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The use of lithium heteropolytungstate as an alternative to bromoform for heavy media separations

Mineralogy, Petrology and Biostratigraphy Facility

Internal Report IR/11/049



BRITISH GEOLOGICAL SURVEY

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INTERNAL REPORT IR/11/049

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Foreword

This report is the published product of a study by the British Geological Survey (BGS) and was produced under the Science Facilities' Maintenance and Development of Capability (MaDCap) project.

This report aims to provide a procedural guide for heavy media separations using non toxic lithium heteropolytungstate (LST) in place of the more toxic bromoform and methylene iodide previously used for this purpose.

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I would like to thank to Gren Turner for his assistance with SEM imaging, Mark Ingham and his team for producing the XRFS data and Simon Kemp for helping to draft this report.

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Summary

This report describes a suggested new procedure for heavy media separation using non-toxic lithium heteropolytungstate as a replacement for bromoform and other halogenated organic solvents. This work was carried out as part of the Science Facilities' Maintenance and Development of Capability (MaDCap) project. Lithium heteropolytungstate is commonly known as LST, relating to its composition of lithium, sodium and tungsten.

The report firstly describes heavy liquids that were traditionally used for this purpose and then details a new heavy media separation methodology using LST.

Results indicate that LST is by far the safest and most efficient heavy liquid suitable for this type of mineral separation.

Issues regarding LST stability indicate potential contamination through initial sample preparation methods. A small study indicates that steel swarf from disc milling is responsible for heteropoly blue contamination of the LST and not pyrite as suggested by the manufacturers. It is therefore critical that extreme care should be taken when undertaking any sample preparation prior to heavy liquid separation.

1 Introduction

Heavy media separations (HMS) are commonly used in the geosciences to divide crushed rocks or soils into their respective light and heavy specific gravity fractions (e.g. Carver, 1971; Koroznikova *et al*, 2007). Possibly the most well known example of this process is panning for gold where water provides the media and the panning action separates the denser gold particles from the lighter gangue minerals.

In the laboratory, HMS are employed to separate mineral grains prior to further analysis such as X-ray diffraction, optical and scanning electron microscopy or dating techniques. A range of chemicals have traditionally been used as heavy media (Table 1) which can be adjusted to specific densities using acetone or water to tailor separations for particular purposes.

Table 1. Common heavy liquids used for heavy media separations.

Heavy Liquid	Density	Viscosity	Vapour Pressure	SG Adjusted	Cost
Bromoform	2.87g/mL	1.8cP	5.9 mm Hg at 25°C	Acetone	1 litre - £970.40 (Fisher.co.uk)
Tetrabromoethane (TBE)	2.95g/mL	9cP	0.02 mm Hg at 25°C	Acetone	1 litre - £203.44 (Fisher.co.uk)
Methylene iodide	3.31g/mL	2.6cP	1.2 mm Hg at 25°C	Acetone	1 litre - £277.38 (Fisher.co.uk)
Lithium heteropolytungstate (LST)	3.3g/mL	10cP	Negligible	De-ionised Water	1 litre - £511.79 www.polytungstate.co.uk
Sodium polytungstate (SPT)	3.1g/mL	20cP	Negligible	De-ionised Water	1 litre - £850.56 http://www.geoliquids.com/

1.1 HALOGENATED ORGANIC SOLVENTS

Traditionally, the most common types of heavy media employed in the geosciences were the halogenated organic solvents such as bromoform, methylene iodide and tetrabromoethane (TBE) but these are now regarded as being highly toxic (<http://msds.chem.ox.ac.uk>) and strict H&S protocols are required for their use.

During the early 1900s, halogenated organic solvents were commonly available but concerns over their toxicity led to their decline and difficulty in obtaining supplies. All can be absorbed through inhalation and ingestion and bromoform can be absorbed through the skin. TBE is toxic when given in small, prolonged and repeated doses. All are known to cause eye, skin and respiratory irritation, headaches, nausea, kidney and liver damage. TBE is also a suspected carcinogen. Animal tests suggest bromoform may be carcinogenic but, as yet, no human information is currently available.

A further disadvantage with using bromoform as a heavy liquid is the post separation contamination of the sample. Even with extensive cleaning of the sample using copious amounts of acetone (which is unsatisfactory from both H&S and environmental perspectives), the sample can remain stained a yellow/brown colour.

The final disadvantage with using organic solvents for HMS is the associated waste from the cleaning of samples and apparatus. Acetone is usually used for cleaning and the acetone/bromoform waste is collected for disposal or, on occasion, reclamation. The reclamation

of bromoform is achieved through several gravity/density separations, both of which can be time consuming and undesirable given the inherent toxicity of bromoform.

1.2 POLYTUNGSTATES

The toxicity of halogenated organic solvents combined with increasing difficulty in obtaining supplies highlighted the need for alternative media. Internet-based research (S.J.Kemp *pers.comm.*) revealed that polytungstate media (Table 1) appeared to offer a suitable alternative.

Apparent advantages of polytungstate media over organic heavy liquids include:

- Polytungstate is non-toxic and can be used in the laboratory in open vessels without the need for forced extraction or specialised personal protective equipment.
- Polytungstate costs are similar to halogenated organic solvents but polytungstates are 99% recoverable through the recycling of undiluted media and further reclamation from washing both samples and apparatus.
- Polytungstate specific gravity (SG) can be altered by the addition or removal of deionised water. Sodium polytungstate (SPT) has an SG of 3.1 g/ml and lithium heteropolytungstate (LST) has a maximum SG of 3.3 g/ml. [However, Li salts can begin to crystallize at 3.3 g/ml, so it is recommended that a limit of 3.15g/ml is achieved.]
- The high solubility of the Li salts reduces the surface contamination of the heavy concentrate from the heavy liquid.
- The viscosity of the LST can be changed through the introduction of heat, although this is not normally required. The viscosity of LST at average room temperature (~20°C), remains favourably low at 10cP and an average separation of c.50 g of 63-250 µm sample taking 10-20 minutes to complete.

LST has three key advantages over SPT:

- LST is considerably less viscous than SPT (Figure 1)
- LST is more soluble in water than SPT, facilitating changes in SG and general handling.
- LST is considerably cheaper than SPT (Table 1)

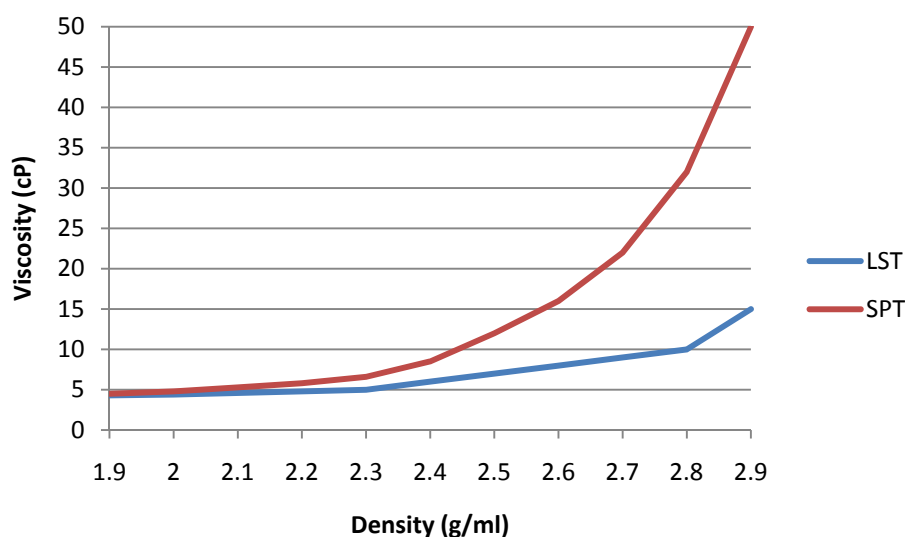


Figure 1. Density as a function of viscosity adapted from <http://www.heavyliquids.com>.

The aim of this project was therefore to explore the use of LST as a replacement for halogenated organic solvents and to produce a new methodology for heavy media separations in BGS.

2 Materials and Methodology

2.1 SAMPLE PREPARATION

Samples typically submitted for HMS are sands, soils and indurated rocks. Indurated rocks are typically jaw-crushed and milled via rotary disc milling before separation.

In order to disaggregate samples prior to HMS, they are typically dispersed in deionised water using ultrasound treatment. Such treatment may need to be repeated if the samples are particularly clay- or organic-rich. Dispersed samples are then wet screened on a 63 μm sieve to remove all fines and then oven dried at 40°C overnight. The removal of fines is necessary to avoid the formation of sediment aggregates within the heavy liquid and to facilitate the distinction between heavy and light fractions.

Typically 50 g disaggregated >63 μm material is then placed in a 250 ml separating funnel with 250 ml LST. The upper-size limit of the material is variable but should not exceed 1 mm due to the size of the separating funnel's tap.

2.2 HEAVY MEDIA SEPARATION

In a fume cupboard, approximately 100 ml of LST was poured into a separating funnel, followed by 50 g of sample and topped up with a further 150 ml LST (Figure 2). This “sandwiching effect” ensures that the sample is thoroughly dispersed. Where necessary the sample was stirred with a glass rod to mobilize any particles which may be stuck to the inside of the glass or any heavy minerals that could be entrained within the light fraction.

The LST can be prone to evaporation if left for any long period of time, potentially changing the SG; therefore it is important to seal any bottles of LST not being used and to plug the inlet of the separation funnels. It is also advisable to turn off the forced extraction within the fume cupboard as this will potentially increase evaporation.

An average sample size of 50 g in 250 ml of LST will take up to 20 minutes to separate but this time can vary depending on the grain-size of the sample and the ratio of heavy to light minerals present. Once a clear division between the heavies and lights is apparent, the heavy fraction can be tapped off, ensuring none of the light fraction is included. The heavy fraction is retained on a Whatman 1 90 mm \varnothing qualitative filter paper. Excess LST was pumped through the filter paper and into a conical vacuum flask. The heavy fraction was thoroughly washed in warm deionised water and left to dry overnight. The remaining LST and light fraction was tapped off in the same manner but with a new filter paper.

In this method a small vacuum pump was used to pull the LST through the filter paper, dramatically reducing the time to separate the solids and liquids.

It is essential to change filter papers between the collection of the heavies and the collection of the lights; this is due to the precipitation of lithium salts into the fibres of the filter paper that can ultimately reduce the permeability of the paper.

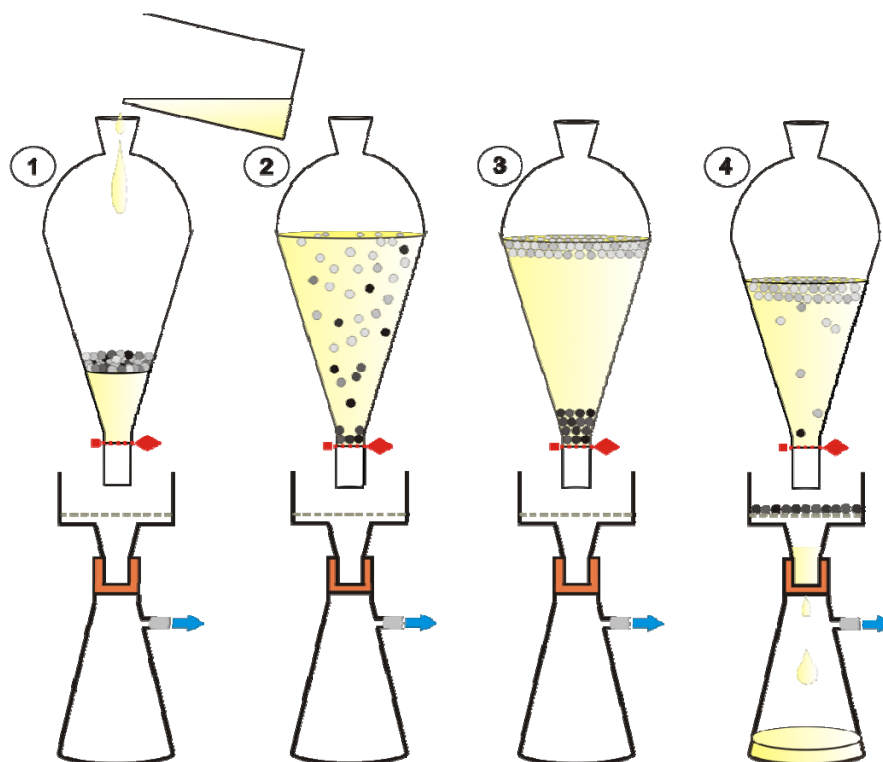


Figure 2. Simplified heavy media separation method. ①. *c.*100 ml LST added to separating funnel followed by *c.*50g sample and a further *c.*150ml LST, ② Particle dispersion following dispersion with a glass rod, ③ Separation and division of minerals (*c.*20 minutes), ④ Heavy mineral concentrate tapped off and recovered on filter paper.

2.3 RECOVERY PROCEDURE

Undiluted LST was retained for further separations. LST washings from the samples and equipment were oven dried at 60°C to form Li salt crystals. These Li salts were ground in a pestle and mortar to produce a fine powder that was then re-dissolved in deionised water. The SG was then finely adjusted to 2.85 g/ml using density beads (2.8 g/ml (green), 2.85 g/ml (mauve) and 2.9 g/ml (yellow)). A free dilution calculator for recombining LST at a specific SG can be found at:

http://www.chem.com.au/products/lstheavyliquid/tech_resources/lstcalc.exe

3 Results

Repeated testing has shown that a typical LST HMS (c.50 g of sample) can be completed within 10-20 minutes (Figure 3) and up to four individual separations can be achieved simultaneously. It is therefore possible to produce 12 separations within a standard working day, as opposed to eight separations when using bromoform, a 50% increase in productivity.

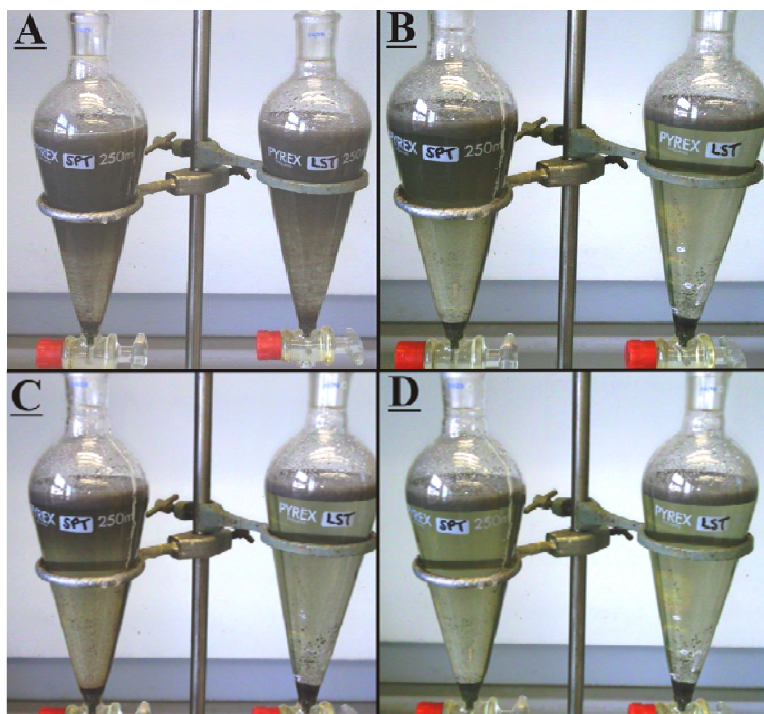


Figure 3 Separation times for SPT (left) and LST (right): A. 0 Minutes, B. 5 Minutes, C. 10 Minutes and D. 20 Minutes.

A concern with using bromoform, especially when SEM analyses are required, was the surface contamination of bromine on the mineral grains; EDXA spectra and imaging results were both reported as being affected. Heavy media separations using LST produced clean heavy and light fractions (Figure 4), EDXA spectra and associated images from samples separated with LST, reveal no indication of lithium/tungsten surface contamination.



Figure 4 Left, Sample of beach sand. Right, heavy and light fractions, separated using LST.

4 Discussion

Repeated separations of different samples suggest that LST provides an excellent replacement for bromoform in routine HMS.

Due to the low viscosity of LST (10cP) there is very little aggregation of particles in suspension; the low viscosity produces a quick and clean divide between the heavy and light mineral phases.

The high solubility of the lithium salts allows for up to 99% recovery of LST, this is not only superior to bromoform but the manner in which it is accomplished is also favourable, both financially and environmentally. Historically, bromoform separates were washed in acetone, although this was not always accomplished to a high standard, as samples were frequently stained (this was due to contact with the bromoform rather than the lack of cleaning, post separation). However, unlike bromoform, LST separates are cleaned with warm de-ionised water, which removes 100% of the LST.

However, a disadvantage noted whilst working with the LST is the occasional blue/black discolouration of the liquid (Figure 5), which has been termed 'heteropoly-blues'; (www.polytungstate.co.uk).

Sometu, the producers of LST Fastfloat and SPT, suggest using 30% weight hydrogen peroxide (1-2 ml) to re-oxidise the LST and remove the discolouration. However, in testing this appears to have little effect. To reduce the blue colouration, the affected LST must be diluted further with de-ionised water and left until transparent; this can take hours or days depending on the degree of discolouration. According to Sometu, this discolouration may be permanent and the heavy liquid deemed unusable. For this reason it is of importance that this phenomenon is understood. A small investigation into the cause of heteropoly blues is shown in Appendix 1.



Figure 5. Example of heteropoly blues during LST testwork.

5 Conclusion and recommendations

Research carried out during this study suggests that LST offers the safest, fastest and most economical method of heavy media separation.

On the basis of a limited study (Appendix 1), it would appear that steel swarf from disc milling reacts with LST to produce heteropoly blues. This may be due to the slightly acidic nature of the LST (pH~4) promoting dissolution and oxidation of the steel resulting in the blue discolouration of the reduced tungstate. This study indicates that this reaction is limited to elemental iron and does not appear to affect iron bearing minerals (pyrite, magnetite, hematite etc). Therefore further emphasis on reducing contamination through sample preparation should be applied for HMS.

To maximise efficiency, reduce contamination from sample preparation and increase the capabilities of this method, three alternative methods could be applied:

- The preferred method for the disaggregation of indurated rock samples has been disc milling; this is because of the reproducibility to create a well constrained particle size. Because of the potential for contamination during disc milling especially when dealing with abrasive rocks, a different method for milling is needed. The best alternative method is to mill with short bursts using a tema mill with agate pots. Between each stage of tema milling the sample should be sieved at the appropriate size required. This recommendation will ultimately reduce sample contamination and eliminate the potential for the heteropoly blue reaction to occur.
- Superpanner/Wilfley/Rodgers Table. These techniques are fundamentally the same, using a vibrating surface to which a feed of wet slurry is added. The action of the water, gravity and shaking will separate minerals into their respective heavy and light fractions. However, the heavy fraction is not specific to a particular SG. For samples that contain a very small percentage of heavy minerals, this method is able to process large volumes of sample to concentrate the heavy minerals. The concentrated HMS can then be used in a LST separation to extract minerals of a particular SG.
- Magnetic separation (Franz). This procedure could be used prior to heavy liquid separation and would have the advantage of creating two distinct separates, one magnetic and one non magnetic.

Appendix 1 Heteropoly blues

The blue/black discolouration of LST and SPT known as heteropoly blues or 'tungsten blues' has previously been described (e.g. Sometu, Berlin www.sometu.de/index.html). These authors found that the polytungstate group is extremely sensitive to reducing agents and suggested that the interaction of iron sulphide (pyrite) was one of the most common causes of heteropoly blues in HMS.

However during this study, pyrite grains placed in contact with LST produced no apparent discoloration of the heavy liquid.

To determine the cause of heteropoly blues, a further study was made during HMS of an aplitic microgranite, sourced from the north-east Grampian granitic suite (Parry, 2004).

A portion of the milled microgranite was submersed in LST in a petri dish and examined under a binocular microscope. Grains that showed a reaction with the LST (Figure 6) were picked and immediately washed in de-ionised water. To avoid potential contamination from the heteropoly blue reaction, representative grains were picked under ethanol and placed onto an aluminium stub for scanning electron microscope analysis (Figure 7).

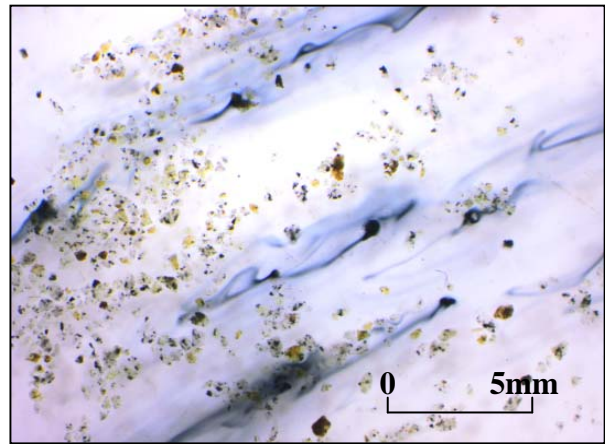


Figure 6. Optical photomicrograph showing heteropoly blues due to disc mill contamination.

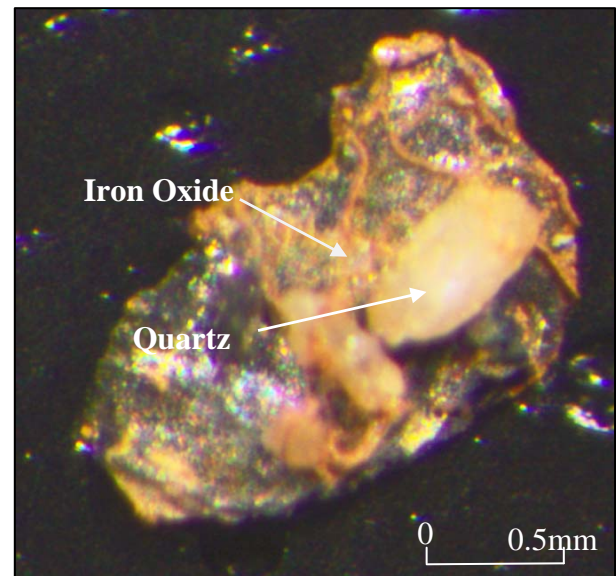


Figure 7. Disc mill swarf with imbedded quartz. Note the iron oxide, a bi-product of wet sieving.

SCANNING ELECTRON MICROSCOPY

SEM analysis was performed using a LEO 435VP digital scanning electron microscope. The instrument was used in variable (for un-coated samples) pressure mode. The SEM instrument was equipped with a KE Developments four-quadrant (4 diode-type) solid-state detector for backscattered electron imaging (BSEM). Phase/mineral identification was aided by qualitative observation of energy-dispersive X-ray spectra recorded simultaneously during SEM observation, using an Oxford Instruments INCA energy-dispersive X-ray microanalysis (EDXA) system. Representative grains were imaged under SEM and point analyses (EDXA) were taken to determine their broad chemistry.

The grains causing the heteropoly blues reaction were identified as small, steel fragments of the disc mill used to prepare the sample (Figures 7, 8 and 9). One of the fragments was replaced in a small quantity of LST and within seconds the heteropoly blue reaction re-occurred. The second fragment was placed onto a SEM stub for analysis. As shown in Figure 8, the fragment is primarily composed of iron and carbon with minor vanadium and chromium, representing a typical hardened steel alloy composition. The aluminium and silica EDXA peaks represent included alumino-silicate contamination produced from the milling abrasion of silica-rich rocks.

A further piece of swarf that had been in contact with the LST for some time, was removed from the milled sample, washed and placed on another SEM stub. Examination of this grain showed white platy crystals of tungsten that appear to be growing from within the swarf as opposed to merely growing on the surface, suggesting that the swarf has undergone a degree of dissolution whilst submersed in the LST (Figure 9).

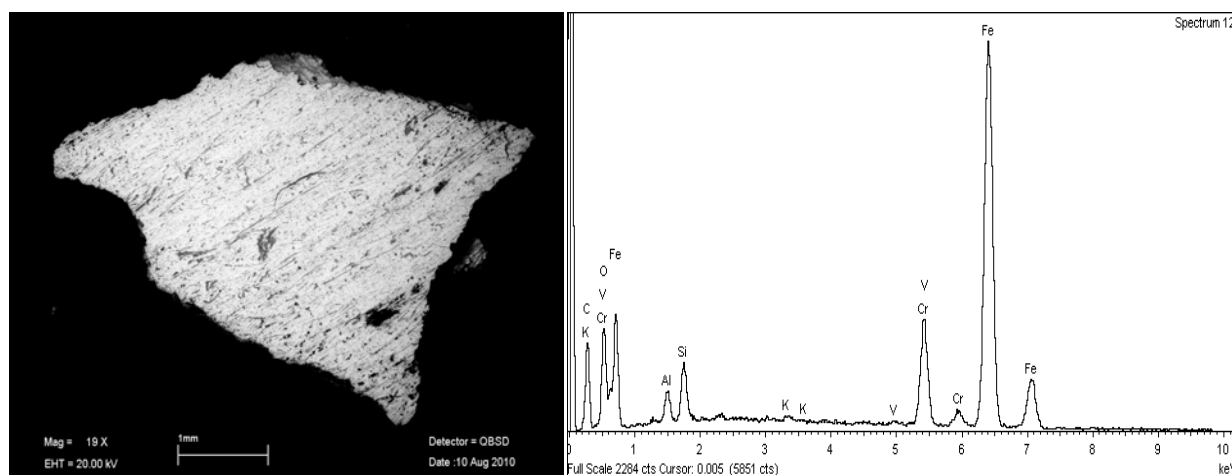


Figure 8. BSEM photomicrograph of a disc mill swarf (un-milled) and associated EDXA spectra.

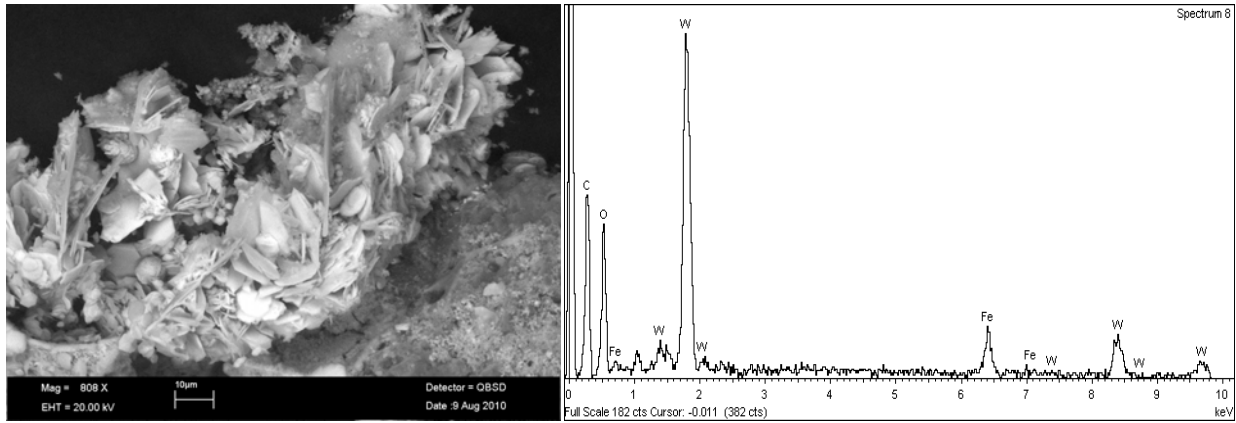


Figure 9. BSEM photomicrograph of disc mill swarf (milled) engulfed with platy tungsten precipitates and associated EDXA spectra.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES)

In order to identify the reactive components of the disc mill swarf that caused the heteropoly blues, samples of both clean and contaminated LST were analysed by ICP-AES. Samples were diluted (x100) with 1% HNO₃ and analysed using a Perkin Elmer Optima 7300 DV OES with WINLab 32 software. The results show that the only major difference in the LST chemistry was an increase of c.13,000 ppm Fe in the ‘heteropoly blue’ contaminated LST sample (Figure 10).

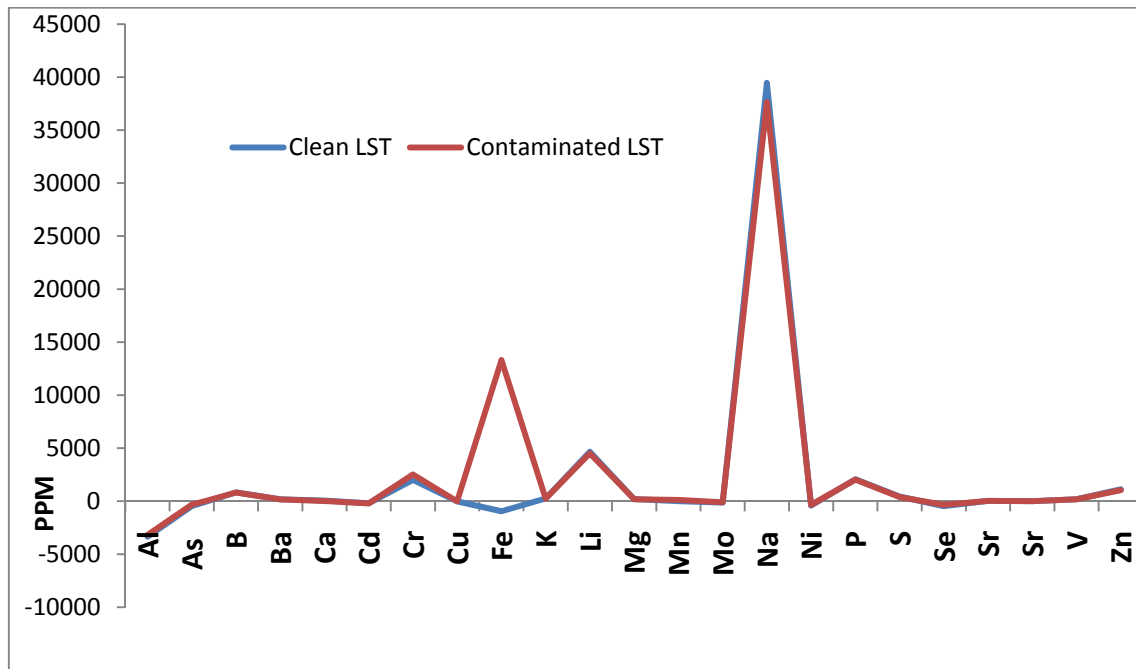


Figure 10. ICP-AES data comparing clean LST with LST contaminated with ‘heteropoly blues’.

X-RAY FLUORESCENCE SPECTROMETRY (XRFS)

In order to test whether disc mill swarf was being produced during routine milling preparation and therefore causing the heteropoly blue problem, trials were performed using a highly abrasive sample of the aplitic microgranite (Parry, 2004).

The microgranite was firstly jaw-crushed to <2 mm and three representative subsamples were taken for milling:

- MPLQ064: Milled using agate planetary ball mill (blank).
- MPLQ065: Disc milled using a set of very worn grinding discs.
- MPLQ066: Disc milled using a nearly new set of grinding discs.

12 g subsamples of the three splits were taken and further milled (agate ball milling) with 3 g binding wax and pressed into 40 mm pellets for XRFS geochemical analysis.

XRFS analysis was performed using a sequential, fully automatic Philips PW2440 MagiX PRO wavelength-dispersive spectrometer, fitted with a 66 kV generator and a 4 kW rhodium end-window X-ray tube and controlled via PCs running PANalytical SuperQ (version 4.0D) XRF application software.

The three samples were analysed for major and trace elements, with particular focus on the known elements found in the hardened steel disc (Table 2).

Table 2. XRFS data.

	Fe ₂ O ₃ (ppm)	V (ppm)	Cr (ppm)	Mo (ppm)	W (ppm)
Agate ball mill	9200	3	<1	<1	2
Worn disc mill	11200	6	124	6	7
New disc mill	10600	4	93	3	5
Disc composition (Retsch Ltd)	835900	5000	120000	7000	6000

Using the agate milled sample (MPLQ064) as a baseline for the microgranite chemistry, the concentration of contamination from both the worn and new disc could be assessed (Table 3). It is clear from this data that the degree of contamination from disc milling increases with wear, as would be expected.

Table 3. Contamination from disc milling.

	Fe ₂ O ₃ (ppm)	V (ppm)	Cr (ppm)	Mo (ppm)	W (ppm)
Worn disc mill	2000	3	123	5	5
New disc mill	1400	1	92	2	3

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