FTIR Analysis of Trace Water in Anhydrous Ammonia

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Introduction:

Anhydrous Ammonia is a versatile compound, with applications in the refrigeration, medical, semi-conductor, and aerospace industries. The demand of high purity NH_3 is ever increasing, with the major contaminant of interest being water. This presentation will outline the advantages of using FTIR (Fourier Transform Infrared) analysis for moisture contamination at low levels. Sampling and analyzing water is challenging since it is ubiquitous in all hardware involved with the analysis. FTIR utilizes the physical property of the chemical bonds ability to absorb infrared radiation. FTIR is advantageous because it allows all of the infrared radiation to interact with the sample, via a 'scan', and continue on to the detector, with characteristic frequencies absorbed by the specimen. The wave number of the infrared radiation is directly proportional to the wave energy. The wave number is defined as the inverse of the radiation wave length or W = 1/λ.

A scan involves an interferometer (Figure 1), to enable the entire spectrum of the radiation to interact with the sample. An interferogram, as seen in Figure 2, displays the constructive and destructive interference that interacts with the sample. Since this is possible, the results obtained are accurate to the sample. Also, unlike a dedicated moisture instrument, analysis by FTIR does not require a large sample size, and can be completed in a timely fashion. Moreover, multiple contaminants can be measured simultaneously with equal reliability.

When using FTIR for the measurement of contaminants in a product, the matrix itself may interfere to a large degree with the contaminants of interest. Figure 3 represents the region of interest, where the ammonia and moisture spectra interfere.

This complication can be overcome by using the vendor's software in order to eliminate the differences in matrices, and focus in on the region of interest. The wave number of interest for moisture being 3720-3760^{cm-1} in this case.

Figure 1

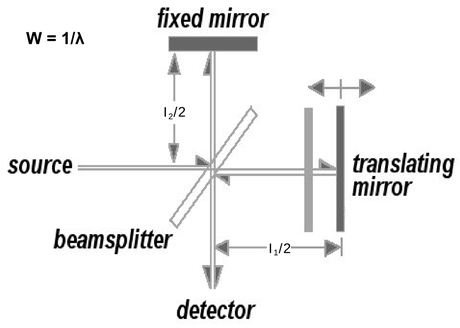


Figure 2

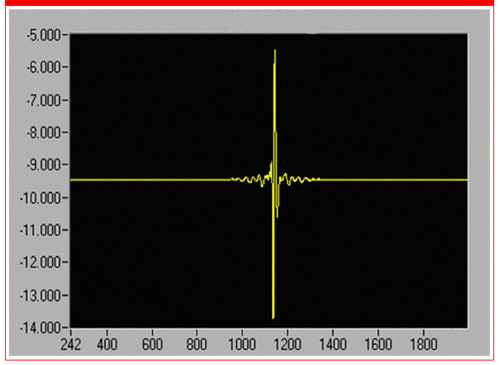
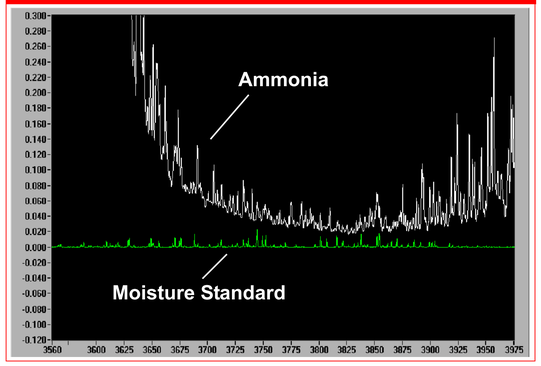


Figure 3



Experiment:

Measurement of H_20 was made using the MKS MultiGas™ 2030 FTIR Analyzer accompanied by MKS's data acquisition software, MG2000.

In this procedure, we eliminate spectral interference with a dry source of ammonia. A schematic of the flow path is described in Figure 4, while the physical representation is shown in Figure 5. The headspace of the dry source ammonia is sampled to give a 'best case' scenario. Since the reference ammonia is taken from the gas phase, any traces of moisture will be further minimized, thus making it an appropriate zero reference. However, we will sample the liquid phase of ammonia when analyzing the unknown source.

Figure 4

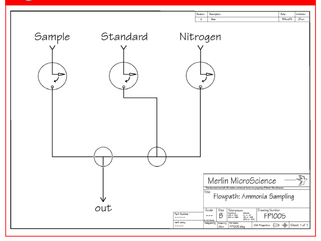
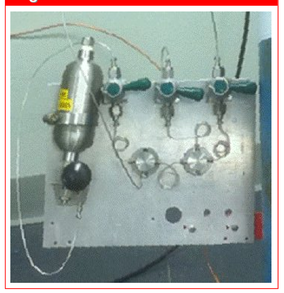


Figure 5



Using the dry source, we can 'zero' the instrument to eliminate interference before measuring the concentration of H_20 in the unknown sample. This task will minimize interference and create a usable baseline for the analysis. The dry source must be observed, over time, against the moisture calibration to confirm the dryness of the source and system.

Figure 6

Sampling indigenous moisture of reference Ammonia until the maximum negative concentration is achieved.

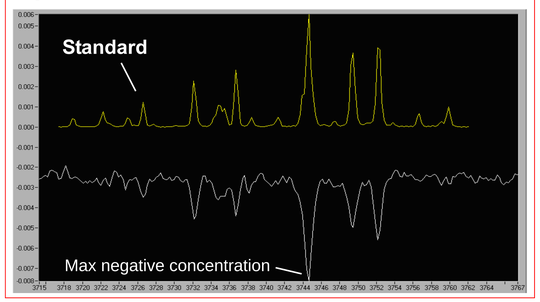


Figure 7

Moisture concentration has been zeroed, producing a useable baseline.

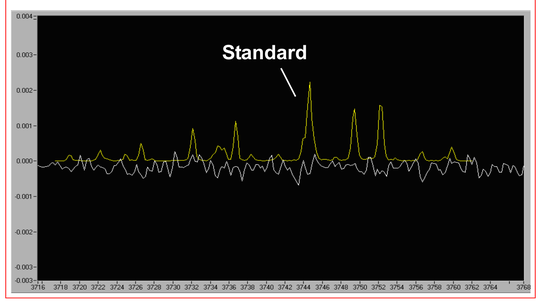


Figure 8

A concentration level of 0.63 ppm was measured against water calibration.