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Remote sensing volatile elements for new signatures from underground nuclear explosions

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Abstract

The detection of radioactive noble gas isotopes is one of the key tools in detecting underground nuclear explosions, but other volatile elemental species, that have historically been overlooked, may also migrate to the surface. To investigate this possibility we have been studying which elements are most readily volatilized into air from solid materials (rocks, concrete, glass, etc.) following short duration (60–120 s) spot heating spanning a wide temperature range (100–2000 °C). The widest possible range of elements (Li–U) was monitored during the experiments that produced some unexpected results with regards to the apparent atmospheric persistence of some volatile signatures long after heating had ceased and the sample had returned to room temperature. Results highlighting elemental volatility as a function of temperature from different materials will be presented along with examples illustrating the extended periods over which some elements remain detectable by real time atmospheric sampling.

Keywords Underground nuclear explosions · Underground chemical explosions · Volatile elements · Laser heating · ICP-MS · Atmospheric sampling

Introduction

During an underground nuclear explosion, large quantities of rock are vaporized (70 tons/kt of explosive yield) along with an even larger mass (350–700 tons/kt) which is melted [1–3]. This leads to the formation of a cavity within the rock and considerable fracturing of the enclosing strata. Even if the explosion is completely contained, the event can still produce many signatures, some of which can be very short lived, like a seismic disturbance lasting seconds [3–6], while others may only emerge slowly, days or weeks later, such as the gradual release of radioactive noble gases [7, 8] or other chemical species [9, 10]. Typically these radioactive gases, including ^{37}Ar, ^{131m}Xe, ^{133}Xe ^{133m}Xe and ^{135}Xe, are transported to the surface along cracks and fissures produced by the explosion and natural fractures that may have already been present prior to the event. The majority of the radioactive material remains trapped in the explosion cavity, largely immobilized in glass formed by melting of the surrounding rock. Methods to detect the release of radioactive noble gases have become extremely sensitive, in part due to their short half-lives (days–weeks) [8]. However, it still requires that they find some release path to the surface within a few half-lives to remain detectable.

Formation of the rock melt glass consumes around 25% of the energy expended during an explosion [2]; however, there will still be considerable heating of the surrounding rock extending out some distance that will remain at an elevated temperature for some time. The development of this thermal halo does not seem to have been investigated to a large extent, possibly because it is highly variable depending on the local geological setting [11, 12], but even relatively small increases (200–300 °C) in local temperature might promote the release of volatile species from the bulk rock which could be utilized as a new signature. This might also include the detection of underground explosions caused by chemical explosives. The amount of heating generated by a small explosion maybe quite limited spatially and largely dissipated after a few days. For larger events, heating could last for weeks and obviously extend

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much further out into the enclosing strata. The development of volatile element signatures both organic and inorganic, even without a driving heat source, is not unheard of. This has become a standard geochemical surveying tool that has been applied to the detection of hydrocarbon, geothermal, and mineral resources. Typical elements most often used in geological surveys include: He, S, and Hg, but Rn is a potential indicator of radioactive minerals. The presence of Hg can be readily detected by fluorescence, S by various chromatographic methods, and Rn by radiometric counting [13–16].

In this paper we report an atmospheric elemental volatility study conducted on various geological and structural materials under confined conditions equating to a "bore hole" type setting and in an open environment subject to turbulent "open-air" conditions. Elements studied covered the whole elemental mass range from Li–U and from ambient temperatures to in excess of 2000 °C. Localized sample heating was performed using a CO_2 laser. A new approach to atmospheric sampling was developed to exploit the high sensitivity and wide mass coverage of ICP-MS for elemental detection. This eliminates the usual pre-requisite of an argon operating atmosphere that allows direct air sampling into the ICP. Furthermore the ICP-MS can be distantly located (> 30 meters) without degrading analytical sensitivity. Instrument backgrounds observed from direct atmospheric sampling were not unduly raised compared to operating in pure Ar. Experiments have shown that some elements (Na, K, Ag, Cs, Hg, and Pb) were emitted from some geological materials at temperatures of just 250 °C. In "open air" these elemental emissions dissipate quite rapidly but under more confined conditions the same signatures persist for many hours, even after only a short period (30 s) of localized heating. While the exact chemical form of the thermally induced elemental emissions was not investigated, inserting a 500 nm particle filter only partially attenuated the detected ICP-MS signal. This would seem to indicate that ~ 30% of the volatized material is still in the form of a gas, nm or smaller sized particulates.

Experimental

The main components of the heating system consist of a laser that acts as the sample heat source in conjunction with an ICP-MS that acts as a very sensitive elemental detector. A 100 W continuous wave (CW) carbon dioxide laser with an output at a wavelength of 10.6 µm (Model: FSTI100SWB Firestar 100, Synrad Inc., Mukilteo, WA, USA) provides a highly flexible heat source with a rapid response that can heat a 5 mm diameter spot on almost any geological material from ambient temperatures to > 2000 °C within a few seconds. For this study, laser powers employed were restricted to the 0–20 W range that corresponded with a setting of 4–15% on the laser energy control unit. At the 100% setting, maximum optical output was measured at 140 W. Every material tested quickly reached its melting point when exposed to the unfocussed power level of 20 W for 30 s or less. To measure sample surface temperature over a range of 300–2000 °C, an optical pyrometer (Model: IGA 50-LOplus, LumaSense Technologies, Santa Clara, CA, USA) with a very small viewing window (0.45–0.6 mm^2 at a distance of 100 mm) was recently incorporated. The sensor possesses a response time of 1 ms and measurement uncertainity is 0.5% of the measured value in °C at temperatures > 1500 °C. As a result of this upgrade conversion to surface temperature measurements is still in progress. The pyrometer is optically blind to the 10.6 µm radiation from the CO_2 laser and incorporates a low power visible pointing laser to aid precise spatial alignment of the viewing window on the heated spot to ensure accurate temperature measurement. The ICP-MS (Model: X-series, Thermo-Fisher, Winsford, England) was operated in Time Resolved Analysis (TRA) mode for all data acquisitions. Typical dwell times range from 0.01 to 1 s/isotope depending on the number of isotopes acquired, with each element mass being sampled between once per second up to once every 5 s. Instrument parameters were typical for dry plasma-laser ablation operation. The basic layout of the main components is given in Fig. 1.

It was observed in early development experiments that the ICP-MS does not need to be in close proximity of the heated sample to make a detection of volatilized elements. This holds irrespective of whether the sample is relatively enclosed during heating or in the open air. Sampling tube lengths > 30 m have been used with no obvious degradation to the observed response, only a slight time delay (20–30 s) in signal appearance at the ICP-MS. Initially the samples were entirely enclosed in a sealed cell much like

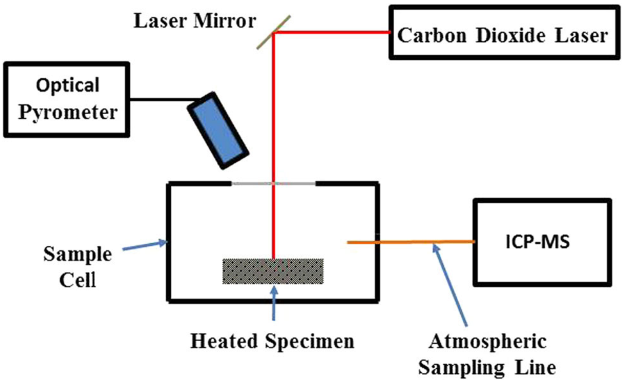

Fig. 1 Basic system layout for detection of volatile element signatures

conventional laser ablation ICP-MS. Argon was continuously fed from the ICP-MS at a flow rate of $\sim$ 0.9 L/min and used to sweep the cell, with the Ar gas exhausted back through the ICP. However, it was noted after several experiments that a gas-tight cell was not essential and good high intensity signals could still be obtained even if the sample chamber was relatively open to the atmosphere. For example, if the heating laser cell window was removed from the system, volatile species could still be sensed without small amounts of air entrainment extinguishing the ICP.

This was carried a stage further to a completely open system where direct air sampling was implemented using a concentric nebulizer (Model: Micromist ARG-1-UM02, Glass Expansion, Pocasset, MA, USA) more routinely used for liquid sample introduction in the ICP. Nebulizer gas from the ICP is applied to the gas inlet port in the standard manner, but the liquid injection port is coupled to a 0.1 ml plastic pipette tip using a 6 mm nylon Swagelok union that now acts as the air sampling port. The third outlet port of the nebulizer is coupled to the 6 mm OD plastic line back up to the ICP using a second 6 mm nylon Swagelok union. In this configuration, a controlled amount of air is drawn through the narrow pipette tip aperture exploiting the Venturi effect in the same manner that liquid would be drawn through the nebulizer without external pumping required when the nebulizer argon gas flow is applied. The nebulizer argon gas flows rapidly, transporting the entrained air containing volatilized species, back to the ICP without causing the ICP to extinguish. Figure 2a, b illustrate the semi-closed and the open approaches to volatile element detection respectively.

By adopting this direct air sampling approach, samples could be tested in a relatively enclosed setting or on the open bench without the strict requirement of an argon atmosphere. The pipette tip gas sampling aperture only needs to be positioned 20–30 mm from the heated region of the sample, preferably slightly above it. The effect on the signal response between open and relatively enclosed setting is quite distinct. Under the former, signal duration is usually quite short at 5–30 s and punctuated by fairly large amounts of instability, making the emission signal profile quite variable, even when peak count rates are relatively high $> 100{,}000$ cps. Much of this variability and short signal duration is attributed to random air currents that quickly dissipate the plume of volatilized species given off during sample heating. Under the more enclosed setting with the same heating conditions, signal duration is greatly extended and can last from minutes to hours with no random air currents to dissipate the evolved material plume. Signal intensity is also usually much higher under these more stable atmospheric conditions. ICP-MS backgrounds were not unduly degraded by sampling in air versus the more typical pure Ar atmosphere. Possible reasons include air dilution in the far larger Ar carrier flow, but also there is always some degree of intrinsic background air entrainment between the ICP and the plasma sampling aperture of the spectrometer. Instrument backgrounds during air sampling operation were < 10 cps at most masses > 80 amu, with the heating laser off. Below 80 amu backgrounds were high where the usual Ar ICP molecular ions are present but no obvious degradation was noted. Formation rates of analyte oxide ions were normal and no obvious nitride molecular ions were detected.

A wide variety of materials were tested in the course of this work that included SRM-610 glass, concrete, limestone (TX), and rhyolite (Taylor Creek, NM). Typical sample dimensions ranged from 1 to 4 cm. The samples were tested intact as solid pieces. The primary interest in the NIST SRM-610 glass stems from the fact it contains most elements in the periodic table at a nominal value of 500 ppm, including many less abundant elements [17]. Therefore the SRM-610 acts as a good multi-element test sample that allows the volatilization behavior of rarer elements to be studied. While these elements may appear at different temperatures in other matrices, this does indicate

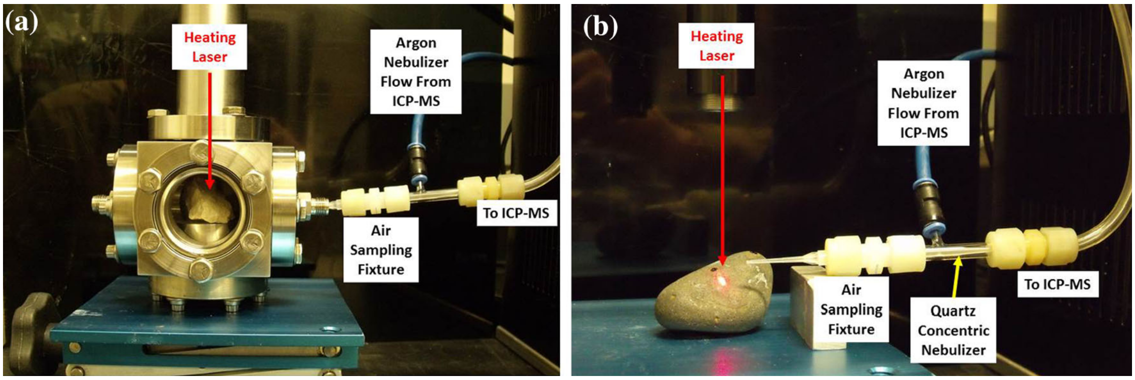

Fig. 2 **a** and **b** Semi-enclosed cell sampling (left) and completely open sampling (right)

the approximate temperature range where they might be observed if present. The concrete, rhyolite, and limestone materials all represent potential geological containment media for an underground nuclear test. Elements that appear to be volatilized from these materials at relatively low temperatures, in some cases below their elemental melting points, may form new detectable signatures even before any radioactive species have managed to escape from an underground nuclear explosion. Further work is required to identify the volatile species responsible for each elements but it is likely related to their chemical form in the host rock and potentially influenced by factors like the presence of water or salt rich brine.

Results

In order to make results comparable between the different matrices, ICP-MS acquisitions were made primarily in 5 or 10 min windows. In a few instances, longer data collection windows lasting several hours were employed to study the relative persistence of some elemental signatures under enclosed conditions. The typical duration of sample heating was limited to 30 s, initiated 120 s after the start of the data acquisition to allow a period of elemental backgrounds to be collected. Heating was applied to the same fixed spot on the sample surface as the laser power was stepped up in 0.5% or 1% increments, collecting one TRA peak jumping mass spectrum at each power setting. For a repeat test on the same sample the laser was re-positioned at a new previously unheated region. Figure 3 highlights the typical laser heating and TRA acquisition profiles under partially enclosed conditions. Air is sampled from the chamber using the nebulizer device described in the previous section with analyte signal delays of ~ 15 s observed using a 5 m long line up to the ICP-MS.

Figure 4a, b illustrate the typical response observed for Pb volatilized from SRM-610 glass where the nominal Pb concentration is 434 ppm [17]. With the sample located in both a relatively enclosed setting and in the open air as laser heating is gradually increased stepwise. Laser heating was applied for a period of 30 s, 120 s after the start of each acquisition. The elemental response from the glass was shorter and much more erratic for completely open air sampling, and the signal response was noticeably lower than that observed from the more enclosed cell setting. This is attributed to the more rapid dispersal of the volatile species plume in the open setting by random air currents. The large signal response observed from the standard glass in both sampling configurations suggests detection of elements at low or even sub-ppb concentrations can be achieved with this approach. The temporal response for most elements released during heating overlapped, however, Hg was consistently observed to lag every other element by 10–20 s. An example of this behavior is shown in Fig. 5 where the temporal response for other elements (Na, Rb, Cs, and Pb) was overlaid with Hg from concrete heated to ~ 250 °C in an open cell setting. This strange behavior of Hg fits its reputation in atomic spectroscopy as a "problem" element, where delayed response and slow or

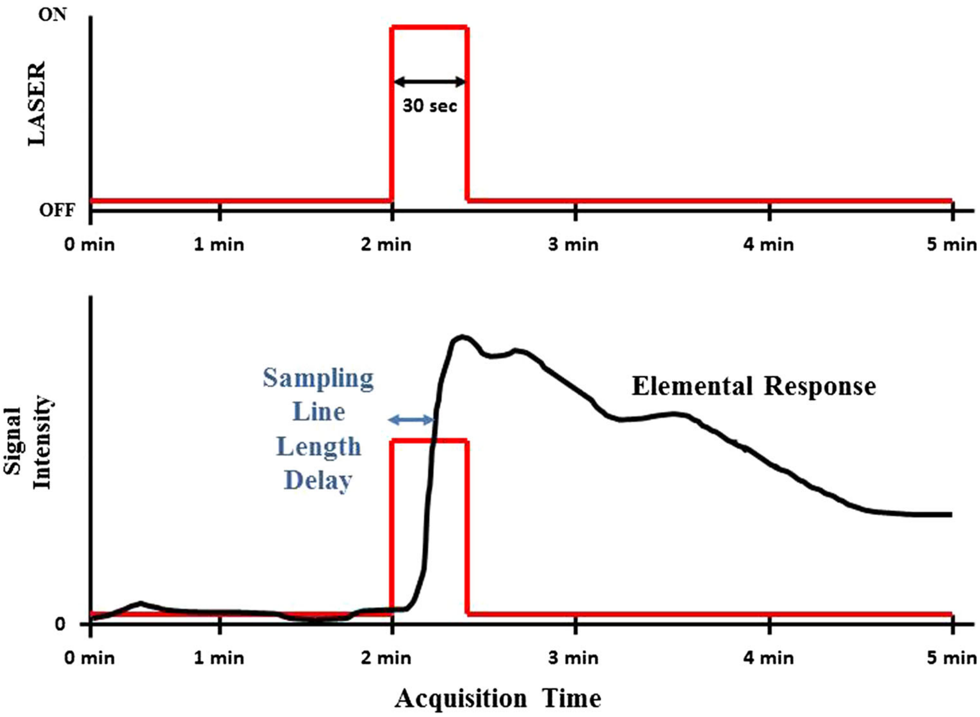

Fig. 3 The typical laser heating and elemental response observed from a semi-enclosed sampling cell open to the air

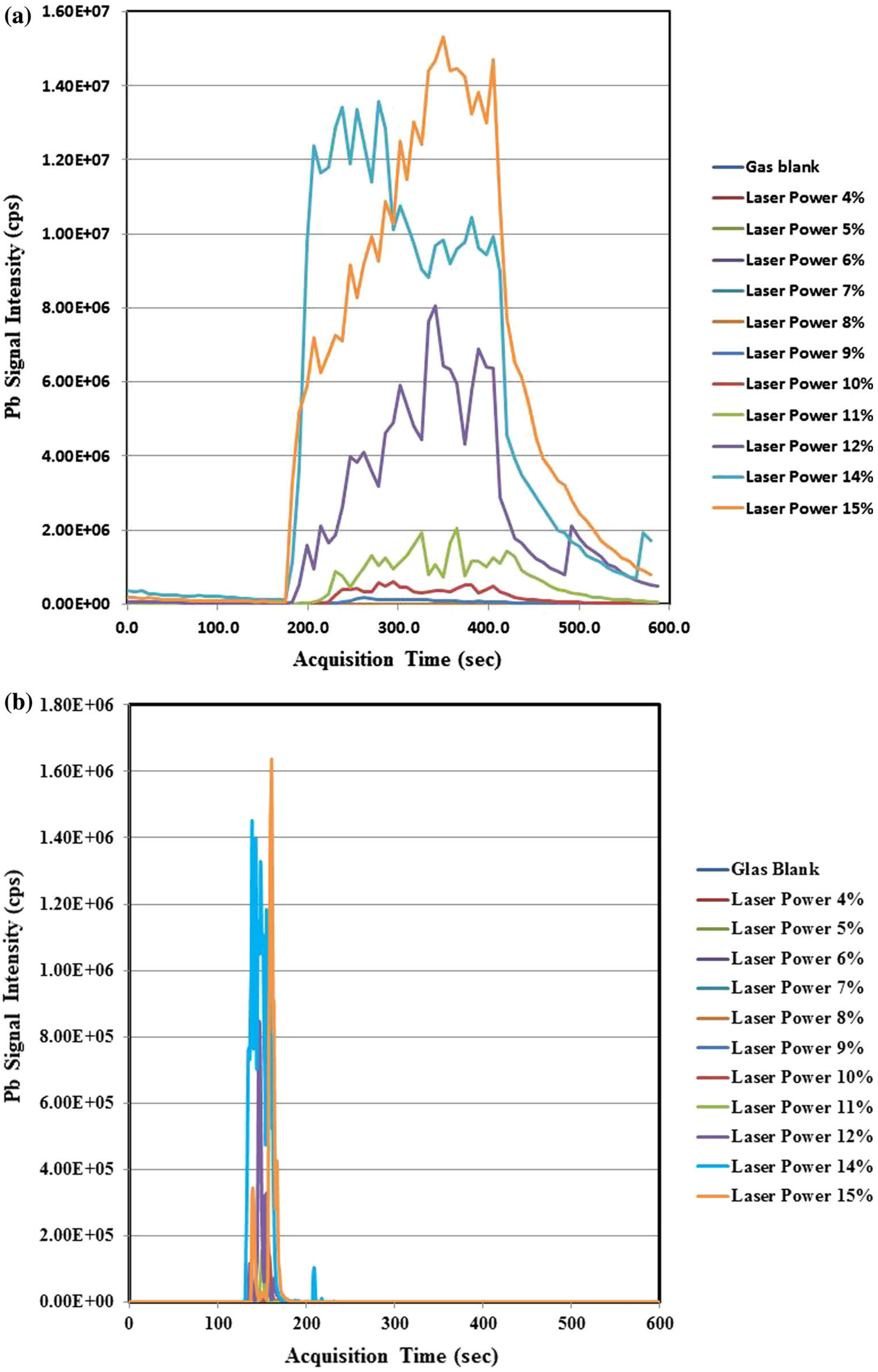

Fig. 4 **a** The volatile Pb signal in air as a function of laser power from SRM-610 glass in a semi-enclosed cell. **b** The volatile Pb signal as a function of laser power from SRM-610 glass in open air

poor washouts in analytical measurements are often observed and have been attributed to its strongly covalent bonding and non-polar behavior [18].

Figure 6a, b graphically display the laser power % on the left vertical scale at which different elements (Li–U) started to become detectable by the ICP-MS using the open air sampling approach for the different materials. Note, for element points falling on the laser power setting of 16%, this equals a “No Detection” at any laser power up to 15%. As such, this may indicate the element was not detected in

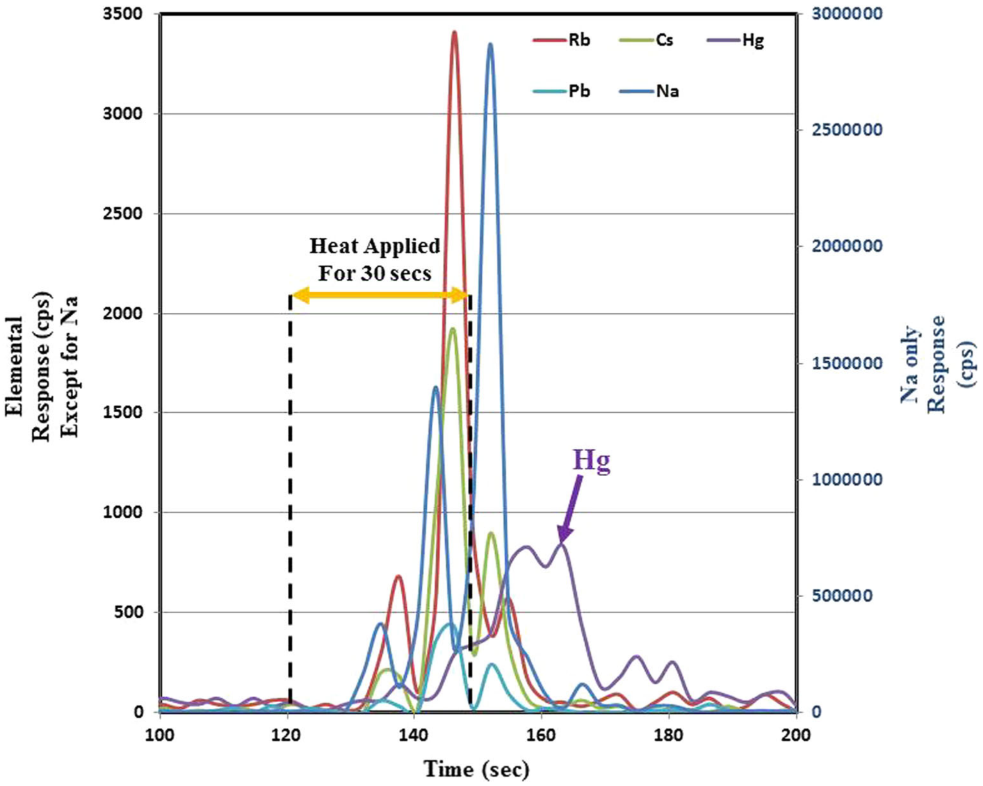

Fig. 5 The Hg signal delay compared to the responses from Na, Rb, Cs and Pb observed from concrete heated to ~ 250 °C

a volatile form, although actual response will also be convoluted with element concentration in that material. The 1800 °C upper limit on the right hand scale represents the highest temperature observed by optical pyrometry. It should be remembered that the laser heating is only applied for 30 s and for most of that time window the sample temperature will be rising very rapidly, therefore the peak temperature is reported. As an example for rare low abundance elements, this might indicate an apparent non-volatile response, but the true underlying reason is a very low concentration, hence the inclusion of the SRM-610 multielement standard. On the right hand vertical scale of the graphs is the approximate sample surface temperature reached after 30 s of laser exposure. This data is also summarized in Table 1. For most elements, once a surface temperature was reached at which it would become detectable in the air above the sample, increased heated always led to a rise in its air concentration. Only Hg sometimes would be detectable with relatively little heating then sometimes decline or even disappear at higher temperatures. Possibly as a result of the available Hg being depleted in the heated region.

Discussion

The elements detected during these heating experiments largely fit into the volatile, intermediate, and refractory categories often referred to in the fractionation of nuclear debris [19], with some exceptions. These differences can always be traced to a volatile or refractory parent in the mass decay chain. For example Freiling classifies ^{89}Sr and ^{90}Sr as volatile, but in this work Sr is classed as a refractory element. The difference is caused by the presence of Br and Kr parents in the masses 89 and 90 decay chains.

The elements most likely to be detected at the lowest temperatures (50–400 °C) include: B, Na, K, Rb, Ag, Cs, Hg, Pb, and Bi. Li would be expected to fall into this most volatile group as a + 1 alkali metal, however, it was not usually detected until slightly higher temperatures were attained, which may reflect its low crustal abundance compared to Na or K in most of the materials analyzed. The Hg response consistently lagged behind other elements by 5–10 s and often seemed to disappear completely at higher temperatures. While mercury memory in ICP-MS is well known, its strange behavior is perhaps more puzzling. Of all the elements examined, it is probably one of the most likely to be evolved in an elemental form even at low temperatures with a melting point of − 38.9 °C and a boiling point of 357.7 °C [20]. The signal for all other elements, in contrast, tended to increase with more intense

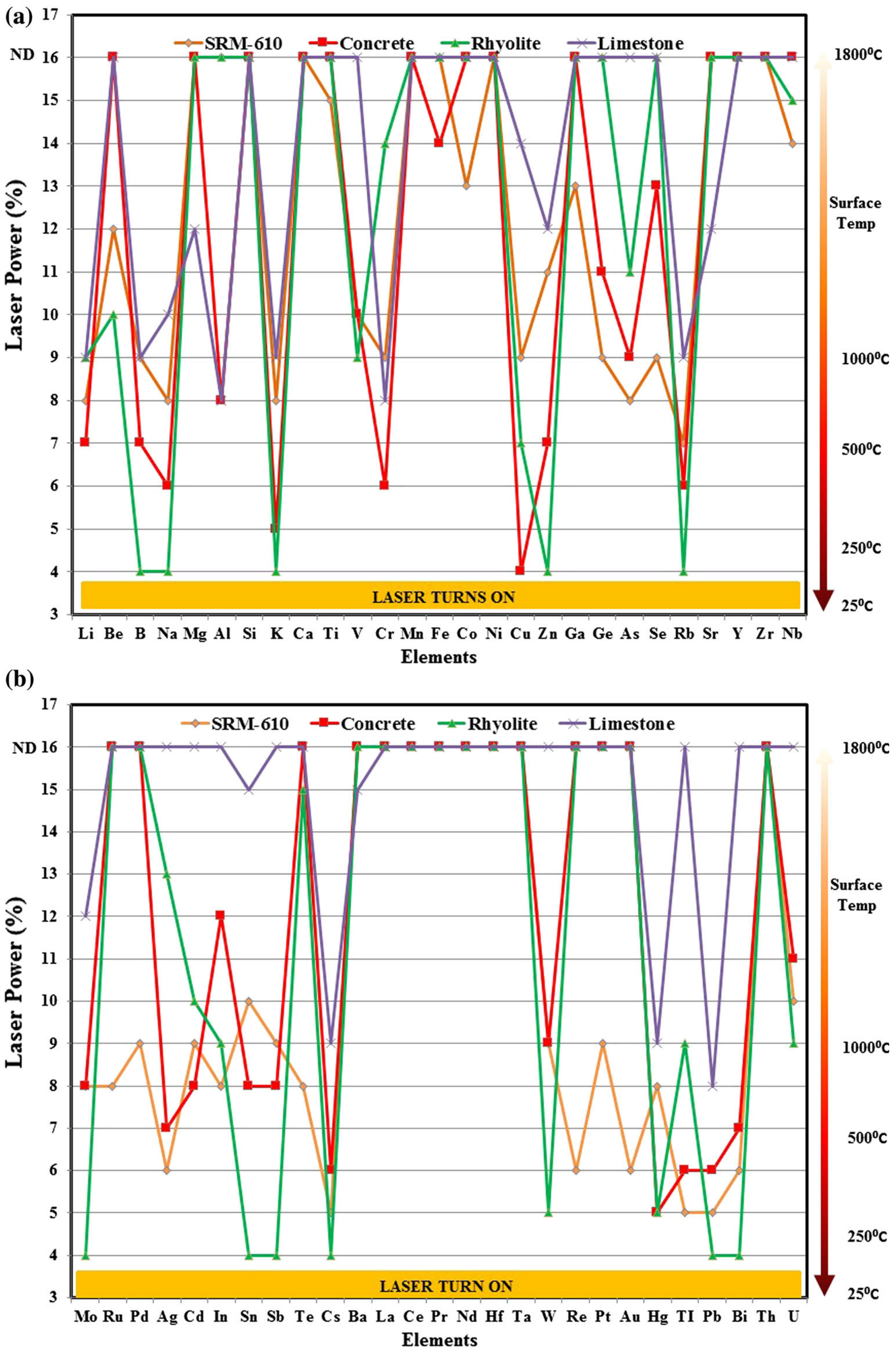

Fig. 6 **a** Evolution of elements Li through Nb from SRM-610 as a function of laser power. **b** Evolution of elements Mo through U from SRM-610 as a function of laser power

heating. Some of this rise may stem from the increase in the heated area as the laser spot size grows at higher powers, but also may be from more rapid element diffusion to the sample surface. Repeatedly heating the same spot a number of times, without necessarily melting the material, did lead to a gradual loss of signal as if the volatile elements forms were being gradually depleted.

The results highlight a suite of non-radioactive elements that might be released at relatively low temperatures after heating of the enclosing rocks by an underground nuclear or chemical explosion. Under the right conditions, such as

Table 1 The laser power setting (%) at which elements (Li–U) become detectable as volatiles for the materials SRM-610 glass, concrete, rhyolite and limestone (ND = no detection)

Element	SRM-610	Concrete	Rhyolite	Limestone	Element	SRM-610	Concrete	Rhyolite	Limestone
Li	8	7	9	9	Mo	8	8	4	12
Be	12	ND	10	ND	Ru	8	ND	ND	ND
B	9	7	4	9	Pd	9	ND	ND	ND
Na	8	6	4	10	Ag	6	7	13	ND
Mg	ND	ND	ND	12	Cd	9	8	10	ND
Al	ND	8	ND	8	In	8	12	9	ND
Si	ND	ND	ND	ND	Sn	10	8	4	15
K	8	5	4	9	Sb	9	8	4	ND
Ca	ND	ND	ND	ND	Te	8	ND	15	ND
Ti	15	ND	ND	ND	Cs	5	6	4	9
V	10	10	9	ND	Ba	ND	ND	ND	15
Cr	9	6	14	8	La	ND	ND	ND	ND
Mn	ND	ND	ND	ND	Ce	ND	ND	ND	ND
Fe	ND	14	ND	ND	Pr	ND	ND	ND	ND
Co	13	ND	ND	ND	Nd	ND	ND	ND	ND
Ni	ND	ND	ND	ND	Hf	ND	ND	ND	ND
Cu	9	4	7	14	Ta	ND	ND	ND	ND
Zn	11	7	4	12	W	9	9	5	ND
Ga	13	ND	ND	ND	Re	6	ND	ND	ND
Ge	9	11	ND	ND	Pt	9	ND	ND	ND
As	8	9	11	ND	An	6	ND	ND	ND
Se	9	13	ND	ND	Hg	8	5	5	9
Rb	7	6	4	9	TI	5	6	9	ND
Sr	ND	ND	ND	12	Pb	5	6	4	8
Y	ND	ND	ND	ND	Bi	6	7	4	ND
Zr	ND	ND	ND	ND	Th	ND	ND	ND	ND
Nb	14	ND	15	ND	U	10	11	9	ND

a nearby bore hole, these elemental signatures might precede any release of radioactive species as they would be released from the strata enclosing the explosion cavity. With a sensitive elemental detector, like an ICP-MS, it was noted that some elemental signatures remained detectable for many hours after only a short duration of modest heating long after the sample had returned to room temperature. Tests demonstrated this was not an ICP-MS memory effect when, with a short 5 s gas purge of the cell enclosing the sample, signal levels would rapidly return to pre-heating background levels. This would seem to suggest that once some elements are volatilized they can remain in the gas phase for a long period of time if not dispersed by local atmospheric conditions. During a small underground nuclear explosion there may be enough heat flow in the surrounding rocks for some days and for larger explosions this timescale might be extended to weeks or even months after the event to generate volatile species.

Elements classed as displaying intermediate volatility usually started to become detectable at higher temperatures, 400–600 °C, after the response from volatile species. These elements include: Be, V, Cr, Cu, Zn, Ga, Ge, As, Se, Mo, Ru, Ru, Pd, Cd, In, Sn, Sb, Te, W, Re, Pt, Au, and U. Measurements were made for molecular oxide peaks, especially $^{232}Th^{16}O^{+}$ and $^{238}U^{16}O^{+}$, and while traces of $^{238}U^{16}O^{+}$ were detected, levels were low, $< 1\%$ of ^{238}U and no $^{232}Th^{16}O^{+}$ was detected. The most refractory element group includes: Mg, Al, Si, Ca, Sc, Ti, Mn, Fe, Ni, Sr, Y, Zr, Nb, Ba, La-Lu, Hf, Ta, and Th with most of these elements not detected even from the 500 ppm multielement SRM-610 glass heated well above its melting point. Data for P, S, Ir, and Os from the materials tested were inconclusive. Sulfur would be expected to be readily volatile, as is probably P. Quadrupole ICP-MS sensitivity for P and S is relatively poor, owing to their low ionization efficiency in the ICP, combined with the presence of many molecular ion interferences that elevate the backgrounds. Certain species of Os, like OsO_4, are known to be very volatile, although given its very low natural abundance (like Ir), it is highly unlikely to be encountered.

While ICP-MS is a very sensitive multi-element detector, transporting one to the field to make measurements is not without logistic problems—namely power, argon supply, and weight. A sensitive, truly fieldable mass spectrometer for elemental-isotopic analysis would be highly desirable for this application. There may be alternative detection techniques for a few elements that could be taken into the field, for example, cold vapor atomic fluorescence for Hg [21]. One possible accidental benefit of this work with development in ICP-MS atmospheric sampling is its application to laser ablation analysis where ns-fs laser pulses are employed. There would be some loss in sensitivity due to reduced sampling efficiency and the response time might be slower, but this method eliminates the requirement to make the sample fit inside an ablation cell. While detection limits with this laser heating approach were not evaluated, high signal intensities $> 10^7$ cps were easily attained for many of the most volatile elements from the 500 ppm glass standard, indicating ppt level detection ought to be possible for these elements.

There is clearly much more work that could be done in this area to identify both the nature and chemical forms of the volatilized species of the elements in order to refine their detection. The highly energetic conditions of the ICP reduce particulates and molecular species to their atomic constituents all too readily. Any closer investigation of volatile element speciation needs to be undertaken using less aggressive methods of atomization and ionization that may well be used for the analysis of organic molecules. The existence of an organic signature from underground explosions, chemical or nuclear, would seem highly likely as organic compounds are widely present in various forms in almost every rock type. It may also turn out, for some elements, that the volatile species involved actually change with temperature, in that different compounds containing the same element are emitted at different temperatures. All measurements were performed on dry samples, but the presence of both water and other chemicals like NaCl will often be present in rocks. These too may also modify the chemistry of the volatilized species and the temperature at which they become detectable.

Conclusions

The work reported herein highlights the potential for new non-radioactive signatures that rely on thermal release elements in a volatile form from the enclosing strata and should be detectable from a small underground nuclear or conventional chemical explosion. The approach also potentially highlights which radionucleides, besides Xe, might be the most mobile in this setting. A new method of atmospheric sampling has been developed and combined with the high sensitivity of ICP-MS, making these volatile signatures readily detectable. It was observed that under relatively stable atmospheric conditions the signature from some elements, like Pb, can persist long (hours) after any heating of the specimen has ceased and sample temperature returned to ambient. Currently ICP-MS may not be the most fieldable approach to make these measurements onsite, but other more portable elemental detection methods could be developed for this application.

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