

## 50 Years of Microanalysis: A Little History of Who's Who, A Perspective from Bruker

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Since Fitzgerald, Heinrich and Keil [1] first introduce using a solid state X-ray detector as a qualitative tool for the EMPA a whole industry has been established in the manufacturing and development of hardware, techniques and algorithms for the analysis of X-ray spectrum. No one source or group is truly responsible for these advancements but there has been a plethora of contributors. Companies like Nuclear Diodes, KeveX, Tracor Northern, Princeton Gamma-Tech and Link Analytical among others and their internal groups of Scientist and Engineers where all major contributors to development of the field.

1968 – 1969 started the commercialization of EDS systems, shortly after the introduction of the solid state Si(Li) detector. Companies such as Princeton Gamma-Tech (PGT), EG&G Ortec and Nuclear Diodes among others, explored the manufacturing of these style of detectors. There was particular interest for the usage on the new Scanning Electron Microscopes (SEM) [2] since WDS were rather large and the chambers of the early SEM were rather small.

During the 1970s as the technique became more accepted, there were major improvements in the design of the detectors and the support electronics. In the early 70's detectors were routinely breaking the 200eV resolution barrier. Detection down Sodium (Na 11) was becoming routine and the first windowless detectors were being offered which improved low-end sensitivity but also detection down to Carbon (C 6). During this period joint venture with companies like PGT (detectors and electronics) and Tracor Northern (Multi-channel Analyzers (MCA) and software) were taking place. Small frame computers were serially connected to the MCA so that data could be plotted and the first programs to do quantitative analysis were being developed. It was during this time that first digital beam control unit was developed for doing digital line profiles. [3]. this also lead to the first all computer based analyzer design, the PGT 1000 Alpha 8. In Europe Link Instruments also designed a similar type analyzer. Nuclear Diode changes its name to EDAX and is bought out by Norelco Philips.

In the 1980s most analyzers are now based on the DEC LSI-11, color monitors are common although with limited color. K, L, M markers for identification as well as the first computer assisted and automatic identification programs are available. Quantification is starting to become common and the first standardless analysis programs are now available thanks to programs like NBS's Frame C and John Colby's Magic which was featured by KeveX. The first systems with digital image and X-ray Maps were now appearing. Detector resolutions were now in the mid-150s to the high-140s. Light element multi-window detectors usually with a turret are now common. KeveX introduced the first sealed light element detectors and no LN<sub>2</sub> to the market with some success and Boron (B 5) detection is becoming routine. [4] To help with stability and linearity of the electronics manufactures started using a Zero Strobe Peak for offset calibration and monitoring the detector's resolution.[5] Toward the end of the 80's transistor reset for the detector is first introduce by Oxford Instruments. The device is called the Pentafet and improves detector resolution and low-end performance. Detectors were now approaching the mid-130s eV. Also the first analyzer using a UNIX workstation was introduced by PGT. High purity Germanium

detectors were being used for better resolution and larger active area detectors with improved resolution. Using GeIG detectors manufacturers are able to achieve 115eV resolution with a 10mm<sup>2</sup> active area while 60mm<sup>2</sup> with 139eV resolution were possible. Link Instruments is bought by Oxford Instruments, Tracor Norther becomes part of Westmark Systems, PGT is purchased by Outokumpu and Ortec drops out of the microanalysis market.

In the 1990s the first digital pulse process is introduced by PGT which added stability to the system, improved throughput with the adaptive shaping time.[6] Shortly thereafter this lead to Spectral Imaging or Position Tagged Spectroscopy (PTS) which revolutionize the way we do X-ray acquisition particular mapping.[7] As Personal Computers became more powerful and companies desire to standardize the first Windows based analyzers came on the market. There was also some movement to Apple as an alternative. Rontec introduces the first Silicon Drift Detectors for XRF analysis and as a high speed X-ray mapping spectrometer. [8] Beryllium detection is now possible. Tracor changes its name to Noran and becomes part of Thermo Scientific and KeveX is merged with Noran and eventually absorbed.

By the turn of the century, the 2000s it is now clear the SDD would replace the Si(Li) and GeIG detectors as companies desired to eliminate the need for LN<sub>2</sub> in the lab as a safety concern. New interest in using multiple detectors to further increase the throughput and process data faster is becoming common. The first detector array the QUAD is introduce and offers the highest solid angle 1.1 steradians allowing high throughput in low kV and low probe current applications. Windowless SDDs detect and map Lithium. Thanks to major improvements in computer technology, increases in CPU speed, larger memories and storage as well as better software development environments more complex techniques for data processing and analysis are now possible. This in turn allows the analyst to process data faster and more accurate. PGT Microanalysis division is merged with Rontec to form Bruker Nano Analytics; EDAX is bought by AMETEK, Inc.

#### References:

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