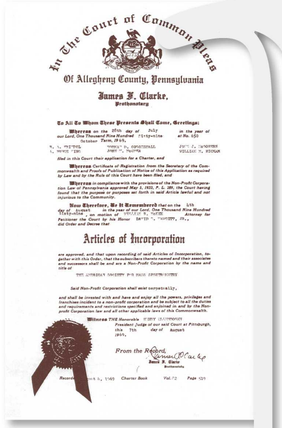
20th Annual Conference • 1972 • Dallas, TX, Southland Life Center

Michael A. Grayson, Washington University in St. Louis
American Society for Mass Spectrometry

ASMS Arranges Annual Conference

The Annual Conference was now "Arranged by the American Society for Mass Spectrometry in cooperation with ASTM Committee E-14." In July of 1969, The Articles of Incorporation of the American Society for Mass Spectrometry were filed in Allegheny County, Pennsylvania. The relationship between the new society and its ASTM progenitor was tenuous and in the process of forming. It was at this meeting that Harold Wiley was invited to regale the Conference banquet attendees with his recollections of the earliest origins of meetings on mass spectrometry; as well as some of difficulties that early practitioners of the art faced. A transcript of his banquet speech is reproduced in a nearby poster.



In The Court of Common Pleas
Of Allegheny County, Pennsylvania
James F. Clarke,
Prothonotary

Articles of Incorporation

By now, the Conference had moved well beyond its earlier emphasis on petrochemical applications of mass spectrometry and session titles included much broader areas of research such as:

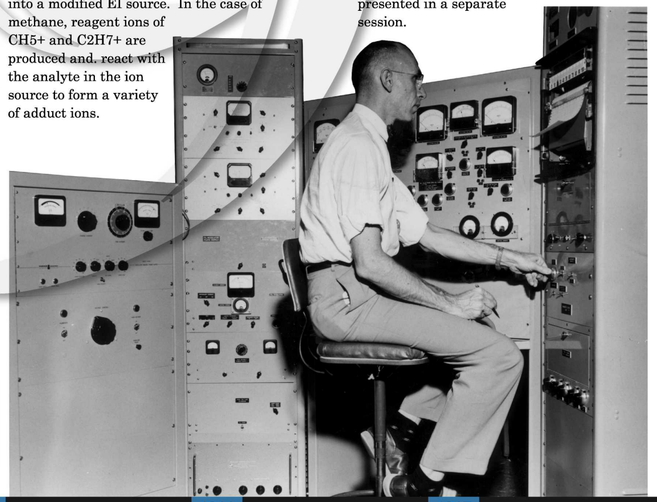
- Studies of Negative Ions
- High Temperature Mass Spectrometry
- Mass Spectra of Biological Compounds
- Identification of Drugs and Drug Metabolites
- Pollution and Environment
- Mass Spectra of Solids
- Chemical Ionization
- Ion Molecule Reactions
- Computer Applications and Data Reduction

Over 200 hundred papers were presented in parallel oral sessions running the entire week. Attendees were excited to get to the meeting upon arrival in Dallas, but by the end of the week, they were just as anxious to get home and rest!

A New Era in Ionization

In the early 60s, a group at the Research and Development Laboratory of the Humble Oil and Refining Company in Baytown Texas headed by Joe Franklin and Frank Field designed and built a mass spectrometer for the measurement of appearance and ionization potentials. It was later used to explore gaseous ionic reactions at increasingly higher pressures in research by Field and Burnaby Munson. An unexpected outcome of this high pressure research was the discovery and application of chemical ionization (CI). In CI, analyte ions are produced by gas phase reactions of the sample with reagent ions. The latter are produced by introducing a high pressure; close to a torr, of a gas, such as methane, into a modified EI source. In the case of methane, reagent ions of CH_5^+ and $C_2H_7^+$ are produced and, react with the analyte in the ion source to form a variety of adduct ions.

This development was significant because the only analytically useful method of ionization at the time was electron ionization. The latter technique had clear limitations since the sample had to be in the gas phase and the EI process imparted a large amount of energy to the analyte molecules. Thus, many non-volatile, polar compounds could not be analyzed by MS; and many labile compounds that could be ionized did not survive the ionization process to produce molecular ions. Chemical ionization was well suited to ionize these more 'difficult' compounds. By the time of the 1972 Annual Conference, a symposium on chemical ionization was organized and an additional dozen contributed papers were presented in a separate session.



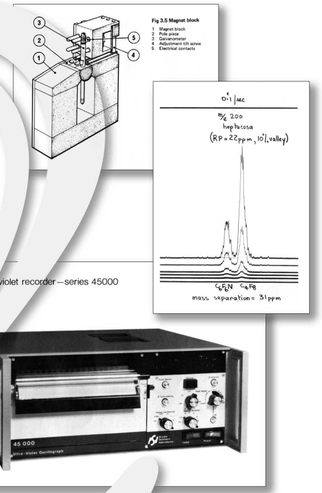
Frank Field at the console of the Humble Chemical Physics Mass Spectrometer ca 1960. Basic research with this instrument eventually led to the development of chemical ionization.

Data Acquisition and Analysis

Modern day users operate the mass spectrometer from a computer terminal where they can examine mass spectra in real-time as they are acquired and can even change the operating mode of the mass spectrometer to perform different experiments based on the data in the most recent scan. Things were not always so easy.

For years, beginning with the sale of the first CEC instrument until well into the early 70's, the standard data acquisition device for mass spectrometry was the oscillographic recorder. This device was ideally suited for recording mass spectra, because it had the required frequency response and dynamic range.

The oscillographic recorder reflected a light beam from a mirror attached to a galvanometer onto photosensitive paper. Several such galvanometers could be placed in the recorder, each operating at a different gain. The photosensitive paper had to be kept in the dark while being transferred from the instrument to the dark room for developing, rinsing and fixing. This was accomplished with the aid of a baggy, black sleeve fitted over the arm up to the shoulder. The end of the sleeve had a light-tight flange which was manually engaged with a mating flange on the front of the recorder. Once the flange was secured, a door in the mating flange could be slid aside and the recording paper transferred into the sleeve. After twisting the end of the sleeve at the flange to keep out light, the door was closed, the flange disengaged and the recording paper transferred to the dark room without exposing it to light.



Developing the photographic paper took time and was messy. Since the chart paper was typically between 3 to 5 feet in length, the developing solutions were in long narrow tanks. When the developing process was complete, one had a length of wet chart paper that had to be hung out to dry. A significant advance in this technology came about when UV sensitive recording paper was introduced. This had the advantage that the exposed paper would 'develop' on exposure to ultraviolet light, eliminating the procedure of wet photo-developing. However, the spectra were not permanent. Stored in a dark file cabinet, they would last for a long time, but if left out on a desk for a day or two, the spectrum would essentially fade away.

After photo-processing and drying the spectrum, the masses had to be marked off and the intensities of pertinent peaks measured. The spectral marking routine was basically a counting exercise, the ions in the vicinity of water and air providing a recognizable starting point. Given the high dynamic range of the instrument, there was almost always a peak at every mass and simple counting could be performed. Measurement of peak intensities was somewhat more time consuming as there were typically five galvanometer traces at various attenuations; 1, 3, 10, 30, and 100 were commonly used. This accommodated a dynamic range in intensity of 1 in 10,000. Mass and intensity information was recorded on a large lined pad for latter analysis and computations. This laborious procedure was followed for every sample that was run and analyzed. There were no shortcuts, save drawing up a mass scale that could make the mass marking exercise a little quicker. Obviously it was a labor intensive exercise just to obtain a spectrum of mass and intensity.

MASS SPECTROMETRY

1963

Early deployment of mass spectrometers occurs on satellites for the study of atmospheric density.

1964

The concept of a computer search of mass spectra is first proposed.

The flowing afterglow technique for gas-phase ion-molecule reaction studies is developed at the U.S. National Bureau of Standards.

A jet separator for GC-MS is demonstrated by Swedish chemist Ragnar Ryhage.

1965

Researchers at MIT describe the molecular effusion GC-MS interface.

1966

Tandem mass spectrometry for ion-molecule reaction studies is developed at Aerospace Research Laboratories, Wright-Patterson Air Force Base.

Researchers at Stanford University describe an ion-cyclotron double resonance instrument.

Chemical ionization mass spectrometry is developed at the Esso Research and Engineering Company.

Researchers at MIT use a computer to sequence oligopeptides from their spectra.

Alternation of accelerating voltage for selected ion monitoring in magnetic sector mass spectrometers permits resolution of mixtures in GC-MS analysis.

1967

A reverse geometry sector instrument is marketed by Mess- und Analysen-Technik (MAT).

1968

The electrospray ionization technique is introduced at Northwestern University for studying macromolecules.

The order of gas phase basicity of neutral methylamines is determined by ion cyclotron resonance-mass spectrometry.

Researchers in Australia use a computer library search to identify spectra of unknown compounds.

1969

The first gas chromatograph mass spectrometer with an integrated computer data system is introduced.

The American Society for Mass Spectrometry (ASMS) is incorporated on August 8.

1970

The annual Conference on Mass Spectrometry and Allied Topics becomes a conference of the ASMS, initially in cooperation with the ASTM Committee E-14.

Development of algorithms is begun for computer-based comparison of mass spectral data to libraries of known spectra for automated identification of unknown compounds.

NASA researchers discover amino acids in carbonaceous chondrites.

Eiji Osawa at Hokkaido University proposes a carbon compound with a three-dimensional "soccer ball" structure.

1971

Glow discharge mass spectrometry is developed at IBM research laboratories in San Jose, California.

"Static" secondary ion mass spectrometry is developed at the University of Cologne.

GC-MS is used for clinical diagnosis of metabolic disorders.

Reflectron time-of-flight mass spectrometry is developed in Leningrad.

1972

Mass-analyzed ion kinetic energy spectrometry (MIKES) is developed.

HISTORY

1963

Soviet cosmonaut Valentina Tereshkova becomes the first woman to travel in space.

Audiocassette tapes are introduced.

President John F. Kennedy is assassinated.

1964

The Civil Rights Act bans many forms of institutional racism.

Nikita Khrushchev is ousted from power in the Soviet Union in a hard-line coup led by Leonid Brezhnev.

Murray Glen-Mann proposes that matter is made of quarks.

1965

An electrical blackout grips the northeastern United States.

Cosmic microwave radiation is discovered by Arno H. Penzias and Robert W. Wilson and is thought to be a residual effect of the big bang.

1966

China's Cultural Revolution begins under the direction of Mao Zedong, throwing nearly all aspects of Chinese life into chaos.

Miranda rules are established by the U.S. Supreme Court.

Suharto comes to power in Indonesia.

1967

Jocelyn Bell discovers pulsars.

The Arab-Israeli Six-Day War ends. Israel occupies the West Bank, Golan Heights, Sinai Peninsula, and East Jerusalem.

Christiaan Barnard performs the first successful human heart transplant on Louis Washansky.

China explodes its first hydrogen bomb.

1968

Martin Luther King, Jr., is assassinated.

Robert F. Kennedy is assassinated.

The Tet Offensive is launched by North Vietnamese forces.

1969

The Cuyahoga River catches fire in Cleveland owing to the presence of high levels of flammable pollutants.

The Woodstock festival takes place.

Apollo 11 lands on the Moon.

1970

The U.S. Environmental Protection Agency is established.

A cyclone leaves between 150,000 and 200,000 dead in East Pakistan (now Bangladesh).

U.S. forces withdraw from Cambodia.

1971

Bangladesh gains independence from Pakistan.

Attica Prison is taken over by inmates.

The Soviets launch Salyut 1, the first space station.

1972

Democratic National Committee headquarters in the Watergate Hotel is burglarized.

DDT is banned in the United States.

The United States and the U.S.S.R. sign the Strategic Arms Limitation Treaty (SALT I).