The MicroAnalyst

ROYAL SOCIETY OF CHEMISTRY | MICROANALYTICAL GROUP

Issue Number 23
• Winter 2021•

Dear Colleagues,

Please accept our apologies for the lack of newsletters recently. Obviously it has been a very difficult period for everyone but now that we are starting to return to a normal existence we hope to resume our activities.

The committee has been having regular meetings over zoom, and are looking at arranging some guest speakers to appear. If you would like to join these meetings, have ideas for topics to discuss or would like to present a talk, please do get in touch for the link.

We also have a new YouTube channel where we will be hosting short videos on practical aspects of microanalysis.

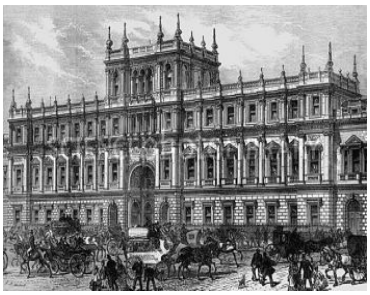
Finally, if you have any questions about the group meetings, wish to take part, or contribute to the newsletter then please do get in touch. We always very happy to hear from you all.

With best regards,

Richard Morris:
Editor, Microanalytical Group

The *London School of Hygiene & Tropical Medicine's* famous Isolation Chamber Orchestra.

NOTICE OF AGM



Tuesday, January 18th 2021

zoom

please contact us for the link

Contact Us

Contact information is on our RSC information page:

http://www.rsc.org/Membership/Networking/InterestGroups/Microanalytical/index.asp

MyRSC …http://my.rsc.org/groups/home/339

An inspirational life

Helen Murray Free 1923-2021

Helen Murray Free receives the National Medal of Technology and Innovation from President Barack Obama. (J. Scott Applewhite/AP)

Helen Murray Free was a chemist who with her husband Alfred Free developed the first dip-and -read diagnostic test for diabetes. Known as Clinistix in 1956.

She originally planned to be a teacher of English and Latin when the US entered the war, and it was suggested to her that there might be a shortage of women in science. She switched to chemistry at college – a move she described as “the most terrific thing that had ever happened to her”. After graduating she joined Miles Laboratories (later Bayer) as quality controller of vitamins but soon joined her husband to be in research.

She remained with Miles Labs until her retirement and won numerous awards and patents and wrote a book “Urinalysis in Laboratory Practice” with her husband. She encouraged many young people to follow chemistry as a career especially girls when she advised that “chemistry sets were for all children” when she was inducted into the National Women’s Hall of Fame in 2011.

Today, dip-and -read strips are routinely used as micro-chemistry tests (really semi-micro) for conditions ranging from diabetes, diseases of the liver, kidney and urinary tract; certain blood disorders and pregnancy.

Louise Dixon: Secretary, Microanalytical Group

Published and printed by the MicroAnalytical Group for its members

What can you analyse with less than 1mg of sample?

Emily Unsworth: Durham University, Dept of Chemistry.

This is a question that is sometimes asked when there is not much sample available and decisions have to be made as to which analysis to carry out, particularly for destructive techniques which will result in the irrecoverable loss of sample during the analysis.

Although CHN analysis usually uses a bit more than 1mg of sample, how much is needed can vary. For example, less sample is needed if it has a high CHN content which will give a larger signal. If less sample is available and it doesn't have a particularly high CHN content the instrument could be recalibrated to reflect the lower signal level by using a lower weight of standard. However this can only go so far as when the signal drops closer to the blank signal level the accuracy and precision of the results will be impacted.

Speaking to a colleague about weighing out small amounts of sample, they said that they often analysed small amounts of sample, going down to 0.2mg of sample if more sample wasn't available. The case they were thinking about was the stable isotope analysis of tooth enamel from archaeological samples. With a limited amount of sample and a destructive technique, they didn't want to use up more than they could help. As the results are quoted as a ratio of the isotopes measured in the sample, the weight of sample is not used in the calculation, as I understand it. The carbon in the sample is converted to carbon dioxide, which is then measured by a mass spectrometer that can detect the different carbon isotopes; unlike CHN analysis that converts the carbon to carbon dioxide but then measures it by a non-specific detection method such as thermal conductivity. For the isotope analysis a reference gas of a certified isotopic composition is used to measure the instrument performance.

Analysis of Nitrocellulose

Paul Hemming: Exeter Analytical (retired).

Nitrocellulose is the common name for a family of explosive materials based on the cellulose unit $C_{12}H_{20}O_{10}$. Included among these are pentanitrocellulose, dekanitrocellulose, and pyroxylin.

Historically, the nitrogen content of this compound, as well as other munitions, has been analyzed with a nitrometer using the standard method

MIL-STD-286B method T209.9. This method is not only labour intensive, but also exposes the operator to carcinogenic mercury vapours.

The Exeter Analytical CE-440 elemental analyzers use thermal conductivity detectors to simultaneously analyze the nitrogen, carbon and hydrogen content. Interferences are removed through column packings and the combusted sample is converted into non-toxic carbon dioxide, water vapor and nitrogen. These gases, along with the helium carrier, are vented during the cycle.

Previously, Exeter Analytical developed a method to successfully analyze the nitrogen content of a similar munition, pentaerythritol tetranitrate (PETN) on the CE-440. It was found that by reducing the available oxygen supplied to the sample, thus preventing gaseous oxides of nitrogen from forming, accurate results could be obtained. Using a slightly modified approach, they were able to achieve the same for nitrocellulose. The following conditions have been established to accurately determine the nitrogen content in nitrocellulose compounds. This procedure also provides consistent carbon and hydrogen data.

* Combustion time - 28 seconds (eliminates one oxygen burst and thus provides a reduced oxygen environment).
* Combustion temperature - 1000^{0}C
* Reduction temperature - 595^{0}C
* Oxygen pressure - reduced to 15 psi
* Tin capsules (P/N 6703-0418), nickel sleeves (P/N 6703-0499) for sample containment
* Broad spectrum combustion aid (P/N 650-00008) added to sample, 8-10 mg per sample. Combustion aid added to capsule first and tared out. Sample is added on top, capsule sealed, then agitated to mix the sample with the combustion aid.

Comparison of the data obtained using the CE-440 Elemental Analyzer versus the standard method (MIL-STD-286B method T209.9)

SAMPLE	CE-440 % NITROGEN	STANDARD METHOD % NITROGEN
1	13.11	13.12
2	12.65	12.6
3	13.25	13.16
4	13.21	13.19
5	13.18	13.09