



EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

U.S. ARMY SOLDIER AND BIOLOGICAL CHEMICAL COMMAND

ECBC-TR-360

**QUANTITATIVE INFRARED REFERENCE LIBRARY
VOLUME II**

**John Ditillo
Christopher C. Keiser**

CB SERVICES DIRECTORATE

Barry R. Williams

**EAI CORPORATION
Abingdon, MD 21009**



December 2003

Approved for public release;
distribution is unlimited.



20040317 154

Aberdeen Proving Ground, MD 21010-5424

Disclaimer

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

REPORT DOCUMENTATION PAGEForm Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY) XX-12-2003		2. REPORT TYPE Final		3. DATES COVERED (From - To) Jun 2002 - Dec 2002							
4. TITLE AND SUBTITLE Quantitative Infrared Reference Library Volume II				5a. CONTRACT NUMBER							
				5b. GRANT NUMBER							
				5c. PROGRAM ELEMENT NUMBER 2VJRZZ							
6. AUTHOR(S) Ditillo, John; Keiser, Christopher C. (ECBC); and Williams, Barry R. (EAI CORPORATION)				5d. PROJECT NUMBER							
				5e. TASK NUMBER							
				5f. WORK UNIT NUMBER							
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) AND ADDRESS(ES) DIR, ECBC, ATTN: AMSRD-ECB-CB-C, APG, MD 21010-5424 EAI CORPORATION, 1308 Continental Drive, Abingdon, MD 21009				8. PERFORMING ORGANIZATION REPORT NUMBER ECBC-TR-360							
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)							
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)							
12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.											
13. SUPPLEMENTARY NOTES											
14. ABSTRACT Recent technology developments in Fourier Transform Infrared (FTIR) spectroscopy (such as stand-off infrared (IR) detection of chemical agents and related compounds for monitoring applications) has created a need for condensed and vapor phase FTIR databases, which use current technology. Old IR 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 IR spectra complications do not adequately address militarily significant or directly related compounds. 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.											
15. SUBJECT TERMS <table border="0"><tr><td>Chemical warfare material</td><td>Vapor phase spectra</td></tr><tr><td>Chemical warfare agents</td><td>Infrared spectra</td></tr><tr><td>Infrared spectroscopy</td><td>FTIR</td></tr></table>						Chemical warfare material	Vapor phase spectra	Chemical warfare agents	Infrared spectra	Infrared spectroscopy	FTIR
Chemical warfare material	Vapor phase spectra										
Chemical warfare agents	Infrared spectra										
Infrared spectroscopy	FTIR										
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Sandra J. Johnson						
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (include area code) (410) 436-2914						

Blank

PREFACE

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

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

This report has been approved for public release. Registered users should request additional copies from the Defense Technical Information Center; unregistered users should direct such requests to the National Technical Information Center.

Blank

CONTENTS

1.	INTRODUCTION	7
2.	OBJECTIVE.....	7
3.	DESCRIPTION	7
	APPENDIX – EDGEWOOD CHEMICAL BIOLOGICAL CENTER QIRL DATABASE.....	9

Blank

QUANTITATIVE INFRARED REFERENCE LIBRARY

VOLUME II

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 $\frac{1}{4}$ " 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⁻¹ 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

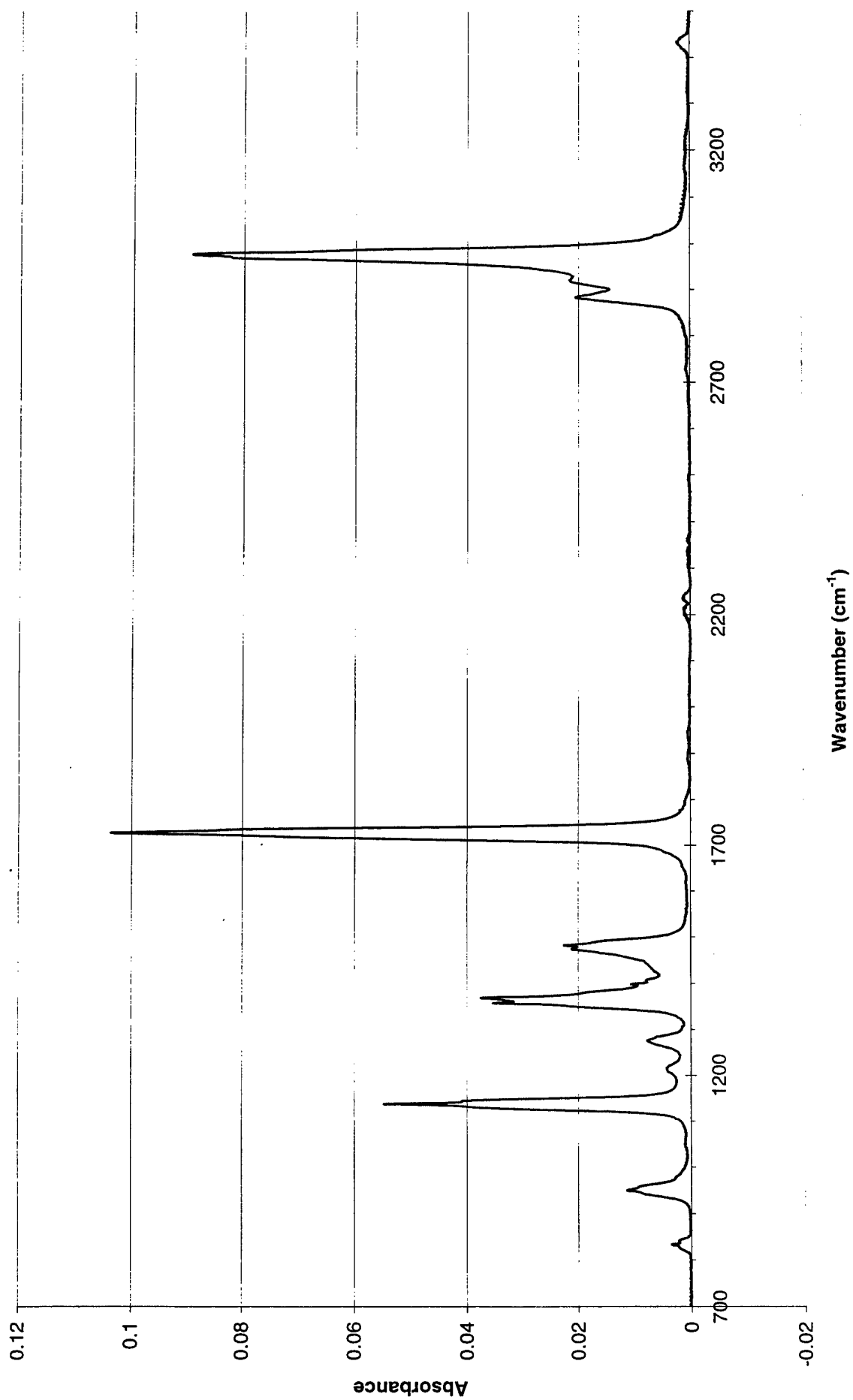
EDGEWOOD CHEMICAL BIOLOGICAL CENTER
QIRL DATABASE

Edgewood Chemical Biological Center
QIRL Database

QIRL Filename	Compound Name	CAS	Molecular Formula	CL (ppm-m)
QIRL0210	Pinacolone	75-97-8	(CH ₃) ₃ CCCOCH ₃	488
QIRL0220	Pinacolyl Alcohol	464-07-3	(CH ₃) ₃ CCCHOHCH ₃	170
QIRL0230	Diethyl Ethylphosphonate	78-38-6	(CH ₃ CH ₂ O) ₂ P(O)CH ₂ CH ₃	37
QIRL0240	N-Methyldiethanolamine	105-59-9	(HOCH ₂) ₂ NCH ₃	10
QIRL0250	Cyanogen Chloride (CK)	506-77-4	CCIN	343
QIRL0260	2-Furaldehyde	98-01-1	C ₅ H ₄ O ₂	74
QIRL0270	Trichloronitromethane (PS)	76-06-02	CCl ₃ NO ₂	177
QIRL0280	2-(Diisopropylamino) ethanethiol	5842-07-9	C ₆ H ₁₄ NCH ₂ CH ₂ SH	20
QIRL0290	2-(Diethylamino) ethanethiol	100-38-9	C ₄ H ₁₀ NCH ₂ CH ₂ SH	72
QIRL0300	2-(Diethylamino) ethanol	100-37-8	C ₄ H ₁₀ NCH ₂ CH ₂ OH	173
QIRL0310	Benzyl alcohol	100-51-6	C ₆ H ₅ CH ₂ OH	84
QIRL0320	Diisopropyl fluorophosphate	55-91-4	(C ₃ H ₇ O) ₂ P(O)F	104
QIRL0330	Diethyl pimelate	2050-20-6	C ₂ H ₅ O ₂ C(CH ₂) ₆ O ₂ C ₂ H ₅	10
QIRL0340	Allyl isothiocyanate	57-06-7	C ₂ CHCH ₂ CNS	128
QIRL0350	Benzyl bromide	100-39-0	C ₆ H ₅ CH ₂ Br	119
QIRL0360	Ethyl chloroacetate	105-39-5	ClCH ₂ CO ₂ C ₂ H ₅	103
QIRL0370	2-Butenal	123-73-9	CH ₃ CHCHCHO	158
QIRL0380	Ethyl chloroformate	541-41-3	ClCO ₂ C ₂ H ₅	209
QIRL0390	Ethyl chloroethiolformate	2941-64-2	ClCOSC ₂ H ₅	128
QIRL0400	Bromodichloromethane	75-27-4	CHBrCl ₂	105

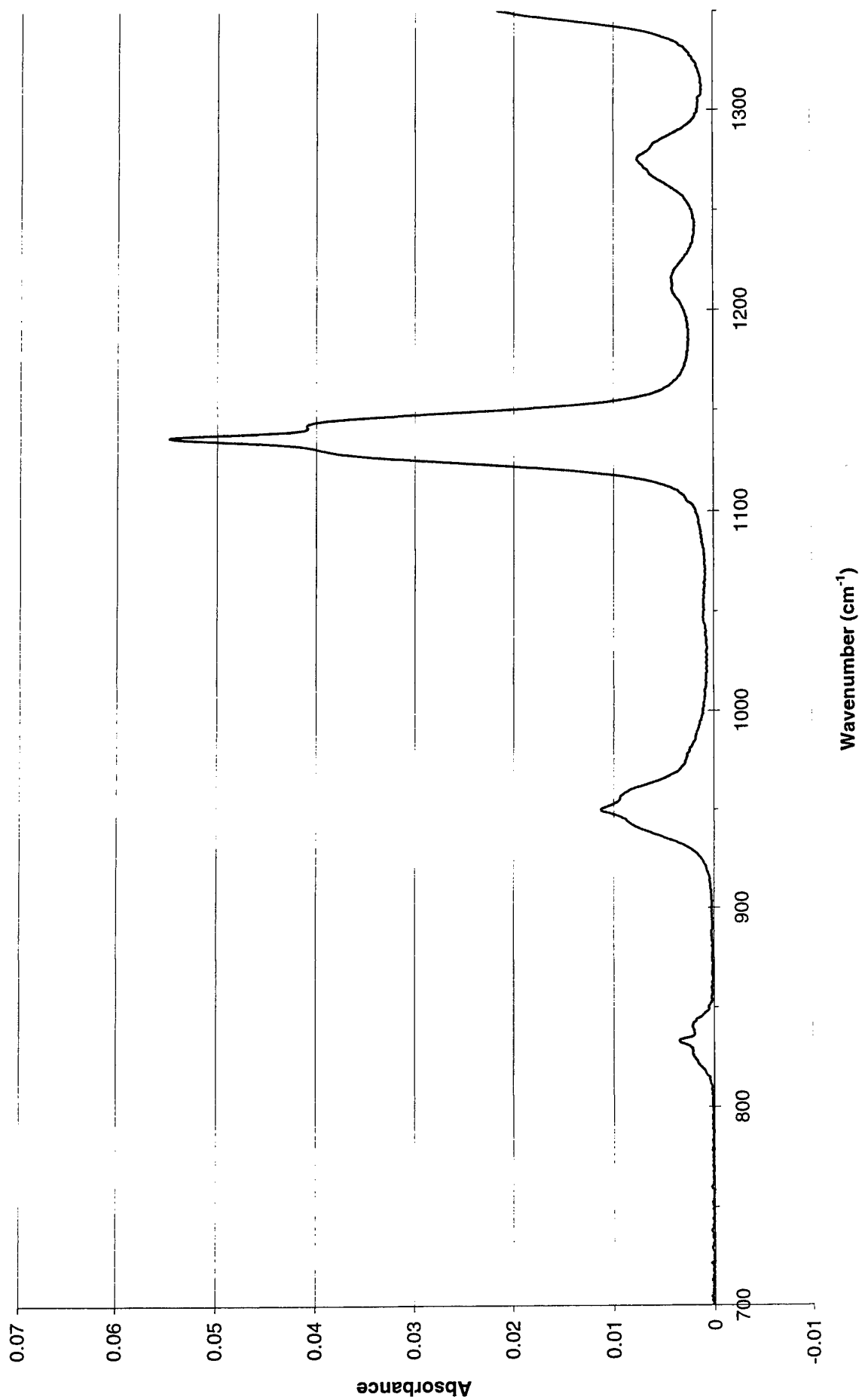


QIRL0210 - Pinacolone
488 ppm-m



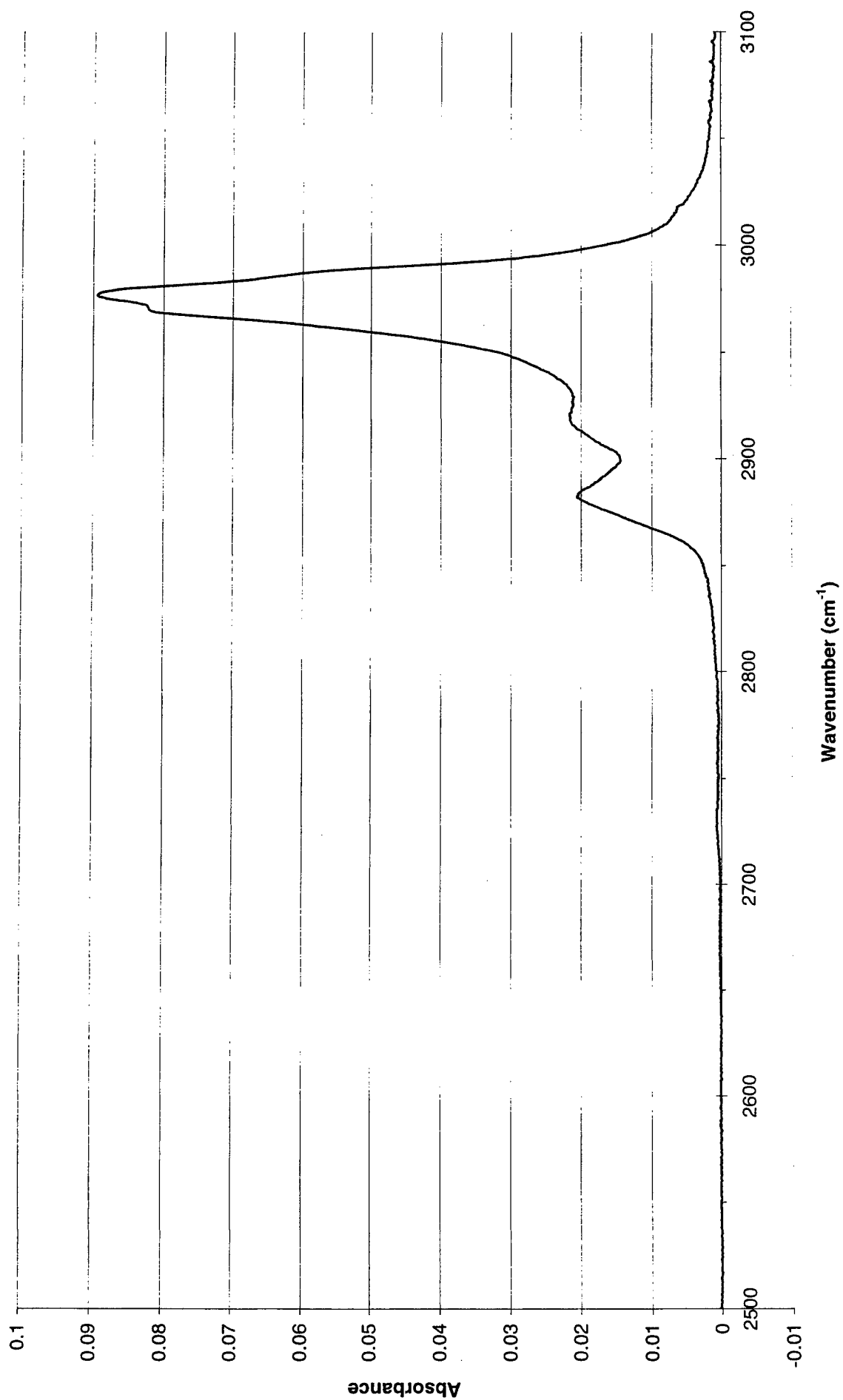


QIRL0210 - Pinacolone
488 ppm-m



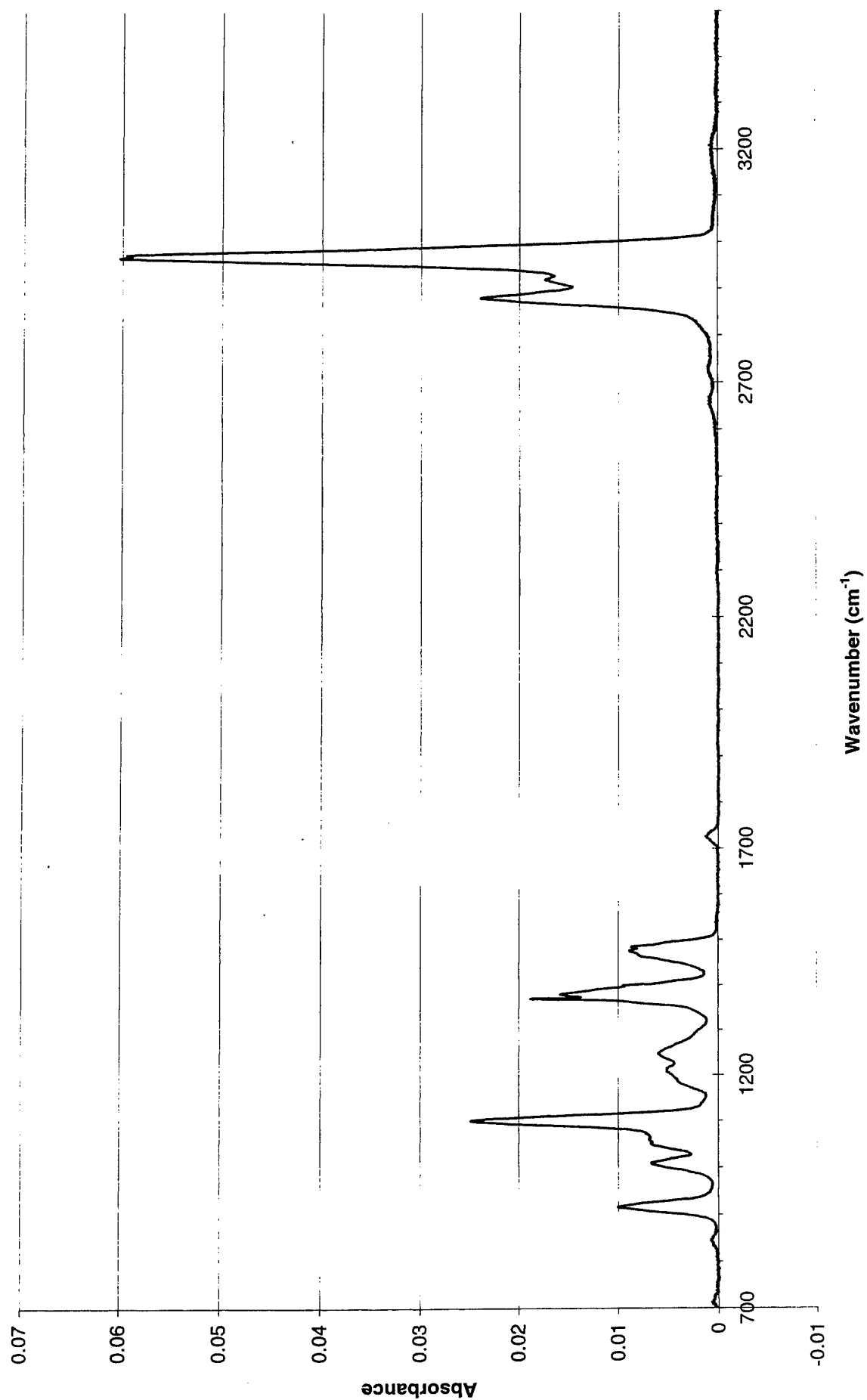


QIRL0210 - Pinacolone
488 ppm-m





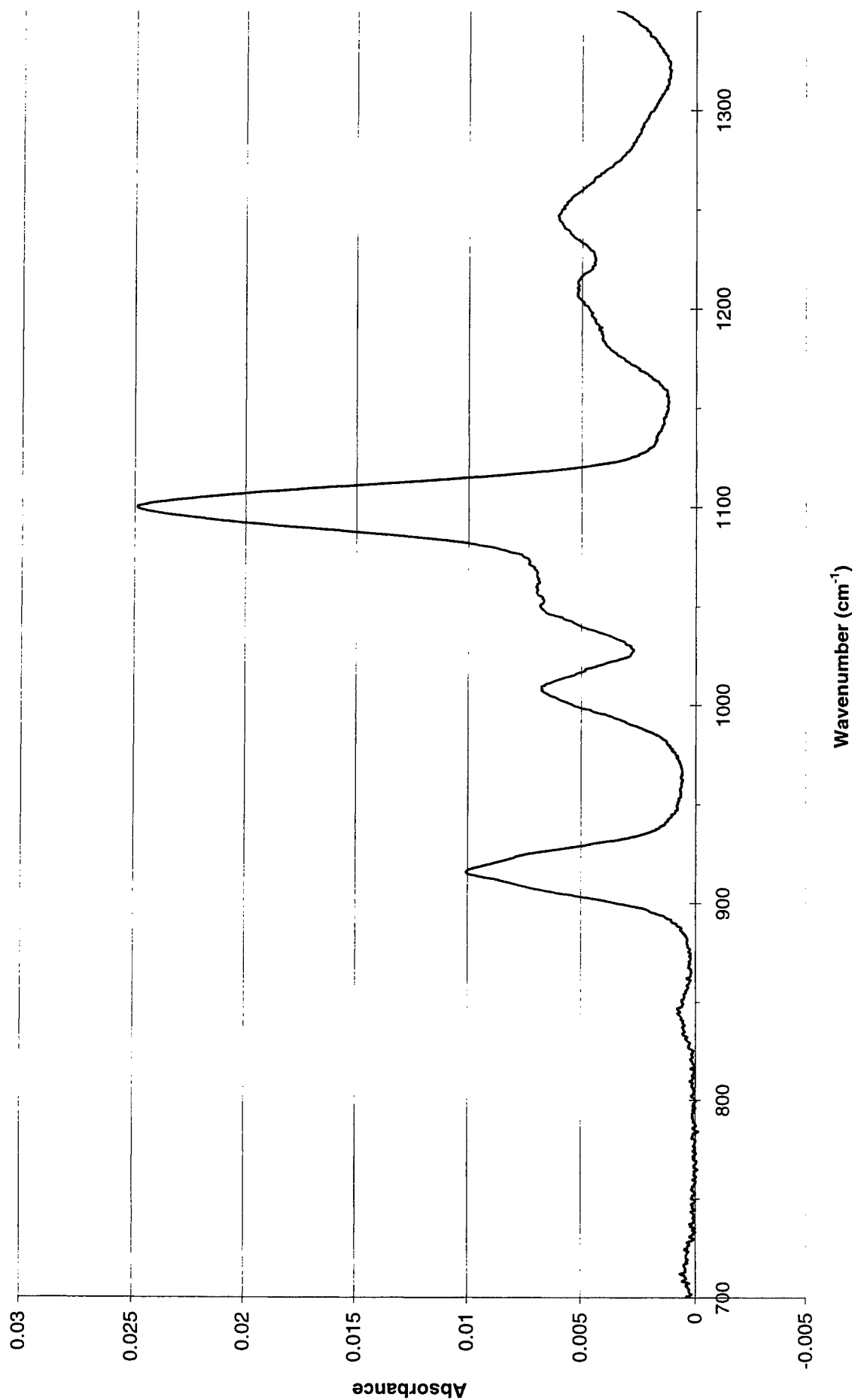
QIRL0220 - Pinacolyl Alcohol 170 ppm-m





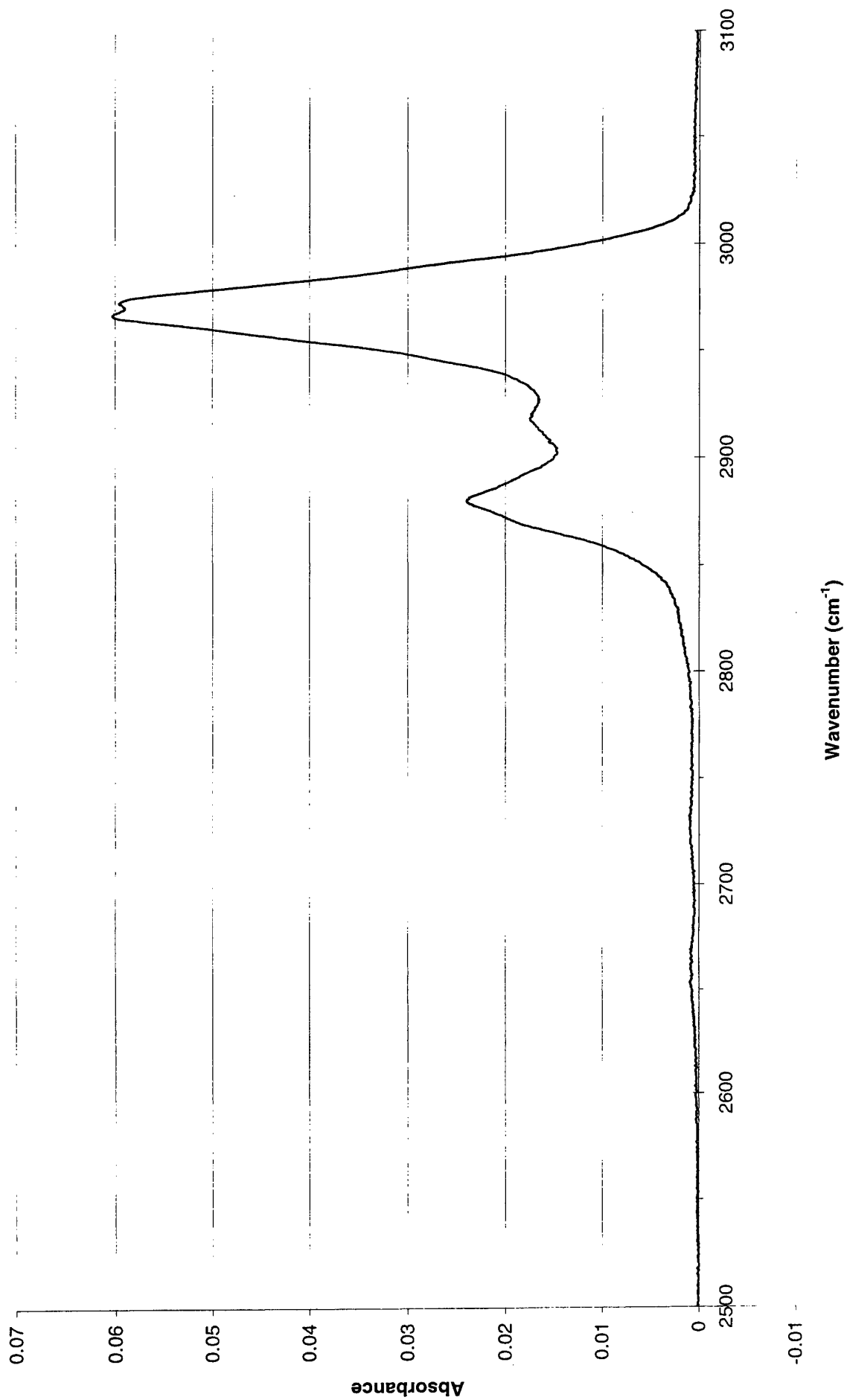
QIRL0220 - Pinacolyl Alcohol

170 ppm-m





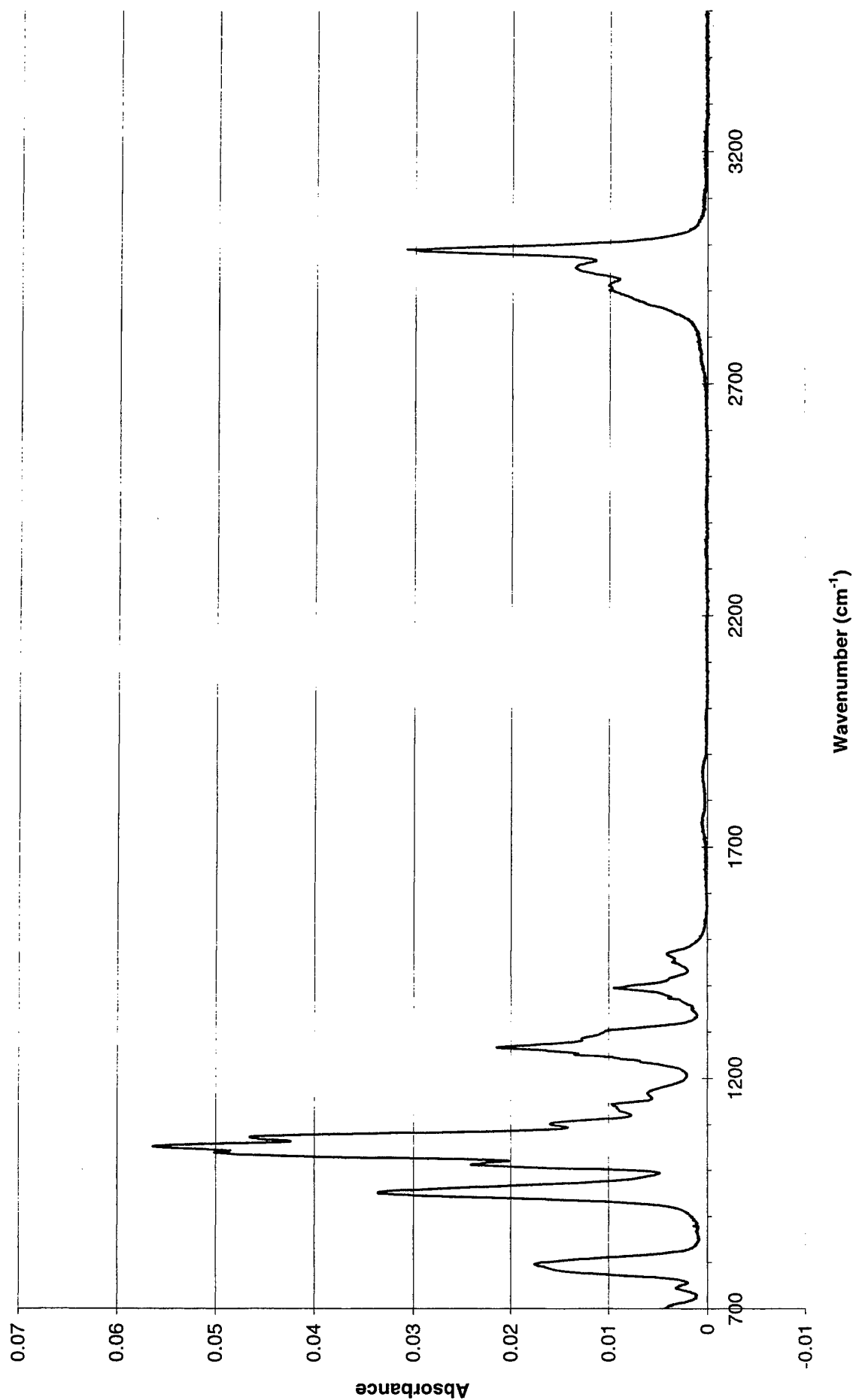
QIRL0220 - Pinacolyl Alcohol 170 ppm-m





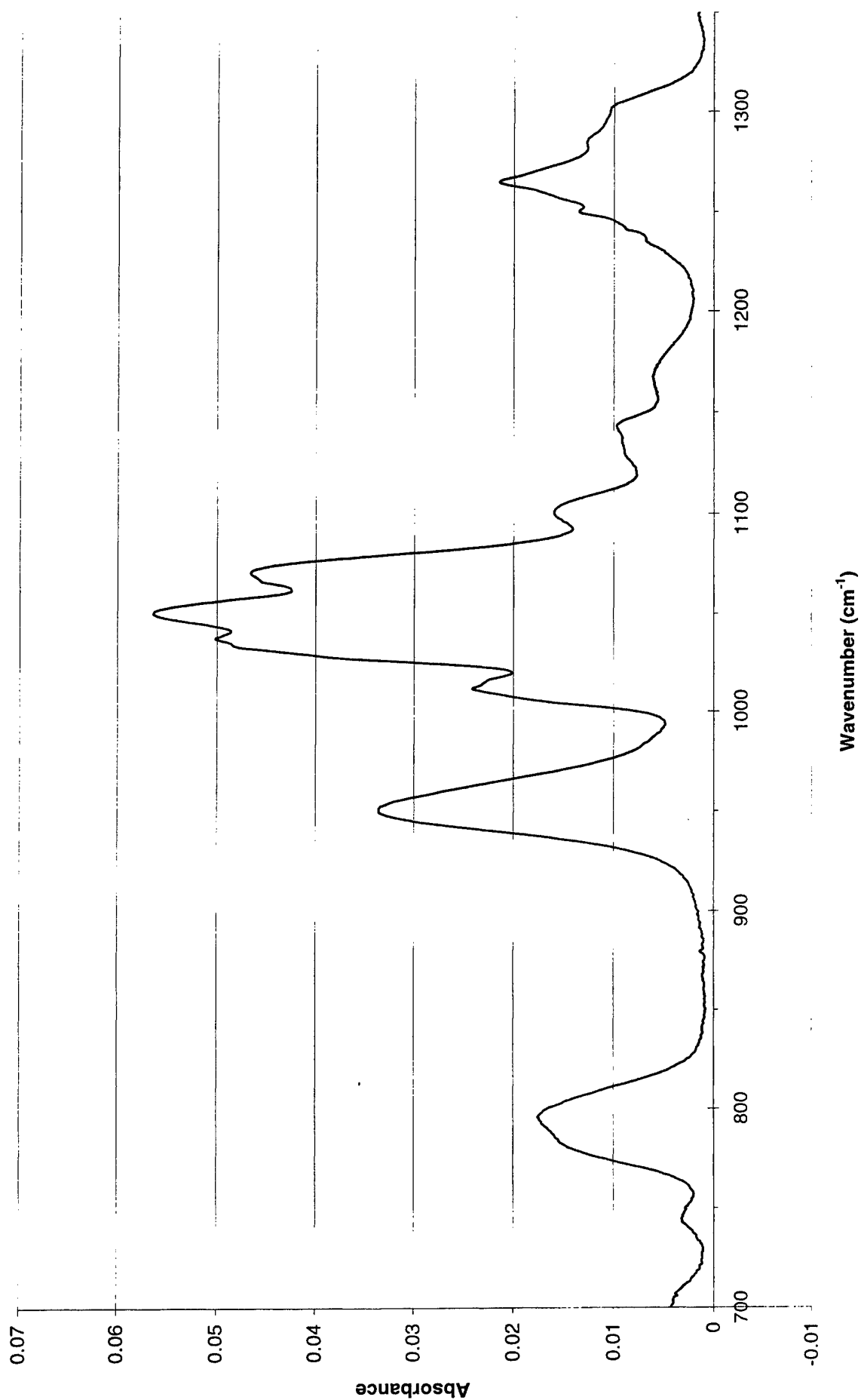
QIRL0230 - Diethyl Ethylphosphonate

37 ppm-m



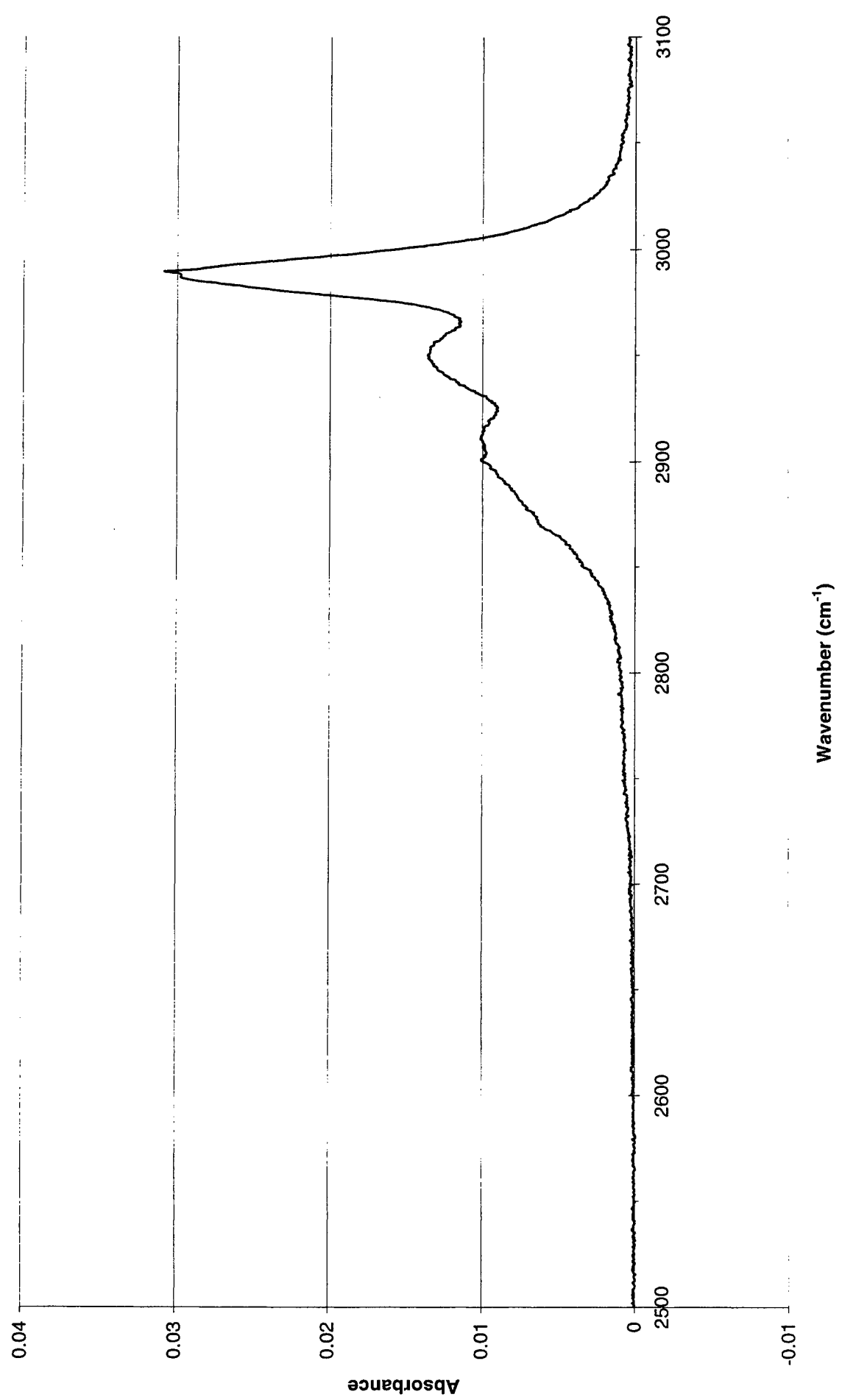


QIRL0230 - Diethyl Ethylphosphonate
37 ppm-m



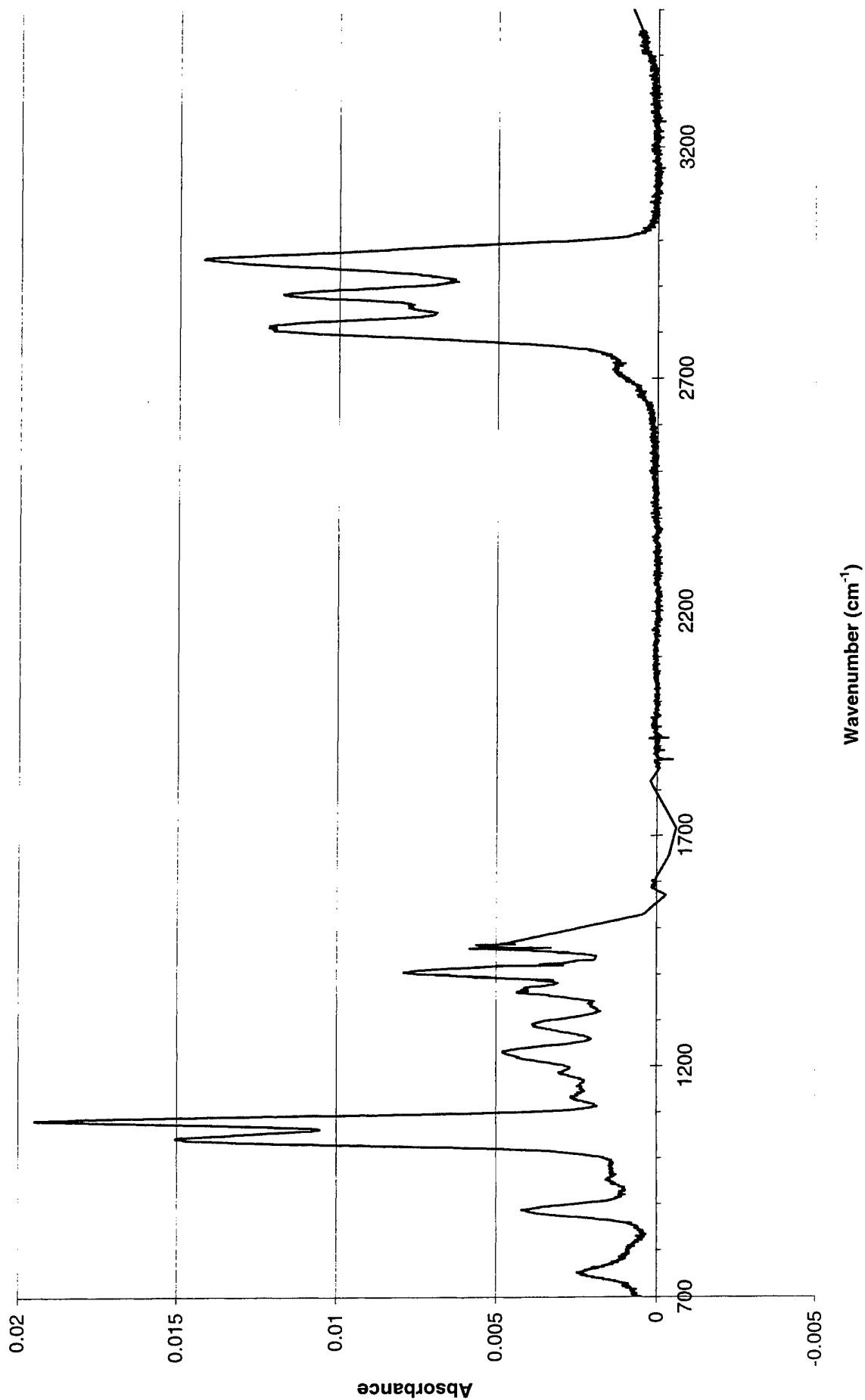


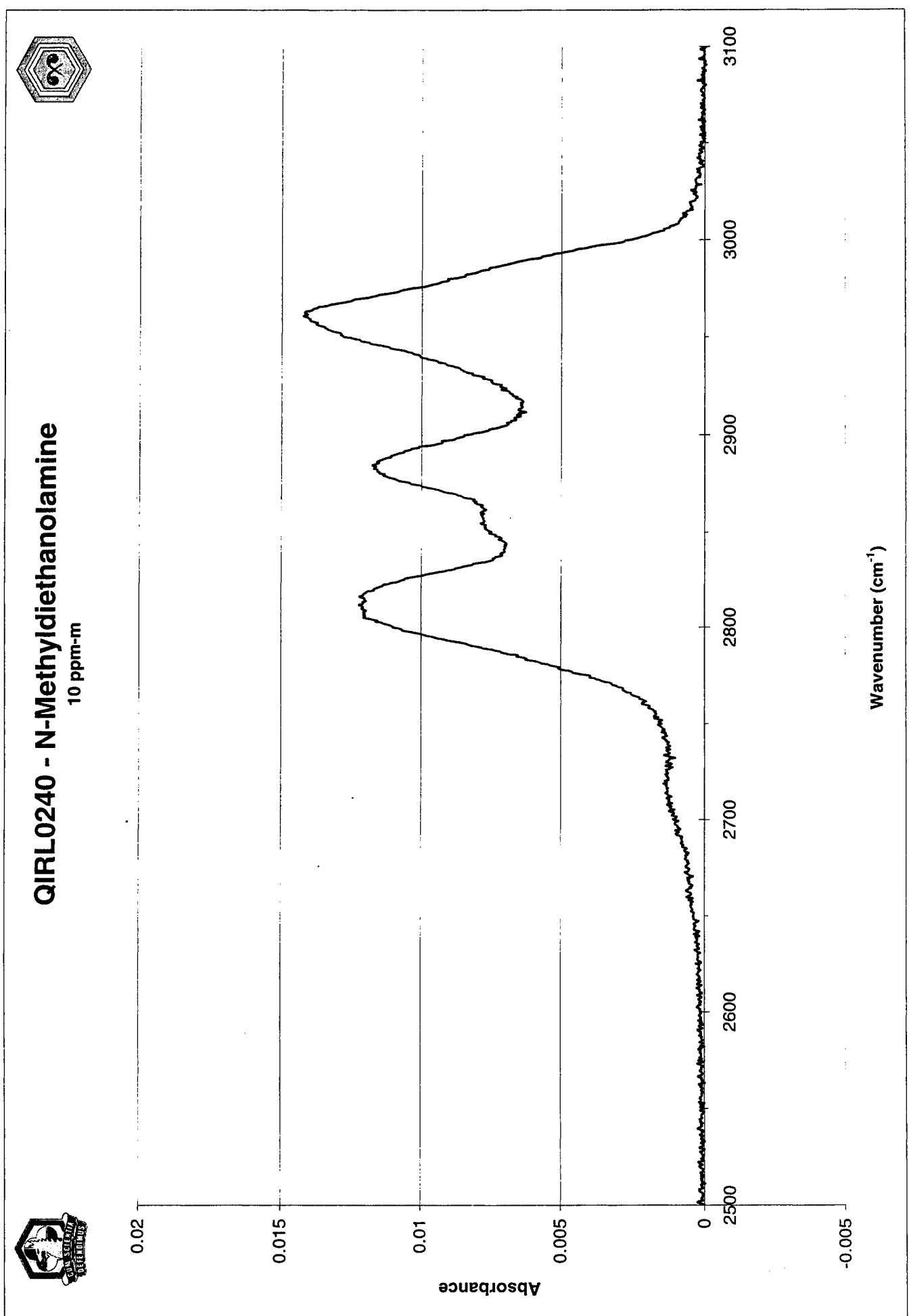
QIRL0230 - Diethyl Ethylphosphonate
37 ppm-m





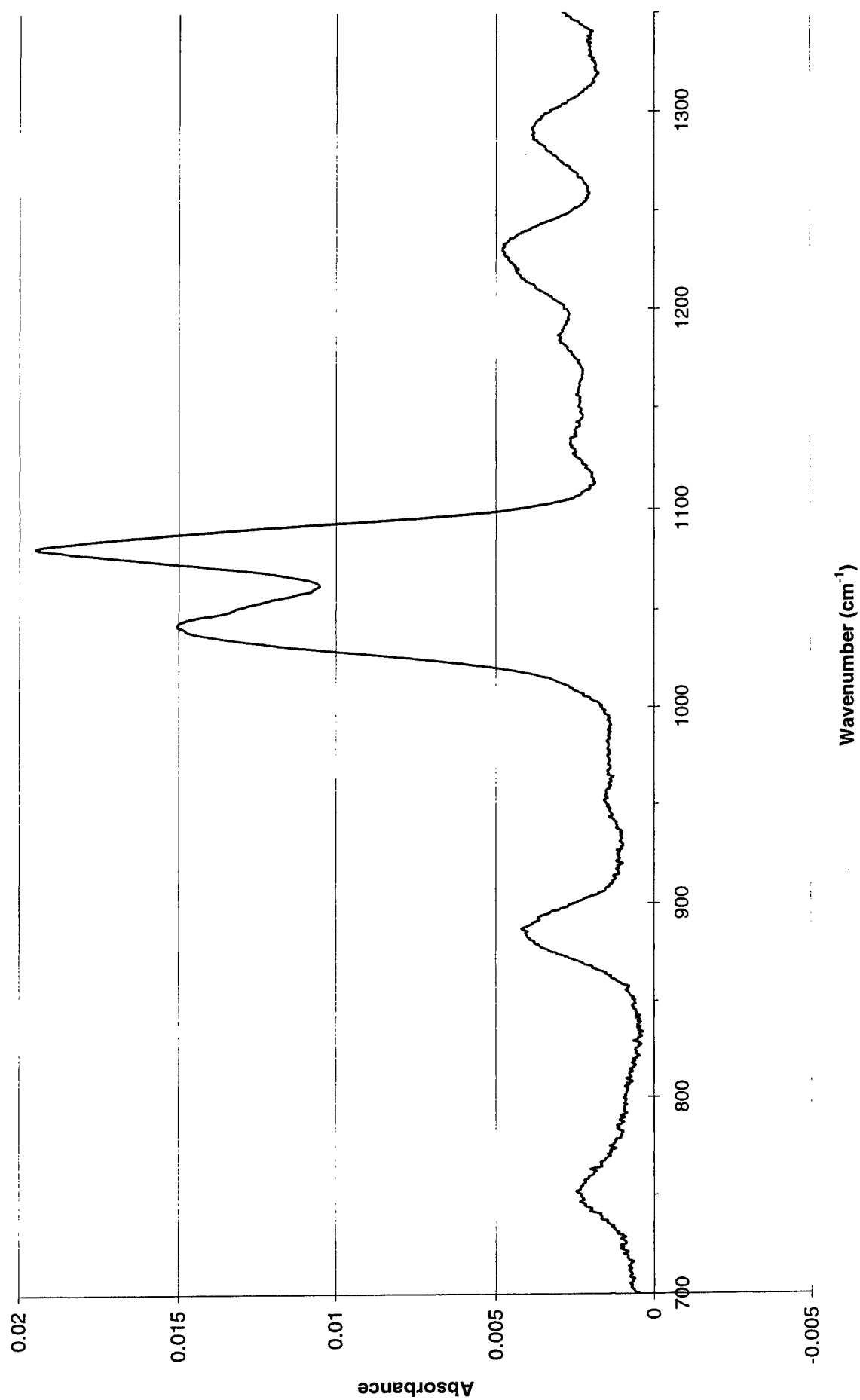
QIRL0240 - N-Methyldiethanolamine
10 ppm-m







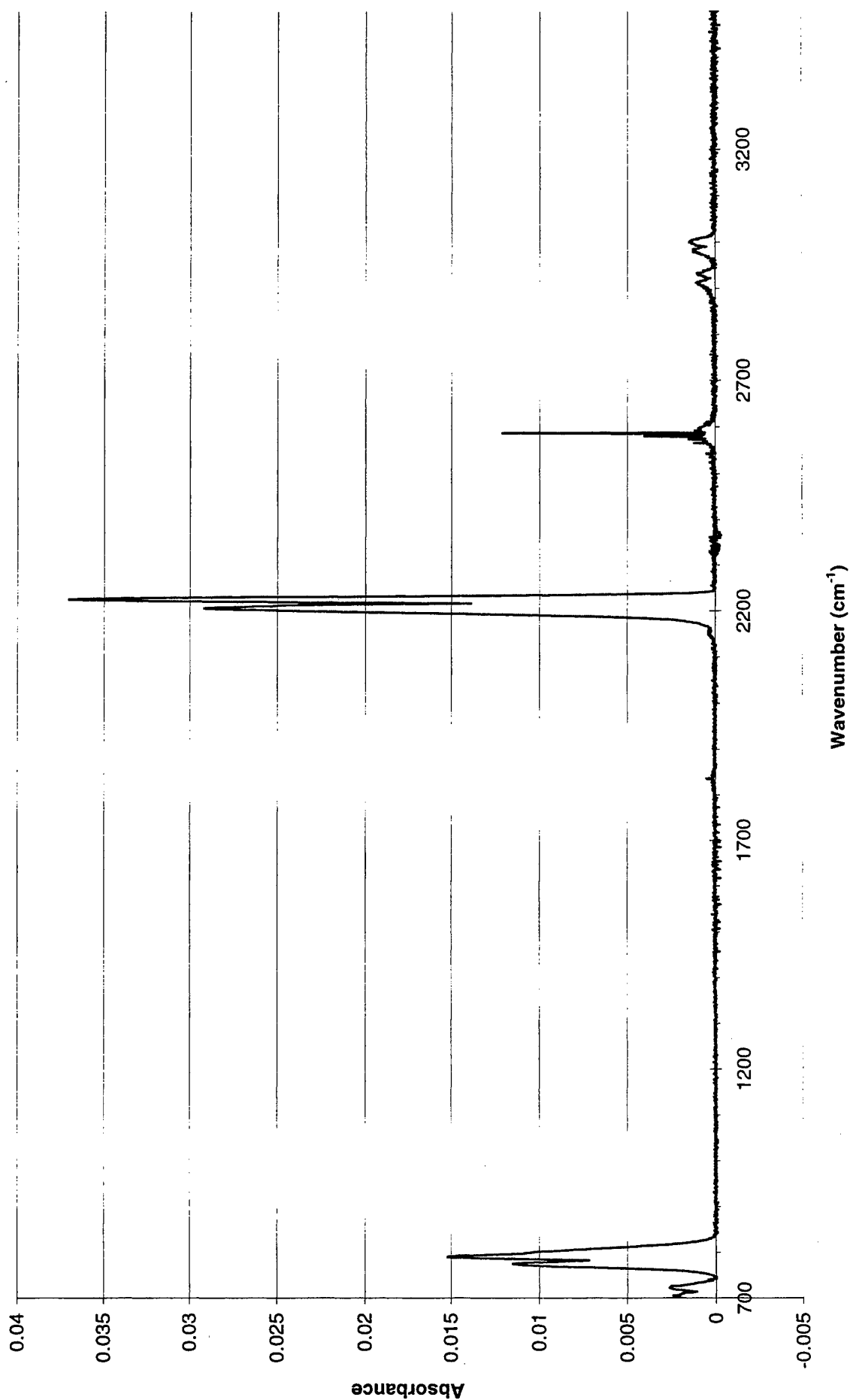
QIRL0240 - N-Methyldiethanolamine
10 ppm-m





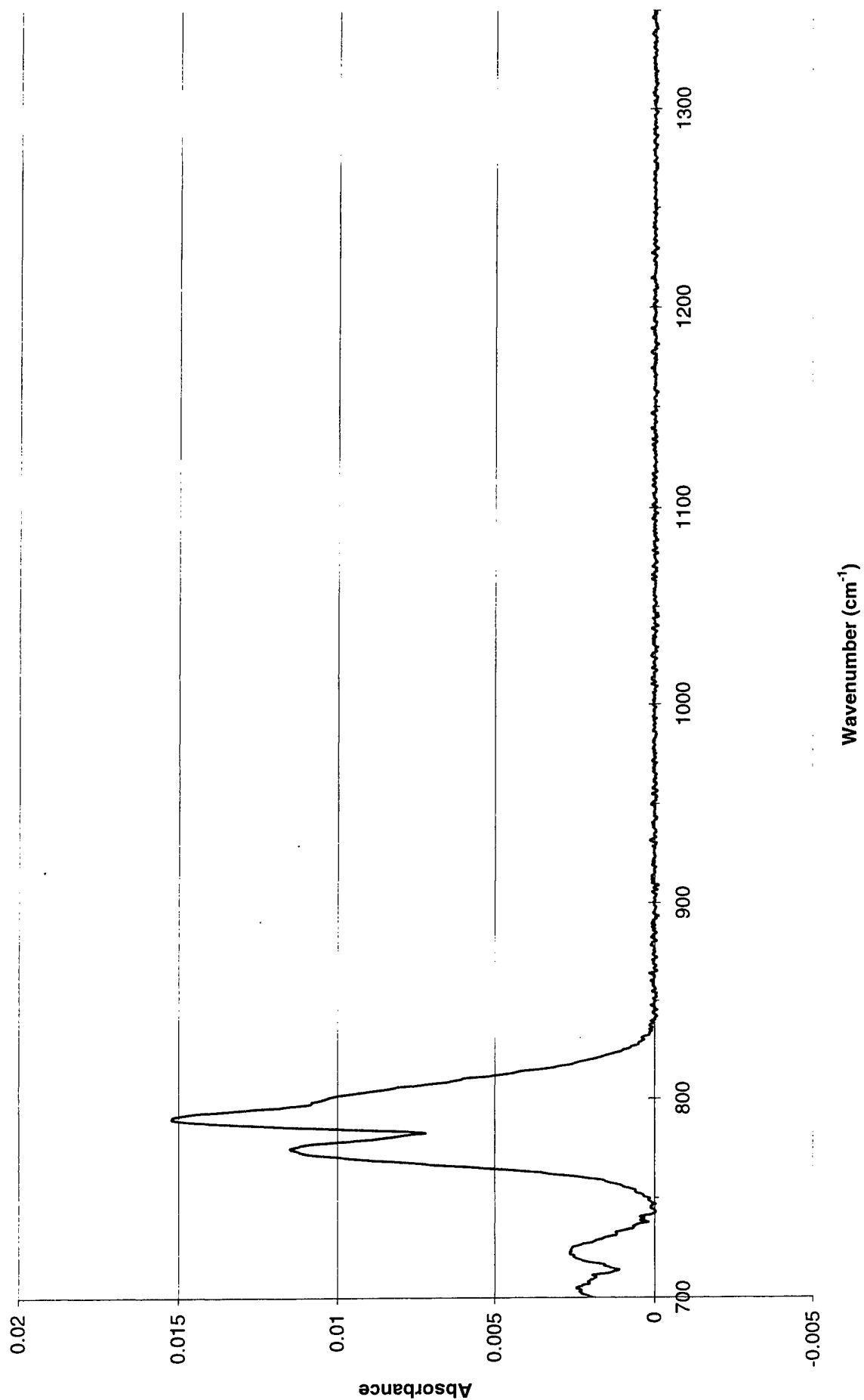
QIRL0250 - Cyanogen Chloride (CK)

343 ppm-m



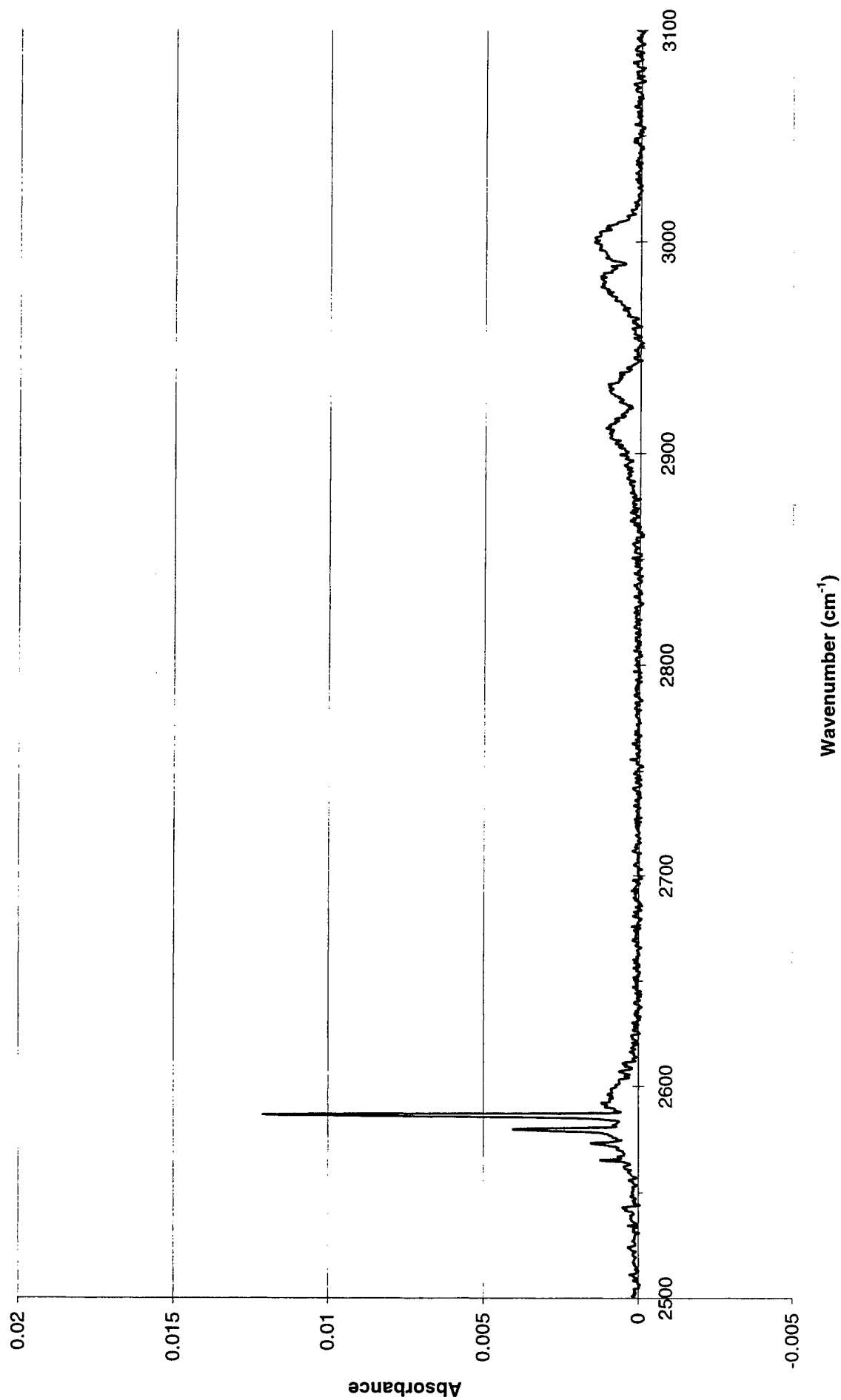


QIRL0250 - Cyanogen Chloride (CK)
343 ppm-m



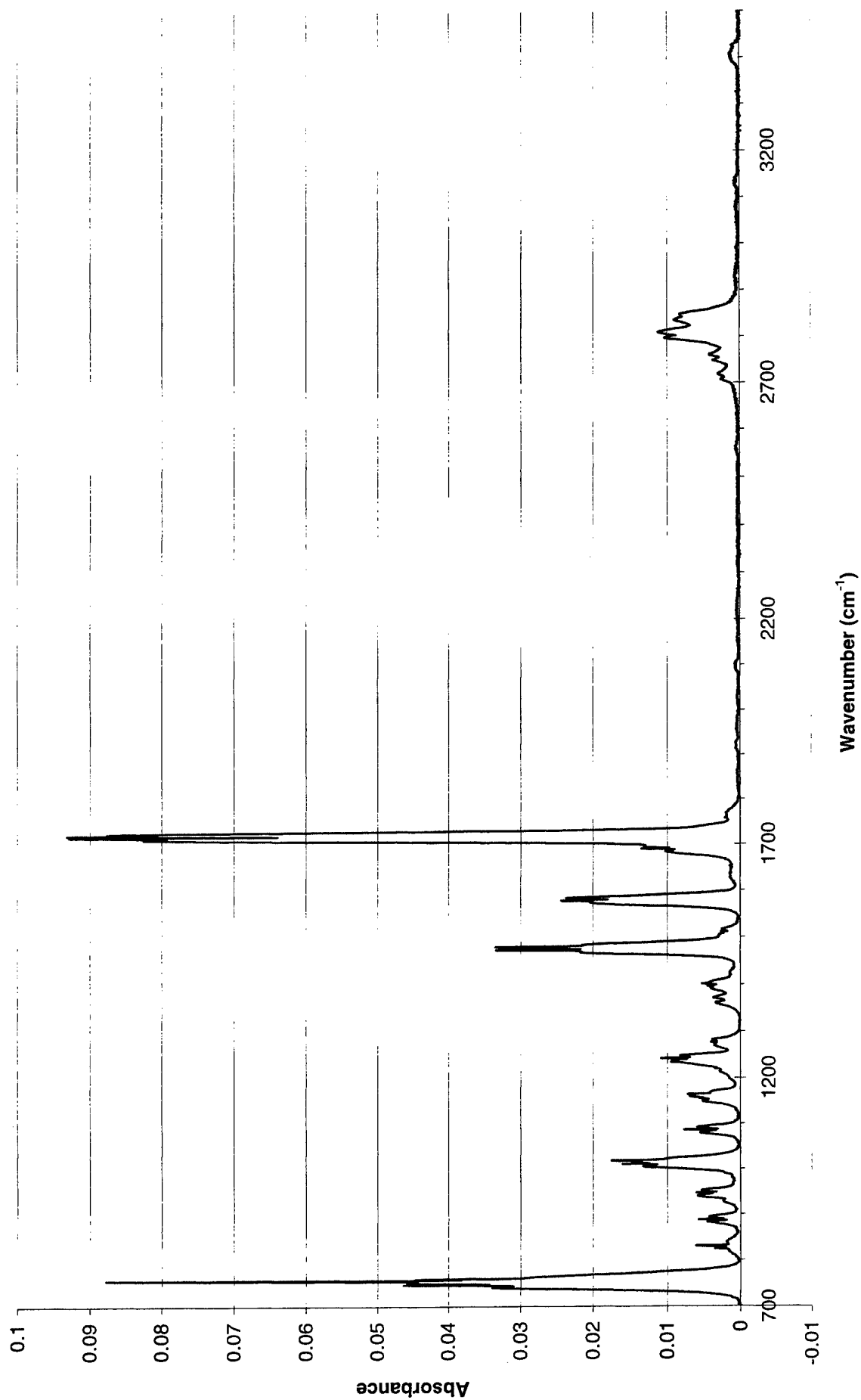


QIRL0250 - Cyanogen Chloride (CK)
343 ppm-m



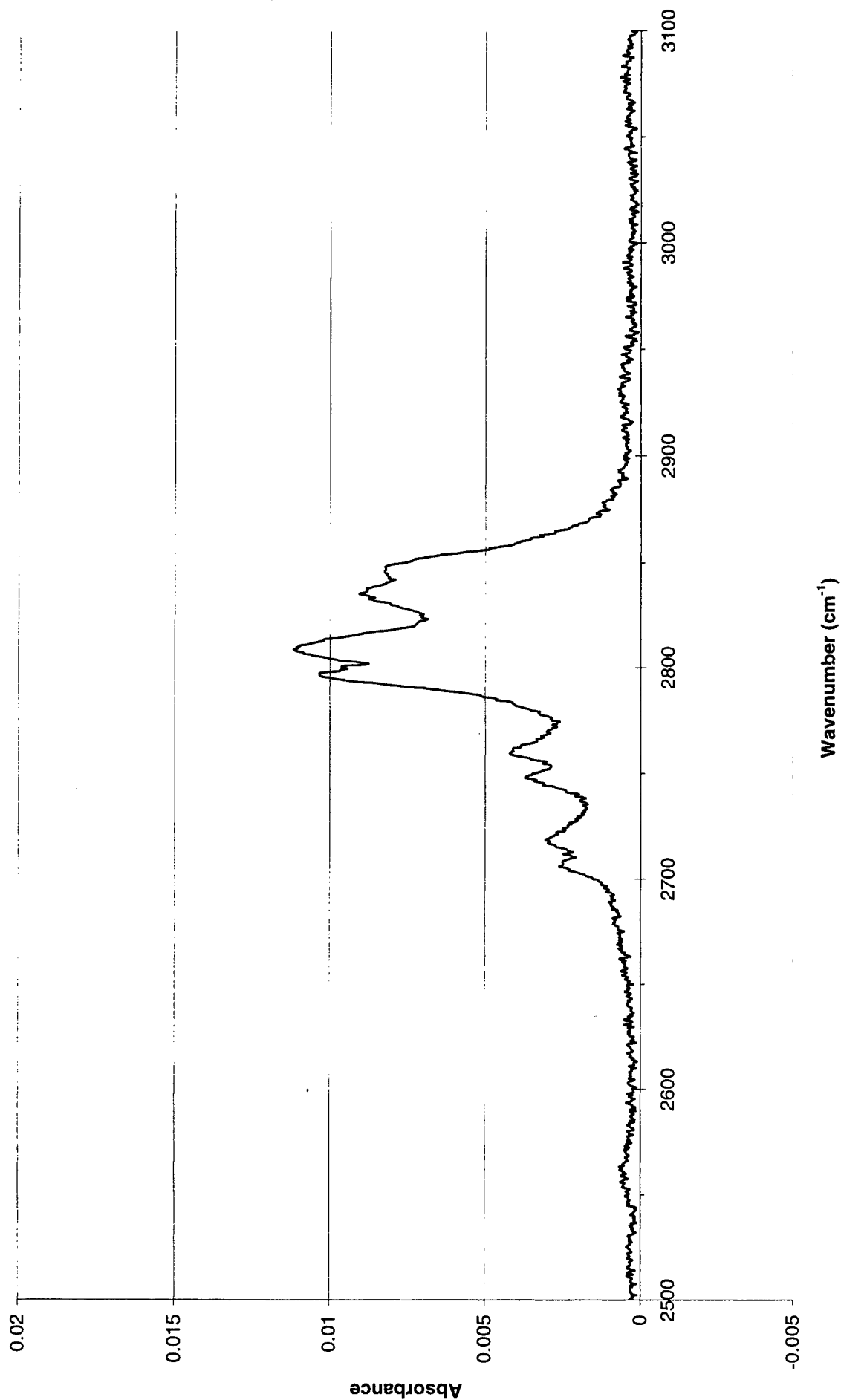


QIRL0260 - 2-Furaldehyde
74 ppm-m





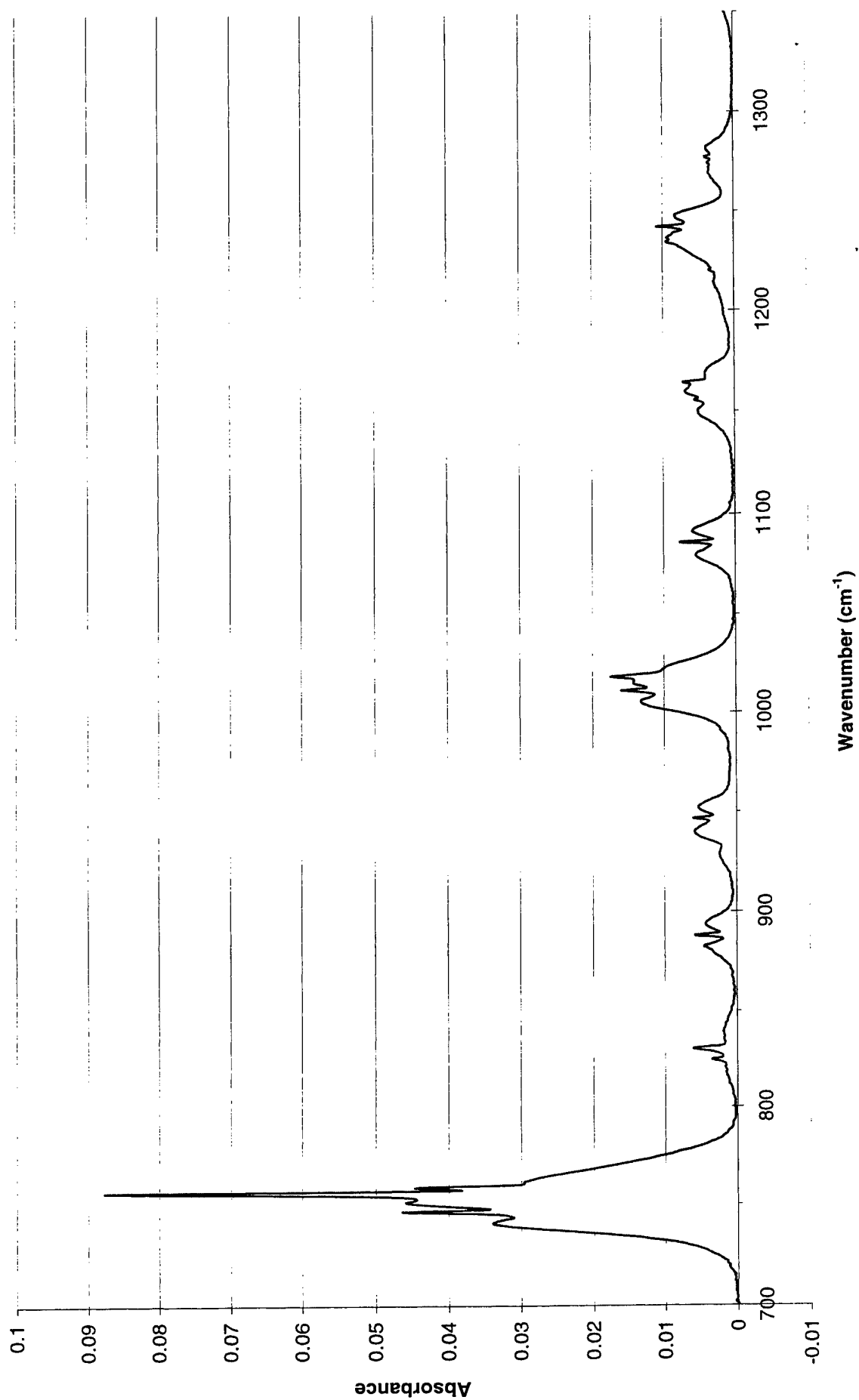
QIRL0260 - 2-Furaldehyde
74 ppm-m





QIRL0260 - 2-Furaldehyde

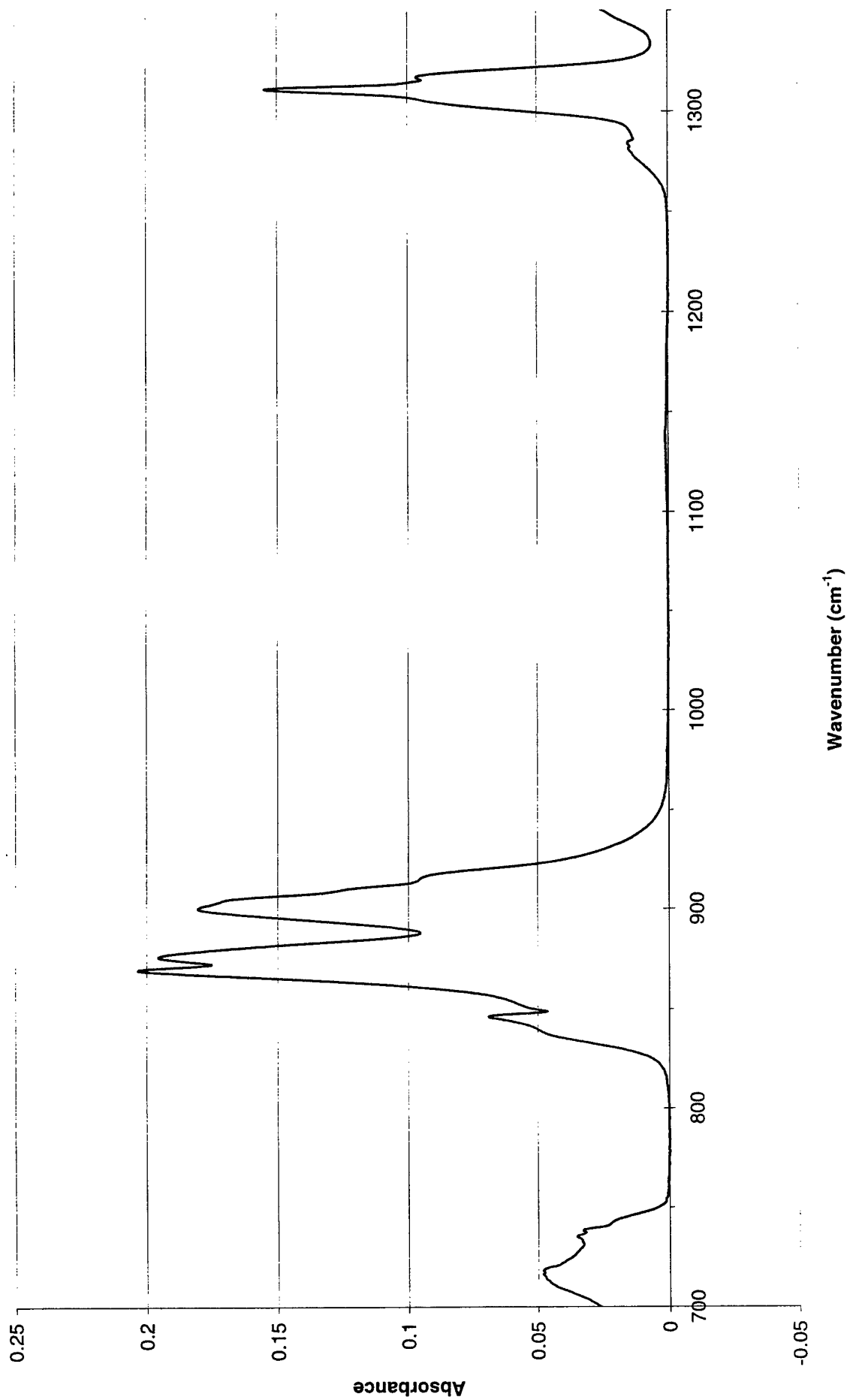
74 ppm-m





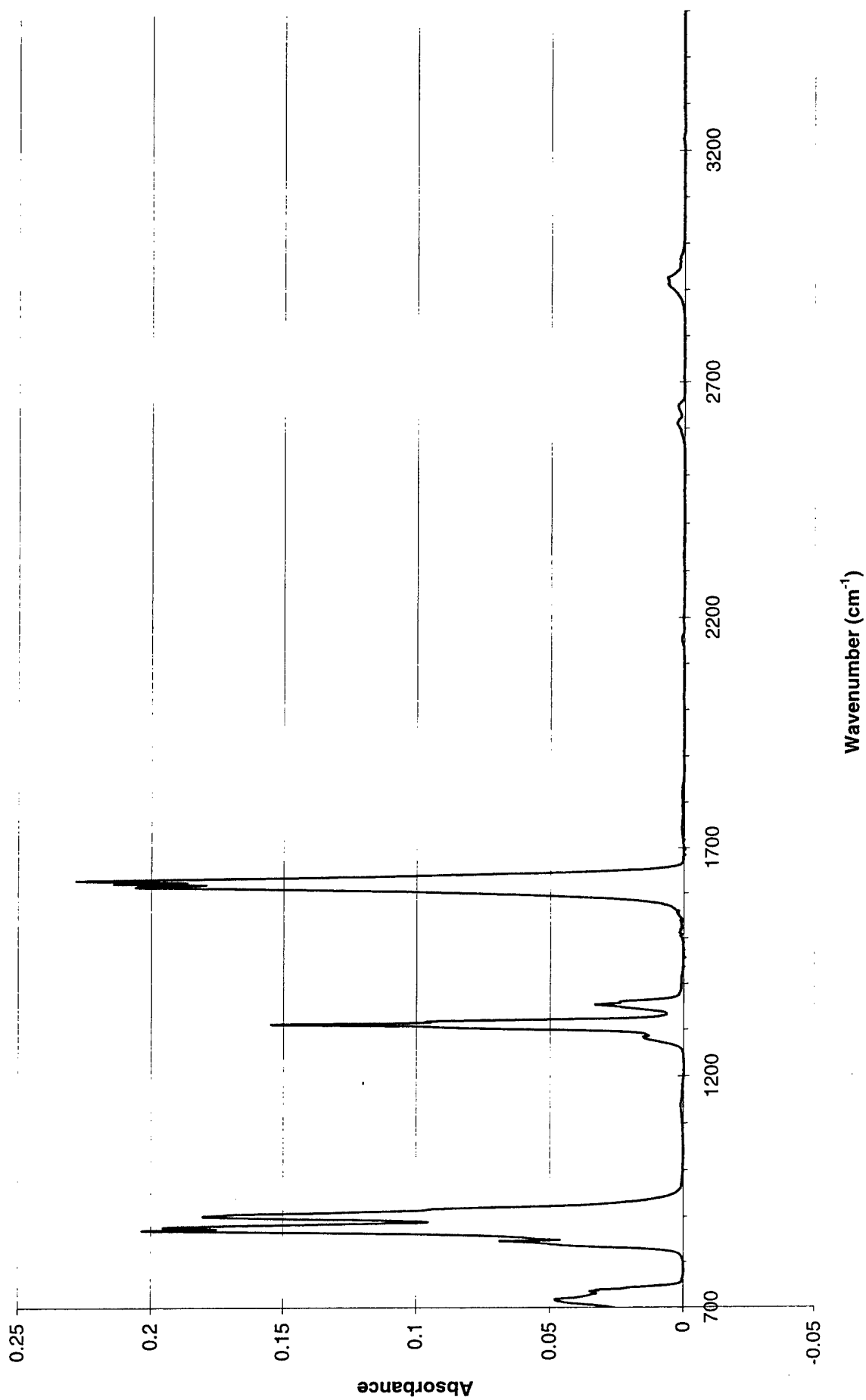
QIRL0270 - Trichloronitromethane (PS)

177 ppm-m



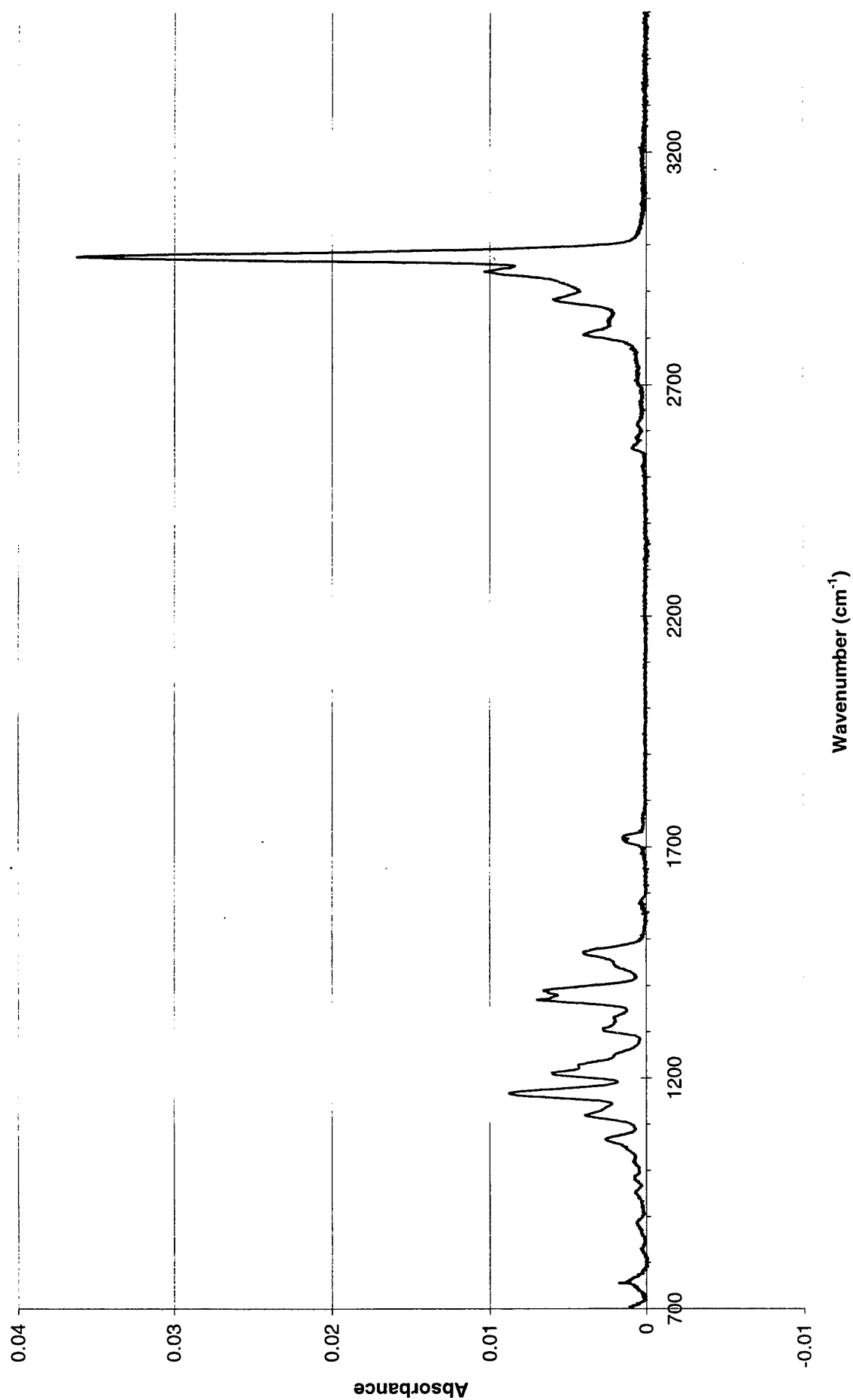


QIRL0270 - Trichloronitromethane (PS)
177 ppm-m



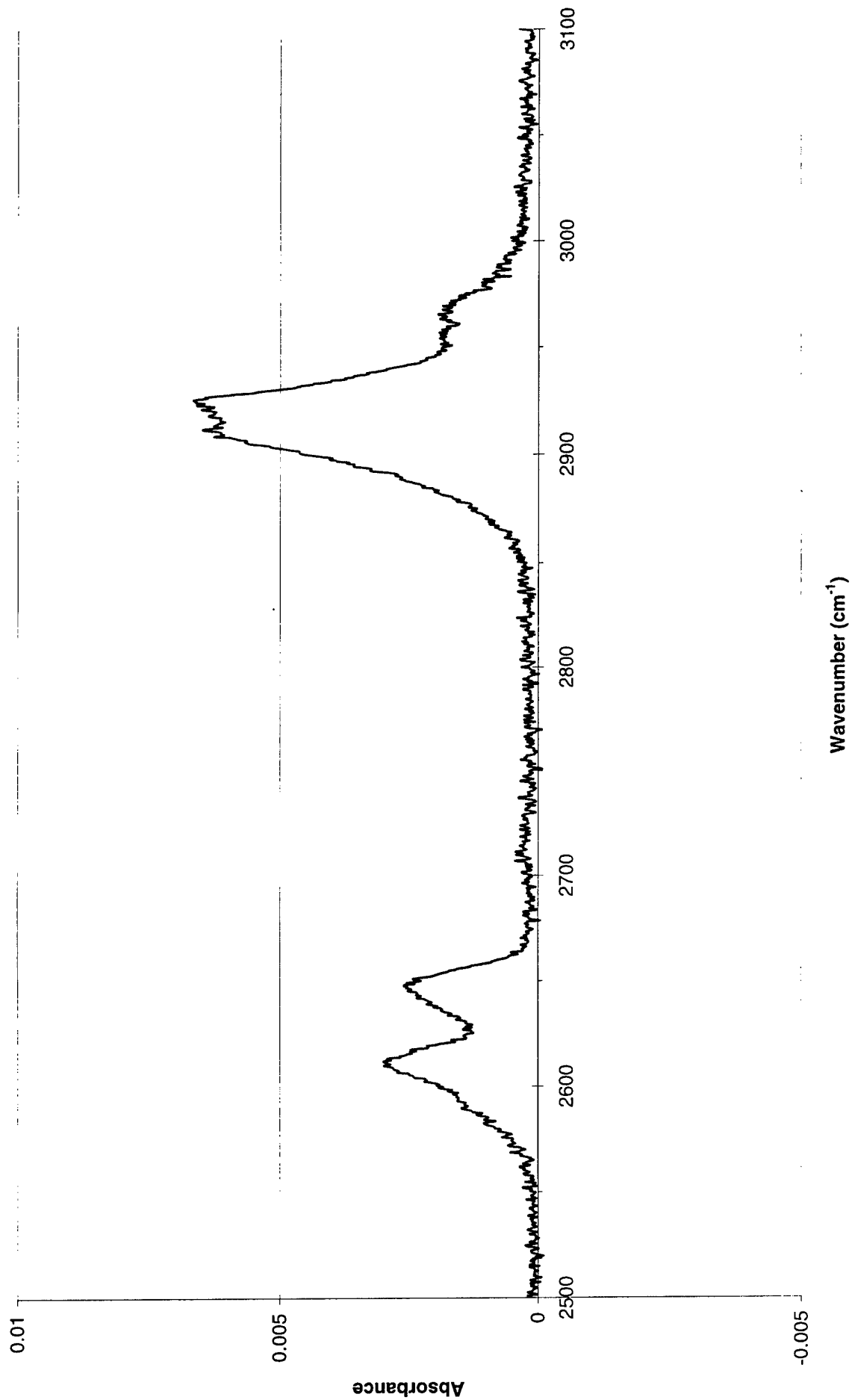


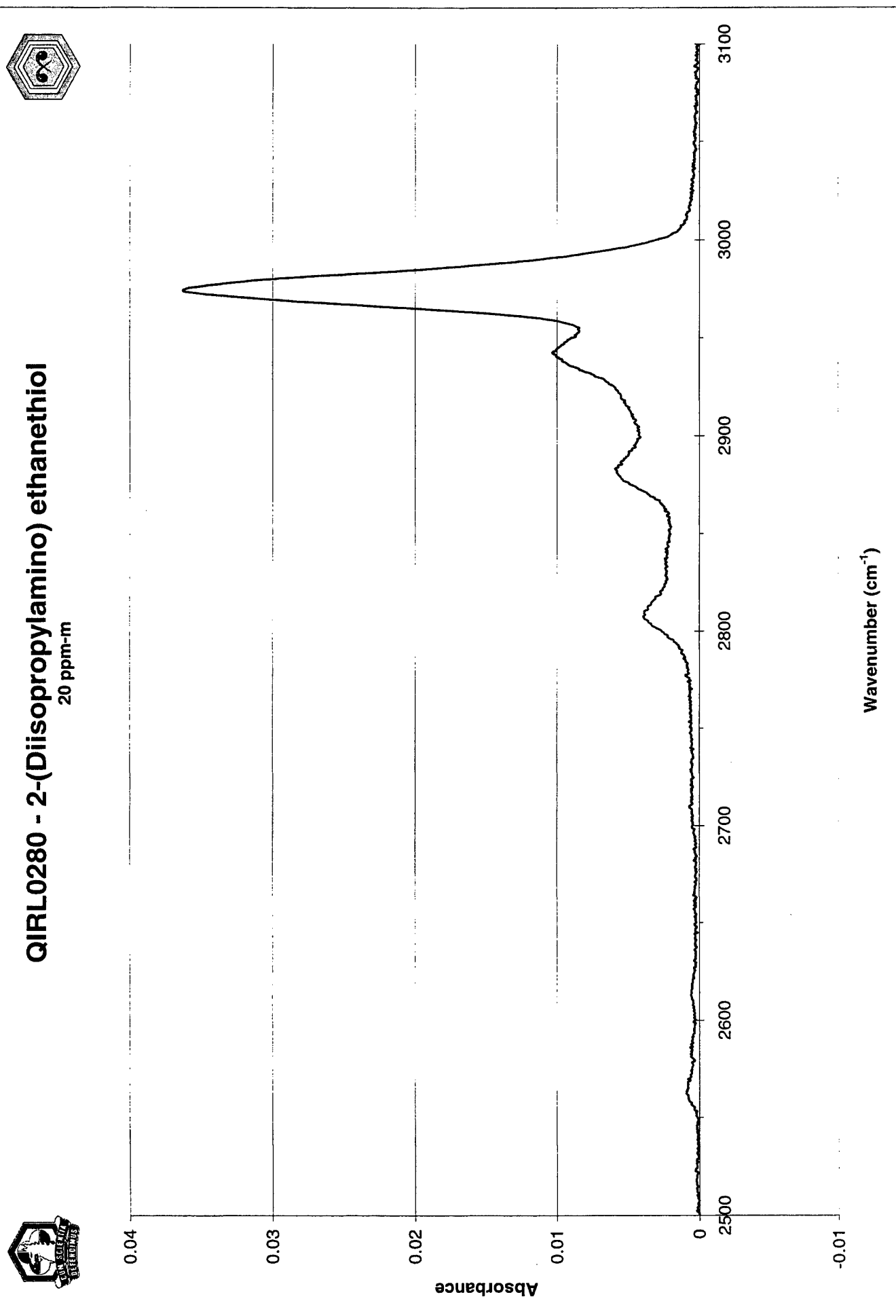
QIRL0280 - 2-(Diisopropylamino) ethanethiol
20 ppm-m





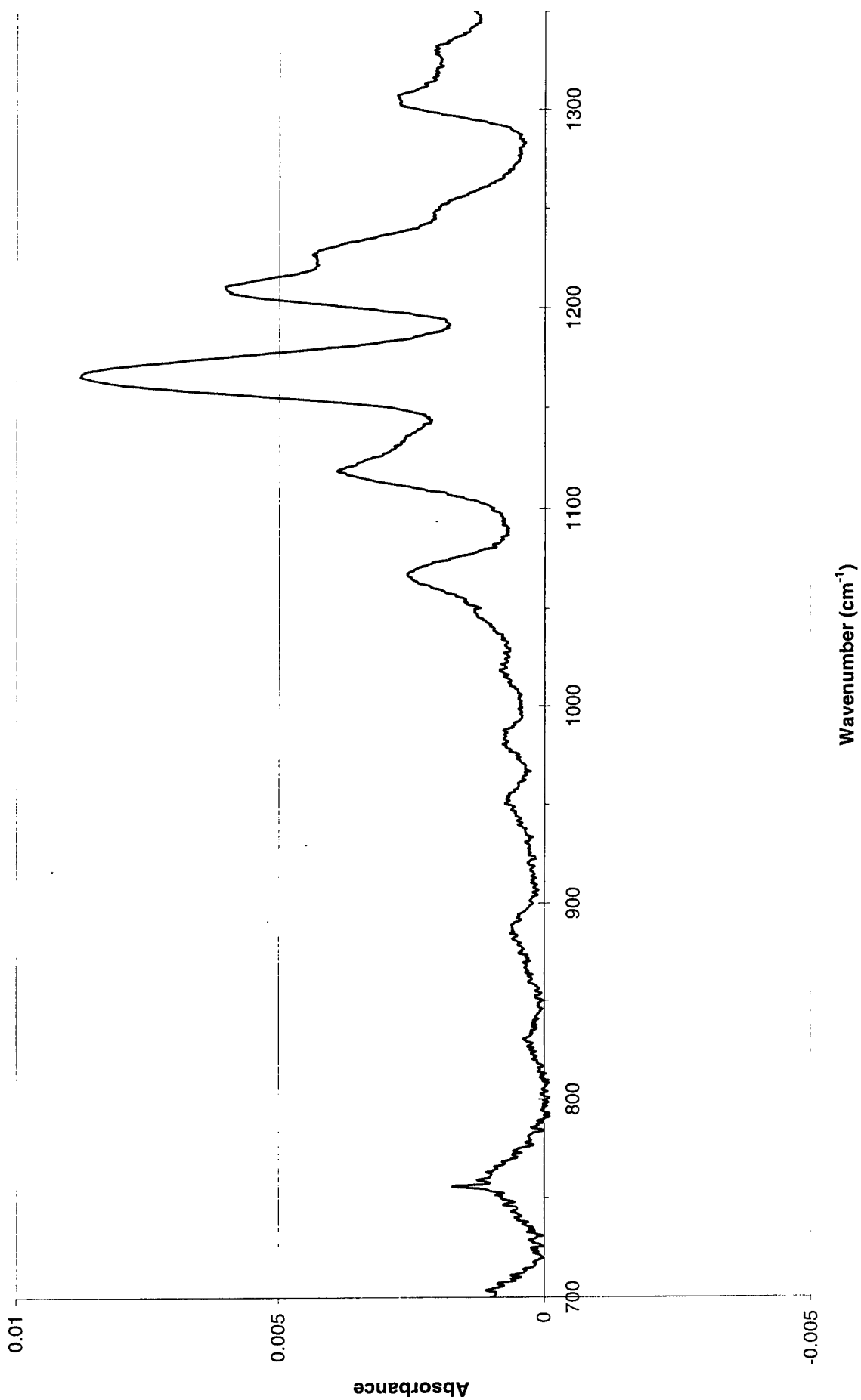
QIRL0270 - Trichloronitromethane (PS)
177 ppm-m





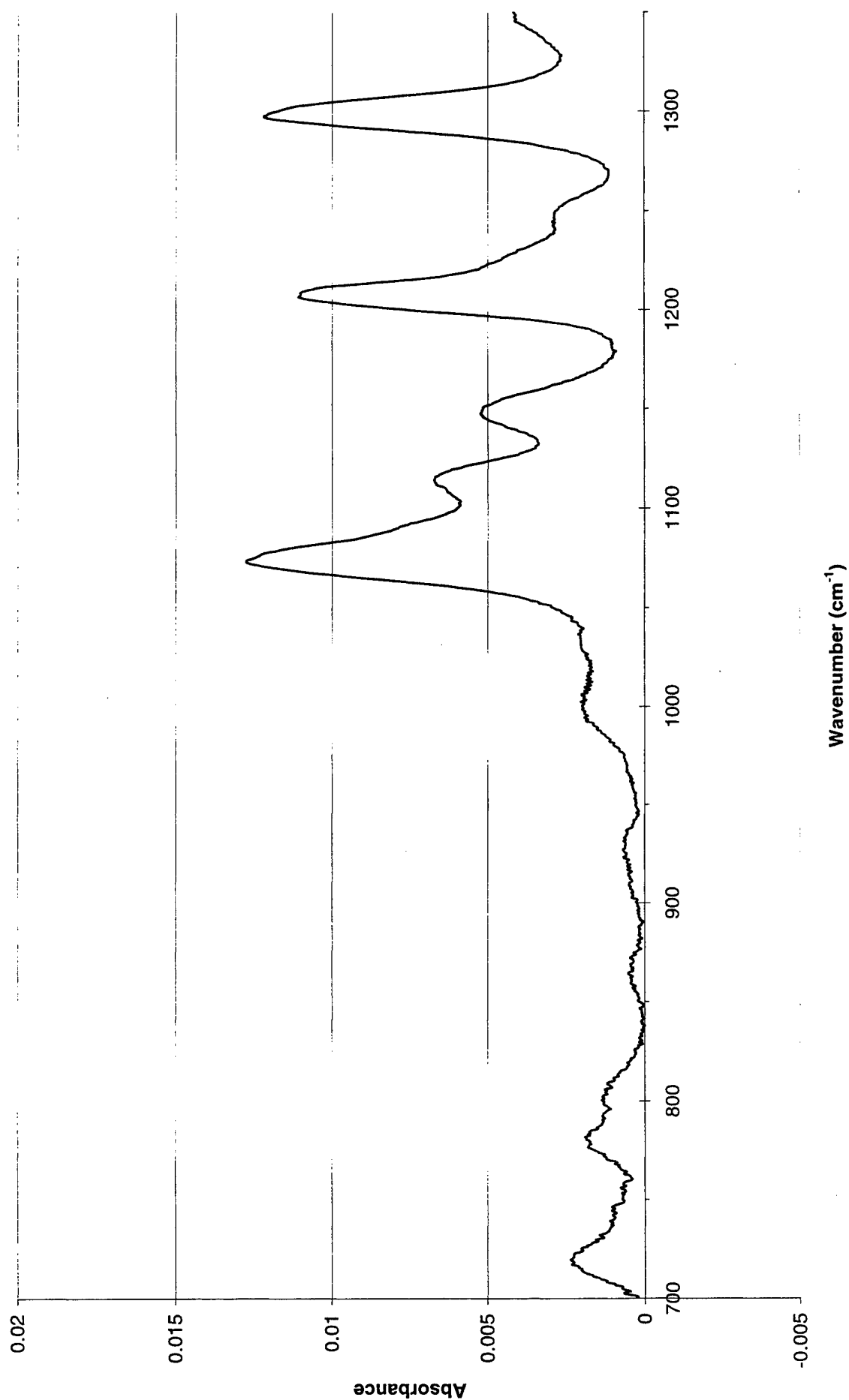


QIRL0280 - 2-(Diisopropylamino) ethanethiol
20 ppm-m



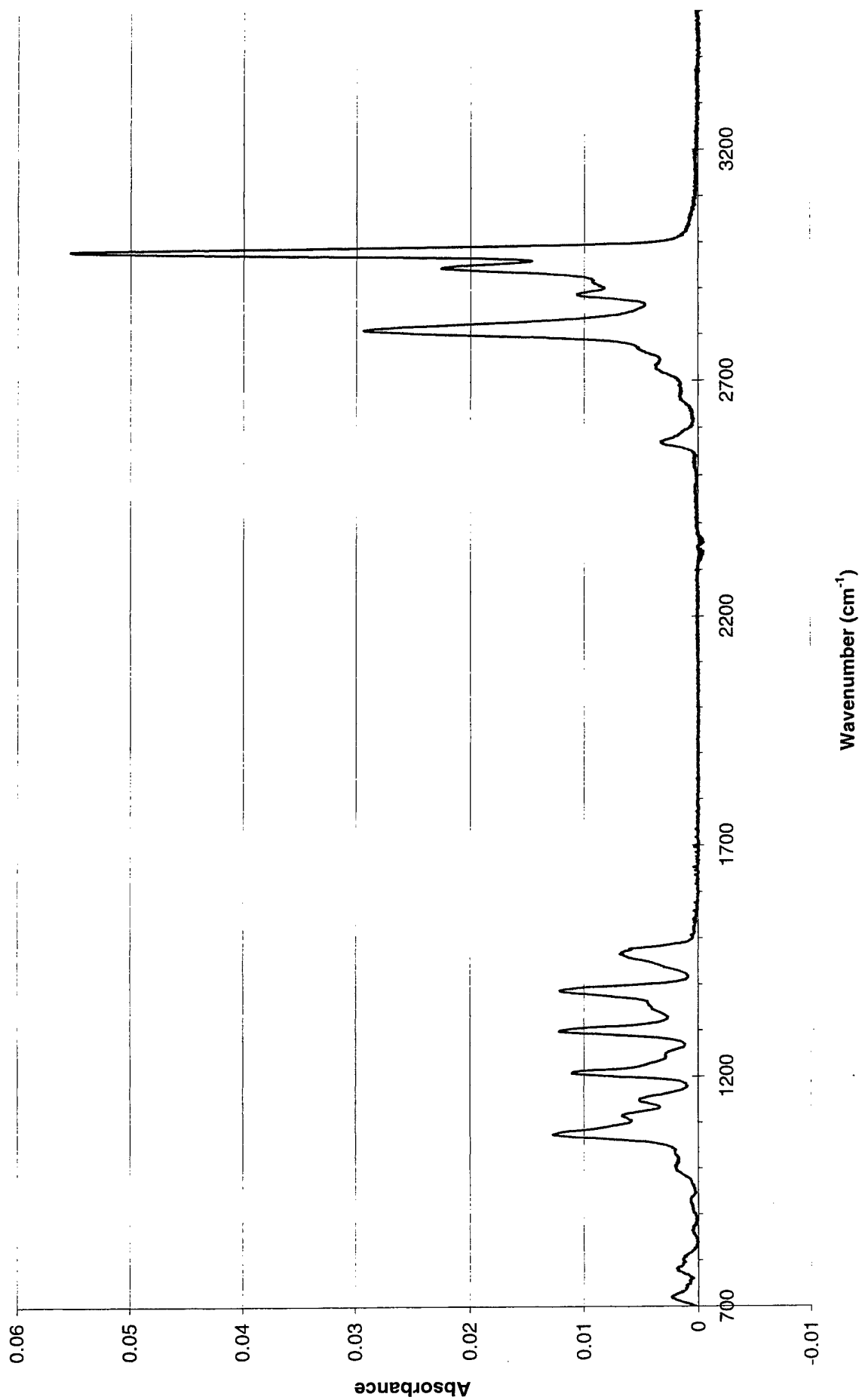


QIRL0290 - 2-(Diethylamino) ethanethiol
72 ppm-m



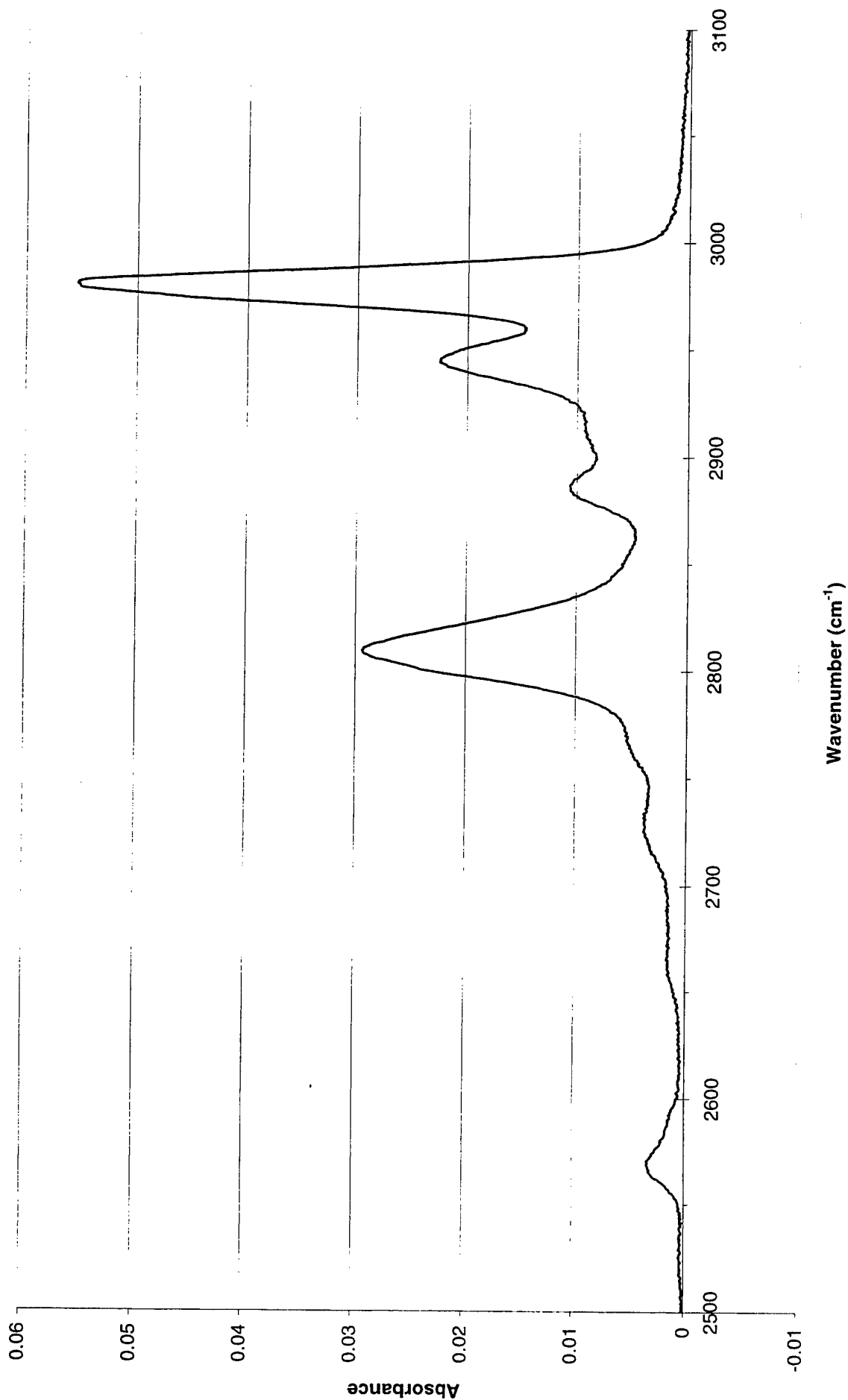


QIRL0290 - 2-(Diethylamino) ethanethiol
72 ppm-m



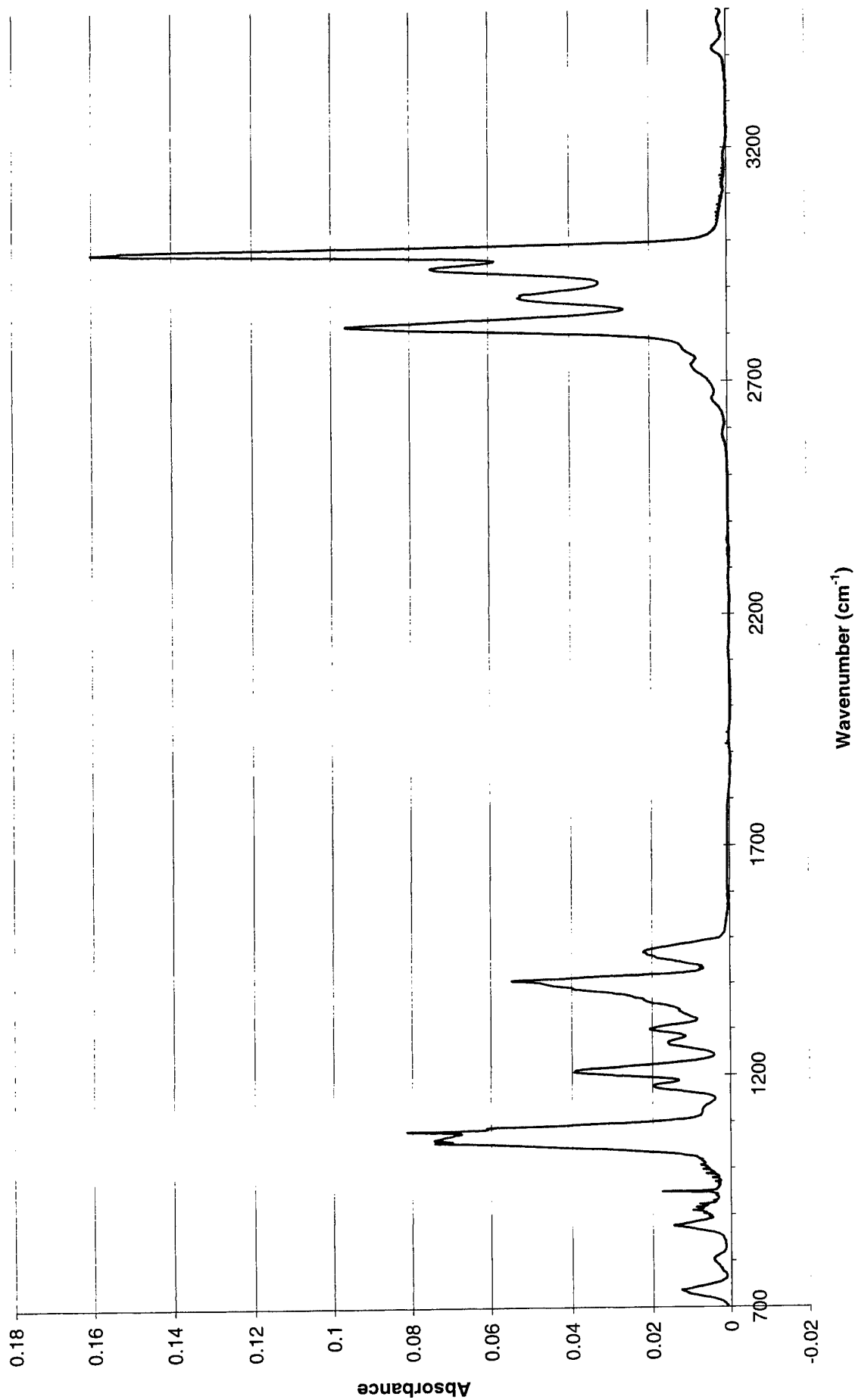


QIRL0290 - 2-(Diethyamino) ethanethiol
72 ppm-m



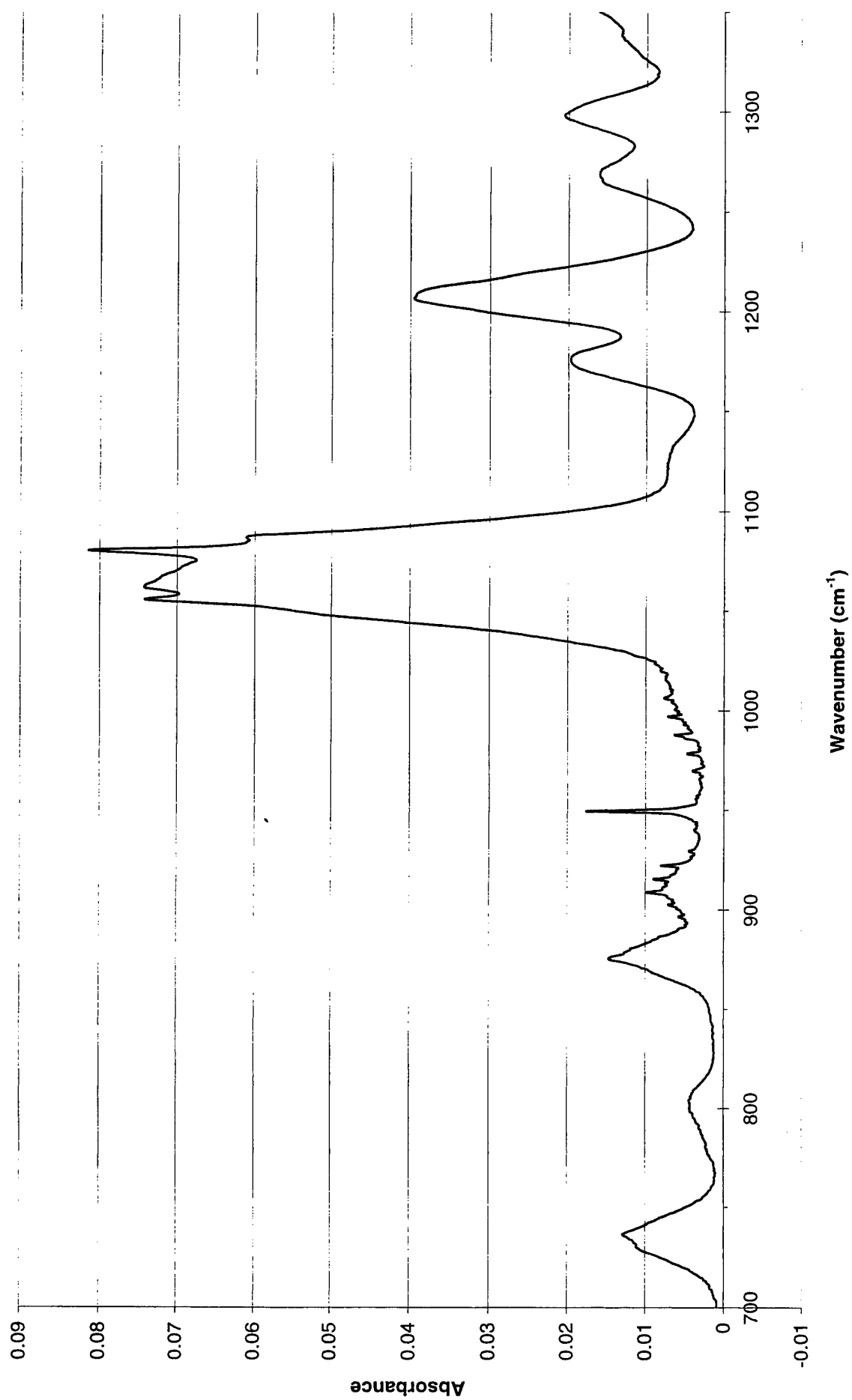


QIRL0300 - 2-(Diethyamino) ethanol
173 ppm-m



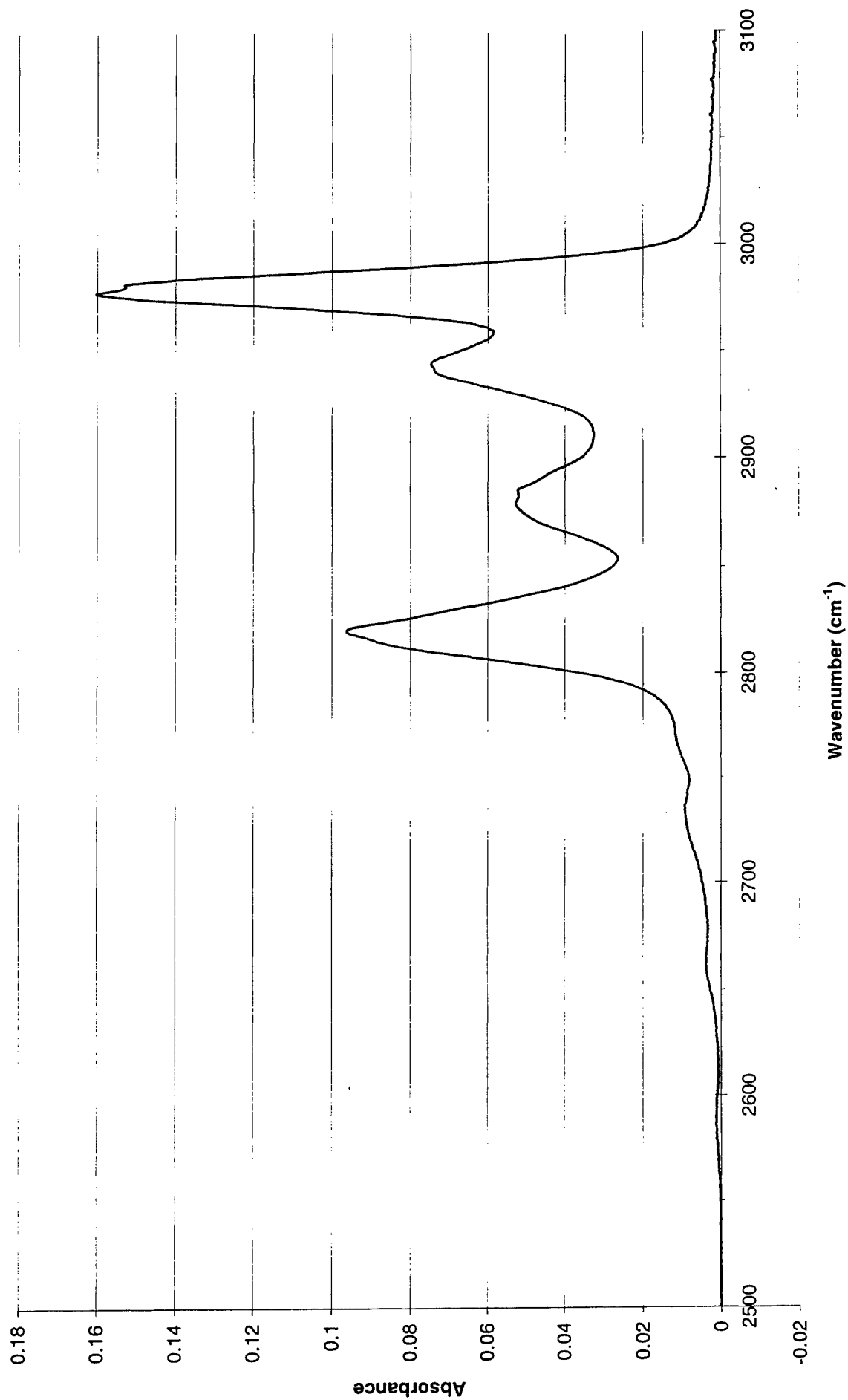


QIRL0300 - 2-(Diethyamino) ethanol
173 ppm-m



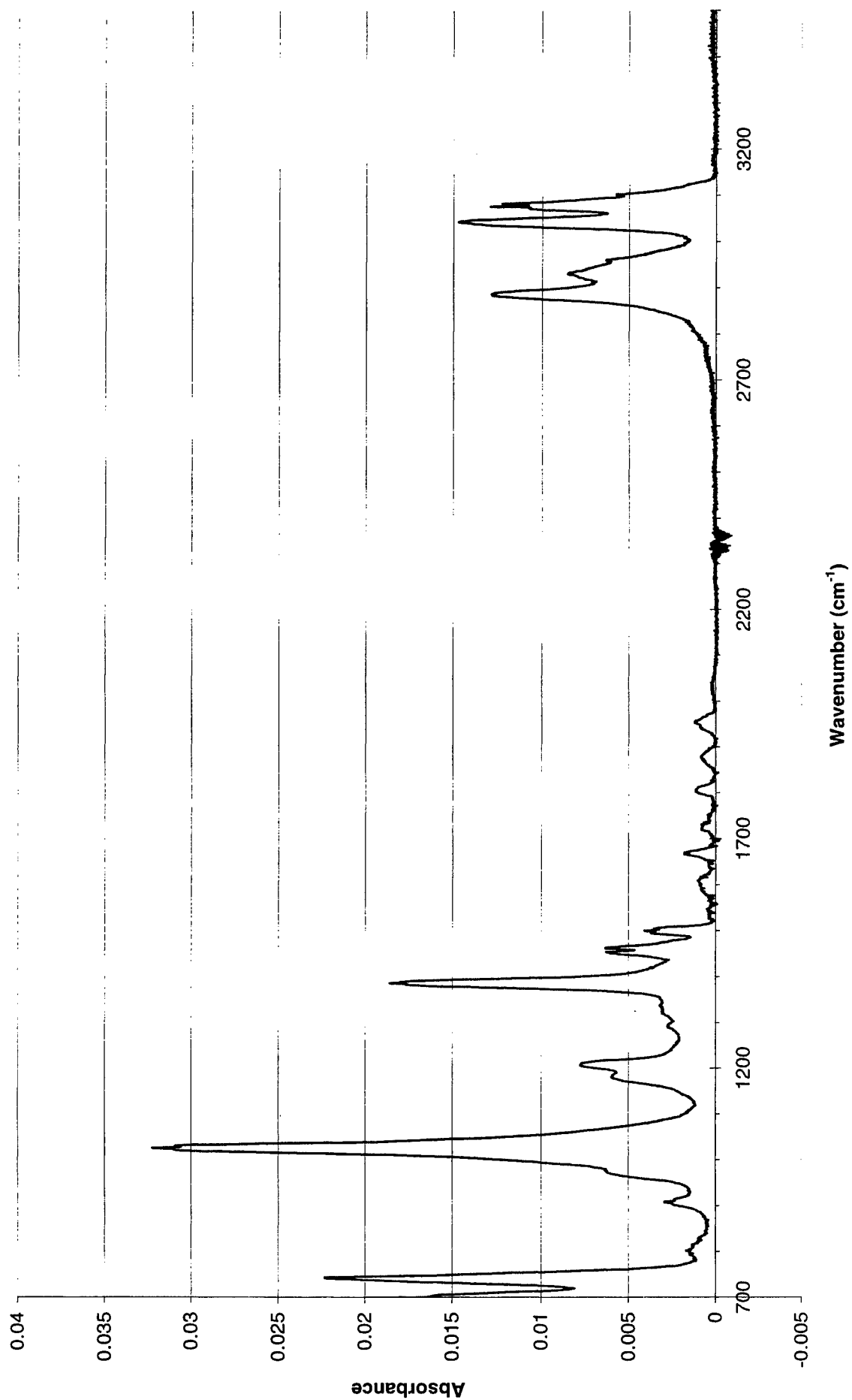


QIRL0300 - 2-(Diethylamino) ethanol
173 ppm-m





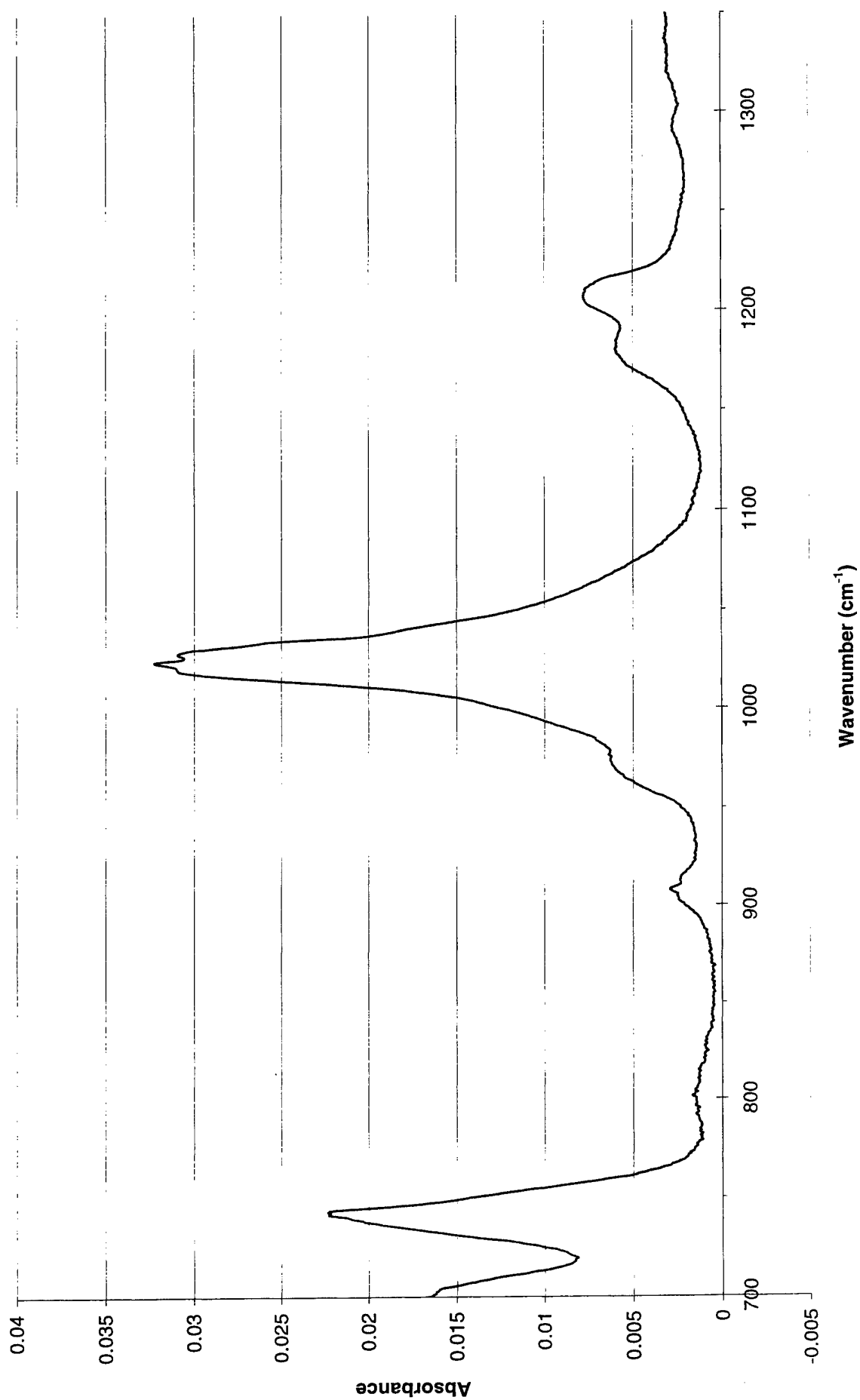
QIRL0310 - Benzyl alcohol
84 ppm-m





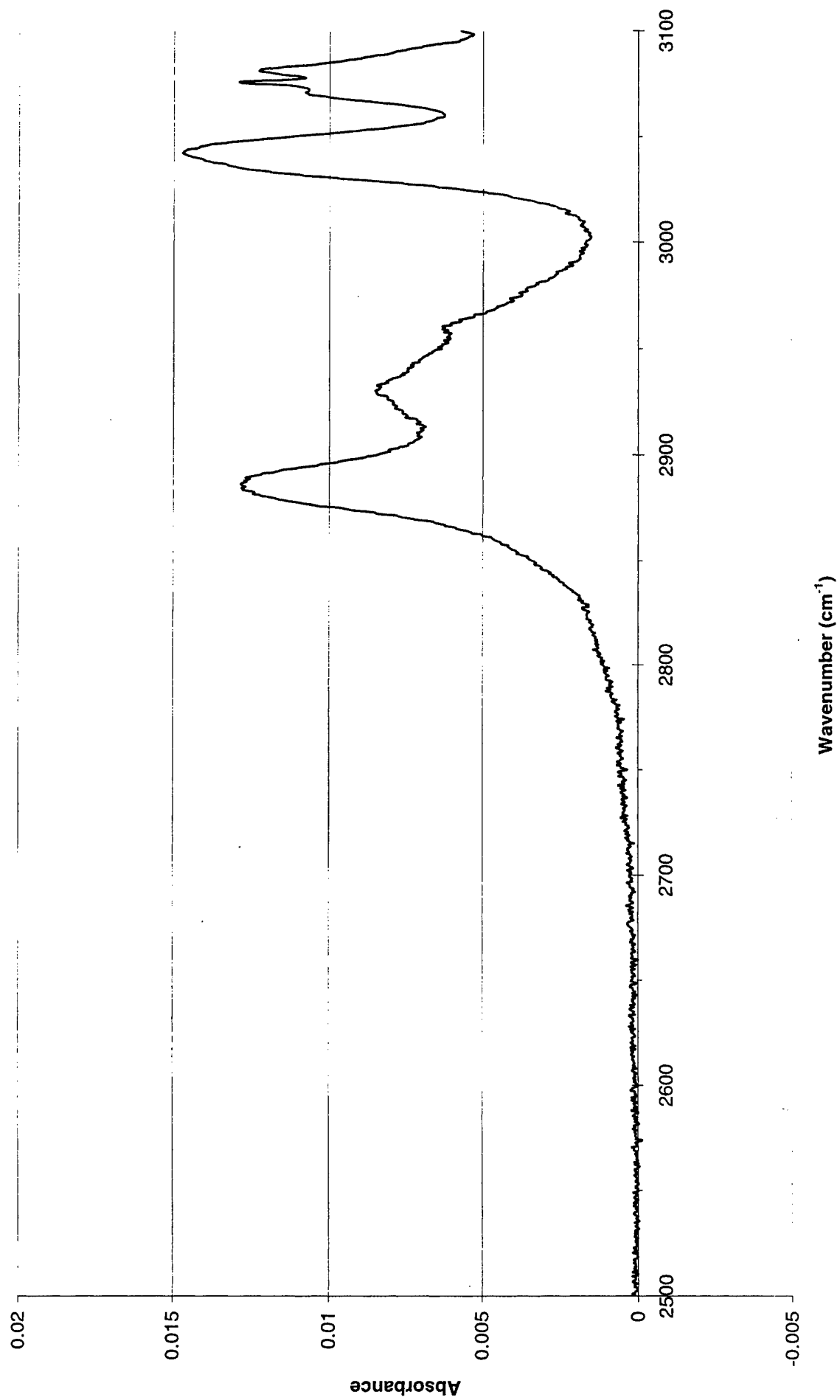
QIRL0310 - Benzyl alcohol

84 ppm-m





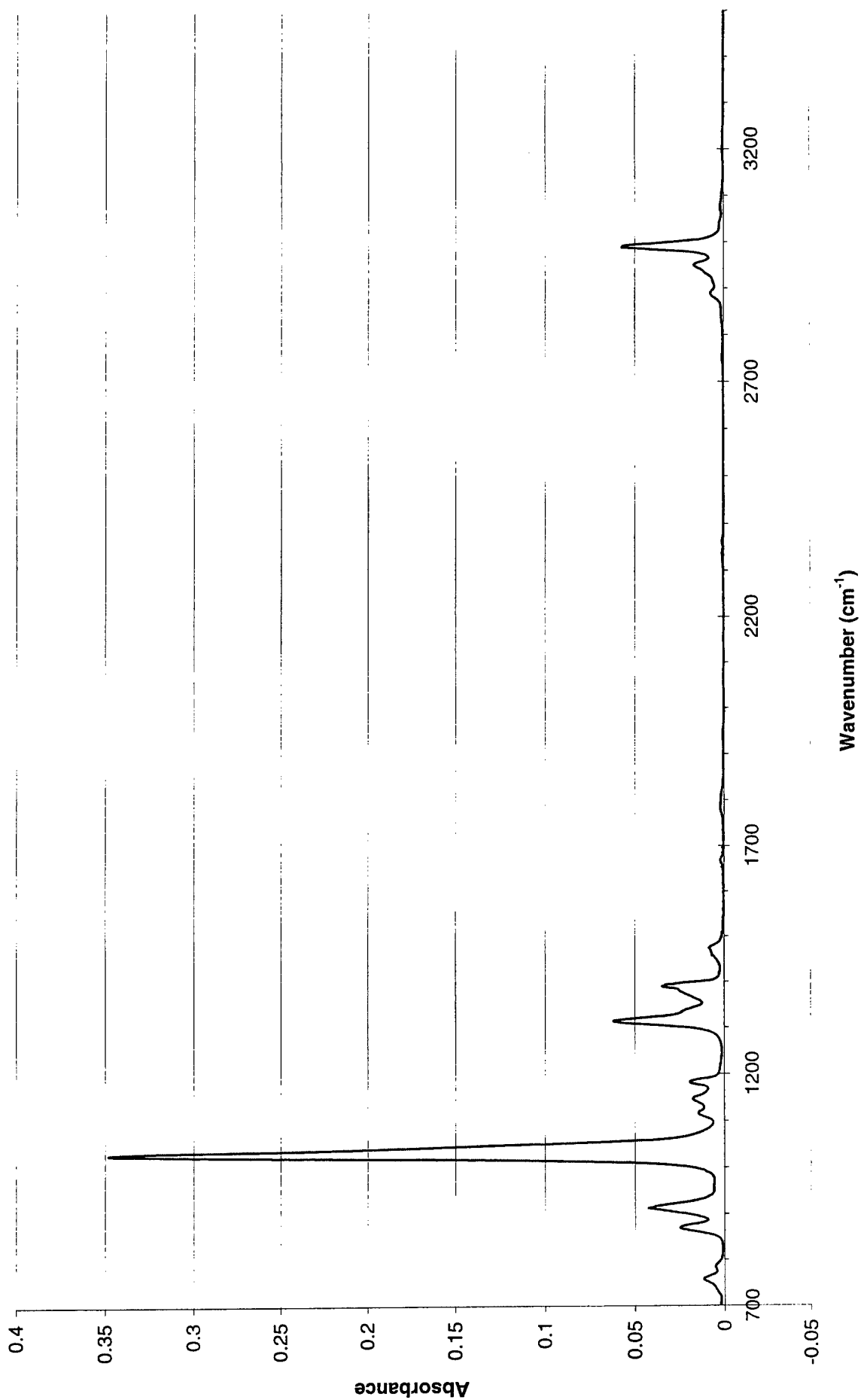
QIRL0310 - Benzyl alcohol
84 ppm-m





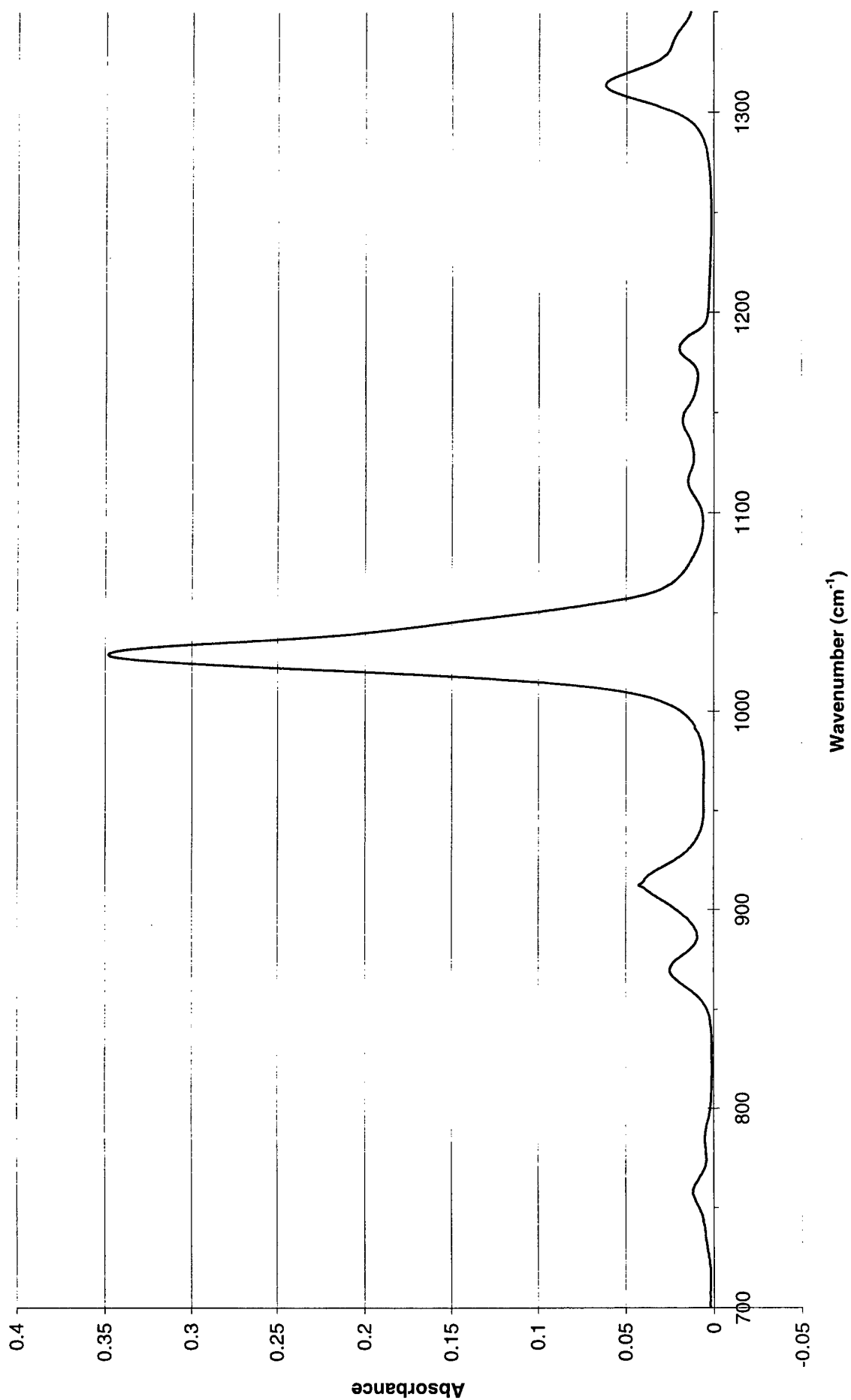
QIRL0320 - Diisopropyl fluorophosphate

104 ppm-m



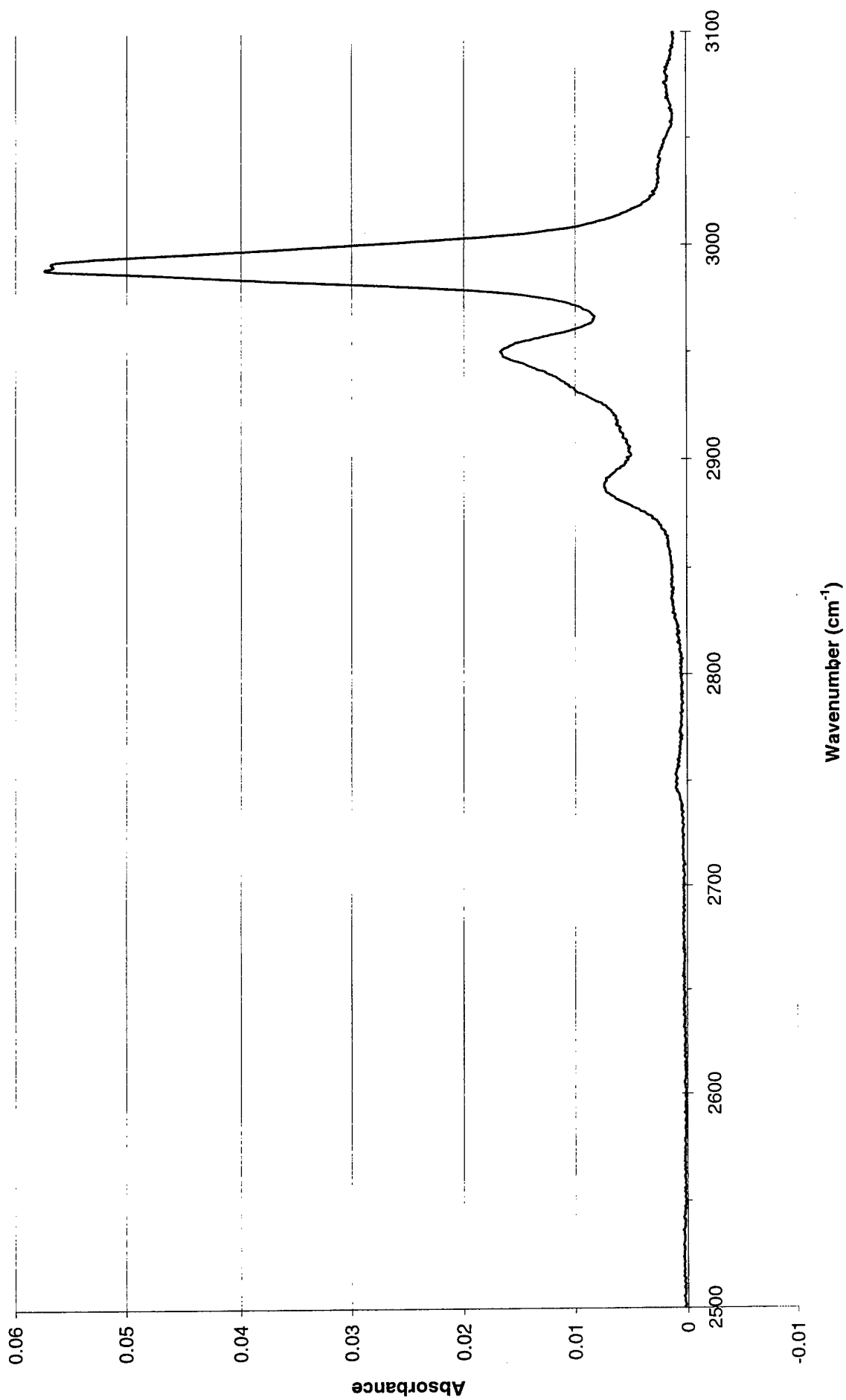


QIRL0320 - Diisopropyl fluorophosphate
104 ppm-m



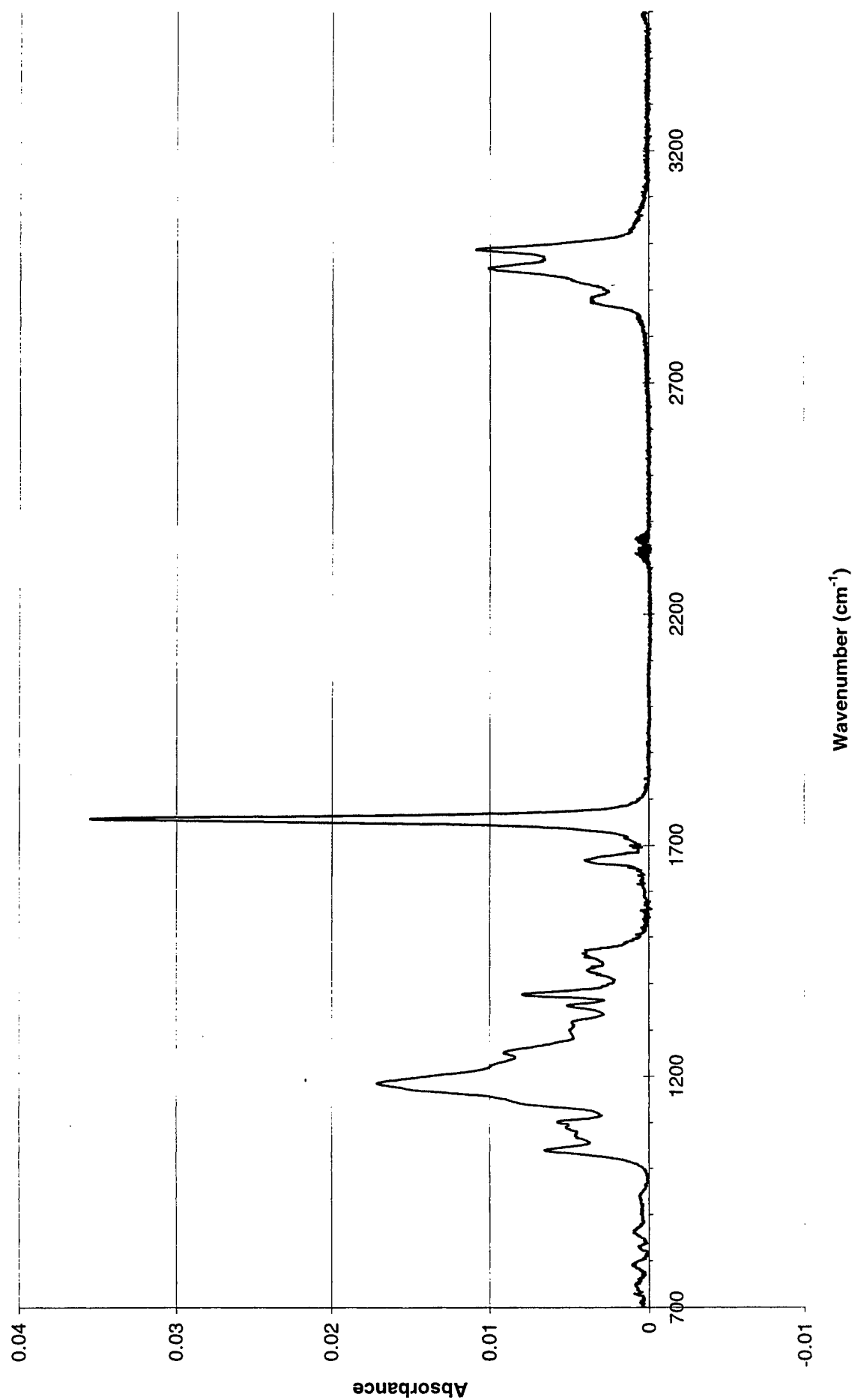
QIRL0320 - Diisopropyl fluorophosphate

104 ppm-m



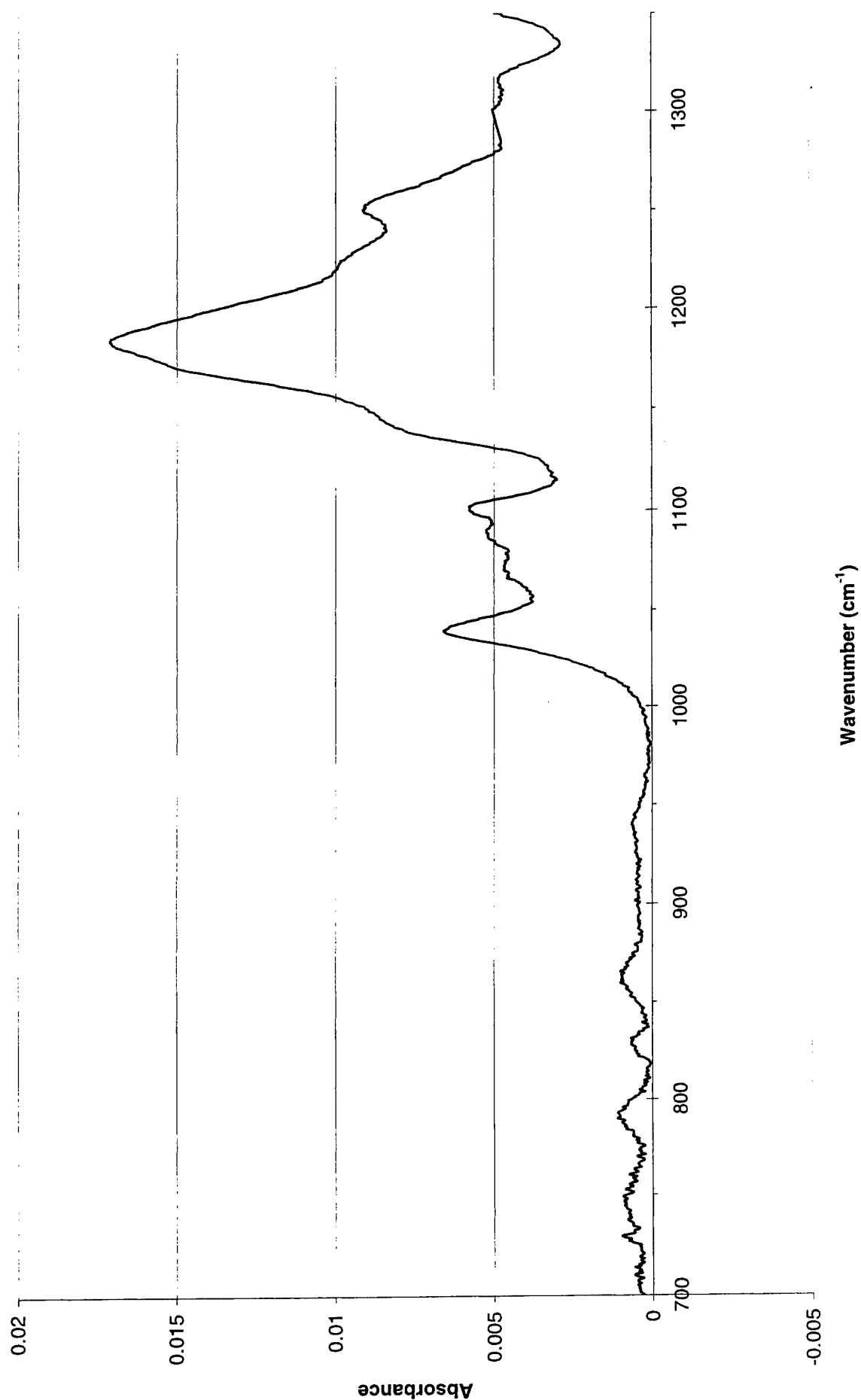


QIRL0330 - Diethyl pimelate
10 ppm-m



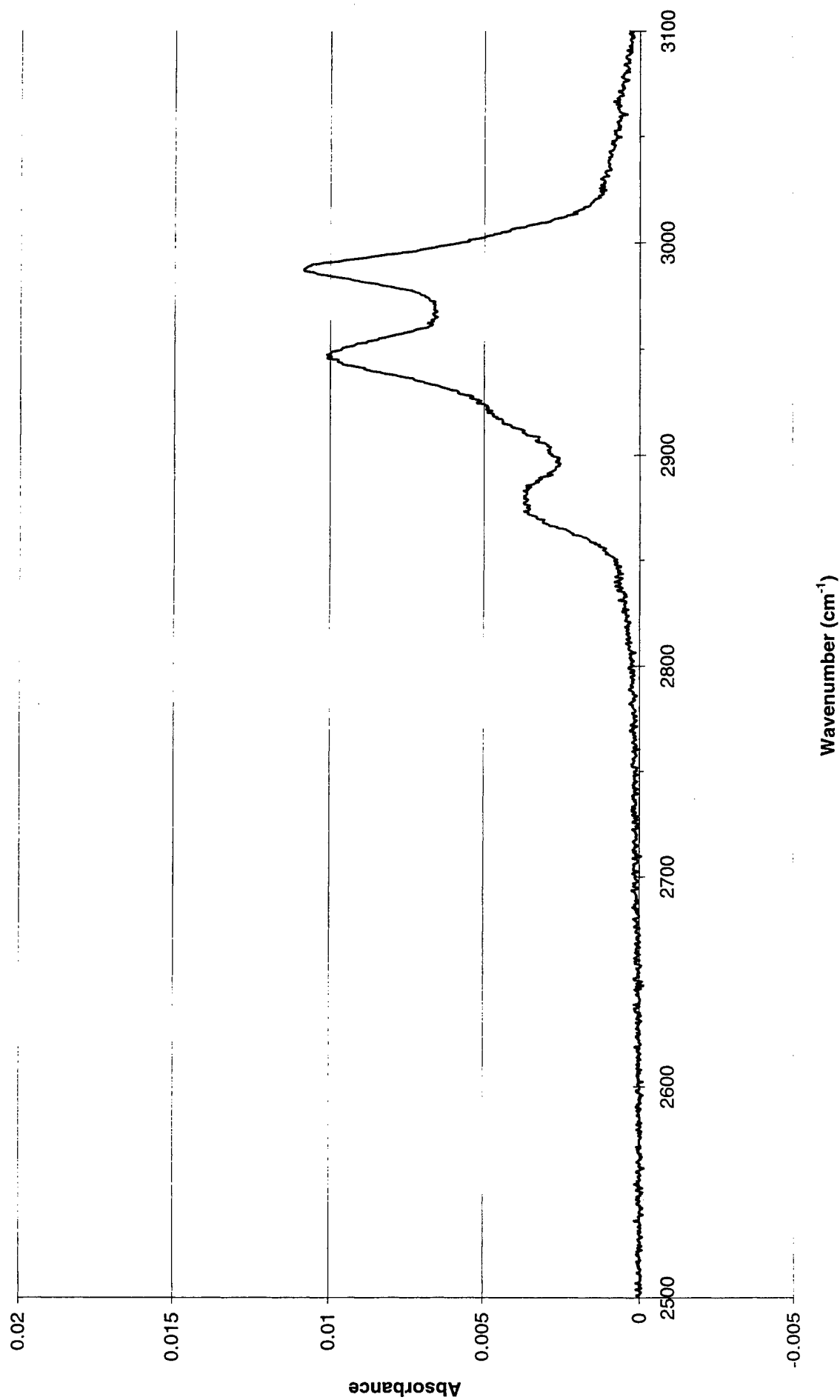


QIRL0330 - Diethyl pimelate
10 ppm-m



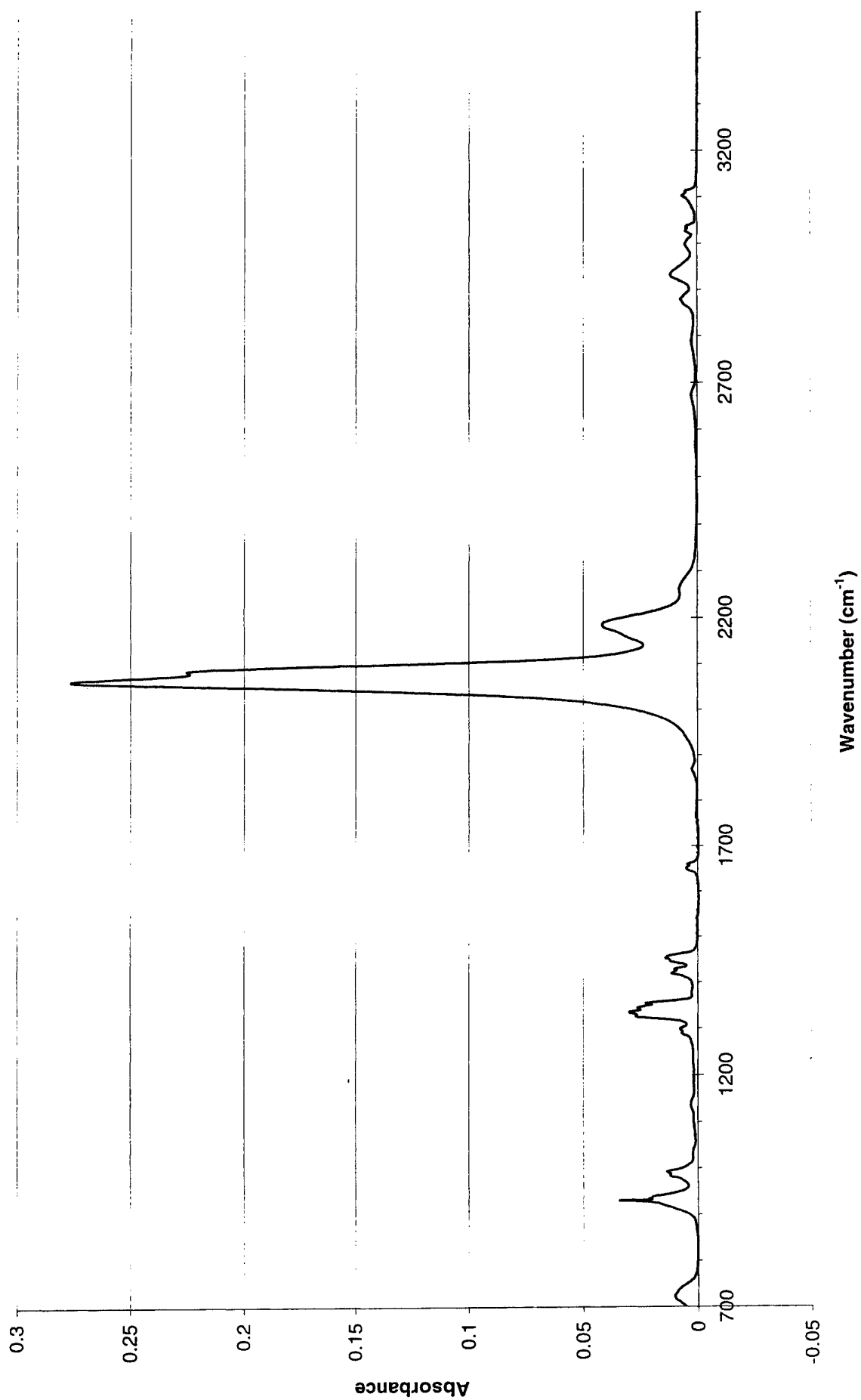


QIRL0330 - Diethyl pimelate
10 ppm-m



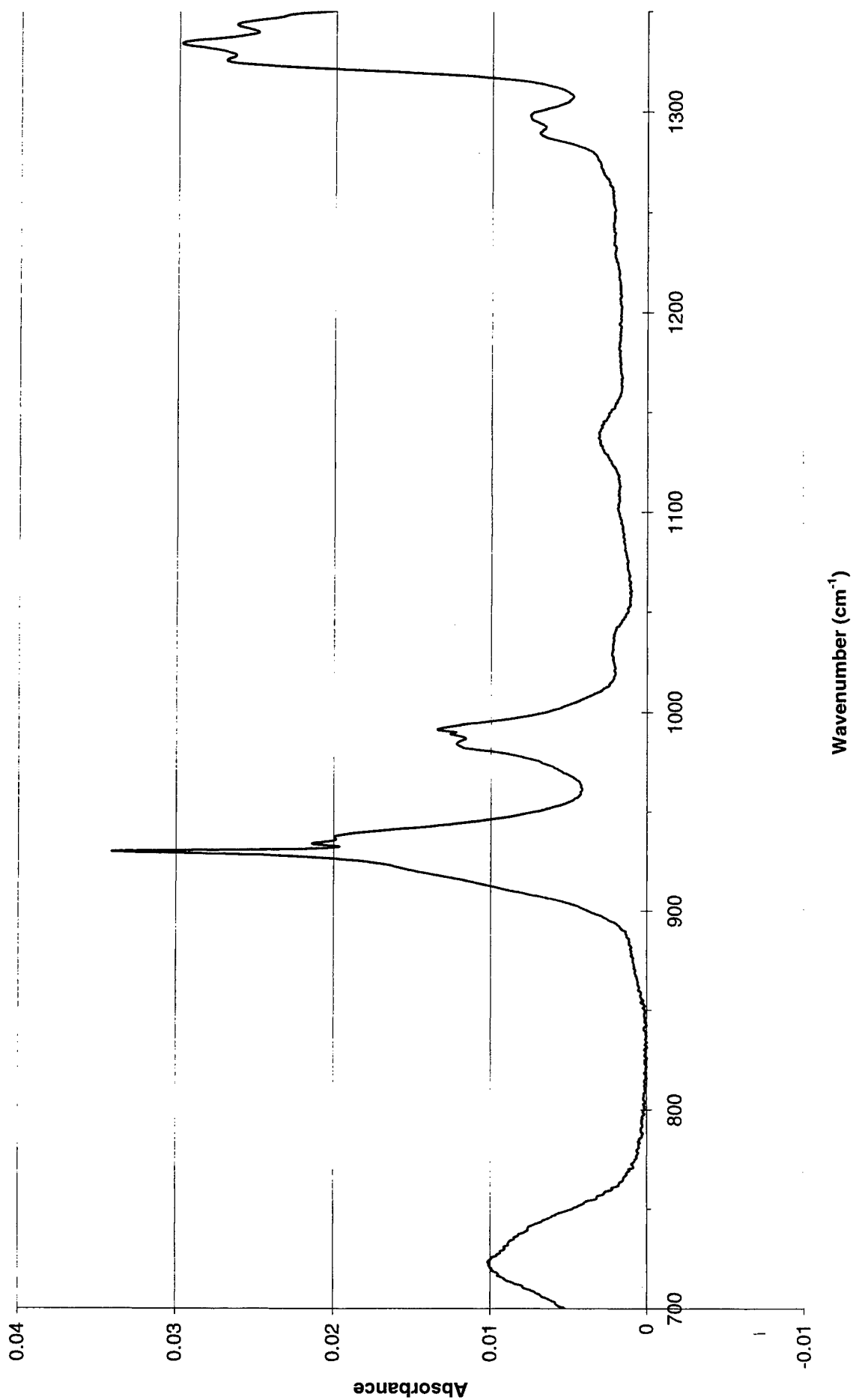


QIRL0340 - Allyl isothiocyanate
128 ppm-m



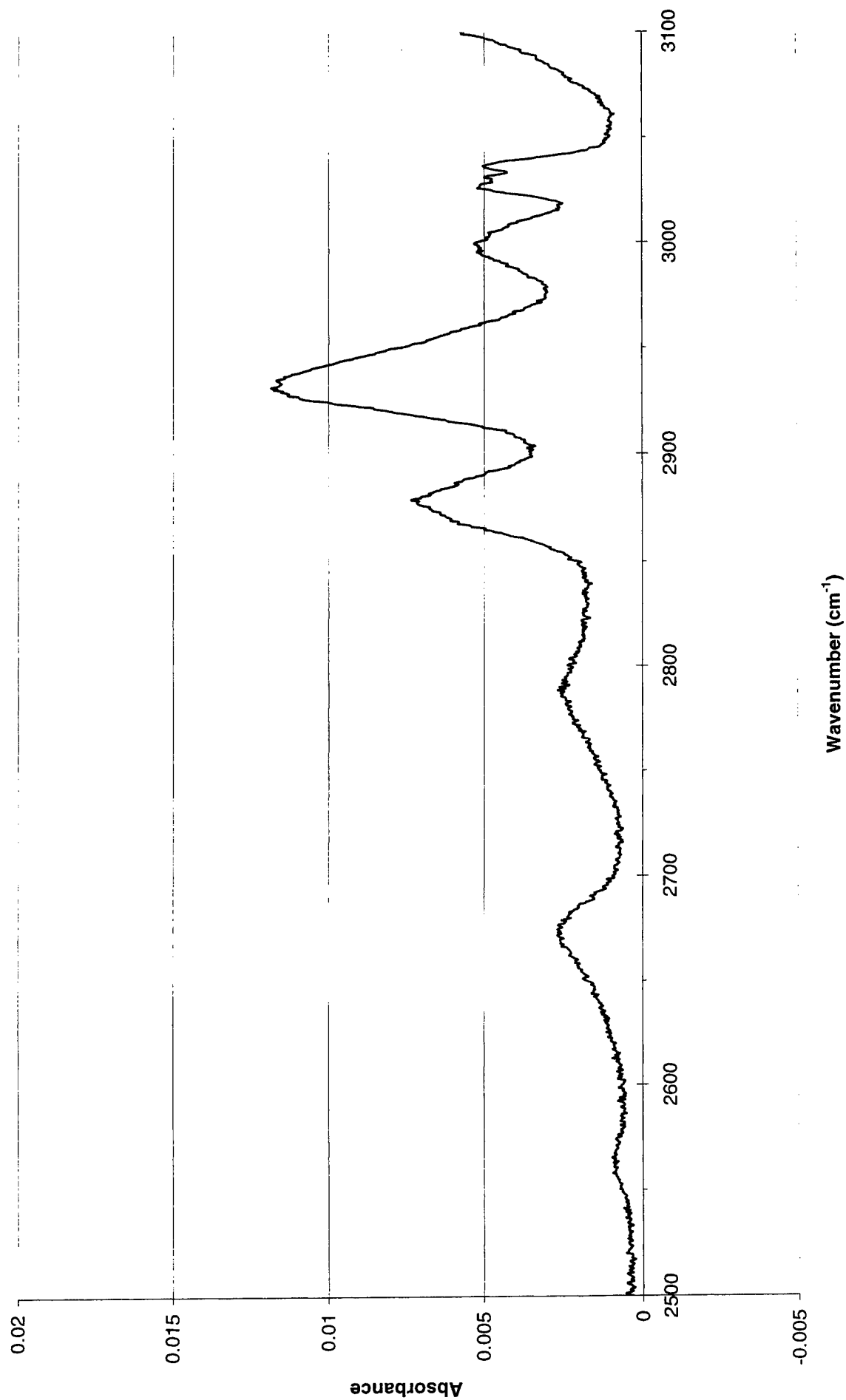


QIRL0340 - Allyl isothiocyanate
128 ppm-m



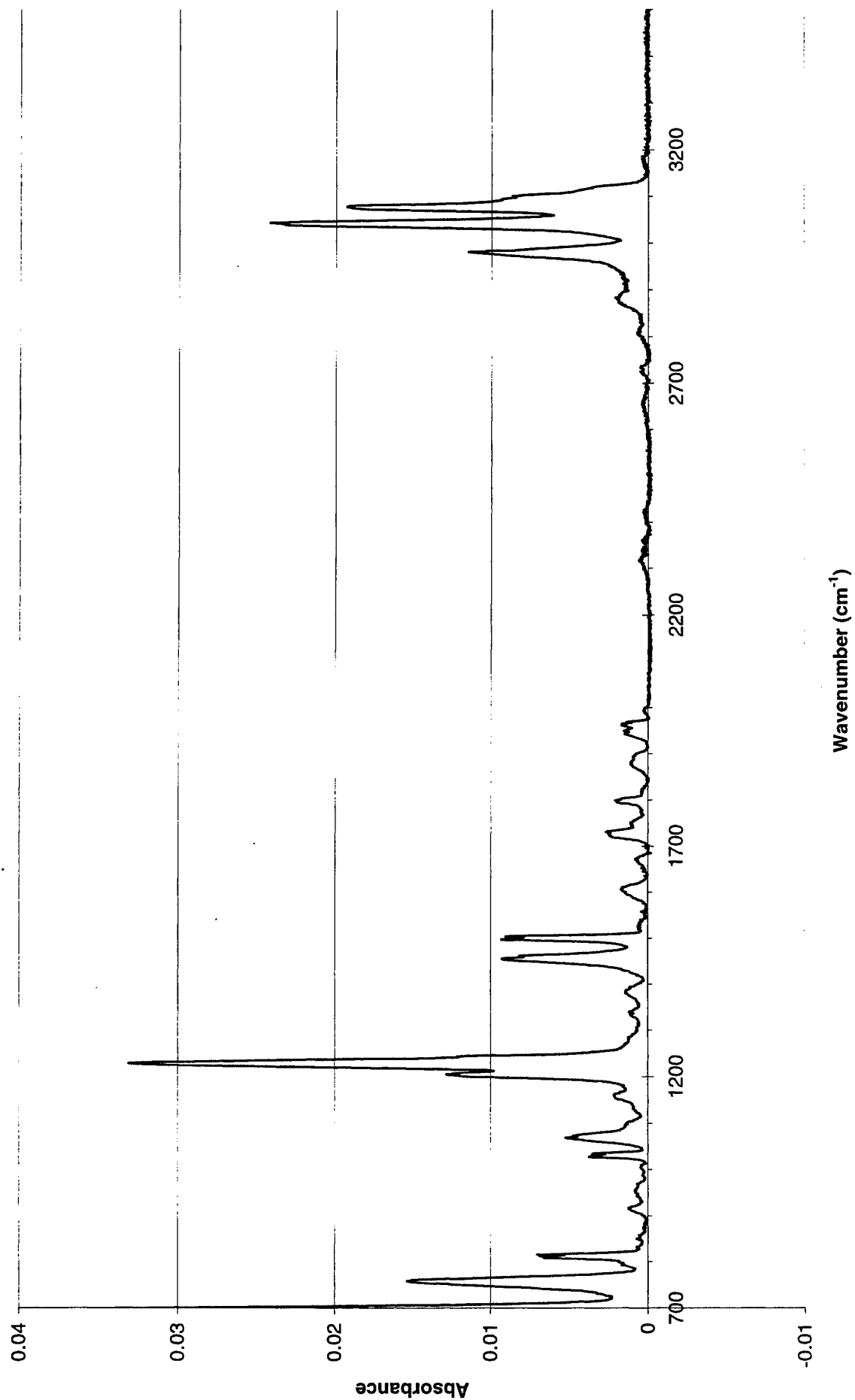


QIRL0340 - Allyl isothiocyanate
128 ppm-m



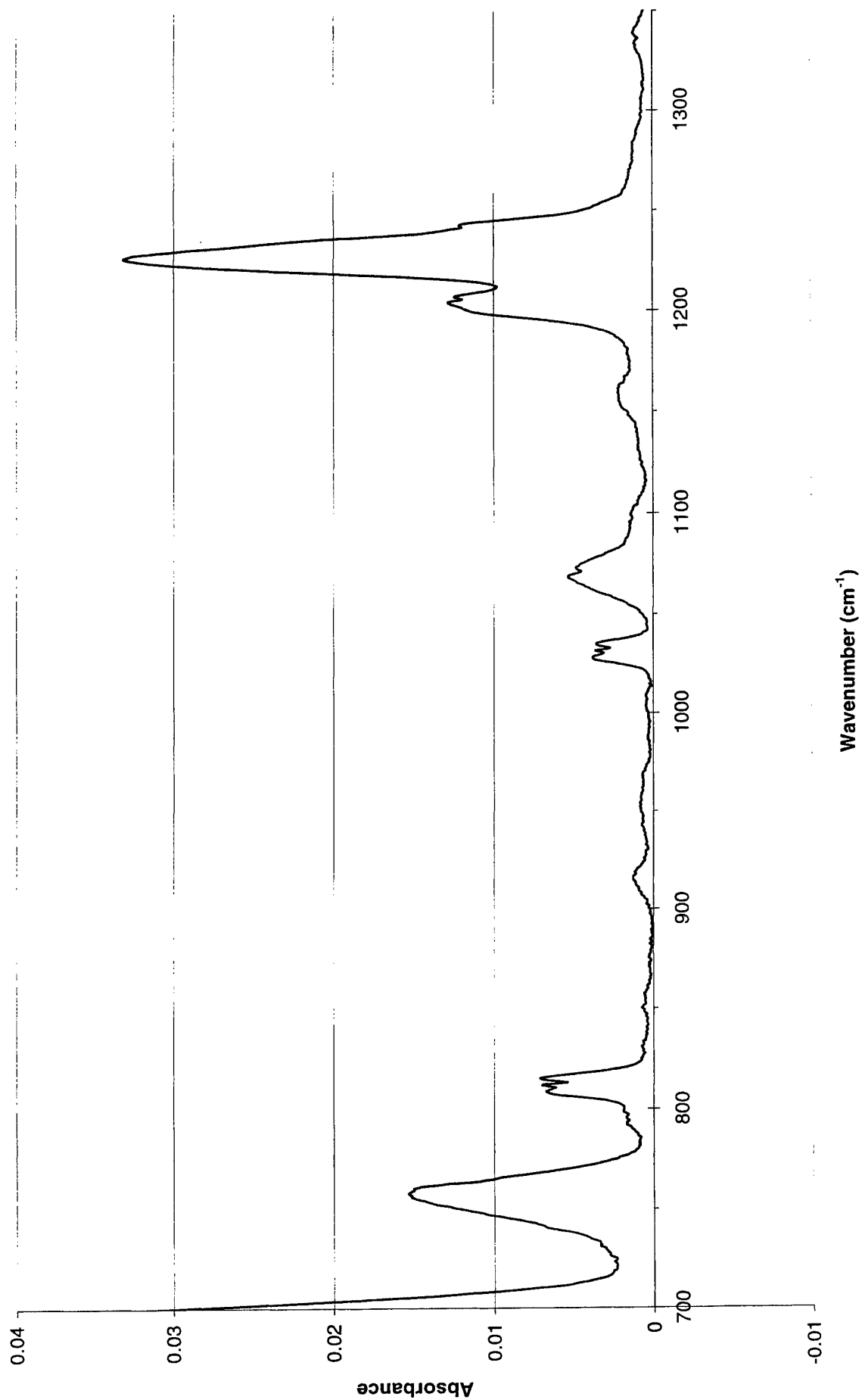


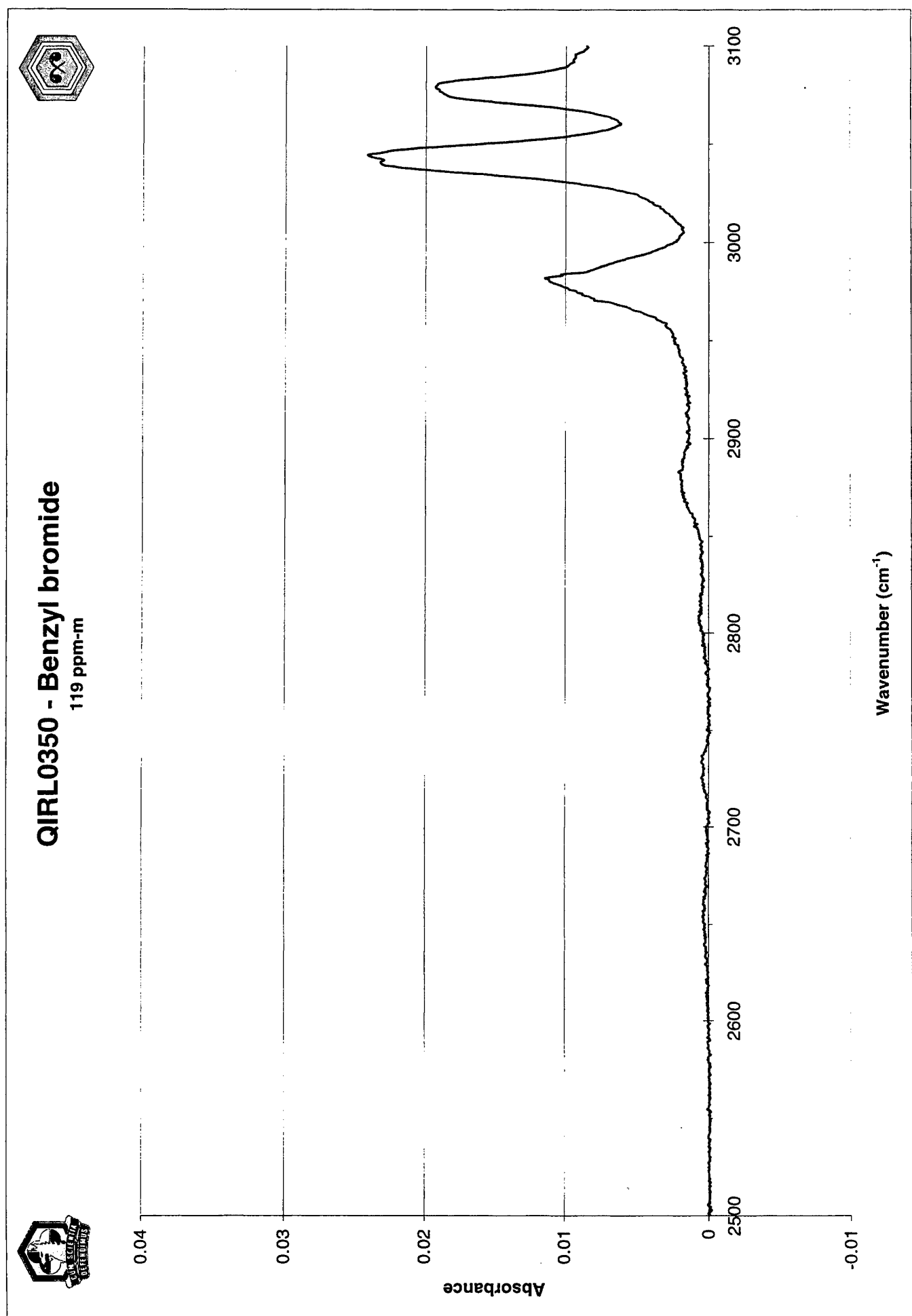
QIRL0350 - Benzyl bromide
119 ppm-m





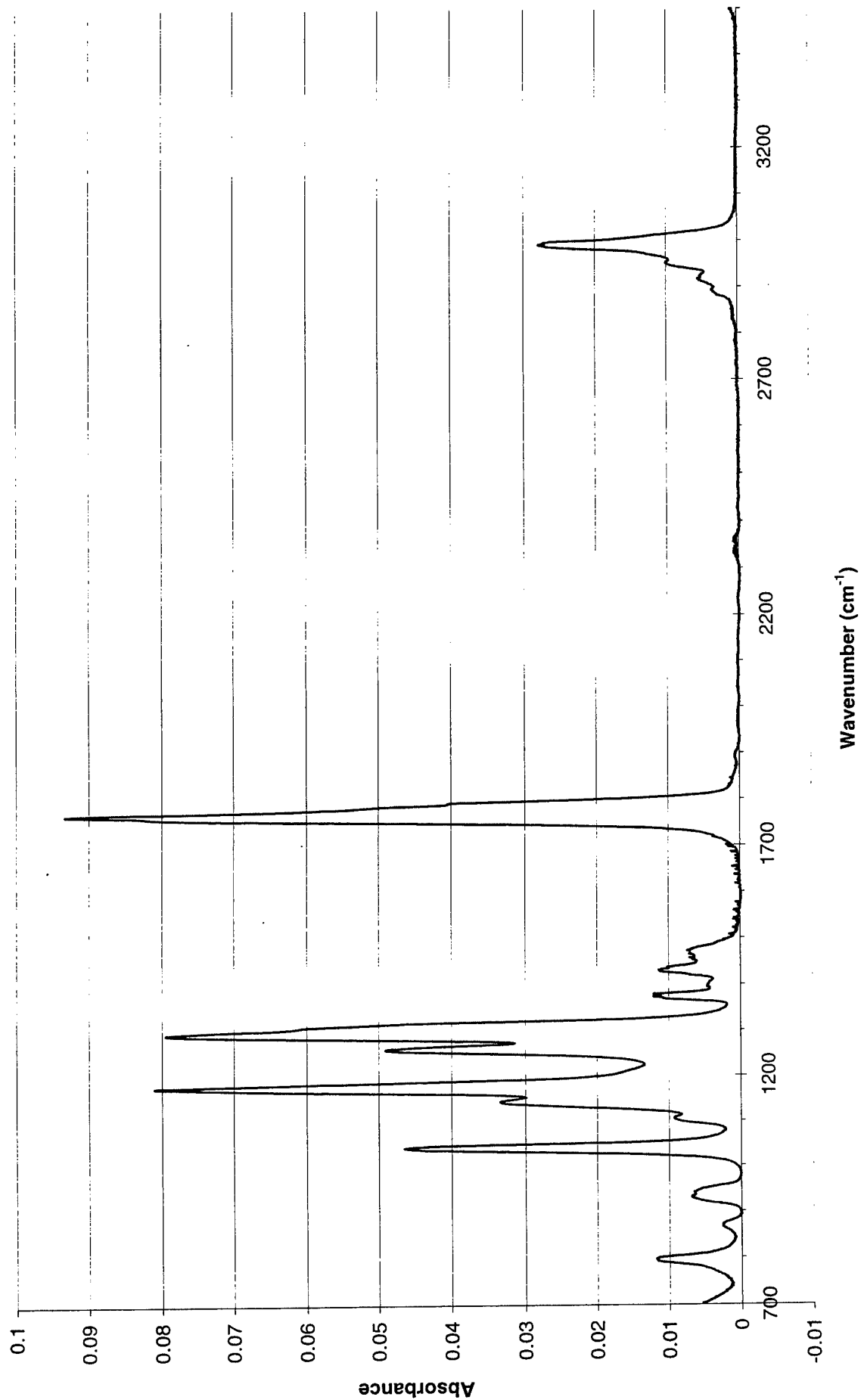
QIRL0350 - Benzyl bromide
119 ppm-m





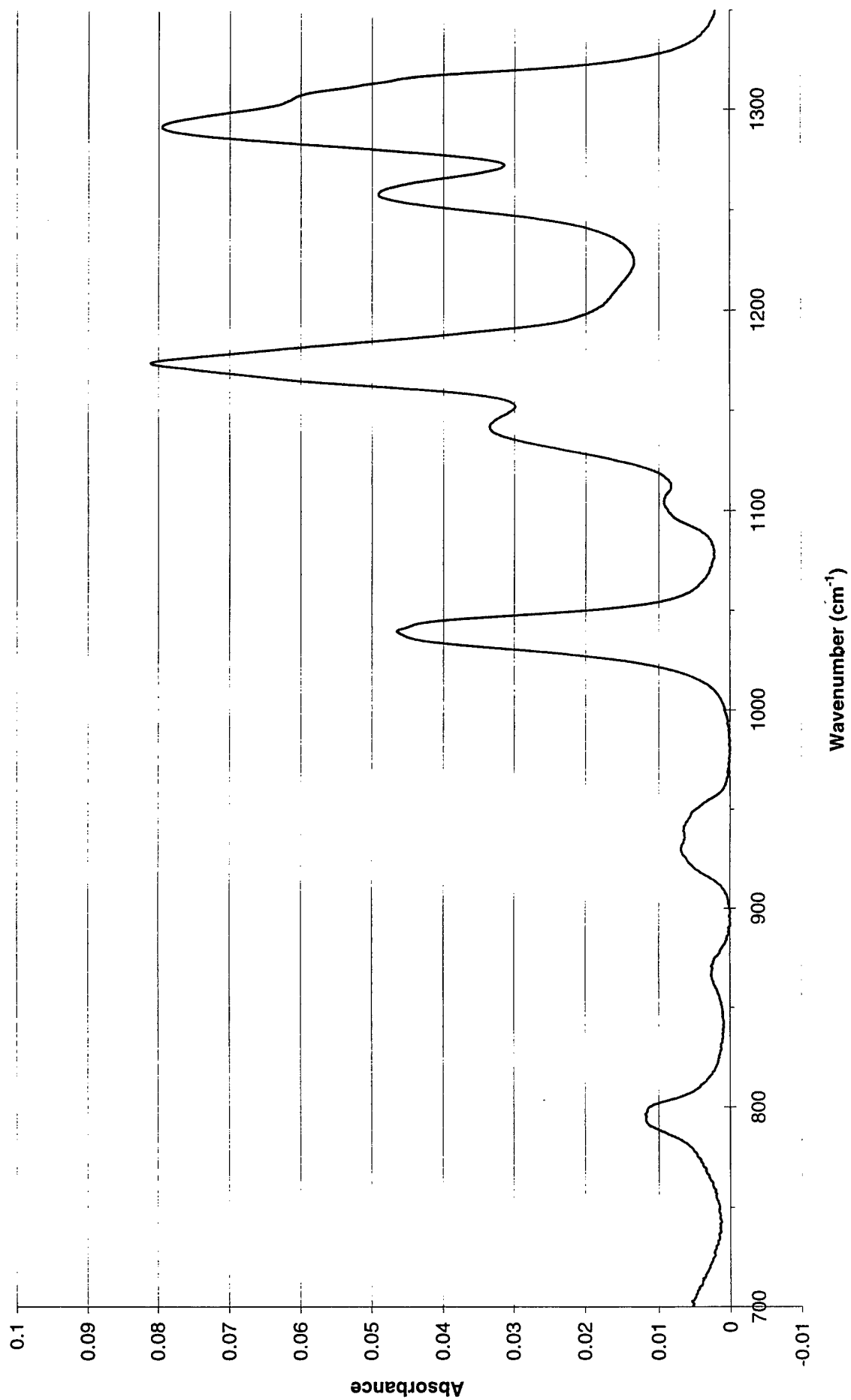


QIRL0360 - Ethyl chloroacetate
103 ppm-m



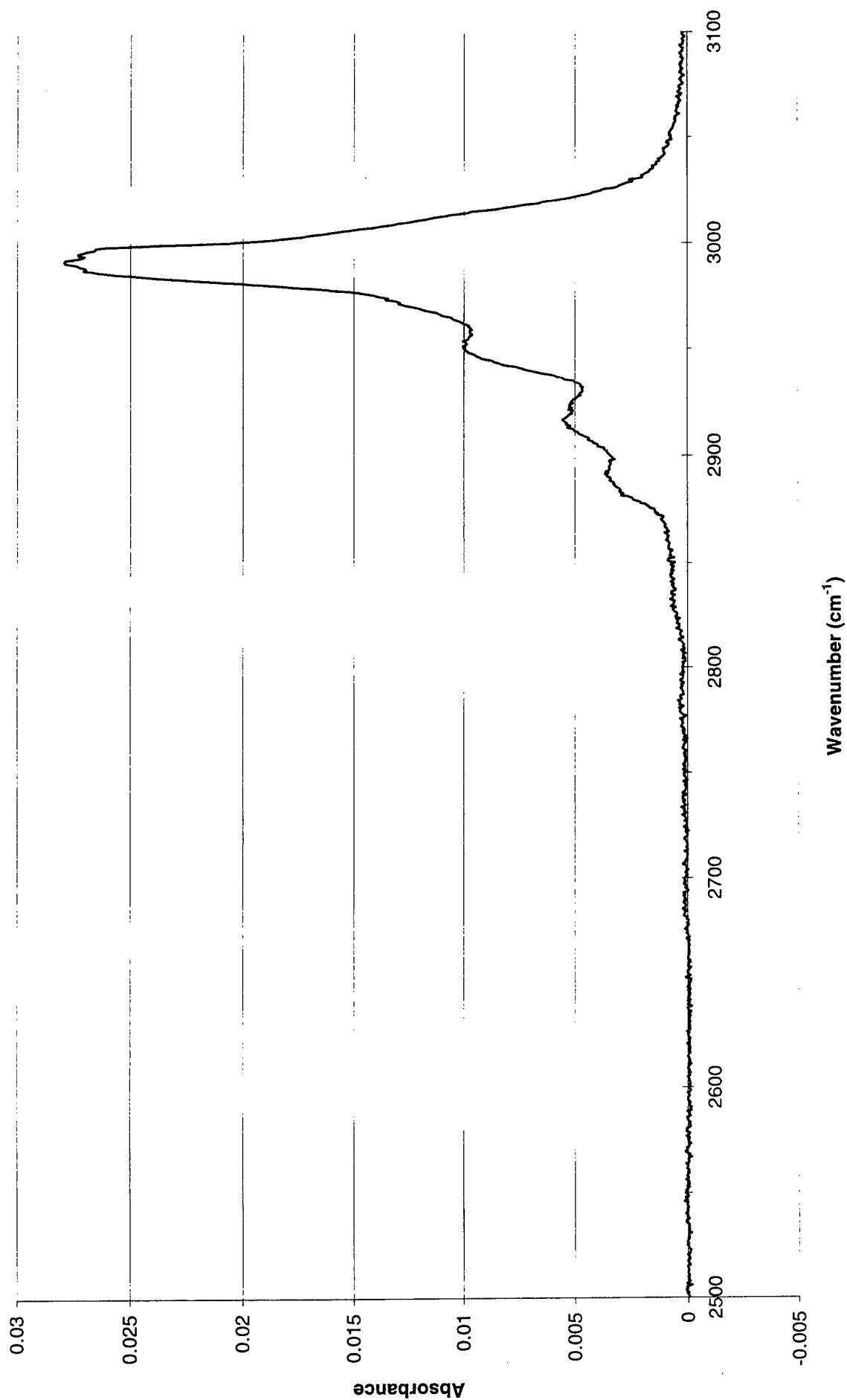


QIRL0360 - Ethyl chloroacetate
103 ppm-m



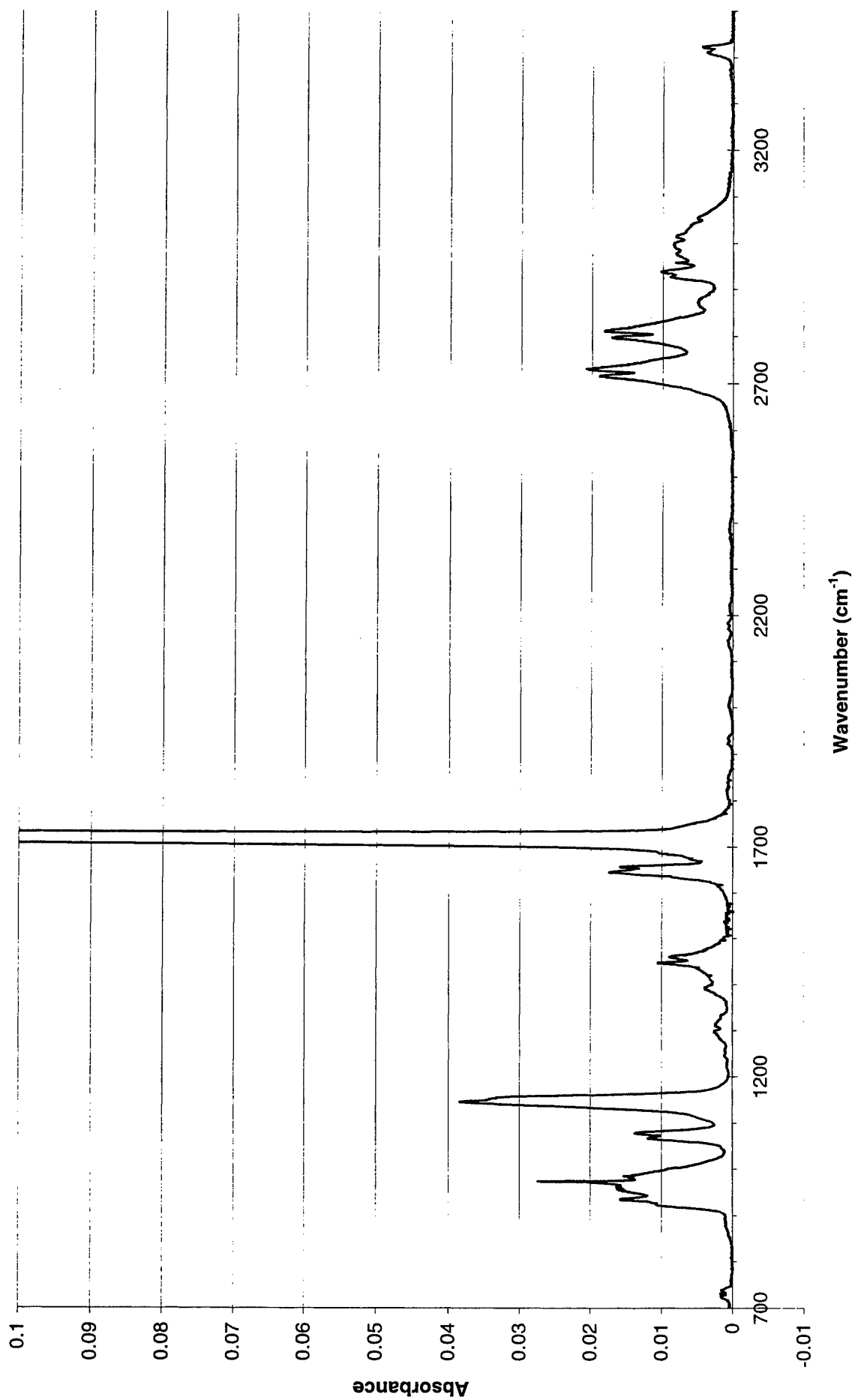


QIRL0360 - Ethyl chloroacetate
103 ppm-m



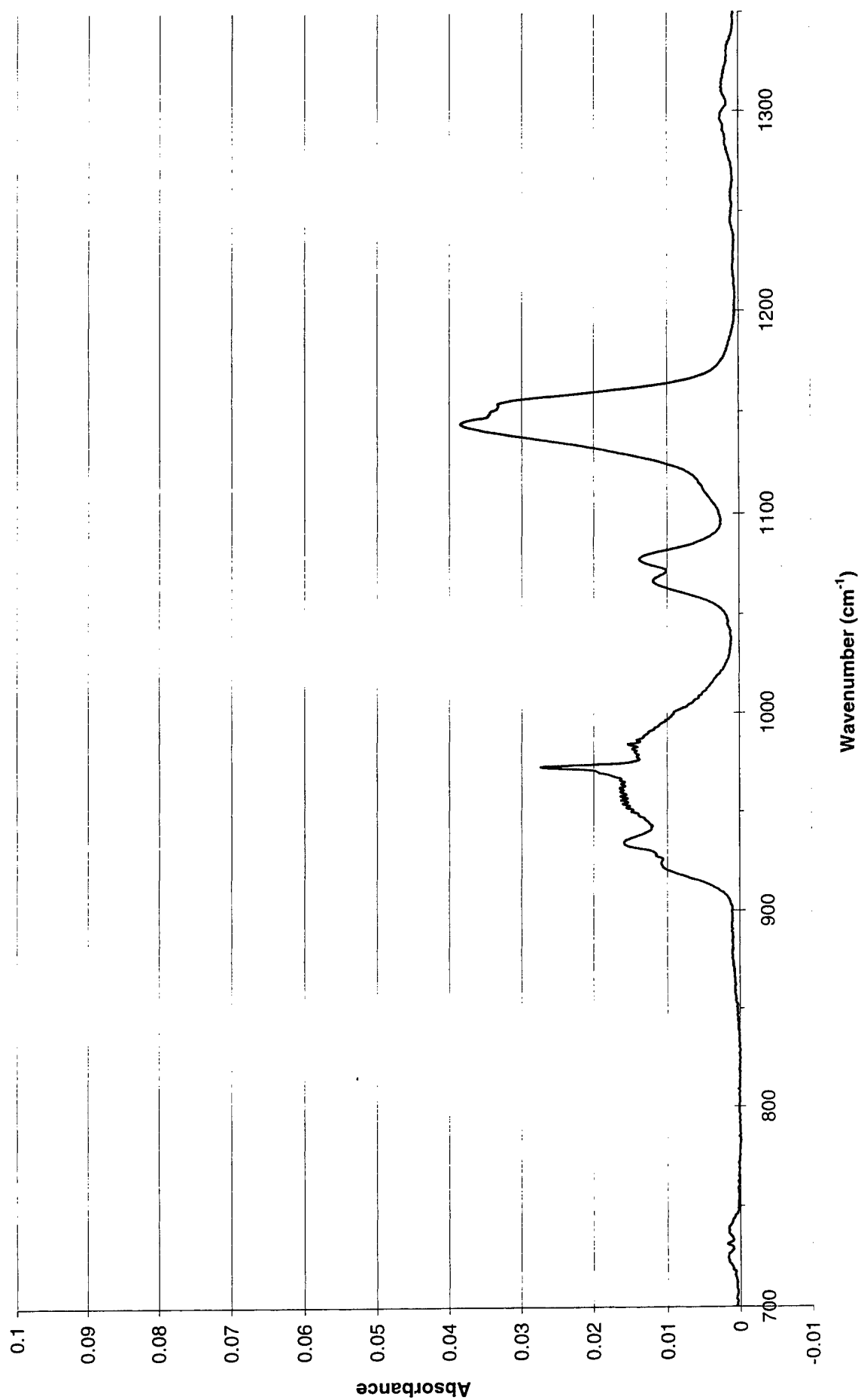


QIRL0370 - 2-Butenal
158 ppm-m



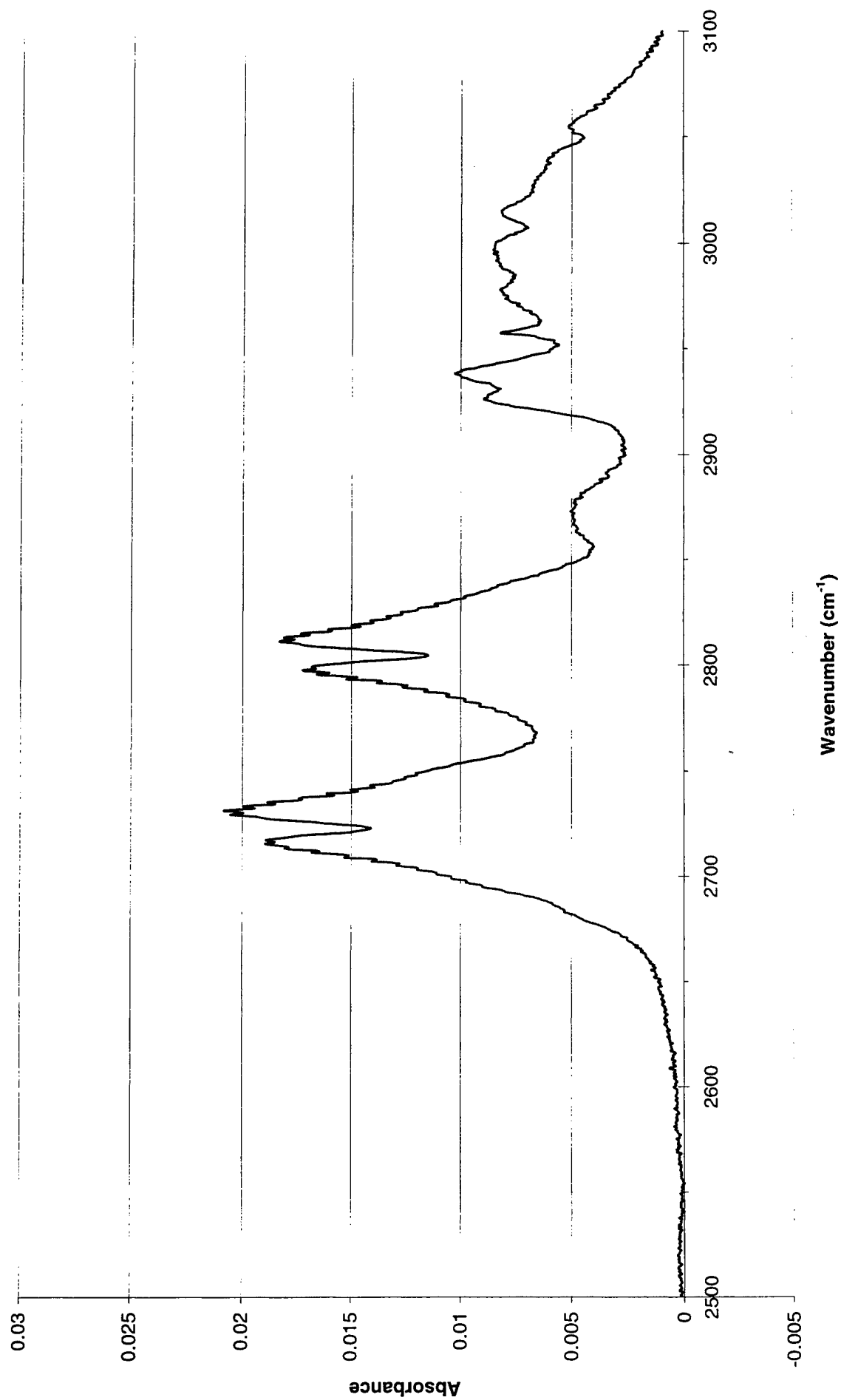


QIRL0370 - 2-Butenal
158 ppm-m



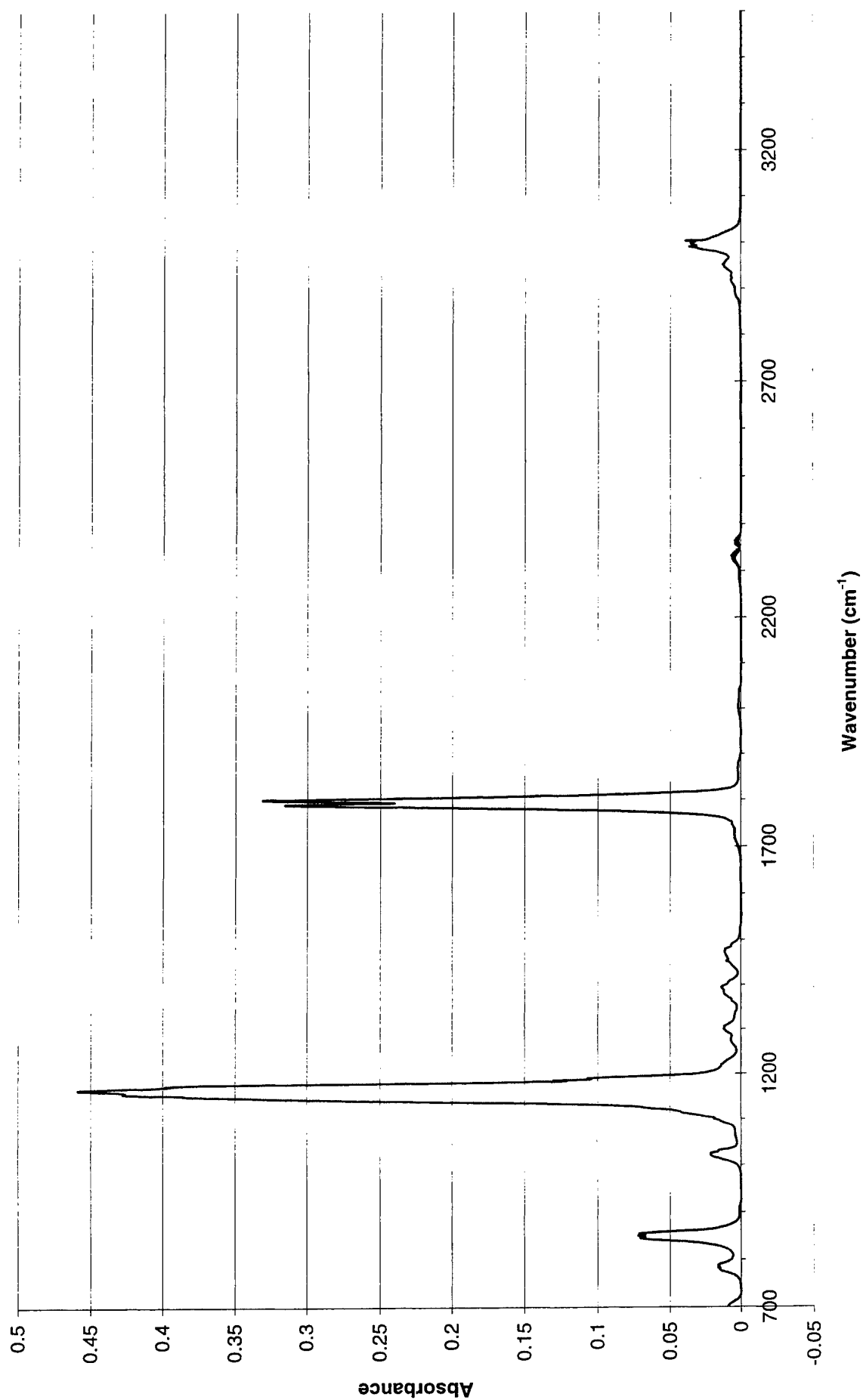


QIRL0370 - 2-Butenal
158 ppm-m





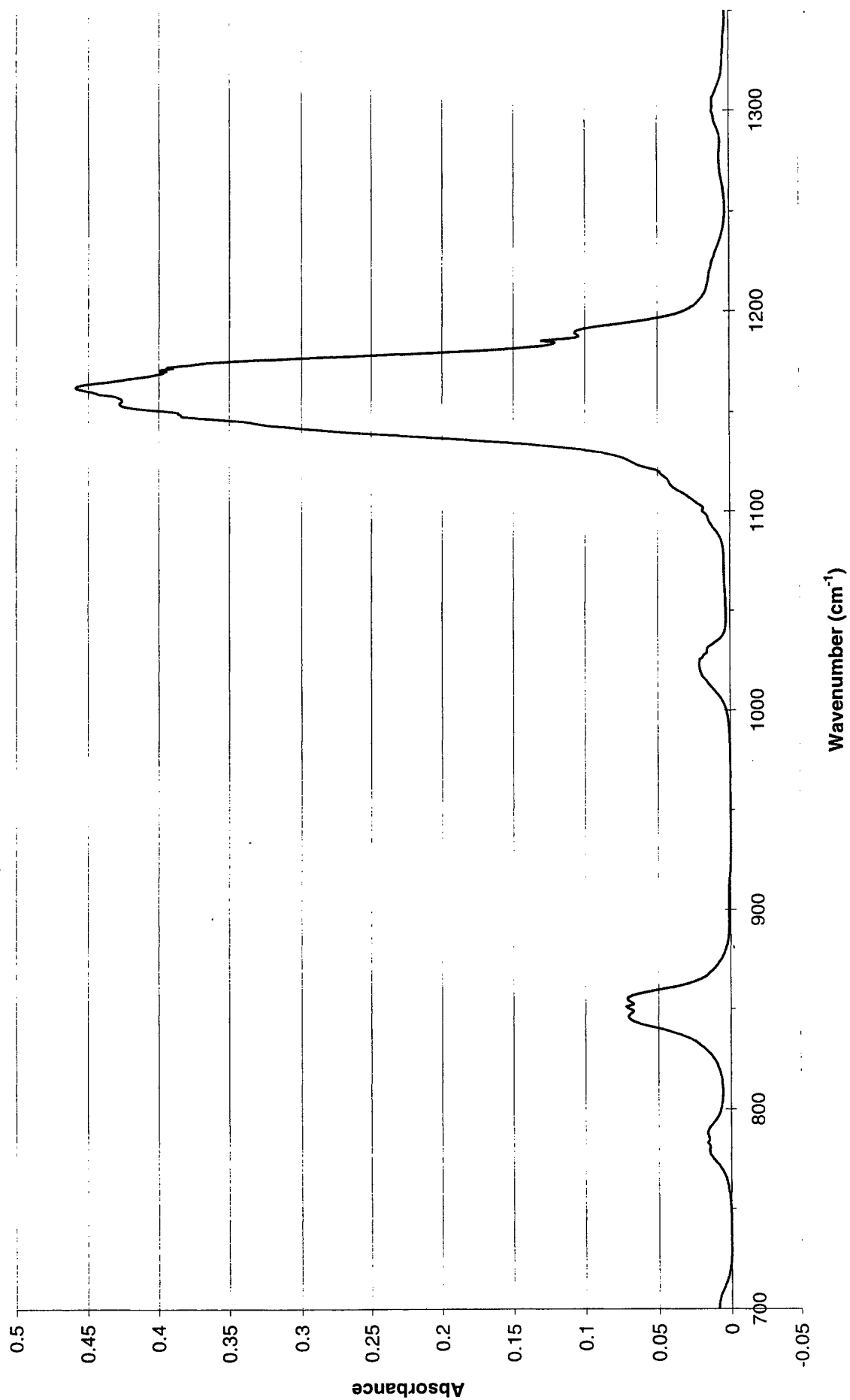
QIRL0380 - Ethyl chloroformate
209 ppm-m





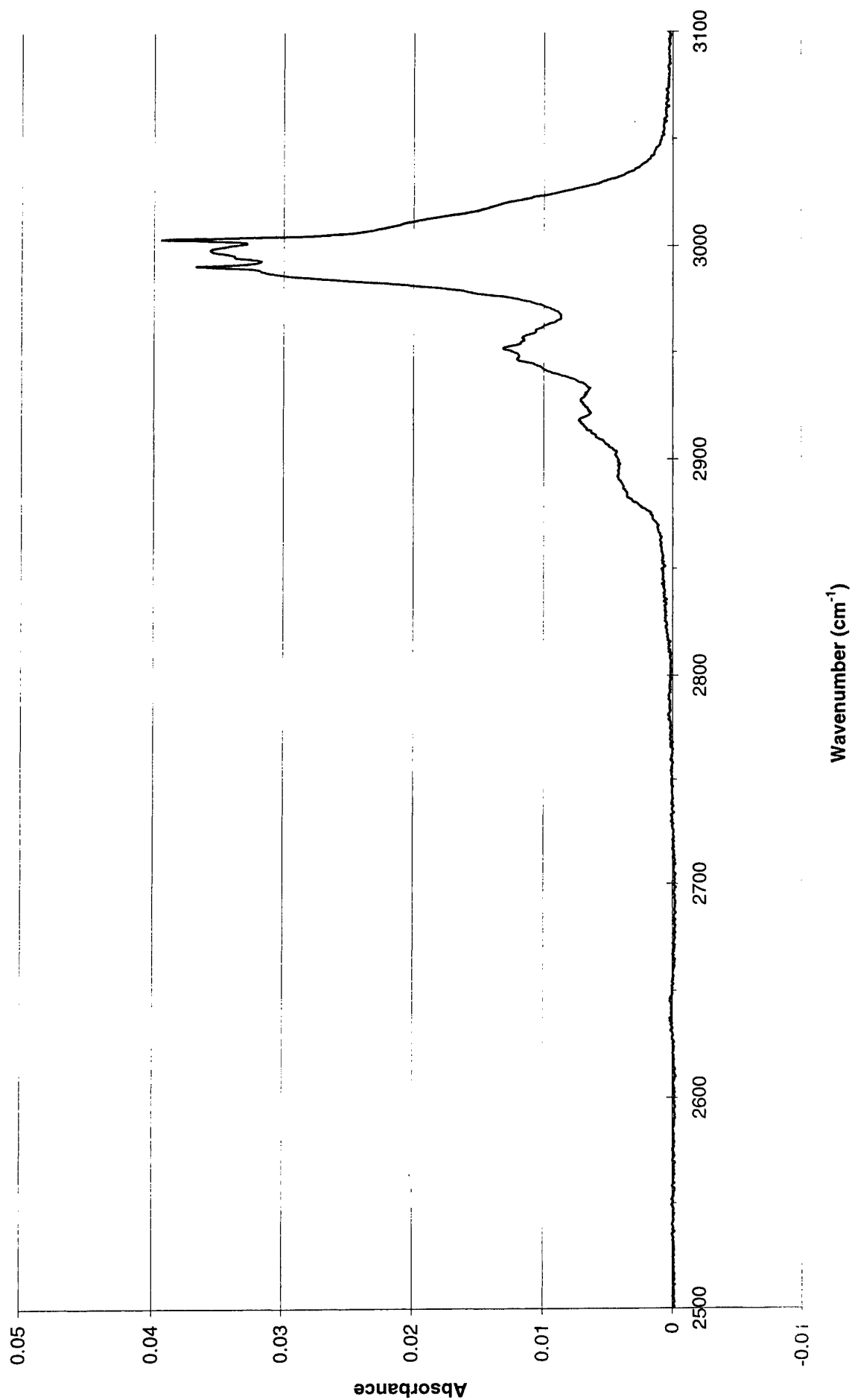
QIRL0380 - Ethyl chloroformate

209 ppm-m



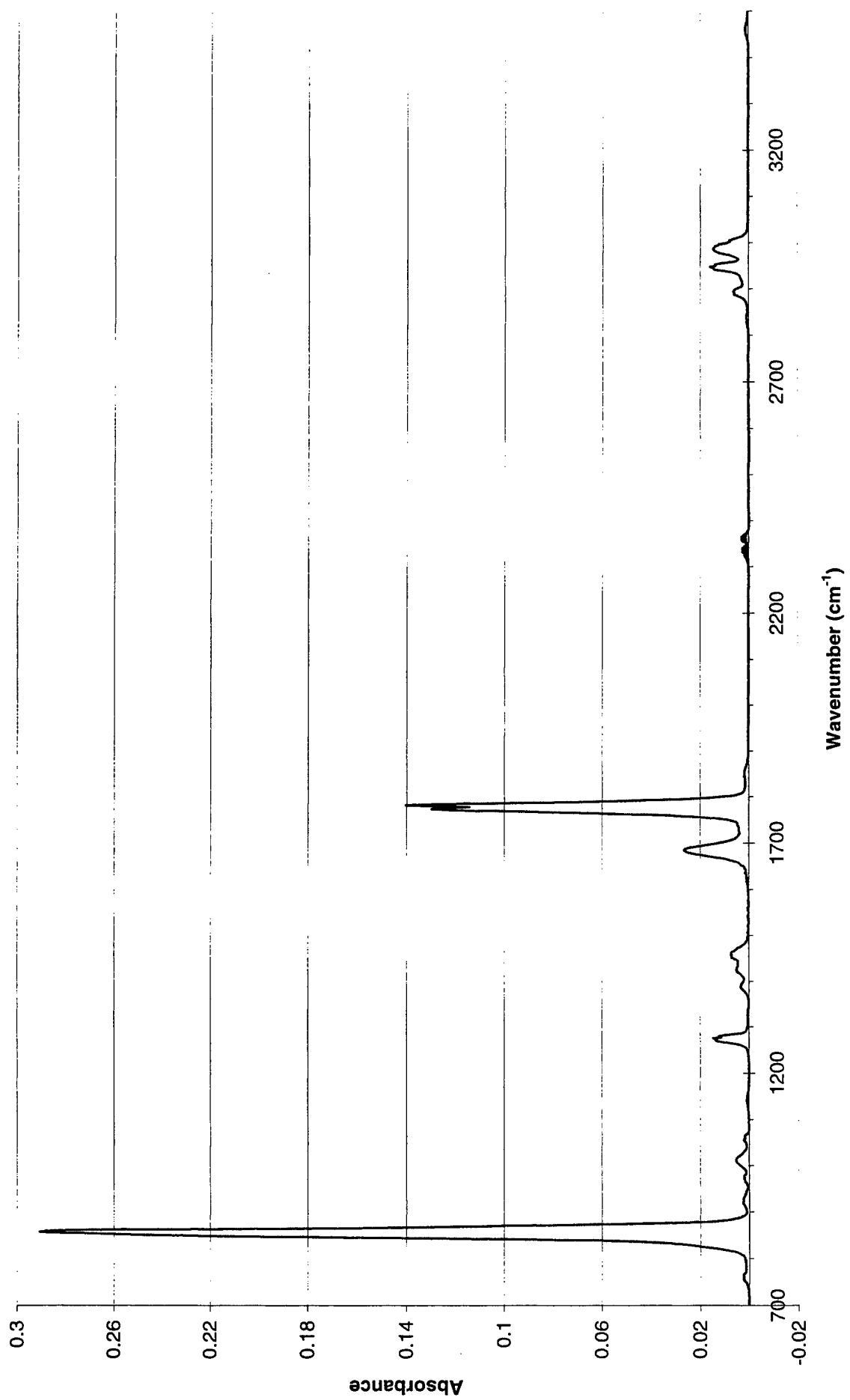


QIRL0380 - Ethyl chloroformate
209 ppm-m



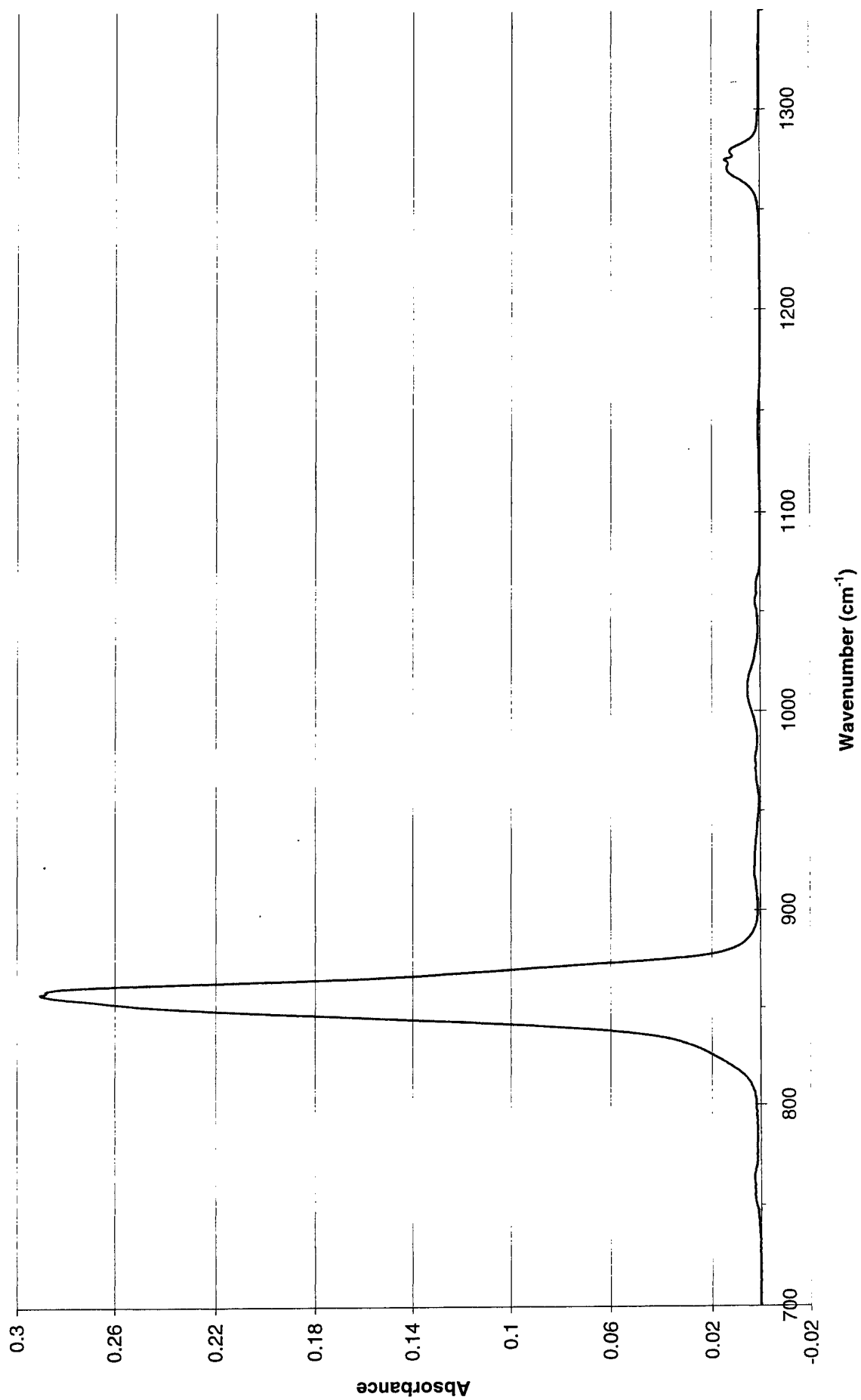


QIRL0390 - Ethyl chlorothiolformate
128 ppm-m



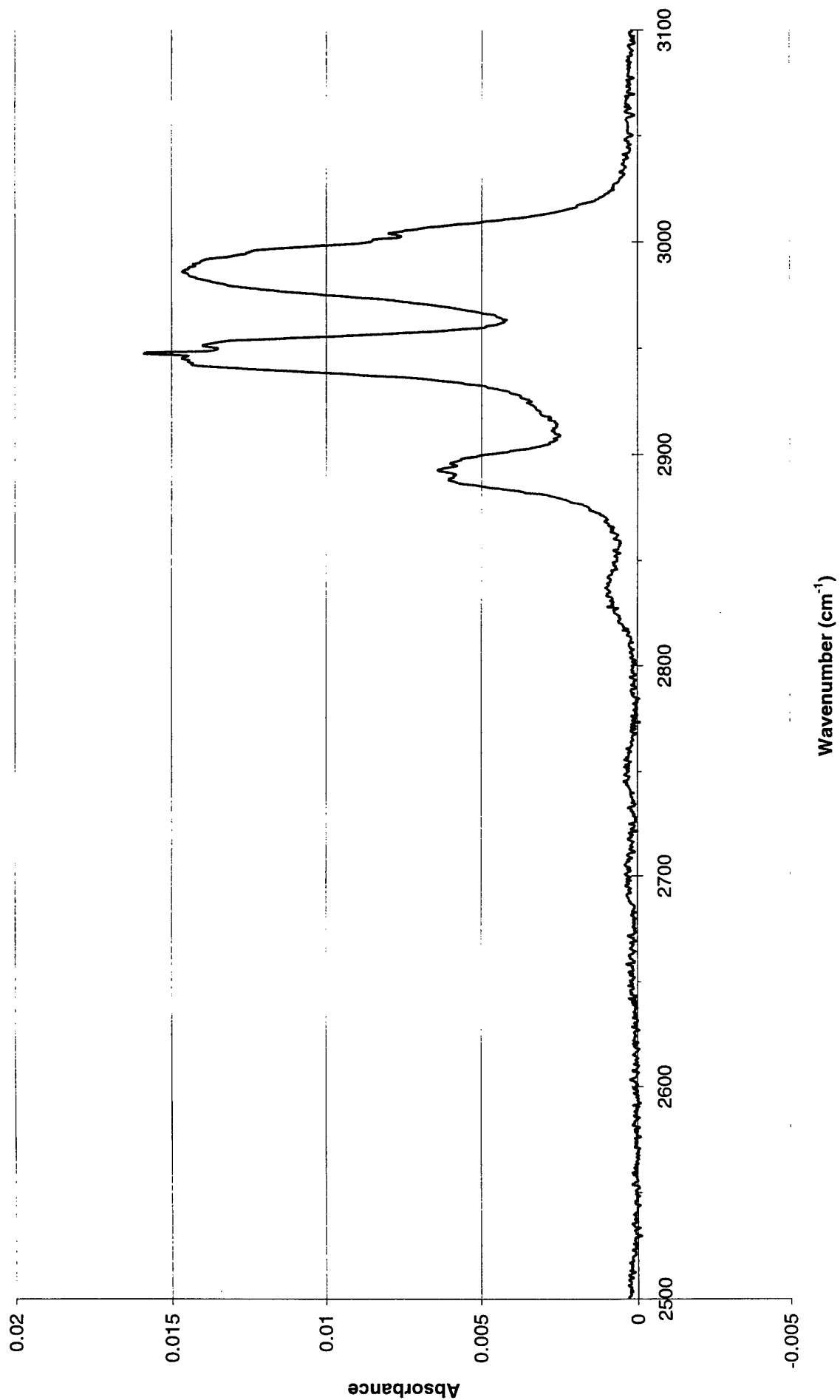


QIRL0390 - Ethyl chlorothiolformate
128 ppm-m



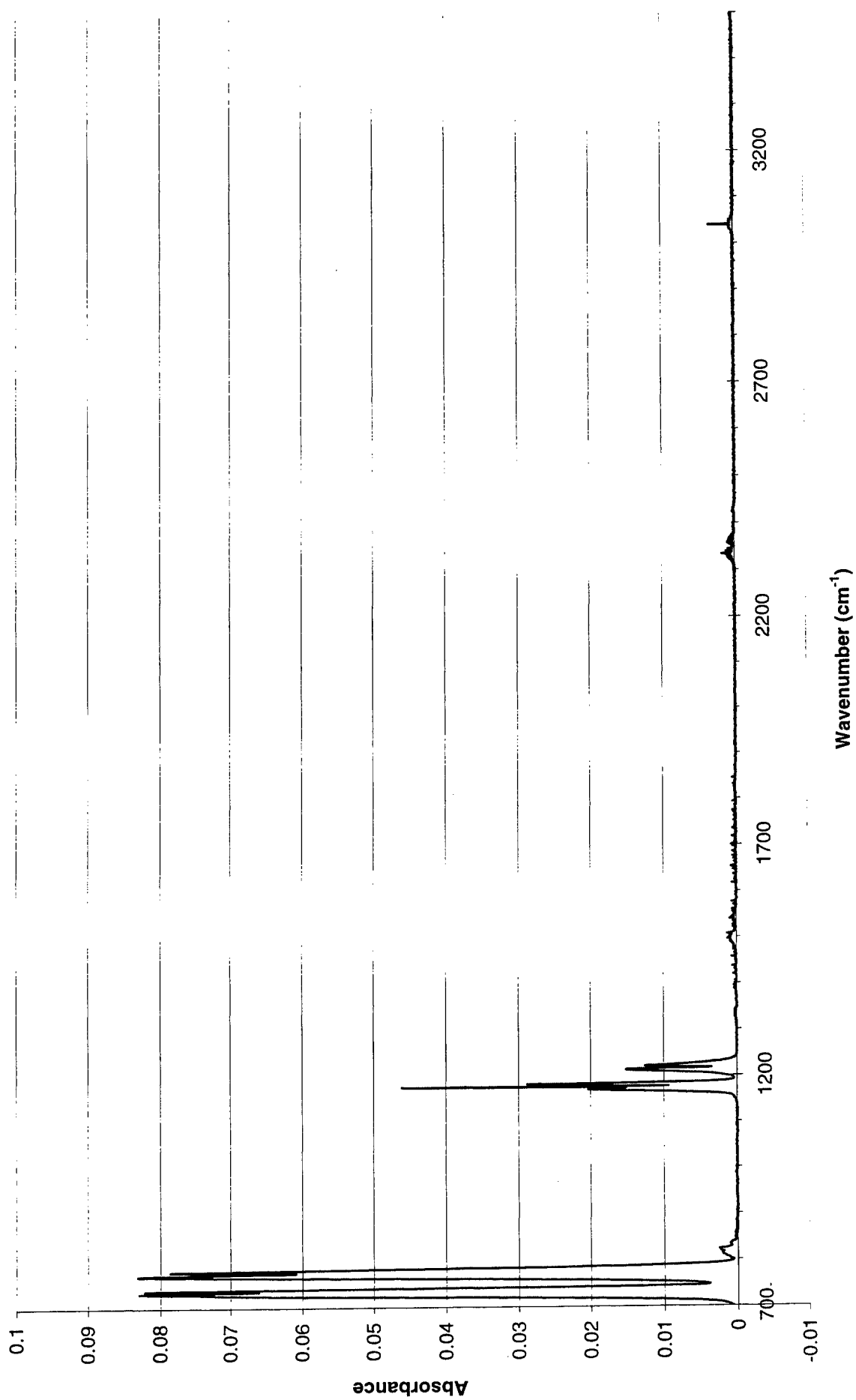


QIRL0390 - Ethyl chlorothiolformate
128 ppm-m





QIRL0400 - Bromodichloromethane
105 ppm-m





QIRL0400 - Bromodichloromethane
105 ppm-m

