FTIR Application – Practical Issues

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FTIR used at WBRC

(Woodland Biomass Research Center)

Model MKS Multigas 2030

Purchased in 2009 ($60,000)

1-day onsite training by MKS representative

1-week training at MKS was never used due to travel constraints, but would be highly recommended.

Used for:

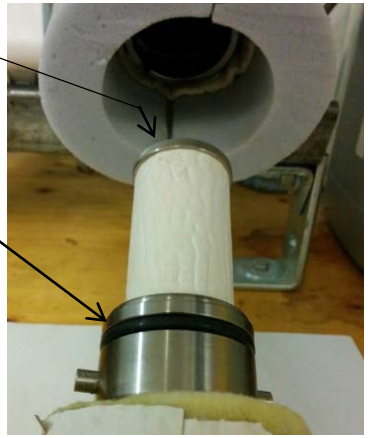
- Producer gas (wet and dry)
- Combustion exhaust
- Tar reforming experiment
- Small projects such as test gas generator, leak check, dilution system, …

FTIR Maintenance

Both dewars lost vacuum (internal and extended dewar)

Mirror contaminated (aerosols from tars or impingers)

Remedy: Use aerosol filter upstream, heated to 50C below FTIR temperature. (Installed two FEP coated viton o-rings.)



Routine overhaul

Quote from MKS:

Repair Required:

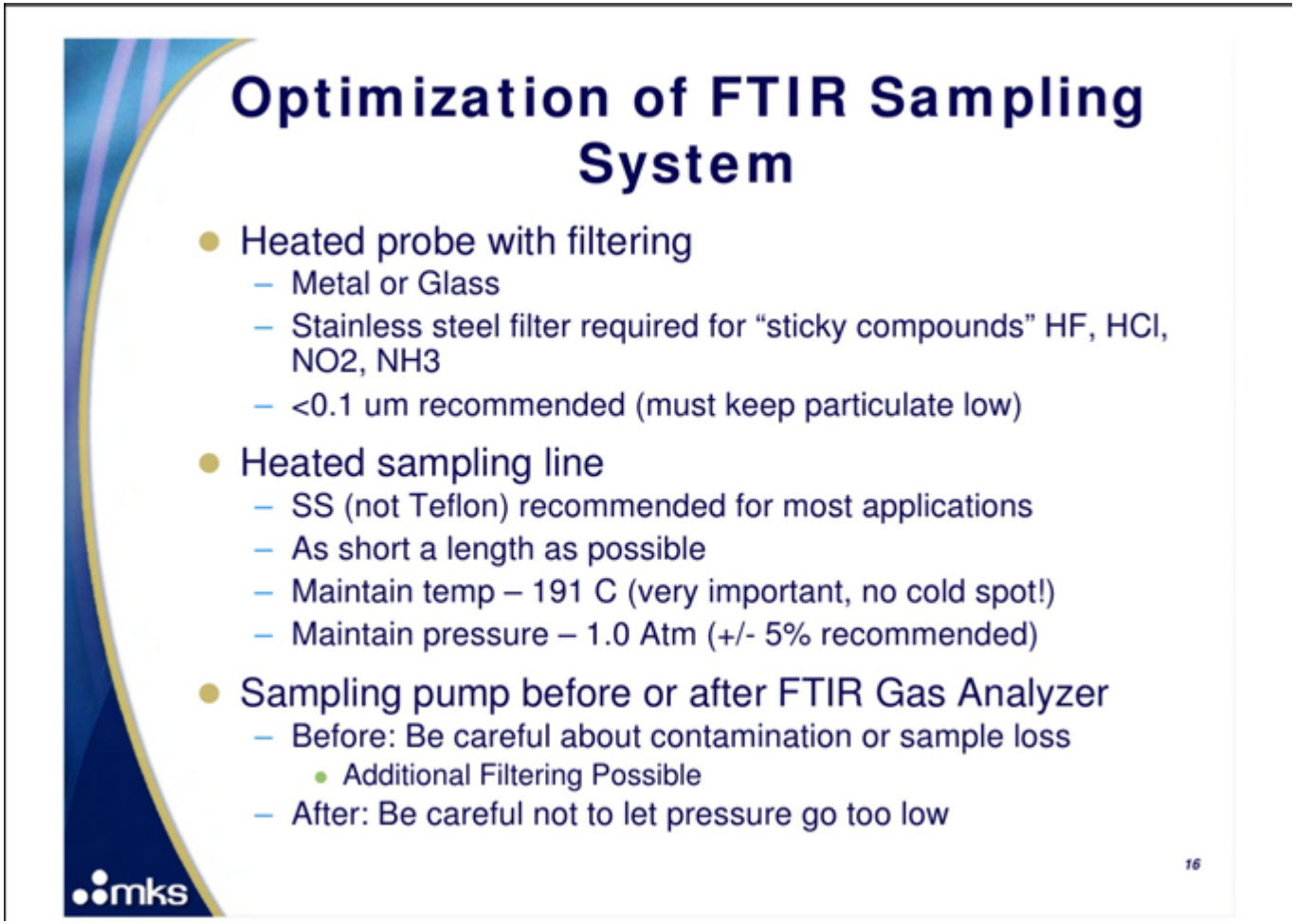
Proposed Action to Resolve Primary Failure: Replace LN2 detector(exchange program).
Replace Hene laser and realign modulator.
Replace IR source assembly(Refurbish).
Replace gas cell windows, O-rings(Kalrez), clean mirrors and rebuild gas cell

Proposed Action to Resolve Secondary Failure: Replace air filter.

Estimate Charges Breakdown:

Charge Type:	Price:
Material	$7,576.00
Total:	$7,576.00

Some Useful Suggestions from MKS



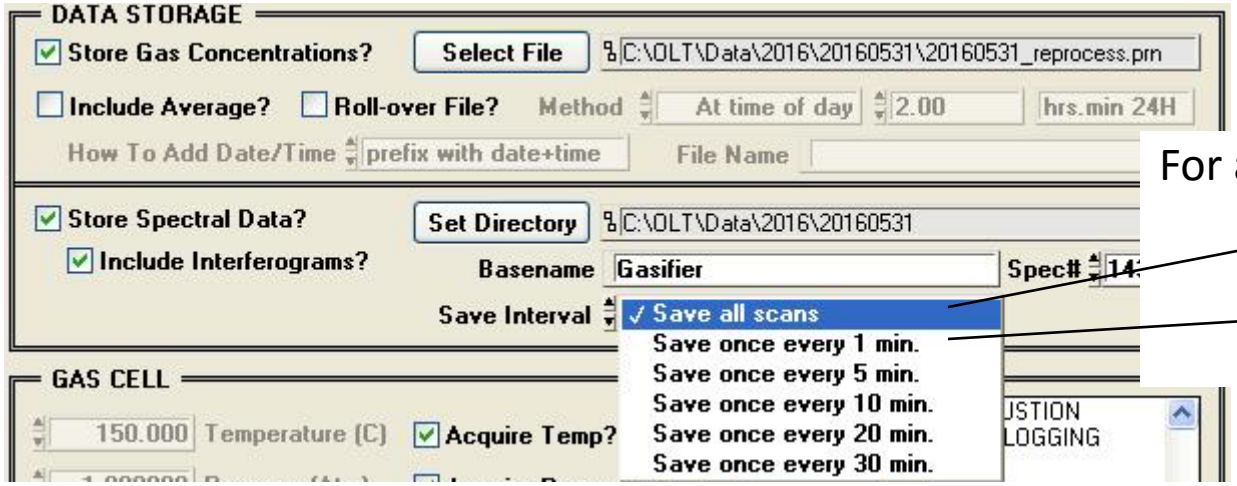
http://www.slideshare.net/jimbelanger33/ftir-for-stack-and-cem

FTIR operation

Two main stages:

1. Collecting absorption spectra (via broadband source, interferometer, mono-wavelength laser, Fourier transform,…). Once per second.
2. Calculation of gas composition based on the absorption spectra.

Step 2 is performed online, but can be repeated offline (reprocessing), as long as spectra are recorded.



For a 100 h measuring campaign:

→ 360,000 files = 120 GB

→ 6,000 files = 2 GB

For research, best to record all spectra and delete 9 of every 10 files later if not needed.

FTIR – Easy to use?

Black Box Aspect

- Canned pre-validated method – Load and GO!

http://cemteks.com/images/FTIR_Technology_Overview_Peter_Zemek_MKS.pdf

- Simultaneous analysis and display of more than 30 gases,
- 10-100 ppb sensitivity for many toxic gases without moisture removal,
- Easily transportable from site to site, with set up time in minutes,
- Permanent calibrations eliminates the need for costly gas cylinders,
- Patented, linearized detector response assures all instruments maintain the same calibration,
- User-friendly software for simple operation with minimal training.

http://www.mksinst.com/product/Product.aspx?ProductID=180

Use calibrations delivered with instrument (based on physical constants, function of temperature, pressure, mole fraction, optical path length). Learning effort: a few days.

Create Own Calibrations

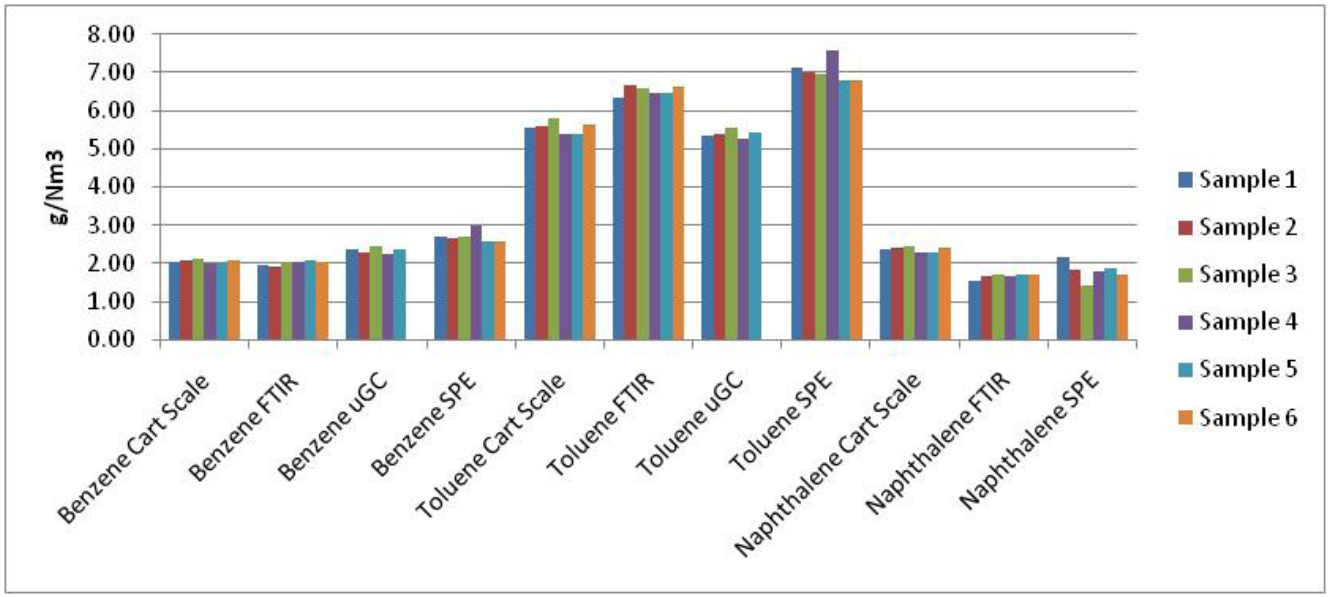
Needed for complex matrices (many compounds), or when small amounts of low-absorbing compounds are mixed with large amounts of high-absorbing compounds. Learning effort: several months.

Good Results with Simple Mixtures

Combustor exhaust

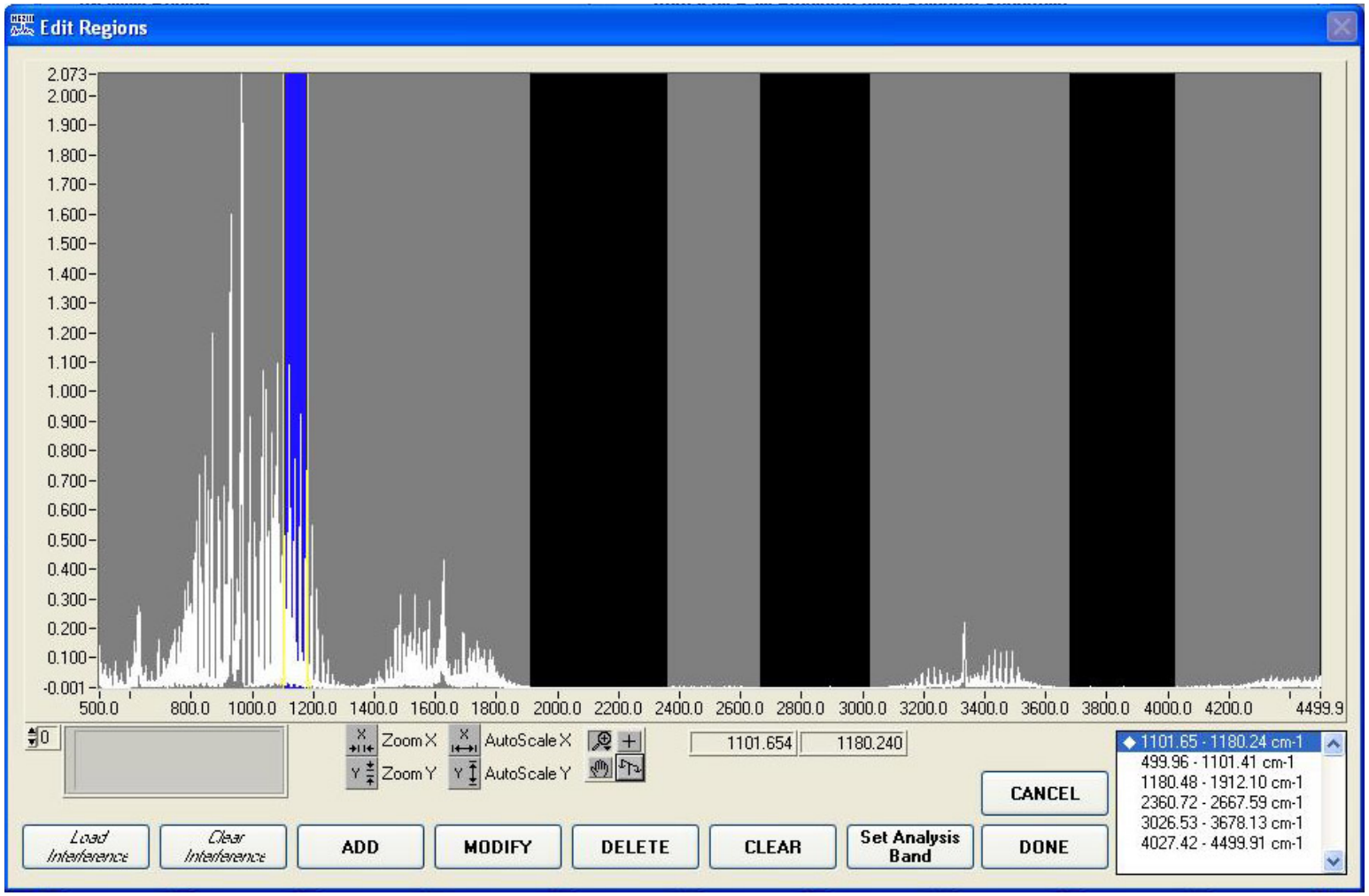
Compound		Mole fraction	
		GC	FTIR
Hydrogen	[%]	0.1%	-
Oxygen	[%]	6.3%	-
Nitrogen	[%]	59.6%	-
Carbon Dioxide	[%]	10.5%	10.7%
Water	[%]	23.3%	22.4%
Carbon Monoxide	[ppm]	2954	2915
Methane	[ppm]	894	851

Synthetic tar mixture



Example: Calibration of NH_3

900ppm NH_3



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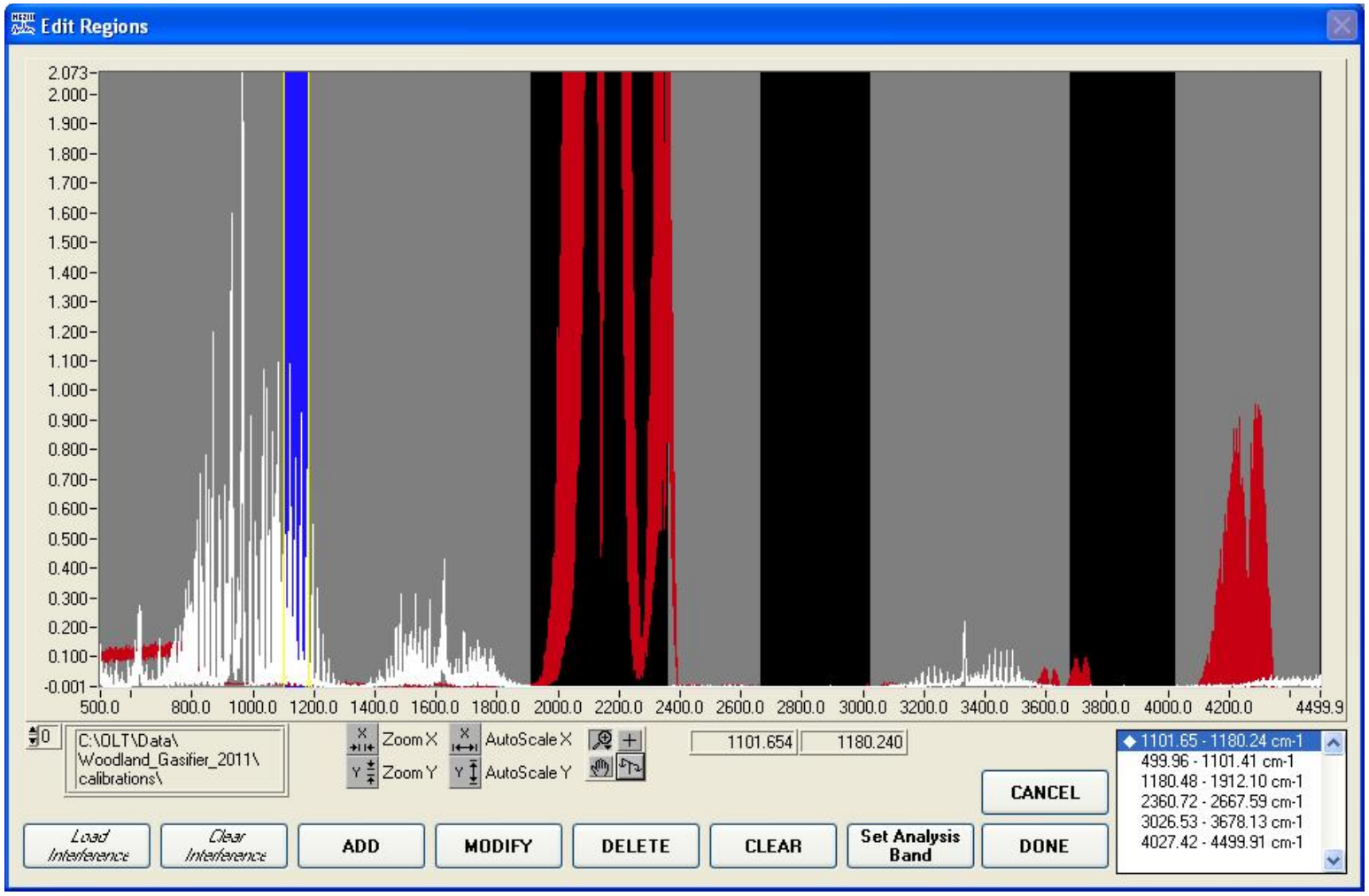
Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO (red)



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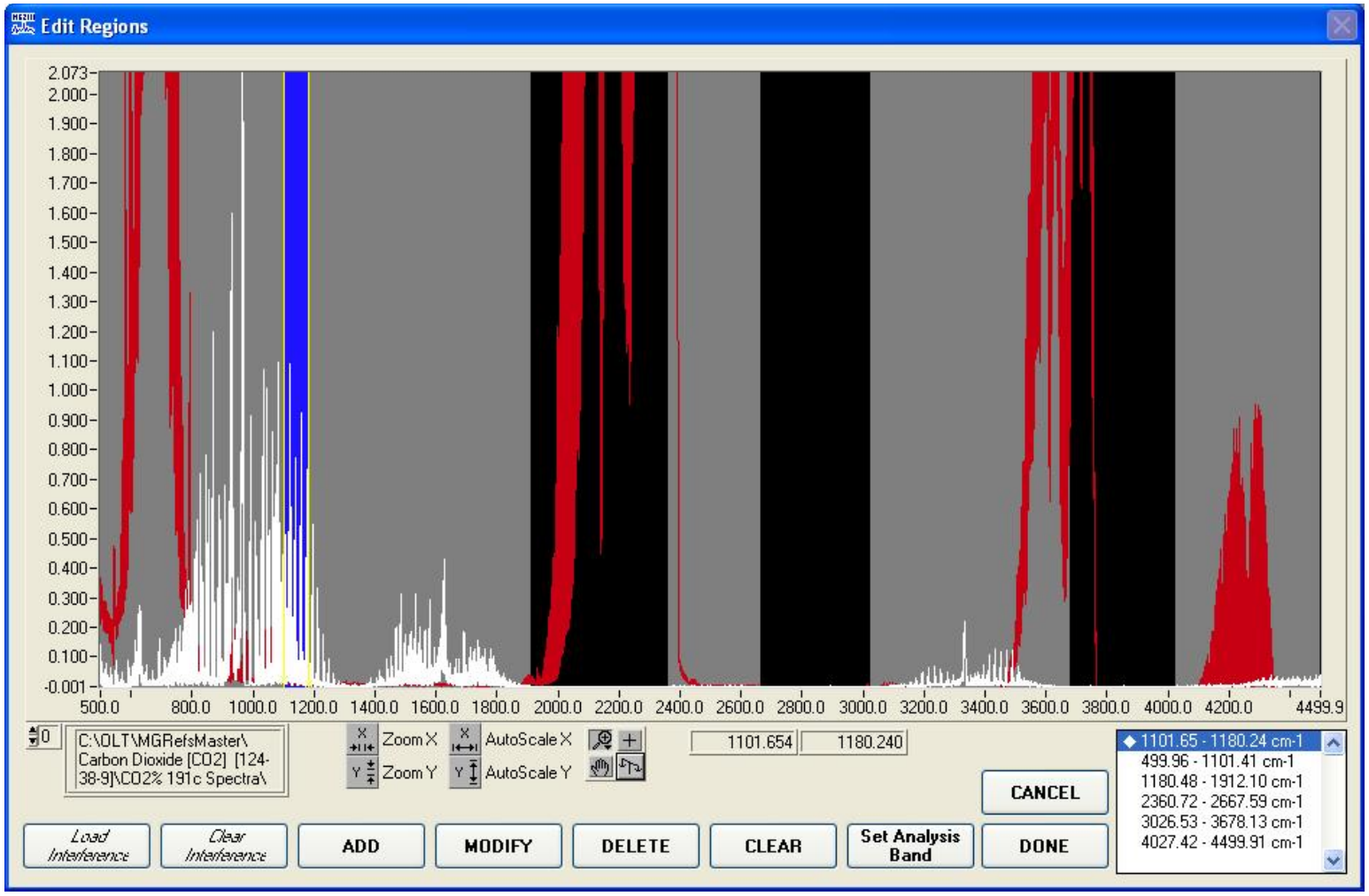
Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO + 18% CO_2 (red)



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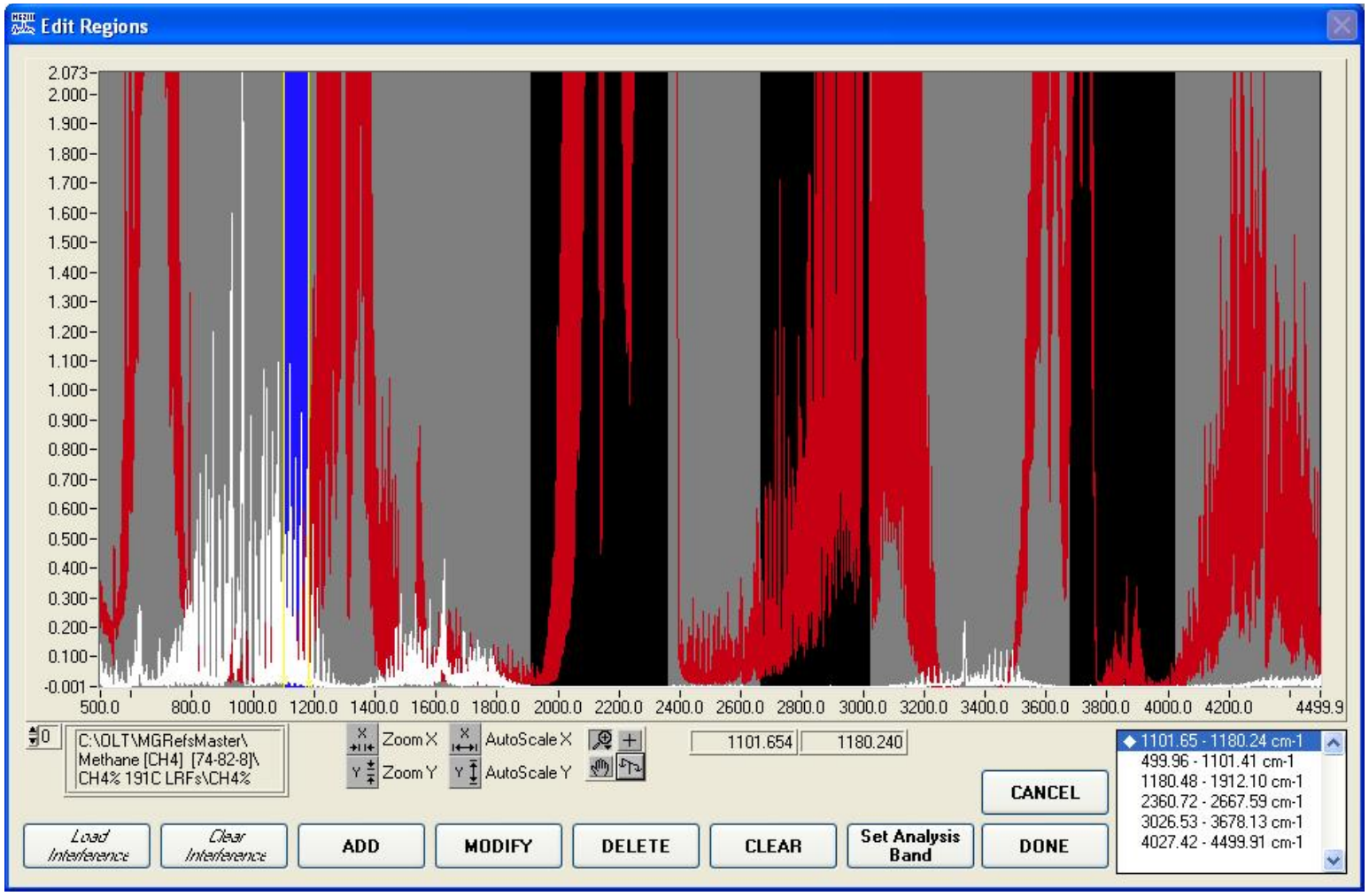
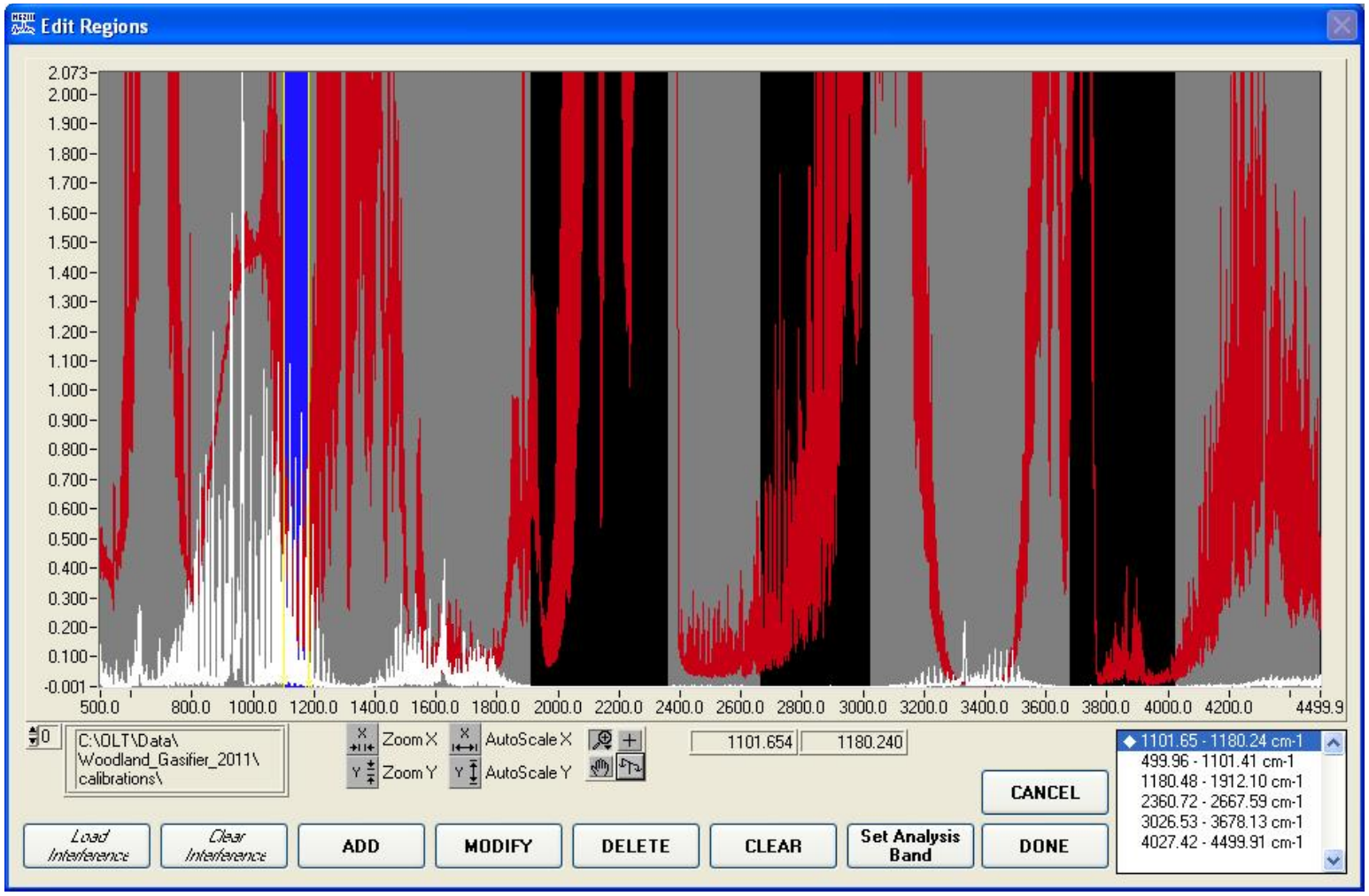
Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO + 18% CO_2 + 9% CH_4 (red)



Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO + 18% CO_2 + 9% CH_4 + 3% C_2H_4 (red)



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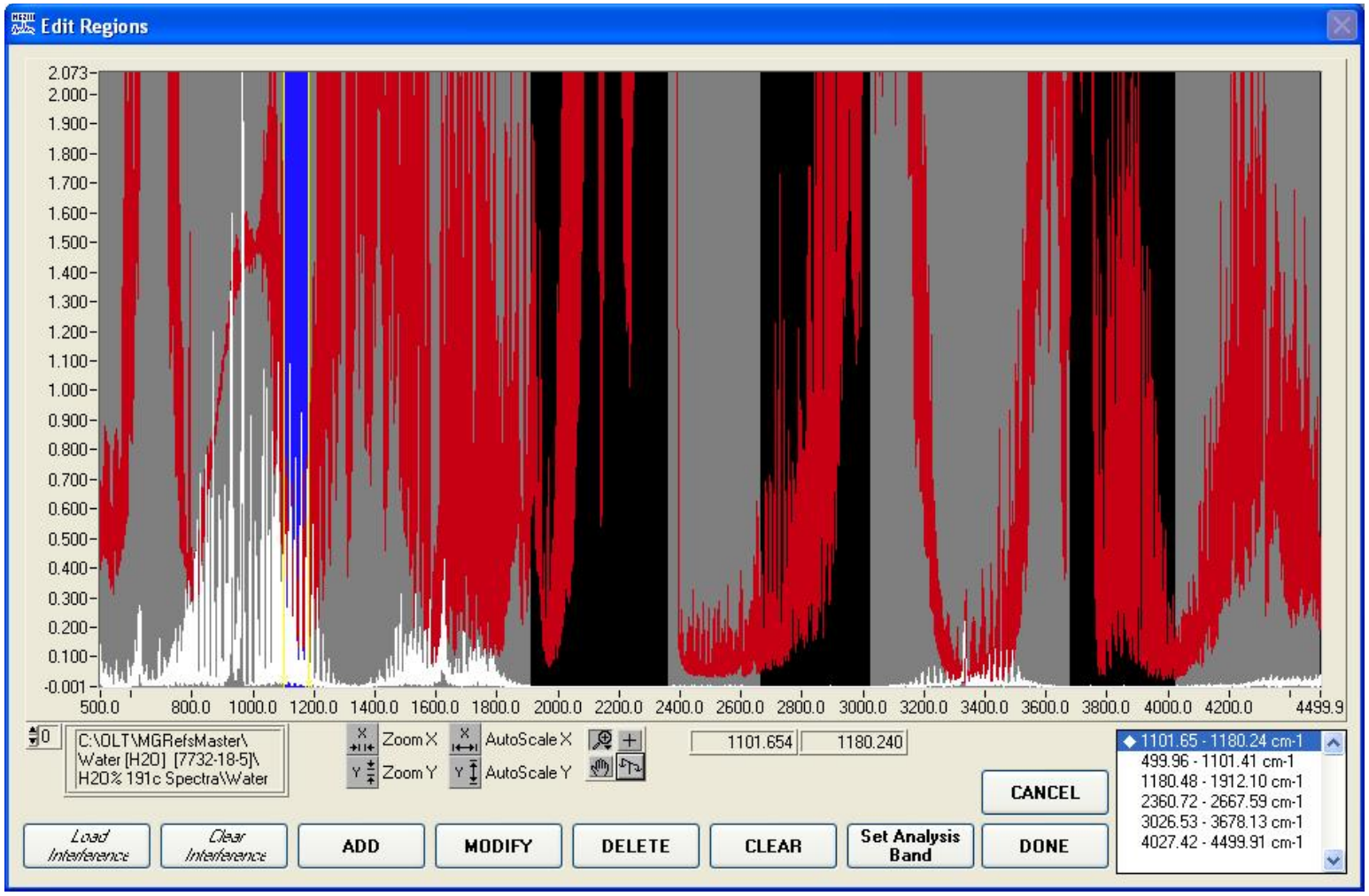
Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO + 18% CO_2 + 9% CH_4 + 3% C_2H_4 + 2% H_2O (red)



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Calibration of a new compound

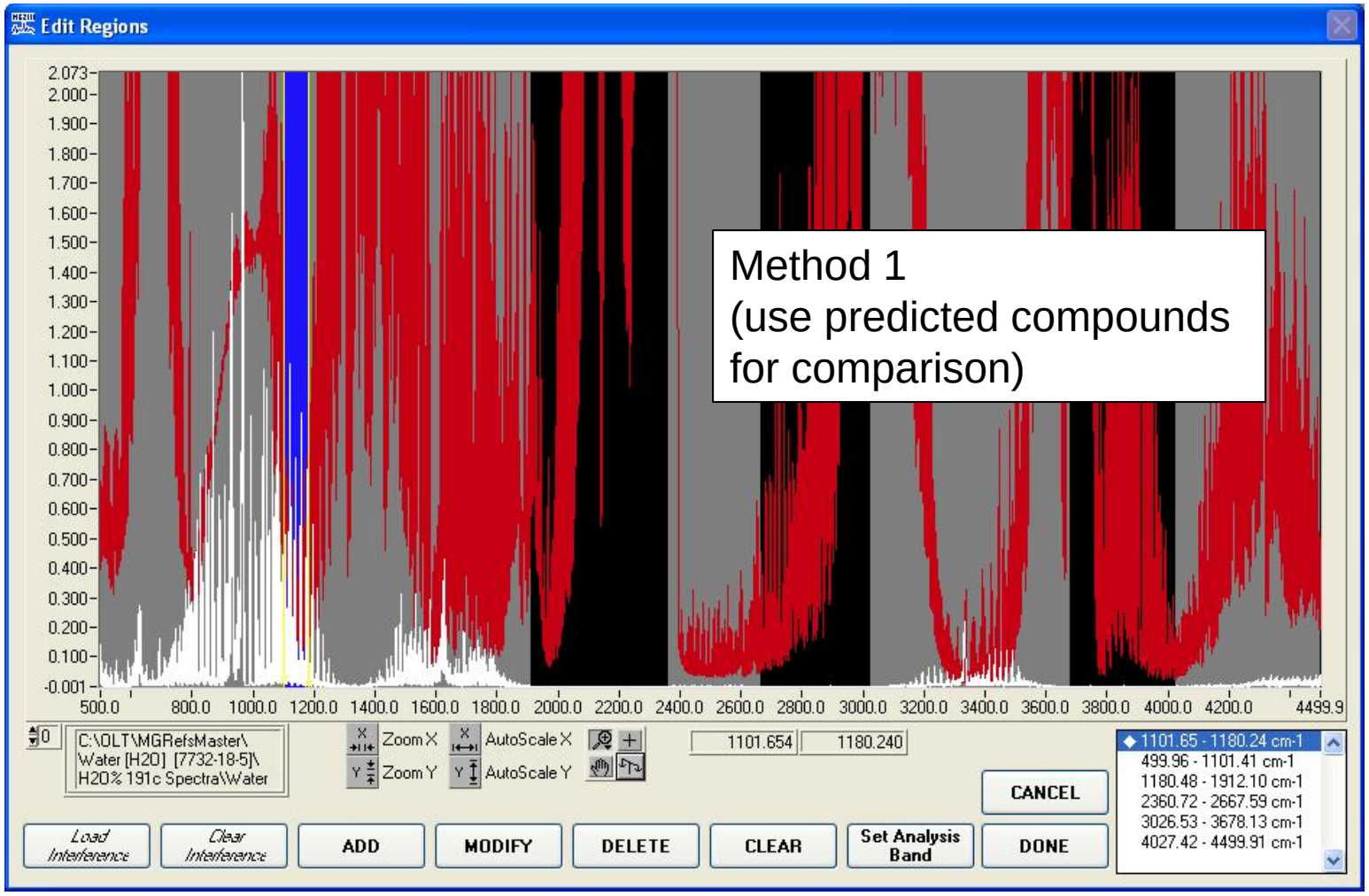
Find absorption region that is most unique to compound being calibrated ("primary region" or "analysis band" in MKS software).

Method 1: Judge against sum of spectra of expected other/all compounds.

Method 2: Judge against actual, recorded, spectrum of the to be measured gas mixture.

Example: Calibration of NH_3

900ppm NH_3 (white), 23% CO + 18% CO_2 + 9% CH_4 + 3% C_2H_4 + 2% H_2O (red)



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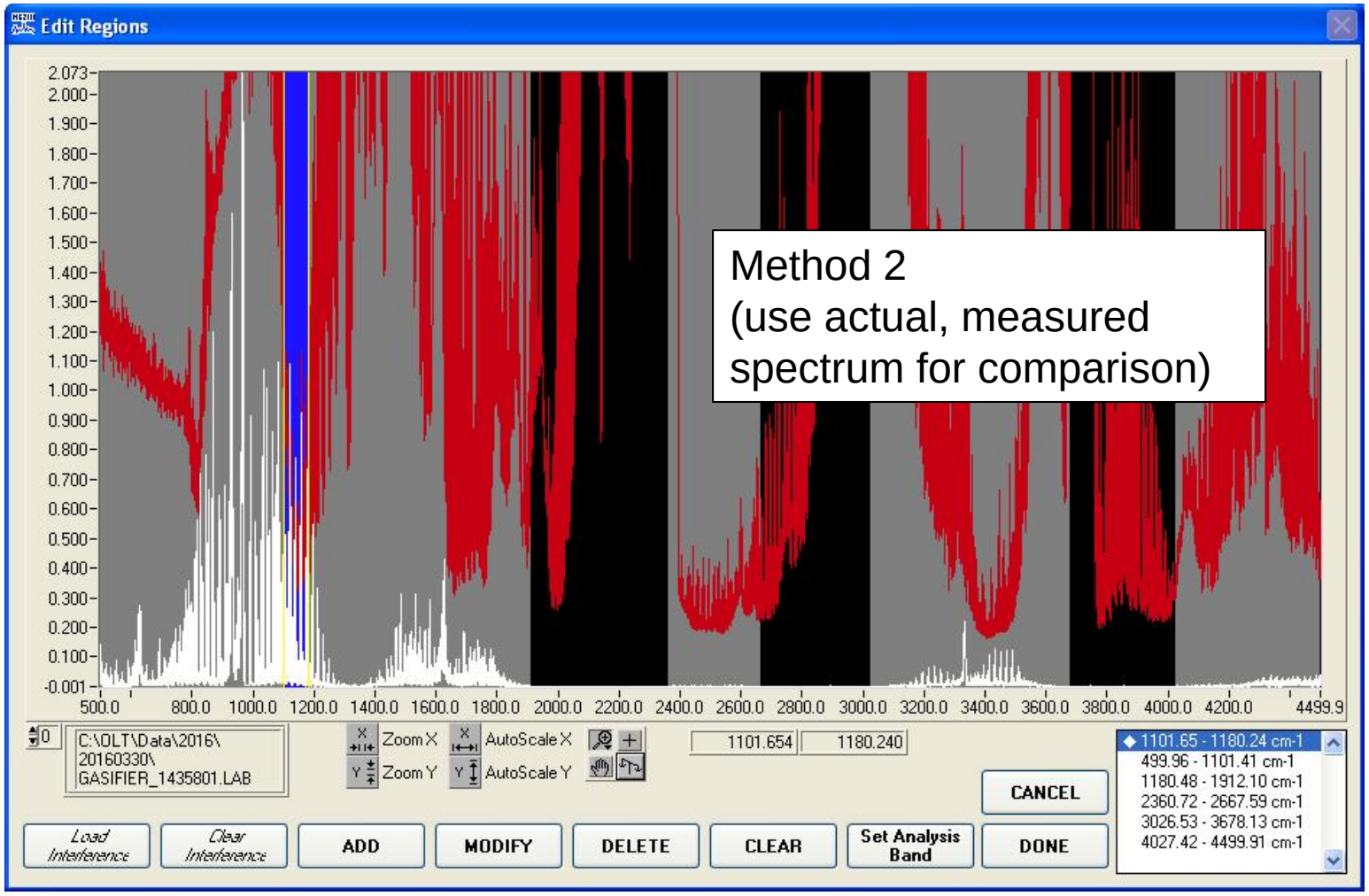
Example: Calibration of NH_3

900ppm NH_3 (white), Real producer gas, dried (red)



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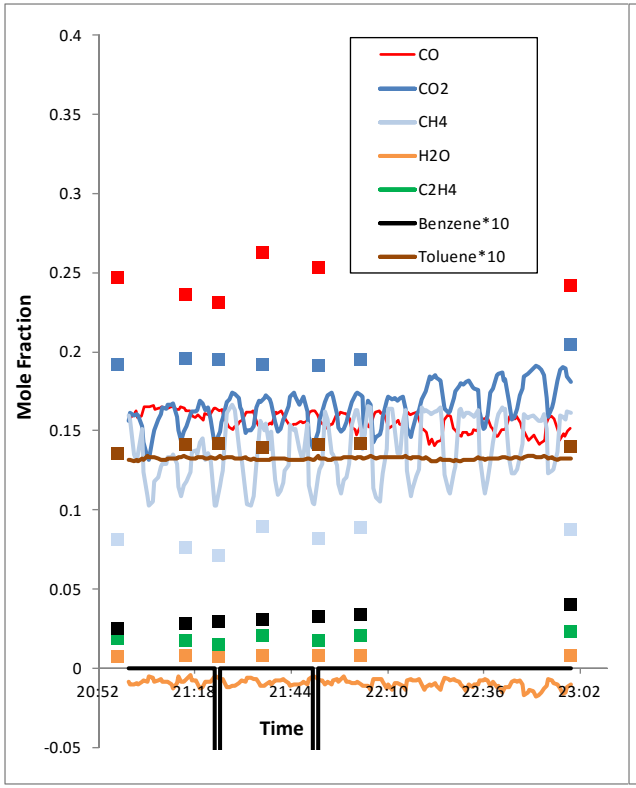
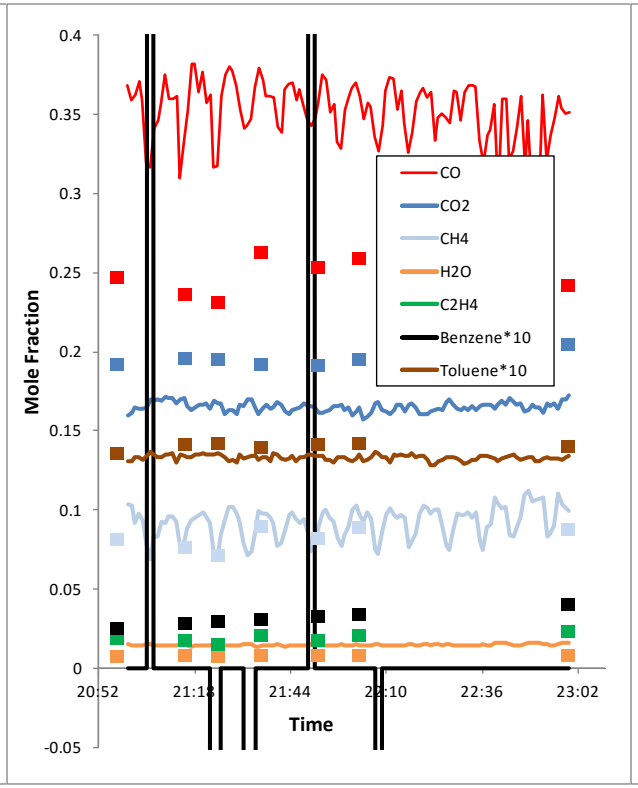
Results of Different Methods

Producer gas from gasifier (dried through toluene impinger)

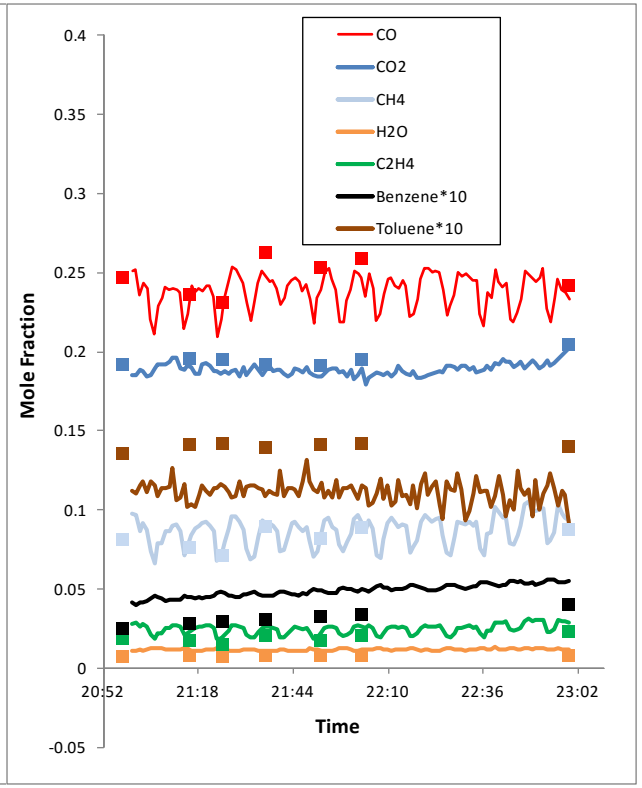
Symbols are measurements using a Micro GC, lines are the results of the FTIR using different calibrations.



Original Calibrations
(some compounds were out of calibration range)



Improved Calibrations
(using Method 1)



Further Improved Calibrations
(using Method 2)

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Some Useful Links on the Theory of Operation

http://chemistry.oregonstate.edu/courses/ch361-464/ch362/irinstrs.htm

http://mmrc.caltech.edu/FTIR/FTIRintro.pdf

http://www.physics.nus.edu.sg/~L3000/Level3manuals/FTIR.pdf

http://www.patarnott.com/atms360/pptATMS360/FTIRSpectrometer.ppt

Conclusion

FTIR is a useful tool for monitoring processes (steadyness).

For mixtures of a few components, precise measurements are possible.

For complex mixtures, such as real producer gas, using FTIR as measurement tool is challenging:

- High amounts of CO_2 and H_2O
- All IR-absorbing components need to be known to include their calibrations .
- The calibrations need to be adjusted to match expected ranges of compounds as well as interfering compounds (which absorption bands to use).