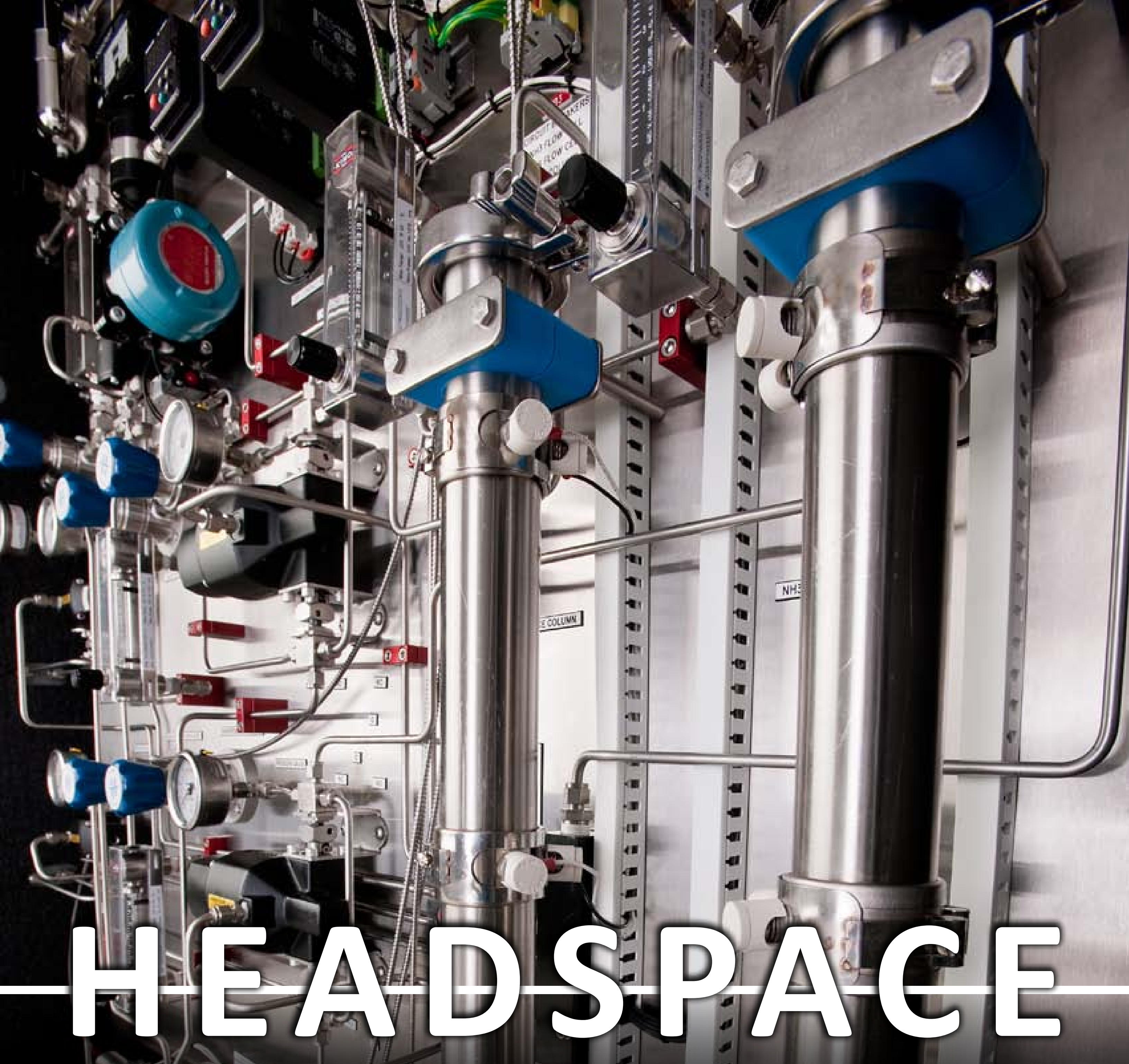


HEADSPACE

DEFINITION

Headspace sampling is designed to strip out a specific chemical from an opaque liquid stream for the purpose of measuring that chemical's concentration optically. When a liquid process is too viscous or dirty to transmit light, a direct sample is not useful for absorbance spectroscopy. A headspace system forces a controlled portion of the liquid sample into the gas phase for unobstructed optical measurement.

APPLICATIONS

contaminants (H_2S) in crude oil

contaminants (H_2S, ammonia) in dirty wastewater

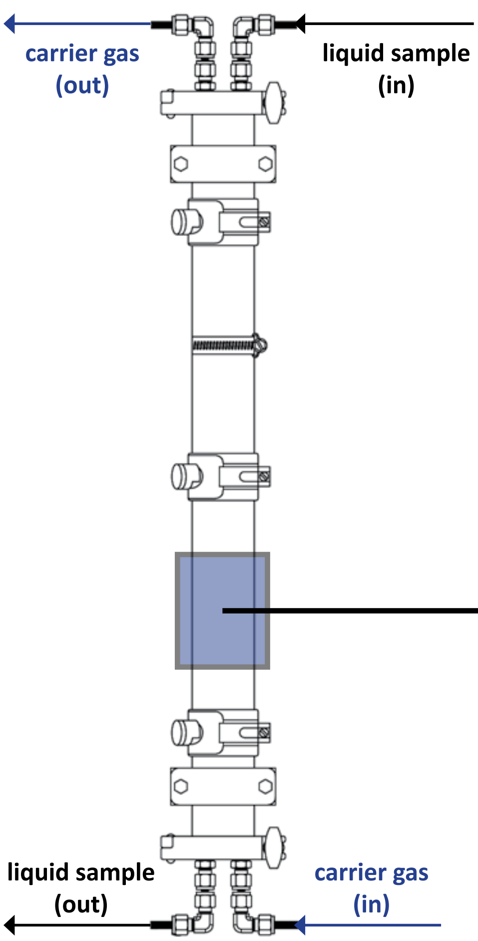
APPLIED™ ANALYTICS

Absorbance spectroscopy is the universal technique for process analysis. The premise is that the concentration of any chemical is directly proportional to its absorbance, or the amount of light it absorbs at a given wavelength. In practice, a light signal is transmitted through a representative sample from the process; a detector measures the signal at the opposite end of the flow cell and correlates the amount of light lost to the concentration of the species in question.

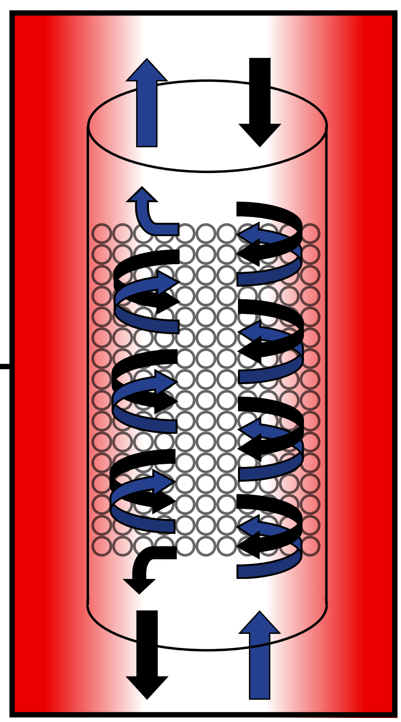
In the context of process analysis, the **problem with opaque liquids** is that they don't allow light to pass through at all. UV spectroscopy is severly impaired in any liquid process that has high opacity, contains particulates which scatter light, or contains chemicals such as phenols which absorb heavily in the UV range and thus interfere with the signal.

As a solution, the **headspace technique exploits Henry's Law** to force part of the liquid sample into the gas phase. This law states that the amount of a certain gas dissolved in a solution at a given temperature is directly proportional to the patrial pressure of that gas above the solution. The takeaway message here is that, given certain constant conditions (temperature, pressure, carrier gas flow rate, and liquid sample flow rate--all of which are regulated by the system), the chemical composition of the headspace gas can be correlated to the composition of the opaque liquid sample.

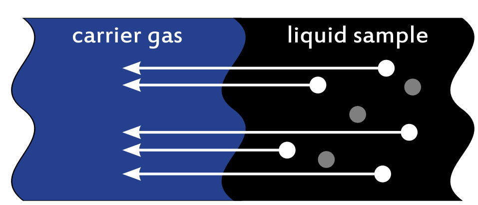
To create a **representative vapor-phase sample** for optical measurement, the headspace system uses a temperature-controlled column--just like any conventional distillation. The design is most advantageous when the component to be measured has high volatility (low boiling point) and interfering components have low volatility (high boiling point). This allows the column to be kept at a temperature range where the measured component evaporates significantly into the headspace gas while absorbance-distorting compounds remain in solution.



An AAI headspace column is 24" long and 2" in diameter. The opaque sample flows in from the top while carrier gas (often N_2) is introduced at the bottom. The carrier gas picks up the molecules that evaporate from the liquid sample and brings them out of the column for optical measurement in the flow cell. The system is calibrated by correlating a gaseous concentration in the flow cell (at a regulated temperature) to the known concentration of a standard liquid sample fed through the column.



The red bands represent a controlled temperature between the boiling point of the species of interest (low) and the boiling point of the interfering, opaque compounds (high). The compound we want to measure (white) transfers easily to the carrier gas while the opaque, problematic compounds (grey) remain largely in solution.



= Raschig rings for maximizing surface area within the column for interaction between liquid and gas. Common in distillation, these rings allow for much more efficient mass transfer.

APPLICATIONS

MEASURING HYDROGEN SULFIDE IN CRUDE

Crude oil with low sulfur content ("sweet crude") is coveted because it is more easily processed into usable gasoline. By contrast, sour crude contains a significant H_2S concentration and requires more expensive processing. Online H_2S analysis is required to determine how much processing a specific crude stream requires and to differentiate different crudes by their commercial value.

Oil naturally contains aromatic compounds which absorb heavily in the UV range and act as spectroscopic interferents; to complicate matters, heavier crudes are too dark and dense to transmit a light signal. The headspace system evaporates H_2S out of the opaque sample by heating the column and flowing the H_2S into the flow cell via carrier gas.

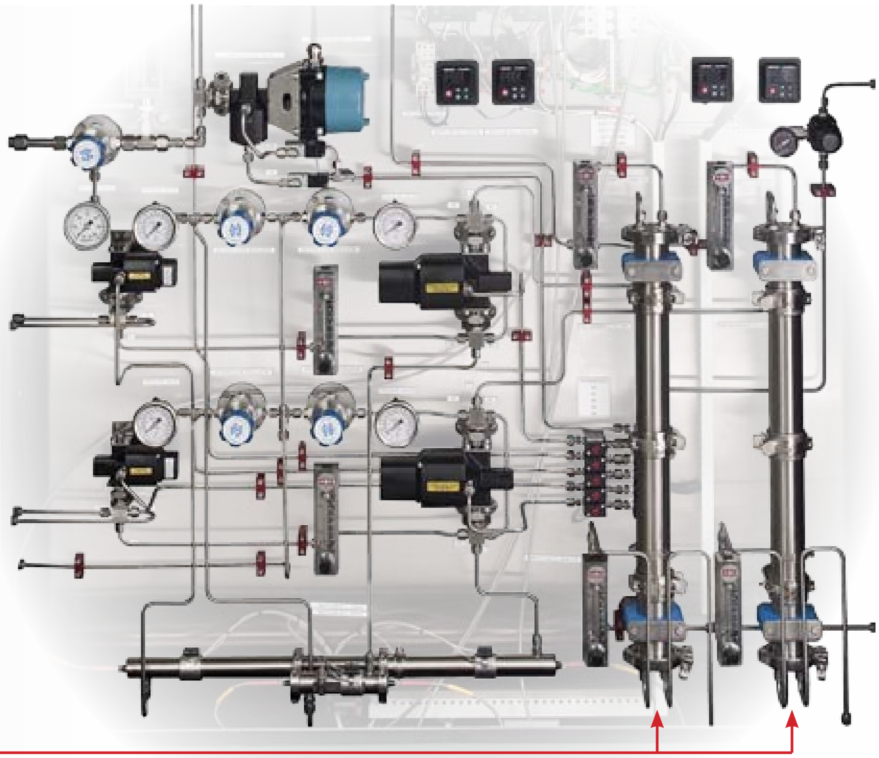
This principle only works because H_2S has a significantly lower boiling point than any of the other compounds present in the oil. This gap in volatility makes headspace an extremely effective crude sampling technique.

single headspace column

MEASURING HYDROGEN SULFIDE AND AMMONIA IN DIRTY WASTEWATER

Rich in H_2S and NH_3, the wastewater from the petroleum refining process is commonly referred to as "sour water." These contaminants are typically stripped to avoid the formation of ammonium bisulfate (a maintenance nightmare) and to curb H_2S pollution. In order to verify the efficiency of the stripping and to validate the water for reintroduction into the process, the strripped stream is monitored for H_2S and NH_3 content.

The double headspace system strips both H_2S and ammonia using two separate columns running in parallel. The carrier gases are fed into two separate flow cells, each with specialized path length and temperature regulation for its target analyte.



dual headspace columns run in parallel

FLOW CELL

Optical measurement interface; the carrier gas travels here from the headspace column. (600mm, 316L stainless steel)

TEMPERATURE CONTROLLER

COLUMN BAND HEATER (3X)

HEADSPACE COLUMN

Representative vapor-phase sample produced from opaque liquid sample.

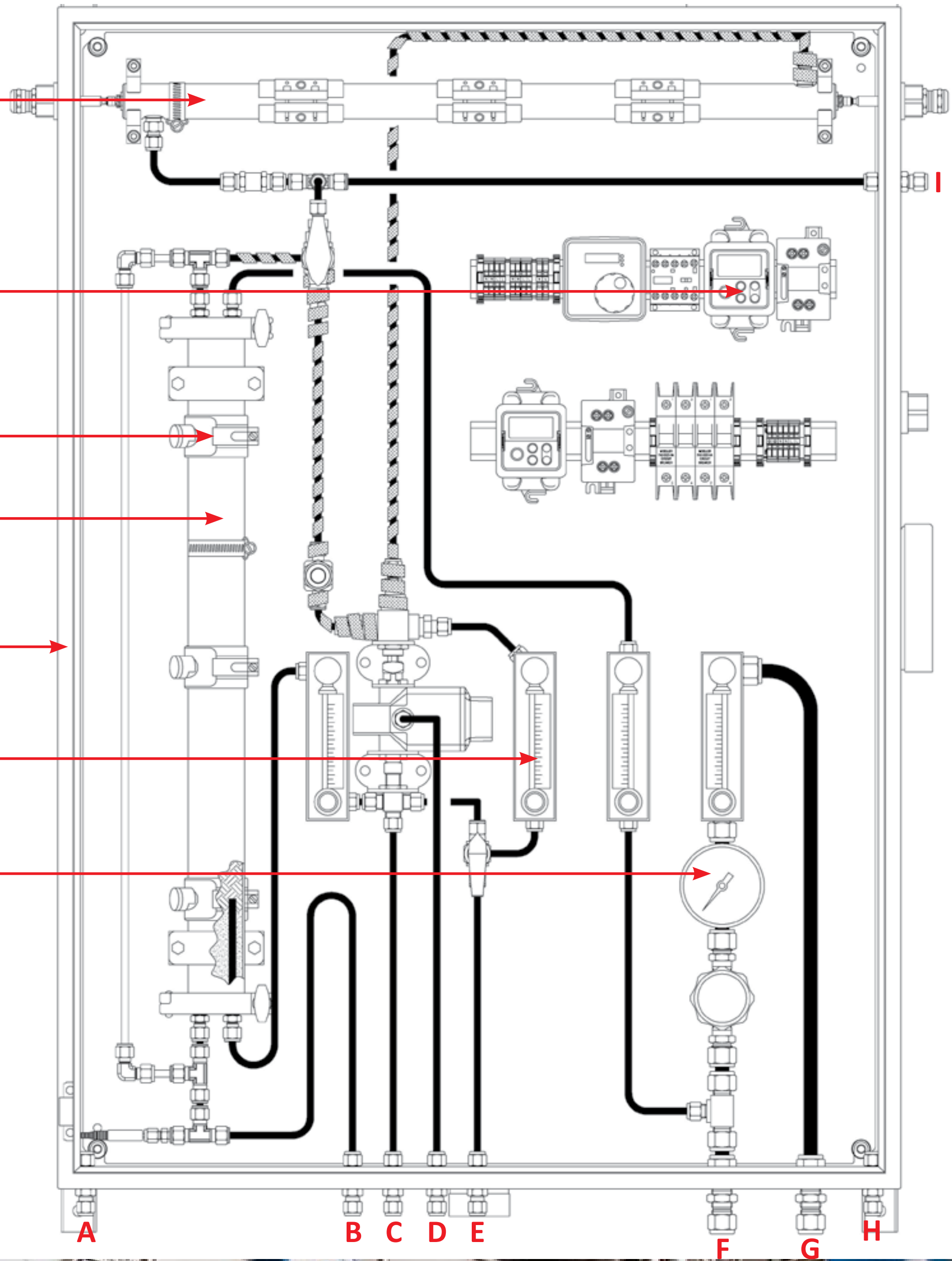
ENCLOSURE

42"x30"x10" 304 stainless steel, NEMA 4X

FLOW METER (3X)

Control flow of carrier gas and liquid sample.

PRESSURE GAUGE & REGULATOR



A. Purge air in/out
B. Water out
C. Zero gas (N_2)
D. Air to valve
E. Span gas
F. Process in
G. Process return
H. Purge air in/out
I. Gas out

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