

TBI-FORENSIC SERVICES DIVISION PERFORMANCE VERIFICATION FORM

Instrument/Method to Verify:

Agilent 8890 GC with ASAP Analytical IRD3 (YODA) - MAN60TO270 Method

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

Laboratory: NCL ☒ KCL ☐ JCL ☐ Unit: Forensic Chemistry

Based on training and competency, the following analyst(s) are authorized by the Technical Leader to participate in this study:

Name Rebecca Hernandez Title SAFS-S Initials RH

Completed: Rebecca Hernandez Digitally signed by Rebecca Hernandez
Date: 2026.04.14 12:33:01 -05'00'

Name _____ Title _____ Initials _____

Completed:

Name _____ Title _____ Initials _____

Completed:

Verification of final review:

Unit Supervisor: Brett Trotter Digitally signed by Brett Trotter
Date: 2026.04.16 08:16:03 -05'00' Section Manager: Glenn Everett Digitally signed by Glenn Everett
Date: 2026.04.16 09:01:08 -05'00'

Unit Supervisor:

Section Manager:

Unit Supervisor:

Section Manager:

Regional Administrator: Jenifer Hall Digitally signed by Jenifer Hall
Date: 2026.04.16 10:09:43 -05'00' IT Manager:

Regional Administrator:

Regional Administrator:

Quality Assurance Manager: Chad D Johnson Digitally signed by Chad D
Johnson
Date: 2026.04.16 11:09:49 -05'00'

By signing below, I have verified this study meets all requirements and I deem this procedure/
instrument fit for the intended use:

Technical Leader: Brett Trotter Digitally signed by Brett Trotter
Date: 2026.04.16 08:16:21 -05'00'

All information must be submitted to the Quality Assurance Manager prior to the implementation of the
procedure/instrument. If applicable, include training plan and competency testing schedule.

**Performance Check of Manual Method “MAN60TO270” on
Agilent GC 8890 ASAP Analytical IRD3 GC-FTIR (vapor
phase) “Yoda”**

Table of Contents

I. Introduction

II. Materials

III. Methods/Procedures

IV. Experiments/Studies Performed

○ Performance Check of Manual Method

V. Results and Discussion

VI. Conclusions

VII. Training

I. Introduction:

This performance check is designed to demonstrate the effectiveness of using the previously validated GC-FTIR vapor phase manual method “MAN60TO270” for headspace analysis by running a series of nitrite standards by headspace injection on a DB-5 column, 20-meter, 0.32 ID, 0.50 um film thickness on ASAP Analytical’s IRD3 (IRD3) “Yoda” instrument. The standards will be evaluated to determine if they elute within the allotted time frame and temperature program, have resolved peaks, and demonstrate spectra that is comparable to known standards in the TBI GC-FTIR library. Once deemed fit for use, all analysts will be notified about the method being available for use on Yoda with a reminder about the manual instructions listed in the instruments operating procedures.

Validation of the following will include:

- Performance Check of Manual Method
- A. Performance Check of Manual Method – A series of nitrite standards will be analyzed by headspace using Hamilton headspace syringe and injecting it on a DB-5, 20 meter column, 0.32 ID, 0.50 um film thickness with a previously validated GC-IR manual method “MAN60TO270” in order to ensure their vapor phase IR spectra can be obtained and compared to nitrite standards previously run and within the TBI GCIR library.

II. Materials:

Serial Number Information:

GC: Model 8890 SN#US2245A073

IR: Model 528A SN#A10054-B16

Autosampler: Model G4513A SN#RO22425083

Tray: Model G4514A SN#RO22387026

Column & Liner:

Agilent J&W GC DB-5, 20 meter, 0.32 ID, 0.50 um column (S/N#US4394326H)

Restek Split/Splitless Straight with wool liner

Hamilton Headspace Syringe

Glass blood alcohol vials with crimped tops (retrieved from Toxicology Unit)

Standards:

Isoamyl Nitrite
Sigma Aldrich Lot#09829LC

Tert-Butyl Nitrite
Sigma Aldrich Lot#BCCB6099

Butyl Nitrite
Sigma Aldrich Lot#MKCL4353

Isobutyl Nitrite
Sigma Aldrich Lot#MKCL3264

III. Methods/Procedures:

The following standards were run with corresponding method on Yoda (Agilent/ASAP Analytical IRD3 GC-FTIR):

The “IRD3” method on the ASAP Analytical IRD3 was used in conjunction with all Agilent GC methods.

Performance Check of Method

Manual:

MAN60TO270 – Tert-Butyl Nitrite, Butyl Nitrite, Isoamyl Nitrite, Isobutyl Nitrite

MAN60TO270

Initial 60C degrees hold for 2 minutes ramped to 270C degrees at 70C/min.
Constant carrier flow rate of 3.0 mL/min, Split ratio of 2.0, Split flow 6.0 mL/min, Pressure 13.827 psi, Septum purge flow 3 mL/min, Inlet temperature of 250C degrees. Total run time 10.00 minutes.

IV. Experiment:

This instrument consists of an Agilent Technologies 8890 Gas Chromatograph with Agilent Autosampler G4513A coupled to an ASAP Analytical Model 528A IRD3 GC-FTIR. It was installed and set up by an ASAP Analytical service engineer. Existing ASAP Analytical IRD3's in our laboratory were previously validated and run at varying conditions to determine optimum values and methods to obtain the best results. Previously validated methods,

including manual injection methods, were introduced for hand injecting more sample volume for lower concentration samples and for headspace samples. A series of nitrite standards will be analyzed by headspace using a Hamilton headspace syringe and injecting it on a DB-5, 20 meter column, 0.32 ID, 0.50 um film thickness with a previously validated GC-IR manual method “MAN60TO270” in order to ensure their vapor phase IR spectra can be obtained and compared to nitrite standards previously run and within the TBI GCIR library. Manual injections used a Hamilton headspace syringe using 1 mL of each standards headspace in glass blood alcohol vials with crimped tops (retrieved from Toxicology Unit) and blank headspace (air) in glass blood alcohol vials with crimped tops were used prior to running the standard to ensure there was no carryover or contamination of the headspace syringe.

A Performance Check of Manual Method “MAN60TO270” was performed by the following:

A. Performance Check of Method- The following standards were analyzed: Isoamyl Nitrite Sigma Aldrich, Tert-Butyl Nitrite, Butyl Nitrite, Isobutyl Nitrite by headspace. These standards were analyzed using a manual method that has previously been validated. A DB-5, 20-meter column, 0.32 ID, 0.50 um film thickness was utilized. Blanks were run before each standard under the same method parameters of the standard. These standards were then compared to the TBI GCIR library database.

V. Results and Discussion:

For Performance Check of the manual method “MAN60TO270” on ASAP Analytical’s IRD3 (IRD3) Yoda, the standards run eluted within the allotted time frame and temperature program, had resolved peaks, and demonstrated spectra that were comparable to known standards in the TBI GC-FTIR library. See attached data for results.

Optical alignment value was monitored on Yoda, and any necessary alignment and instrument inlet maintenance was performed prior to analyzing standards. The chart below outlines results obtained.

Date	Optical Alignment Value
03/31/26	1.42

VI. Conclusions:

After a Performance Check of the manual method “MAN60TO270”, the ASAP Analytical IRD3 instrument showed comparable results in instrument responses and to existing TBI GC-FTIR libraries. The instrument method is now fit for use.

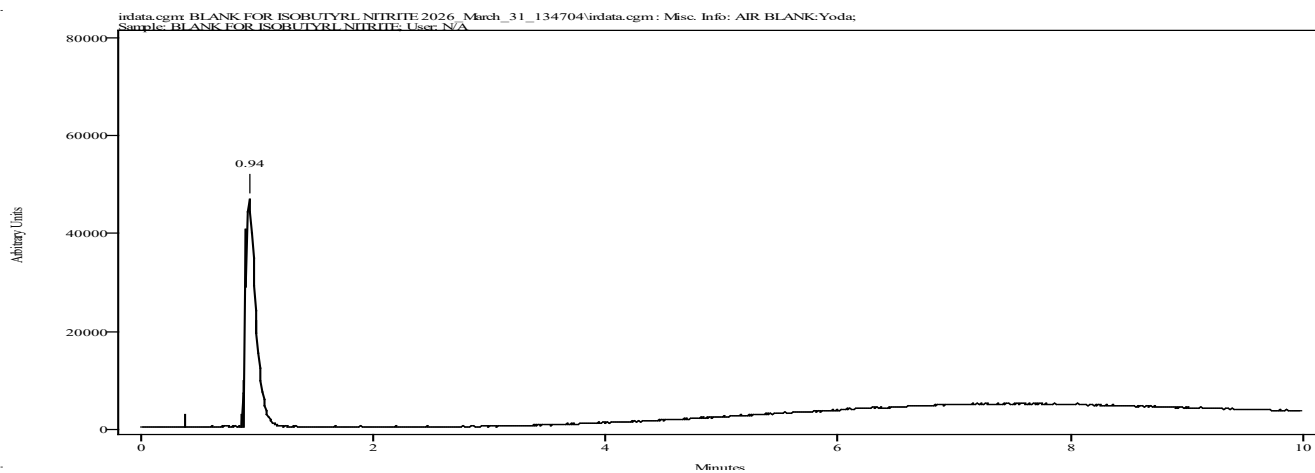
VII. Training:

No training is required at this time, but an email will be sent to notify scientists that the method is available for use on Yoda with a reminder about the manual instructions listed in the instruments operating procedures.

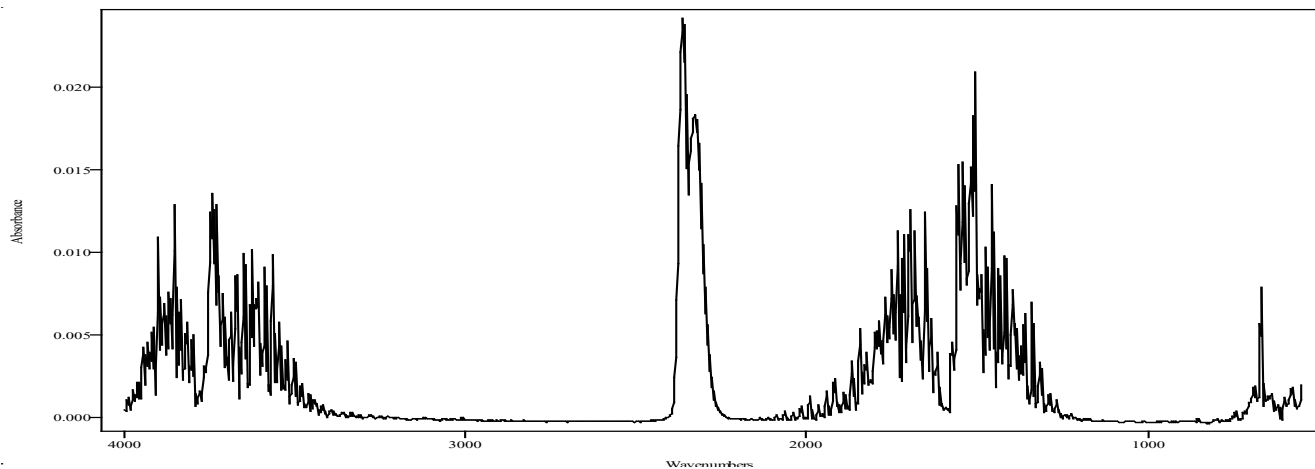
RH

IRD Data Analysis Report

Data Filename	BLANK FOR ISOBUTYRL NITRITE 2026_March_31_134704
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BLANK FOR ISOBUTYL NITRITE
Misc. Info	AIR BLANK (Headspace):Yoda
User ID	N/A
Injection Time	Tue Mar 31 13:52:33 2026
Date Processed	Tue Apr 07 15:00:19 2026



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.892	1.003	0.111	0.379	0.869

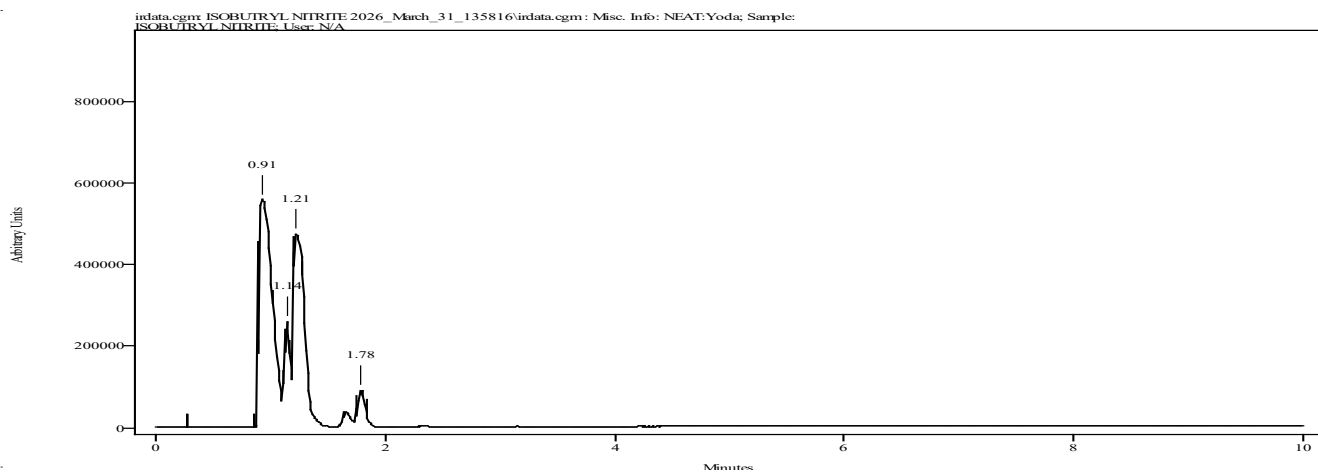


IRDATA.SPC: 0.94 minutes: AVE (0.89: 1.00) Ref. (0.38: 0.87) of BLANK FOR ISOBUTYL NITRITE
2026_March_31_134704.cgm

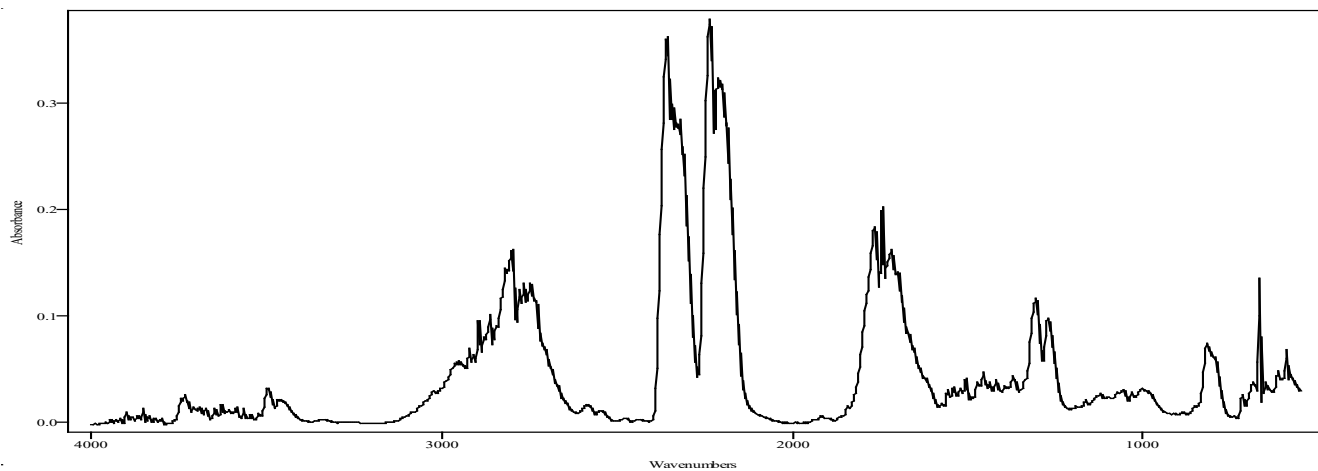
RH

IRD Data Analysis Report

Data Filename	ISOBUTRYL NITRITE 2026_March_31_135816
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	ISOBUTYL NITRITE Sigma Aldrich Lot#MKCL3264 RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 14:06:35 2026
Date Processed	Tue Apr 07 15:06:28 2026



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.880	1.014	0.134	0.267	0.847
1.103	1.159	0.056	0.267	0.847
1.194	1.268	0.078	0.267	0.847
1.750	1.828	0.078	0.267	0.847

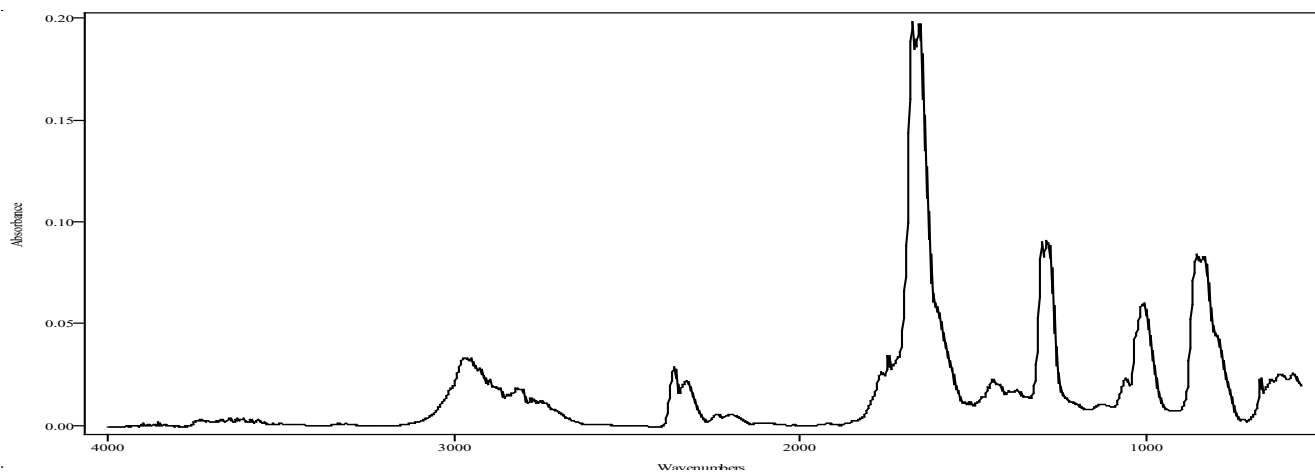


IRDATA.SPC: 0.91 minutes: AVE (0.88: 1.01) Ref. (0.27: 0.85) of ISOBUTYL NITRITE
2026_March_31_135816.cgm

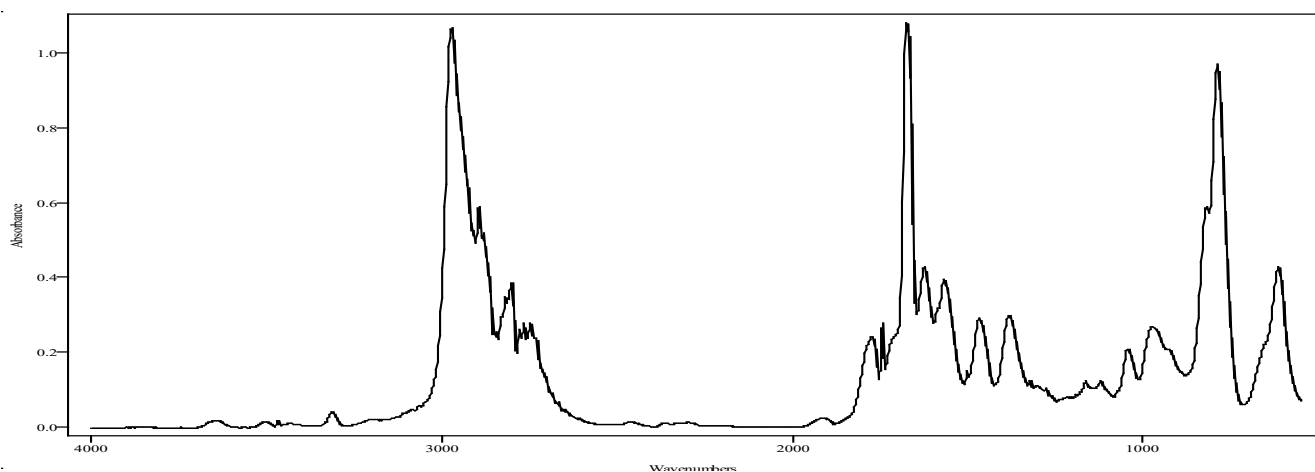
RH

IRD Data Analysis Report

Data Filename	ISOBUTRYL NITRITE 2026_March_31_135816
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	ISOBUTYL NITRITE Sigma Aldrich Lot#MKCL3264 RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 14:06:35 2026
Date Processed	Tue Apr 07 15:06:28 2026

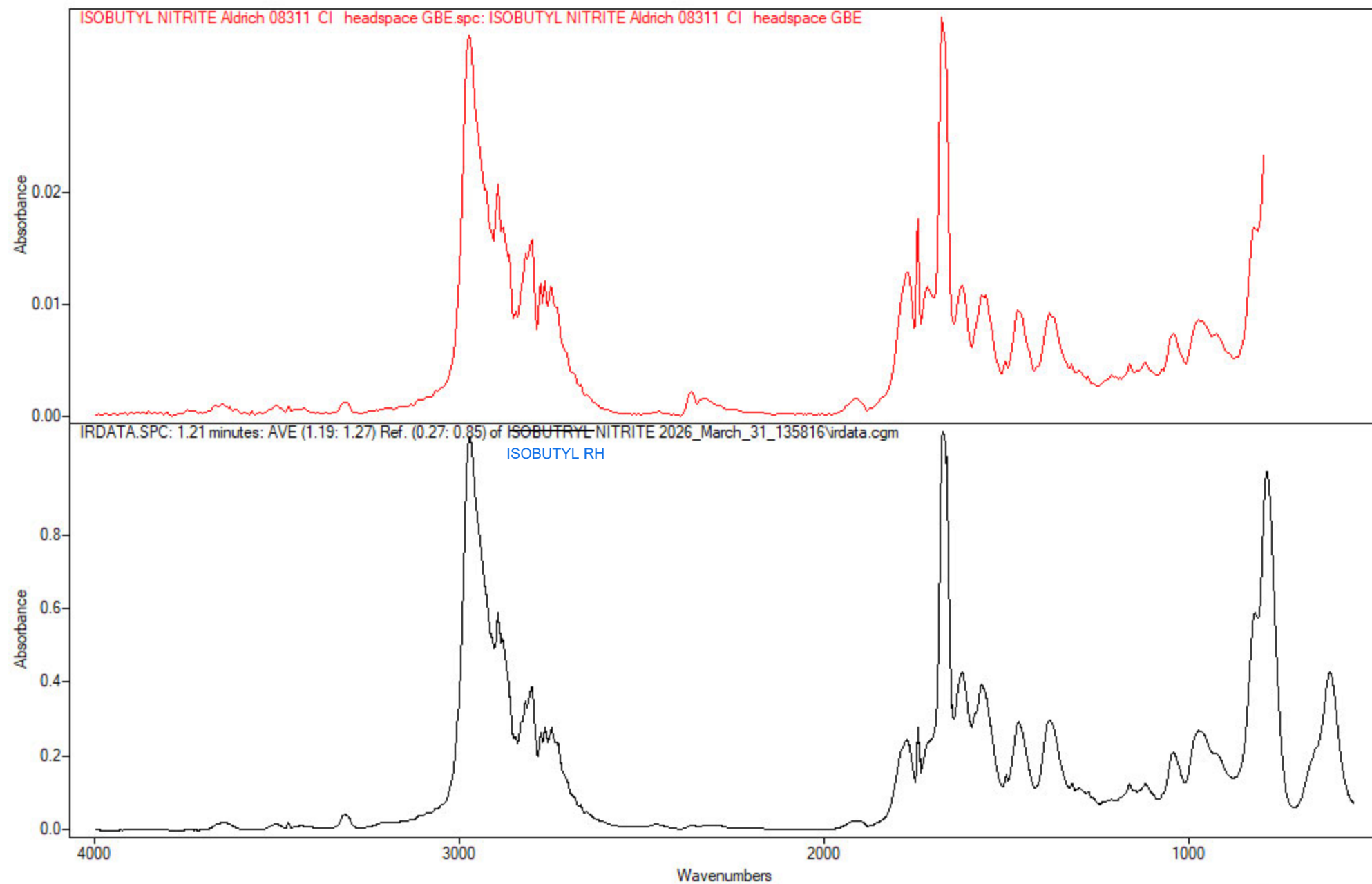


IRDATA.SPC: 1.14 minutes: AVE (1.10: 1.16) Ref. (0.27: 0.85) of ISOBUTYL NITRITE
2026_March_31_135816.cgm



IRDATA.SPC: 1.21 minutes: AVE (1.19: 1.27) Ref. (0.27: 0.85) of ISOBUTYL NITRITE
2026_March_31_135816.cgm

ISOBUTYL NITRITE

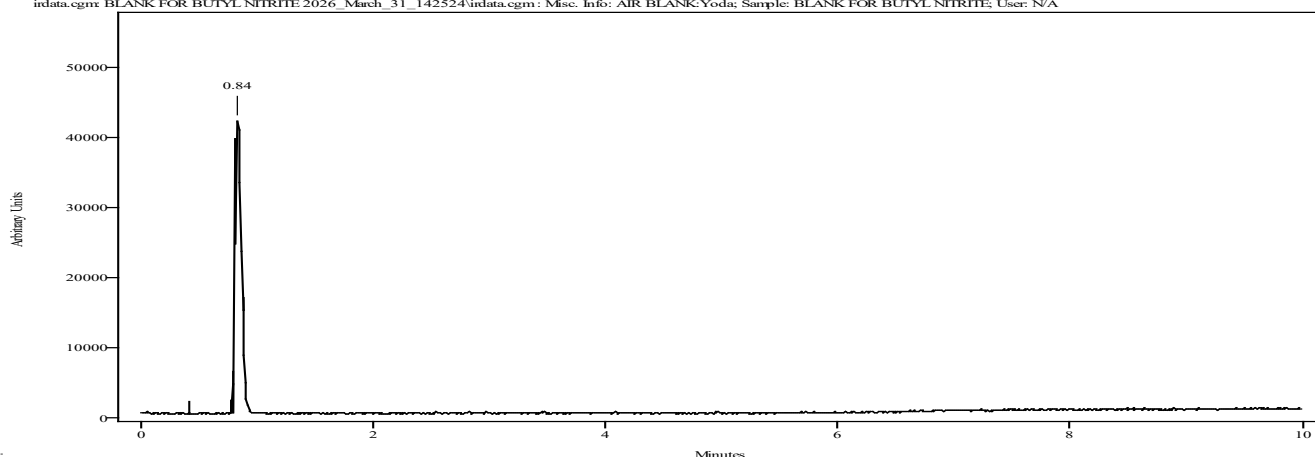


RH

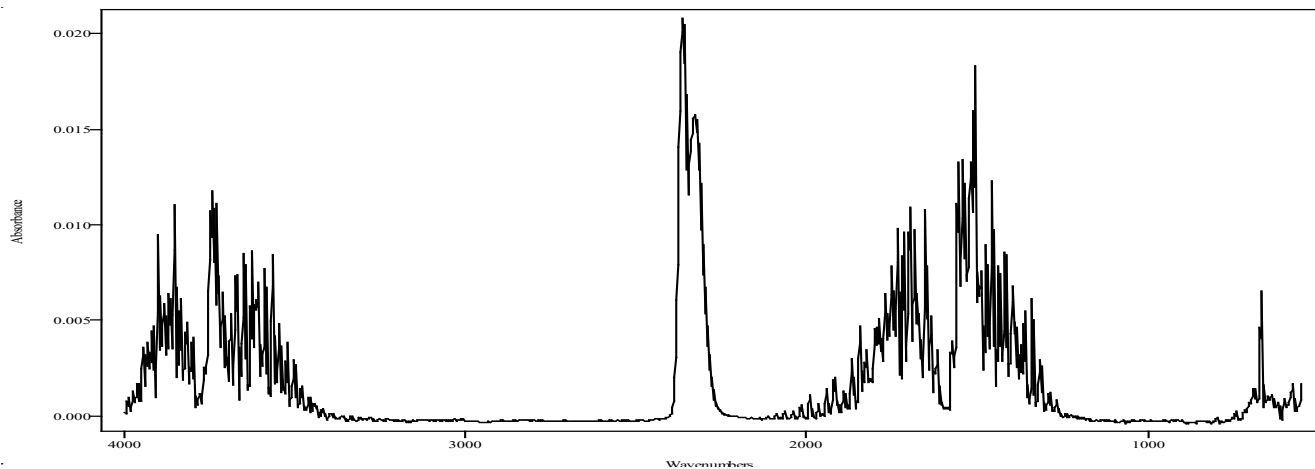
IRD Data Analysis Report

Data Filename	BLANK FOR BUTYL NITRITE 2026_March_31_142524
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BLANK FOR BUTYL NITRITE
Misc. Info	AIR BLANK (Headspace):Yoda
User ID	N/A
Injection Time	Tue Mar 31 14:32:52 2026
Date Processed	Tue Apr 07 12:32:13 2026

irdata.cgm: BLANK FOR BUTYL NITRITE 2026_March_31_142524\irdata.cgm : Misc. Info: AIR BLANK:Yoda; Sample: BLANK FOR BUTYL NITRITE; User: N/A



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.802	0.880	0.078	0.424	0.780

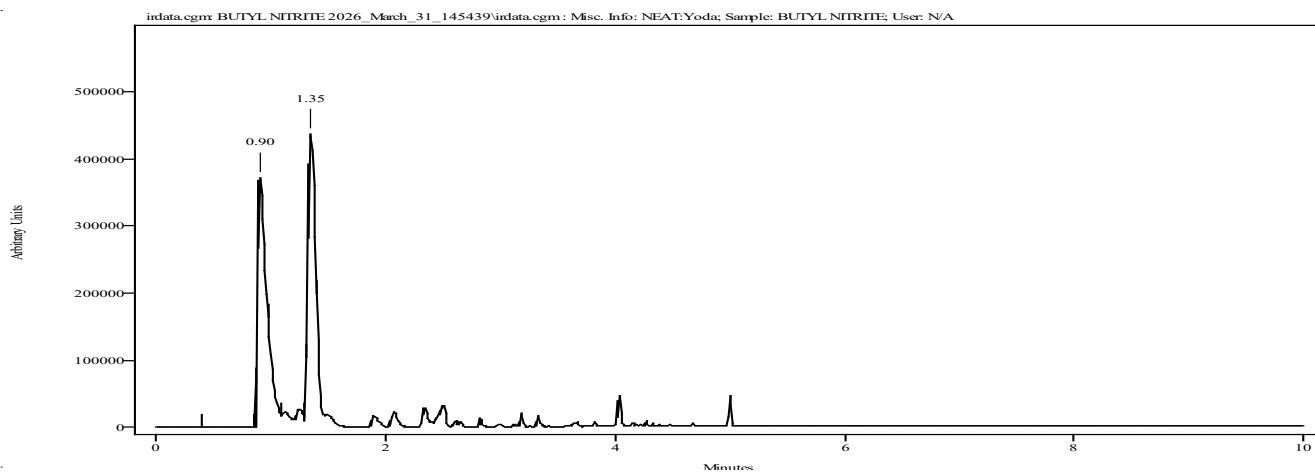


IRDATA.SPC: 0.84 minutes: AVE (0.80: 0.88) Ref. (0.42: 0.78) of BLANK FOR BUTYL NITRITE
2026_March_31_142524.cgm

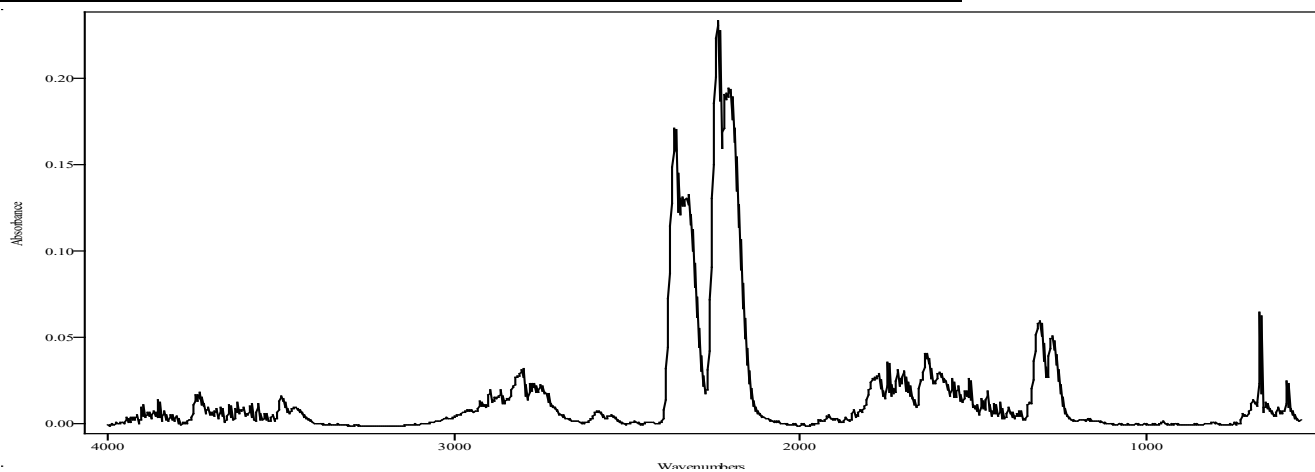
RH

IRD Data Analysis Report

Data Filename	BUTYL NITRITE 2026_March_31_145439
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BUTYL NITRITE Sigma Aldrich Lot#MKCL4353 RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:03:09 2026
Date Processed	Tue Apr 07 12:36:25 2026



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.870	0.970	0.100	0.401	0.847
1.304	1.393	0.089	1.092	1.282

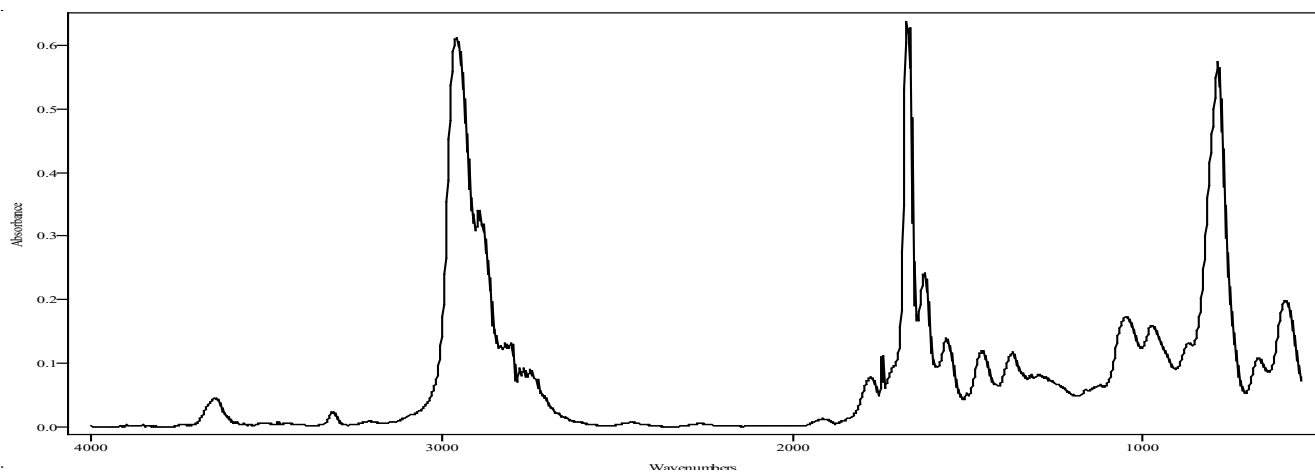


IRDATA.SPC: 0.90 minutes: AVE (0.87: 0.97) Ref. (0.40: 0.85) of BUTYL NITRITE
2026_March_31_145439.cgm

RH

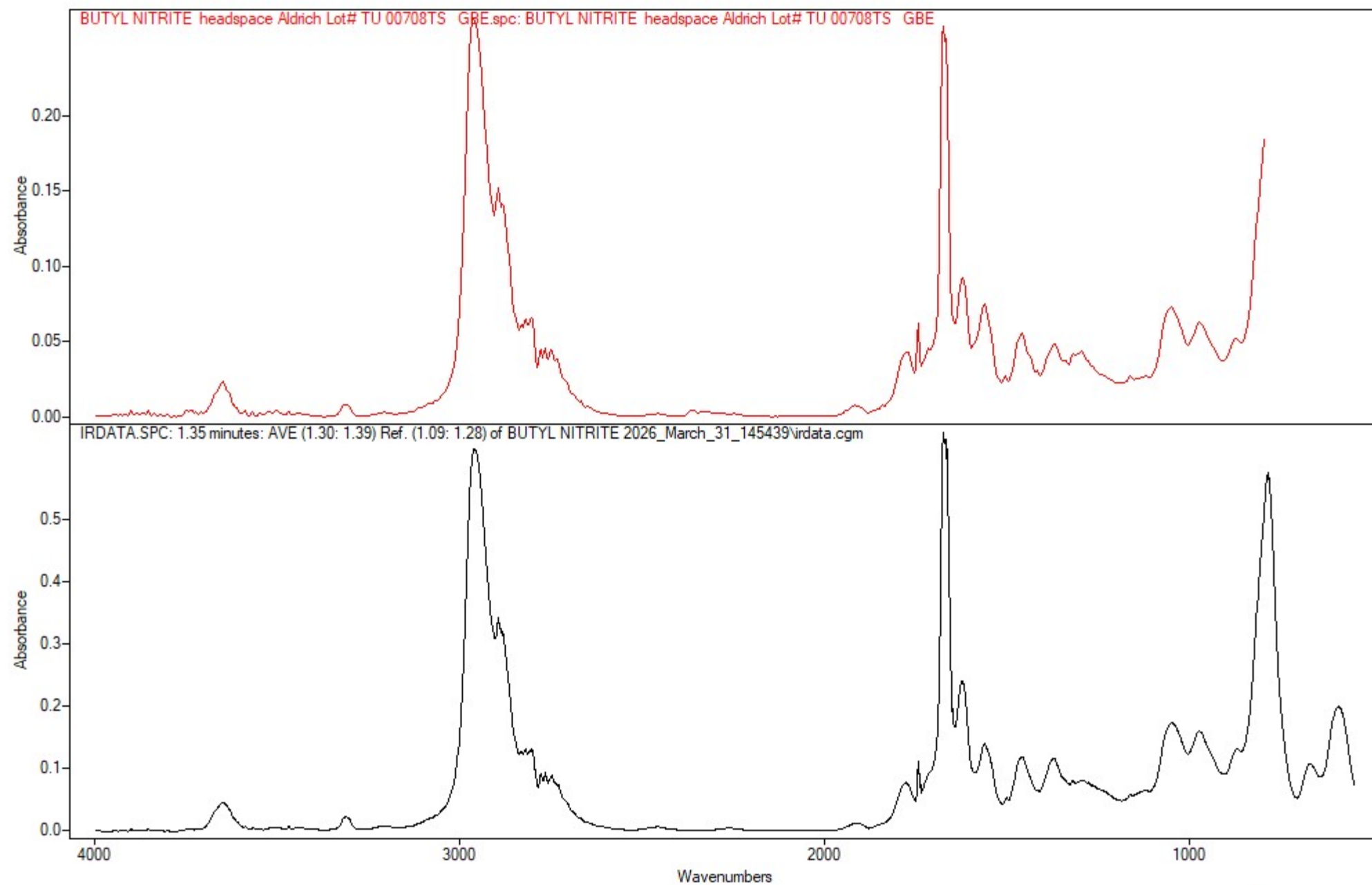
IRD Data Analysis Report

Data Filename	BUTYL NITRITE 2026_March_31_145439
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BUTYL NITRITE Sigma Aldrich Lot#MKCL4353 RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:03:09 2026
Date Processed	Tue Apr 07 12:36:25 2026



IRDATA.SPC: 1.35 minutes: AVE (1.30: 1.39) Ref. (1.09: 1.28) of BUTYL NITRITE
2026_March_31_145439.cgm

BUTYL NITRITE

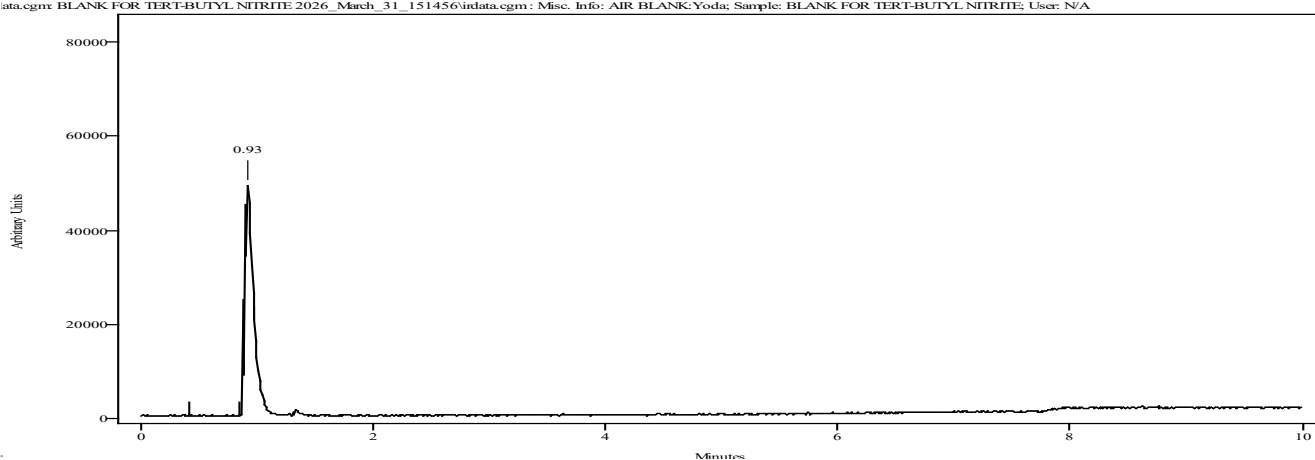


RH

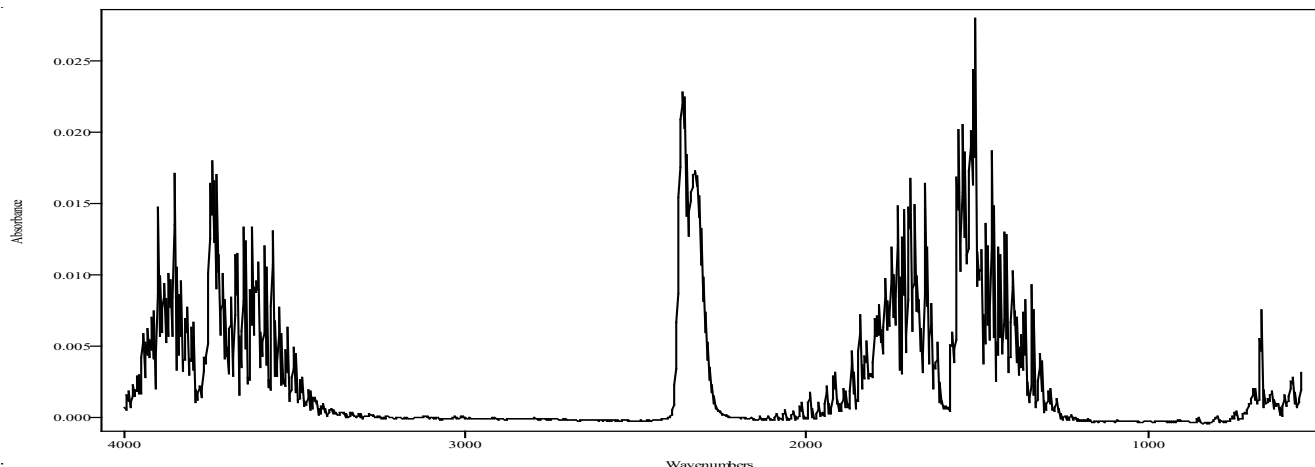
IRD Data Analysis Report

Data Filename	BLANK FOR TERT-BUTYL NITRITE 2026_March_31_151456
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BLANK FOR TERT-BUTYL NITRITE
Misc. Info	AIR BLANK (Headspace):Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:20:59 2026
Date Processed	Tue Apr 07 14:45:10 2026

ata.cgm: BLANK FOR TERT-BUTYL NITRITE 2026_March_31_151456\data.cgm : Misc. Info: AIR BLANK:Yoda; Sample: BLANK FOR TERT-BUTYL NITRITE; User: N/A



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.881	0.982	0.100	0.413	0.859

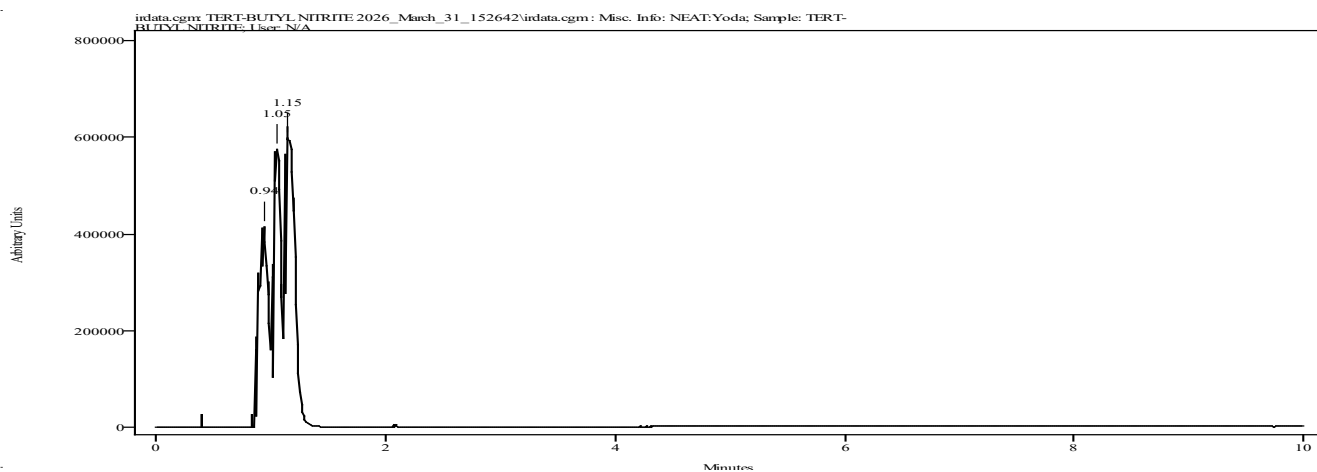


IRDATA.SPC: 0.93 minutes: AVE (0.88: 0.98) Ref. (0.41: 0.86) of BLANK FOR TERT-BUTYL NITRITE 2026_March_31_151456.cgm

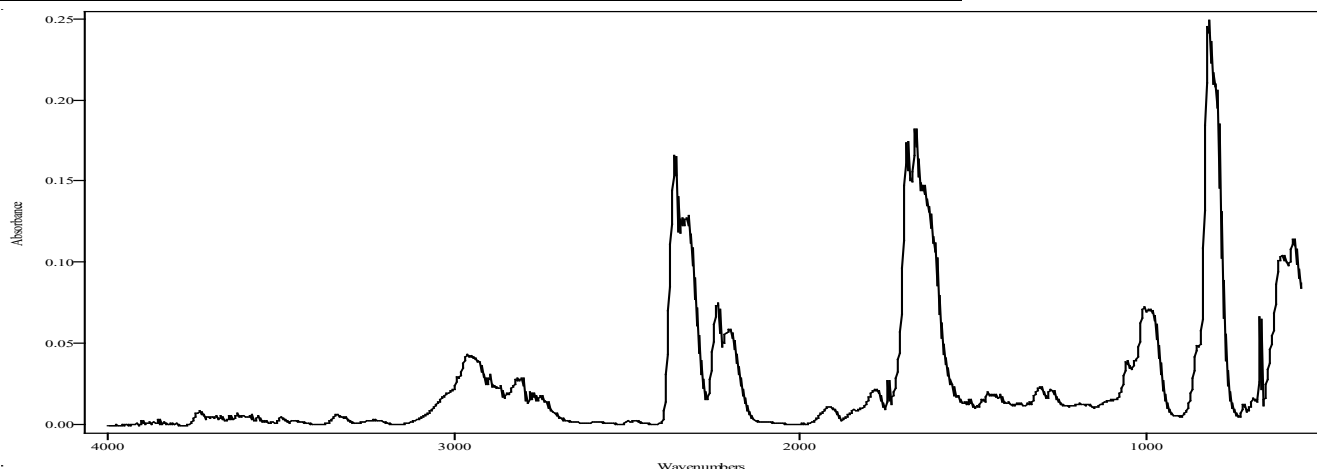
RH

IRD Data Analysis Report

Data Filename	TERT-BUTYL NITRITE 2026_March_31_152642
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	TERT-BUTYL NITRITE SIGMA ALDRICH LOT#BCCB6099 RH
Misc. Info	HEADSPACE:Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:34:09 2026
Date Processed	Tue Apr 07 14:56:50 2026



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.870	0.970	0.100	0.390	0.836
1.015	1.093	0.078	0.390	0.836
1.134	1.195	0.056	0.390	0.836

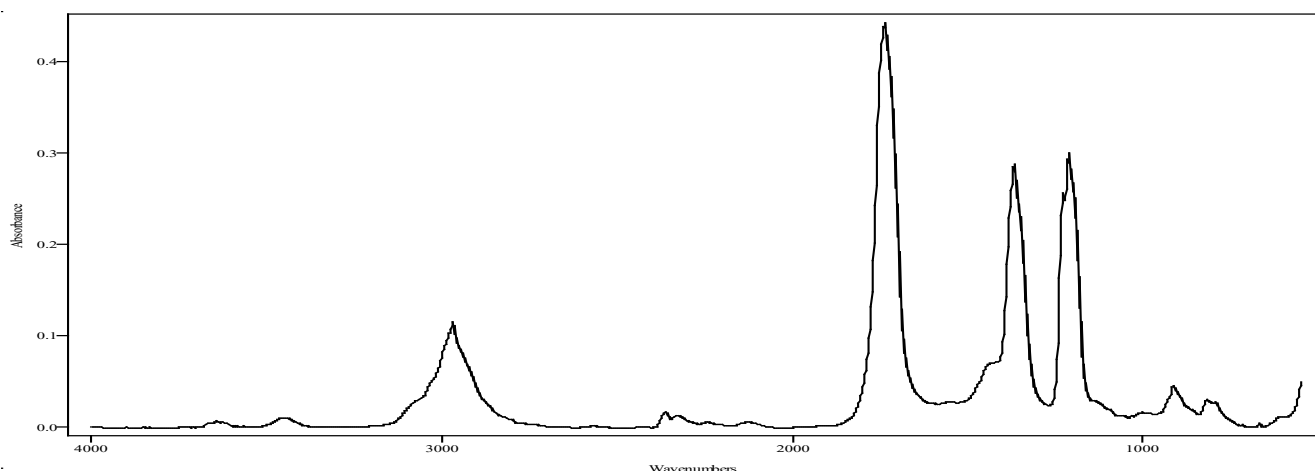


IRDATA.SPC: 0.94 minutes: AVE (0.87: 0.97) Ref. (0.39: 0.84) of TERT-BUTYL NITRITE
2026_March_31_152642.cgm

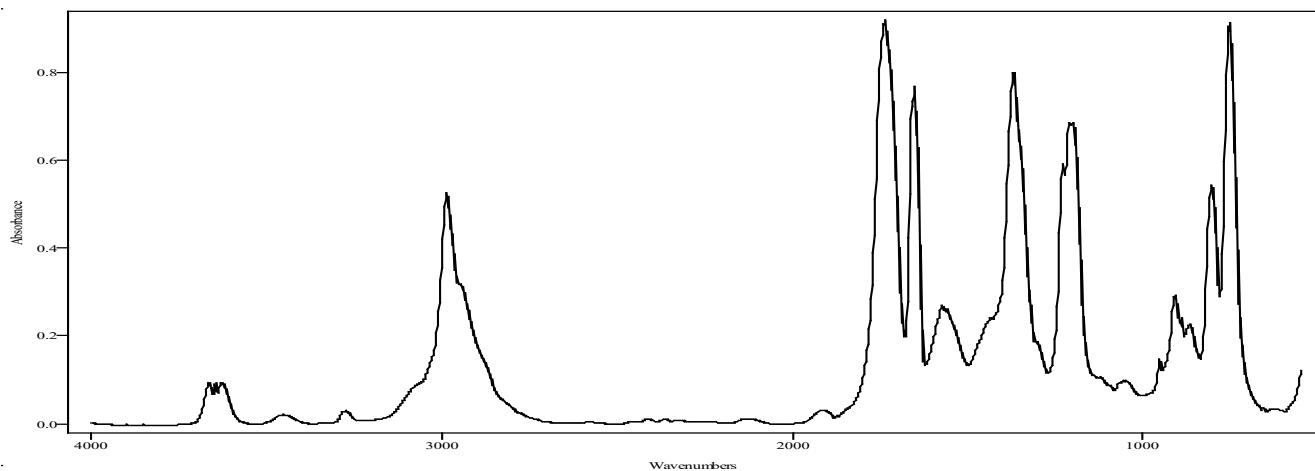
RH

IRD Data Analysis Report

Data Filename	TERT-BUTYL NITRITE 2026_March_31_152642
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	TERT-BUTYL NITRITE SIGMA ALDRICH LOT#BCCB6099 RH
Misc. Info	HEADSPACE:Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:34:09 2026
Date Processed	Tue Apr 07 14:56:50 2026



IRDATA.SPC: 1.05 minutes: AVE (1.01: 1.09) Ref. (0.39: 0.84) of TERT-BUTYL NITRITE
2026_March_31_152642.cgm

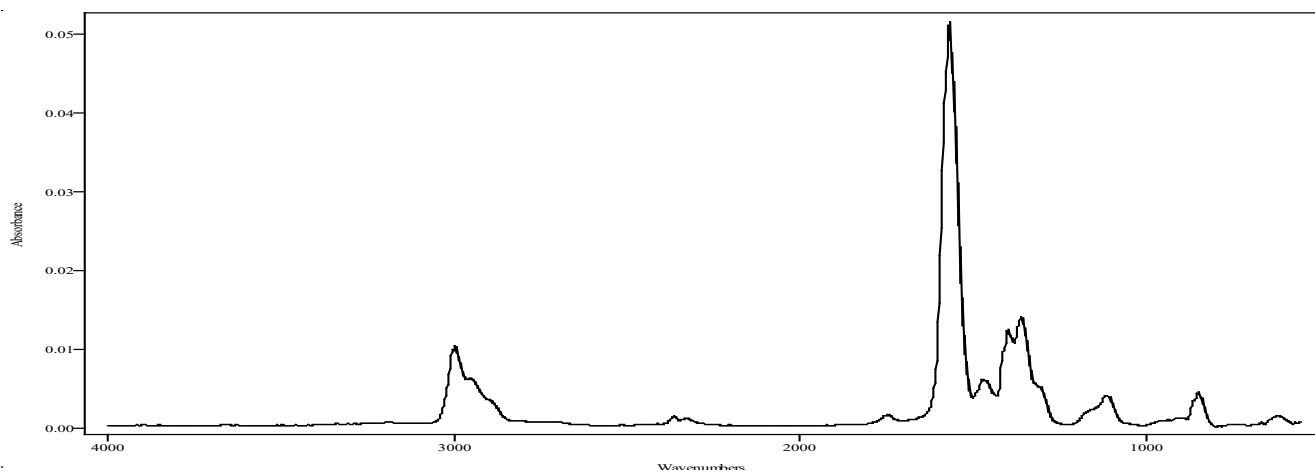


IRDATA.SPC: 1.15 minutes: AVE (1.14: 1.19) Ref. (0.39: 0.84) of TERT-BUTYL NITRITE
2026_March_31_152642.cgm **TERT-BUTYL NITRITE**

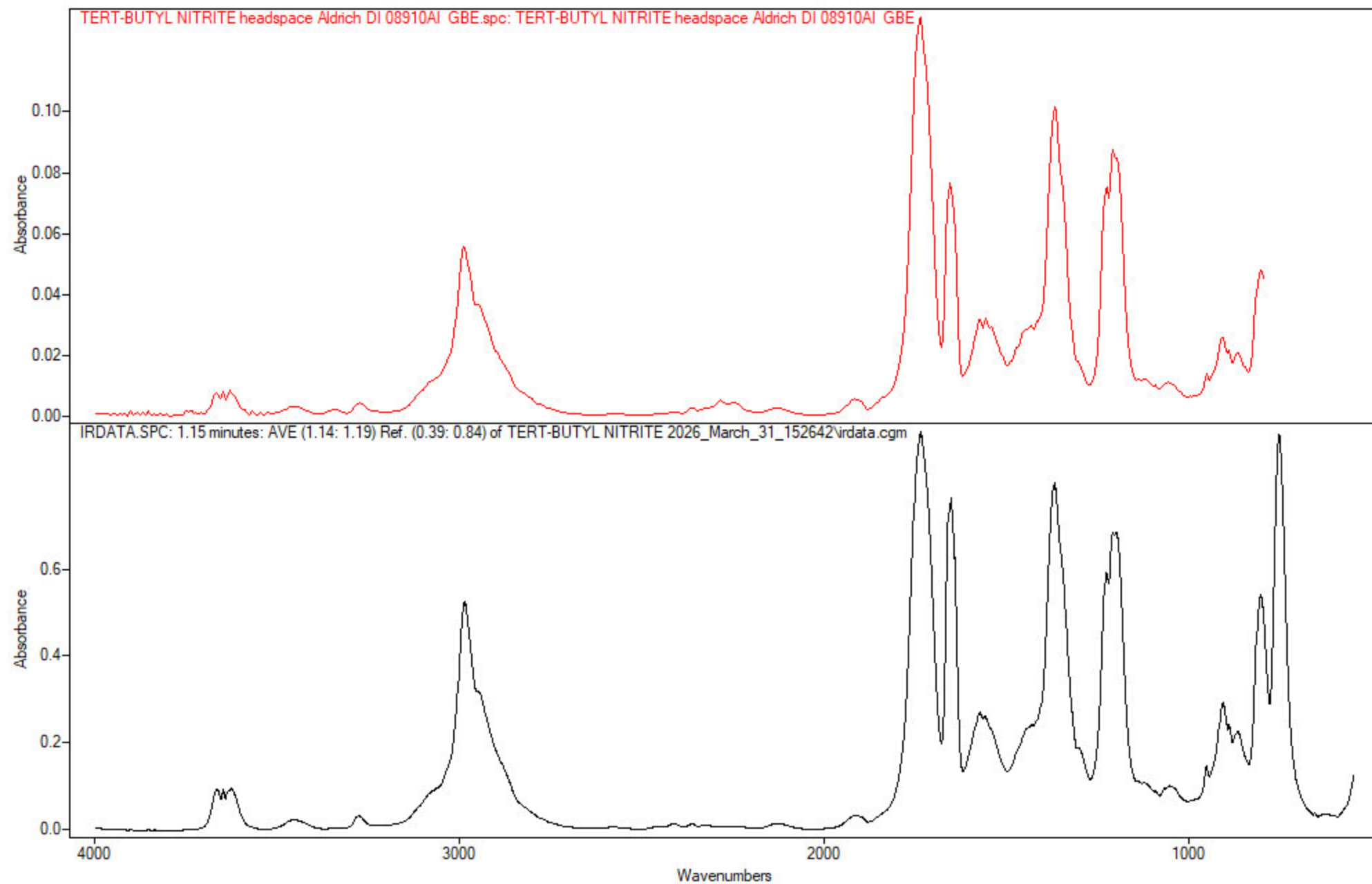
RH

IRD Data Analysis Report

Data Filename	ISOBUTRYL NITRITE 2026_March_31_135816
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	ISOBUTYL NITRITE Sigma Aldrich Lot#MKCL3264 RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 14:06:35 2026
Date Processed	Tue Apr 07 15:06:28 2026



IRDATA.SPC: 1.78 minutes: AVE (1.75: 1.83) Ref. (0.27: 0.85) of ISOBUTYL NITRITE
2026_March_31_135816.cgm

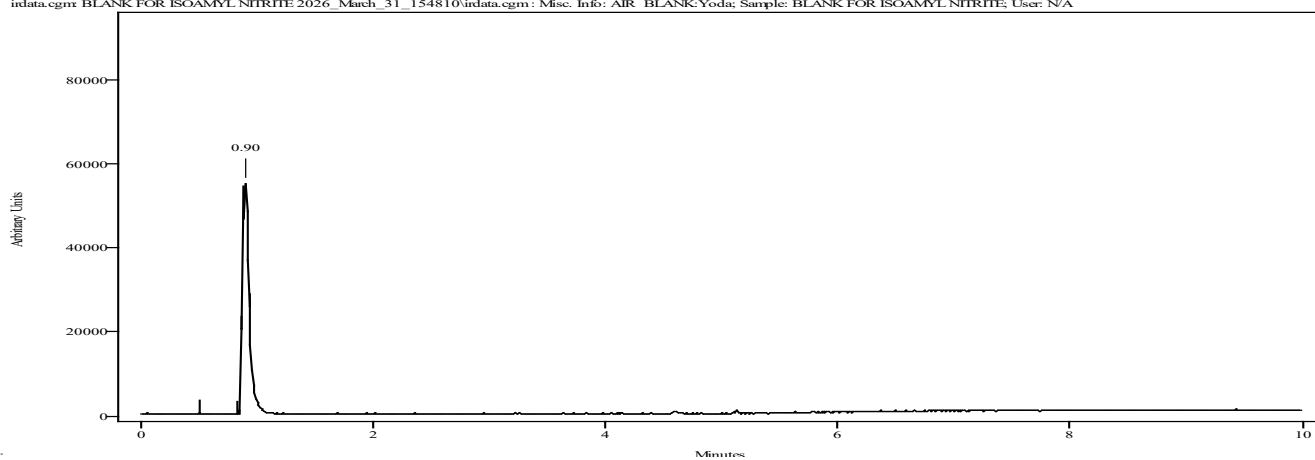


RH

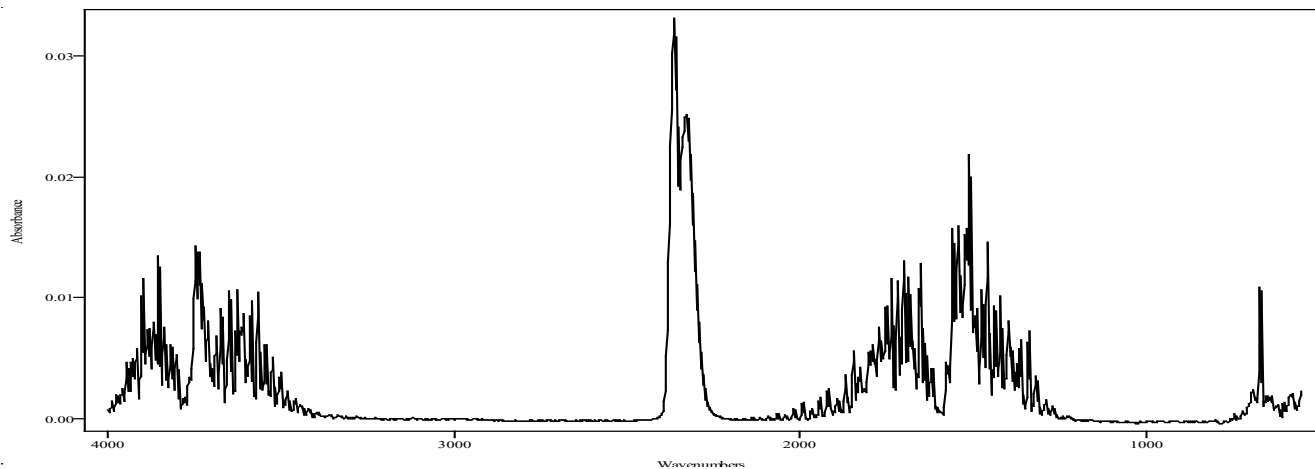
IRD Data Analysis Report

Data Filename	BLANK FOR ISOAMYL NITRITE 2026_March_31_154810
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	BLANK FOR ISOAMYL NITRITE
Misc. Info	AIR BLANK (Headspace):Yoda
User ID	N/A
Injection Time	Tue Mar 31 15:54:06 2026
Date Processed	Tue Apr 07 12:47:09 2026

irdata.cgm: BLANK FOR ISOAMYL NITRITE 2026_March_31_154810; Misc. Info: AIR BLANK:Yoda; Sample: BLANK FOR ISOAMYL NITRITE; User: N/A



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.870	0.939	0.069	0.515	0.836

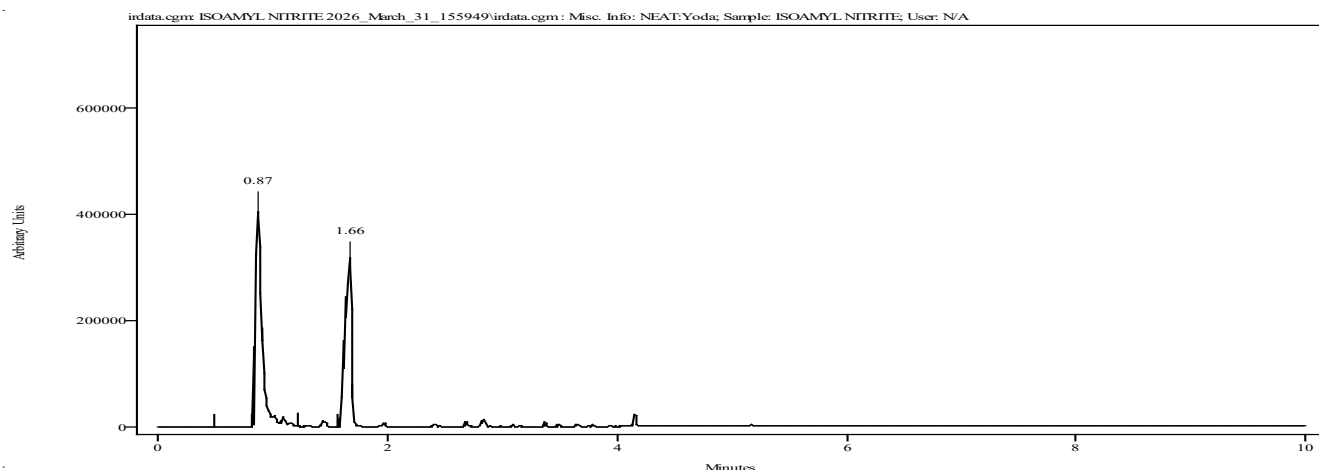


IRDATA.SPC: 0.90 minutes: AVE (0.87: 0.94) Ref. (0.52: 0.84) of BLANK FOR ISOAMYL NITRITE
2026_March_31_154810.cgm

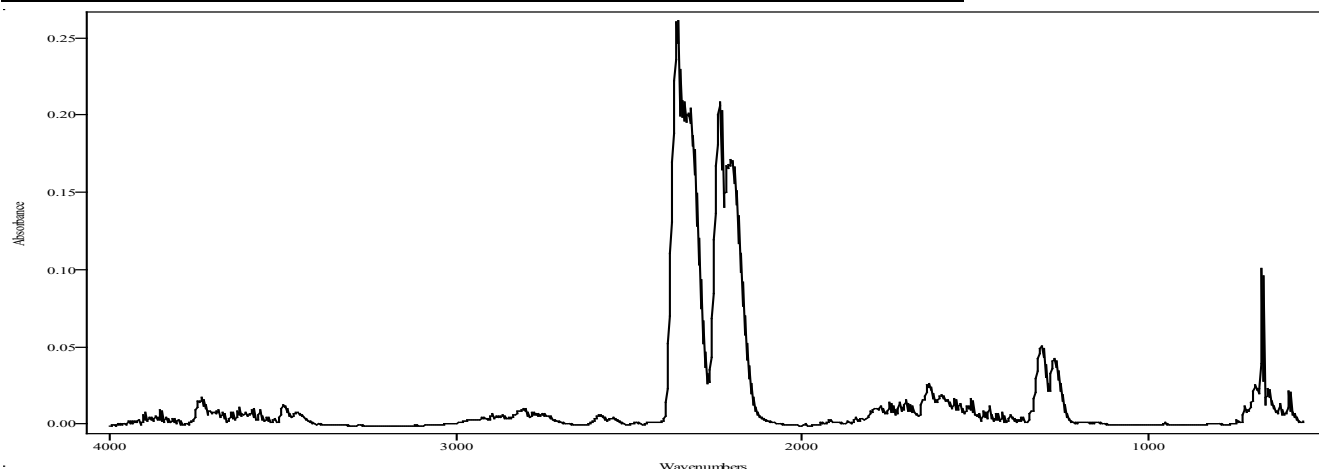
RH

IRD Data Analysis Report

Data Filename	ISOAMYL NITRITE 2026_March_31_155949
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	ISOAMYL NITRITE Sigma Aldrich Lot# 09829LC RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 16:07:34 2026
Date Processed	Tue Apr 07 14:42:32 2026



Peak Start	Peak End	Peak Width	Reference Start	Reference End
0.836	0.903	0.067	0.491	0.803
1.606	1.695	0.089	1.204	1.561

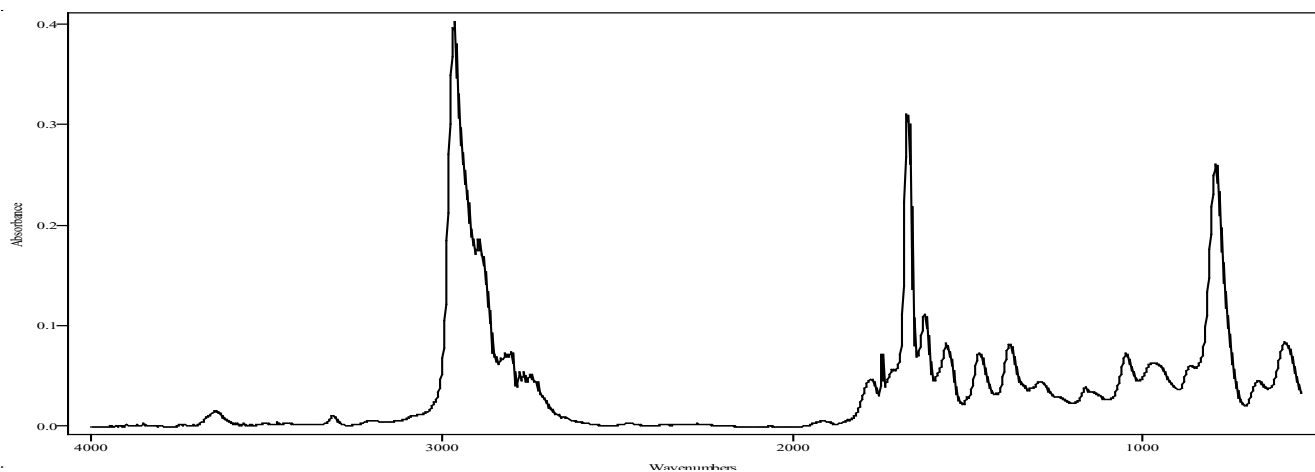


IRDATA.SPC: 0.87 minutes: AVE (0.84: 0.90) Ref. (0.49: 0.80) of ISOAMYL NITRITE
2026_March_31_155949.cgm

RH

IRD Data Analysis Report

Data Filename	ISOAMYL NITRITE 2026_March_31_155949
Data Path	H:/Forensic Chemistry/Instruments/GCIR/Yoda/YODA 2026/03-31-26
IRD Method	IRD3 EXT
GC Method	MAN60TO270
GC Vial	1
Sample Name	ISOAMYL NITRITE Sigma Aldrich Lot# 09829LC RH
Misc. Info	Headspace:Yoda
User ID	N/A
Injection Time	Tue Mar 31 16:07:34 2026
Date Processed	Tue Apr 07 14:42:32 2026



IRDATA.SPC: 1.66 minutes: AVE (1.61: 1.69) Ref. (1.20: 1.56) of ISOAMYL NITRITE
2026_March_31_155949.cgm

