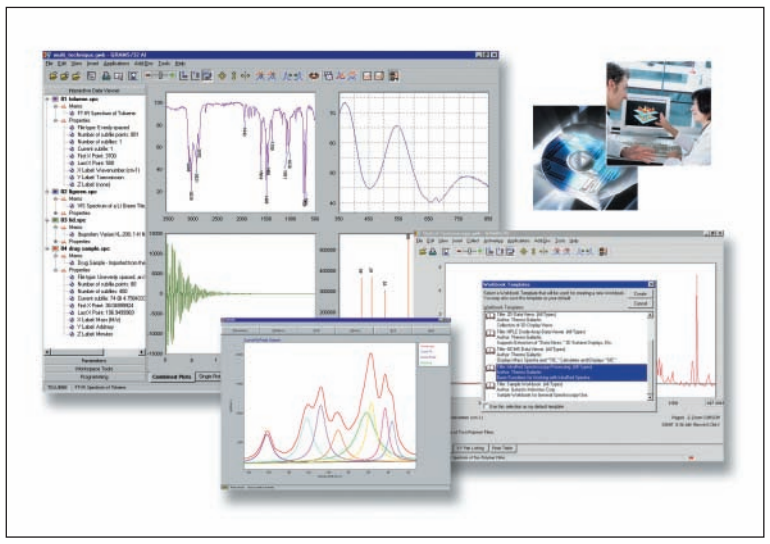
Cool Analysis of So Much Hot Air

Discovering unexpected compounds in the stack emissions of a process plant requires a degree of detective work, supported by the best in infra-red spectrometers and the most powerful of spectroscopic software.



The monitoring and control of industrial emissions has been high on the environmental agenda for almost 50 years, since the first large-scale programmes to measure sulphur dioxide and 'black smoke' were set up in 1961. Since then, air quality in the UK has greatly improved – to the extent that the Hollywood-cliché 'pea-souper' smogs are now but a distant folk memory.

Today, industrial plants have an ever-increasing barrage of legislation and regulation to adhere to with strict requirements regarding the nature and quantity of allowable stack emissions and tight timetables of monitoring and control to be met. The stakes are high at this end of the pipe: lose your emissions monitoring capability and within hours your plant may be shut down completely.

Even the best-maintained site can lose a sensor with little or no warning, meaning that the availability of a rapid back up is critical. Protea, a Company specialising in process investigation, uses techniques such as Fourier Transform infra-red (FT-IR) and mass spectrometry and carries out MCERTS-accredited monitoring of gas emissions from all types of manufacturing processes in the UK and Ireland.

The Company also manufacturers portable analysers to its own design such as the ProtIR 204M portable FTIR instrument, designed specifically for stack emissions testing and process investigation, which can be deployed rapidly on site should a monitoring system go offline.

"This is very much a 'wheel it in, plug it in' machine", said Protea's Senior Applications Specialist Andy Toy. "A man in a van can get it onto site, roll it up to the stack, put a line into wherever it is that the samples are to be drawn from and press go".

This model was the first analyser to be specifically designed for use as a back-up continuous emissions monitor (CEM) for operation within the European Standard EN 14181 and simultaneously measures all gases required on Waste Incineration Directive permits, according to the Company.

"The thing with emissions monitoring", continued Mr Toy, "is that you don't always know what it is you're looking for. With combustion industries like waste incineration it may be fairly predictable – carbon monoxide and oxides of nitrogen are the main gases of interest here. But in the chemical and pharmaceutical markets you can find quite a complex mixture of organic compounds. This is why we made the machine as versatile as we could."

Garbage In, Garbage Out

With any sensor technology, the value of the information yielded depends on both the quality of the data collected and the power with which it is analysed. Protea uses GRAMS software from Thermo Scientific, supplied by UK distributor Adept Scientific, to validate the spectra collected on the ProtIR 204M.

GRAMS enables data to be visualised, processed and managed and is compatible with many different types of instrument. "It has a number of really nice features which are useful for producing calibration spectra like the spectral maths facility and the way you can add and subtract spectra," Mr Toy added.

"We use a partial least squares plug-in called PLSplus/IQ which extends it even further to build models of the spectra. We plug the calibration spectra into the model – and you can put hundreds of them in – then using the tools in GRAMS we can optimise the models to pick out features that best suit the job.

"At this stage we haven't even seen a [stack emission] sample yet – we're just testing the model to see if it could predict, say, benzene. We can see what we would expect with benzene, then add acetone or water or both, and review it.

"When it gets really interesting is when you find something unexpected in the sample – in what we call the component matrix. The customer might tell us that there are five compounds in the matrix, but in reality the analysis might show twice that number, so then we have to revisit the model and start building it up.

"There was a good example of this a few months ago, when we spent a little while being quite puzzled about the spectra we obtained at a pharmaceutical plant. After a bit of detective work, and using the power of GRAMS to build several spectral models, we came up with some far-fetched oxide of silicon. When we presented the information to the client, he said: 'Oh yes, we use that in the process – but it shouldn't be in the emissions' – but we'd found it there."

This sort of example shows the power of modern analytical technology and its potential for not only monitoring compliance with regulations but also as a plant maintenance indicator. The detection of such unexpected components could indicate a fault in the scrubbers or perhaps further upstream.

Andy Toy clearly revels in the power. "As far as I know, we are the only Company that can do the things we do: we can work on any batch chemical and pharmaceutical plant, incineration plant or just about any other industrial site, with a quick response and long monitoring periods. In the old days you would put a tube into the flow, suck out a sample for an hour and then send it off to the lab for analysis.

"Now we can do it continuously for weeks and with our ProtIR spectrometer and GRAMS software we can speciate out all the organics in the stream. Nobody else can do that."

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