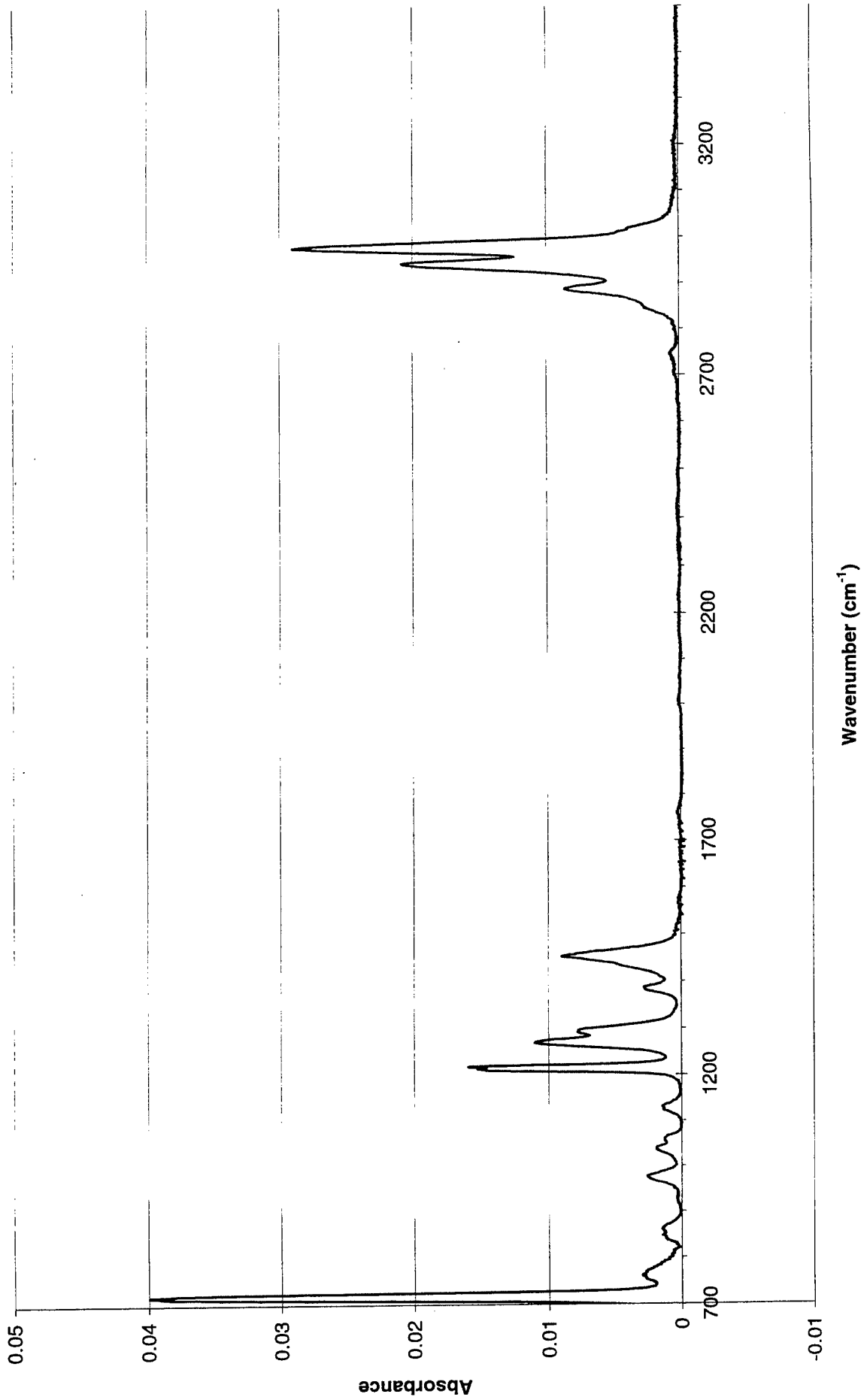
**QIRL0150 - 1,4 Thioxane**  
147 ppm-m





# QIRL0160 - 2-Chloroethyl ethyl sulfide (CEES)

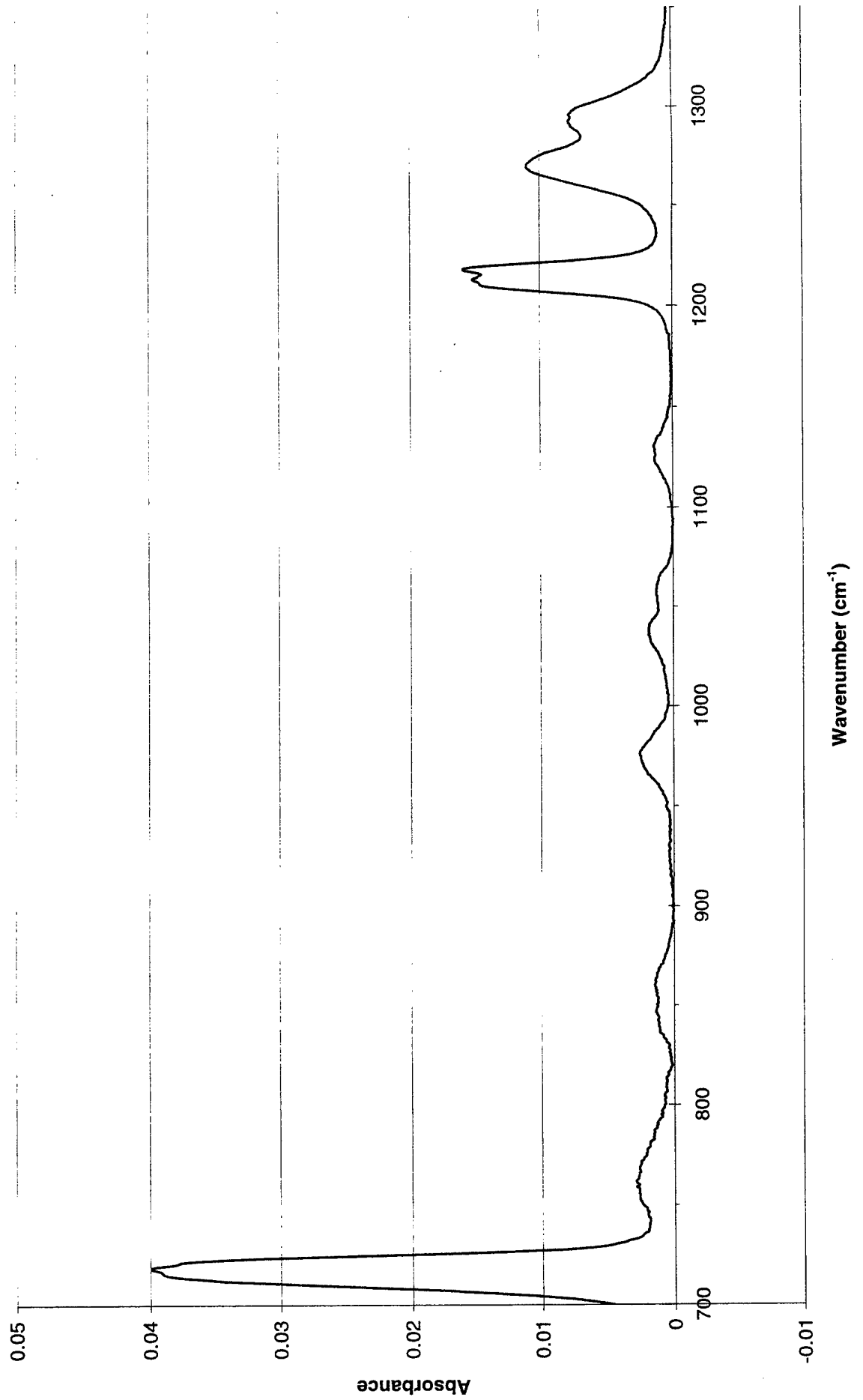
98 ppm-m





# QIRL0160 - 2-Chloroethyl ethyl sulfide (CEES)

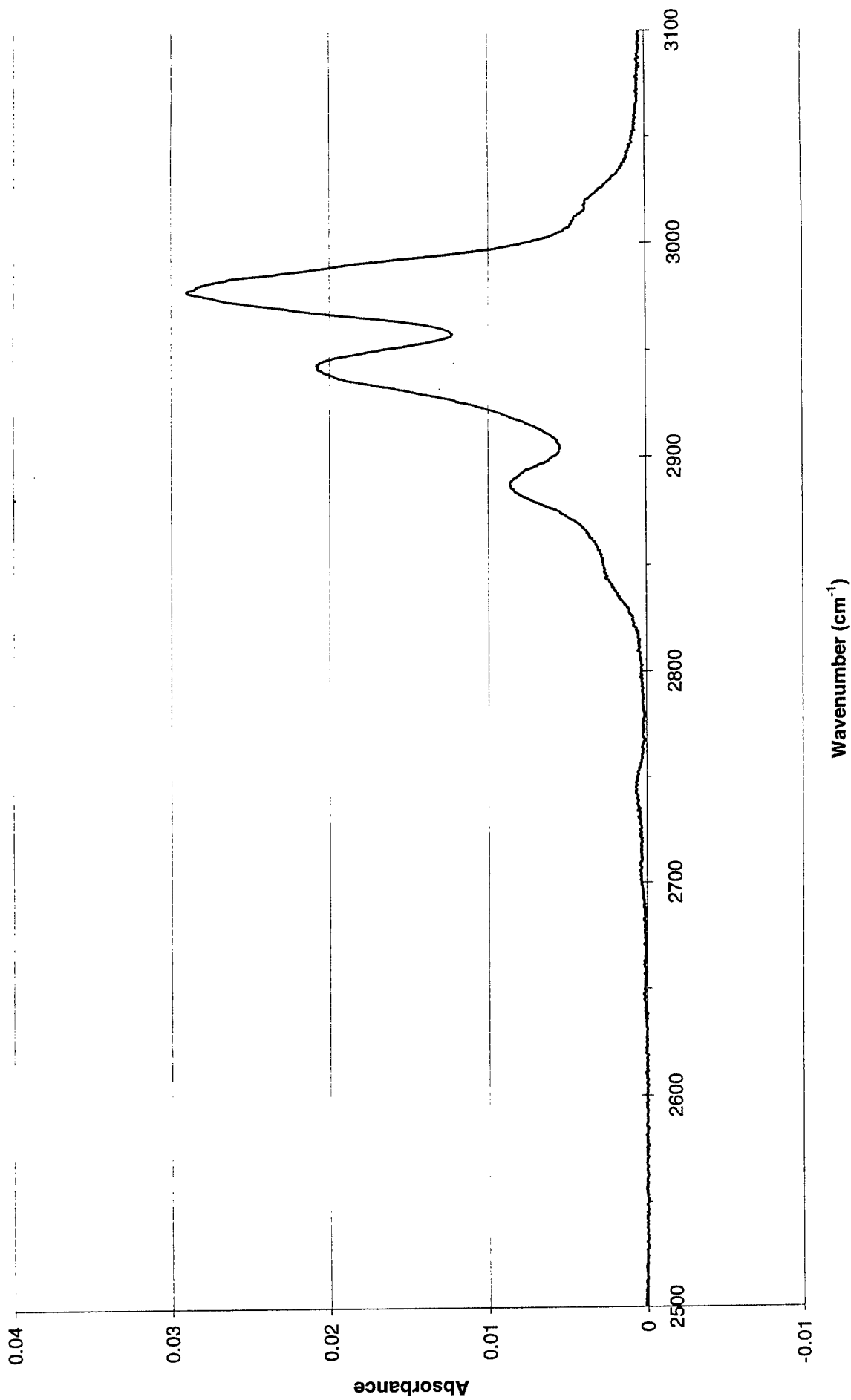
98 ppm-m





# QIRL0160 - 2-Chloroethyl ethyl sulfide (CEES)

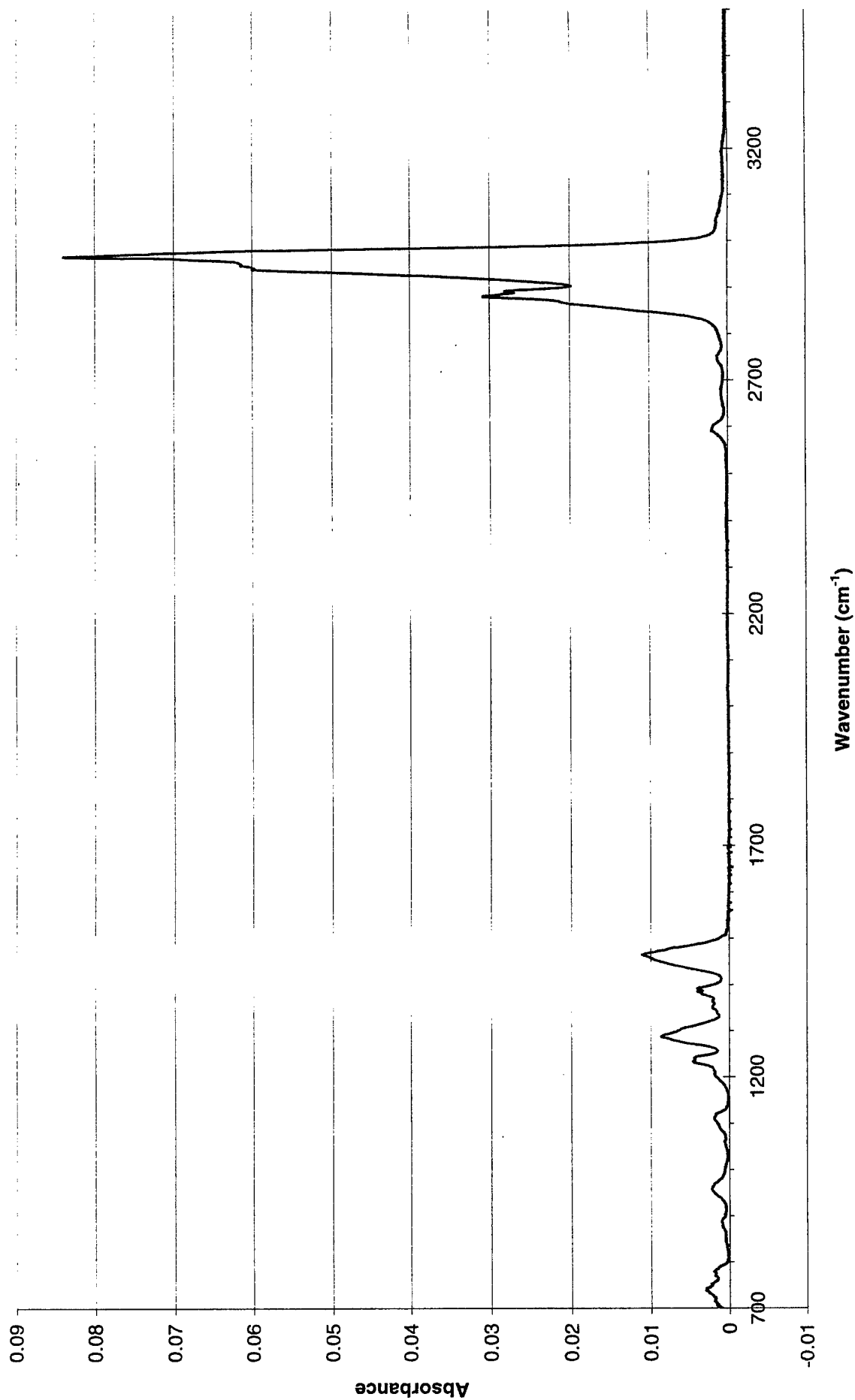
98 ppm-m



Wavenumber (cm<sup>-1</sup>)

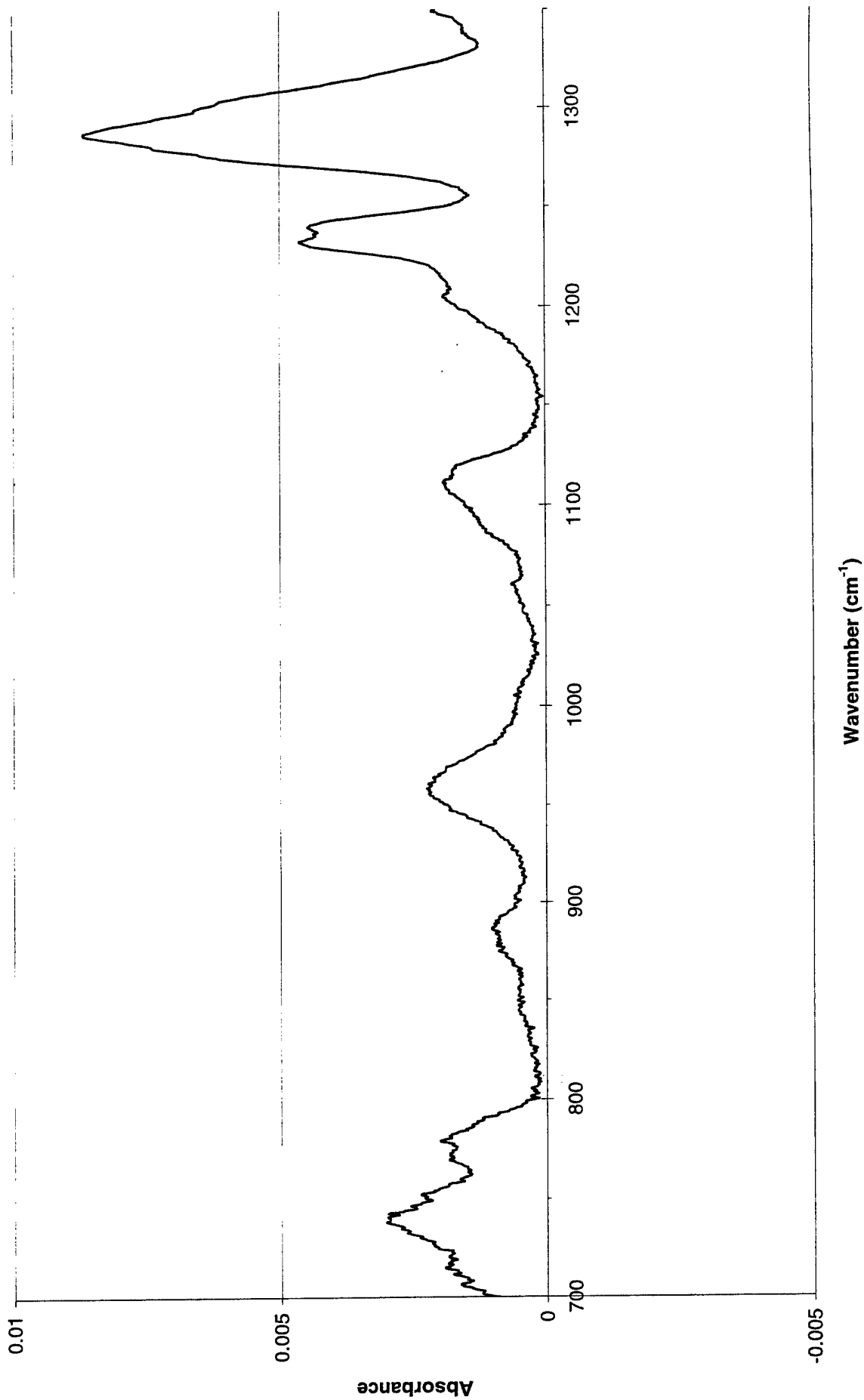


**QIRL0170 - n-Butyl mercaptan (BUSH)**  
156 ppm-m



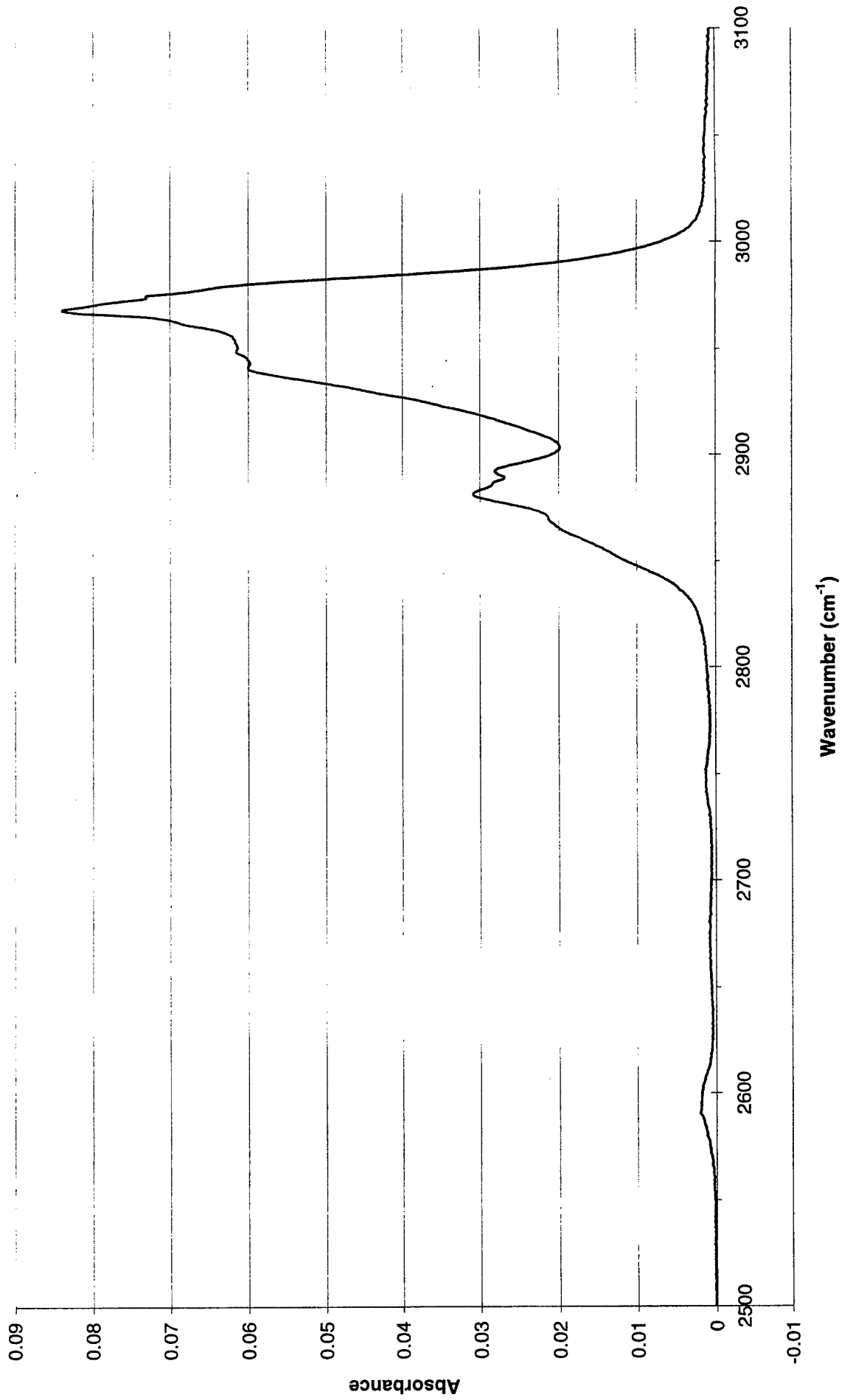


**QIRL0170 - n-Butyl mercaptan (BUSH)**  
156 ppm-m



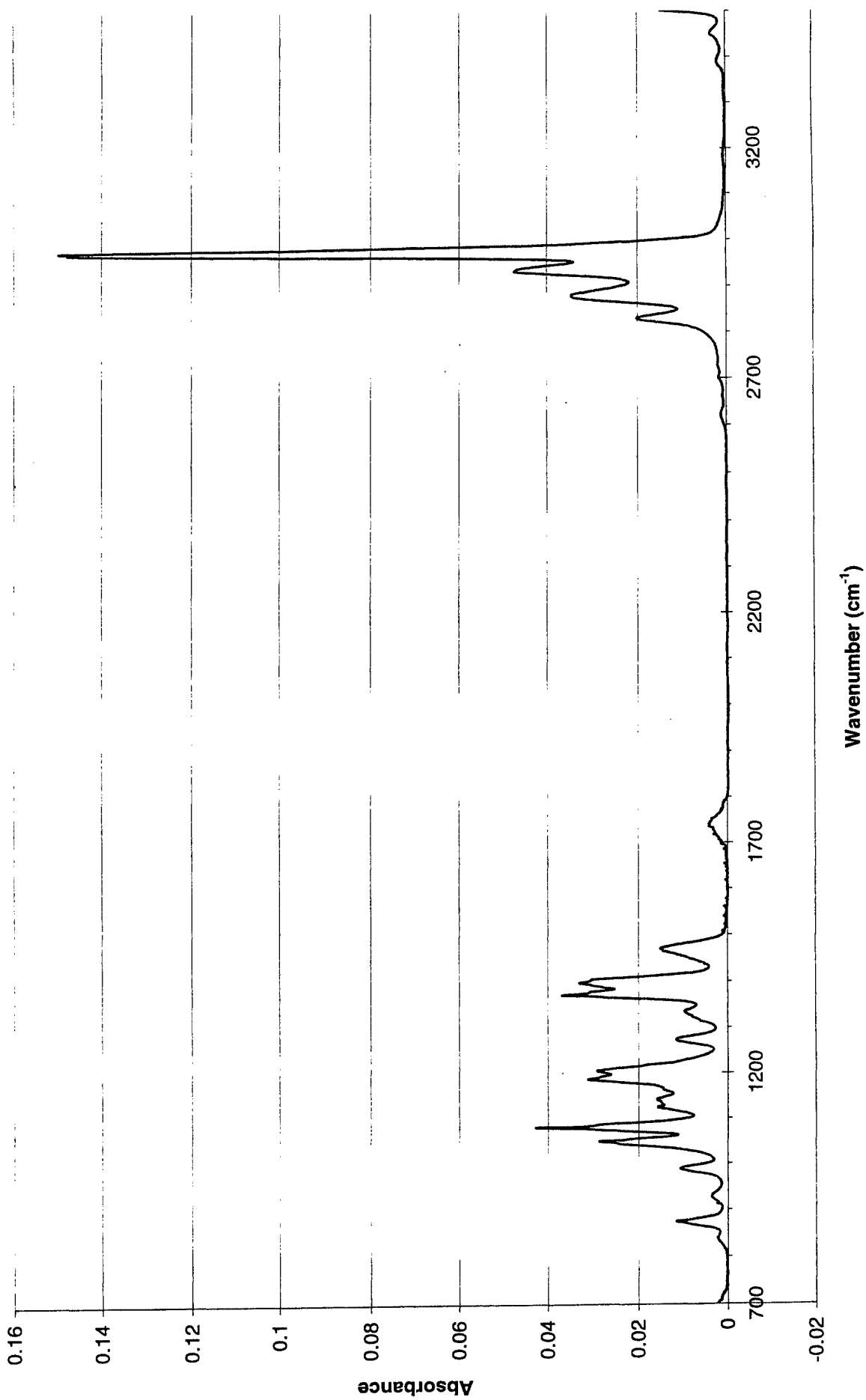


**QIRL0170 - n-Butyl mercaptan (BUSH)**  
156 ppm-m



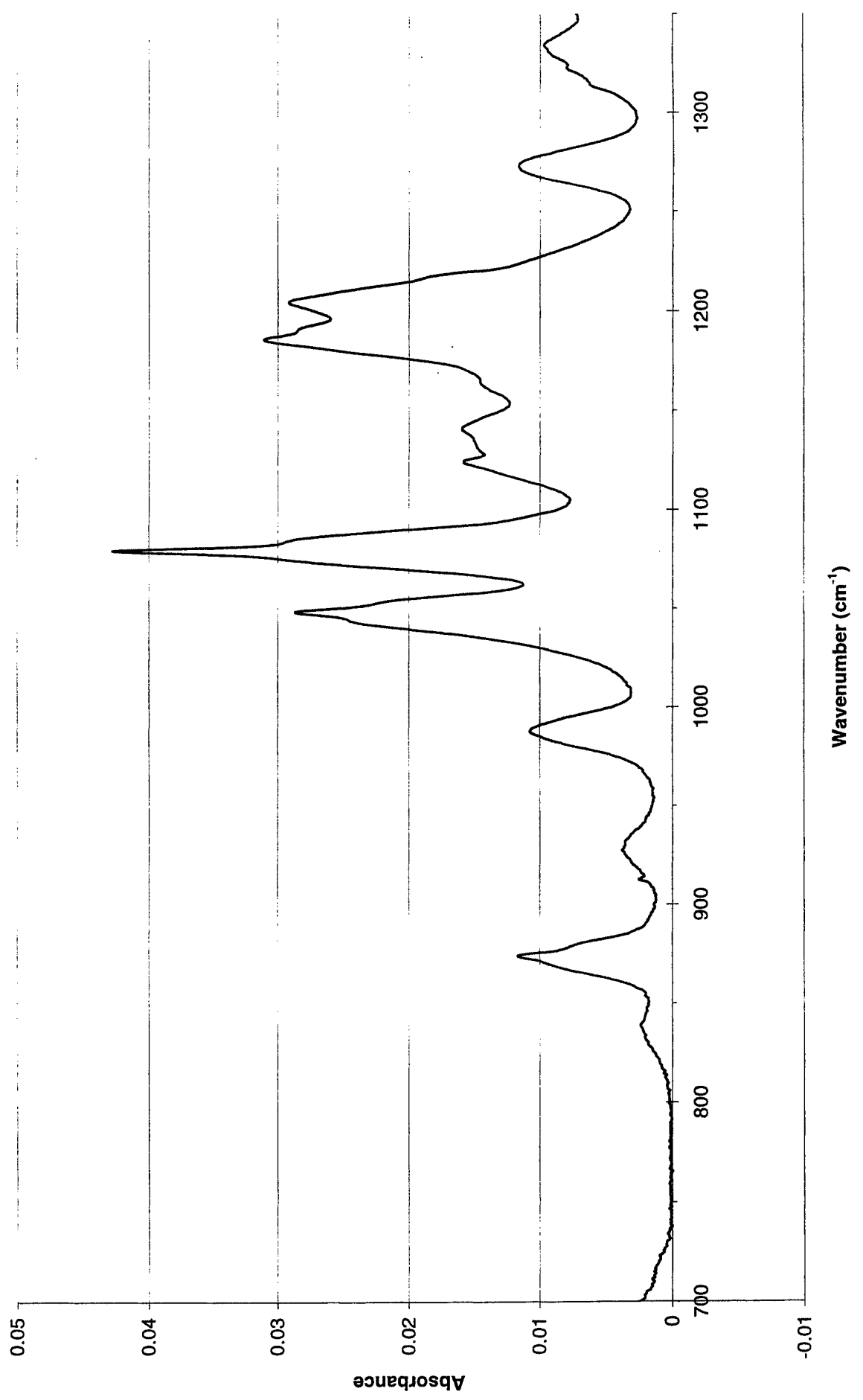


**QIRL0180 - 2-(Diisopropylamino) ethanol**  
97 ppm-m



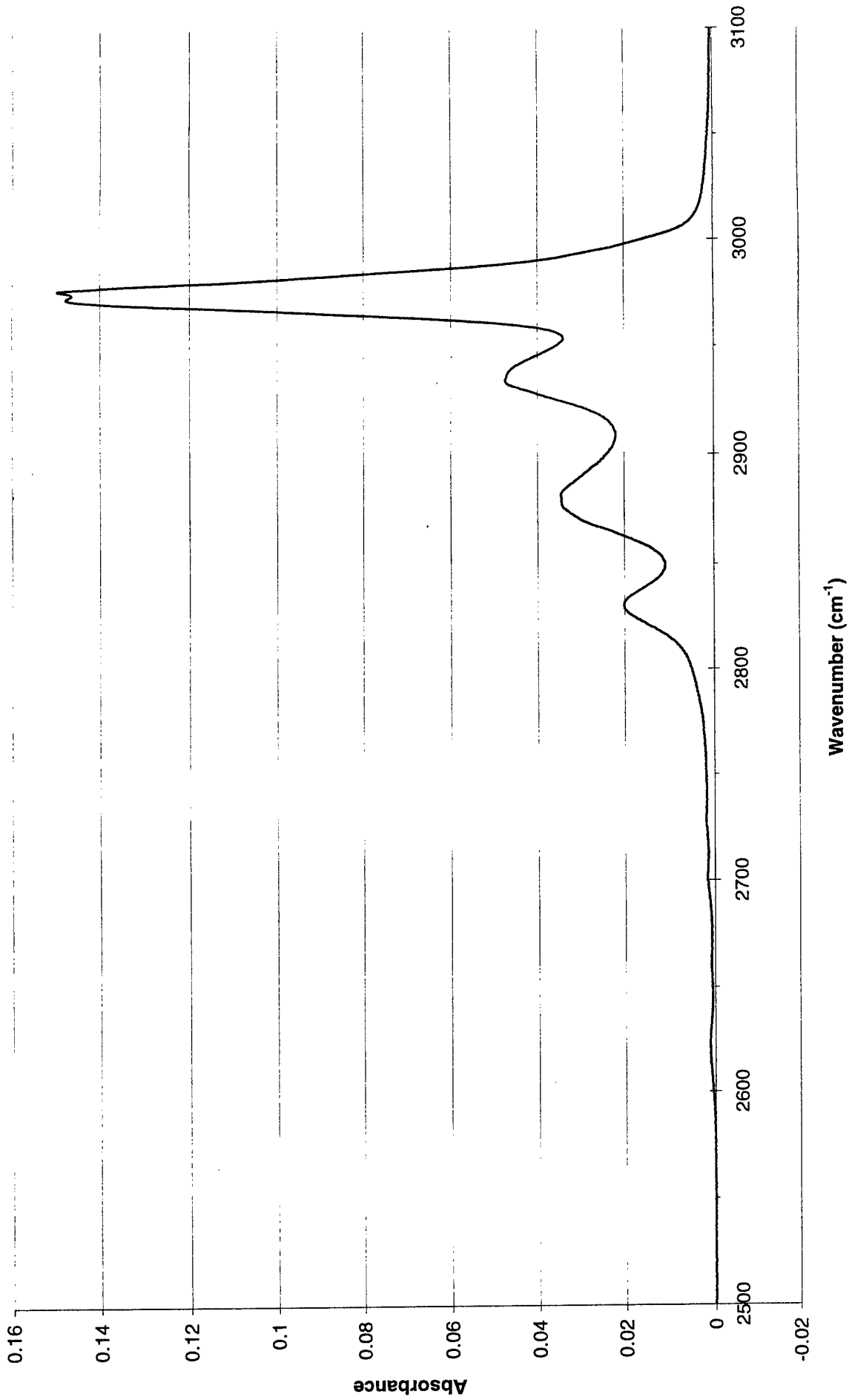


**QIRL0180 - 2-(Diisopropylamino) ethanol**  
97 ppm-m



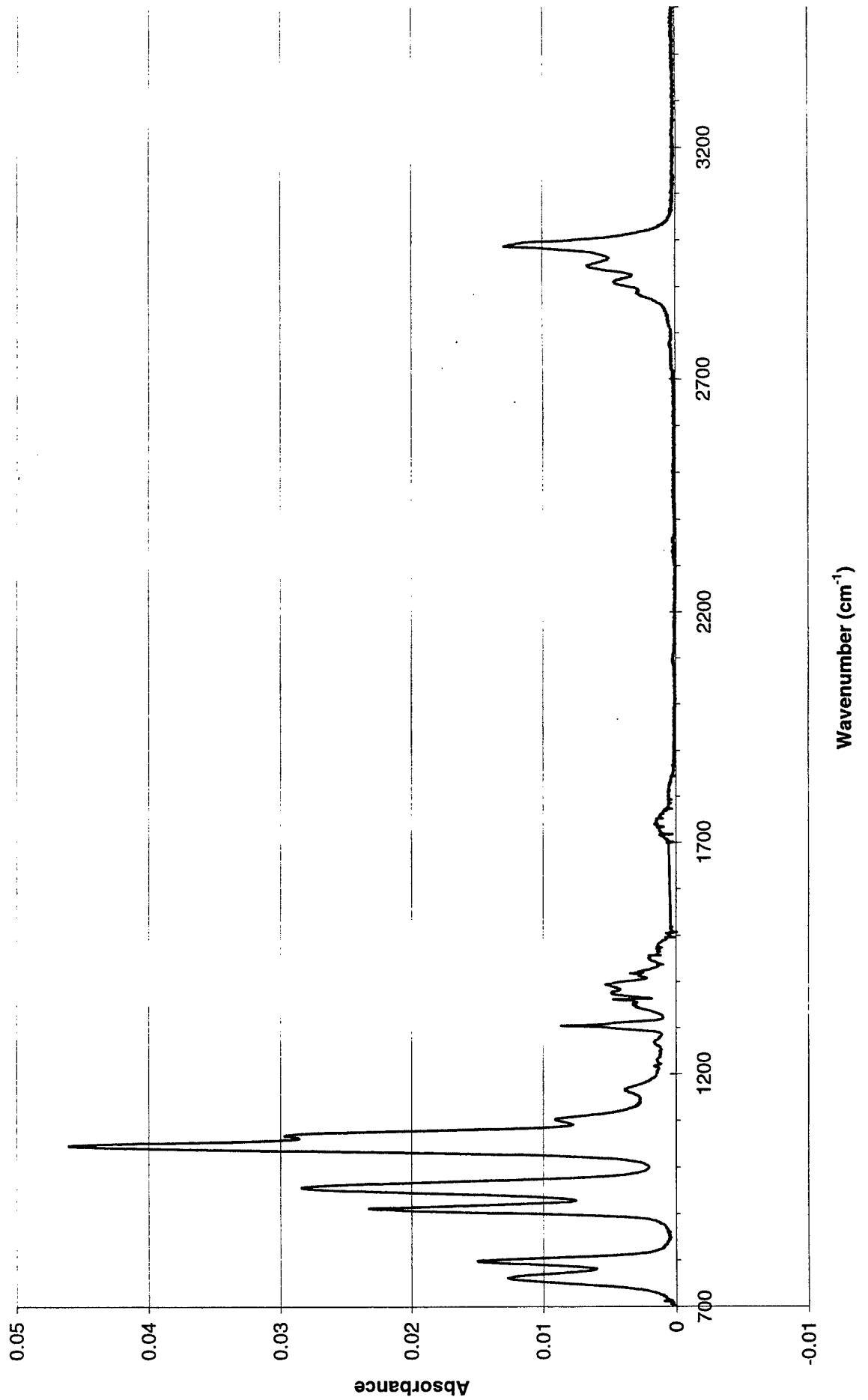


**QIRL0180 - 2-(Diisopropylamino) ethanol**  
97 ppm-m



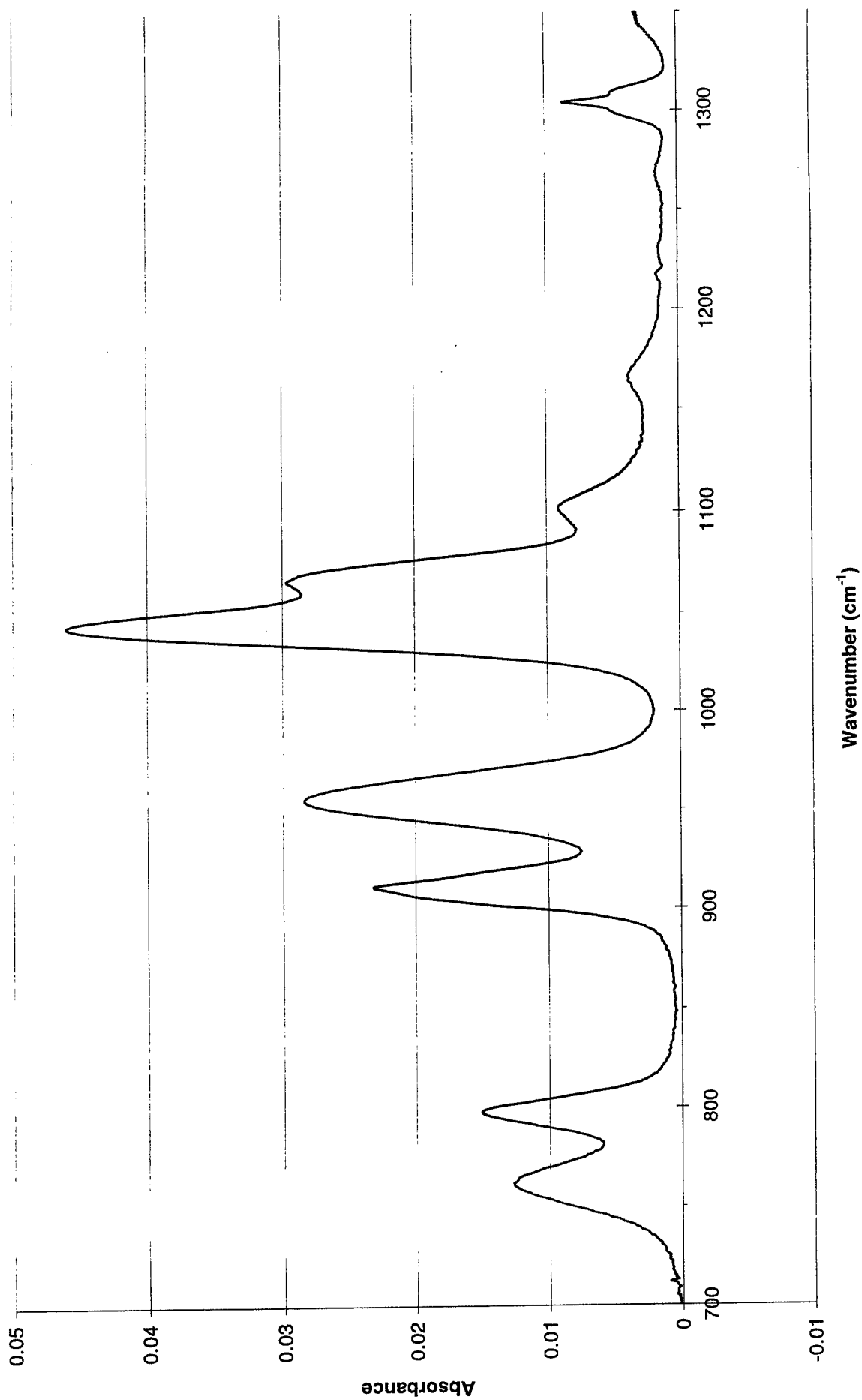


**QIRL0190 - O,O-Diethyl methylphosphonothionate**  
25 ppm-m



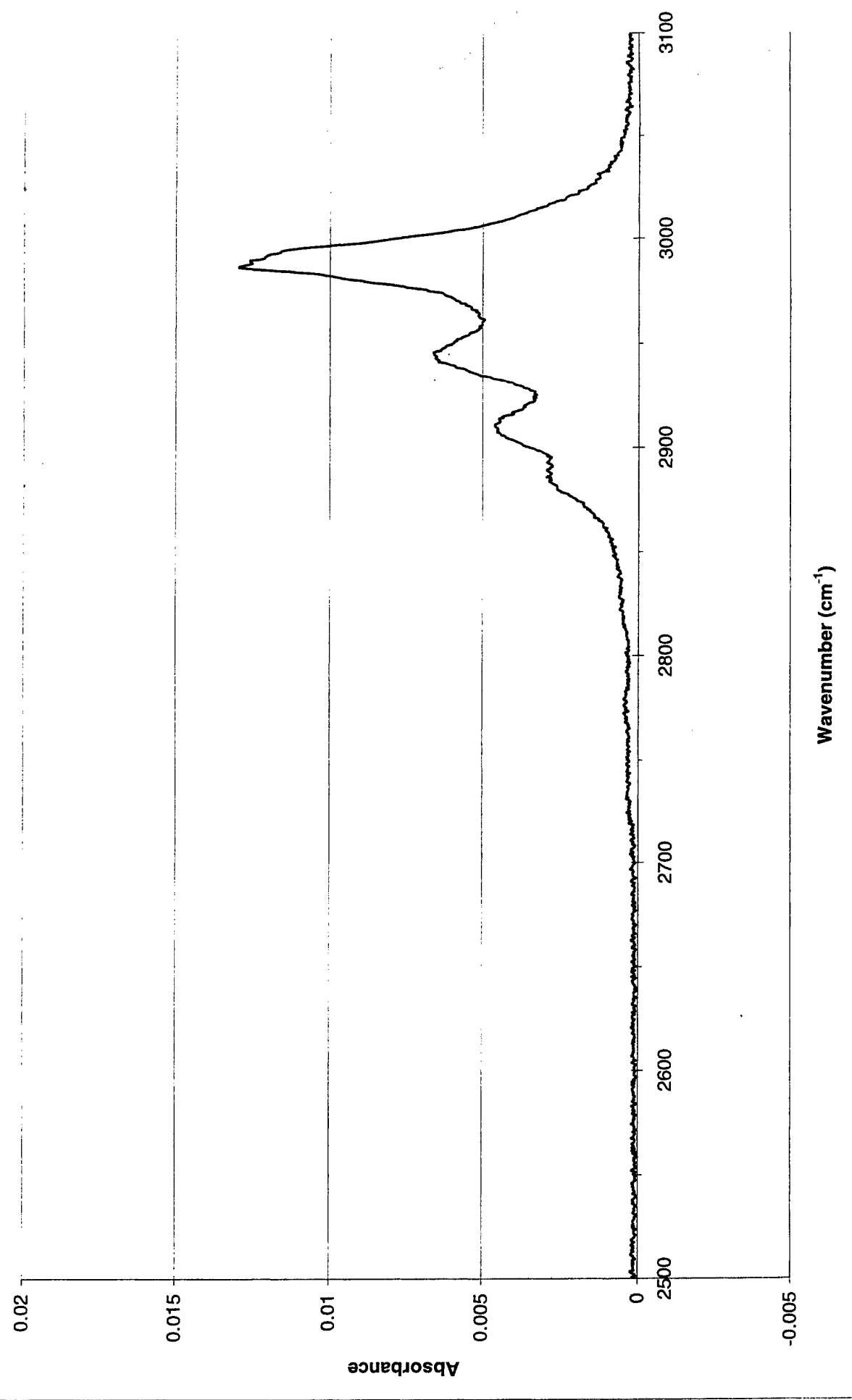


**QIRL0190 - O,O-Diethyl methylphosphonothionate**  
25 ppm-m



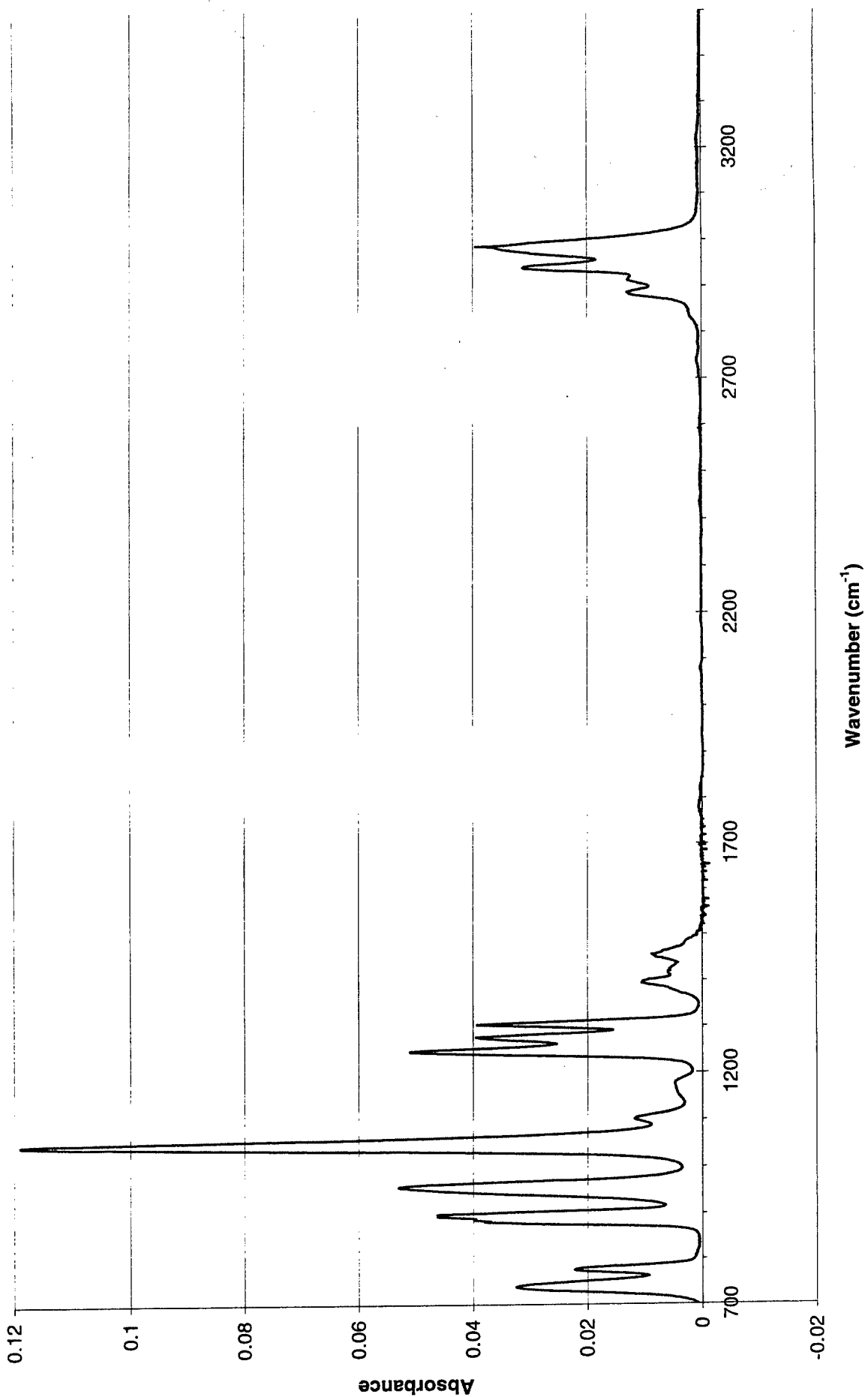


**QIRL0190 - O,O-Diethyl methylphosphonothionate**  
25 ppm-m



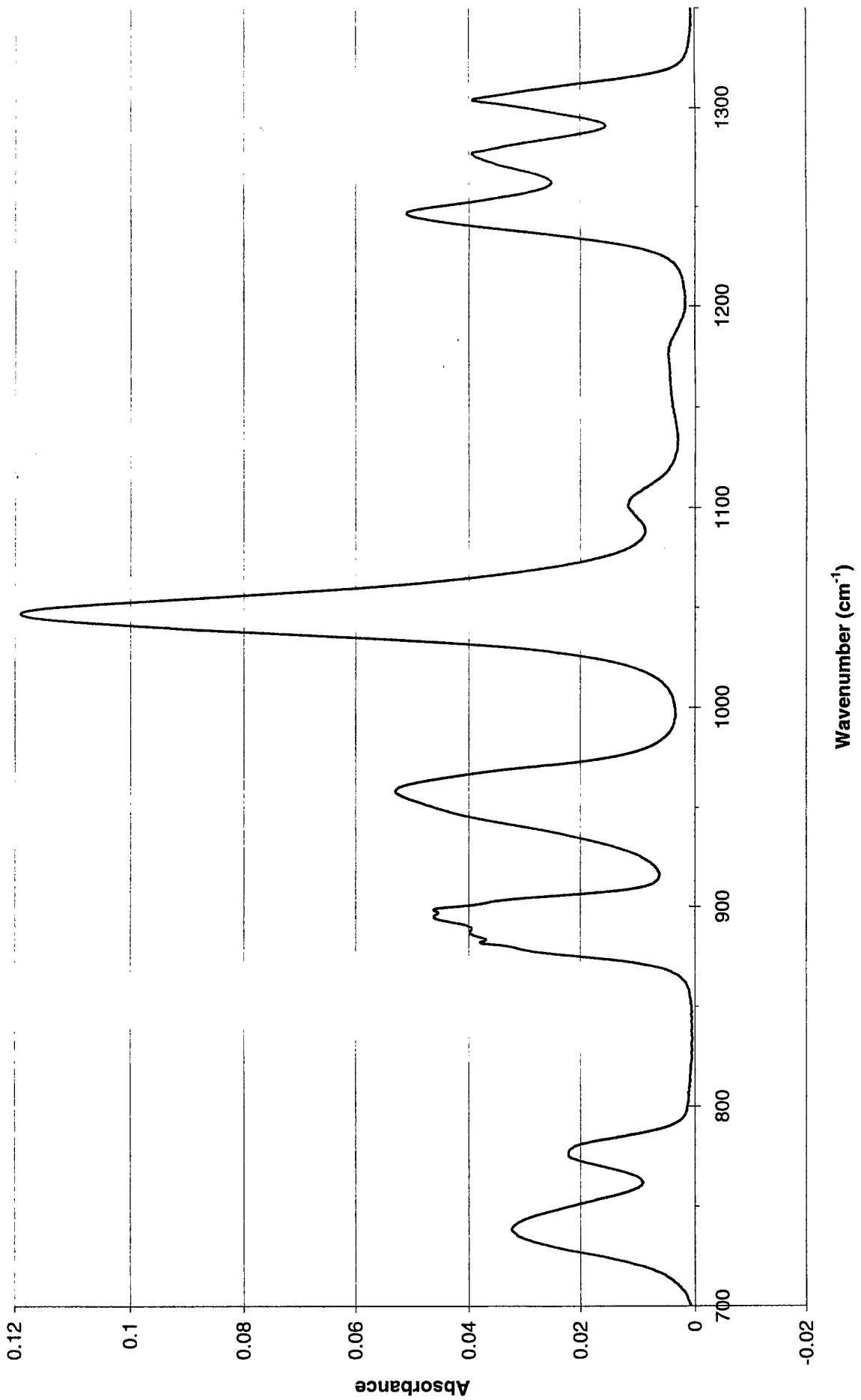


**QIRL0200 - O,S-Diethyl methylphosphonothionate**  
74 ppm-m





**QIRL0200 - O,S-Diethyl methylphosphonothionate**  
74 ppm-m





**QIRL0200 - O,S-Diethyl methylphosphonothionate**  
74 ppm-m

