MaSC

Users' Group for **Mass Spectrometry and Chromatography**

MaSC Meeting

Van Gogh Museum
Amsterdam, The Netherlands

9 - 10 September 2005

Preface

We would like to welcome you to the second meeting of the Users' Group for Mass Spectrometry and Chromatography (MaSC).

Since MaSC was founded at the 'Discussion Meeting on Binding Media Identification in Art Objects' at the Netherlands Institute of Cultural Heritage, Amsterdam, in 2003 and our first workshop and meeting at the National Gallery of Art, Washington DC, in 2004 several developments have taken place which will be presented at this meeting.

Our initial mission was to explore the possibility for creating a mass spectral database and to establish a forum for encouraging information exchange among those using mass spectrometry and chromatography techniques in the field of conservation science.
We have established a format for the compilation of mass spectral data and, with your input, will soon have a database ready for distribution. At this meeting, we will present the details of the submission process as well as plans for the upcoming web-based procedure and database.

This meeting also offers a forum for information exchange, and a range of topics will be covered in the presentations, abstracts of which can be found in this programme. Although in the beginning the Group was comprised largely of scientists involved in the study of works of art, an increasing number of lectures demonstrate that scientists engaged in archaeometrical research are becoming an important part of our Users' Group.

We thank our colleagues from the Infrared and Raman Users' Group - Beth Price, Boris Pretzel and Janice Carlson - for sharing their invaluable experience in establishing a users' group, and to Dr. Steve Stein (NIST) and Oliver Stahlmann (University of Applied Sciences Cologne, Department of Conservation) for their help in setting up the database.

We would also like to extend our thanks to AMOLF, ICN and the staff of the van Gogh Museum for their assistance in the organisation and hosting of this meeting, which we hope will be both inspiring and thought provoking. As ever, we thank you for your support and will welcome your feedback on the meeting, and your continued input on the activities and direction of the Group.

The organizing committee:
Ester Ferreira
Klaas Jan van den Berg *(European Coordinator)*
Christopher Maines *(North American Coordinator)*
Catherine Higgitt
Ken Sutherland

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HPLC and MS signatures for ancient Andean textile dyes: Initial progress on establishing protocols and shared libraries

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10.50 **Shuya Wei *et al.* (Vienna University of Technology, Vienna):** Effects of pigments and accelerated ageing on drying and degradation of oils as binding media.

11.15 **Shirly Montero (Netherlands Forensic Institute, Den Haag)**: Elemental profiling with LA-ICP-MS for glass and automotive paints.

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Users' Group for **Mass Spectrometry and Chromatography**

Abstracts

DE-MS analysis of triterpenoid resinous materials in archaeological findings.

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Chemical studies on resinous materials in archaeological remains are generally aimed at identifying the origin of the material, at studying the production and manufacturing techniques, and at improving techniques for conservation, restoration and storage procedures in museums.

This paper presents the most significant results obtained for a series of terpenoid resin materials by using a fast analytical volatilisation/pyrolysis technique, namely direct exposure mass spectrometry (DE-MS). This technique allows sample manipulation and analysis time to be minimized, and in a few minutes provides a mass fingerprint of organic material which highlights the classes of compounds that are the main components in the archaeological organic residue.

The ability of DE-MS technique was tested to distinguish not only between several different organic materials as triterpenic resins, diterpenic resins and beeswax, but also between different triterpenoid materials such as mastic, frankincense and birch bark tar. Despite the complexity of the chemical composition of raw materials, DE-MS was found to be a rapid and efficient tool for identification purposes.

The chemometric evaluation of mass spectral data by DE-MS has been performed by principal component analysis (PCA). The pattern analysis permitted to compare mass spectra of triterpenoid materials highlighting differences and similarities between samples, and to identify the more significant mass fragments for their characterisation.

The paper also reports applications to characterise organic residues in archaeological objects such as in an Egyptian censer (V-VII cent. AD) from the Necropolis of Antinoe, and in a Palaeolithic lithic tool recovered in the site of Campitello (Arezzo, Tuscany).

Possible roads to the identification of organic residues in archaeological ceramics

Hans Barnard
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Among the focal points of archaeological ceramic analysis are the provenance and the function of ancient vessels. Determining the origin of the raw geological materials can be facilitated by creating fingerprints of the ceramic matrix using trace elements or selected stable isotopes. In favorable circumstances the golden standard for this, instrumental neutron activation analysis (INAA), can be replaced by inductively coupled plasma mass spectrometry (ICP-MS) after either chemical digestion or laser ablation.

The former contents of unglazed vessels have often left organic residues attached to the ceramic matrix. The study of such residues has mainly concentrated on analyzing the lipids by gas chromatography and mass spectrometry (GC/MS). Methods to better understand these residues include comparing fatty acid ratios, searching for marker molecules, and establishing stable carbon and nitrogen isotope ratios. A summary will be presented of the contributions to a symposium on this subject, during the Annual Meeting of the Society for American Archaeology, on 31 March 2005 in Salt Lake City (Utah).

Full characterization of poly(ethylene glycol) based additives in acrylic paints by matrix-assisted laser desorption/ionisation mass spectrometry and nano-electrospray ionisation mass spectrometry.

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2 Tate Gallery, Mill Bank, London, United Kingdom

Acrylic paints are complex systems that contain water, acrylates, pigment and numerous additives. One important additive is based on poly(ethylene glycols) (PEG). It acts as a surfactant with a hydrophilic tail and a hydrophobic head group. The polymer plays an important role in the stabilization of acrylic polymers in the aqueous phase. However, the surfactant properties become obsolete when the paint dries. Water evaporates and the acrylic particles fuse together to form a film. From this point it is not well understood what will happen with the PEG compounds in the paint. Recent cleaning studies shows that PEG compounds migrate to the surface of the paint film[1], giving it a matt appearance. Another study showed the formation of small inclusions in an unpigmented acrylic paint film[2].

These problems may be due to the chemical properties of the PEG compounds. A detailed structural analysis of these components is therefore necessary. This will be the focus of this study. Mass spectrometry was used to determine the distribution of the oligomeric/polymeric components and end-group properties.

Both dry and liquid acrylic paint were used in this study. Liquid samples included base emulsions, modified emulsions, varnishes and paints. They were painted on aluminium foil and dried for two weeks. Dry paints included paint films, samples from a palette and samples from paintings. The samples were extracted with water for two weeks. Matrix-assisted laser desorption/ionisation mass spectrometry was used to determine the polymeric properties, the molar mass distribution. Mass distributions found vary between 500 and 4200 Dalton. Nano-electrospray ionisation mass spectrometry in combination with nozzle skimmer dissociation and tandem mass spectrometry allowed end-group identification. The end-groups found include: octylphenyl, nonylphenyl, hydroxyl and sulphate.

Combining the molar mass distribution and end-groups allowed the complete characterisation for each PEG compound found in the water extracts.

1 Digney-Peer S.; Burnstock, A.; Learner, T.; Khanjian, H.; Hoogland, F. and Boon, J. *The migration of surfactants in acrylic emulsion paint films on curing, aging and after selected surface treatment* paper presented at 20th IIC Congress, Bilbao, Spain, 2004.

2 Whitmore, P.M.; Colaluca, V.G. and Farrell, E. *Stud. Conserv.* 1996, *41*, 250-255.

HPLC and MS signatures for ancient Andean textile dyes: Initial progress on establishing protocols and shared libraries

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Textiles were valued and prized by the inhabitants of the Ancient Andean (South American) cultures. Decorated with complex designs in a range of colours and dyes, these were important objects that are now key for our understanding of ancient life in this continent. They are also considered some of the most significant art works of the ancient world. Research of dyes used for the creation of the elaborate designs progressed in sporadic fashion during the past hundred years and has been primarily based on the UV-vis spectrometry. Only limited HPLC had been done an all research has been dependent on the limited range of possible known standard collections. This paper will discuss attempts to employ contemporary physical characterisation methods for component separations and characterisation. Particular emphasis will be placed on mass spectrometry to generate biosignatures and positive identification Andean dyes, even when comparative biological standards are not available. The presentation will also explore investigations into the creation of easy to follow protocols for dye extraction and analysis that can be replicated in our labs. Our work is at its initial stages, and we will present proposals for method standardization and file sharing for comparative purposes.

ıNvestigation of mixed techniques by GC/MS and AAA

A case study on Vermeer's 'The Procuress' from the Dresden Gallery

Ursula Baumer, Irene Fiedler and Johann Koller
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According to Giorgio Vasari the historical (egg) tempera medium in Italian pictorial art has been replaced by an oil medium in the second half of the 15th century. A clear distinction was made later on in historical art between oil and tempera painting. As a consequence, whenever during modern binding media analysis drying oils were found, the whole painting was accepted as an oil based one. Since one could not imagine the existence of oil and tempera media side by side, analysis for additional binding media were only performed in exceptional cases. Our latest binding media analyses, however, revealed different results, and 'mixed techniques' were often detected.

For example, Vermeer's painting 'The Procuress' from the Gallery of the Old Masters in Dresden (Zwinger), which was generally accepted as an oil painting, contained oil and tempera media as well. By gas chromatography/mass spectrometry (GC/MS) analysis high amounts of a boiled linseed oil media were identified in all paint layers. Although the fatty acid profiles by GC/MS gave not even a slight hint to lipids from egg, in all investigated Vermeer paint layers an additional tempera medium was found by amino acid analysis (AAA). Since we were able to distinguish between egg yolk and egg white (with AAA), we could show, that egg white was used as a second medium in the light colours (like blue) and egg yolk as a second medium in the dark colours (like red and the ground).

Structural characterisation of archaeological resins using mass spectrometric and chromatographic procedures

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A series of several pieces of natural resins along with potsherds characterised by the presence of charred surface residues were recently discovered at the site of Sharma, located on the south coast of Yemen (10^{th}-12^{th} century AD).

Although the samples were supposed to contain frankincense that is known to be naturally produced in the region of interest, nothing was known on their exact nature. The first step of the research consisted in analysing the samples by direct inlet electron ionisation mass spectrometry to obtain a spectral fingerprint. The mass spectra were recorded on a Finnigan GCQ mass spectrometer equipped with an ion trap analyser. The spectra obtained show that two kinds of resins were exploited at Sharma: triterpenoid resins (base peak at *m/z* 189, 203 or 218, depending of the samples and molecular peaks at *m/z* > 400) in the ceramic vessels and a resin with a spectrum close to that of copal, a diterpenoid polymerised resin. This latter material was identified in most of the raw pieces of resin.

These preliminary results by EI-MS allowed us to choose the analytical conditions adapted to the properties of such resins by GC techniques: solvent soluble triterpenoid resins were analysed after extraction and derivatisation by GC/MS whereas resins close to copal were analysed by Py-GC/MS after TMAH treatment.

Boswellic acids were then identified in triterpenoid resins by the study of their mass spectra, indicating that frankincense was manufactured in some vessels at Sharma. Py-GC/MS provided pyrograms and mass spectra characteristic of copal from Madagascar.

Beyond the structural characterisation of resins by EI-MS, GC/MS and Py-GC/MS, these results shed new light on the commercial roads of resins at the site of Sharma since copal was probably imported from Africa whereas frankincense is produced by a species that naturally grows in Yemen.

New approaches in the GC-MS investigations in the Kunsthistorisches Museum Vienna

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The specific character of the Kunsthistorisches Museum collection with its variety of outstanding art and historical objects from different periods results in broad responsibility for staff of the single collections and the conservation departments, and therefore, in 1998 the Conservation Science Department of the Kunsthistorisches Museum was established. With its research programs involving both organic and inorganic analyses of art objects the department assists with scientific information on e.g. the painting technique and alteration due to former restoration treatments, the development of the new conservation treatments, or the preventive conservatoin measures within the museum's collection.

The installation of a GC-MS system in 2002 with the financial support of the Austrian Science Fund allowed the department to increase and extend its research activities to the field of organic materials analysis. A series of analytical procedures for the investigation of binding media composition (lipids, resins, waxes, proteins, and polysaccharides) was optimised and already applied to a variety of samples from works of art, which vary from analysis of binding media from Old Masters paintings, analysis of varnish from bronze sculptures, lacquer analysis from antique music instruments, binders from wall paintings to paint layers analysis of Baroque furniture.

For better understanding the changes in the composition of organic matrix caused in time an accelerated ageing under controlled conditions (UV, temperature and relative humidity) has been also introduced and studied on a set of pigmented reference standards.

A combined GC-MS analytical procedure for the characterisation of drying oils, waxes, natural terpenoid resins, pitch, tars and proteinaceous materials in a unique microsample from painted works of art

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Samples from painted works of art are generally characterised by the concomitant presence of several different organic materials together with pigments and other inorganic components (support, metal leaf). The organic content is generally quite low compared to the inorganic content. Moreover, since any damage to the artwork needs to be kept to the absolute minimum, samples have to be very limited in size (frequently less than 100 mg). All these reasons represent a challenge in the characterisation of lipids, waxes, proteins and resinous materials that may be simultaneously present in the same sample.

A novel analytical procedure for the identification of the above mentioned materials in the same paint micro-sample is presented. It is based on a sample pre-treatment that is able to separate the various organic components into different fractions, which are suitably treated and derivatised before being analysed by means of GC-MS. In particular, this method achieves :

- the identification of the proteinaceous binder (egg, collagen, casein) on the basis of the quantitative determination of the amino acid profile submitted to the principal component analysis together with the data obtained from reference samples;
- the identification of lipids (linseed oil, poppy seed oil, walnut oil and egg), plant resins (*Pinaceae* resins, sandarac, mastic and dammar), animal resins (shellac), tar or pitches and natural waxes (beeswax, carnauba wax) on the basis of the determination of fatty acid, alcohol and hydrocarbon profiles and of significant terpenic molecular markers.

Recent dye analysis research at the National Museums of Scotland

Anita Quye
Department of Conservation and Analytical Research, National Museums of Scotland, Edinburgh, United Kingdom

Over the past eighteen months, dye analysis at the NMS has involved method development and textiles collection studies as well as the completion of a European collaborative project, Monitoring of Damage to Historic Tapestries (MODHT).

The NMS' routine gradient HPLC-PDA method for natural dyes has been improved following the evaluation of different reversed phase stationary phases. This work has been complimented with preliminary studies to improve the extraction of indigoid dyes and the analysis of lichen dyes. Collection studies have included seventeenth century textiles excavated from Scottish peat bogs and eighteenth century Scottish tartans for women's wear.

The results from these studies, along with the latest HPLC-PDA and LC-MS outcomes from MODHT research in collaboration with the University of Edinburgh's School of Chemistry, will be presented.

Rapid analysis of exhibition materials using solid phase microextraction with on-fiber derivatization

Christopher A. Maines
Scientific Research Department, National Gallery of Art, Washington, USA

Solid phase microextraction (SPME) coupled with gas chromatography / mass spectrometry (GC/MS) overcomes many of the shortcomings of the conventional methods for analyzing evolved gasses: identification, rapid sampling, and quantitation. SPME with on-fiber derivatization allows for simultaneous analysis of all species of interest and can result in stable derivatives with on-fiber lifetimes at room temperature of several hours allowing for remote sampling followed by later chromatographic analysis

The so-called “Oddy Test” has long been used as a non-specific test for damaging airborne pollutants, but takes 28 days to complete and is notoriously susceptible to operator error.[1] SPME has been in use since the early 1990’s for the analysis of trace amounts of volatile compounds[2] and since the late 1990’s with on-fiber derivatization for the analysis of gaseous compounds.[3] The latter is particularly suited for analysis of small organic pollutants of concern to conservators and conservation scientists. The derivatizing reagent PFBHA reacts with aldehydes to form stable oximes that can be analyzed using standard GC conditions.

GC analysis of samples introduced via solution injection or pyrolysis will not necessarily distinguish among compounds that will be off-gassed from those that will remain in the material. A sample of asphalt that had been intended for a natural history museum was shown to off-gas appreciable amounts of aldehydic compounds. The “Oddy Test” for the asphalt had been inconclusive, while pyrolysis-GC/MS was unable to show which compounds within the asphalt could cause damage to surrounding objects.

[1]Green, L.R. and D. Thickett, Testing materials for use in the storage and display of antiquities - a revised methodology. Studies in Conservation, 1995. 40: p. 145-152.
[2]Zhang, Z. and J. Pawliszyn, Headspace solid phase microextraction. Analytical Chemistry, 1993. 65: p. 1843-1852.
[3]Martos, P.A. and J. Pawliszyn, Sampling and Determination of Formaldehyde Using Solid-Phase Microextraction with On-Fiber Derivatization. Analytical Chemistry, 1998. 70(11): p. 2311-2320.

Effects of pigments and accelerated aging on the drying and degradation of oils as binding media

Shuya Wei, Erwin Rosenberg
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Oils including linseed oil, linseed standard oil, poppy seed oil and walnut oil present in paintings can be identified by gas chromatographic analysis of their derivatives formed with trimethylsulfonium hydroxide (TMSH) as derivatization reagent. With parallel flame ionization and mass spectrometric detection, a systematical study of the effects of pigments under the conditions of accelerated aging on the drying and degradation of oils as binding media has been conducted.

Surprisingly, for some pigments including titanium white, chalk, yellow ochre and burnt umber, not supposed to have much effect on drying and degradation of binding media according to the literature, different results were found. In addition to this, suberic acid and azelaic acid are claimed to be the degradation products, but the concentration of neither of them increased during the aging process, even though oleic acid apparently has a decreasing trend in most of the samples.

Although the palmitic acid to stearic acid (P/S) ratio of linseed and linseed standard oil is stable during the accelerated aging process, the P/S ratio of walnut and poppy seed oil obviously has a declining trend during aging. From this observation, it can be deduced that the differentiation of oils based merely on their P/S ratio can be misleading under conditions of serious deterioration.

Elemental profiling with LA-ICP-MS for glass and automotive paints.

Shirly Montero
Netherlands Forensic Institute, The Hague, The Netherlands

At the Netherlands Forensic Institute, elemental analysis by LA-ICP-MS for characterisation of materials of forensic interest has an important place. This technique combines the sensitivity and precision of ICP-MS with the advantages of laser ablation sampling. The amount of material ablated for the complete analysis is very small (~ng), allowing the analysis (including replicates) of very small samples. The destruction is minimal with craters in the order of 10^{-9} m^2, in contrast to the solution approach where the whole sample to be analysed is irreversibly digested. In addition, with this sampling technique depth profiling as well as surface profiling is possible.

The system used at the institute is an ICP-MS (Perkin Elmer ELAN6100 DRC) in combination with a 3 mJ- 213 nm-laser ablation system (New Wave UP-213). Two applications have been selected for this presentation, quantitative analysis of glass and qualitative analysis of automotive paints. The method for the analysis of glass has been used already in the general scheme for casework, in addition to the traditional analyses such as the refractive index measurements and semi-quantitative µXRF measurements. The methods, the selection of the matching criteria and the results of the comparisons of the samples using elemental concentrations and refractive index (measured with GRIM II) will be discussed. Finally, the development of a method for the comparisons of automotive paints, using depth profiling as well cross sections, will be also discussed.

Quantitative comparison of the derivatization efficiency for drying oil paint related substances using different reagents

Masahiko Tsukada
The National Museum of Western Art, Japan

Poster

The efficiency of different reagents and methods for the derivatization of oil paint related substances has been studied comparing the quantitative yields of methyl ester of fatty acids with gas chromatography / mass spectrometry. The following derivatization methods and reagents were compared; thermally assisted hydrolysis and methylation (THM) with tetramethylammonium hydroxide (TMAH), (m-trifluoromethylphenyl) trimethylammonium hydroxide (TMTFAH), trimethylphenylammonium hydroxide (TMPAH), and trimethylsulfonium hydroxide (TMSH), and conventional saponification with KOH in methanol and methylation with diazomethane (DM) or TMS-diazomethane (TMSDM).

The substances studied were free fatty acids (2C9, C16:0, C18:0, C18:1, C18:2, and C18:3), triglycerides of each mono acid, metal soap (stearate salts of zinc and lead), raw linseed oil, and lead white / linseed oil paint sample dried for 10 years.

The injection of the samples to GC was carried out by splitless mode, and for the THM method the methylation reaction was took place in the injector heat block of GC. The highest yields of fatty acid methyl esters (FAMEs) were obtained by THM with TMTFAH for all samples. TMAH gave FAME yields to the same extent as TMTFAH for saturated fatty acids, but was less efficient for azelaic acid and the base catalysed and thermally induced isomerization of polyunsaturated fatty acids occurred.

The yields of FAMEs with TMPAH and TMSH were smaller than with TMTFAH, and TMPAH seemed to isomerize polyunsaturated fatty acids. TMSDM and DM showed the efficiency for the methylation of free fatty acids similar to TMTFAH and TMSH respectively, but the yields of FAMEs decreased for other substances probably because of the sample losses during the procedure.
For all methods, fatty acids in the metal soaps were more difficult to derivatize than other samples.

Analysis of the binding media of two painting on metal using GS-MS

Kamilla B. Kalinina, Marina. E. Ilyina
State Hermitage Museum, St.-Petersburg, Russia

Poster

The application of gas chromatography/mass spectrometry has made it possible to obtain more information about complex organic materials used by artists. The examination of painting materials of two pictures on the metal from the interior of the Gallery of the History of Ancient Painting of the Hermitage Museum permitted to compare painting technique of German and Russian schools. One of the pictures was created in studio of Hiltensperger in Munich, another one was made in studio of Hosroe Duzzi in St. Petersburg Academy of Fine Arts after the model of drafts of Hiltensperger.

As part of a technical examination accompanying the recently fulfilled restoration of these two paintings binding media were analyzed by using gas chromatography/mass spectrometry. Samples were analyzed after derivatization by methanolic (m-trifluoromethylphenyl) trimethylammonium hydroxide (Meth Prep II). The transesterification products of autoxidized oils in the form of fatty acid methyl esters were examined by GC-MS. Using Meth Prep II, the acidic components of diterpenoid and triterpenoid resins are also methylated.

The cross-sections of samples from the several places of both pictures show that structure of color layers was different. There were three-four of color layers on cross-sections of the picture made in German and five color layers on cross-sections of the picture made in Russia. In the second case color layers were considerable thicker. Pigment composition differed too. GC-MS analysis of binding media of each layer was carried out.

Results of examination of picture made by German artists showed the presence of a beeswax, small amount of rosin and, perhaps, copaiba balsam in upper and lower layers. We observed the presence of asphalt in these layers too. The intermediate thin layer consists of stearin wax.

GC-MS analysis of binding media used by Russian artists indicated the presence of considerable amount of rosin in the very thin lower layer. The dried oil and asphalt were determined in this layer too. We found out a presence of a rosin as main component with addition small amount of wax in two intermediate layers. GC-MS analysis of binding media of two upper layers showed the presence of beeswax with traces of diterpenoid resin.

Our investigation of the composition of binding media showed the difference in painting technique of German and Russian artists and allowed to choose the correct approach to restoration of these objects.

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MaSC

Users' Group for **Mass Spectrometry and Chromatography**

Database

Procedure for generating MaSC JCAMP-DX mass spectral data:

There are two parts to a JCAMP-DX file – 1.) detailed header information about the sample itself, the instrumental conditions, the submitter, and the data processing, and 2.) the mass spectral data in X, Y format. Currently, the preferred method of submission is the **MaSC Mass Spectrum Template** (a MS Excel file), which is available on the MaSC website: http://www.mascgroup.org/datasubmission.html. You will also need an ASCII text editor in preparing your mass spectral files for submission. Notepad or Wordpad, which come standard with Microsoft Windows, will work but are not recommended. If you don't have a good ASCII text editor, try Notetab Light (http://www.notetab.com) which is freeware.

You will need to have previously saved the mass spectral data you wish to submit as an ASCII text file containing x,y values, where x corresponds to mass, and y corresponds to the intensity at that mass. There are instructions on the MaSC website (http://www.mascgroup.org/datasubmission.html) for several commonly used data collection systems. We apologize if your system is not represented, and if you can supply a procedure for getting x,y data from your system's datafiles we will gladly include it on the MaSC website.

General Notes on JCAMP-DX files:

1. Basic structure:
   A. A JCAMP-DX file consists of a series of linked data records (fields).

   B. Data records are stored in one of three types of field: a *generic labeled data record* (LDR), a *data-type-specific record*, or a *user-defined labeled record*. Only the *user-defined labelnames* are modifiable by MaSC.

   C. General JCAMP: The *generic labeled data records* have the format: ##labelname= dataset
   MS specific: The *data-type-specific records* have the format: ##.labelname= dataset
   MaSC specific: The *user-defined labeled records* have the format: ##$labelname= dataset

   D. The first three data records must be ##TITLE=, ##JCAMP-DX=, and ##DATA TYPE= in that order. The file must terminate with ##END=

2. Data records may be no longer than 80 characters per line (including the label), but are allowed to continue on subsequent lines.

3. Comments, which are ignored by data importers and translators, begin with $$ or ##=, and must finish at the end of the line on which they start.

4. Data records can be in any order with the exception of ##TITLE=, ##JCAMP-DX=, ##DATA TYPE=, and ##END= as described above.

INSTRUCTIONS:

1. Open the Excel template and fill in the datafields/metadata tags (rows 2 through 46) as completely as possible. Refer to the notes in the table below and in column C of the Excel template as an aid. You might want to re-save

the Excel template at this point to preserve information that will be used in all your mass spectral submissions.

2. Insert your x,y mass spectral data starting in cell C47, which is marked with a red border. This can be accomplished in at least two ways described below. When finished, the x values should be in column C and the y values should be in column D.
    either:
    a. With the x,y mass spectral data already open in an ASCII text editor, simply highlight the x,y data only, and then select Edit, and then Copy. Click on cell C47 in the Excel template, and then select Edit, and then Paste.
    or:
    b. From within Excel, select File, and then Open, and then navigate to the directory where the x,y mass spectral data files are stored. Make sure to select the appropriate "Files of type:" in the dialog box to enable Excel to see the data files, and double-click on the appropriate data file to open it. You'll be confronted with the text import wizard, which will allow you to select the first x,y data line in the file, and to select the appropriate type of delimiter that separates your x and y values: space, tab, comma, etc. Once you've clicked "Finish," the x,y data will open in an Excel file with the x values in column A and the y values in column B. Highlight these x,y values, select Edit, and then Copy. Click on cell C47 in the MaSC Excel template, and then select Edit, and then Paste.
3. Edit the formula in cell B47 Locate the last y value in column D, and note its corresponding row number. Substitute this row number for "row 1000" in the formula. For instance, if the last y value in column D is in row 193, then change D$1000 to D$193.
4. Next you need to transfer the y values in column D as scaled values into column B as described in a. through c. below.
    a. Copy cell B47, by highlighting it, and then selecting Edit, and then Copy.
    b. Highlight all the cells in column B starting with cell B48 and ending with the cell in column B with the same row number of the last y value in column D. For example, if the last y value in column D is in row 193, then you would highlight all the cells in column B from B48 to B193.
    c. Select Edit, and then Paste. The y values in column D should now be in column B, but scaled appropriately. The base peak will be set to 999 and all other y values will be scaled to this peak and rounded to the nearest whole number.
5. Transfer the x values in column C to column A as described below:
    a. Highlight all the x values in column C, starting with cell C47, then select Edit, and then Copy.
    b. Highlight cell A47, and then select Edit, and then Paste.
6. You then need to remove extraneous information from the file which is done by selecting only the data in columns A and B as described below:
    a. Highlight the column headings A and B which will select everything in columns A and B.
    b. Select Edit, and then Copy.
    c. Switch to the blank worksheet 3 by clicking on the tab labeled "Sheet3" near the bottom of the screen.

    d. Highlight cell A1, then select Edit, and then Paste Special, and then select Values, and then select OK. The columns might have to be resized to show their full contents.
7. Next save the resulting spreadsheet with the name of the mass spectrum, as a tab-delimited ASCII text file:
    a. Select File, and then Save as …., and then be sure to select *Text (Tab delimited) (*.txt)* in the dialog box labeled "Save as type:"
    b. Enter the name of the mass spectrum as the filename, and select Save.
    c. Answer OK to the dialog box that pops up stating that the selected type does not support multiple worksheets. We no longer care about the other worksheets in this file.
    d. Answer Yes to the dialog box that pops up stating that the file you are about to save has features that are not compatible with tab delimited text. We don't care about losing "special features" that can not be saved as ASCII text.
8. Open the resulting ASCII text file in a text editor (see note in the introduction regarding suitable text editors, Notetab is recommended). It is then necessary to remove the tabs that will have appeared in the metadata/data fields. The quick method for doing this if you are using Notetab is described below:
    a. Within Notetab, highlight all the rows up to, but not including, the actual x,y data.
    b. Select Search, and then Replace.
    c. Enter ^T into the "Find what:" box and simply click in the "Replace with:" box. Now click on "Replace All"
9. Now use the first line of the file, the number line, to determine where lines need to be broken to conform to the 80 characters per line maximum. Once the lines have been corrected (by pressing the Enter key to enter a "carriage-return" to break lines as appropriate), remove the number line, and make the ##TITLE= line the first line in the file.
10. Add ##END= as the final line in the file.
11. Resave the completed file.
12. Check that the file has been formatted properly and contains the correct data by using one of the freely available JCAMP-DX viewers available on the web, such as MSView32 (http://merian.pch.univie.ac.at/pch/download/spectroscopy) or WSearch32 (http://www.wsearch.com.au)
13. Submit both the MS Excel file of the mass spectrum as well as its associated JCAMP-DX file to MaSC. Rename each Excel file and its associated JCAMP-DX file identically – the only difference will be that the Excel file should have the extension .xls and the JCAMP-DX file should have the extension .dx

J-CAMP Field Name	Contents [brief description of contents]	Notes on contents
##TITLE=	[first 8 characters assigned by MaSC]; [Sample name]	This must be the first line in any J -CAMP file. The name of the compound should be entered here, and will most often be the common or trivial name. Once accepted into the MaSC library, the editing committee will insert an 8 character indexing number.
##JCAMP-DX=	5.01 [Version number of JCAMP -DX used, invariant]	This must follow the ##TITLE= line. We are using the field definitions from J-CAMP version 5.01
##DATA TYPE=	MASS SPECTRUM	This is the designation for a single mass spectrum. This should remain as is.
##DATA CLASS=	XYDATA	This denotes a set of x,y data. This should remain as is.
##ORIGIN=	[Analyst name, Institut ion name, address, phone, fax, e-mail]	At minimum, the analyst name, institution name, and institution address should be entered here. Additional contact details are welcomed, however.
##OWNER=	COPYRIGHT (C) [Year] by [Institution Name]	Enter the year the spectrum was submitted as well as the institution's name. The submitting institution retains copyright over the spectrum.
##$LICENSE=	[a licensing statement to be determined at a later date]	This is a field defined by MaSC, and not by J -CAMP. This statement will include words to the effect of "user of this library agrees to abide by MaSC user's license, contributor agrees to abide by the MaSC contributor's license, any reference made to this spectrum oral or written should credit both the contributing individual/institution and MaSC."
##$INSTITUTION FILENAME=	[originating institution's filename]	Enter the filename, including extension, under which the data were originally saved by the analyst. This will serve as a reference if the original data need to be accessed at a later date. This is a field defined by MaSC, and not by J-CAMP.
##SAMPLE DESCRIPTION=	[composition or origin, collection date, state (solution/solid), etc.]	Enter a brief description of the sample analysed (include information such as source/supplier, age, physical state, or purity).
##SAMPLING PROCEDURE=	[no pretreatment, no derivatisation, derivatisation (diazomethane, BSTFA, Methprep I/II, TMAH, BF3, etc.)]	Enter a brief description of all sample preparation before introduction to any instrumental analysis. Include derivatising reagents, reaction times, solvent, etc. Sample quantitation, such as mass, volume, and/or concentration, can be included here. *No pretreatment* would be appropriate where online (in situ) pyrolysis/methylation is carried out as part of the instrumental analysis, or for direct insertion mass spectrometry with no prior sample pretreatment.
##TEXT=	YES [if the spectrum has been published]; NO [if the spectrum is unpublished]	Future plans: complete bibliographic information for published spectra available on www.mascgroup.org .
##LONG DATE=	[YYYY/MM/DD HH:MM:SS.SSSS ±UUUU]	The date YYYY/MM/DD is required. The precise time is optional. For example, 2004/06/28 09:55:49 or 2004/06/28 are acceptable.
$$ Equipment		

J-CAMP Field Name	Contents [brief description of contents]	Notes on contents
##.INLET=	GC (gas chromatograph), LC (liquid chromatograph), DIRECT (direct insertion probe), BATCH (batch inlet), CZE (capillary zone electrophoresis system), PB (particle beam inlet), MOB (moving belt inlet), etc.	Enter the standard abbreviation for the instrument that introduces the sample into the mass spectrometer. The inlets named here are only a partial list.
##INSTRUMENTAL PARAMETERS=	[For GC: column brand, stationary phase, i.d., o.d., film thickness, column length, pressure/flow control, temperat ure program, carrier gas, flow rate, injection parameters (pyrolysis, split/splitless, on -column), etc.]	All instrumental components and parameters with reference to the INLET only. Mass spectrometer parameters are entered in subsequent fields. Mass spectr ometer details may be entered here, if such information is not included in subsequent fields. If no GC, LC, pyrolysis equipment, etc. was used, leave this field blank.
##SPECTROMETER/DATA SYSTEM=	[Manufacturer and Model of Mass Spectrometer and of inlet s ystem, software system(s)and version(s)]	The J-CAMP definition only includes the name of the mass spectrometer and data collection hardware/software. MaSC includes the name of the inlet system (GC, LC, etc.) to prevent defining an additional field.
##.SPECTROMETER TYPE=	Q (quadrupole), TRAP (ion trap), TOF (time -of-flight), B (magnetic sector field spectrometer), BE or EB (double -focussing spectrometer), etc.	Designation of the mass spectrometer type
##.SOURCE TEMPERATURE=	[temperature of ion source in °C]	Do not include units.
##.IONIZATION MODE=	EI+/– (electron impact), CI+/ – (chemical ionisation), FAB+/ – (fast-atom bombardment), TSP+/ – (thermospray), ESI+/– (electrospray), APCI+/ – (atmospheric pressure chemical ionisation), LD +/– (laser desorption)	Designation of the mass spectrometer ionization mode
##.IONIZATION ENERGY=	[ionization energy in eV]	
##.AQUISITION RANGE=	[lowest mass, highest mass in amu]	This refers to the acquisition range of the current spectrum only, and not to the range the mass s pectrometer is capable of. Do not include units and separate values by a comma
##.SCAN RATE=	[scan rate in scans/sec, masses/second or seconds/decade with units defined]	Enter the scan rate, with units (scans/sec is defined by MaSC).
##RESOLUTION=	[mass resolution with units]	
##$THRESHOLD=	[threshold value for data acquisition]	Enter the threshold value for data acquisition if applicable.
$$ Compound Information		
##CAS NAME=	[if known]	Chemical Abstracts Service (CAS) name if known.
##NAMES=	[common names]	Include synonyms, common or trivial names or other chemical names

J-CAMP Field Name	Contents [brief description of contents]	Notes on contents
##MOLFORM=	[molecular formula]	The molecular formula should be given in Hill Order, i.e. for organic compounds, C is listed first, followed by H, followed by the remaining elements in alphabetical order, e.g. palmitic acid, trimethylsilyl ester = C19H40O2Si
##CAS REGISTRY NO=	[CAS number]	Chemical Abstracts Service (CAS) registry number if known
##MW=	[molecular weight, to two decimal places]	Molecular weight is defined as the relative molecular mass averaged over all isotopes and can be easily calc ulated using various free web -based calculators such as http://www.ch.cam.ac.uk/magnus/MolWeight.html or http://medlib.med.utah.edu/m asspec/mole.htm
##.MONOISOTOPIC MASS=	[accurate weight of the most common isotope peak, to two decimal places]	Monoisotopic mass (also referred to as the HRMS (high resolution mass spectrometry) mass) is the accurate mass of the molecule/ion containing only the most common elemental isotopes. It is obtained by summing up the masses of the most abundant isotopes of all elements in the molecule/ion. The monoisotopic mass can be easily calculated using various free web -based calculators such as http://www.ch.cam.ac.uk/magnus/MolWeight.html or http://medlib.med.utah.edu/masspec/mole.htm .
##.NOMINAL MASS=	[nominal mass, integer value]	Nominal mass is the sum of the integer atomic masses of the most common isotopes of the elements in the molecule, that is H=1, C=12, N=14, O=16, F=19, Si = 28, P = 31, S = 32 etc. See http://www.cem.msu.edu/~reusch/OrgPage/mass.htm for table of nominal masses. The calculator at www.ch.cam.ac.uk shows the nominal mass as the first integer value in the table giving the “molecular ion isotope pattern.” A nice example of the different values for these three masses is given by Ubiquitin (C378H630N105O118S) where MW = 8565.89, monoisotopic mass = 8560.62 and nominal mass = 8556.
$$ Spectrum		
##.SCAN NUMBER=	[scan number(s) of spectrum]	Enter scan number of submitted spectrum
##.RETENTION TIME=	[retention time in seconds]	Convert retention times in minutes:seconds to seconds and do not include the units.
##$KOVATS INDEX=	[Kovats Index]	Kovats Retention Index is a logarithmic scale on which the adjusted retention time of a peak on a non -polar column is compared with those of linear n -alkanes as reference compounds. For polar phases a modified version of this is used with linear n -FAMEs as the reference, partly due to the poor peak shape of alkanes on these polar phases. See http://www.chromtech.net.au/kovats_ri.cfm for a more detailed definition.

J-CAMP Field Name	Contents [brief description of contents]	Notes on contents
##.BASE PEAK=	[m/z value of base peak]	Mass peak in the spectrum that has the greatest intensity.
##.BASE PEAK INTENSITY=	[unscaled Y-value of the base peak] COUNTS	
##.RIC=	[relative ion count or reconstructed ion current as recorded by instrument softw are]	
##DATA PROCESSING=	[average, background subtraction, etc.]	
##$SPECTRUM REMARKS=	[additional remarks on the mass spectral data]	This field is included for additional comments on the mass spectral data. Examples of useful comments might include tent ative identification, suspected contamination, or high background. This field is defined by MaSC.
##XUNITS=	m/z	Invariant field. This should remain as is.
##YUNITS=	Relative Abundance	Invariant field. This should remain as is.
##NPOINTS=	[Total number of x,y data pairs]	
##XYDATA=	(XY..XY)	Invariant field. JCAMP field name for the type of data in the file.
x1 y1 x2 y2 etc.	[Mass spectral data where x is the m/z value and y is the relative intensity of the peak at that m/z value.]	x-values are sepa rated from y-values by a space or a tab. The intensity of the base peak is set to 999 and all other y -values are scaled to the base peak and rounded to the nearest whole number. Scaled y -values can be calculated using the MaSC -Mass Spectrum Template in MS Excel format.
##END=	[This remains blank.]	JCAMP definition for the end of the data.