History of Magnetic Sector Mass Spectrometry at MV - AEI - GEC - Kratos - Shimadzu

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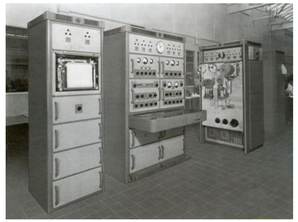
British atomic bomb project (1944)

In 1944, Metropolitan Vickers (MV) received a contract to manufacture four mass spectrometers for the Tube Alloys Directorate, which was responsible for the British development of the atomic bomb (before the project was replaced by the Manhattan project in collaboration with the USA). At the time, MV was already known for engineering innovation, particularly in vacuum technology and high energy physics. The mass spectrometers built (MS1) were 6 inch radius 90º magnetic sector instruments with an electron impact (EI) ion source and 2 kV accelerating voltage. They were based on the 1940 glass mass spectrograph designs by US physicist Alfred Nier, and specifically made for measurements of $^{235/238}$uranium isotope abundance. Pivotal to the design and production of MS1 was Jack Blears, research and development lead at MV, who was keen on mass spectrometry since reading Nier's 1936 paper (in the air-raid shelter!). As Jack would later describe it, nothing important was left to chance. At the time, there were no stabilized power supplies, no emission regulators, no suitable electrometer valves, no 10"ohm resistors and only an engineer's knowledge of chemistry and physics. The first MS1 delivery was made to James Chadwick in the Physics Department of Liverpool University, in March 1946. Previously in 1938, MV had also built the 37 inch Cyclotron Particle Accelerator, used by Chadwick's team to determine the critical mass of uranium required for explosion of an atomic bomb. Chadwick would later lead the British delegation to the Manhattan project. MS1 was the first commercial mass spectrometer built outside the USA.

MS1 (1946): Analysis of isotope ratios in uranium hexafluoride (UF_6) contained in gas cylinders on the side of the instrument by mathematician Anne Mettrick from MV. This is likely to be the fourth MS1 which remained at MV and was used to develop MS2

MS2 (1950 - 1960s)

Following the end of the war, a new instrument (MS2) was built to be multi-purpose, and take advantage of other markets besides uranium isotope analysis. Blears orchestrated several key improvements to achieve this. (1) The glass blown vacuum envelopes for the source and collector regions were replaced with all-metal housings because of difficulties presented by combined glass-metal mass spectrometer tubes in MS1. Once glass-blown, routine maintenance required a 'crack and rejoin'; (2) A stronger magnetic field was used resulting in increase of the mass range to m/z 700, and the resolution to between 600 and 700; (3) A thermal inlet system fitted to the source increased sampling capability so that not only gases, but liquids and some organic solids could be analysed; (4) The signal recorder was significantly improved. This was based on a chart mechanism using wartime Type 74 motors, and achieved 2 s full-scale response (when most competition managed 30 s). It also featured automatic range-changing with printing of the range number, together with automatic increase of the ion-accelerating voltage to prevent loss of peak-top during range changes on large peaks.



MS2 (1950): The general-purpose mass spectrometer was used in analysis of uranium, deuterium, bond-energy determinations and hydrocarbon gas mixtures, with customers including universities, the UK Atomic Energy Authority (UKAEA), and major petroleum and chemical companies such as Shell and BP.

MS3 (1952)

This was a smaller footprint version of the MS2 with a 4 inch radius 90º magnetic sector, and the vacuum and electronics placed together, in order to offer a cheaper product.

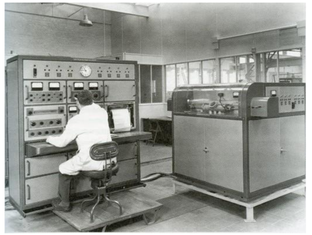
MS3 (1952): The instrument was designed to be a more affordable option and sold for half the price of MS2.

MS4 (1959)

Designed for rapid respiratory gas analysis which was made possible with the fast response of the instrument, due to the rapid mass analyser scan rate (25 scans/second) of the mass range. It was able to measure changes in concentration of lung gases (N_2, O_2, CO_2) in less than 0.04 secs – well within 1 expiration cycle, which was astounding at the time. MS4 was used in medical research and led to understanding of lung function and development of rapid diagnostic tests for respiratory disease. The subject breathed directly into the mass spectrometer through a connection between the mouth piece and the sample inlet tube.

MS5 (1955 - 1970)

This featured a new method of thermal ionisation and a novel vacuum sliding bar lock which produced unprecedented high throughput analysis (20 x 1 µg uranium samples per day) without closing down the instrument. MS5 also featured the first commercial use of an electron multiplier detector (built at MV R&D), thereby allowing observation of arrival of individual ions.



MS5 (1955): Features new thermal ionisation source, a 12 inch radius 90º magnetic sector, with a 6 kV source voltage. Operated by Barry Russell from the test department.

Robert Craig (Physicist)

In 1950, Craig was recruited by MV, and his work alongside that of **John Waldron,** the physical chemist, and **Jack Blears**, would be key to innovation in MV, including the double focusing mass spectrometers.

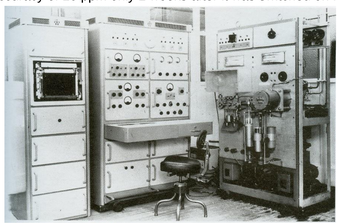
MS7 Double-focusing 1955

MS7 was the first commercially built double focusing instrument in the world and designed for the impurity analysis of solids. The first instrument built by Blears and Craig was the Mattauch-Herzog type, which used a spark source and a photoplate (2x 10 inches) detector. The spark source was good for analysis of mixtures of inorganic solids because the relative sensitivities for all the solids were nearly the same, and the photoplate also suited this analysis because of the ability to integrate simultaneously ions from all the solid elements in the mixture. The photoplate could also be moved sideways so up to 30 separate exposures could be made on one plate. Vacuum improvement (up to 10^{-8} torr) permitted the highest detection limit in the world, said to be equivalent to a grain of sugar in a lorry load of sand. MS7 was a commercial success (about 150 sold) and the first export to USA. It became the workhorse in many US laboratories engaged in semiconductor research.

MS7: The spark source Mattauch-Herzog type double focusing sector instrument operated by J. Rogers Woolston at RCA, USA, for analysis of solids in 1961. The first MS7 to reach North American shores, it shared its DC7-F (freighter) plane flight across the Atlantic with cages containing 100 screaming monkeys. In later years, Woolston delighted in telling the story of going to Idlewild (now JFK) airport to rescue 'his baby.'.

MS8 Double-focusing 1955

Development of MS8 at MV was driven by a commission from the Physicist J.H. Beynon from ICI in Manchester. He believed this would significantly improve mass resolution and it did. Beynon provided the geometry design, which was based on the Nier-Johnson geometry but changed to a 90˚ electric/ 90˚ magnetic sector and a short ion path. Beynon worked with Craig at MV for 6 months and although the specification was for a mass resolution of 2,500, they optimised it further to a mass resolution of 13,500 and mass accuracy of 10 ppm only 2 weeks after it was switched on !



MS8: A 7.5 inch radius 90º electric sector was added to the MS2 magnetic sector and a electron multiplier detector. This provided high resolution and high abundance sensitivity for Beynon's determination of elemental composition of unknown organic compounds (to complement his nitrogen rule calculations which he originated), and was sought by organic chemists.

AEI building, Trafford Park, Manchester (1965): Although MV was acquired by Associated Electrical Industries Ltd (AEI) in 1928, the trading name was not changed until 1960. The AEI Scientific Apparatus Department was created and MV mass spectrometry research department at Trafford Park relocated to Barton Dock Road.

MS9 Double-focusing 1962

MS9 (1962): Final testing performed by Nigel Bean, Test Manager at AEI

MS9 was a similar design to Beynon's MS8 but twice the size of the magnetic and electric sectors. It had a major influence on the development of organic mass spectrometry, eventually achieving a resolution specification of 100,000 and accurate mass measurement of 1 ppm (using peak matching method of Beynon). This exceeded any other commercial instrument at the time. MS9 also provided for the first time, a means to see precursor and fragment ions in a metastable decay reaction

MS30 Double beam 1970

AEI become a subsidiary of General Electric Company Ltd. (GEC) in 1967 and produced the first double-beam mass spectrometer (MS30). There were 2 separate ion sources, each with its own collector. The ions from each travelled side by side through a double-focusing arrangement. It was no longer necessary to have high resolving power for separation of reference and sample ions since the second source was exclusively used for the reference compound.

MS50 150,000 ultrahigh resolution

MS50 (1972): MS50 was in an unassailable position as the only ultrahigh resolution mass spectrometer on the market, so much so that sales staff joked they only sat in the office to receive orders. It was also coupled to advanced computing power (DS50) for automated acquisition and 10 pre-programmed operations. Along with the Daly fluorescent detector for metastable ion detection and multiple ion sources (EI, EI/CI, EI/FD, GC), it was used for multiple applications in petroleum, biomedical research, pharmaceuticals and organic chemistry.

MS50TC (1982): The Kratos MS50TC had an advanced computer processor for innovative operational control

1946	1950	1952	1955	1955	1955	1959	1960	1962	1967	1970	1972	1976	1979	1981	1988	1989	1989
MS1: MV delivers first commercial mass spectrometer built outside the USA	**MS2**: MV builds general –purpose commercial all-metal mass spectrometer	**MS3**: MV builds small footprint cheaper mass spectrometer	**MS5**: MV builds high throughput novel thermal ionisation mass spectrometer	**MS7**: MV builds first commercial spark source double-focusing mass spectrometer in the world	**MS8**: MV builds first commercial E/B double-focusing mass spectrometer in the world. 13,000 resolution	**MS4**: MV builds fast voltage scan 25 Volts/sec mass spectrometer	**Trading name of MV changes to AEI**: Relocation from Trafford Park to Barton Dock Road	**MS9**: AEI builds first commercial mass spectrometer in the world with 100,000 resolution, 1 ppm accuracy	**AEI becomes subsidiary of GEC**	**MS30**: GEC builds First mass spectrometer to have double beam technique	**MS50**: GEC builds First mass spectrometer to have resolution of 150,000	**Kratos acquires AEI from GEC**	**MS50EBE**: Kratos builds First triple sector mass spectrometer. Records CID-MIKES spectra	**MS80RF**: Kratos builds First mass spectrometer with 'rolling-field' magnets	**Concept:** Kratos builds horizontal configuration multi-sector mass spectrometer	**Profile:** Kratos builds fully computer controlled multi-sector mass spectrometer	**Kratos becomes subsidiary of Shimadzu**

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Bibliography:

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- John H. Beynon, interview by Michael A. Grayson at Swansea, Wales, United Kingdom, 22 April 2008 (Philadelphia: Chemical Heritage Foundation, Oral History Transcript # 0420).