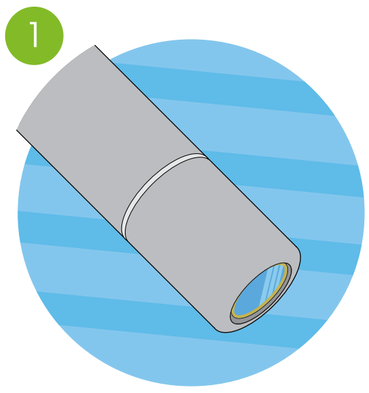
Guide to Inline Monitoring of Reaction Mechanisms

METTLER TOLEDO

www.mt.com/ReactIR

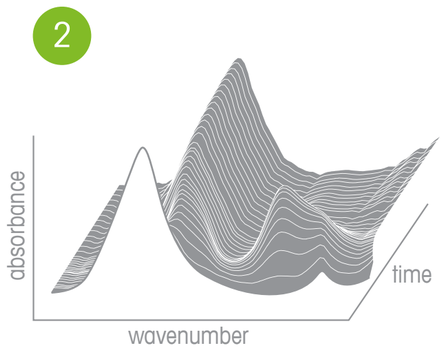
ReactIR Method of Measurement

1



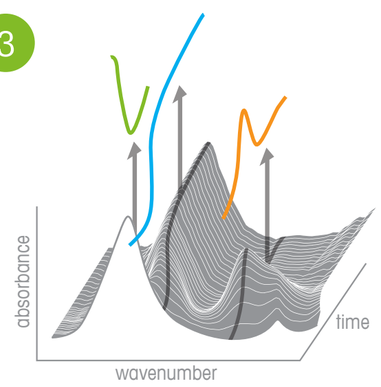
The Sensor: ReactIR™ Comp™ sampling technology places the sensor directly into the reaction for continuous, real-time monitoring

2



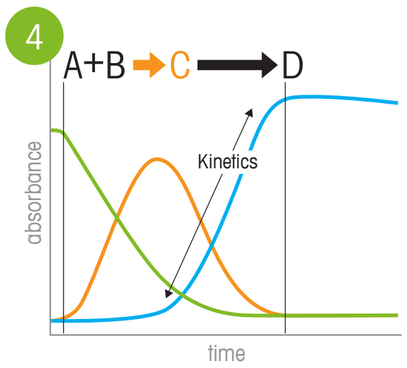
The Measurement: Spectra are continuously collected and presented in a water fall plot - absorbance vs. wavenumber vs. time

3



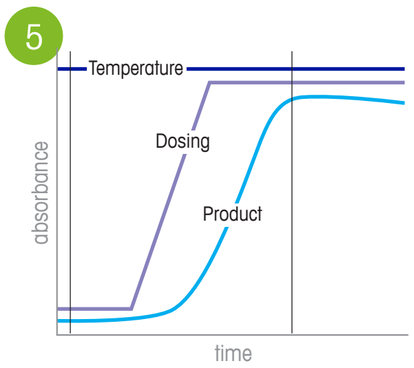
The Peaks: Use iC IR™ software to identify isolated peaks that change over time. 'Profiling' those peaks shows how they change over time. Consider using the Find Trends function.

4



The Trends: Beer's Law tells us that absorbance is proportional to concentration resulting in reaction profiles which show key reaction events - start, stop, intermediate formation, and kinetics

5



The Chemistry: By studying how chemistry changes as process conditions are varied, scientists can determine which process parameters will deliver optimized reactions

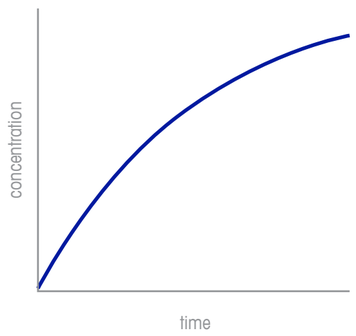
The Molecule Must be Infrared Active

The Compound is in Solution

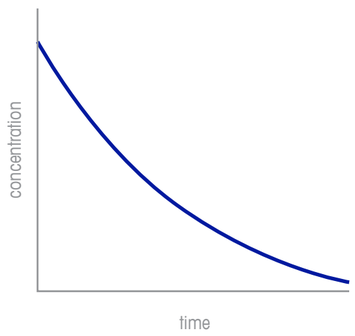
The Concentration is Greater than 0.1 %

Tracking Common Reaction Mechanisms

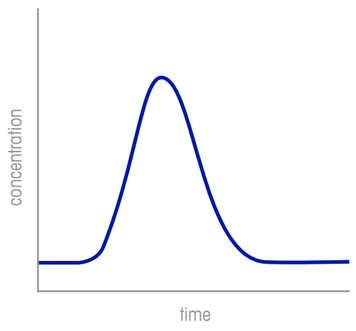
Product Formation / Dissolution



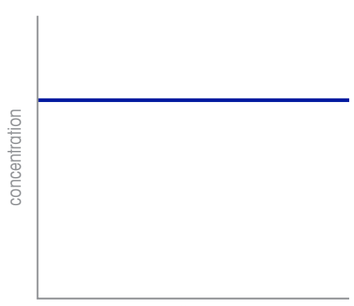
Material Consumption / Crystallization



Reaction Intermediate



Flow - Steady State



Hints and Tips

Additional Parameters
Integrate stirring, temperature dosing, and pH data to see how parameters impact the reaction

Correct Location
Avoid poorly mixed regions of the vessel or pipeline to ensure a representative measurement

Compare with Offline Methods
Use traditional offline methods like HPLC to turn infrared trends from absorbance into quantified trends

Find Trends
Use the new Find Trends feature to quickly search the data to identify the best peaks to trend

Clean Data
Make sure you get the best data by following the probe cleaning methods

Collect References
To aid analysis, collect or calculate reference spectra whenever possible

Collect Background
Always collect a new background for every experiment to obtain the most accurate data

Contact Information

Our TAC _____

Our Account Manager _____

Our Service Rep _____

Other _____

Correlation Table

ReactIR collects data in the mid-infrared spectral region, which provide a characteristic fingerprint absorbance that is associated with fundamental vibrations in the molecules of interest. Once a peak is profiled, embedded correlation tables in iC IR help you associate each trend with the constituents in the reaction.

● Strong; ● Medium; ● Weak

3400 to 3000 cm^{-1}

3410-3390	●	N-H	Amine, aryl secondary (aryl-NH-R)
3420-3380	●	N-H	Amine, aryl primary (aryl-NH_2)
3400-3370	●	O-H	Alcohol, tertiary (R_3-COH)
3430-3370	●	O-H	Alcohol, phenol (aryl-OH)
3400-3350	●	N-H	Amine, primary (R-NH_2)
3360-3340	●	O-H	Alcohol, secondary (alkyl-CHOH-alkyl)
3370-3340	●	N-H	Amide, primary (R-CO-NH_2)
3350-3320	●	O-H	Alcohol, primary (R-CH_2-OH)
3350-3320	●	N-H	Amine, secondary (R-NH)
3320-3280	●	N-H	Amide, secondary (R-CO-NH-R)
3340-3270	●	C≡C-H	Acetylene (R-C≡C-H)
3260-3220	●	N-H	Amide, lactam, five membered ring
3220-3180	●	N-H	Amide, lactam, four membered ring

2750 to 1900 cm^{-1}

2750-2710	●	C-H	Aldehyde (R-CO-H), aliphatic
2590-2540	●	S-H	Thiol (R-SH)
2275-2265	●	N=C=O	Isocyanate (R-N=C=O)
2255-2245	●	C≡N	Cyanate (R-C-O-C≡N)
2260-2240	●	C≡N	Nitrile (R-C≡N)
2225-2210	●	N-C≡N	Cyanamide (R-N-C≡N)
2170-2150	●	C≡N	Thiocyanate (R-S-C≡N)
2135-2110	●	C=N=N	Diazo (R-C=N=N)
2180-2110	●	N=N=N	Azide (R-N=N=N)
2140-2100	●	C≡C	Acetylene (R-C≡C-H)
2165-2100	●	C=C=O	Ketene (R-C=C=O)
2110-2090	●	N=C=S	Isothiocyanate (R-N=C=S/broad)
2000-1900	●	C=C=CH_2	Allene (C=C=CH_2)

1870 to 900 cm^{-1}

1870-1835	●	C=O	Anhydride (R-CO-O-CO-R), cyclic
1835-1815	●	C=O	Anhydride (R-CO-O-CO-R), noncyclic
1810-1795	●	C=O	Acid Chloride (R-CO-Cl), aliphatic
1800-1775	●	C=O	Anhydride (R-CO-O-CO-R), cyclic
1785-1765	●	C=O	Acid Chloride (R-CO-Cl), aromatic
1790-1760	●	C=O	Amide, lactam, four membered ring
1795-1760	●	C=O	Ester (RCOOR), lactone
1765-1745	●	C=O	Anhydride (R-CO-O-CO-R), noncyclic
1750-1740	●	C=O	Ketone (RCOR), cyclic
1750-1735	●	C=O	Acid Chloride (R-CO-Cl), aromatic
1750-1735	●	C=O	Ester (RCOOR), noncyclic
1740-1720	●	C=O	Aldehyde (R-CO-H), aliphatic
1725-1705	●	C=O	Ketone (RCOR), dialkyl
1710-1685	●	C=O	Aldehyde (R-CO-H), aromatic
1720-1680	●	C=O	Carboxylic Acid (RCOOH), Aliphatic
1700-1670	●	C=O	Carboxylic Acid (RCOOH), Unsaturated/Aromatic
1700-1670	●	C=O	Ketone (RCOR), singly conjugated
1720-1670	●	C=O	Amide, lactam, five membered ring
1685-1645	●	C=O	Amide, primary (R-CO-NH_2)

1870 to 900 cm^{-1} (cont.)

1661-1640	●	C=CH_2	Vinylidene (R-C=CH_2)
1680-1640	●	C=O	Ketone (RCOR), doubly conjugated
1645-1635	●	O-H	Water
1662-1630	●	C=C-H	Olefin cis (R-C=C-R)
1670-1630	●	C=O	Amide, secondary (R-CO-NH-R)
1670-1630	●	C=O	Amide, tertiary (R-CO-NR_2)
1670-1630	●	C=N	Imine (R-CH=N-R)
1620-1590	●	N-H	Amine, primary (R-NH_2)
1620-1590	●	N-H	Amine, aryl primary (aryl-NH_2)
1640-1580	●	C=O	Ketone (RCOR), diketone enol form
1610-1570	●	C=C	Aromatic ring
1565-1535	●	R-NO_2	Nitro (R-NO_2)
1570-1530	●	C-N-H	Amide, secondary (R-CO-NH-R)
1535-1475	●	C=C	Aromatic ring
1475-1455	●	CH_2-CN	Nitrile (R-CH_2 -C≡N)
1455-1410	●	C=C	Olefin cis (R-C=C-R)
1410-1380	●	R-NO_2	Nitro (R-NO_2)
1420-1380	●	C-N	Amide, primary (R-CO-NH_2)
1400-1350	●	C-F	Fluoro compound (C-F)
1350-1250	●	P=O	Phosphates, organic (P=O)
1260-1220	●	C-O	Ether, aromatic (alkyl-O-aryl)
1225-1200	●	C-O	Ether, vinyl (R-O-C=CH_2)
1255-1185	●	C-O	Ester, aliphatic (alkyl-O-C=O)
1250-1180	●	C-O	Alcohol, phenol (aryl-OH)
1150-1120	●	C-N	Amine, Secondary (R_2 -NH)
1130-1110	●	C-O	Ether, aliphatic (alkyl-O-alkyl)
1150-1110	●	C-O	Cyanate (R-C-O-C≡N)
1200-1100	●	C-O	Alcohol, tertiary (R_3-COH)
1120-1080	●	C-O	Alcohol, secondary (alkyl-CHOH-alkyl)
1075-1045	●	C-N	Amine, Primary (R-NH_2)
1060-1030	●	C-O	Ether, aromatic (alkyl-O-aryl)
1060-1000	●	C-O	Alcohol, primary (R-CH_2-OH)
1080-1000	●	C-O	Alcohol, aromatic secondary (phenyl-CHOH)
1000-950	●	C=C-H	Olefin trans (R-C=C-R)
950-900	●	C-S	Thiol ester (R-CO-S)

894 to 600 cm^{-1}

895-885	●	=CH_2	Vinylidene (R-C=CH_2)
900-870	●	C-O-C	Ethylene oxide (C_2H_4O)
870-825	●	=CH_2	Allene (C=C=CH_2)
820-780	●	N-H	Amine, primary (R-NH_2)
825-750	●	C-H	Aromatic ring
710-680	●	C-S	Thiol (R-SH)
720-680	●	N-H	Amine, secondary (R-NH)
700-650	●	C-Cl	Acid Chloride (R-CO-Cl)
730-650	●	C=C-H	Olefin cis (R-C=C-R)
750-650	●	C-Cl	Chloro compound (C-Cl)
680-610	●	≡C-H	Acetylene (R-C≡C-H)
650-600	●	C-Br	Bromo compound (C-Br)

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