

HEADSPACE

SAMPLE CONDITIONERS

When a liquid is too opaque or dirty to transmit a light signal, direct optical analysis may be impossible. In this case, the composition of the liquid can be measured by heating the sample and analyzing the vaporous 'headspace' gas. The headspace sampling system design uses Henry's Law to provide a highly effective analytical solution for opaque liquids, including:

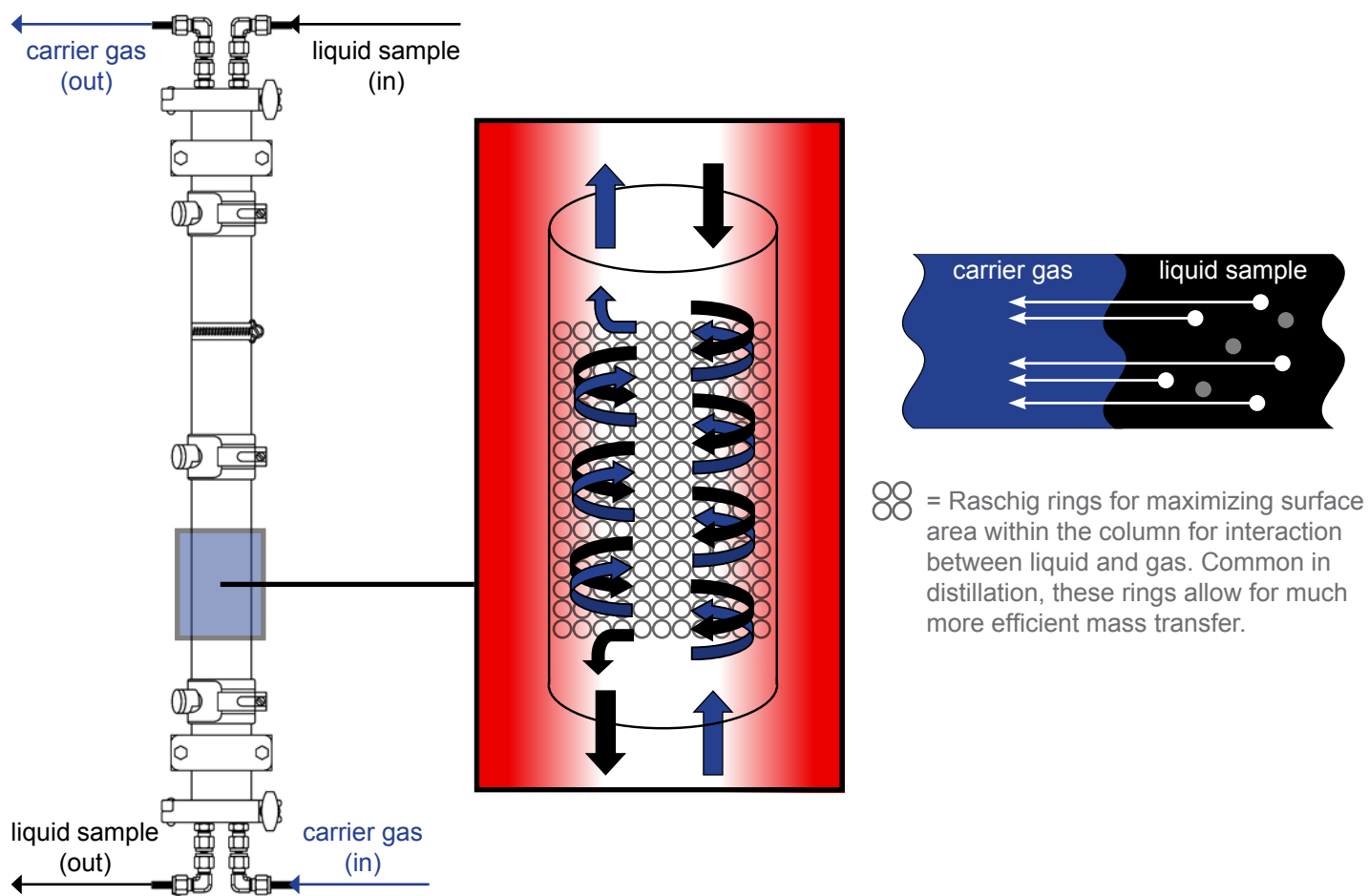
- » crude oil
- » amine solution
- » dirty wastewater

Headspace Design & Henry's Law

Henry's Law states that the amount of a certain gas dissolved in a solution at a given temperature is directly proportional to the partial pressure of that gas above the solution. The major implication of this for opaque liquid analysis is that—given certain constant conditions—the chemical composition of the headspace gas correlates directly to the composition of the opaque liquid sample. The conditions which must remain constant (temperature, pressure, carrier gas flow rate, and liquid sample flow rate) are all held constant by the headspace system.

To create a representative vapor-phase sample from the liquid, the headspace system uses a temperature-controlled column (24" high by 2" diameter). The opaque liquid sample flows in from the top while carrier gas (typically nitrogen) flows in from the bottom. The carrier gas picks up the molecules that evaporate from the liquid sample and carries them out of the column and into the flow cell for optical analysis.

The system is calibrated by correlating the analyte concentration in the flow cell (headspace gas sample) to the analyte concentration of a standard liquid sample.



The held temperature determines the partial pressure mix of compounds in the headspace gas. This allows us to hold the temperature of the column at a point where we get an extremely useful vapor-phase sample that contains the analyte in a spectroscopically significant range while containing very low levels of interfering compounds like aromatics and phenols. This is possible due to the difference in Henry's law constants between the analyte and the interfering compounds.

Key Applications

Measuring H₂S in Crude Oil

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 H₂S concentration and requires more expensive processing. Online H₂S analysis is required to determine how much processing a specific feed of crude oil will require and to differentiate different crudes by their commercial value.

The composition of oil presents significant challenges to direct optical analysis. These challenges include aromatic hydrocarbons and/or phenols which absorb heavily in the UV range and act as spectroscopic interferences, particulates which scatter light, and the opacity of heavier crudes (too dark to transmit a light signal).

The headspace system strips H₂S out of the crude for straightforward vapor phase analysis.

Measuring H₂S and Ammonia in Dirty Wastewater

Rich in H₂S and NH₃, the wastewater from 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 H₂S emissions. In order to verify the efficiency of the stripping and to validate the water for recycling into the process, the stripped stream is monitored for H₂S and NH₃ loading.

The double headspace system strips both H₂S and NH₃ using two separate columns running in parallel. Each column is held at a specific temperature to strip out its dedicated analyte. The headspace gas from each column flows into a dedicated flow cell with path length specific to the analyte.

Measuring H₂S and CO₂ in Rich Amine Solution

Amine gas treating is used to remove H₂S and CO₂ from sour gas for environmental reasons. In the absorber unit, an amine solution absorbs H₂S and CO₂ molecules from the feed gas in order to “sweeten” the upflowing gas stream.

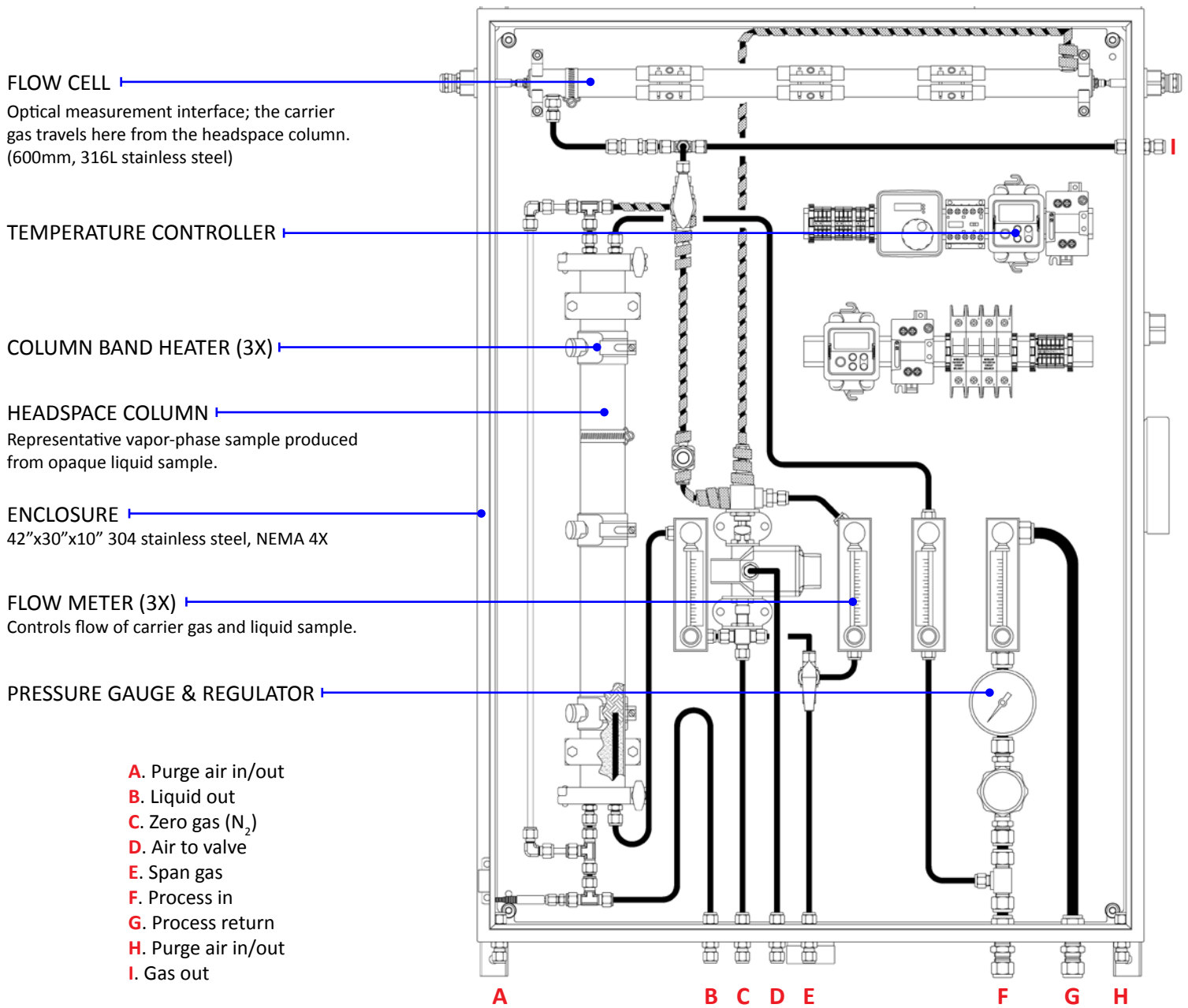
In order to minimize energy costs of the operation, the saturated “rich” amine should only be sent to the regenerator when fully saturated to prevent unnecessary regenerator activity. This circulation can be optimized by implementing a system to monitor H₂S/CO₂ loading in the rich amine and determine current saturation level.

The headspace system strips both H₂S and CO₂ out of the rich amine solution for simple optical analysis.



Double headspace system for H₂S and NH₃ in sour water.

This drawing represents a standard headspace system design for typical applications (e.g. H₂S in crude oil). System design and specifications will vary by measurement application.



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