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ABSTRACT

Extractive Fourier Transform Infrared (FTIR) spectrometry was used to continuously monitor the emissions of several different hazardous air pollutants (HAPs) from a Portland cement manufacturing plant. The target analytes included HCl and both volatile and semivolatile compounds. In order to comply with the Clean Air Act Amendments of 1990, the Portland cement industry may be required to continuously monitor the stack emissions of these compounds.

Results of this program indicate that extractive FTIR is a viable monitoring alternative for most of the target compounds. The extractive FTIR approach also has a significant advantage over traditional manual sampling methods by providing continuous and real-time results from a single instrumentation system.

The FTIR measurements were made using an extractive system developed by Clean Air Engineering and Argonne National Laboratories. The experimental program followed two draft protocols for the measurement of HCl and volatile organic compound emissions from Portland cement kilns. These protocols were prepared by the Portland Cement Association and submitted to the EPA Emission Measurement Center for review in July, 1995.

This paper presents the results of the FTIR measurements, and evaluates the success of the approach for each analyte based on the relative attainment of the quality assurance objectives. The paper also describes the details of the FTIR system and procedures employed.

INTRODUCTION

Background

In order to comply with the Clean Air Act Amendments of 1990, the Portland cement industry may be required to continuously monitor the stack emissions of several compounds ubiquitous to the cement-making process. These compounds include hydrogen chloride (HCl) and several organic hazardous air pollutants (HAPs).

The Portland Cement Association (PCA) and the American Portland Cement Alliance (APCA) have jointly formed a HAPs Task Force[1]. This group has taken the lead in working with EPA's Office of Air Quality Programs and Standards (OAQPS) in developing maximum achievable control technology (MACT) emission standards for Portland cement facilities.

The group is working to develop an emission database for cement plants that only burn conventional (fossil) fuels. This database, which will consist of actual emission test results obtained from a cross section of plants throughout the industry, may ultimately be used to develop MACT emission standards for the industry.

The industry is considering the use of extractive Fourier Transform Infrared (FTIR) spectrometry as a means to continuously monitor cement kiln emissions for several of the HAPs. FTIR has several advantages over other technologies, most notably of which is its ability to detect a number of different compounds simultaneously and unambiguously. To support the use of FTIR for these measurements, the HAPs Task Force prepared two separate measurement protocols[2,3], one for HCl and one for volatile organic compounds (VOCs), and submitted these to the EPA Emission Measurement Center for review.

These protocols provide specific sampling, analytical and quality assurance procedures for the use of extractive FTIR to make measurements of HCl and VOC concentrations in Portland cement kiln emissions. The intent of the protocols is to provide an appropriate level of built-in quality assurance so that each method will be self-validating. In this way, the need for separate EPA Method 301 validations for each individual analyte is eliminated.

Scope of Work

Clean Air Engineering, in conjunction with Argonne National Laboratories and Holnam Cement Company, conducted an experimental program to evaluate the feasibility of using an FTIR system to measure HAPs at a cement kiln[4]. The project was conducted at the Holnam facility located in Florence, Colorado.

The target analytes for the program included inorganic compounds and both volatile and semivolatile organic compounds. Specifically, the following compounds were measured:

hydrogen chloride
acetaldehyde
benzene
chlorobenzene
ethylene
formaldehyde
hexane
methylene chloride
naphthalene
phenol
styrene
toluene
meta-xylene
ortho-xylene
para-xylene

All of these compounds except ethylene are listed as HAPs by the EPA. Ethylene was included in the program because it has been identified in cement kiln emissions and it has the potential to form polynuclear aromatic hydrocarbons (PAHs) under certain conditions[3].

In addition to the primary target compounds listed above, ammonia (NH_3), carbon dioxide (CO_2), carbon monoxide (CO), methane (CH_4) and water vapor were also measured during the program. These compounds were included because they are known spectral interferents in the FTIR measurement of many of the target compounds[2,3,5], and all are present in cement kiln emissions. In addition to being spectral interferents, NH_3 and water vapor are also potential measurement system interferents for HCl. For example, NH_3 and HCl may react at certain sampling temperatures to form ammonium chloride, which would deposit in the sampling system and not be seen by the FTIR instrument.

EXPERIMENTAL PROGRAM

The Holnam facility manufactures Portland and masonry cement from raw products using three long, wet rotating kilns. The kilns are fired with pulverized coal. Electrostatic precipitators (ESPs) control particulate emissions from each kiln.

All testing for this project was performed at the exhaust stack of the No. 3 kiln. The program was conducted while the kiln was operating under normal manufacturing conditions. A schematic of the No. 3 kiln is shown in Figure 1.

The program consisted of several different measurement objectives and approaches. These included:

- FTIR Measurements of Target Compounds and Interferents
- Flame Ionization Analyzer (FIA) Measurements of Methane
- Manual EPA Sampling for HCl and NH_3

Three test runs were conducted for each parameter. Each test run was a minimum of one hour in length.

Other measurements of metals, dioxins and total particulate were also performed during the program. The results of these measurements are not discussed in this paper.

FTIR Measurements

The FTIR measurements were made on a hot-wet basis using an extractive system developed by Clean Air Engineering and Argonne National Laboratories. Table 1 lists the physical specifications for the FTIR system. The FTIR measurements followed the draft protocols for the measurement of VOC and HCl emissions from Portland cement kilns. In accordance with these methods, flue gas was continuously extracted through heated Teflon sample lines and directly interfaced with the FTIR instrumentation. Protocol requirements for dynamic analyte spiking were used in order to validate the method. Figure 2 summarizes the sample and QA sequencing used for the FTIR runs. Details of the FTIR system and procedures are summarized as follows.

Sampling System. Figure 3 contains a general schematic of the FTIR sampling system. The system utilized a heated stainless steel probe for gas withdrawal. The end of the probe was equipped with a sintered stainless steel filter for coarse particulate removal. The exit of the probe was followed by a heated glass fiber filter. A heated three-way stainless steel tee preceded the filter and facilitated system calibrations and spiking. A heated Teflon sample line delivered the sample gases from the stack to the instrumental system, which was located at ground level in an environmentally controlled trailer. Sampling line and filter temperatures of 180°C were maintained to reduce the potential of analyte loss in the sampling system. The gas stream remained heated all the way into the FTIR Analyzer.

FTIR Instrument. The FTIR analyzer consisted of a medium-high resolution interferometer, heated fixed path absorption cell, a mercury cadmium telluride (MCT) detector (liquid nitrogen cooled), electronics package and computer. The gas transport path inside the FTIR was heated to 180°C, while the absorption cell was maintained at 150°C.

The interferometer/electronics package was produced by Mattson Instruments, Inc. It was operated at a nominal spectral resolution of 0.5 wavenumber (0.5 cm^{-1}). The heated absorption cell was produced by Nicolet with a fixed pathlength of 10 meters. The mirrors and cell interior were gold plated. The IR beam splitter and all optical windows were made of zinc selenide.

Software. Computer software was used to control the sampling system, acquire spectra and post process the spectra to provide quantitation of the analytes in the sample. TEAM (Toxic Emissions Air Monitoring) software was incorporated by the FTIR for this project. TEAM was jointly developed by Argonne National Laboratories and Clean Air Engineering.

Sample Analysis. The FTIR operated in continuous purge mode during analysis of sample gas. A spectrum of the sample gas was obtained by passing the infrared beam through the sample as it flowed through the gas cell. After identification of the compound from the infrared spectrum of a sample mixture, its concentration was measured by comparing band intensities in the sample spectrum to band intensities in reference spectra of the compound. All reference spectra were created by Clean Air Engineering in the laboratory at one atmosphere and 150˚C.

Table 2 lists the specific spectral regions used for the analytical determinations.

The sample sequencing consisted of the following events:

- Spectral scan of flowing gas (64 scans in 0.9 minutes)
- Data transfer to computer
- FTIR processing of spectra
- Processed data saved to file
- Additional data processing and quantification
- Ready for next scan

The entire sample analysis sequence lasted approximately six minutes per analysis. During this period, the gas cell was continuously purged by sample gas. By operating in this way, a gas volume equivalent to at least five cell volumes purged the cell between subsequent spectral scans.

Spectral Calculations and Quantitation. The TEAM software utilized a partial least squares regression program to analyze for the analytes and known interferants. The program creates a series of equations which, when applied to spectra of unknown mixture samples, accurately predicts the quantities of the components of interest. In order to calculate these equations, a set of known standard mixture spectra (a training set) were prepared which reflect the composition of the unknown as closely as possible. The program uses the training set to build the equations which predict the concentration of the analytes in the sample gas.

FTIR Calibration. The analyzer was calibrated according to the draft protocol procedures using a calibration transfer standard (CTS) of ethylene in nitrogen. Before testing, the analyzer cell and system were calibrated by introducing the CTS both directly into the cell and into the sampling line at the exit of the heated probe. The direct cell calibrations were made by operating the cell in batch mode (i.e., the cell was isolated and no gas flow occurred during the scan). The system calibrations were made using the purge mode described above.

The system bias was determined by comparison of the spectral band areas from the direct (cell) and system calibrations. Appropriate sampling system repairs were made if this bias exceeded 5%.

Immediately following each of the test runs, the CTS was introduced again into the sampling system to record the calibration drift. In order for a test run to be considered valid, the calibration drift between the pre-test and post-test calibrations was required to be less than 5%.

Analyte Spiking. Analyte spiking was used to verify the effectiveness of the sampling system for the target compounds in the flue gas matrix. Two separate gas mixtures were used. One mixture contained hydrogen chloride and sulfur hexafluoride (SF_6) in a nitrogen balance. The other mixture contained acetaldehyde, chlorobenzene, p-xylene and SF_6 in a nitrogen balance. The SF_6 was included in the matrix spiking mixtures as an inert tracer gas to accurately determine the spike/total sample flow ratio.

Analyte spiking was performed before and after each test run by introducing the spike gases at the probe tip in front of the secondary filter. The spiking was performed after the acquisition of the pre- and post-test CTS spectra. A direct-to-cell measurement was performed before each analyte spike by introducing spike gas to the FTIR cell (batch mode). Separate spiking sequences were required for the HCl and VOC calibration mixtures. The system biases calculated from the analyte spiking are required by the protocols to be within 30% for the spiked recovery check compound analytes.

The entire calibration and analyte spiking quality assurance sequences depicted in Figure 2 required approximately three hours to complete prior to each run, and an additional three hours after each run. For Runs 2 and 3, the QA/QC sequence after Run 2 was also the QA/QC sequence before Run 3, thus saving three hours in the overall testing period.

FIA Measurements

As an alternate comparison procedure to the FTIR measurements, methane emissions were continuously monitored using a flame ionization analyzer following EPA Method 25A procedures.

A slip-stream of the FTIR gas sample was transported directly into a J.U.M. Engineering Model 109A Flame Ionization Analyzer. This analyzer incorporated a sample filter, sample pump, sample valve and a flame ionization detector (FID), all mounted in a heated oven operating at approximately 190°C. A catalytic reactor was installed in front of the FID. This reactor operated at approximately 220°C to strip away all hydrocarbons except methane.

Manual EPA Sampling

A modified EPA Method 5/26 sampling train was used as an alternate comparative procedure to measure HCl and NH_3 emissions. This approach used a dilute solution of sulfuric acid to absorb the HCl and NH_3 from the gas stream. This solution was then analyzed at the laboratory for chloride and ammonium ions using standard ion chromatography procedures.

Procedures for selecting sampling locations and for operation of the apparatus were derived from EPA Method 5 and associated EPA Methods 1 through 4. Sampling was performed isokinetically. The impinger solutions were analyzed for chloride and ammonium ions using ion chromatography following the general procedures contained in EPA Method 26.

EXPERIMENTAL RESULTS AND DISCUSSION

Emissions Quantification

VOCs. Table 3 summarizes the FTIR results obtained for the VOCs. All of the VOC concentrations were below quantifiable limits except for ethylene. The ethylene concentration averaged 8.7 ppmdv. As described below, the detection limits for the VOCs were around 3.7 ppmdv.

These results are consistent with other studies which have quantified cement kiln VOC emissions and shown relatively low concentrations of organic HAPs from non-hazardous waste burning facilities.

The practical lower quantification limit (LQL) is determined by the measurement system design, the composition of the gas stream, and the nature of the gas stream being analyzed. Specifically, the LQL depends on:

- the absorption coefficient of the compound in the analytical frequency region;
- the spectral resolution;
- interferometer sampling time;
- detector sensitivity and response;
- absorption pathlength;
- the presence of interfering species (notably H_2O, CO_2 and SO_2);
- analyte losses in the sampling system;
- optical alignment of the gas cell and transfer optics;
- optical throughput of the cell.

The LQLs represented in Table 3 were determined according to FTIR Protocol. They are believed to be relatively conservative estimates of the actual detection limits. These values are based on the calculation of the minimum analyte uncertainty, or MAU. The MAU is the minimum concentration for which the analytical uncertainty limit, based on spectral data in the analytical region, can be maintained. The MAU is an estimate of the absolute concentration uncertainty when spectral noise dominates the measurement error.

The determination of the MAU depends upon several assumptions, and is generally a trial and error procedure. The MAU can be influenced by the starting assumptions which go into its calculation. Because of this, care must be taken when interpreting detection limits based upon this approach.

HCl and Ammonia. The HCl and NH_3 results are presented in Table 4. Both the FTIR and the EPA Method 5/26 results are presented in the table.

The HCl concentration measured by the FTIR averaged 2.7 ppmdv. The HCl measured according to EPA Method 5/26 procedures was very low, averaging 0.09 ppmdv. The NH_3 concentrations measured by the FTIR and EPA Method 5/26 showed good agreement, with values averaging 118 ppmdv and 116 ppmdv, respectively.

The 2.7 ppmdv HCl concentration reported for the FTIR may be biased somewhat high due to inadequate purging of spike gas from the system before collection of sample spectra. One of the primary problems associated with HCl measurement is physical adsorption and/or chemical absorption of the analyte within the system components. Because of this, a relatively long time is required to reach a new steady state concentration within the system after a step-change in the source concentration. This leads to relatively long system response times for HCl, on the order of 30 minutes or greater for the system used in this project.

Because of this long response time for HCl, it was difficult to precisely define the point after a matrix spike at which the analyzer was seeing only sample gas. Thus, it is possible that the first several spectral samples for each run may have actually included a small amount of residual influence from the previous HCl matrix spike. As Figure 4 shows, the HCl concentration throughout each run showed a continuous decline over time, which would be consistent with this phenomena. However, no apparent steady-state value was achieved during any of the runs.

As Figure 4 shows, the actual HCl concentration measured with the FTIR is probably below 2 ppmdv, and may even be below 1 ppmdv. Strictly speaking, however, the FTIR detection limit for HCl in this program was around 3.7 ppmdv. This is based upon the determination of the MAU as described above for VOCs. However, because of some idiosyncrasies related to the mathematical interpretation of the spectral data for HCl, it is believed that a considerably lower detection limit for HCl is achievable. Many of the spectral peaks for HCl are strong but very narrow. The mathematical treatment of these peaks in determining the MAU (and correspondingly, the detection limit) tends to result in an overestimation of the true detection limit.

Methane. A comparison of the methane concentrations measured separately using the FTIR and the FIA is shown in Table 4.

The methane concentration measured by the FTIR averaged 21.6 ppmdv. This compares relatively well with the concentration measured using the FIA, which averaged 28.3 ppmdv. The reason that the FIA result may have been somewhat higher could be due to some limitations of the hydrocarbon stripper used by the J.U.M. 109A FIA. Low-weight hydrocarbons (e.g., ethane, ethylene) present in the sample gas may not be totally removed by the stripper. These compounds would then be counted as methane, thus imparting a slight positive bias to the measurements.

Quality Control

Calibration. Results from the CTS direct and system calibrations for each test run are shown in Table 5.

The HCl and VOC FTIR protocols require that the CTS spectral band area of the system calibration must be within 5% of that of the direct calibration (i.e., the system bias must be less than 5%). The CTS system bias before each test run ranged from 0.1% to 0.2%, well within the protocol requirements.

The protocols also require that the CTS spectral band areas for the system calibrations before and after each test run be within 5% of each other (i.e., the system drift must be less than 5%). The CTS system drift ranged from 0.03% to 1.9% for the three test runs.

Dynamic Spiking. Results from the analyte spiking for each test run are also shown in Table 5.

Most of the matrix spike recoveries were within protocol requirements of 100% ±30%. There were three exceptions, however. The HCl matrix spike at the conclusion of the final run was slightly below 70%. This is believed to be due to a sudden drop in ambient temperature accompanied by snow/sleet and high winds that probably resulted in a cool spot in the sample line. This did not appear to impact the results of the measurements prior to introducing the spike. However, this incident re-emphasizes the importance of proper sampling system heating for HCl measurements.

Two other spike recoveries were low - the Run 1 post-test for p-xylene and the Run 3 post-test for chlorobenzene. No definitive cause for these results have been identified.

CONCLUSIONS

The following conclusions resulted from this project.

- FTIR is a viable approach to monitoring HCl, NH_3, and several different HAPs from cement kilns. Good agreement between the FTIR measurements and the manual EPA sampling approaches indicates that the FTIR method is a suitable alternative to the more cumbersome and less versatile manual methods.

- Most of the VOCs and likely the HCl were below the detectable range of the FTIR. Despite this, however, the overall success of the QA/QC program for these analytes demonstrated that the FTIR approach is suitable for measuring these compounds.

- Prior concern for adequate heating of sampling system components for HCl measurements is well-founded. Extensive efforts were made to ensure that all sample-exposed components of the sampling system were maintained at 180°C. Despite this, a potential problem related to localized cold-spots in the sampling system appears to have arisen during one of the runs. Although this problem was a result of an unexpected weather change, it highlights the necessity of heat-tracing in this application and the need to engineer the system for “worst-case” conditions.

- Standard methods for determining detection limits for FTIR analyses yield a somewhat arbitrary end result. Limits for this program were purposely set at high levels because of this, even though much lower levels may be quantifiable in some circumstances. This issue of detection limits is not thoroughly addressed in detail in the specific FTIR protocols. More definitive procedures for determining detection limits may be in order.

- The actual test duration of a 1 to 2-hour FTIR run was on the order of 7 hours due to the time required to complete all of the necessary QA/QC before and after the run. This fact must be considered when planning this type of measurement program.

- Problems were encountered in obtaining a “clean” sample with respect to HCl due to difficulties in purging all of the matrix spike from the sampling system. Because of this, the reported FTIR values for HCl may be biased high. In order to prevent this problem, additional time should be allowed to purge the system between the matrix spike and sampling steps.

ACKNOWLEDGMENTS

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Table 1. FTIR System Specifications

Sample Delivery	
Sampling System Temperature	180°C
Nominal Sample Flow Rate	12 LPM
Analytical	
Cell Path Length	10 meters
Cell Temperature	150°C
Spectral Resolution	0.5 wavenumber
Number of Scans	64/0.9 minutes
Detector Type	MCT (Liquid N2 cooled)
Optical Window Material	Zinc Selenide
Data Analysis	
CPU Type	486DX/66
RAM	24 MB
Disk Storage	1 GB
Operating System	WFW 3.11
Software	TEAM 2.1

Table 2. FTIR Analytical Spectral Regions

Compound	CAS #	Analytical Region (cm-1)
ammonia	7664-41-7	840-1275
hydrogen chloride	7647-01-1	2679-2840
acetaldehyde	75-07-0	2679-2840
benzene	71-43-2	3020-3124
chlorobenzene	108-90-7	1012-1036
formaldehyde	50-00-0	2679-2840
ethylene	74-85-1	920-960
hexane	110-54-3	2850-2950
methylene chloride	75-09-2	1241-1290
naphthalene	91-20-3	770-819
phenol	108-95-2	1111-1284
styrene	100-42-5	886-931
toluene	108-88-3	3018-3054
o-xylene	95-47-6	2859-3095
m-xylene	108-38-3	2971-3138
p-xylene	106-42-3	770-819

Table 3. VOC Results

Run No.	1	2	3	Average
Ethylene	7.5	9.1	9.6	8.7
Acetaldehyde	<3.69	<3.72	<3.71	<3.70
Benzene	<3.69	<3.72	<3.71	<3.70
Chlorobenzene	<3.69	<3.72	<3.71	<3.70
Formaldehyde	<3.69	<3.72	<3.71	<3.70
Hexane	<3.69	<3.72	<3.71	<3.70
Methylene Chloride	<3.69	<3.72	<3.71	<3.70
Napthalene	<3.69	<3.72	<3.71	<3.70
Phenol	<3.69	<3.72	<3.71	<3.70
Styrene	<3.69	<3.72	<3.71	<3.70
Toluene	<3.69	<3.72	<3.71	<3.70
o-Xylene	<3.69	<3.72	<3.71	<3.70
m-Xylene	<3.69	<3.72	<3.71	<3.70
p-Xylene	<3.69	<3.72	<3.71	<3.70

Table 4. HCl, NH_3 and CH_4 Results

Run No.	1	2	3	Average
HCl Results (ppmdv)				
FTIR*	3.51	2.48	1.98	2.66
EPA 5/26	0.11	0.10	0.09	0.09
NH_3 Results (ppmdv)				
FTIR	118	112	123	118
EPA 5/26	138	117	111	116
CH_4 Results (ppmdv)				
FTIR	20.4	20.0	24.3	21.6
EPA 25A	29.6	24.8	31.1	28.3

* Due to problems in adequately purging the system of HCl matrix spike gas, the FTIR HCl results are suspected to be biased high.

Table 5. FTIR Quality Assurance Results

Run Number	**1**		**2**		**3**	
Sequence	**Pre-Test**	**Post-Test**	**Pre-Test**	**Post-Test**	**Pre-Test**	**Post-Test**
CTS Calibrations						
Direct Calibration (ppm)	96.53		103.53		103.28	
System Calibration (ppm)	96.71	95.65	103.45	103.42	103.42	105.44
System Bias	0.18%		0.08%		0.13%	
System Drift		1.09%		0.03%		1.95%
Matrix Spikes						
<u>HCl</u>						
Predicted Response (ppm)	12.05	23.00	7.92	7.99	7.99	9.47
Actual Response (ppm)	10.10	16.52	7.83	7.21	7.21	6.45
Percent Recovery	83.8%	71.8%	98.7%	90.2%	90.2%	68.2%
<u>ACETALDEHYDE</u>						
Predicted Response (ppm)	5.20	5.65	5.20	5.31	5.31	5.98
Actual Response (ppm)	5.27	5.17	4.70	5.30	5.30	5.36
Percent Recovery	101.5%	91.5%	90.4%	99.8%	99.8%	89.6%
<u>CHLOROBENZENE</u>						
Predicted Response (ppm)	5.60	6.09	5.60	5.70	5.70	6.45
Actual Response (ppm)	5.21	6.65	4.51	4.47	4.47	3.22
Percent Recovery	93.1%	109.2%	80.4%	78.5%	78.5%	49.9%
<u>p-XYLENE</u>						
Predicted Response (ppm)	5.56	6.04	5.56	5.66	5.66	6.39
Actual Response (ppm)	5.52	3.76	4.25	4.50	4.50	4.60
Percent Recovery	99.3%	62.3%	76.3%	79.5%	79.5%	72.0%

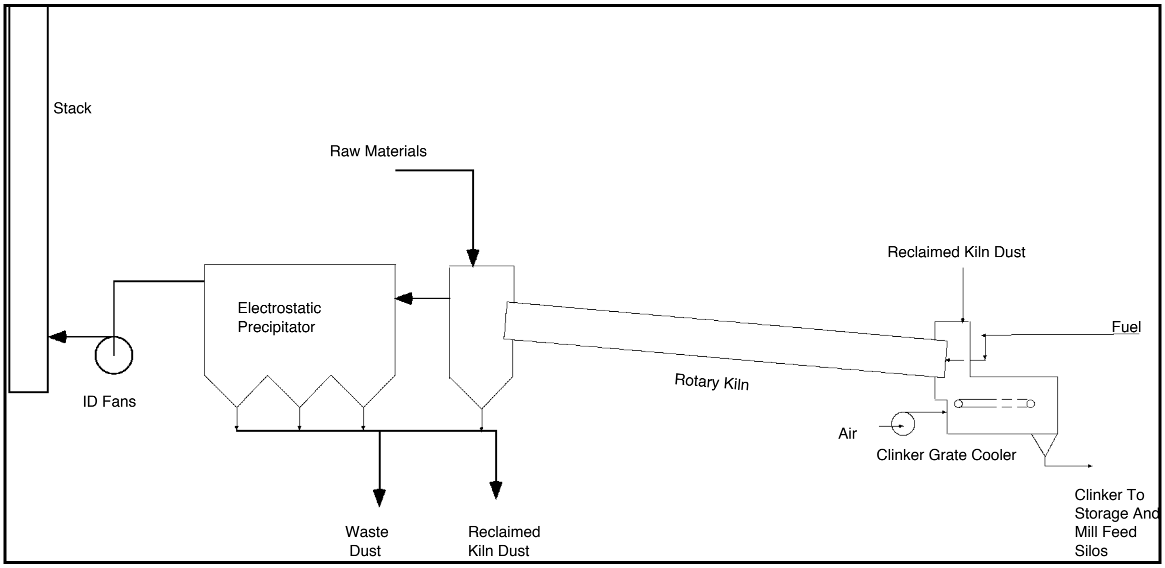

Figure 1. Process Schematic

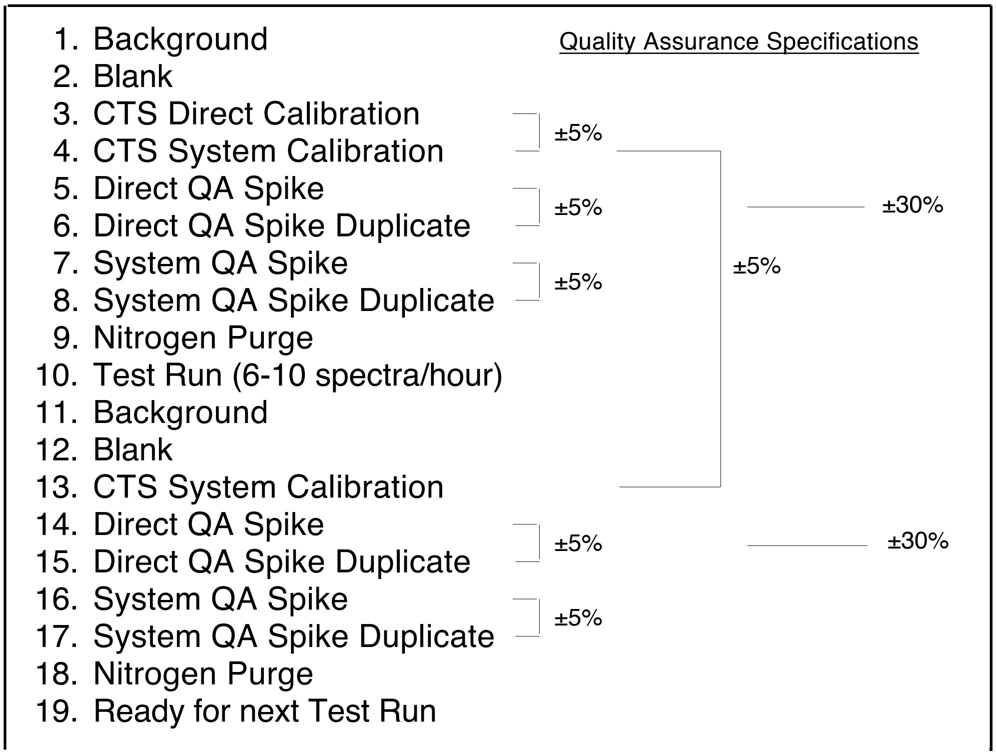
1. Background
2. Blank
3. CTS Direct Calibration
4. CTS System Calibration
5. Direct QA Spike
6. Direct QA Spike Duplicate
7. System QA Spike
8. System QA Spike Duplicate
9. Nitrogen Purge
10. Test Run (6-10 spectra/hour)
11. Background
12. Blank
13. CTS System Calibration
14. Direct QA Spike
15. Direct QA Spike Duplicate
16. System QA Spike
17. System QA Spike Duplicate
18. Nitrogen Purge
19. Ready for next Test Run

Figure 2. FTIR Run Sequencing

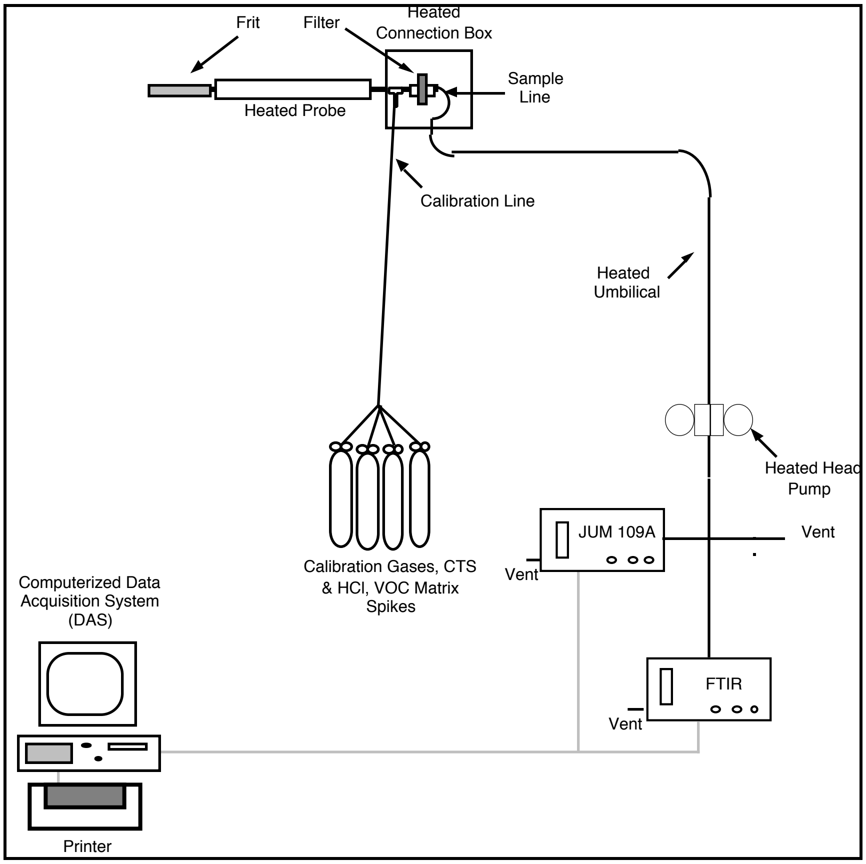

Figure 3. JUM 109A and FTIR Monitoring System Schematic

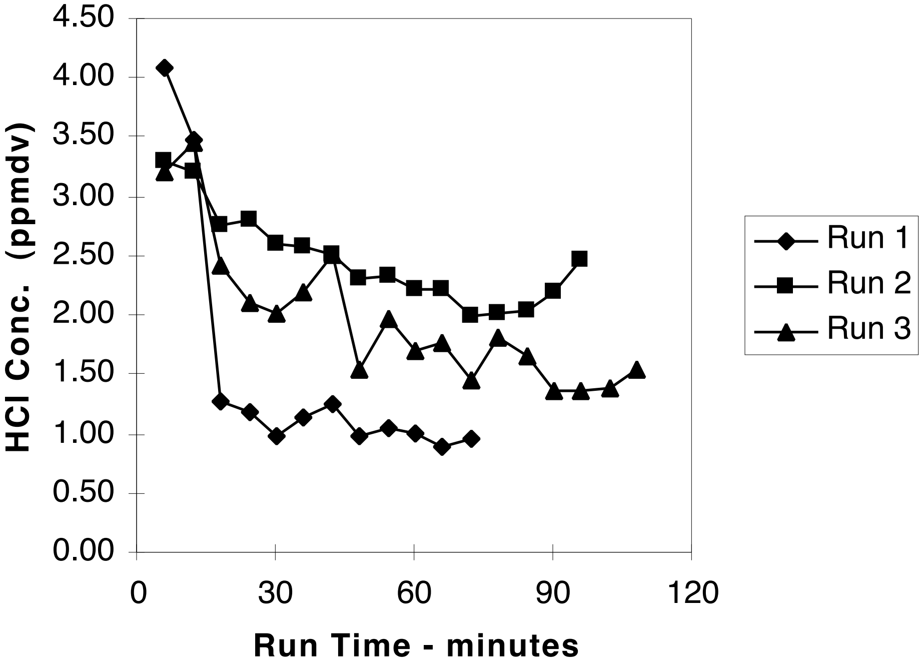

Figure 4. FTIR HCl Concentration versus Run Time