



In-Line Color Measurement of In-Process Streams

July 19, 2017 / John Caton – Tijmen Ros

HCN Industry Safety Conference – Color Monitoring and Instrumentation Panel

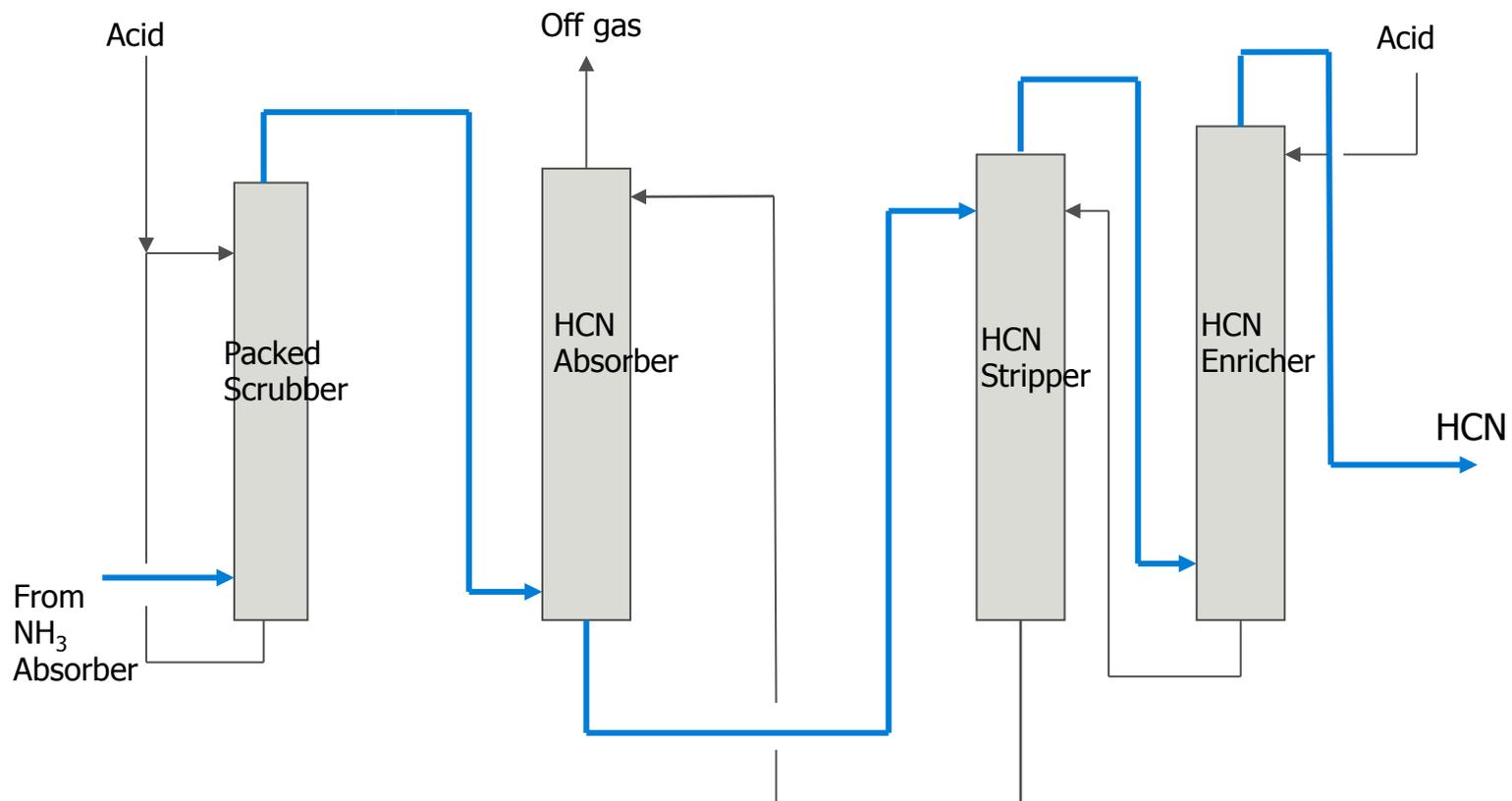


Background

- INVISTA's Victoria facility produces HCN for the production of adiponitrile
 - Steps for producing HCN are: Synthesis, NH₃ Removal, and HCN Refining
 - Acid addition is used to stabilize the HCN in the refining section
 - One of the first indicators of HCN oligomer and/or polymer formation is an off-line measurement of % transmittance in HCN Refining
 - Sampling and analysis require special procedures to assure personnel safety
 - High HCN-containing samples are a challenge
 - Sampling frequency and time-to-analyze determine the reaction time to a start of polymerization
 - Low analysis frequency makes correlation with other process parameters more challenging
- **In-line color analysis of in-process streams can be advantageous**



HCN Train

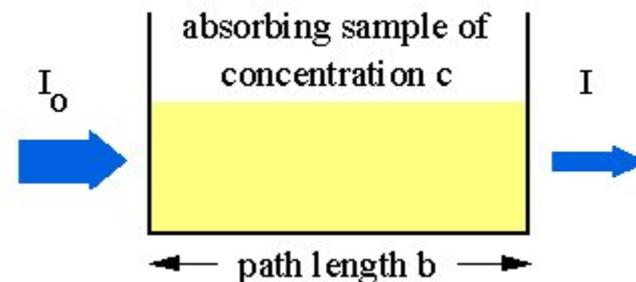




Data obtained in collaboration with the vendor

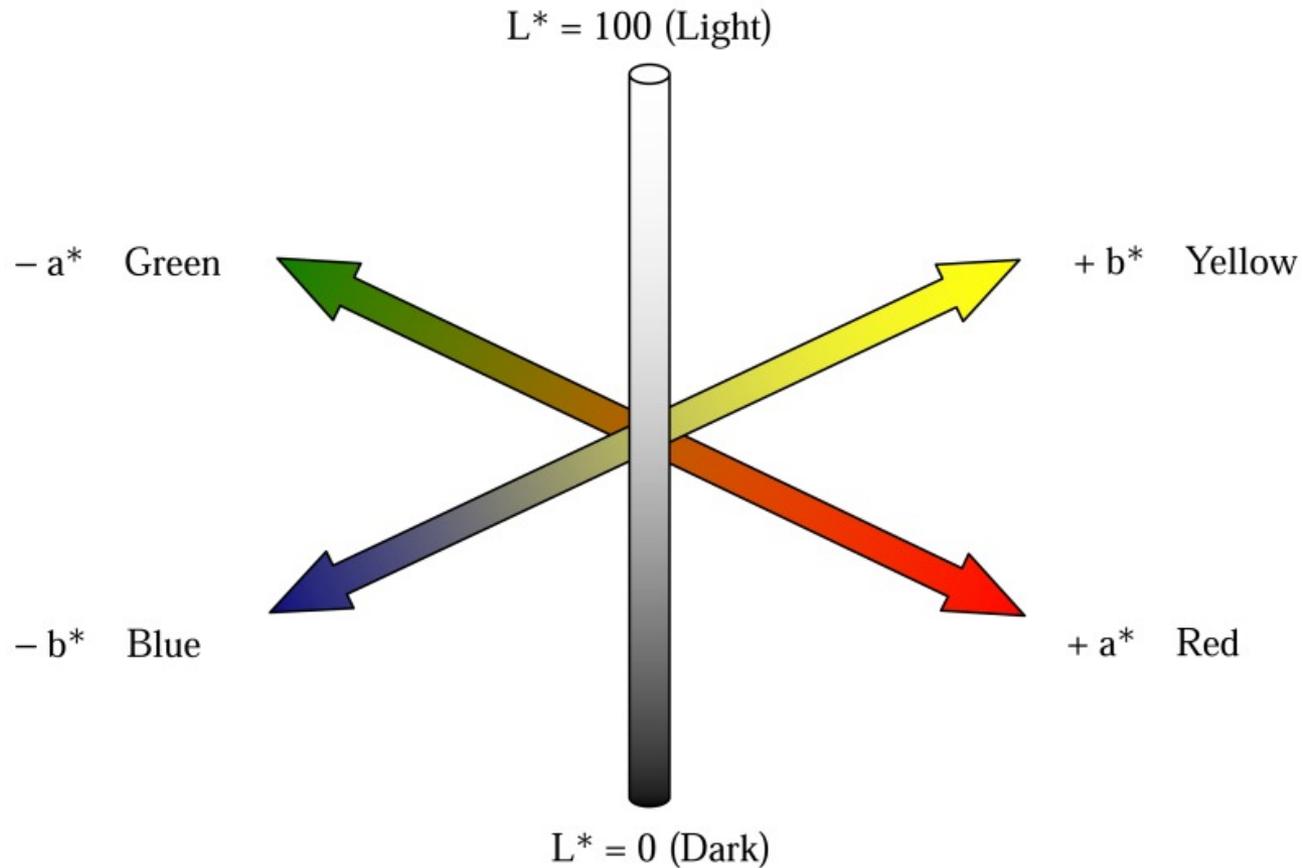
Process Analyzers – Color or Chemical

- Intensity of light absorbed by chemicals proportional to their concentration
- The spectral pattern of the absorption is characteristic of the compound and its chemical environment
- Chemical compounds of interest must absorb in the UV-Vis-NIR wavelength range (200-1000 nm)
- Probe path length determined by concentration range
- Calculate $L^*a^*b^*$ color values of the CIE color space





Simple Illustration of Color Coordinates





HCN Measurement – vendor laboratory

- Measured at an optical path length of 1-2 cm
- Instrument collects transmittance data at all wavelengths simultaneously across full instrument range (230-820 nm)
- Color values calculated ($L^*a^*b^*$ and Yellow Index)
- Individual wavelength responses can be tracked, if desired
- Polymer/oligomer formation can be detected by a change in color values and by changes in transmittance, particularly at shorter wavelengths



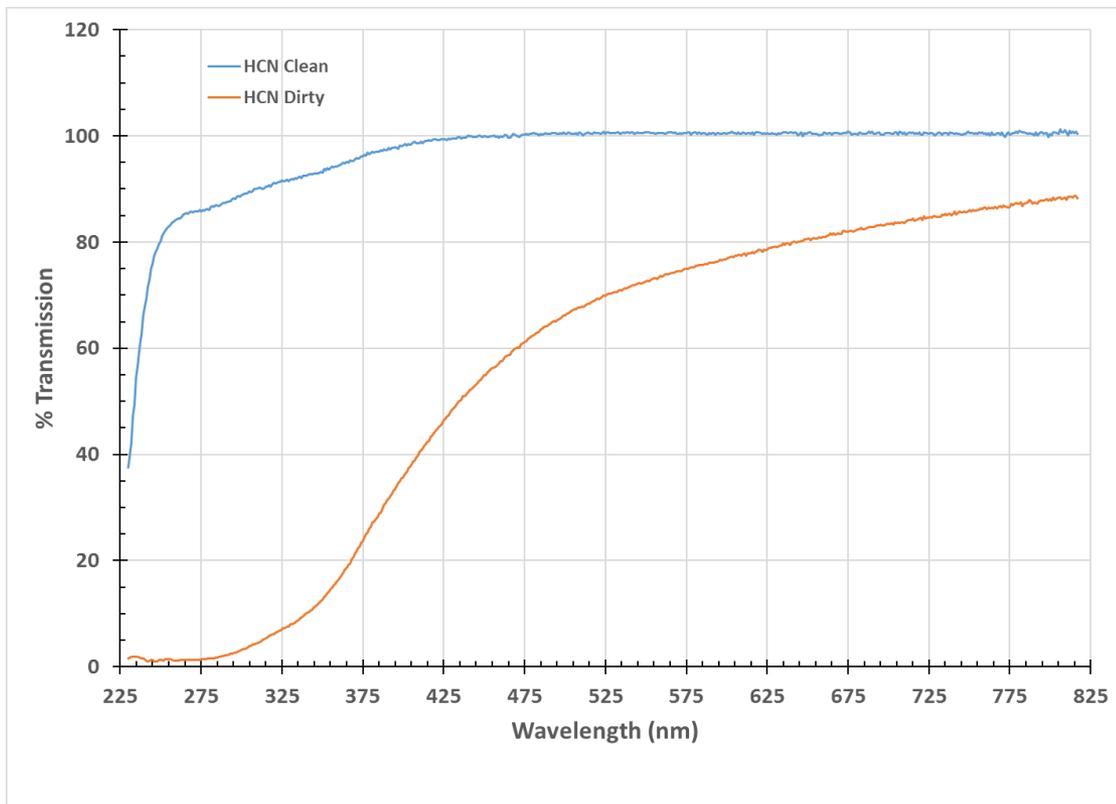
HCN Measurement – two approaches

1. Color – regard the polymer formation as a change in color of the product stream and track by calculating various color parameters
2. Concentration – regard the polymer formation as an increase in concentration of undesired species and track at specific wavelengths

Both approaches are feasible. For the data shown in this presentation approach #1 was used



HCN Samples – UV-vis Spectra



Diluted and acidified in-process HCN from INVISTA's Victoria facility, during normal operation and during a color event



HCN Measurement - Results

Sample	L*	a*	b*	YI
Water	99.96	0.01	0.01	0.05
HCN "Clean"	100.16	-0.18	0.42	0.67
HCN "Dirty"	87.89	-0.30	15.50	29.12

- Water was the optical reference material
- HCN "Clean" is slightly more "yellow" than the water
- HCN "Dirty" is substantially different in both overall transmittance (L*) and yellowness (b*, YI)
 - The color numbers are calculated using a large portion of the instrument wavelength range (360-780 nm)
 - The YI calculation uses the range of 375-450 nm and is more sensitive than b*



Online Analyzers – General Configuration

- Standard
 - NEMA 4 Enclosure with TE Heater/Cooler
 - UV-vis spectrophotometer
 - Xenon flash lamp (230-820 nm)
 - Industrial panel PC with touch screen
 - Standard operating system and Vendor Software
 - USB ports and ethernet port with OPC

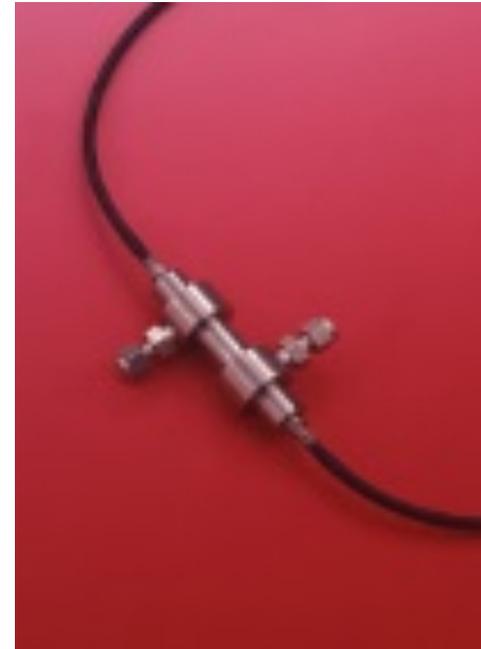
- Optional
 - Class I, Div 1 or 2
 - Deuterium lamp (200-400 nm)
 - Serial port with Modbus





Probes: Transmission Cell

- Path length: 10-20 mm
 - 2 – 1000 mm possible
- Optical fiber 400 micron UV
- UV fused silica windows
 - Sapphire possible (limited UV range)
- Temperature range -20°C to 260°C
- Maximum pressure 3000 psi/207 bar
- HCN service compatible



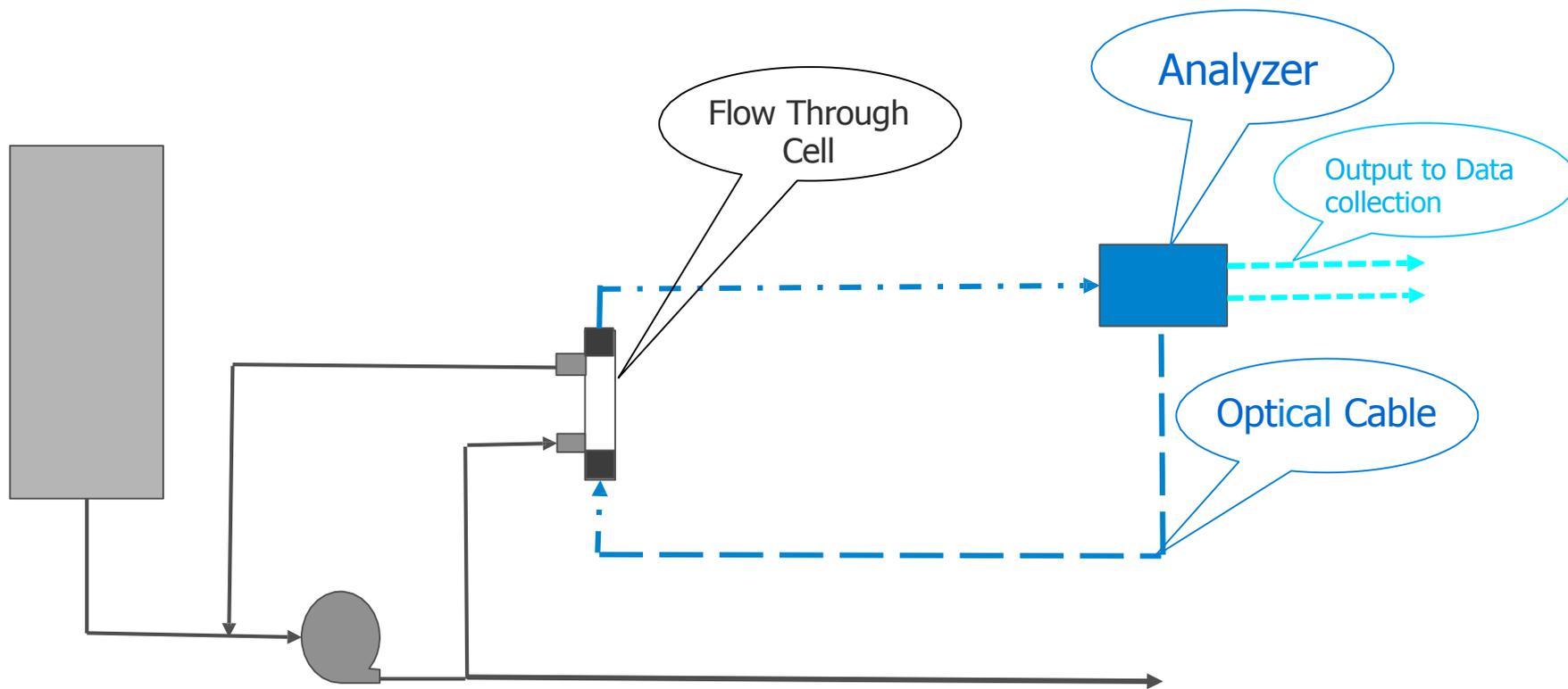


Other specifics - considerations

- Per probe 4 analog outputs are possible (software limited)
 - Calculated values or specific wavelength responses
 - For instance: L^* , YI or b^* , and two wavelength responses
- The ethernet port allows export of up to 10 parameters
- Complete spectra can be saved
- Once installed the configuration can be changed to get the most useful data
- The flash lamp allows data acquisition every 5 s
- Important optical fiber lengths will diminish sensitivity, especially in the UV range
 - Fiber length of 200 feet is maximum for measurements down to 300 nm (100 feet distance)
 - At 1,000 feet: range narrowed to 420-720 nm → YI cannot be determined

Planned installation at INVISTA's Victoria facility

- Cell mounted in a box with a flow measurement device
- Flow through cell from column tails pump discharge to pump suction
- The signal travels by optical cable to the analyzer





Thank you for your attention