

14.9

TENNESSEE BUREAU OF INVESTIGATION  
FORENSIC SERVICES DIVISION

Validation Review Form

Procedure Standard Verification / Performance Check on Nicolet 380 FTIR/ATR

Check the appropriate: New Validation ☐ Modification ☐ Performance Check ☒

This form is to be used as a coversheet for any new method, change in method, reagent or kit change. The Forensic Scientist appointed for procedure development/validation is to initial and date the appropriate box and attach documentation of required validation study results. Each applicable area listed below should be clearly labeled in the Validation Notebook (NO POST-IT NOTES).

Submit all information to the Quality Assurance Manager prior to implementation of the procedure/instrument.

| NOTE: Each applicable area listed below should start with a typed summary about that particular section.  | REVIEWED BY: | DATE COMPLETED: |
|-----------------------------------------------------------------------------------------------------------|--------------|-----------------|
| Overall Validation Summary and Section Summaries are included                                             | JBE          | 3/7/08          |
| Parallel Testing Procedure                                                                                | JBE          | 3/7/08          |
| Accuracy                                                                                                  | JBE          | 3/7/08          |
| Limit of Detection (LOD) (if applicable)                                                                  | N/A          |                 |
| Limit of Quantitation (LOQ) (if applicable)                                                               | N/A          |                 |
| Linearity & Range Established (if applicable)                                                             | N/A          |                 |
| Specificity Range Established (if applicable)                                                             | N/A          |                 |
| Precision                                                                                                 | N/A          |                 |
| Robustness - to include: Matrix                                                                           | N/A          |                 |
| Effects                                                                                                   | N/A          |                 |
| Cross Reactivity                                                                                          |              |                 |
| PH, Ionic Strength, Temperature                                                                           |              |                 |
| Laboratory Procedure Approved (if applicable)                                                             | N/A          |                 |
| LIMS Messages(s) and sample of Final Report Established (if applicable)                                   | N/A          |                 |
| Training and Competency Testing for each Examiner/Technician Completed or Proposed Training Plan Enclosed | N/A          |                 |
| References are listed                                                                                     | N/A          |                 |
| Technical Review of Completed Notebook                                                                    | JBE          | 3/7/08          |

\*If all technical staff training has not been completed, please include a proposed training plan and completion schedule.

Sign and date to indicate your acceptance of the procedure/instrument:

Unit Supervisor (if applicable) Glenn B. Everett  
 Technical Leader (if applicable) Glenn B. Everett  
 Regional Laboratory Supervisor Philip R. Smith 3-11-08  
 Quality Assurance Manager Margaret R. Bash

THIS IS AN UNCONTROLLED COPY OF A CONTROLLED DOCUMENT

## **Standard Verification Performance Check for Nicolet 380 FTIR with Golden Gate ATR Attachment**

### **Validation Procedure:**

Ten certified drug standards were selected and tested for identification with this instrument. The standards selected are as follows:

Alprazolam – manufactured by Upjohn, Lot # 487RA  
Caffeine – manufactured by Eastman Kodak, Lot # 355  
Carisoprodol – manufactured by Alltech, Lot # 311 DC-0852  
Cocaine base – manufactured by Merck, Lot # V7472  
Cocaine HCl – manufactured by Sigma, Lot # 50K1124  
Diazepam – manufactured by Sigma, Lot # 105F0451  
(+)-Methamphetamine – manufactured by Sigma, Lot # 34H01444  
3,4-MDMA – manufactured by Sigma, Lot # 60K4096  
Pethidine – manufactured by SB Penick, Lot # 413NMN-027  
Testosterone – manufactured by Sigma, Lot # 50H0345

Several of these standards were chosen because they are frequently encountered in case work and/or analyzed by ATR. All standards were run on the instrument neat – no extractions or washes were performed. When available, the same chemical lots utilized to create the current spectral library were used. The instrument's sampling area was cleaned and a background spectrum was collected between each standard run to rule out any possible cross-contamination

### **Accuracy:**

Spectra collected from these samples were compared to the current spectral library on the instrument and a high match percentage (>95%) was obtained for each standard. The spectra were also compared to the standards by a trained forensic scientist, and each match was deemed as satisfactory for identification. The collected spectra (in red) as well as the standard spectra used for comparison are included.

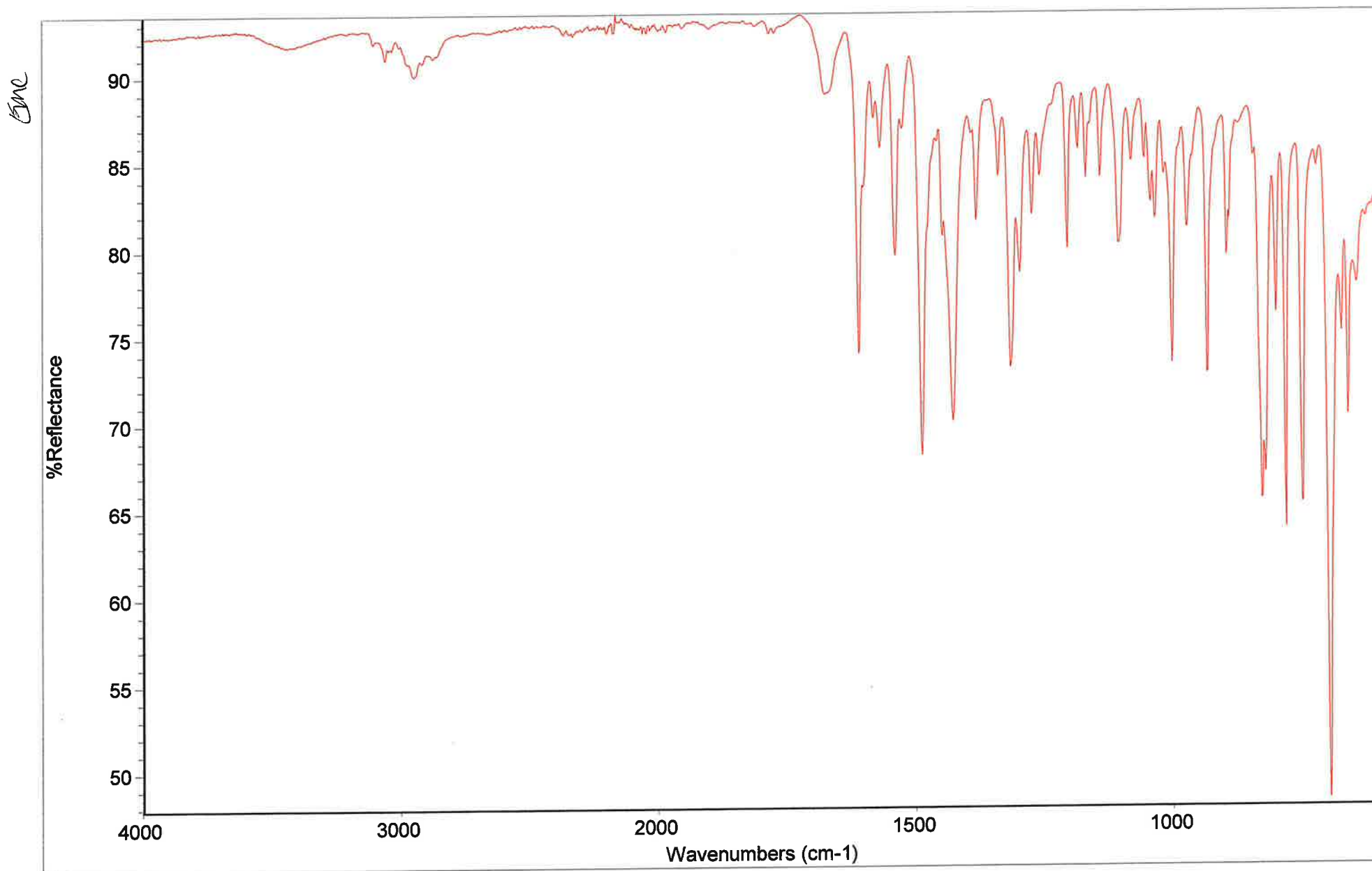
Since the Crime Lab does not use ATR for quantitations, Limits of Detection (LOD), Limits of Quantitation (LOQ), Linearity and Range, and Precision were not determined. Specificity and Robustness do not apply to this method.

## **Proposal for Performance Check on FTIR with Golden Gate ATR**

All three TBI Crime Labs are currently using the Golden Gate ATR. Before being accepted for use by all three labs, it was treated by each lab as a new invalidated and unaccepted sampling technique. Therefore each lab proceeded to obtain ATR spectra of every unknown in their possession and to establish their own library of standards. ATR was found to be a valid sampling technique in all three labs. In addition ATR is a well-accepted scientific technique throughout the scientific community.

The Nashville Laboratory has recently purchased a new Nicolet 380 FTIR with an ATR sampling accessory to be used by the Forensic Chemistry Section. The new FTIR/ATR is exactly the same configuration as the original FTIR/ATR that has already been validated. In addition, the software on the instrument it will be placed on is the same as the software on the instrument that the original was placed on. Since this is an established sampling technique, there is no need to completely reprove the validity of ATR for drug identification. In addition FTIR is a scientifically proven technique and has been shown to give consistent and reliable data. Data that can be used to unmistakably identify a compound. Therefore, essentially all that needs to be done is to verify that the new FTIR/ATR gives the same data as the original one gives.

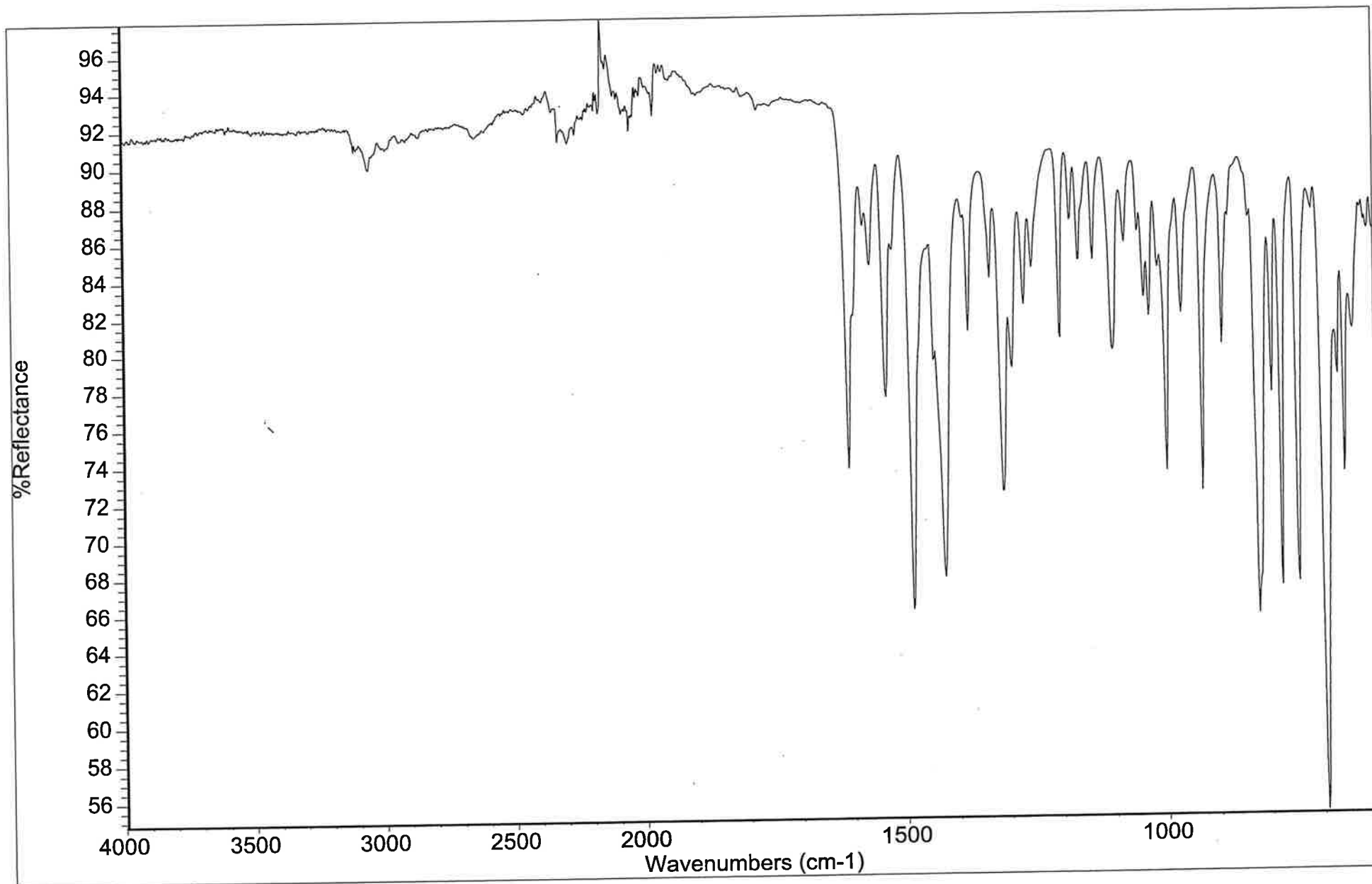
To accomplish this we propose to run a series of 10 standards on the new ATR. These will include Cocaine HCl, Cocaine Base, and Methamphetamine, as these are the compounds that we analyze by FTIR most often. All the samples that are to be used have been previously analyzed on the original ATR (in fact the exact same lot numbers will be used) and the new data will be compared to these spectra. After obtaining this data and comparing it to our FTIR/ATR standards, the new FTIR/ATR will be submitted for approval.



IR VERIFICATION - ALPRAZOLAM NEAT UPJOHN LOT# 487RA EMC

IR #1

Collection time: Thu Mar 06 11:50:06 2008 (GMT-06:00)

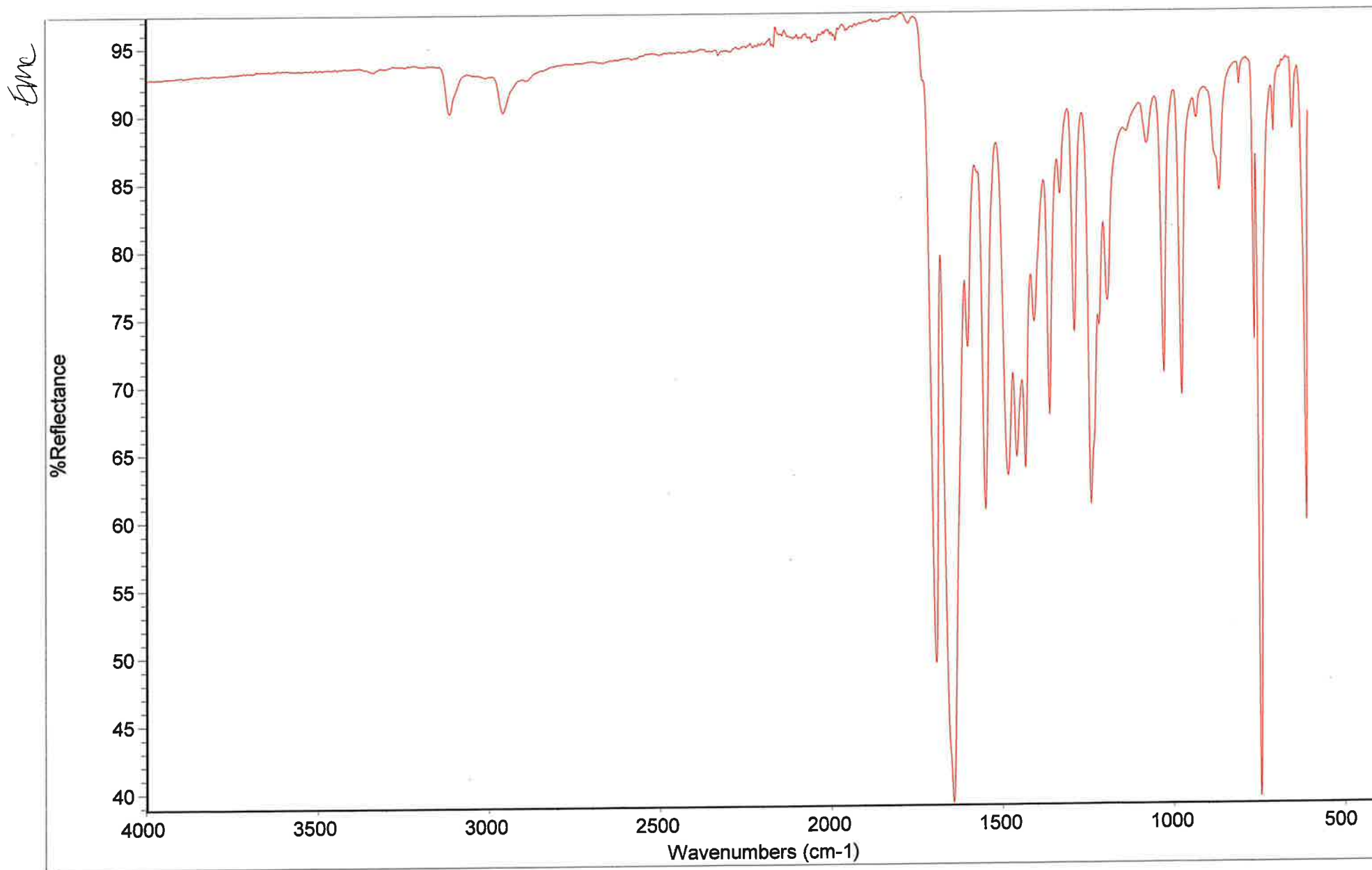


ALPRAZOLAM U31889 LOT #487RA

IR #4

*WKS*

Collection time: Mon Sep 15 14:10:07 2003

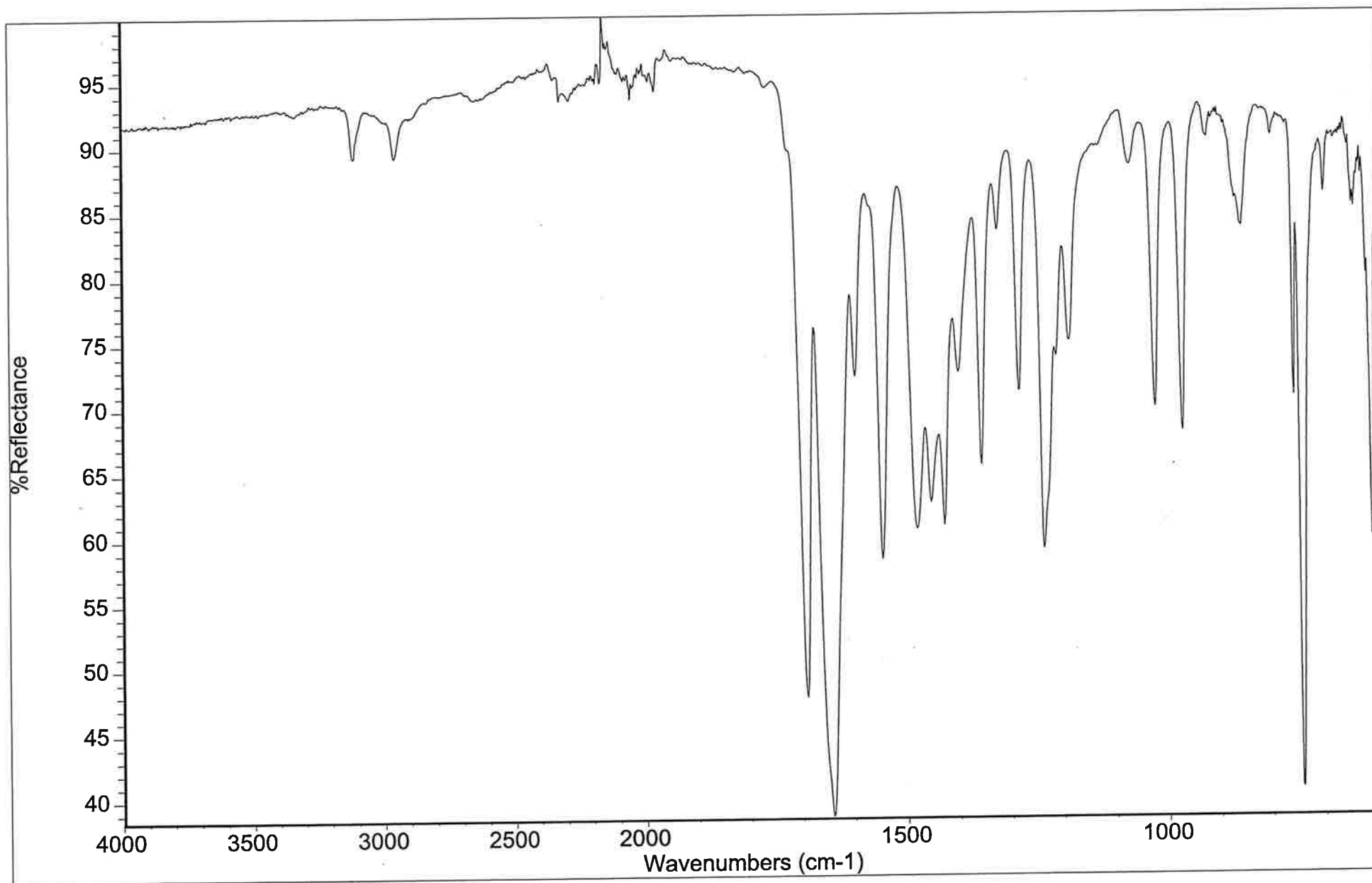


IR VERIFICATION - CAFFEINE STD NEAT EMC

IR #1

Lot# Eastman Kodak -355

Collection time: Thu Mar 06 11:16:42 2008 (GMT-06:00)

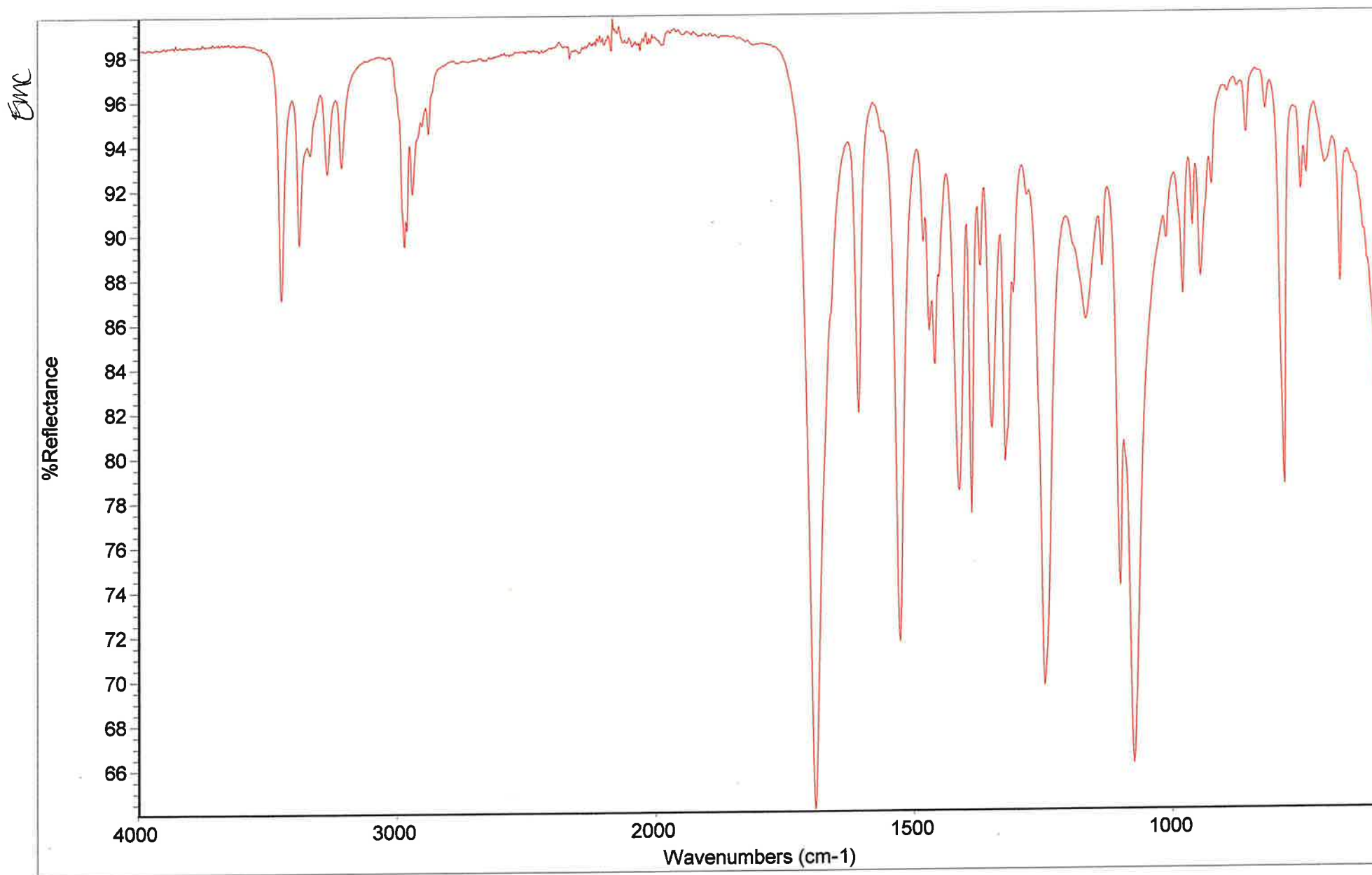


Caffeine Eastman Kodak Lot #355

IR #4

*WKS*

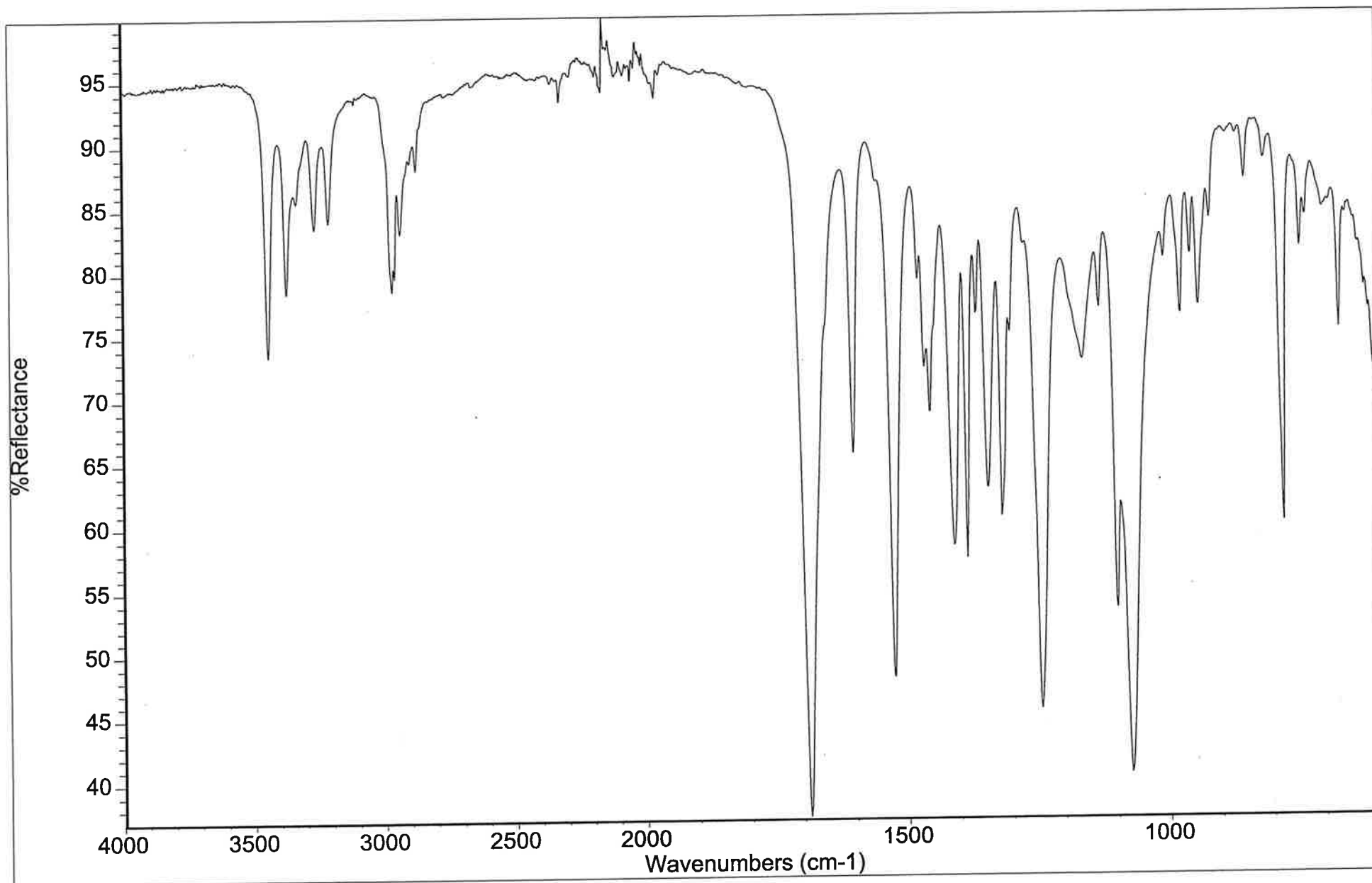
Collection time: Tue Sep 16 13:18:23 2003



IR VERIFICATION - CARISOPRODOL STD ALLTECH LOT# 311 DC 08-52

IR #1

Collection time: Thu Mar 06 11:42:37 2008 (GMT-06:00)



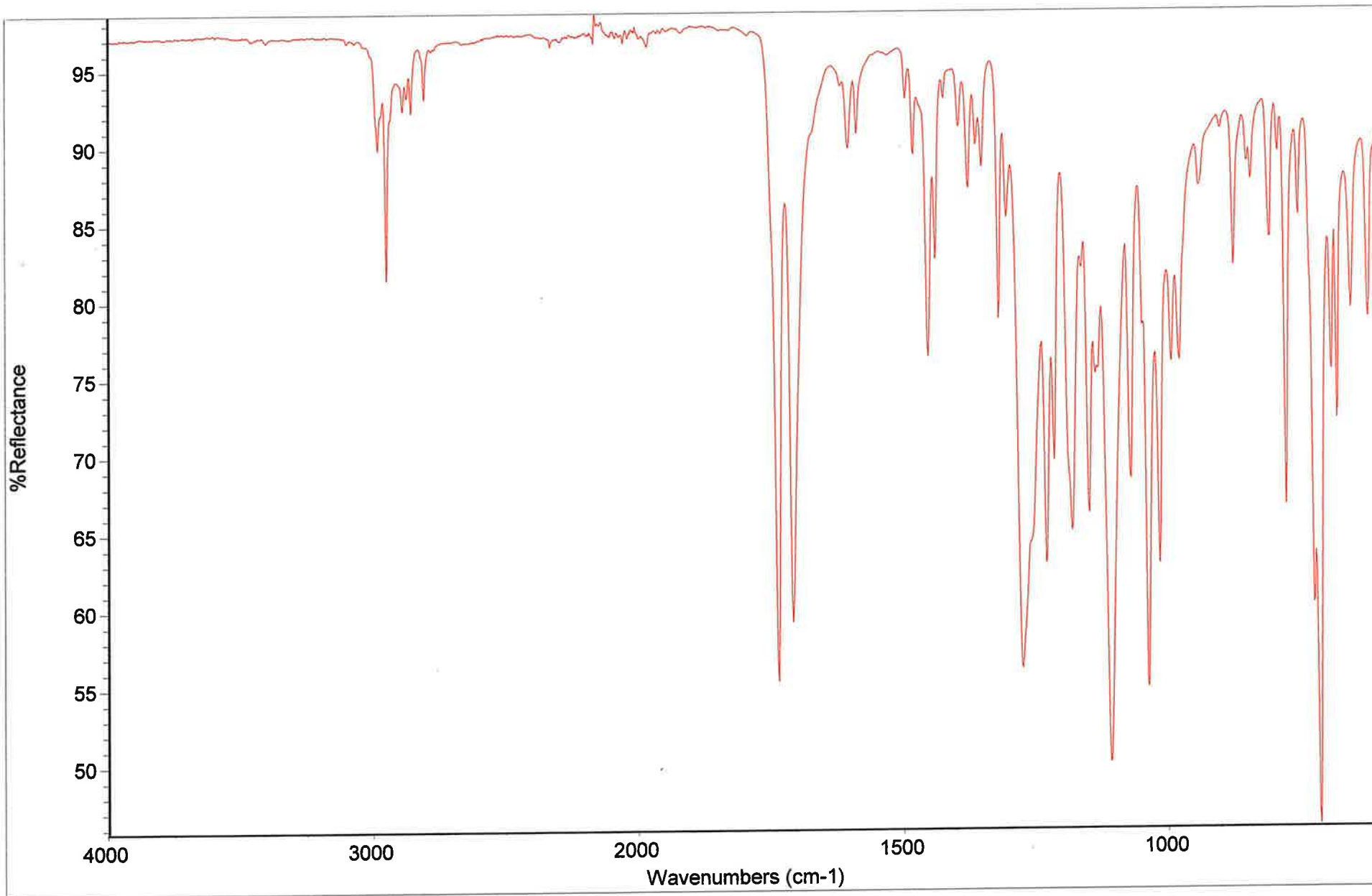
Carisoprodol Alltech Lot #311

IR #4

*(Handwritten signature)*

Collection time: Tue Sep 16 11:24:37 2003

Emc

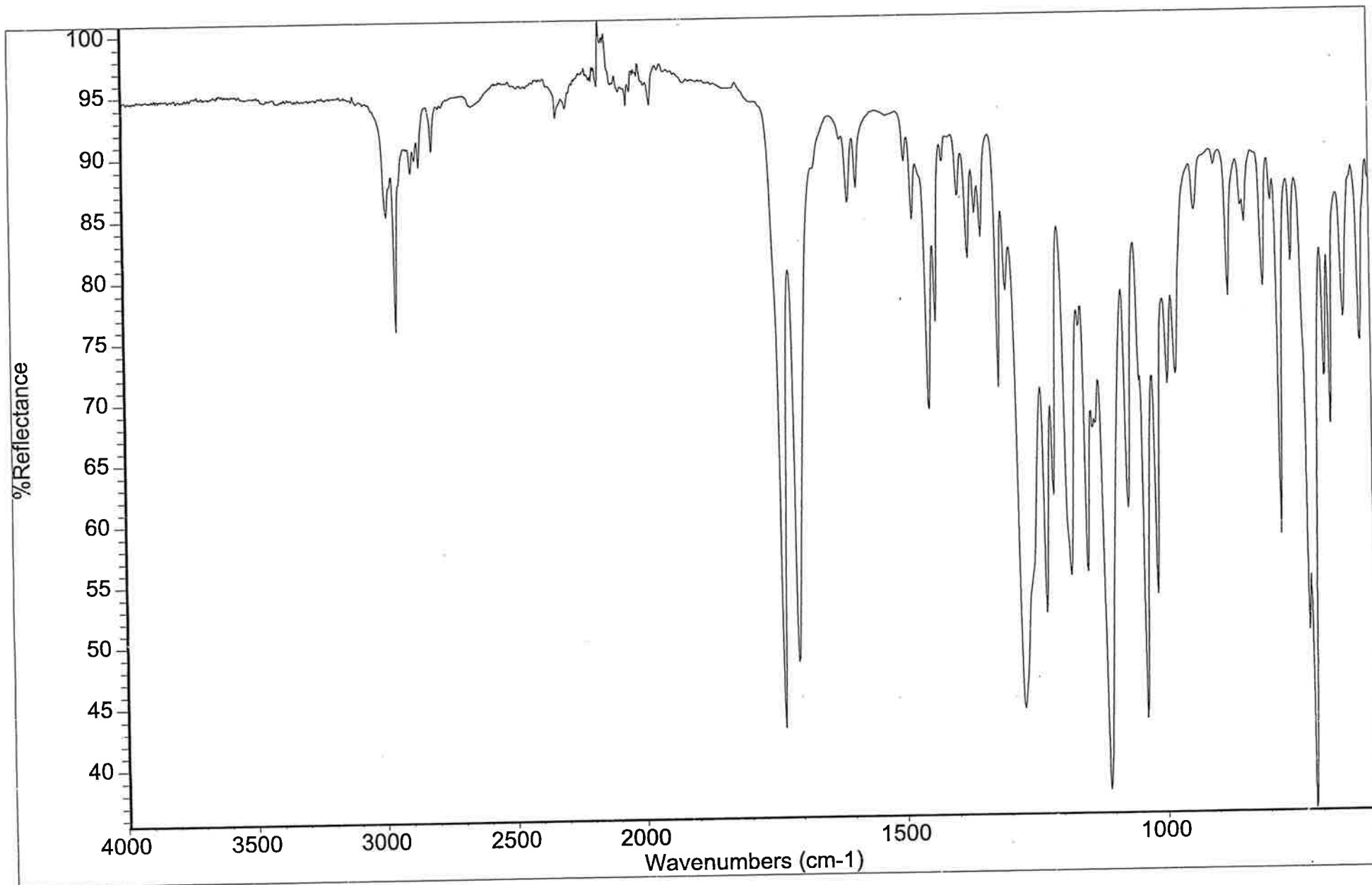


IR VERIFICATION - COCAINE BASE NEAT MERCK LOT#V7472 EMC

u(Emc)

IR #1

Collection time: Thu Mar 06 11:56:16 2008 (GMT-06:00)

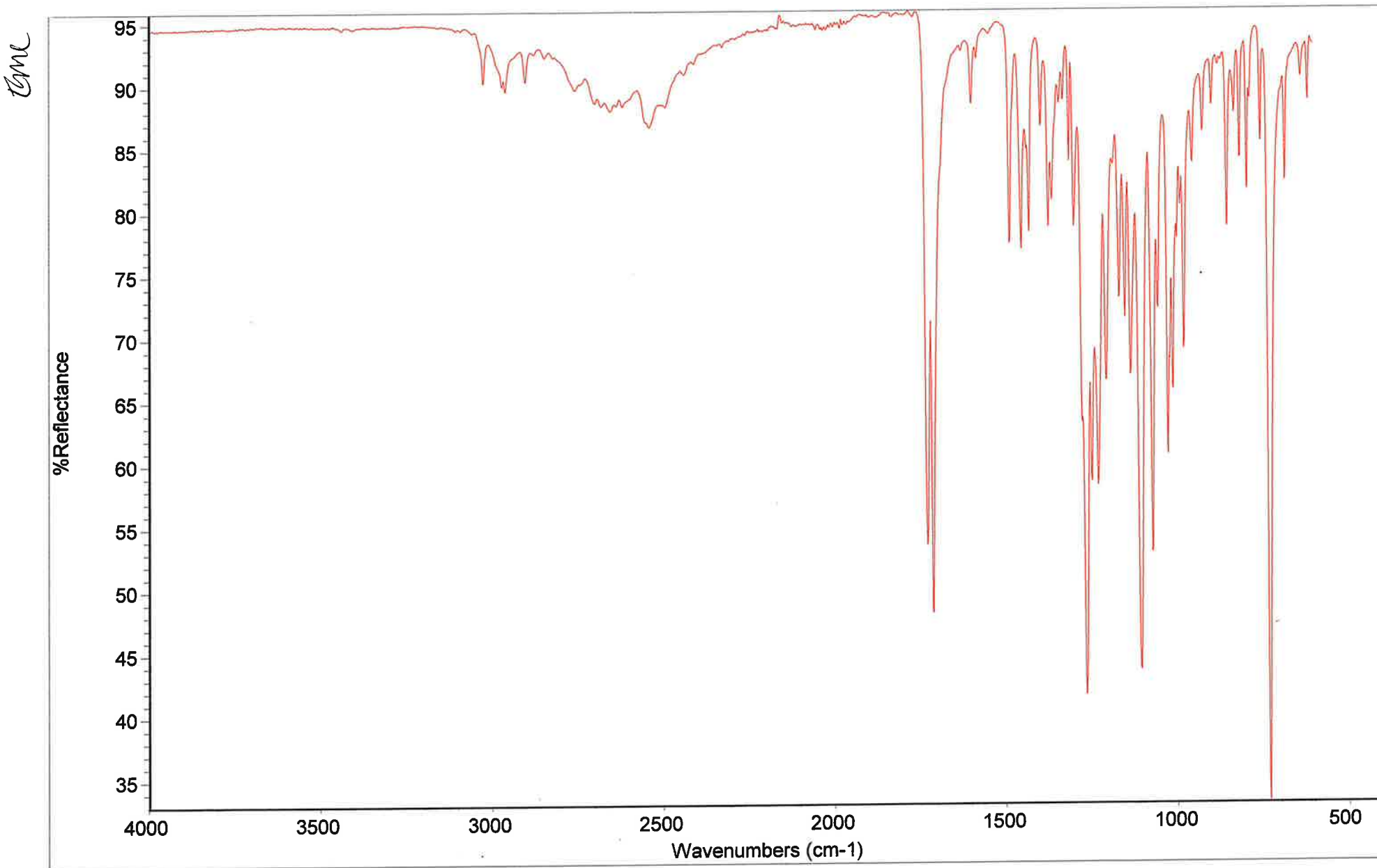


Cocaine Base Merck Lot #U7472

IR #4

*Handwritten signature/initials*

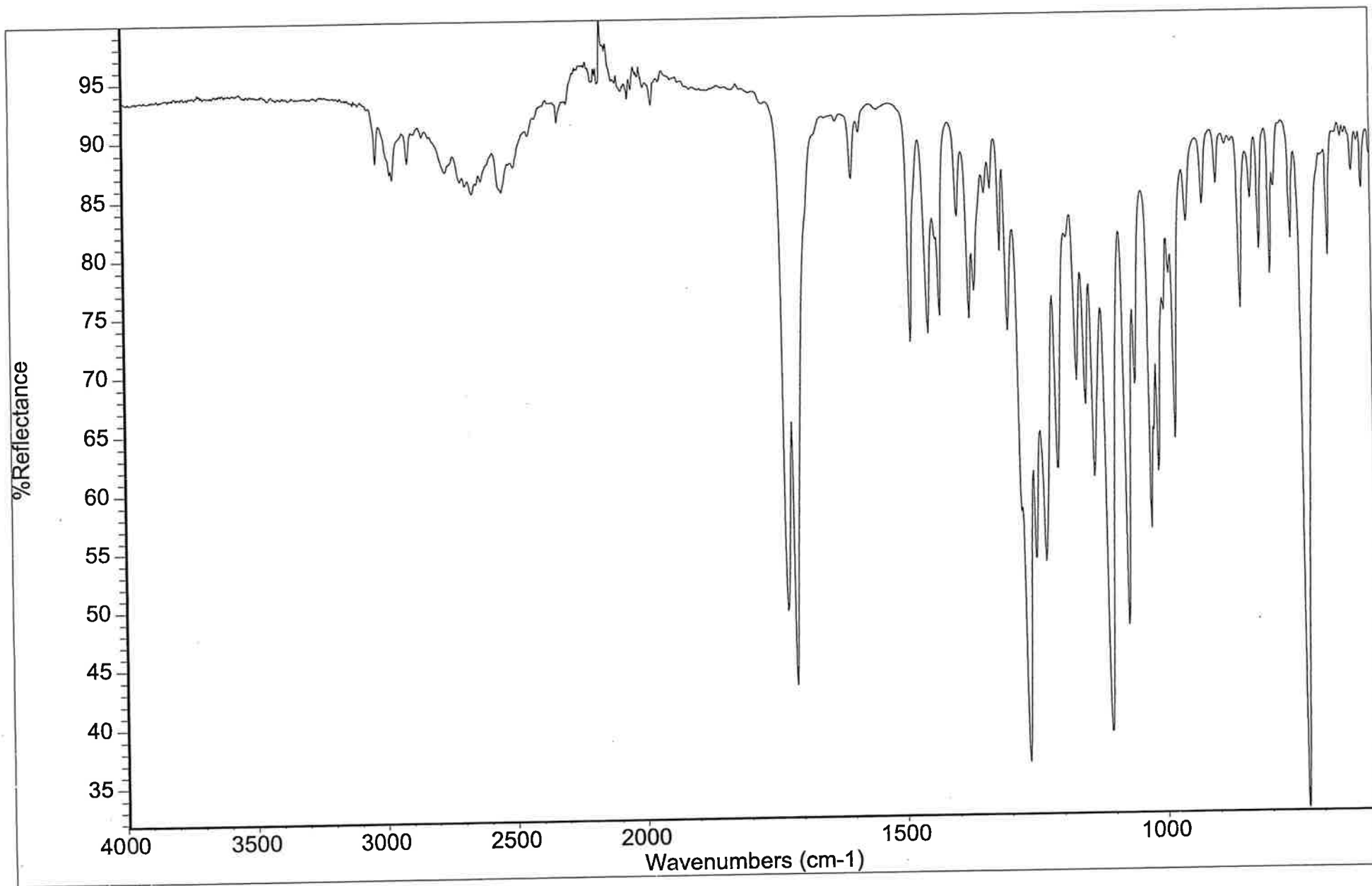
Collection time: Tue Sep 16 15:18:05 2003



IR VERIFICATION - COCAINE HCL STD SIGMA LOT 50K1124 EMC

IR #1

Collection time: Thu Mar 06 11:19:45 2008 (GMT-06:00)

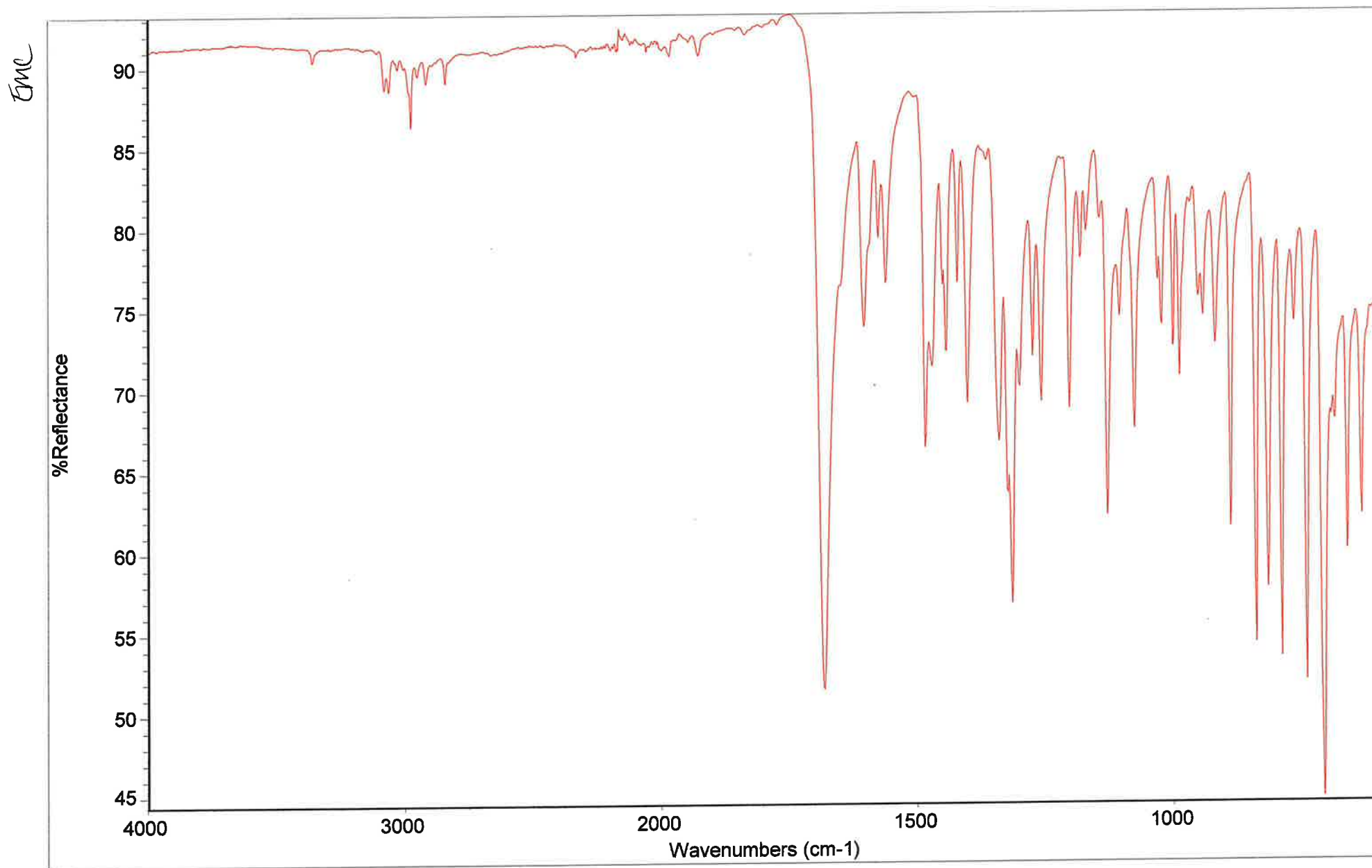


Cocaine HCl Sigma Lot #50K1124

IR #4

*(Handwritten signature)*

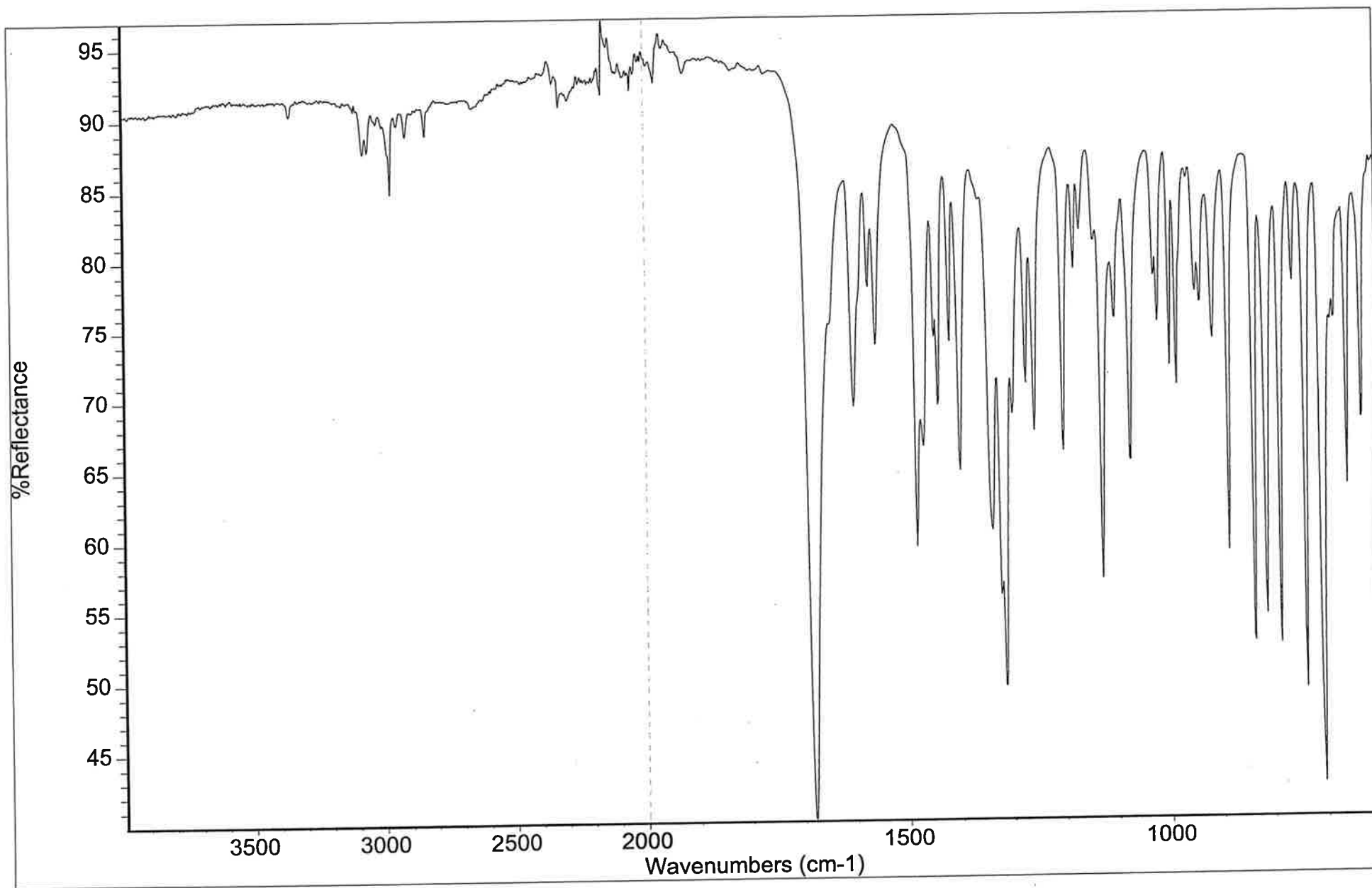
Collection time: Tue Sep 16 15:14:25 2003



IR VERIFICATION - DIAZEPAM NEAT SIGMA LOT# 105F0451

IR #1

Collection time: Thu Mar 06 11:45:47 2008 (GMT-06:00)

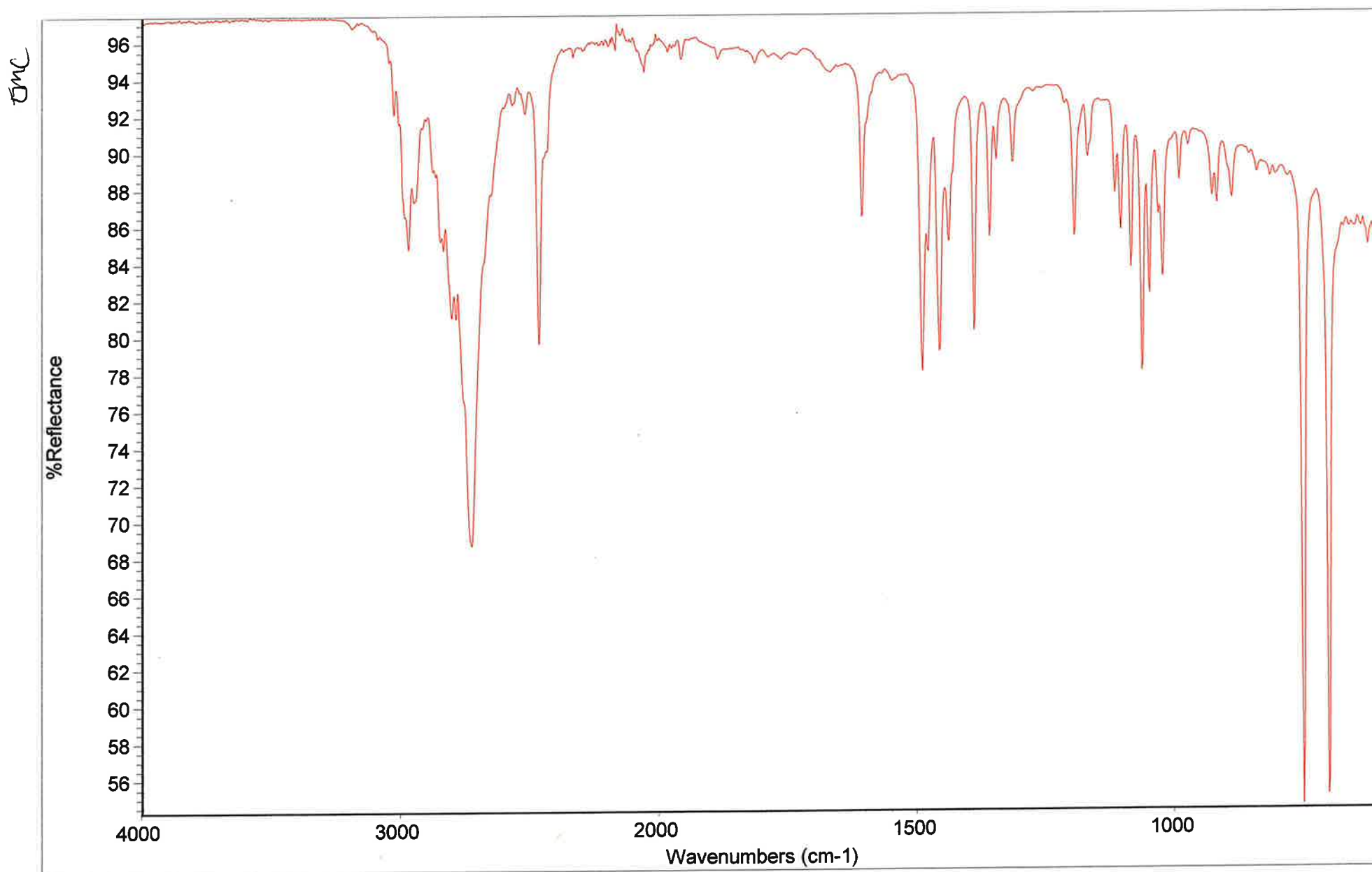


Diazepam Sigma Lot #105F0451

IR #4

*WHS*

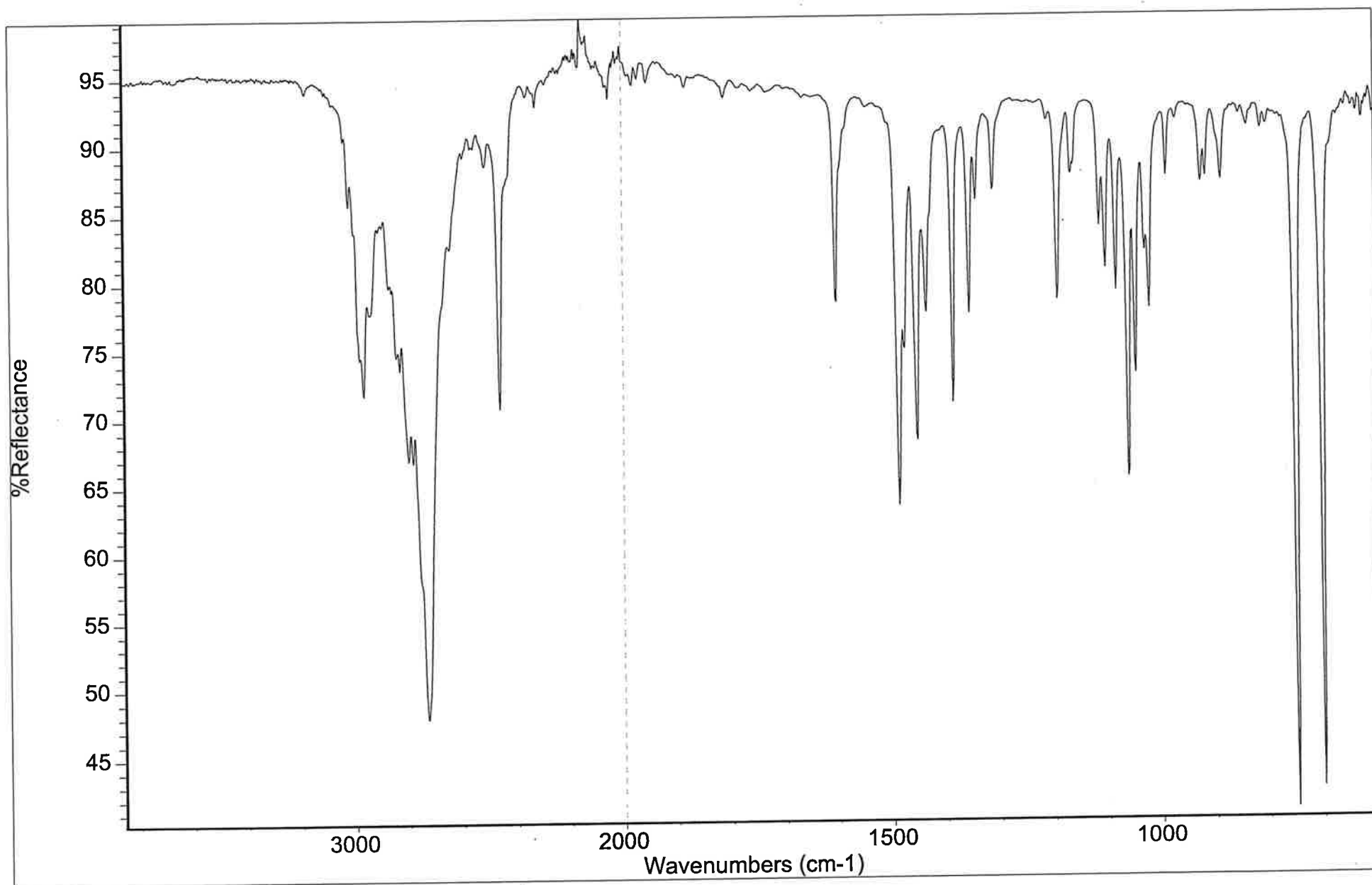
Collection time: Fri Sep 19 08:19:57 2003



IR VERIFICATION - (+)METHAMPHETAMINE NEAT SIGMA 34H0144 EMC

IR #1

Collection time: Thu Mar 06 11:53:35 2008 (GMT-06:00)



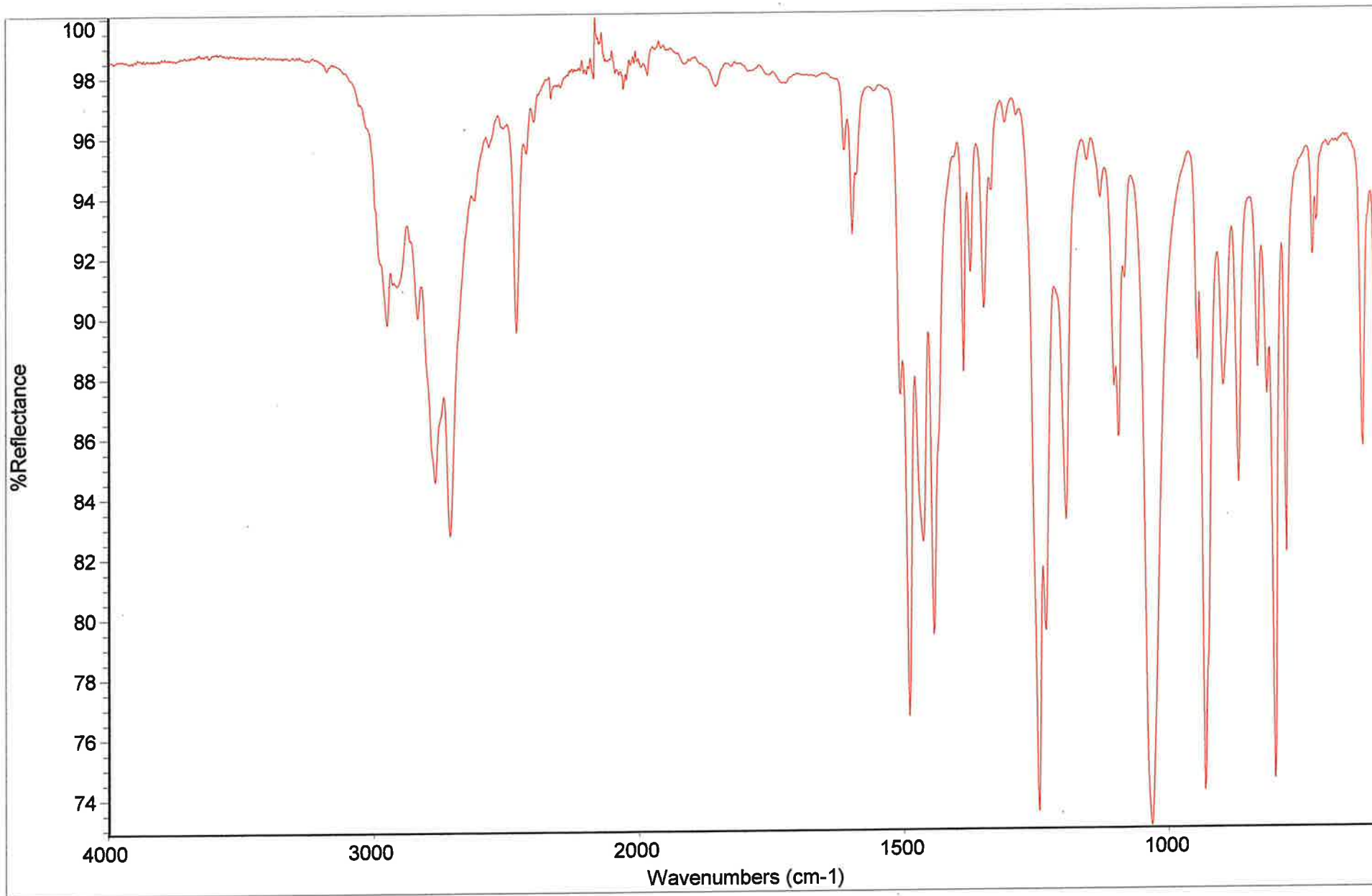
(+)-Methamphetamine HCl Sigma Lot #M-8750

IR #4

*(Handwritten signature)*

Collection time: Mon Sep 22 14:23:20 2003

EMC

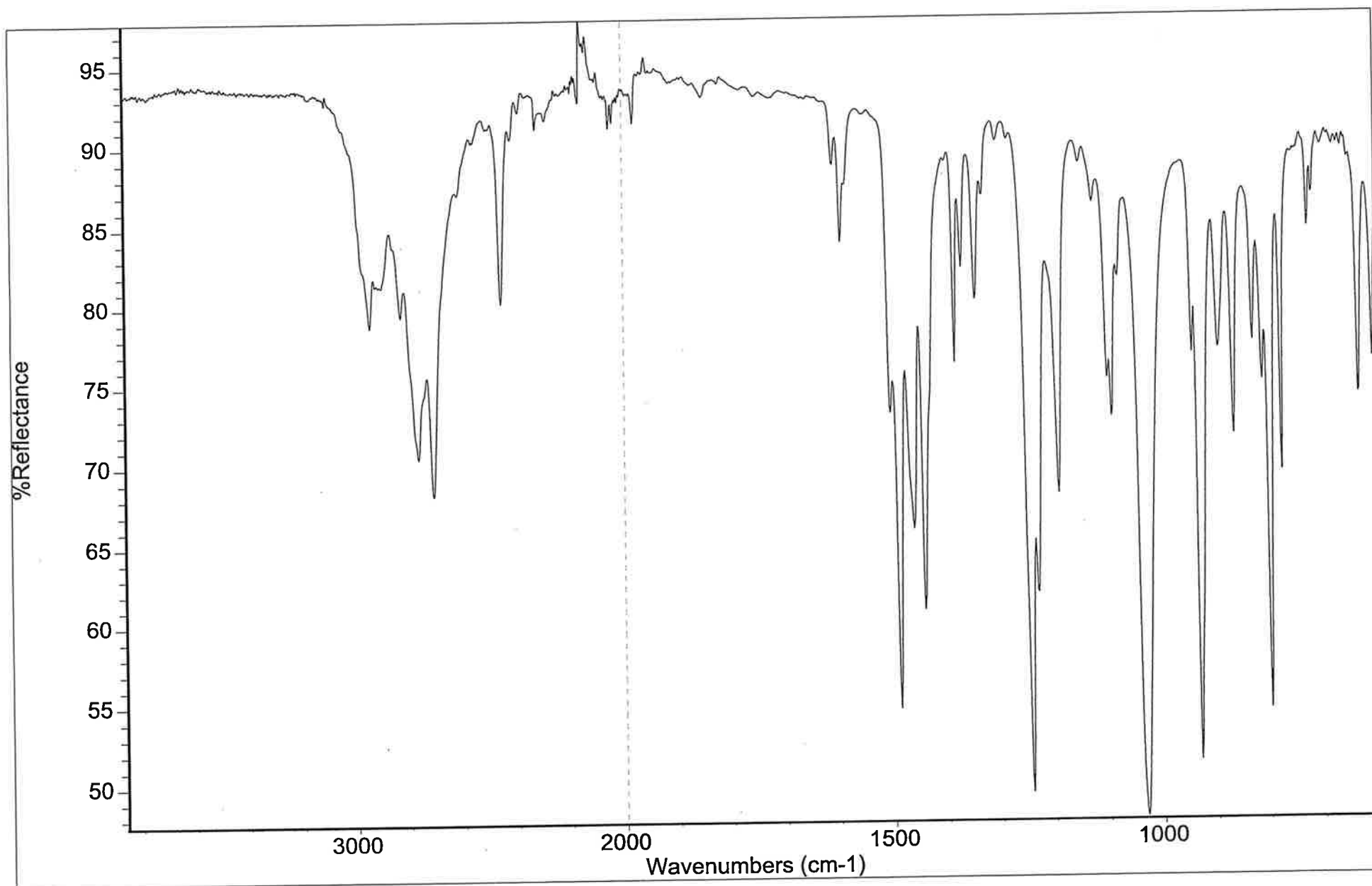


IR VERIFICATION - 3,4-MDMA NEAT SIGMA LOT #60K4098 EMC

6 (EMC)

IR #1

Collection time: Thu Mar 06 11:34:58 2008 (GMT-06:00)



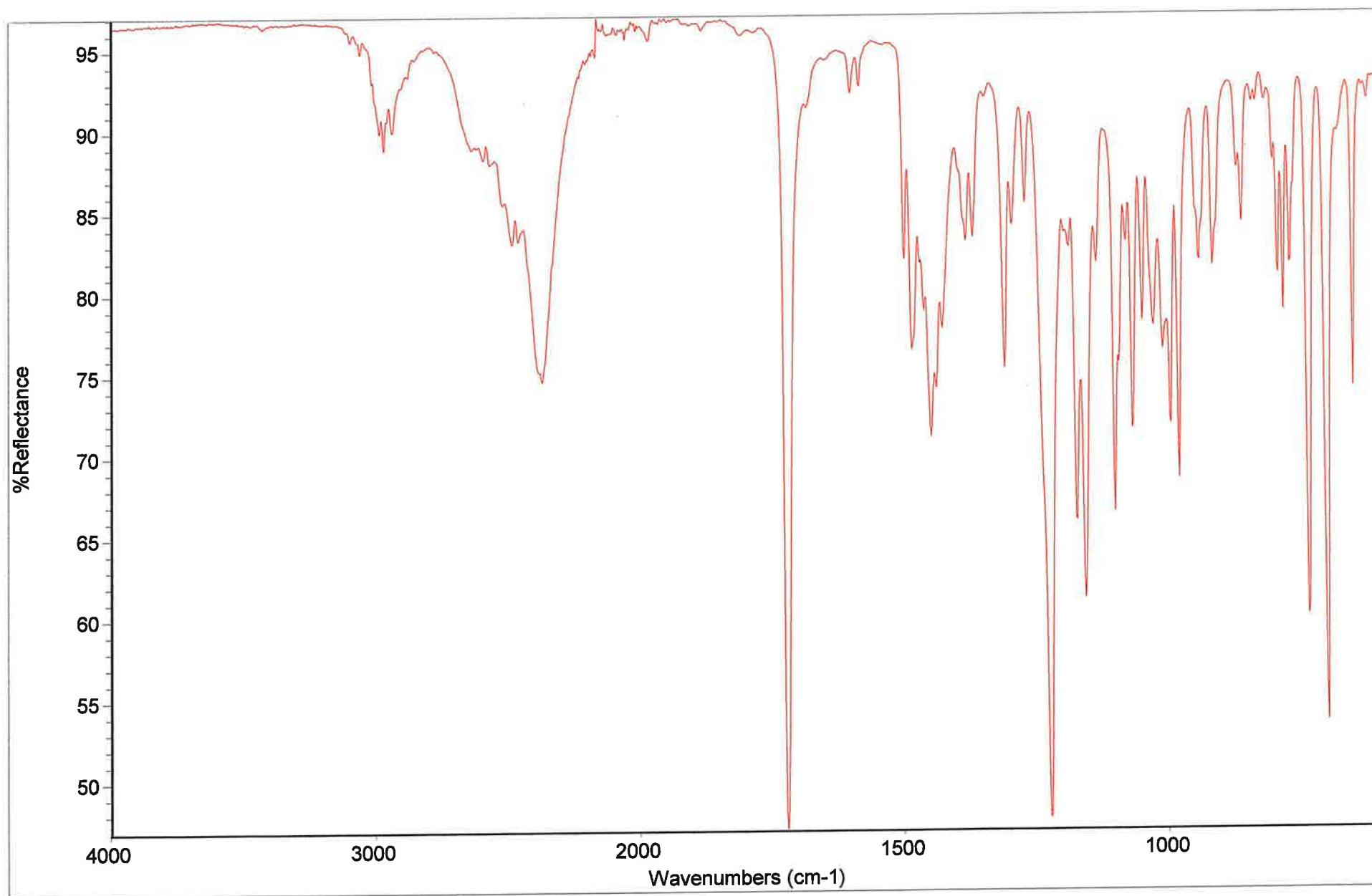
(+,-)-3,4-Methylenedioxymethamphetamine (MDMA) HCl Sigma Lot #60K4096

IR #4

*CSK*

Collection time: Tue Sep 23 11:18:07 2003

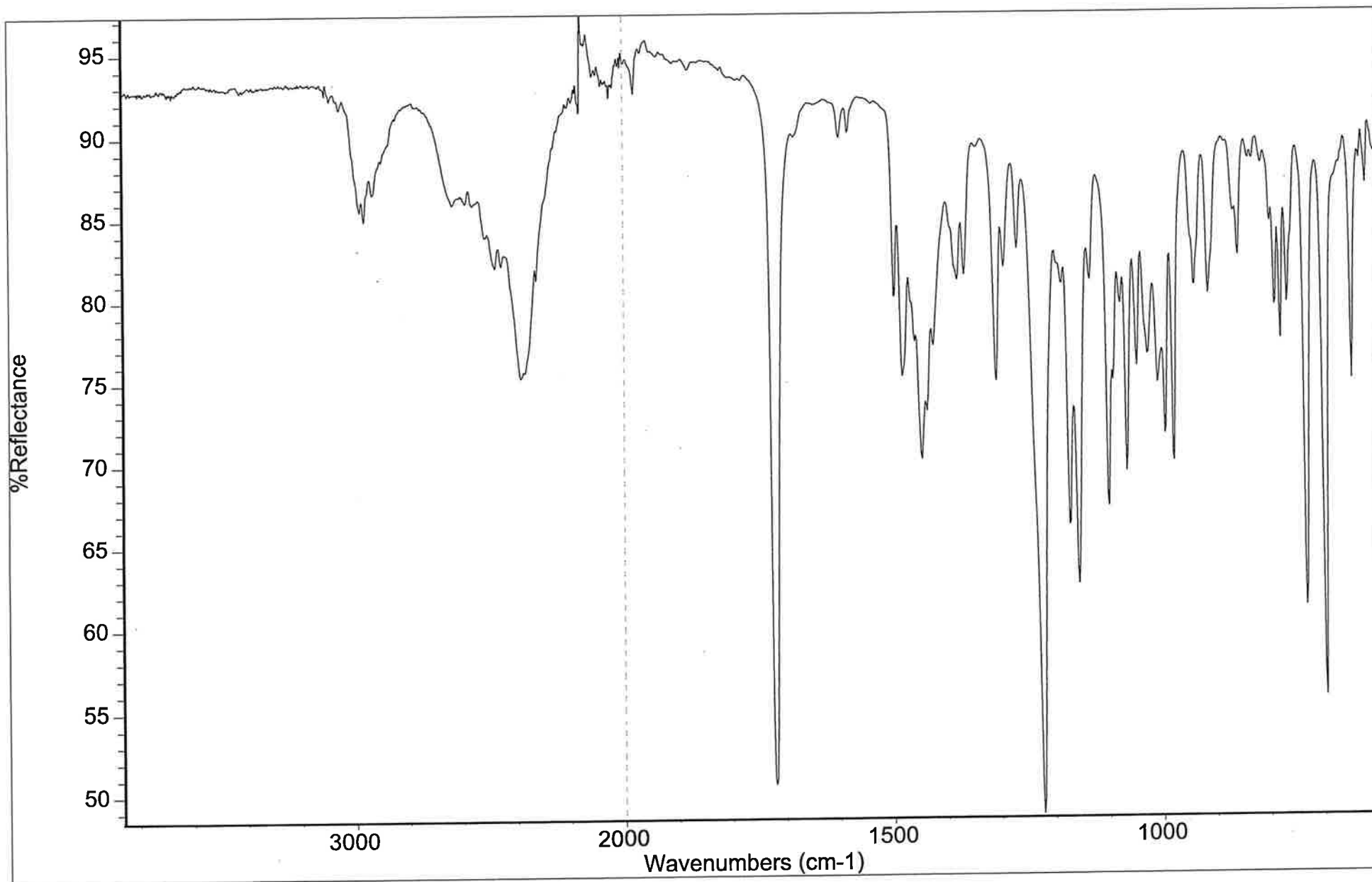
EMC



IR VERIFICATION - PETHIDINE NEAT SB PENICK LOT#413NMN-027 EMC

IR #1

Collection time: Thu Mar 06 11:38:43 2008 (GMT-06:00)



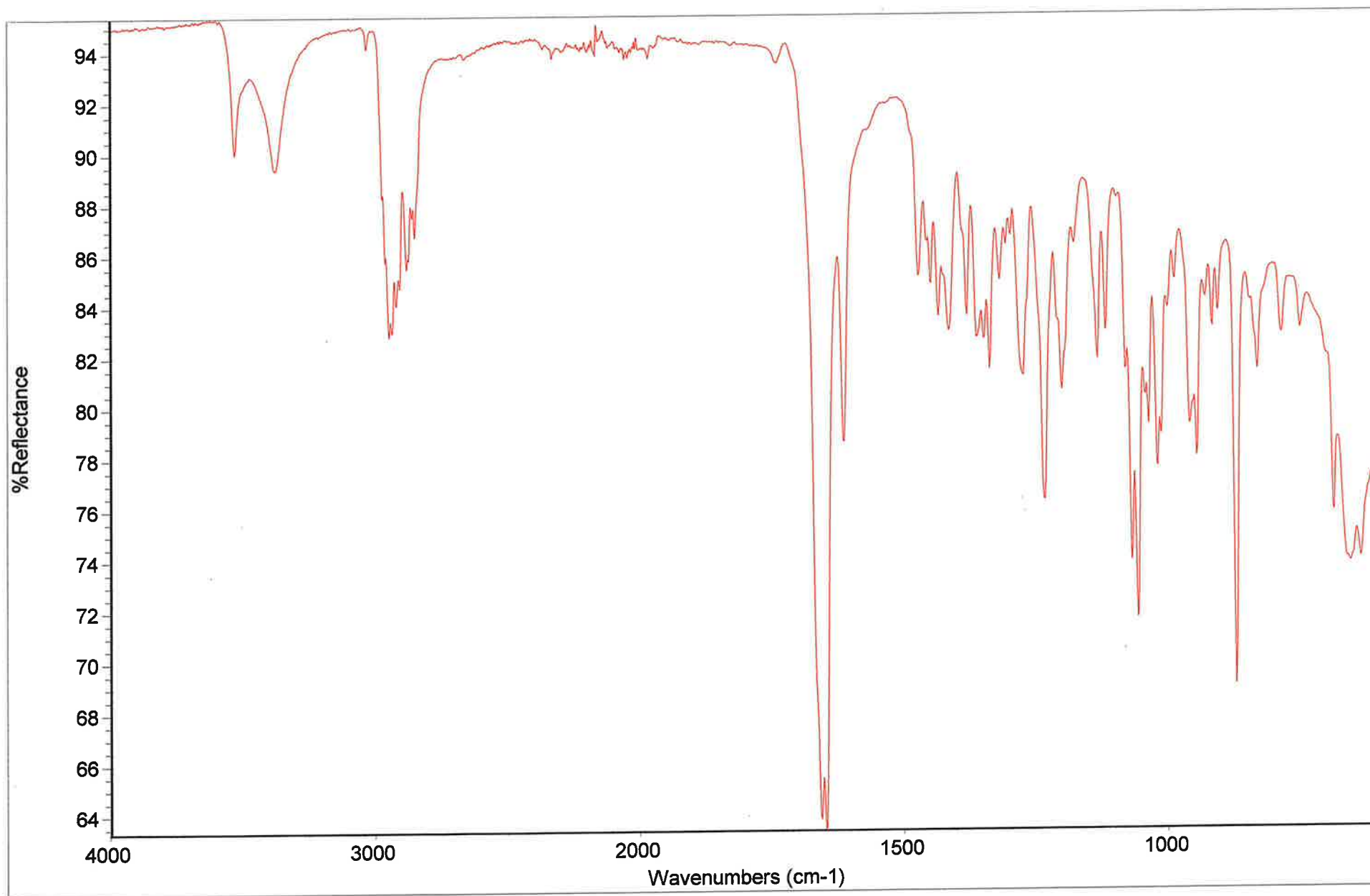
Pethidine HCl S. B. Penick Lot #0501

IR #4

W.B.

Collection time: Thu Sep 25 15:39:22 2003

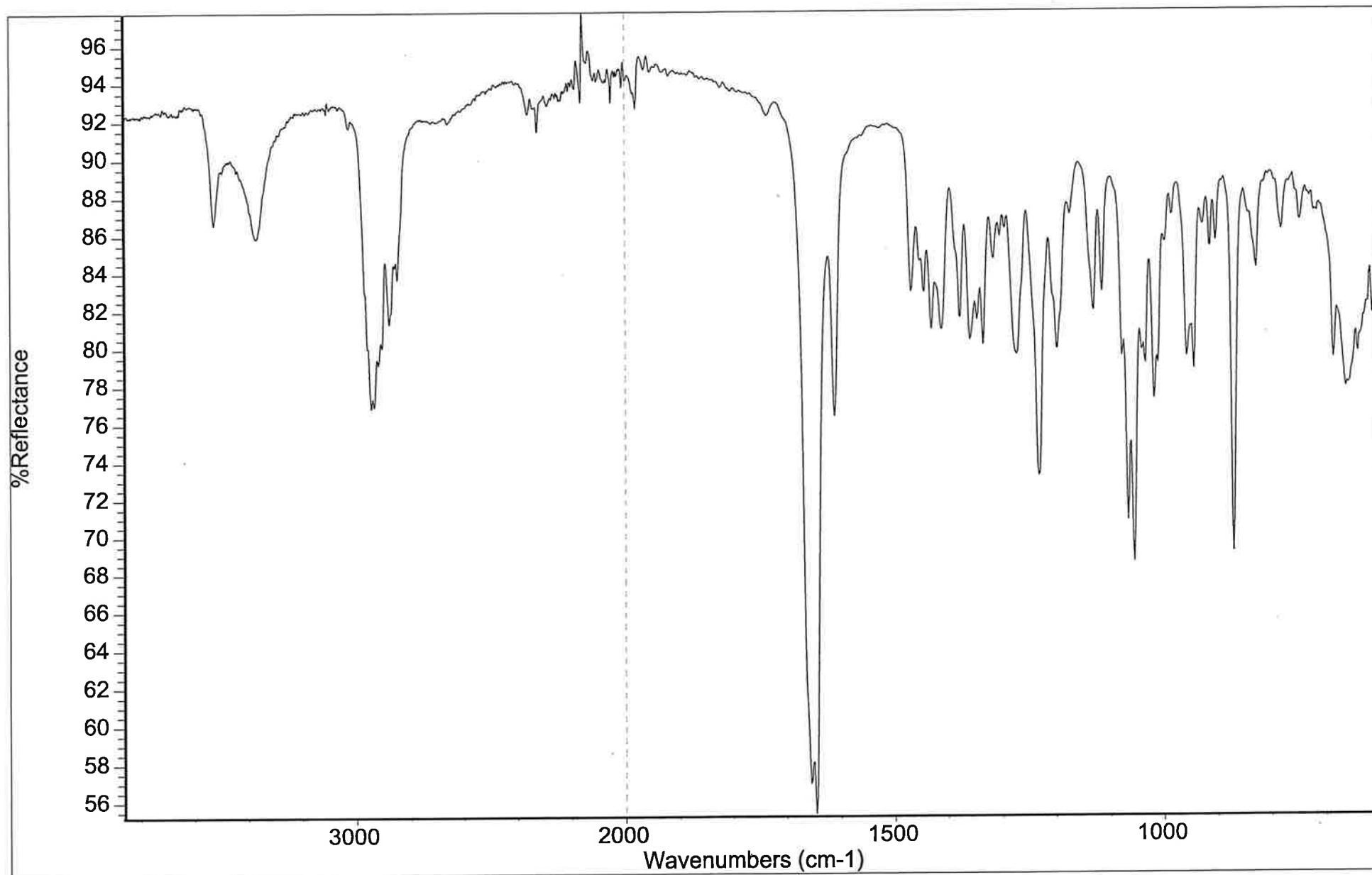
me



IR VERIFICATION - TESTOSTERONE NEAT SIGMA LOT#50H0345 EMC

IR #1

Collection time: Thu Mar 06 11:47:52 2008 (GMT-06:00)



Testosterone Sigma Lot #50H0345

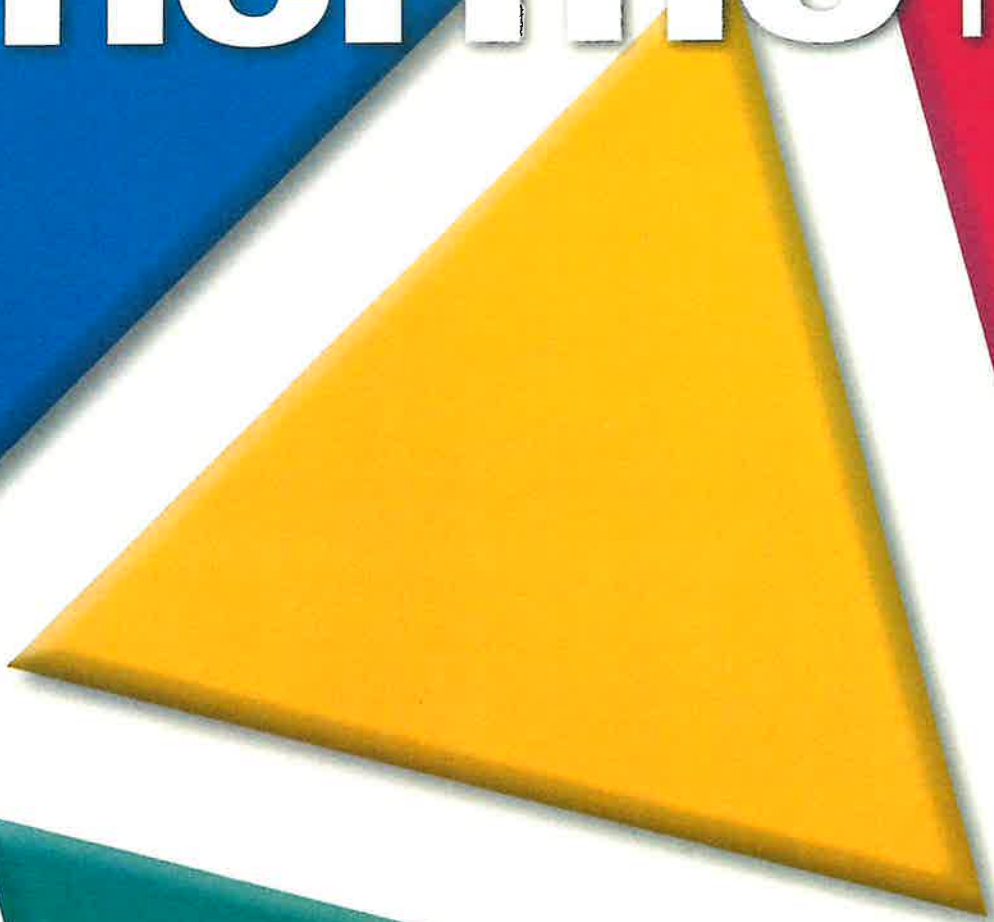
IR #4

*WLB*

Collection time: Fri Sep 26 14:27:09 2003

# Thermo Nicolet

System Test Spectra



# Shipment Authorization

Customer Name: Tennessee Bureau of Investigation

Job Number: 137320 System Serial Number: AEPO301050

☒ This instrument meets or exceeds all applicable performance specifications.

I validate the performance of this instrument:

Bill Barden

Test Technician

3/21/03

Date

☒ All components of your order are present unless otherwise noted on the Notification of Partial Shipment form included in this envelope.

I authorize shipment of this order:

Bill Hill

System Configuration Specialist

3/24/03

Date

*If you have any problems concerning this order please contact your nearest Sales/Service Office or Representative.*

%Transmittance

Nicolet Instrument Corp.  
3.0 mil Polystyrene  
Mon Mar 03 15:02:25 2003  
Number of sample scans: 36  
Number of background scans: 36  
Resolution: 4.000  
Sample gain: 1.0  
Mirror velocity: 0.6329  
Aperture: 100.00  
Detector: DTGS KBr  
Beamsplitter: KBr  
Source: IR  
Aperture: 100.000000  
AEP0301050

85  
80  
75  
70  
65  
60  
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45  
40  
35  
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10  
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-0

5000

4000

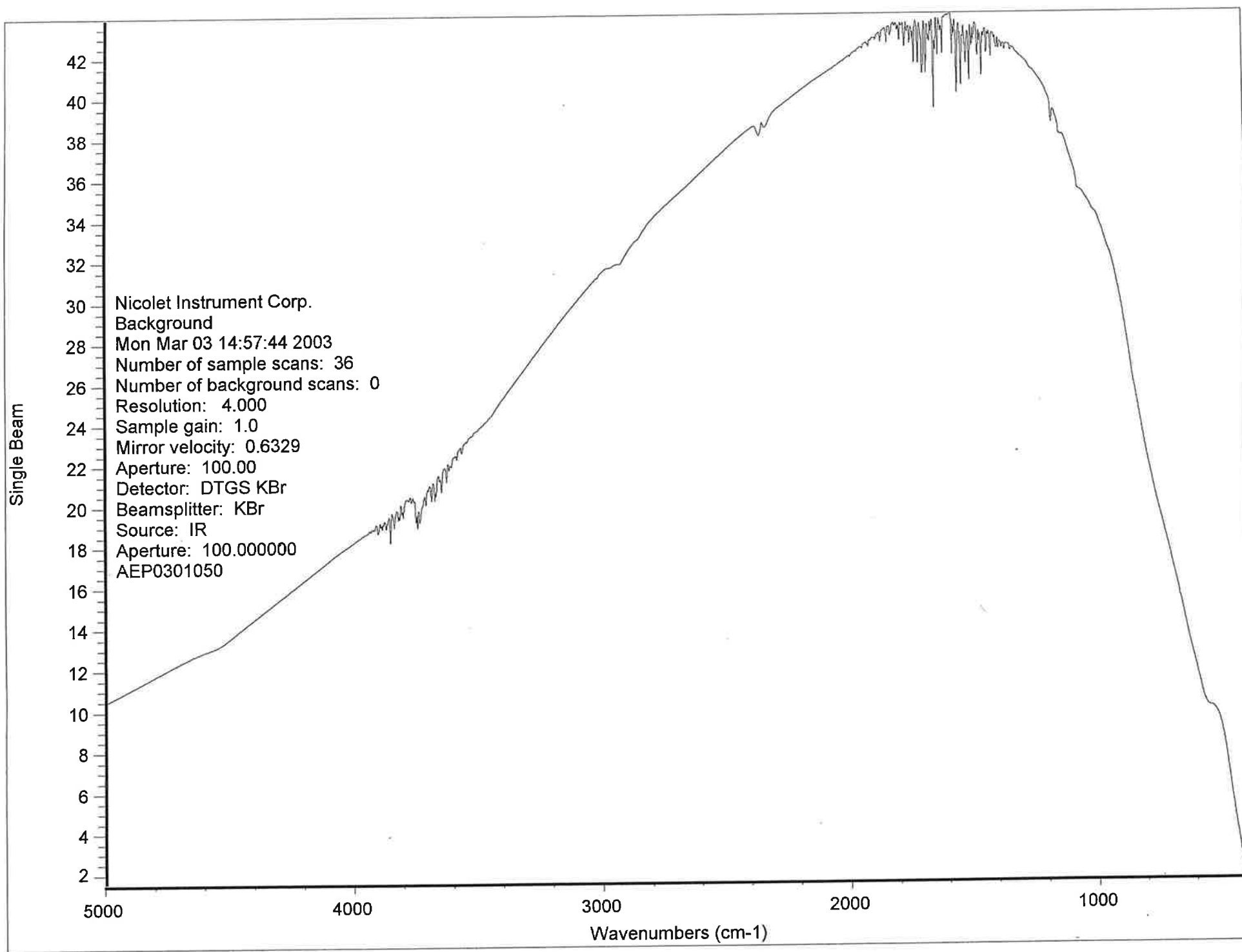
3000

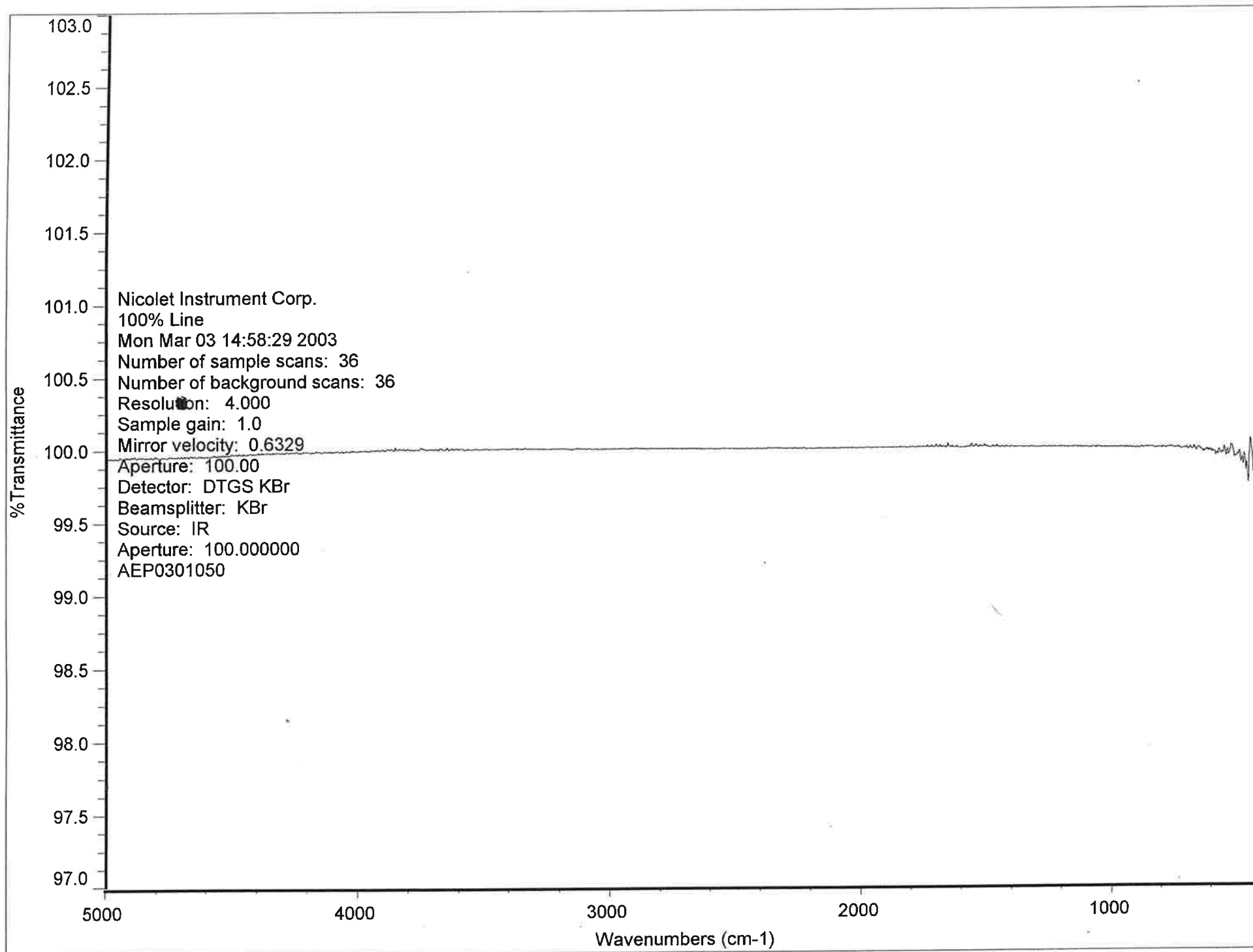
2000

1000

Wavenumbers (cm-1)

Nicolet Instrument Corp.  
Background  
Mon Mar 03 14:57:44 2003  
Number of sample scans: 36  
Number of background scans: 0  
Resolution: 4.000  
Sample gain: 1.0  
Mirror velocity: 0.6329  
Aperture: 100.00  
Detector: DTGS KBr  
Beamsplitter: KBr  
Source: IR  
Aperture: 100.000000  
AEP0301050





# Shipment Authorization

Customer Name: Tennessee Bureau of Investigation

Job Number: 137320 System Serial Number: AEO 0300167

☒ This instrument meets or exceeds all applicable performance specifications.

I validate the performance of this instrument:

John B. Cabren  
Test Technician

3/21/03  
Date

☒ All components of your order are present unless otherwise noted on the Notification of Partial Shipment form included in this envelope.

I authorize shipment of this order:

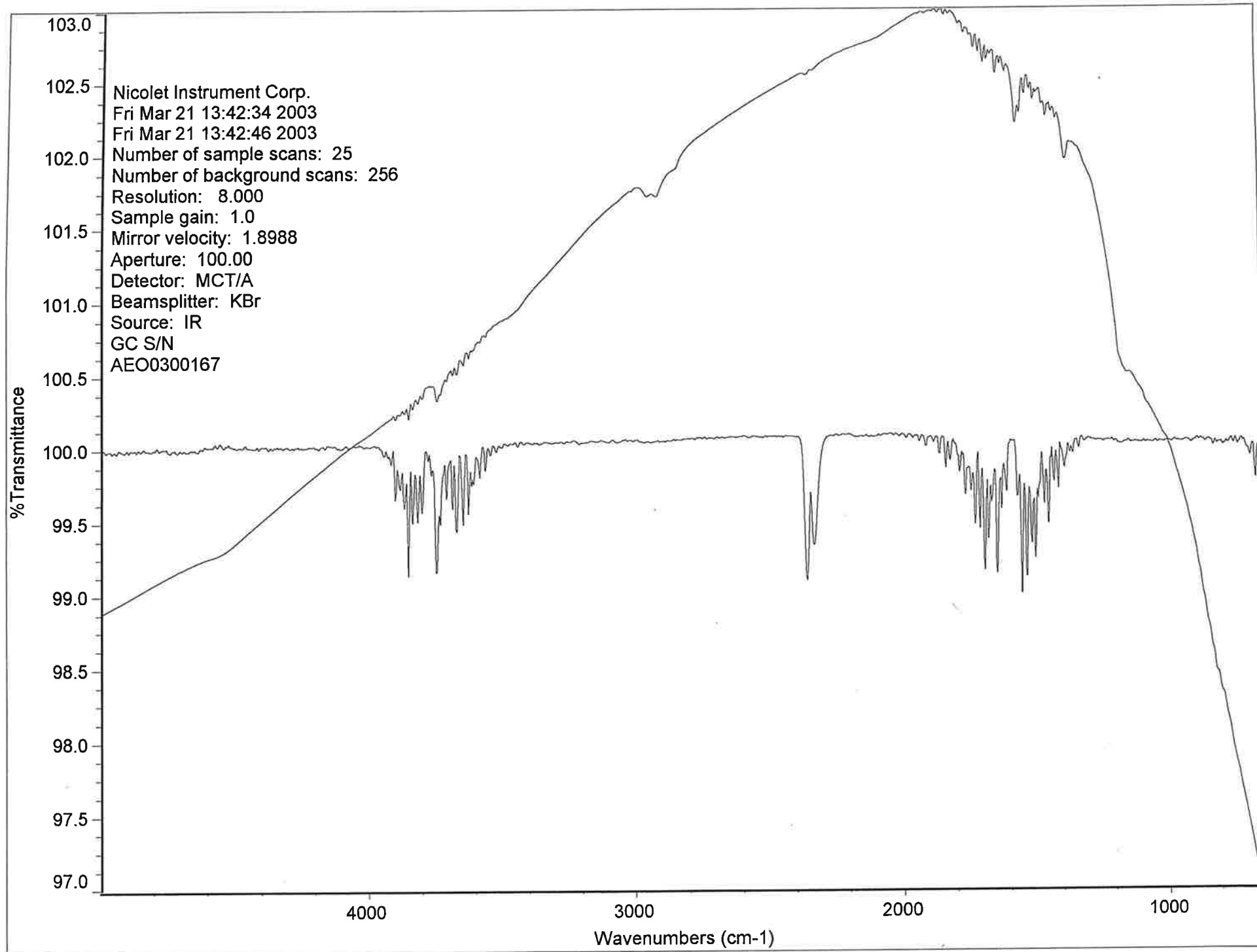
Ben Hill

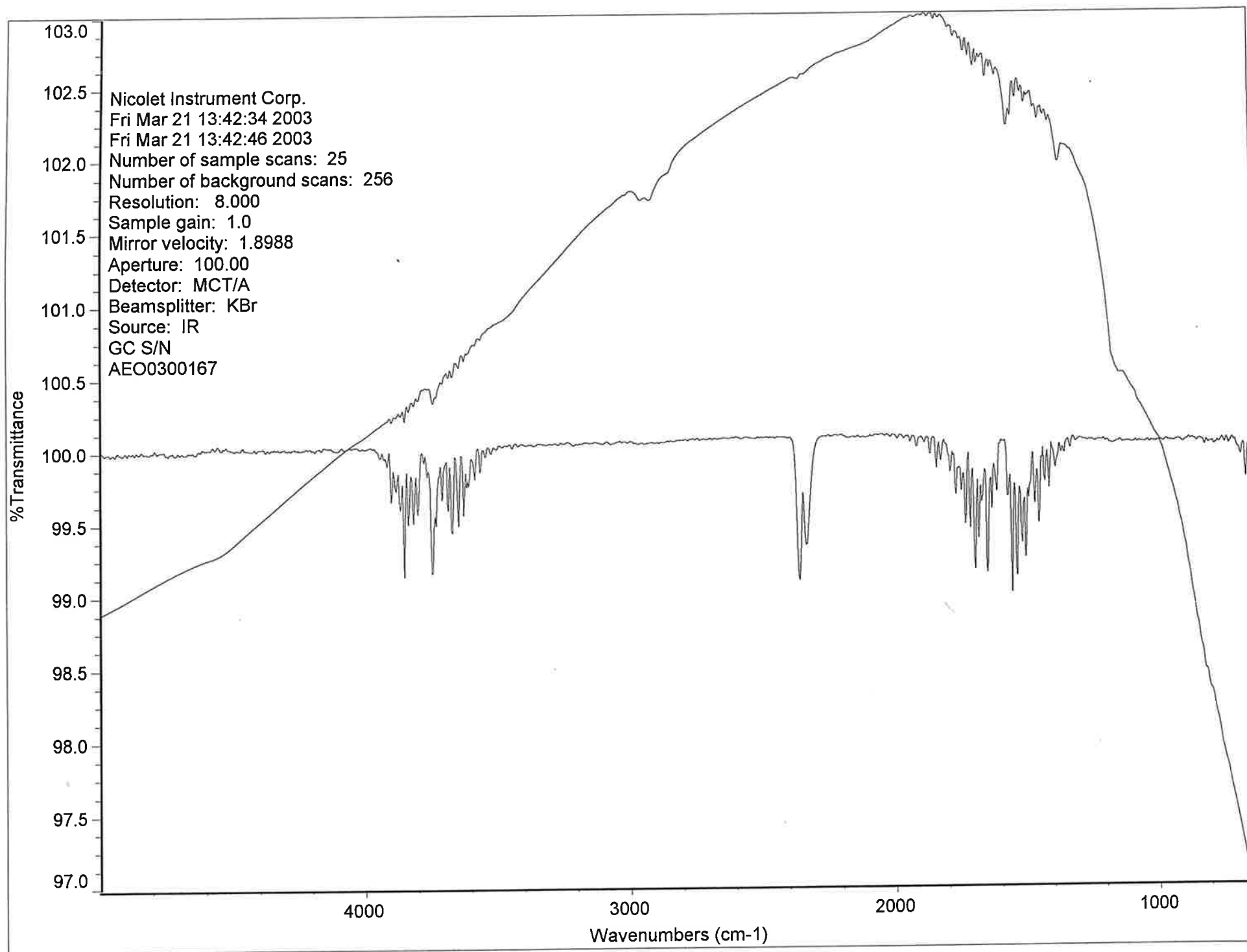
System Configuration Specialist

3/24/03

Date

***If you have any problems concerning this order please contact your nearest Sales/Service Office or Representative.***







# DECLARATION OF CONFORMITY

According to ISO/IEC Guide 22 and CEN/CENELEC EN 45014



**Manufacturer's Name:**  
**Manufacturer's Address:**

Agilent Technologies, Inc.  
Little Falls Site 2350  
2850 Centerville Road  
Wilmington, De. 19808

Agilent Technologies, Inc.  
ASA Site  
12 Cangwu Road  
Shanghai 200233, P. R. China

**Declares, that the product**

**Product Name:** Integrated Automatic Liquid Sample Injection System  
**Model Number:** 7683x Comprised of:  
G2612x, G2613x, G2614x Where x=Any Letter  
**Product Options:** All options, accessories, and customized products based on the above listed models

## Conforms with the following European Directives:

The product herewith complies with the requirements of the Low Voltage Directive 73/23/EEC and the EMC Directive 89/336/EEC (including 93/68/EEC) and carries the CE Marking accordingly.

## Conforms with the following Product Standards:

|             |                                                     |                                  |
|-------------|-----------------------------------------------------|----------------------------------|
| <b>EMC:</b> | IEC 61326-1:1997 +A1:1998/EN 61326-1:1997 +A1:1998) | EN 50082-1                       |
|             | Standard                                            | Limit                            |
|             | CISPR11, 3rd:1997/EN 55011, 3rd:1998                | Group1, ClassA <sup>1)</sup>     |
|             | IEC 61000-4-2:1995+A1:1998/EN 61000-4-2:1995'       | 4kV CD, 8kV AD                   |
|             | IEC 61000-4-3:1995/EN 61000-4-3:1995                | 3V/m, 80-1000 MHz, 80% AM        |
|             | IEC 61000-4-4:1995/EN 61000-4-4:1995                | 0.5kV sig. lines 1kV power lines |
|             | IEC 61000-4-5:1995/EN 61000-4-5:1995                | 0.5kV L-L 1kV L-G                |
|             | IEC 61000-4-6:1996/EN 61000-4-6:1998                | 3V, 0.15-80 MHz                  |
|             | IEC 61000-4-11:1994/EN 61000-4-11:198               | >-95%: 0.5, Cycles               |

**Safety:** IEC 61010-1:1990 +A1:1992 +A2:1995  
EN 61010-1:1993 +A2:1995  
Canada/CSA-C22.2 No. 1010.1-92 <sup>2)</sup>  
UL 3101, 1st Edition

## Supplementary Information:

- [1] The product was tested in a typical configuration with Agilent Technologies test systems.  
[2] CSA Certified

**March 21, 2001**

Date

*Nancy J. Adams*

Name  
Quality Manager  
Title

For further information, please contact your local Agilent Technologies sales office, agent or distributor.