IMPROVED PLASMA TORCH AND DATA ANALYSIS SOFTWARE FOR AN ON-LINE, MULTIELEMENT ICP SPECTROMETER DESIGNED FOR APPLICATION TO HIGH TEMPERATURE AND PRESSURE FOSSIL FUEL PROCESS STREAMS

Joseph Yip, Kelly D. Pearce, C. Edward Christy, Brooke A. Barill, Shelly M. Monteleone, William P. Chisholm

US Department of Energy, Morgantown Energy Technology Center, Morgantown, WV 26507-0880 telephone (304)291-4730

KEY WORDS: ICP, On-Line Analysis, Fossil Fuel Analysis, Artificial Neural Networks

ABSTRACT: METC is continuing to improve its real-time, multielement ICP spectrometer system for application to high temperature and high pressure fossil fuel process streams. The ICP torch operates on a mixture of argon and helium with a conventional annular swirl flow plasma gas, no auxiliary gas, and a conventional sample stream injection through the base of the plasma flame. A new, demountable torch design comprised of three ceramic sections allows bolts passing the length of the torch to compress a double O-ring seal. This improves the reliability of the torch. A battery of monochromators, controlled by a microcomputer, is the detection system. In lieu of conventional data reduction techniques, a neural net analysis of emission spectra is being developed to analyze the data.

Rising concerns about the potential release of harmful elements into the environment from coal utilization have driven the development of new analytical capabilities. Especially useful to the suite of advanced technologies under development by the Morgantown Energy Technology Center, (METC), would be a process monitor to perform real-time, multi-element trace analysis in a high temperature and high pressure environment. The inductively coupled plasma (ICP) spectrometer has the potential to perform this kind of process monitoring.

The role of the inductively coupled plasma, (ICP) as a process stream monitor for trace elements is only beginning to be realized, although it has been widely used for a number of years as a spectrometric emission source in elemental analysis laboratories. Some details of METC's efforts in this direction have been reported previously [1]. The raison d'etre of METC's ICP work is to address process monitoring under conditions relevant to METC's advanced fossil fuel technologies.

The shortcomings of conventional ICP systems plumbed directly to high temperature and pressure process systems have been previously discussed [1]. In brief, these systems cannot sustain a plasma in a gas stream containing a large proportion of molecular gases, they cannot be connected to hot, pressurized sample lines, and the plasma emission spectrum contains strong interfering molecular bands. Our previously reported torch design solved these problems, but suffered from a tendency for the plasma to erode some of the O-rings after several hours of operation. These o-ring seals eroded because they tended to develop leaks. They developed leaks because they were not compressed, and were sealed only by forcing the quartz auxiliary tube into the torch, without additional compression. Also, the brass base of the old torch design spanned several centimeters between the electrically hot side of the radio frequency (RF) coil and the chassis ground. This tended to aggravate the tendency of the plasma to discharge back into the torch towards ground. We have developed a new torch which is sectioned into several pieces. By running bolts through the length of the torch, all the o-ring seals in it can be compressed by tightening the bolts. The new torch willbe made of a insulating ceramic capable of direct connection to a hot sample line, but which also minimizes the tendency of the discharge to follow plasma gas leaks towards ground. Also, the design protects some of the O-rings by providing water cooling between the O-rings and the hotter parts of the torch.

The detection system has previously been described [1]. In a typical experiment, integrated line intensities from the emission spectrum of an element of interest are recorded from solutions of known concentration nebulized into the sample gas stream, and a calibration curve is drawn to relate element concentration to integrated intensity. Although this established approach works well for simple systems, the ICP optical emission from a fossil fuel sample stream is very complex, is influenced by the presence of easily ionized elements, and has a variable and noisy background. Given the limitations of present curve fitting algorithms and other methods of ICP analysis it was desired that a "better" way to determine elemental concentration was needed; a method that could account for the interferences and complications present in these spectra.

It was decided that an ArtificialNeural Network (ANN) might be a very powerful tool in analyzing this data. ANNs have become very popular in recent years in applications of pattern recognition, speech recognition and other areas due to their adaptability to different types of data. ANNs were inspired by and function similarly to biological neural networks.

Figure 1 shows a typical Back Propagation neural network architecture. This particular architecture is the one used for the METC ICP application i.e. two layers and one output. Data values ranging from 0 to 1 enter the network at the input layer one point per node. Each of these values is then multiplied by the hidden weights and summed at the hidden layer nodes. These values are then multiplied by the output weights and are summed at the output node(s).

Training the network entails first gathering training data representative of a pattern of known, experimental inputs and outputs, i.e. known concentrations and integrated line intensities. The data could include the intensities of several emission lines from one element, the intensities of lines of easily ionized elements, lines from elements that overlap the emission lines of elements of interest, background spectral regions, molecular emission band intensities, and even ancillary data on experimental conditions, such as flow rates, RF power level, etc. Given the training data input, the network adapts and adjusts until its calculated output becomes very close to the output values of the training data. Once trained, unknown input data may be entered and the network will generate an answer based on the data for which it was trained. Back propagation neural networks function well in pattern recognition and classification, but are often very slow to train if large, i.e. if they contain a large number of inputs and/or hidden neurons.

The back propagation neural network used for the METC ICP is a commonly used, general network architecture. The uniqueness of its application to the ICP system derives from knowledge of which peaks to use to give a good representation of the concentrations of the elements. Background noise and interactions between elements play a large part in determining which peaks to use since the number of inputs (data points) needs to be minimal, for reasonable computational times, but must be large enough to give the network information sufficient to make reliable predictions.

Initial experiments with the neural net were performed by generating data with a mercury pen lamp or an iron hollow cathode lamp. Using neutral density filters, training data of intensity versus filter attenuation was generated. Several emission lines and a background region were included in the data set. It was found that the neural net works well over a decade of data, but did not work well over a larger range. Some experiments with data from the actual ICP system were also performed, with promising results. Efforts to further develop the neural net continue.

The new torch design was constructed in teflon and operated on argon and argon and helium mixtures. Improved reliability was found under a variety of operating conditions. The new design also provides for adjustments of the alignment of the plasma, auxiliary, and sample gas tubes. Planned work includes constructing a ceramic version and operating with hot sample gas input.

References

[1] Robert R. Romanosky, Anthony S. Viscomi, Steven S. Miller, William P. Chisholm, Proceedings of March 1993 American Chemical Society Conference, Denver, Colorado, March 28 - April 2, 1993.

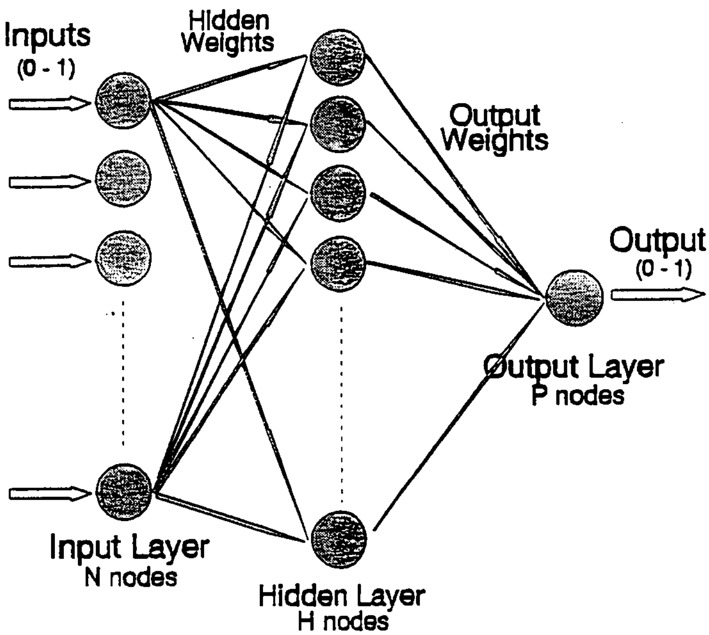

Figure 1. Input, Hidden, and Output Layers of an Artificial Neural Network.