



## FIRST Newsletter

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### History of Handheld XRF

By *Alexander Seyfarth and Dr. Bruce Kaiser, Bruker Elemental, Kennewick, WA, USA*

Handheld XRF (HH XRF) today is a staple in the Bruker instrument family and serves a large and diverse market.

You will see it working on any scrap yard to identify metal and help sort it for recycling. You'll also find HH XRF at any oil refinery or chemical industrial process, where it is used to safety-check the chemistry of the pipes under pressure and temperature.

HH XRF units are used to screen imported toys and consumer products at the borders and at manufacturing sites to ensure that the products do not contain any hazardous chemicals such as lead (Pb), mercury (Hg), cadmium (Cd) and other regulated elements (RoHS, CPSIA, California prop 65).

HH XRF enables faster cleanup of contaminated areas by mapping the concentrated areas and, after remediation, checking that the cleanup was successful.

New ore deposits are mapped in similar fashion, allowing mining companies to be more efficient in their prospection campaigns and to assess elemental content information on site, rather than 2 to 6 weeks later in the lab.

When you trade in your gold and other precious metals, you might see HH XRF being used to authenticate your trade. Similarly, HH XRF is used to investigate paintings and works of art for authenticity and for conservation purposes. Another very intriguing application is determining the sources of obsidian used in stone age tools to ascertain old trading and migration routes.

Worldwide there are now over 40,000 units from a handful of manufacturers in use. Ten years ago the units were used just for niche applications. How could a segment grow that quickly?

How did it all start? What were the "killer" applications? Let us look back and follow HH XRF from the beginning to where we are today. Let's journey into the past and live through the development as experienced by a small group of people in Kennewick, Washington.

A spin off from the DOE in the Hanford Nuclear Reserve in the early 1980's, United Nuclear built a prototype "hand held" energy-dispersive XRF probe with a radioactive source as a proof of concept. This tool was used to investigate the highly radioactive holding tanks where no X-ray source was needed, but also to test soil. The main element they were looking for was uranium (U). Figure 1 shows the measurement head which was connected to a cart on which the electronics displayed data. This "hand held" XRF system weighed in at over 70 pounds.



Figure 1: DOE/United Nuclear “hand held” XRF measurement head.

Commercialization then moved towards detecting U and other elements in soil through the creation of a wearable “front pack” as shown in Figure 2. The electronics pack was reduced to a mere 50 pounds.



Figure 2: United Nuclear commercial MAP-1 device, 1982-1983.

Due to its high price, weight and limited elemental range, the MAP-1 device was only marginally successful commercially. The unit morphed into the MAP-2 (Figure 3) using a higher resolving CdTe detector and was the market leader in soil and mining related instrumentation.

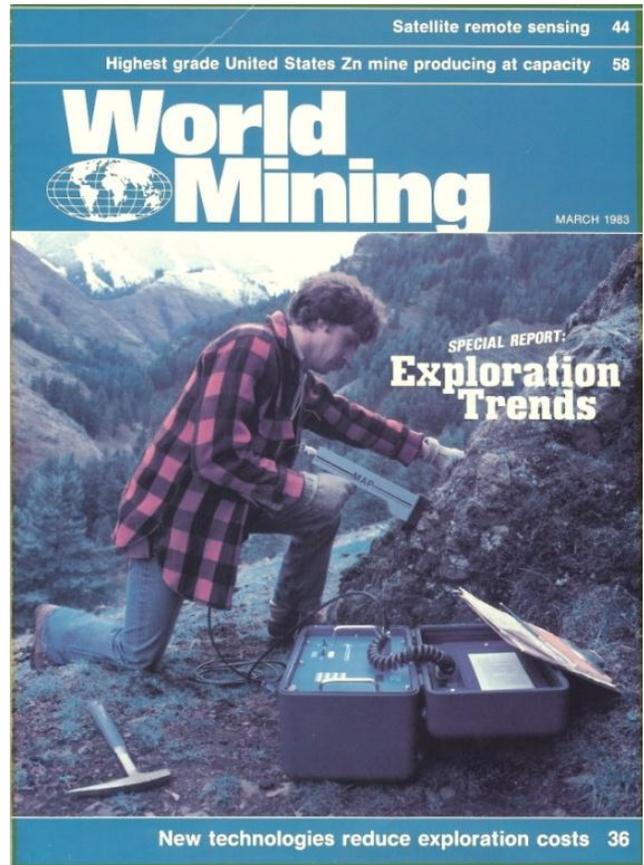


Figure 3: MAP-2 front page cover in 1983.

Based on the MAP-2 unit, the “lead in paint” application was covered by being able to measure Pb in wipes as well as in surface and hidden paint. In 1996, the MAP-2 was one of the first units approved for EPA SW846.

The MAP-3 analyzer (Figure 4) was derivative with smaller electronics, still using a proportional counter detector, and priced around \$16,000 USD in 1997.



Figure 4: The MAP-3 analyzer in 1997, the beginning of true HH XRF.

The breakthrough-enabling component was developed in 1991: the SiPin detector (Figure 5). Taking advantage of its experience in building hybrid circuits and satellite instrumentation, Amptek developed the XR-100 X-ray detector for commercial use. After winning the coveted R&D 100 award in 1994, this simple to use, thermoelectrically cooled X-ray detector was the technological breakthrough responsible for replacing cumbersome liquid nitrogen cooled detectors in many applications. Although developed for commercial use, the XR-100 was selected for the Pathfinder Mission to Mars, where it successfully analyzed rocks and soil using X-ray fluorescence techniques. This successful instrument is the detector of choice in numerous original equipment manufacturers' (OEM) applications using the XRF

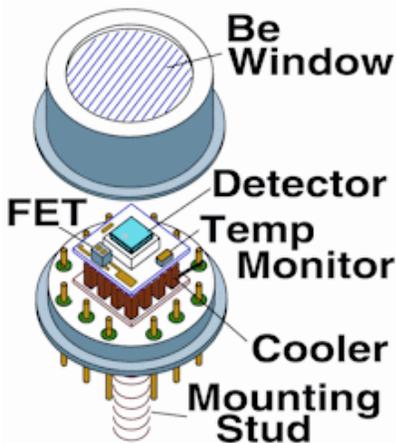


Figure 5: XR-100 SiPin detector (courtesy Amptek Inc.).

method. When Amptek began development of the thermoelectrically cooled detector, there was virtually no hand-held or portable XRF market. No cost-effective technology existed with good enough performance in a small enough package that could be incorporated into a portable XRF analyzer.

The XR-100 SiPin detector was rapidly adopted for “portable” or “hand held” XRF products with radio isotope sources. 1995 marked the introduction of the MAP-4, which was primarily designed for the lead in paint market. This first fully handheld unit by SciTec entered the market late since, at that time, a Massachusetts-based company called Niton Inc. had already launched (in 1994) an all-in-one unit later called the “Brick”.



1994: NITON XL-309  
1<sup>st</sup> one piece, handheld XRF with real-time digital signal processing and silicon PIN diode detectors



Figure 6: Niton and MAP-4 handheld XRF units, 1994-1995.

The Niton XL-309 offered remarkably better analytical performance at a lower price than the MAP-3 and, within a few years, was able to grab a dominant share of the lead in paint market. This prompted SciTec to fall on hard times; the company was taken over by a C-through, and then acquired by an investor group, Advent, and combined with the assets of EDAX. Advent reorganized the HH XRF into Keymaster Technologies in 2001 when it sold all but the XRF part of EDAX to AMETEK.

EDAX, and later Keymaster, only retained a small segment of the lead in paint market. They also lost a majority of the soil and mining market to the “Brick” through 2002.

The game-changing year 2001 brought a new revolution: the miniature X-ray tube became commercially available and could replace selected isotope sources.

Keymaster designed and presented at PITTCON 2001 the first TRACER II unit (Figure 7) which included a new tube (Ag transmission target) and the Amptek SiPin detector as a proof of concept well before the first commercial units of others.



Figure 7: TRACER II handheld XRF introduced by KeyMaster Technologies at PITTCON 2001.

In 2001 NASA was investigating a way to conduct quality control for critical aluminum alloy parts used in the Space Shuttle Program. Representatives of KeyMaster Technologies visited NASA Marshall Space Flight Center to demonstrate its XRF analyzer. The instrument, which was about the size of an electric drill, quickly determined the identity and amount of chemical elements in many materials. Marshall engineers recognized the device could have an immediate benefit if the range of elements detected were expanded. NASA and KeyMaster worked together to create a portable vacuum XRF analyzer that performed on-the-spot chemical analyses — a task previously only possible in a chemical laboratory. The first application for NASA involved the Shuttle's Return to Flight activities.

But steel alloy composition was only one field of need. NASA engineers wanted to analyze aluminum alloys because vast amounts of high-strength aluminum alloys are used in the Space Shuttle propulsion system — the external tank, main engine and solid rocket boosters. Just as KeyMaster and NASA engineers theorized, subsequent tests showed that when the XRF analyzer was operated

in a vacuum, aluminum alloys were easily analyzed. According to NASA, the ability to quickly and accurately determine composition on large objects such as a rocket motor case was a major breakthrough. KeyMaster and NASA patented the Vacuum XRF analyzer jointly and implemented the commercial product as the TRACER III-V.



Figure 8: KeyMaster Technologies' TRACER III-V unit.

The TRACER III-V was able to measure magnesium (Mg) and aluminum (Al) in aluminum alloys and was controlled by a Microsoft Windows-powered HP personal digital assistant (PDA) and used in quantity by NASA.

2002 brought the introduction of X-ray tube-based commercial units by both Niton (XLT) and a new competitor in the HH XRF segment, Innov-X (Alpha). These tube-based units (Figure 9) were all gun shaped and revolutionized not only the way metal sorting in scrapyards was done, but also positive material identification (PMI) applications essential for the safe operation of refineries and pressure vessels.



Figure 9: Niton XLT and Innov-X Alpha tube-based units, 2002.

Meteorex, a strong benchtop EDX company based in Finland and a pioneer on the portable XRF market, selected KeyMaster as the OEM hardware source for its XMET 3000 instrument. Oxford instruments acquired Meteorex in 2004 and continued the XMET product line with numerous upgrades and accessories.



Figure 10: XMET 3000 (KeyMaster hardware OEM unit), 2003.

Driven by scrap and PMI applications, the HH XRF market has been exploding in the metals segment since 2002 (Figure 11).

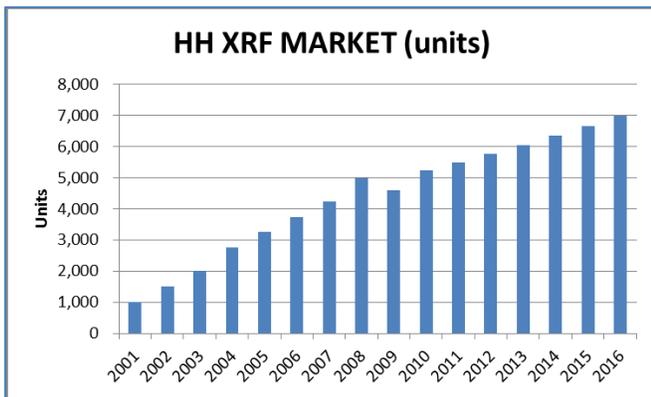


Figure 11: HH XRF market development 2001-2012 and projected 2013-2016. (Data from combined sources: SDI, Bruker, various literature sources, vendor data).

2005 and 2006 brought an additional growth spurt with the European Union, and later also China, requiring screening of hazardous substance (Pb, Hg, Cr, Br, Cd) in consumer products. This increased the market size again substantially.

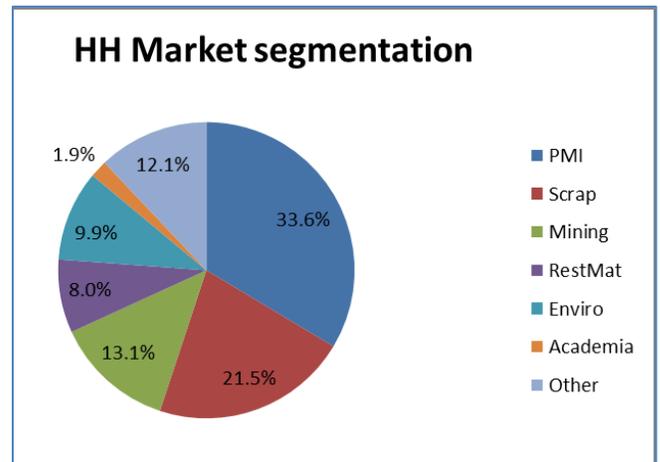


Figure 12: HH XRF market segments 2012-2013. (Data from combined sources: SDI, Bruker, vendors and trade groups).

2005 also marked the introduction of what became the “gold standard” instrument for academia and research: the TRACER III V series.

The TRACER III-V (Figure 13) was designed for the investigation of inhomogeneous materials, for all applications where no “generic” calibration could be made. It is a tool which allows the researcher to conduct an interactive investigation using the full spectrum. All settings on this instrument are user-configurable, similar to what researchers are accustomed to on benchtop XRF systems.

The KeyMaster TRACER series was given the coveted seal of certified space technology in 2006, the same year that KeyMaster was acquired by Bruker Corporation. Dr. Frank Laukien commented in the press release at that time: “We cordially welcome the excellent KeyMaster team. After careful exploration, we have decided that KeyMaster is the best partner for us to enter the rapidly growing hand-held XRF market. We are very pleased with KeyMaster’s innovative and versatile, yet robust technology to serve this market both via OEM partners as well as via our own applications and market expertise. The KeyMaster technology is perfectly complementary to our existing X-ray analysis portfolio, and will be an enabling technology to pursue new miniaturized analytical solutions, particularly in fast-growing industrial applications.”



Figure 13: TRACER III-V Series of HH XRF instruments.

Within 2 years, Bruker XFlash™ silicon drift detector (SDD) technology was integrated into the first ever SDD-based HH XRF unit and the S1 TRACER shown in Figure 14 was born.



Figure 14: S1 TRACER, the first SDD-based HH XRF, June 2008.

This giant leap in performance allowed the analysis of light elements (Mg, Al, Si), in air and, using the Bruker/NASA patented vacuum approach, at 10 times lower concentration levels than before.

The competition followed within months to match the SDD integration. Still, Bruker's S1 TRACER remained the unit with the best resolution, < 145 eV at a count rate of 120,000 cps.

The mining and geochemistry market was the next driver since a correct quantification of Al and Si greatly improved the accuracy compared to the prior "soil" type calibrations. With an increased concentration range and the ability to use Fundamental Parameters, exploration and mining companies as well as geological surveys started buying SDD-based units in larger quantities.

Bruker then split the instrumentation line into an academic/research based TRACER platform, which is sold with user calibration software and extensive training, and the S1 platform, which is the typical industrial point-and-shoot analyze. Read more about the TRACER platform here:

An updated S1 platform was just released in 2012 and includes all the latest available technology and has driven the compactness, weight and performance to new levels. The S1 TITAN Series now also features the first and patented TITAN Detector Shield which protects the most costly and vulnerable component of the analyzer: (video link!).

With this 2012 all-new platform and all common applications available, Bruker's HH XRF has established itself as one of the market trendsetters and is part of the top 3 vendors (SDI 2012). Now part of the Bruker Elemental division, the HH group still resides in Kennewick, WA and produces all of its analyzers in the US. Recently the operation has been named the Manufacturer of the Year in the Pacific Northwest, which is known for its high tech workforce and companies.

"We are delighted to be the Smartmap Expo 2012 Manufacturer of the Year," said John H. Landefeld,

Bruker Elemental executive vice president. “It is a great honor to be recognized as one of the leading manufacturing companies in the Pacific Northwest—a great compliment to our management and employees.

What is next for HH XRF? For the academic and research segment, HH XRF now has its firm place and a growing set of applications as indicated in Volume 3 of the Studies in Archeological Sciences handbook titled “[Handheld XRF for Art and Archeology](#).” This book can be used as a manual for everything from the analysis of obsidian to the identification of pesticides in ethnographic collections!

For the industrial segment, HH XRF ensures that our world remains a safe and healthy place to live, with

more regulations to be enforced and legislation on the way.

The light element capability also allows for more crossover into the laboratory segment as can be seen by the application of limestone analysis ([view webinar](#), 58 minutes).

As a recent example of how HH XRF helps us be safe, the upcoming Reduction of Lead in Drinking Water Act was enacted on January 4, 2011 to amend Section 1417 of the Safe Drinking Water Act (SDWA Act) regarding the use and introduction into commerce of lead pipes, plumbing fittings or fixtures, solder and flux. HH XRF is *the* technique to detect Pb, a HH XRF favorite from the very beginning.