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(54) Title of the Invention: **Method and system for low level metal analysis of mineral samples**
Abstract Title: **Method and system for metal analysis of mineral samples**

(57) A metal value collector device **10** for adsorbing a metal value from a digestion mixture, where the metal value collection device is sized and shaped for association with a detector. The metal value collector may be a plate of adsorbent material, preferably activated carbon cloth, or may be a bed of adsorbent material, preferably carbon granules or ion exchange resin beads. A sample preparation device **14** comprises a vessel **16** for receiving a sample, a digestion composition, a closure **18** for the vessel, and metal value collector **10**. A method for preparing a sample comprises mixing the sample and digestion medium and contacting the mixture with the metal value collector. A system for metal value analysis comprises the digestion mixture and metal value collector and a detector. Preferably the detector is an x-ray fluorescence detector, especially a portable XRF detector (pXRF). Also claimed are: a digestion composition comprising a metal lixiviant and an alkali compound; and a metal value collector device reference standard having a known amount of adsorbed metal value.

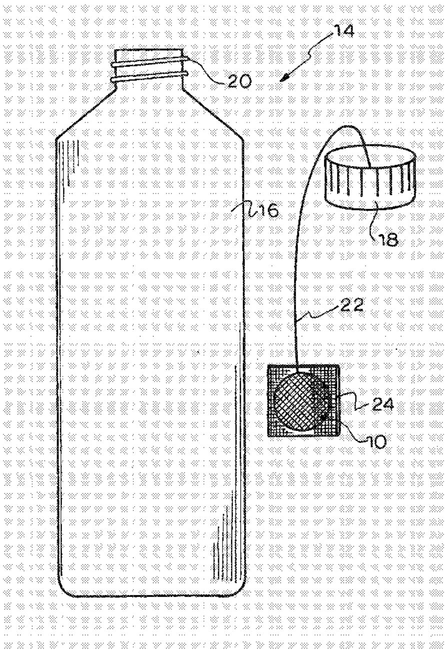

FIG. 2a

At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

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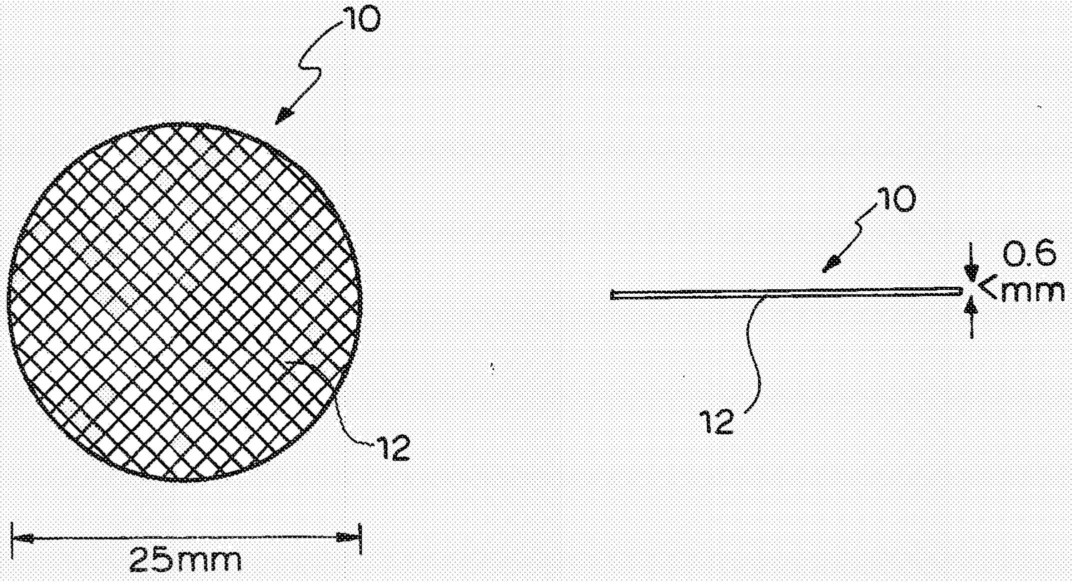

FIG.1

18 09 17

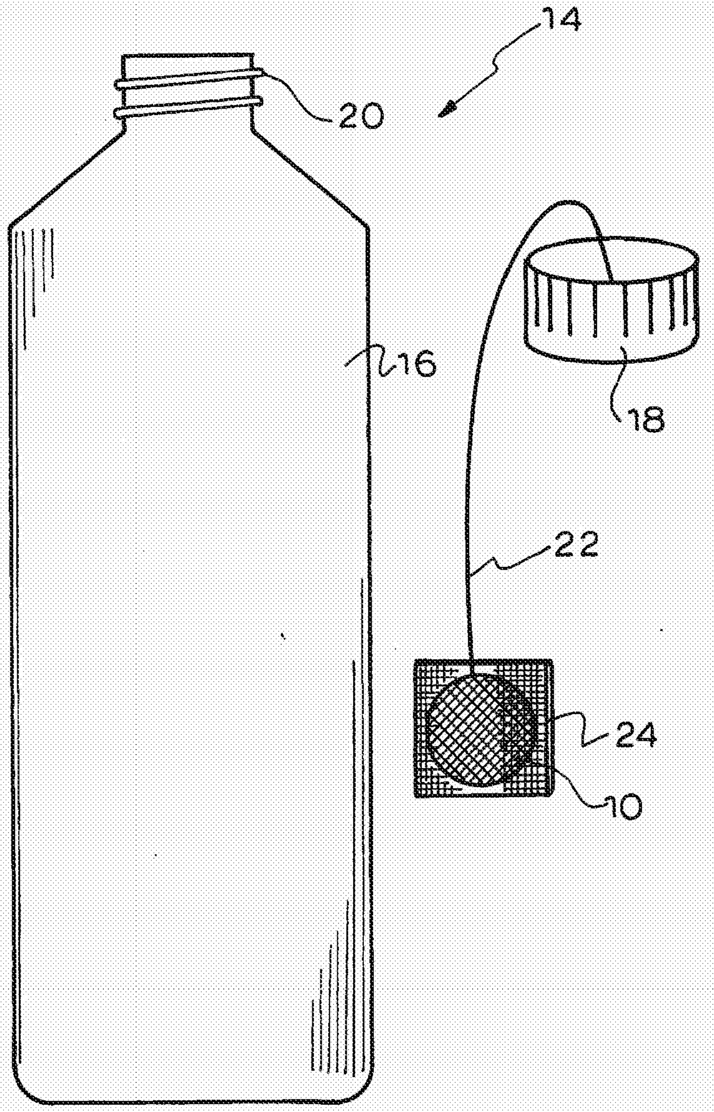

FIG. 2a

18 09 17

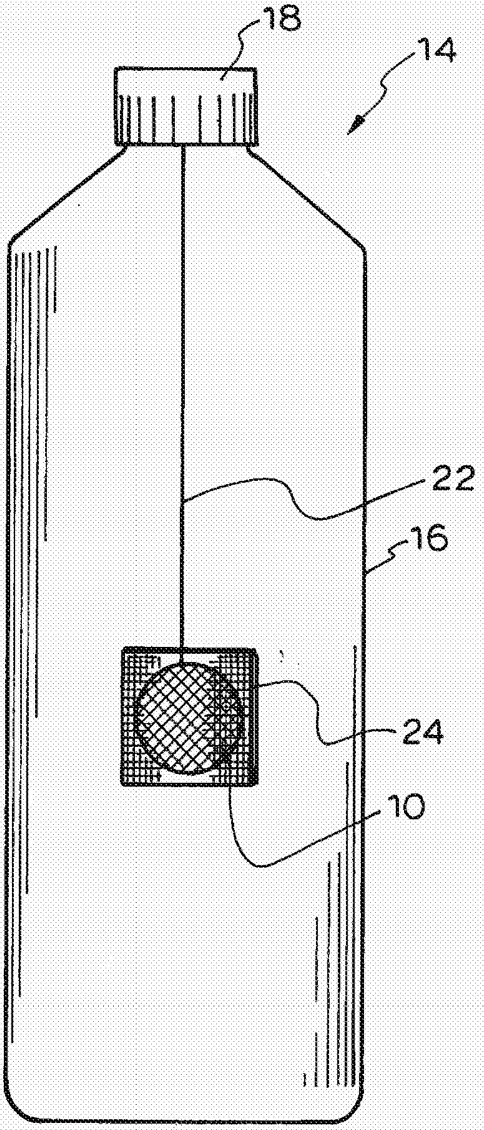

FIG. 2b

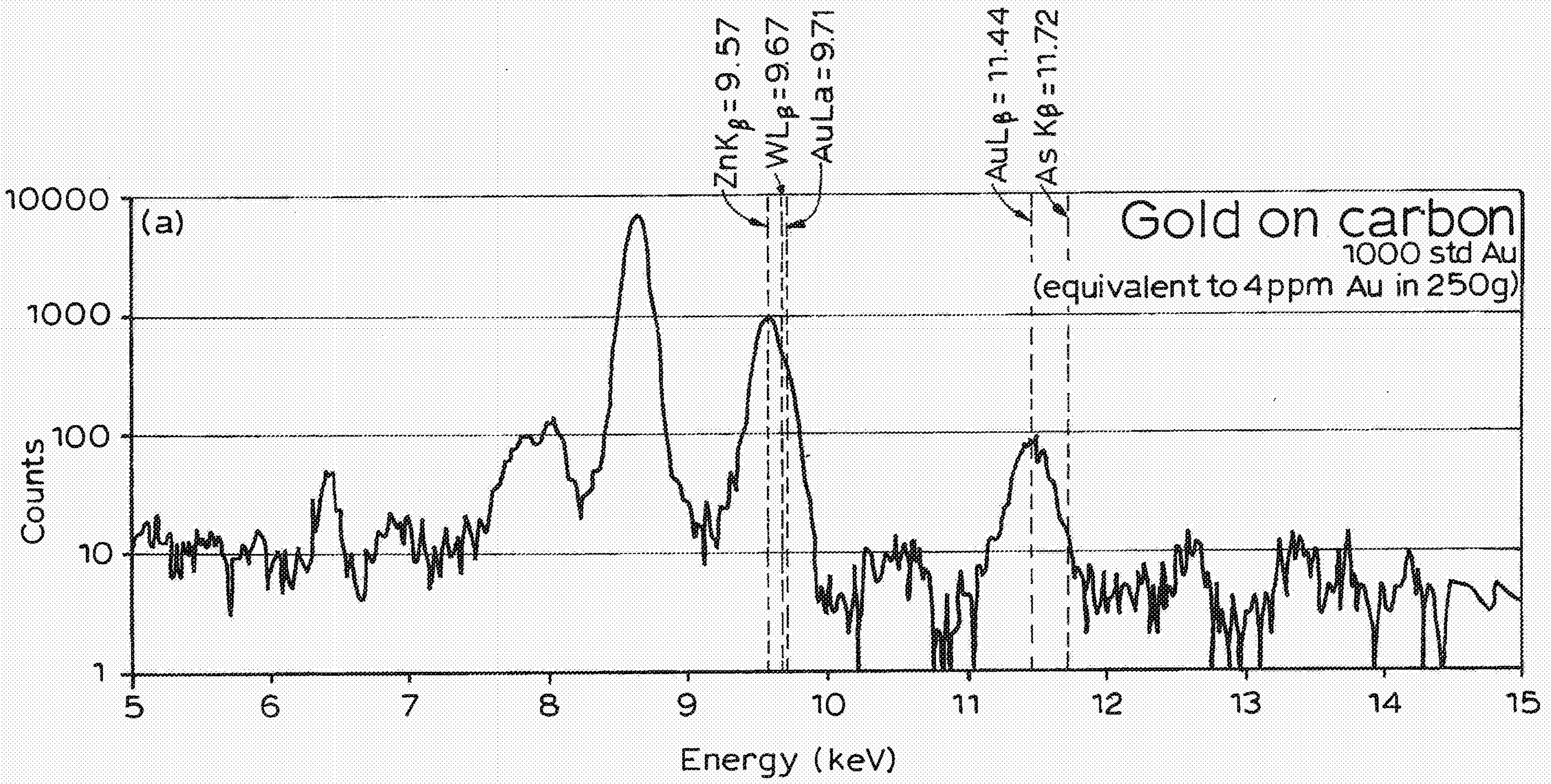
ZnK$_\beta$ = 9.57
WL$_\beta$ = 9.67
AuLa = 9.71
AuL$_\beta$ = 11.44
As Kβ = 11.72
(a)
Gold on carbon
1000 std Au
(equivalent to 4ppm Au in 250g)
Counts
10000
1000
100
10
1
5
6
7
8
9
10
11
12
13
14
15
Energy (keV)

FIG. 3a

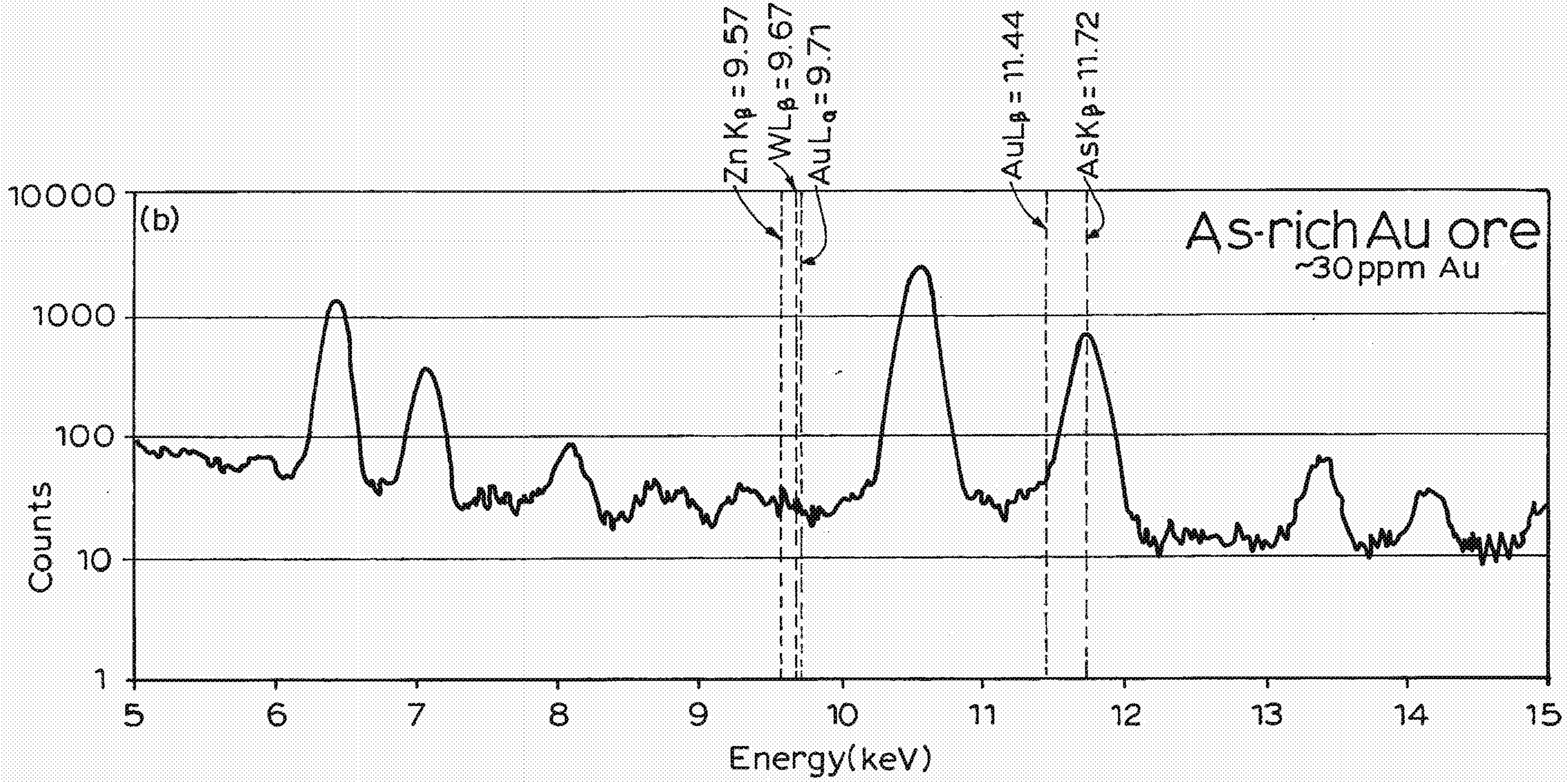
(b)
As-rich Au ore
~30ppm Au
Zn Kβ = 9.57
WLβ = 9.67
AuLα = 9.71
AuLβ = 11.44
AsKβ = 11.72
Counts
10000
1000
100
10
1
5
6
7
8
9
10
11
12
13
14
15
Energy(keV)

FIG. 3b

18 09 17

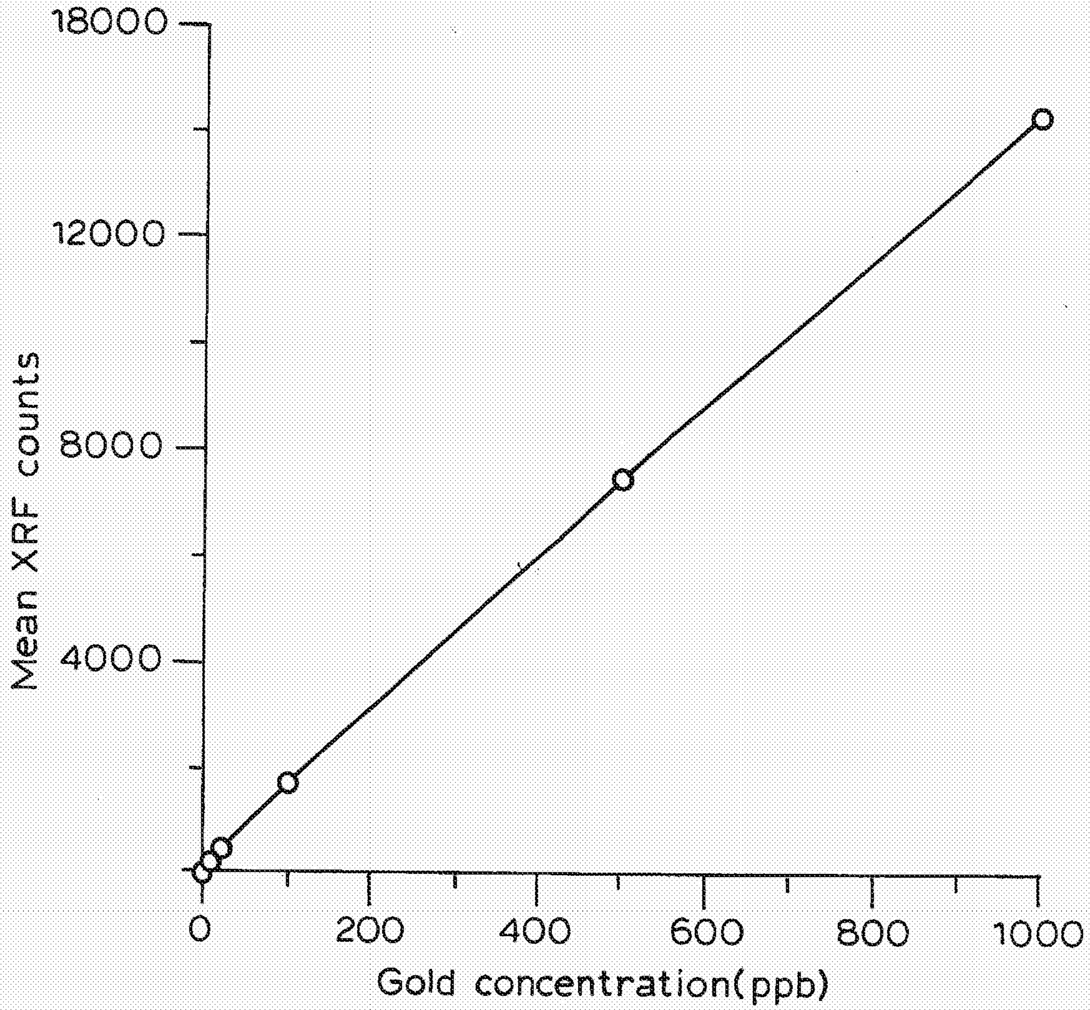
18000
12000
8000
4000
Mean XRF counts
0
200
400
600
800
1000
Gold concentration(ppb)

FIG. 4

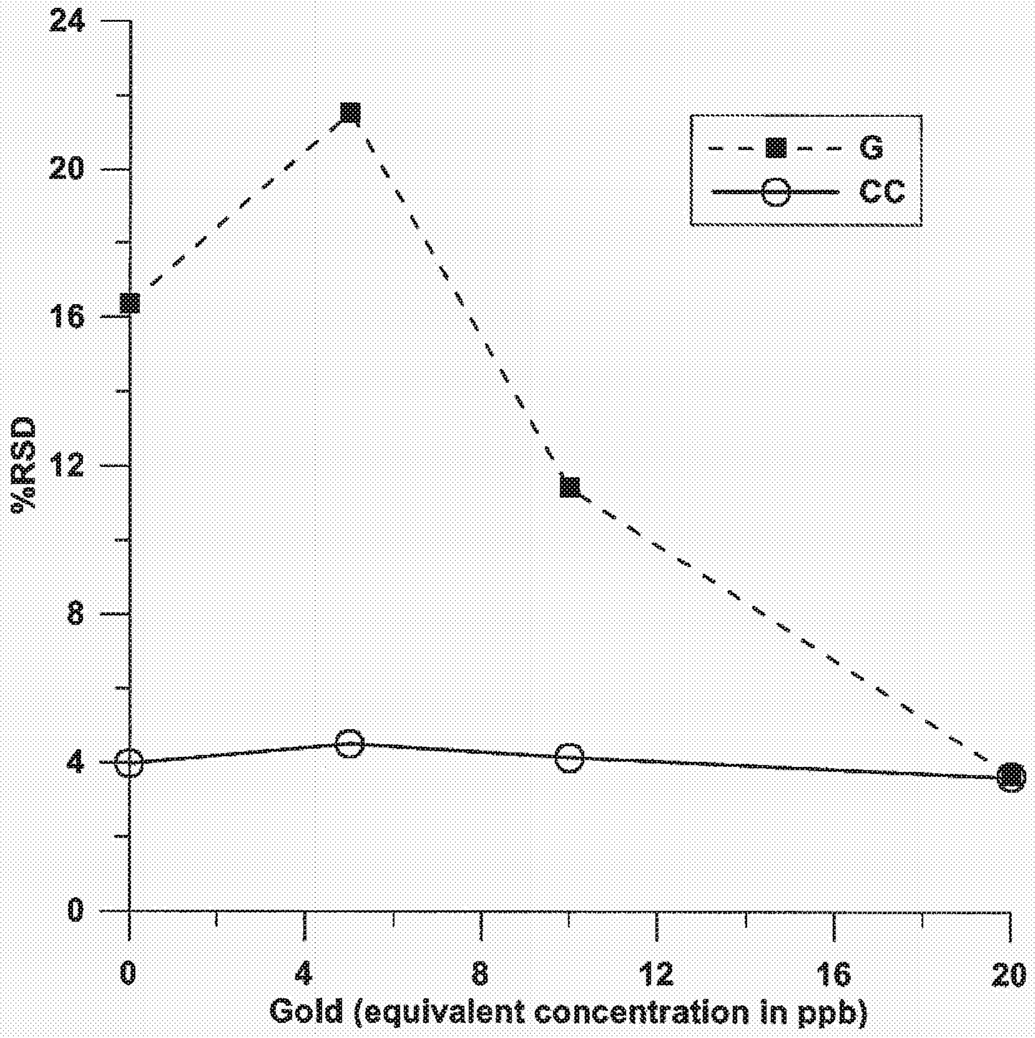
24
20
16
12
8
4
0
%RSD
0
4
8
12
16
20
Gold (equivalent concentration in ppb)
G
CC

FIG. 5

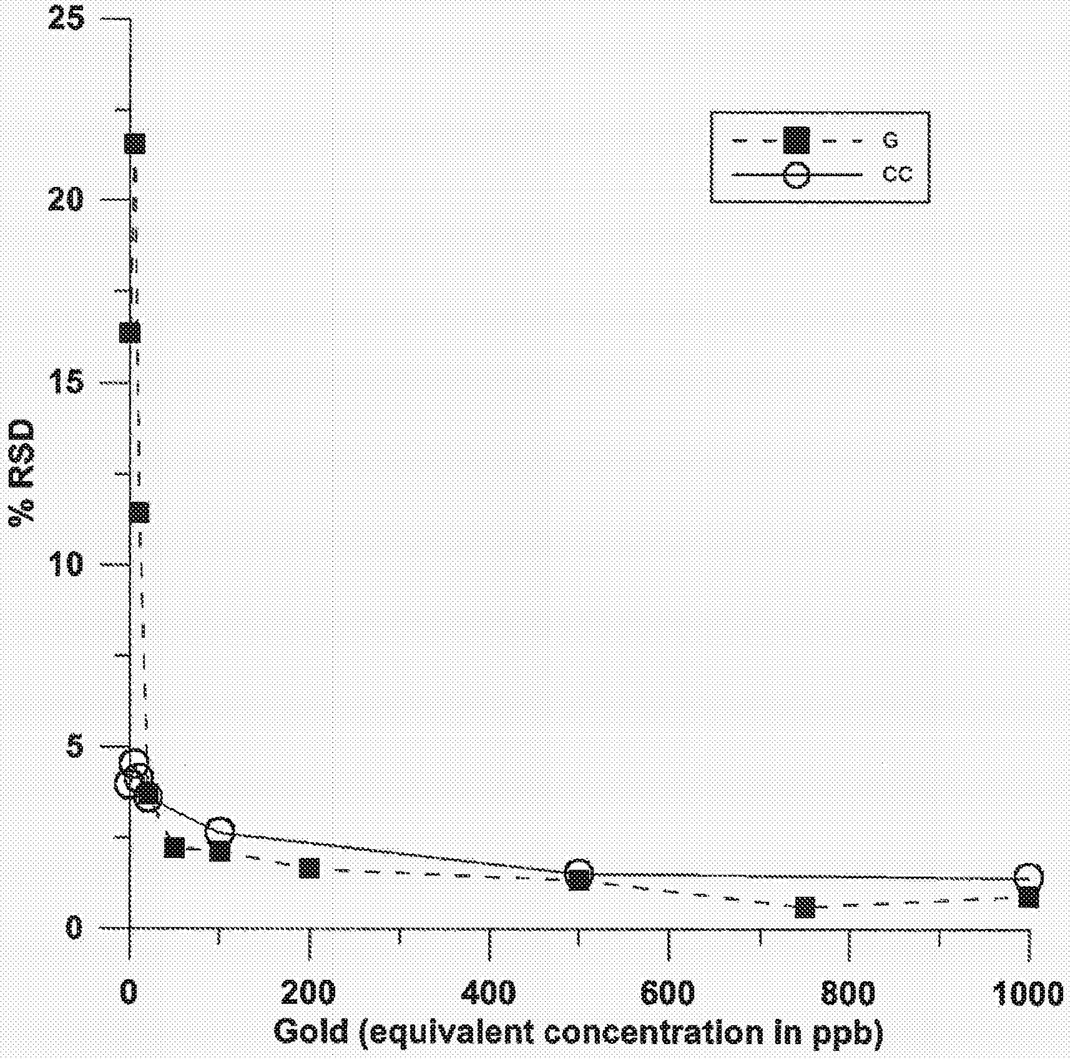
25
20
15
10
5
0
% RSD
0
200
400
600
800
1000
Gold (equivalent concentration in ppb)
G
CC

FIG. 6

18 09 17

18 09 17

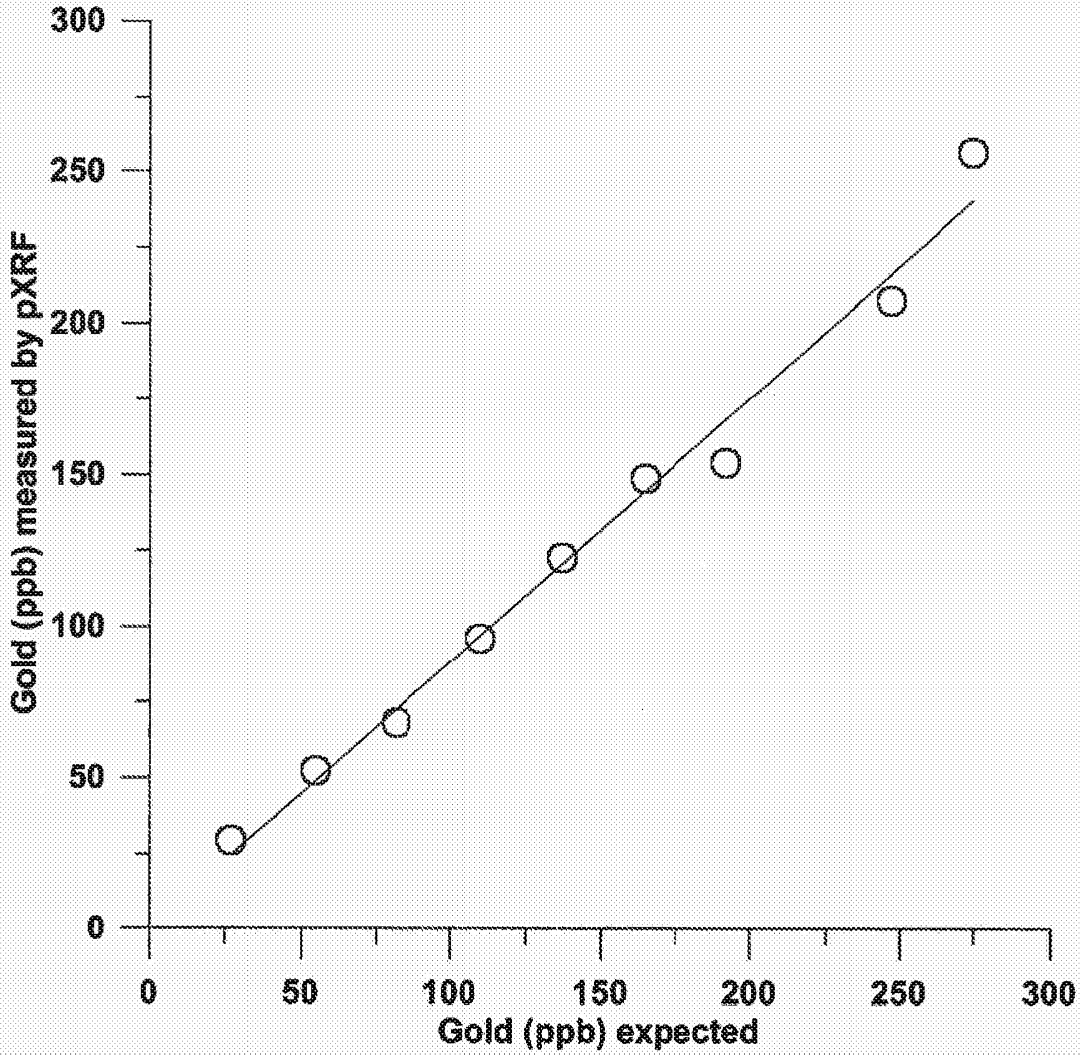
300
250
200
150
100
50
0
Gold (ppb) measured by pXRF
0
50
100
150
200
250
300
Gold (ppb) expected

FIG. 7

"Method and system for metal analysis of mineral samples"

Field

[0001] The present disclosure generally relates to a method and system for metal analysis, in particular to a method and system for metal analysis of mineral samples, for use in a laboratory, mineral processing plant or during a field mineral exploration sampling campaign. The present disclosure also relates to a digestion composition and a digestion tablet which may be used to prepare mineral samples for metal analysis according to the method and system described herein. The present disclosure further relates to a sample preparation device, method and system which may be used to prepare mineral samples for metal analysis according to the method and system described herein. The present disclosure also relates to a metal value collector device for collecting extracted metal values from a digestion mixture. The metal value collector device interfaces with a detector arranged to measure the amount of extracted metal value collected on the metal value collector device.

Background

[0002] Mineral exploration campaigns are frequently conducted in remote areas and involve collection of numerous geological samples including, but not limited to soil samples, drill cutting samples and stream sediment samples. Once collected, the samples are typically transported to a laboratory for analysis to determine the concentration of metal values in the sample. Many of the available analytical techniques involve acid extraction, fire assay or fusion techniques followed by spectrometric or spectrophotometric analysis for quantification of the metal values. These relatively sophisticated chemical methods are generally not suitable for remote field use and require high power consumption, clean conditions, stable temperatures and constant electrical output. A fire assay furnace, for example, commonly requires tens of kW of power and 460V.

[0003] As most laboratories capable of performing the necessary analysis are located in urban areas, it can take a period of several days, weeks or even months to have the samples analysed and data interpreted, leading to prolonged periods before the results of a mineral exploration campaign are known.

[0004] One of the more difficult metals to analyse in geological samples is gold. Electrochemical and colorimetry have been previously proposed as being suitable for analysing gold in the field. Both these techniques are time consuming and have not been adopted by the mineral exploration industry in recent times. They are not suitable for rapid routine analyses as they require sophisticated set ups in the form of a stand-alone chemical laboratory, skilled personnel to prepare the samples for analysis, and expert knowledge to perform and interpret the sample analyses.

[0005] Although portable X-ray fluorescence (pXRF) instruments are used in the field for mineral analysis, they are not generally used for gold analysis because a) the detection threshold for gold is high, b) the concentration of gold in collected samples is typically low (<1 ppm), and c) many samples include other metals or elements that provide spectral interferences which preclude the reliable detection of gold by this method.

[0006] Thus, there is a need to develop a simple on-site method for preparing and analysing samples containing very low concentrations of gold in the field. Furthermore, on site analysis is particularly advantageous to a mineral explorer as prompt access to analytical results permits near-real time decision making in respect to continuation or amendment of an exploration program.

Summary

[0007] The present disclosure provides a digestion composition and a digestion tablet which may be used to prepare a sample for metal analysis. The metal may be a precious metal, in particular gold or silver.

[0008] Certain embodiments of the disclosure provide a digestion composition comprising a metal lixiviant and an alkali compound.

[0009] Alternative embodiments of the disclosure provide a digestion tablet comprising a core portion containing a metal lixiviant, a layer of alkali compound encompassing the core portion, and an overcoat layer comprising a water soluble or water dispersible polymer.

[0010] Still other embodiments of the disclosure provide a digestion tablet comprising a core portion containing a mixture of a metal lixiviant and an alkali compound, and an overcoat layer comprising a water soluble or water dispersible polymer.

[0011] The digestion composition or the digestion tablet may comprise the metal lixiviant in an amount sufficient to extract a metal value from a digestion mixture comprising a sample containing low levels of the metal value, the digestion composition or the digestion tablet, and a digestion medium. The digestion medium may be a solvent, such as an aqueous solvent, in which the digestion composition or the digestion tablet is soluble. In some embodiments, the digestion mixture comprises less than 10000 ppm metal lixiviant. It will be appreciated to those skilled in the art that the rate of digestion will be normally faster for higher concentrations of metal lixiviant in the digestion mixture.

[0012] In one embodiment, the metal lixiviant in the digestion composition or the digestion tablet may be a cyanide compound. The cyanide compound may be a water soluble cyanide salt such as sodium cyanide or potassium cyanide.

[0013] In an alternative embodiment, the metal lixiviant in the digestion composition or the digestion table may be a halide compound, in particular a water soluble iodide such as potassium iodide.

[0014] In another embodiment, the metal lixiviant in the digestion composition or the digestion tablet may be a thiosulphate compound, in particular a water soluble thiosulphate salt, such as ammonium thiosulphate. Alternatively, the metal lixiviant in the digestion composition or the digestion tablet may be an iodide compound, in particular a water soluble iodide salt, such as ammonium iodide or sodium iodide.

[0015] In some embodiments the alkali compound may be an alkali metal hydroxide, an alkaline earth metal hydroxide, lime (CaO), soda (Na_2O), or a combination thereof.

[0016] One purpose of the alkali compound is to raise the pH of the digestion mixture to minimise the risk of forming hydrogen cyanide when the metal lixiviant is a cyanide compound. Accordingly, in one embodiment the alkali compound is present in an amount sufficient to maintain the pH of the digestion mixture above pH 10.

[0017] In some embodiments, the digestion composition or the digestion tablet may further comprise an ionic salt in an amount sufficient to maintain an ionic strength of the digestion mixture to at least 0.1M. Suitable examples of ionic salts for this purpose include, but are not limited to sodium chloride, potassium chloride and other inert water soluble salts.

[0018] In some embodiments, the digestion composition or the digestion tablet may further comprise an accelerant. The term 'accelerant' as used herein refers to one of more substances capable of increasing the rate of digestion of a metal value in a digestion medium. Suitable examples of accelerants include, but are not limited to, alkali or alkali earth metal peroxides and percarbonates or organic peroxides and percarbonates.

[0019] The present disclosure also provides a metal value collector device for collecting extracted metal value from a digestion mixture and interfacing with a detector arranged to measure the amount of extracted metal value collected on the metal value collector device.

[0020] Certain embodiments of the disclosure provide a metal value collector device capable of adsorbing a metal value thereon from a digestion mixture, thereby concentrating the extracted metal values from the digestion mixture to a concentration at or above a detectable threshold, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value. The detector may be an XRF detector, in particular a portable XRF (pXRF) detector.

[0021] In one embodiment the metal value collector device comprises a plate of adsorbent material. The plate may be shaped and sized for association with the detector. For example, the plate may be a 25 mm disc of adsorbent material for association with a window of the detector.

[0022] In one embodiment, the adsorbent material may be a textile material. In one form the textile material may be an activated carbon cloth.

[0023] In another embodiment, the metal value collector device comprises a bed of adsorbent material, such as activated carbon granules or anion exchange resin beads.

The bed may be shaped and sized for association with the detector. For example, the bed may be contained in a vessel having an interior space which defines the bed of adsorbent material. In this particular embodiment, the vessel may be configured to allow X-ray radiation to interact with the bed of adsorbent material.

[0024] In one embodiment the metal value collector device may further comprise a protective covering. The protective covering prevents particulate material in the sample from abrading adsorbed metal value from the plate of adsorbent material. In one form, the protective covering may be liquid permeable. For example, the protective covering may be a mesh bag containing the plate of adsorbent material.

[0025] Another embodiment of the disclosure provides a metal value collector device reference standard comprising a plate of adsorbent material having a known amount of metal value adsorbed thereon, wherein the plate is shaped and sized for association with a detector arranged to detect the known amount of metal value.

[0026] The present disclosure also provides a sample preparation device for preparing a sample containing low level metal value for metal value analysis by a detector.

[0027] The sample preparation device comprises:

a vessel for receiving a sample containing a metal value, a digestion composition or a digestion tablet, and a digestion medium;

a closure for the vessel to allow the vessel to be agitated and thereby produce a digestion mixture comprising extracted metal value and the digestion composition or the digestion tablet dissolved in the digestion medium; and,

a metal value collector device capable of adsorbing the extracted metal value thereon from the digestion mixture, thereby concentrating the extracted metal value, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the extracted metal value adsorbed on the metal value collector device.

[0028] In some embodiments, the closure and the metal value collector device may be coupled in a manner whereby the metal value collector device is retrieved from the vessel by removing the closure therefrom.

[0029] In one example, the closure and the metal value collector device are coupled by an elongate element. The elongate element may be flexible. For example, the elongate element may be a lanyard. Alternatively, the elongate element may be rigid. For example, the elongate element may be a permeable housing for the device.

[0030] The present disclosure also provides a system and a method for preparing a sample containing low level metal value for metal value analysis by a detector.

[0031] The system for preparing a sample containing low level metal value for metal value analysis by a detector comprises:

a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

a vessel for receiving the sample containing the extracted metal value, a digestion composition or a digestion tablet, and a digestion medium, the vessel having a closure for the vessel to allow the vessel to be agitated and thereby produce a digestion mixture comprising said extracted metal value and the digestion composition or the digestion table dissolved in the digestion medium; and,

a metal value collector device capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal values, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

[0032] The method for preparing a sample containing low level metal value for metal value analysis by a detector comprises:

mixing said sample, a digestion medium and a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in the digestion medium to produce a digestion mixture; and,

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal value, wherein the metal value collector device is shaped and sized for association with a detector arranged to measure the extracted metal value.

[0033] The present disclosure also provides a digestion composition as described above for use with a method or a system for preparing a sample containing low-level metal value for metal value analysis by a detector.

[0034] In another aspect, the present disclosure also provides a digestion tablet as described above for use with a method or a system for preparing a sample containing low-level metal value for metal value analysis by a detector.

[0035] The present disclosure also provides a system and method for metal value analysis of a sample containing low-level metal values

[0036] The system for metal value analysis of a sample containing low-level metal value comprises:

a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

a metal value collector device capable of adsorbing the extracted metal value from a digestion mixture comprising said extracted metal value and the digestion composition or the digestion table dissolved in the digestion medium, thereby concentrating the extracted metal value; and,

a detector arranged to detect the metal value adsorbed on the metal value collector device, wherein the metal value collector device is shaped and sized for association with the detector.

[0037] In one embodiment the system further comprises a control device for controlling the detector. The control device may comprise a memory for storing data and operation software, a controller that provides a user interface, and an external port for export of data.

[0038] The method of analysing a sample containing a metal value comprises the steps of:

providing a digestion mixture comprising said sample and a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value thereon, thereby concentrating the extracted metal values;

associating the metal value collector device having the extracted metal value adsorbed thereon with a detector; and,

measuring a concentration of adsorbed metal value on the metal value collector device with the detector.

[0039] The present disclosure also provides a digestion composition as described above for use with a method or a system of analysing a sample containing a metal value.

[0040] In another aspect, the present disclosure also provides a digestion tablet as described above for use with a method or a system of analysing a sample containing a metal value.

[0041] Other features, objects and advantages of the present disclosure and its embodiments will become apparent from the detailed description, examples and claims that follow.

Brief Description of Drawings

[0042] Preferred embodiments of the present invention will now be further described and illustrated, by way of example only, with reference to the accompanying drawings in which:

[0043] Figure 1 provides a schematic representation of a metal value collector device according to one embodiment described herein;

[0044] Figures 2a and 2b provide a schematic representation of a sample preparation device according to one embodiment described herein;

[0045] Figure 3 compares two respective XRF spectra for gold adsorbed on an activated carbon cloth in accordance with one embodiment as described herein; and a geological sample of As-rich gold ore containing about 30 ppm gold;

[0046] Figure 4 shows a calibration curve for gold (ppb) vs pXRF counts;

[0047] Figure 5 is a graphical representation showing a measure of error in XRF measurements of gold at low concentrations adsorbed on carbon granules (G) in comparison with gold adsorbed on activated carbon cloth;

[0048] Figure 6 is a graphical representation showing a measure of error in XRF measurements of gold at high concentrations adsorbed on carbon granules (G) in comparison with gold adsorbed on activated carbon cloth; and,

[0049] Figure 7 is a graphical representation of gold concentrations measured by XRF versus expected gold concentrations.

Description of Embodiments

[0050] The present invention is described in the following various non-limiting embodiments, which relate to systems and methods for preparation and analysis of samples containing low-level metal values. Various non-limiting embodiments relating to digestion compositions and digestion tablets which can be used in said systems and methods to extract a metal value from a sample containing low-level metal value are also described. Further, a metal value collector device which can be used in said systems and methods to concentrate the extracted metal values is also described.

[0051] GENERAL TERMS

[0052] Throughout this specification, unless specifically stated otherwise or the context requires otherwise, reference to a single step, composition of matter, group of steps or group of compositions of matter shall be taken to encompass one and a plurality (i.e. one or more) of those steps, compositions of matter, groups of steps or groups of compositions of matter. Thus, as used herein, the singular forms "a", "an" and "the" include plural aspects unless the context clearly dictates otherwise. For example, reference to "a" includes a single as well as two or more; reference to "an" includes a single as well as two or more; reference to "the" includes a single as well as two or more and so forth.

[0053] Each example of the present disclosure described herein is to be applied *mutatis mutandis* to each and every other example unless specifically stated otherwise. The present disclosure is not to be limited in scope by the specific examples described herein, which are intended for the purpose of exemplification only. Functionally-equivalent products, compositions and methods are clearly within the scope of the disclosure as described herein.

[0054] The term "and/or", e.g., "X and/or Y" shall be understood to mean either "X and Y" or "X or Y" and shall be taken to provide explicit support for both meanings or for either meaning.

[0055] Throughout this specification the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

[0056] It will be clearly understood that, although a number of prior art references are made herein, this reference does not constitute an admission that any of this information forms part of the common general knowledge in the art, in Australia or in any other country.

[0057] Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Although methods and materials similar or equivalent to those described herein can be used in the practice or testing of the present invention, suitable methods and materials are described below. In case of conflict, the present specification, including definitions, will control. In addition, the materials, methods, and examples are illustrative only and not intended to be limiting.

SPECIFIC TERMS

[0058] The term "low-level metal analysis" as used herein refers to detection, identification and/or measurement of a concentration of a metal value in a sample containing the metal value, wherein the concentration of the metal value in the sample may be from 5 ppb to 10 ppm. In the embodiments described herein, the detection, identification and/or measurement of the concentration of the metal value in the sample may be performed with an X-ray fluorescence (XRF) spectrometer, in particular a portable XRF (pXRF).

[0059] The term "sample" refers to a geological sample. The sample may include, but is not limited to, an ore, mineral, concentrate, tailings, soil, drill cutting, stream sediment or alluvial sediments. Samples may be collected from one or more sites in accordance with field sampling for a mineral exploration campaign, as will be known to those skilled in the art. The field sampling or mineral exploration campaign will determine the sample size, size fraction, site selection, sample density, sample interval and so forth. The field sampling campaign may be modified in response to results obtained by the methods described herein; this may be called 'interactive sampling'.

[0060] A minimum sample size may be a function of the metal value abundance and the particle-size distribution. Larger samples are generally required for coarse metal value particles than for fine metal value particles, in particular when the metal value is gold. For large particles, for example, only one sample in ten equal samples may be expected to contain gold and that fraction may report a value of ten times the average value of the sample. An equal weight of fine-grained gold would more likely report in each fraction at about a common concentration. For the purposes of the embodiments described herein, the sample may be about 250-500 g. Advantageously, preparation and analysis of the sample in this weight range in accordance with the methods described herein increases the analytical precision in samples containing coarse gold particles.

[0061] The term "metal lixiviant" as used herein refers to one or more chemical compounds capable of selectively extracting a desired metal value from a sample into a liquid medium. The extracted metal value may then be recovered from the liquid medium in a concentrated form. The metal lixiviant may selectively extract the metal value by altering the redox state of the sample or the pH of the liquid medium.

[0062] The term "digestion medium" as used herein refers to a liquid medium capable of dissolving the metal lixiviant, one or more compounds other than the metal lixiviant for adjusting and/or maintaining the liquid medium under a desired condition (e.g. pH, ionic strength, reduction potential (Eh), dissolved oxygen and so forth), and the metal value extracted from the sample.

[0063] The digestion medium may be an aqueous solvent, such as water. The water may be rainwater, deionised water, distilled water, ultrapure water, municipal water, groundwater, produced water, process water, brackish water, or brine.

DIGESTION COMPOSITION

[0064] The present disclosure provides a digestion composition which may be used to prepare a sample for low-level metal analysis, in particular for low-level gold analysis, in accordance with methods described herein.

[0065] The digestion composition comprises a metal lixiviant and an alkali compound.

[0066] The metal lixiviant may be a cyanide compound. The cyanide compound may be a water soluble cyanide salt such as sodium cyanide or potassium cyanide.

[0067] Alternatively, the metal may be a thiosulphate compound in particular a water soluble thiosulphate salt, such as ammonium thiosulphate. Alternatively, the metal lixiviant may be an iodide compound, in particular a water soluble iodide salt, such as ammonium iodide or sodium iodide.

[0068] The alkali compound may be an alkali metal hydroxide, an alkaline earth metal hydroxide, lime (CaO), soda (Na_2O), or a combination of two or more.

[0069] In use, a fixed amount of the digestion composition and the sample are mixed with a digestion medium, such as water, to produce a digestion mixture. The metal lixiviant is present in the digestion composition in an amount sufficient to leach the metal value in the sample into the digestion mixture. In one embodiment, the metal lixiviant is present in the digestion composition in an amount sufficient to provide a concentration in the digestion mixture of between 1 ppm and 10000 ppm It will be appreciated that a high cyanide concentration in the digestion mixture may require extended treatment to neutralise the cyanide for disposal.

[0070] The digestion composition may also comprise an accelerant selected from a group comprising: an alkali or alkali earth metal peroxide, an alkali or alkali earth metal percarbonate, an organic peroxide, or an organic percarbonate.

[0071] In some embodiments, the digestion composition may further comprise a pH indicator compound, for example a compound that is a distinct colour below or above a specific pH. The pH indicator compound may be included in the digestion composition to indicate if the digestion medium or digestion mixture falls below a pH at which hydrogen cyanide gas forms.

[0072] For ease of use, the fixed amount of the digestion composition may be pre-measured and packaged into a sachet. The sachet may then be opened and the fixed amount of the digestion composition may be readily dispensed into a sample preparation vessel for preparing a sample for analysis as described herein.

[0073] In an alternative embodiment, the fixed amount of the digestion composition may be pre-measured and packaged into a frangible pouch or capsule associated with a closure of a sample preparation vessel, wherein the closure is configured to break or tear the frangible pouch or capsule when the closure closes the sample preparation vessel, thereby releasing the digestion composition into said vessel.

[0074] In a further alternative embodiment, the fixed amount of the digestion composition may be pre-measured and packaged in the sample preparation vessel.

[0075] In acidic environments, cyanide salts may generate toxic hydrogen cyanide (HCN). Some samples may have a high sulphide content and/or a low buffering carbonate content. Such samples, when mixed with the digestion medium may lower the pH of the digestion mixture thereby providing conditions under which hydrogen cyanide may be generated.

[0076] Accordingly, the alkali compound is present in the digestion composition in an amount sufficient to maintain the pH of the digestion mixture above 10.

[0077] The digestion composition may further comprise one or more compounds other than the metal lixiviant and the alkali compound for adjusting and/or maintaining the liquid medium under a desired condition (e.g. pH, ionic strength, oxygen concentration, reduction potential (Eh) and so forth). For example, the digestion composition may further comprise an ionic salt in an amount sufficient to maintain an ionic strength of the digestion mixture to at least 0.1M. Suitable examples of ionic salts for this purpose include, but are not limited to sodium chloride, etc.

DIGESTION TABLET

[0078] The fixed amount of the digestion composition may be conveniently provided in the form of a digestion tablet which may be used to prepare a sample for low-level metal analysis, in particular for low-level gold analysis, in accordance with methods described herein.

[0079] In one embodiment, the digestion tablet may comprise a core portion of the digestion composition as described above and an overcoat layer comprising a water soluble or water dispersible polymer.

[0080] In an alternative embodiment, the digestion tablet may comprise a core portion containing the metal lixiviant, a layer of alkali compound encompassing the core portion, and an overcoat layer comprising a water soluble or water dispersible polymer. The layer of alkali compound may also comprise an accelerant and/or a pH indicator compound.

[0081] The overcoat layer allows the digestion tablet to be safely handled because it reduces the risk of contact contamination of personnel with the metal lixiviant, in particular in embodiments where the metal lixiviant is a cyanide salt such as sodium cyanide or potassium cyanide.

[0082] Many water-soluble or water-dispersible polymers are known in the prior art that may serve the present purposes. By way of illustrating the polymers of this character that may be employed herein, mention may be made of the following: hydroxypropylmethylcellulose, polyvinyl pyrrolidone, hydroxypropyl cellulose, polyethylene glycol 3350, 4500, 8000, methyl cellulose, pseudo ethylcellulose. One such polymer that has been found to be very suitable is hydroxypropyl methylcellulose which is a propylene glycol ether of methylcellulose. This is available from several manufacturers as for example from Dow under the trade names HPM Cellulose.

[0083] Aside from the water-soluble or water-dispersible polymers, the overcoat layer may also contain other ingredients which may aid in the application of the coating to the tablet or to improve the character of the coating. These may be such ingredients as surfactants, plasticisers, antifoaming agents, solubilising agents, colouring agents.

[0084] The digestion tablet as described herein can be prepared by any known techniques like direct compression, wet granulation, dry granulation, spray drying and solid dispersion.

[0085] The overcoat layer as described herein can be prepared by any known techniques like dip coating, compression coating, film coating including organic film coating and vacuum film coating.

[0086] In an alternative embodiment, the digestion tablet may take the form of a capsule, wherein the digestion composition as described above may be encapsulated in a water soluble or water dispersible polymeric capsule, such as a gelatin capsule.

METAL VALUE COLLECTOR DEVICE

[0087] The present disclosure also provides a metal value collector device for collecting extracted metal value from a digestion mixture and interfacing with a detector arranged to measure the amount of extracted metal value collected on the metal value collector device. The detector may be an XRF detector, in particular a pXRF detector.

[0088] The metal value collector device is capable of adsorbing an extracted metal value thereon from a digestion mixture, thereby concentrating the extracted metal values from the digestion mixture. The concentration of metal value in the sample may be low-level (e.g. 5 ppb to 1 ppm), in which case it is not possible to directly measure the concentration of gold in the sample with XRF techniques because the concentration of gold in the sample is below a detectable threshold. It will be appreciated that the 'detectable' threshold may vary between individual XRF detectors and samples but will typically be from 1 ppm to 10 ppm depending on the matrix of the sample. Advantageously, adsorbing the extracted metal values from the digestion mixture concentrates the extracted metal value to a concentration at or above the detectable threshold for the metal value.

[0089] The term "detectable threshold" as used herein refers to the lowest concentration of a specific metal value (e.g. gold) that can be consistently quantified by a detector (e.g. pXRF) within a desired error margin. It will be appreciated that the detectable threshold will vary according to the specific metal value, the type of detector, the substrate on which the specific metal value is adsorbed or the solvent in which the specific metal value is dissolved, the nature and concentration of contaminants, the nature and concentration of elements that provide spectral interferences(e.g. arsenic, tungsten, and zinc in the case of gold), and so forth. The detectable threshold of the specific metal value for the detector may be determined by techniques known to those skilled in the art

[0090] In one embodiment, wherein the metal value is gold and the detector is a pXRF, the detectable threshold for the pXRF may be from 1 ppm to 10 ppm gold.

[0091] The metal value collector device may be shaped and sized for association with a detector arranged to detect the extracted metal value collected on the metal value collector device. In particular, the metal value collector device may be shaped and sized to conform to an analysis window of the detector.

[0092] The metal value collector device may comprise an adsorbent material, in particular a textile material. The textile material may be a randomly or non-randomly arranged fibrous mesh, woven, braided, or felt-like textile material. In one form the textile material may be an activated carbon cloth. Activated carbon cloth is particularly effective at adsorbing gold cyanide.

[0093] Tungsten and/or arsenic are typically associated with samples containing gold, generally in much higher concentration than gold. Tungsten and arsenic are also elements that cause spectral interferences for analysis of gold by XRF techniques. Accordingly, the presence of tungsten and/or arsenic in a sample containing gold make direct gold analysis by XRF of limited use. Therefore, XRF analysis of low-level gold samples containing tungsten and/or arsenic is not practical. Advantageously, tungsten and arsenic in the sample are poorly dissolved in the digestion mixture and furthermore are only adsorbed onto the activated carbon cloth at very low (i.e. non-interfering) concentrations.

[0094] The metal value collector device may further comprise a protective covering. The protective covering minimises (1) particulate material in the digestion mixture from abrading adsorbed metal value from the plate of adsorbent material and (2) particulate material from clogging the pores of the adsorbent material. In one embodiment, the protective covering may be < 40 µm mesh. For example, the protective covering may be a sealed mesh bag containing the plate of adsorbent material. It will be appreciated that the metal value collector device may be retrievable from the protective covering prior to association with the detector. For example, the protective covering may be temporarily sealed around the metal value collector device and then cut or torn open after the metal value collector has been contacted with the digestion mixture to release the plate, prior to analysis by the detector.

[0095] It will be appreciated that the protective covering may be fabricated from a material which is inert to the digestion composition and non-adsorbent to the metal value of interest. In one embodiment, the protective covering may be < 40 µm mesh.

[0096] The metal value collector device may be fabricated by any suitable technique. A consistency in the shape and size of the metal value collector device is desirable as it enhances overall precision and accuracy of the measurement of extracted metal values from a plurality of samples by the detector.

[0097] One or more metal value collector device reference standards may also be fabricated and used in association with the methods and systems described herein. The metal value collector device reference standard may comprise a plate of adsorbent material having a known amount of metal value adsorbed thereon, wherein the plate is shaped and sized for association with a detector arranged to detect the known amount of metal value. It will be appreciated by those skilled in the art that the metal value collector device reference standard must be fabricated from an identical adsorbent material, and be shaped and sized in an identical manner, to the metal value collector device for which it is intended to be employed as a reference standard.

[0098] In use, a plurality of metal value collector device reference standards, each having a different known amount of metal value adsorbed thereon, may be used to establish a calibration curve to allow determination of a concentration of metal value in a sample after analysis by the methods and systems described herein. The calibration curve may be established by suitable analytical techniques corresponding to the detector and the specific metal value, as will be understood by those skilled in the art.

[0099] Referring to Figure 1,a metal value collector device (10) comprising a disc (12) of activated carbon cloth is shown. The disc (12) is circular-shaped and sized so as to conform to the shape and size of an analysis window (not shown) of a detector, such as a pXRF detector.

[0100] In an alternative embodiment, the metal value collector device may comprise a bed of adsorbent material, such as activated carbon granules or anion exchange resin beads. The bed may be shaped and sized for association with the detector. For example, the bed may be contained in a vessel having an interior space which defines the bed of adsorbent material.

[0101] In this particular embodiment, the vessel may be configured to allow X-ray radiation to interact with the bed of adsorbent material. For example, the vessel may be provided with a base wall or a closure that is at least near-transparent to X-ray radiation. The vessel containing the bed of adsorbent material may be positioned with respect to the analysis window of the pXRF so that the transparent base wall or closure abuts the analysis window of the pXRF.

[0102] In use, a fixed amount of the activated carbon granules or the anion exchange resin beads may be contained within a mesh bag. After adsorbing the gold thereon, the contents of the mesh bag may be washed and a bed of said activated carbon granules or anion exchange resin beads may be laid flat on the analysis window of the pXRF.

[0103] Alternatively, the activated carbon granules or anion exchange resin beads may be poured into a vessel having an interior space which defines the bed of adsorbent material. In this particular embodiment, the vessel may be configured to allow X-ray radiation to interact with the bed of adsorbent material formed therein. For example, the vessel may have a base fabricated from a material that is near-transparent to X-ray radiation. The base of the vessel may be placed on the analysis window of the pXRF to facilitate measurement of the extracted metal value adsorbed on the bed of activated carbon granules or anion exchange resin beads.

SAMPLE PREPARATION DEVICE

[0104] The present disclosure also provides a sample preparation device for preparing a sample containing low-level metal value for metal value analysis by a detector.

[0105] Referring to Figure 2, the sample preparation device (14) may include a vessel (16) for receiving a sample containing a metal value, a fixed amount of a digestion composition or a digestion tablet, and a digestion medium. Preferably, the vessel (16) is fabricated from a material that is inert to reaction with the sample, digestion composition or digestion tablet and the digestion medium. Suitable materials may include, but are not limited to, glass or plastics materials commonly used in analytical laboratories, as will be well known to those skilled in the art. Generally, these materials will be rigid.

[0106] In alternative embodiments (not shown), the vessel may be fabricated from a flexible material, such as a plastics material used for the production of food or beverage storage pouches or bags. Such 'bladder-like" vessels may be convenient in a remote location, as they can be stored and transported flat and occupy less space than conventional rigid plastic bottles.

[0107] The vessel (16) is provided with a closure (18) to allow the vessel (16) to be closed and its contents to be agitated without spillage. The vessel (16) and the closure (18) may be complementary to one another. For example, a neck (20) of the vessel (16) and the closure (18) may be provided with complementary threading so that the closure (18) may be screwed on and off the neck (20) of the vessel (16). In alternative embodiments (not shown), the closure may be configured to be either snap-fitted over the neck of the vessel or bunged into the neck of the vessel. Other suitable complementary arrangements will be apparent to the skilled person in the art.

[0108] The sample preparation device (14) also includes the metal value collector device (10) as described previously. It will be appreciated that the neck (20) of the vessel (16) will be shaped and sized to allow the metal value collector device (10) to be readily inserted and retrieved from the vessel (16).

[0109] The closure (18) and the metal value collector device (10) may be coupled in a manner whereby the metal value collector device (10) may be conveniently retrieved from the vessel (16) by removing the closure (18) therefrom. For example, the closure (18) and the metal value collector device (10) may be coupled by an elongate element (22). The elongate element (22) may be fabricated from a material that is inert to reaction with the sample, digestion composition or digestion tablet and the digestion medium.

[0110] The elongate element (22) may be flexible. For example, the elongate element (22) may be a plastic cable, wire or thread fabricated from nylon or polyethylene or any other plastics material that is commonly used in analytical laboratories.

[0111] In this particular embodiment, the metal value collector device is housed in a protective covering (24), such as a sealed mesh bag as previously described.

[0112] In alternative embodiments (not shown), the elongate element may be rigid. For example, the elongate element may be provided with a permeable housing for the device (10).

[0113] In other embodiments, the closure and a protective covering of the metal value collector device may be coupled in a manner whereby the metal value collector device and the protective covering may be conveniently retrieved from the vessel by removing the closure therefrom.

[0114] In still further embodiments, the protective covering containing the metal value collector device may not be coupled to the closure by the elongate element. In these particular embodiments, the metal value collector device and the protective covering may be retrieved from the vessel by pouring out the contents of the vessel.

METHOD AND SYSTEM FOR PREPARING A SAMPLE

[0115] The present disclosure also provides a method and a system for preparing a sample containing low-level metal value for metal value analysis by a detector.

[0116] The method for preparing a sample containing low-level metal value for metal value analysis by a detector comprises:

mixing said sample, an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium to produce a digestion mixture; and,

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal values, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

[0117] In some embodiments, the extracted metal values in the digestion mixture are concentrated by adsorption on the metal value collector device to a concentration at or above a detectable threshold of a pXRF detector.

[0118] The samples that are prepared by the methods as described herein may be 'as-received' or may undergo comminution by one or more crushing, grinding and milling processes prior to being prepared for analysis. The sample may undergo classification to select a fraction having a particle size < 250 microns, or even a fraction having a particle size < 75 micron.

[0119] It will be appreciated that a desired particle size may be selected with regard to the nature and type of sample. Even though a particular size fraction of the sample may have the highest gold concentrations, it may well be that another particle size fraction of the sample generates more consistent and meaningful data with the analysis methods described herein. The desired particle size for a sample may be determined by experimental techniques by "interactive sampling" as will be well understood by those skilled in the art.

[0120] In one embodiment, the alkali compound and the metal lixiviant may be provided in a fixed amount of the digestion composition as previously described.

[0121] In another embodiment, the alkali compound and the metal lixiviant may be provided as a digestion tablet as previously described.

[0122] In one embodiment, the metal lixiviant is present in an amount sufficient to provide a concentration in the digestion mixture of from 1ppm, to 10000 ppm. The amount of alkali compound is sufficient to maintain pH > 10 in the digestion mixture.

[0123] The metal value collector device may be as previously described. In some embodiments, the metal value collector device may be pretreated to optimise the surface for adsorbing metal value. For example, the metal value collector device may be contacted with dilute (e.g. 0.1 M) nitric acid for three hours then washed in deionised water.

[0124] The mixing step and the contacting step may be performed simultaneously.

[0125] The mixing step and the contacting step may be performed over a period of about 12-24 hours. It will be appreciated that the period may be shortened by increasing the temperature of the digestion mixture above ambient temperature or by increasing the concentration of metal lixiviant in the digestion mixture. In locations with ambient temperature < 10 °C, it may be necessary in practice to take suitable measures to ensure that the temperature of the digestion mixture is maintained at a temperature ≥15 °C.

[0126] The mixing step and the contacting step may be performed by agitating the metal value collector device, the sample, the alkali compound, the metal lixiviant and the digestion medium in a container, such as a vessel. The vessel may be agitated to mix its contents. The vessel may be closed so that the vessel may be agitated without spillage of its contents. The vessel may be agitated by any one of several conventional techniques such as rolling, shaking, spinning. Alternatively, the contents of the container may be agitated by mechanically stirring the contents.

[0127] After completion of the mixing and contacting steps, the metal value collector device with the adsorbed metal value thereon (and, optionally the protective covering) may be retrieved from the container, rinsed with water to remove any residual sample particulate materials, then air-dried.

[0128] In embodiments where the metal lixiviant is a cyanide salt, the digestion mixture remaining in the container must be neutralised prior to disposal to reduce the potential hazard for generation of toxic hydrogen cyanide gas. A stoichiometric excess of an oxidising agent agent such as chlorine dioxide (e.g. in the form of swimming pool tablets or household liquid bleach) or potassium permanganate ($KMnO_4$) (Condy's crystals) may be used to oxidise the cyanide into less toxic reaction products (e.g. manganese dioxide, nitrogen gas, carbon dioxide and potassium hydroxide). The neutralised solution may then be disposed without harm to the environment or personnel.

[0129] Various embodiments of the above method may be performed by using a system for preparing a sample containing low-level metal value for metal value analysis by a detector, such as an XRF, in particular a pXRF detector.

[0130] Said system comprises a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium. The digestion composition as previously described may be provided in a fixed amount (or multiples thereof).

[0131] The system may also comprise a vessel for receiving the sample containing the metal value, the digestion composition or the digestion tablet, and the digestion medium. In one embodiment, the vessel has a closure to allow the vessel to be agitated without spillage of its contents and thereby produce a digestion mixture comprising said sample and the digestion composition or the digestion tablet dissolved in the digestion medium.

[0132] The vessel and the closure may be as previously described.

[0133] The system may also comprise a metal value collector device. The metal value collector device is capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal values. The metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

[0134] In one embodiment, the metal value collector device may be as previously described. The metal value collector device may be coupled to the closure by an elongate element as previously described to assist retrieval of the metal value collector device after sample preparation.

METHOD AND SYSTEM FOR METAL VALUE ANALYSIS

[0135] The present disclosure also provides a method and system for metal value analysis of a sample containing low-level metal values.

[0136] The method of analysing a sample containing a metal value at low-levels comprises the steps of:

providing a digestion mixture comprising said sample and a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value thereon, thereby concentrating the extracted metal values;

associating the metal value collector device having the extracted metal value adsorbed thereon with a detector; and,

measuring a concentration of adsorbed metal value on the metal value collector device with the detector.

[0137] The digestion mixture may be prepared as previously described. Similarly, the step of contacting the digestion mixture with a metal value collector device may be performed as previously described.

[0138] Associating the metal value collector device with the detector comprises placing the metal value collector device on the analysis window of the detector. It will be appreciated from the foregoing discussion that the metal value collector device is shaped and sized to conform to the detection window of the detector.

[0139] Measuring the concentration of adsorbed metal value on the metal value collector device comprises operating the detector according to the manufacturer's instructions.

[0140] The system for metal value analysis of a sample containing low-level metal value comprises:

a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

a metal value collector device capable of adsorbing the metal value from a digestion mixture comprising said sample and the digestion composition or the digestion table dissolved in the digestion medium, thereby concentrating the metal values; and,

a detector arranged to detect the metal value adsorbed on the metal value collector device, wherein the metal value collector device is shaped and sized for association with the detector.

[0141] In one embodiment, the detector is an XRF, preferably a pXRF detector. An example of a commercially available pXRF detector that is suitable for performing the methods as described herein include, but are not limited to, the pXRF detector manufactured by Olympus (e.g. Olympus Innovx Delta Premium DP-6000-C).

[0142] In one embodiment the system further comprises a control device for controlling the detector. The control device may comprise a memory for storing data, interpreting concentrations and operation software, a controller that provides a user interface, and an external port for export of data.

[0143] A touch responsive display (not shown) may be provided to present information to an operator (for instance data representing the concentration of gold adsorbed on the metal value collector device) and to accept operator input. The XRF analyzer will typically include a wired (for instance a USB) or a wireless communications port to enable the uploading and downloading of data, software, and other information to and from an external/remote computer.It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the above-described embodiments, without departing from the broad general scope of the present disclosure. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.

Example

[0144] The invention is further illustrated by the following example. The example is provided for illustrative purposes only. It is not to be construed as limiting the scope or content of the invention in any way.

[0145] Geological samples from a selection of sites were collected and brought back to a field base camp. The samples were sieved and 250-500 g of each sample having a particle size less than 250 micron was separated for sample preparation and analysis by pXRF.

[0146] The sample (250 g) and water (0.7L) were placed in a plastic bottle together with a digestion tablet as described previously A disc (25 mm diameter) of activated carbon cloth is also placed in the plastic bottle. One end of a length of plastic cable thread is attached to the protective mesh covering which contains the disc and the opposing end of the length of plastic cable thread is attached to a screw cap closure which threadably mates with the plastic bottle.

[0147] The screw cap closure is threaded onto the plastic bottle and then the plastic bottle is gently agitated using a powered rolling device for 24 hours at ambient temperature to mix the digestion tablet, the sample with the water thereby producing a digestion mixture, and to contact the digestion mixture with the disc of activated carbon cloth to adsorb the extracted gold therefrom.

[0148] After 24 hours, the plastic bottle and its contents are removed from the rolling device and the screw cap closure is removed from the plastic bottle to retrieve the disc of activated carbon cloth with adsorbed extracted gold.

[0149] The disc may be rinsed with water to remove any loose particulate material (e.g. particles of rock, clay, sand or soil), then air-dried and placed on the analysis window of the pXRF.

[0150] An XRF analysis for gold (and optionally other metals) is performed and the concentration of gold adsorbed on the disc of activated carbon cloth is reported. It will be appreciated that the measured concentration of gold adsorbed on the disc of activated carbon cloth is not an absolute measurement of the concentration of gold in the geological sample but rather it is directly proportional to the concentration of gold in the geological sample and the size of the disc.

[0151] After completion of the sample preparation as described above, the free cyanide remaining in the digestion mixture in the plastic bottle may be oxidised to remove a potential hazard of generating toxic hydrogen cyanide gas. A stoichiometric excess of an oxidising agent such as chlorine dioxide (e.g. in the form of swimming pool tablets or household liquid bleach) or potassium permanganate (Condy's crystals) oxidises the cyanide into less toxic reaction products (e.g. manganese dioxide, nitrogen gas, carbon dioxide and potassium hydroxide). The resulting solution may then be disposed without harm to the environment or personnel.

[0152] Figures 3a and 3b shows respective XRF spectra for gold adsorbed on the metal value collector device as described herein and an As-rich geological sample containing about 30 ppm gold. The spectrum shown in Figure 3a is a spectrum (5 – 15 keV) of a carbon cloth 1000 standard containing gold extracted from 1L of a 1 ppm solution (equivalent to 4 ppm Au in a 250 g sample), using a 40 kV, 4W Rh-tube, Delta Innov-X Olympus pXRF unit, location of relevant energy peaks as annotated. Note the close correspondence between the location of the Zn, W and gold peaks at ~9.6 keV, and the gold and As peaks at ~11.5 keV. There is no As or W adsorbed on the metal value collector device. Gold can be measured on its L-beta peak at 11.44 keV, but not its L-alpha peak at 9.71 keV due to the presence of Zn.

[0153] With respect to Figure 3b, the As peak at ~11.7 keV interferes with the gold peak at ~11.4 keV; for this sample, gold may be measured on its L-alpha peak at 9.71 keV but this has spectral interferences from W and Zn which are typically present in gold-bearing samples. Note that the deconvolution algorithms in this pXRF unit relies on region-of-interest methods to deconvolve the spectra and are not capable of switching peaks/regions to bypass potentially interfering elements present in the sample.

[0154] Figure 4 shows a calibration curve of concentration of gold (ppb) v pXRF counts from which gold concentrations in a sample may be calculated.

[0155] Figures 5 and 6, show the margins of error in measurement of gold adsorbed on carbon granules (G) and activated carbon cloth (CC) at low and high gold concentrations, respectively. The size of the error measured at low gold concentrations is consistently lower when gold is adsorbed on activated carbon cloth rather than carbon granules, whereas the error for high gold concentrations is similar regardless of whether gold is adsorbed on activated carbon cloth or carbon granules.

[0156] Figure 7 shows a graphical representation of XRF concentrations for gold adsorbed on carbon from a sample that has been digested by the methods described herein compared with gold expected in the sample based on independent laboratory measurements and results. Comparisons between expected data and actual data produced $R^2 = 0.9093$. Aliquots of in-house standards were diluted with barren quartz to a set weight of 250 g to produce a regression coefficient for expected vs actual data of $R^2 = 0.9093$ (Figure 7).

CLAIMS:

1. A metal value collector device capable of adsorbing a metal value thereon from a digestion mixture, thereby concentrating the metal value from the digestion mixture, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

2. A metal value collector device reference standard comprising a plate of adsorbent material having a known amount of metal value adsorbed thereon, wherein the plate is shaped and sized for association with a detector arranged to detect the known amount of metal value.

3. The metal value collector device of claim 1 comprising a plate of adsorbent material.

4. The metal value collector device of claim 3, wherein the plate is shaped and sized for association with an analysis window of the detector.

5. The metal value collector device of any one of claims 1 to 4, wherein the adsorbent material is a textile material.

6. The metal value collector device of claim 5, wherein the textile material is an activated carbon cloth.

7. The metal value collector device of any one of claims 1 or 3 to 6, further comprising a protective covering.

8. The metal value collector device of claim 7, wherein the protective covering comprises a mesh bag.

9. The metal value collector device of claim 1 comprising a bed of adsorbent material.

10. The metal value collector device of claim 9, wherein the bed of adsorbent material is defined by an interior space of a vessel containing the adsorbent material.

11. The metal value collector device of claim 9 or claim 10 comprising activated carbon granules or ion exchange resin beads.

12. A sample preparation device for preparing a sample containing low-level metal value for metal value analysis by a detector, wherein said device comprises:

a vessel for receiving a sample containing a metal value, a digestion composition or a digestion tablet, and a digestion medium;

a closure for the vessel to allow the vessel to be agitated and thereby produce a digestion mixture comprising extracted metal value and the digestion composition or the digestion tablet dissolved in the digestion medium; and,

a metal value collector device capable of adsorbing the extracted metal value thereon from the digestion mixture, thereby concentrating the extracted metal value, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the extracted metal value adsorbed on the metal value collector device, wherein the closure and the metal value collector device are coupled in a manner whereby the metal value collector device is retrieved from the vessel by removing the closure therefrom.

13. The device of claim 12 wherein the closure and the metal value collector device are coupled by an elongate element.

14. The device of claim 11, wherein the elongate element is flexible.

15. The device of claim 11 wherein the elongate element is rigid.

16. The device of claim 15, wherein the elongate element is a permeable housing for the metal value collector device.

17. The device of any one of claims 13 to 16, wherein the closure is coupled to a protective covering containing the metal value collector device by the elongate element.

18. A system for preparing a sample containing low-level metal value for metal value analysis by a detector comprising:

a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

a vessel for receiving the sample containing the metal value, a digestion composition or a digestion tablet, and a digestion medium, the vessel having a closure for the vessel to allow the vessel to be agitated and thereby produce a digestion mixture comprising extracted metal value and the digestion composition or the digestion tablet dissolved in the digestion medium; and,

a metal value collector device capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal values, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

19. A method for preparing a sample containing low-level metal value for metal value analysis by a detector comprising:

mixing said sample, a digestion medium and a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in the digestion medium to produce a digestion mixture; and,

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value from the digestion mixture, thereby concentrating the extracted metal value, wherein the metal value collector device is shaped and sized for association with a detector arranged to detect the metal value.

20. A system for metal value analysis of a sample containing low-level metal value comprising:

a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in a digestion medium;

a metal value collector device capable of adsorbing extracted metal value from a digestion mixture comprising the digestion medium, said sample and the digestion composition or the digestion table dissolved in the digestion medium, thereby concentrating the extracted metal value; and,

a detector arranged to detect the extracted metal value adsorbed on the metal value collector device, wherein the metal value collector device is shaped and sized for association with the detector.

21. The system of claim 20 further comprising a control device for controlling the detector.

22. The system of claim 21, wherein the control device comprises a memory for storing data and operation software, a controller that provides a user interface, and an external port for export of data.

23. The system of any one of claims 20 to 22, wherein the detector is an X-ray fluorescence (XRF) detector.

24. A method of analysing a sample containing a metal value at low-levels comprising the steps of:

providing a digestion mixture comprising said sample, a digestion medium and a digestion composition or a digestion tablet containing an alkali compound and a metal lixiviant in an amount capable of extracting the metal value from said sample in the digestion medium;

contacting the digestion mixture with a metal value collector device capable of adsorbing the extracted metal value thereon, thereby concentrating the extracted metal value;

associating the metal value collector device having the extracted metal value adsorbed thereon with a detector; and,

measuring a concentration of adsorbed metal value on the metal value collector device with the detector.

25. A digestion composition comprising a metal lixiviant and an alkali compound.

26. The digestion composition according to claim 25 further comprising an accelerant and/or a pH indicator.

27. A digestion tablet comprising a core portion containing a metal lixiviant, a layer of alkali compound encompassing the core portion, and an overcoat layer comprising a water soluble or water dispersible polymer.

28. A digestion tablet comprising a core portion containing a mixture of a metal lixiviant and an alkali compound, and an overcoat layer comprising a water soluble or water dispersible polymer.

29. The composition of claim 25 or claim 26, or the digestion tablet of claims 27 or 28, wherein the metal lixiviant is in an amount sufficient to extract a metal value from a digestion mixture comprising a sample containing low-levels of the metal value, the digestion composition or the digestion tablet, and a digestion medium.

30. The composition of any one of claims 25, 24 or 29 or the digestion tablet of any one of claims 27 to 29, wherein the digestion medium is a solvent in which the digestion composition or the digestion tablet is soluble.

31. The composition of claim 30 or the digestion tablet of claim 30, wherein the solvent is an aqueous solvent.

32. The composition of any one of claims 25, 26, 29 to 31 or the digestion tablet of any one of claims 27 to 31, wherein the digestion mixture comprises from 1ppm to 10000 ppm metal lixiviant.

33. The composition of any one of claims 25, 26, 29 to 32 or the digestion tablet of any one of claims 27 to 32, wherein the metal lixiviant is a water soluble cyanide compound.

34. The composition of any one of claims 25, 26, 29 to 32 or the digestion tablet of any one of claims 27 to 32, wherein the metal lixiviant is a water soluble thiosulphate compound.

35. The composition of any one of claims 25, 26, 29 to 32 or the digestion tablet of any one of claims 27 to 32, wherein the metal lixiviant is a water soluble iodide compound.

36. The composition of any one of claims 25, 26, 29 to 35 or the digestion tablet of any one of claims 27 to 35, further comprising an accelerant selected from a group comprising: an alkali or alkali earth metal peroxide, an alkali or alkali earth metal percarbonate, an organic peroxide or an organic percarbonate..

37. The composition of any one of claims 22, 23, 24 to 36 or the digestion tablet of any one of claims 24 to 36, wherein the alkali compound is an alkali metal hydroxide, an alkaline earth metal hydroxide, lime (CaO), soda (Na_2O), or a combination thereof.

38. The composition of any one of claims 22, 23, 26 to 37 or the digestion tablet of any one of claims 24 to 37, wherein the alkali compound is present in an amount sufficient to maintain the pH of the digestion mixture above pH 10.

39. The composition of any one of claims 22, 23, 26 to 38 or the digestion tablet of any one of claims 24 to 38 further comprising an ionic salt in an amount sufficient to maintain an ionic strength of the digestion mixture to at least 0.1M.

40. A digestion composition of any one of claims 22, 23, 26 to 39 for use in a system according to claim 18 or method according to claim 19 for preparing a sample containing low-level metal value for metal value analysis by a detector.

41. A digestion tablet of any one of claims 24 to 39 for use in a system according to claim 18 or method according to claim 19 for preparing a sample containing low-level metal value for metal value analysis by a detector.

42. A digestion composition of any one of claims 22, 23, 26 to 39 for use in a system according to claim 20 or method according to claim 24 for analysing a sample containing a metal value at low levels.

43. A digestion tablet of any one of claims 24 to 40 for use in a system according to claim 20 or method according to claim 24 for analysing a sample containing a metal value at low levels.

Application No:	GB1611434.0	**Examiner:**	Dr Richard Wood
Claims searched:	1, 3-24	**Date of search:**	11 April 2017

Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
X	1, 3-6, 9-11, 18-24	EP 0049172 A1 (HUNTER) - see especially Example 1.
X	1, 7-11, 20-24	CN 202136838 U (GUANGXI DIBO MINING GROUP) - see especially EPODOC abstract, WPI abstract accession no. 2012-C33922.
A	-	RO 77952 A2 (INST DE FIZICA SI INGINERIE NUCLEARA)
A	-	GB 2066456 A (GEN MINING UNION CORP)
A	-	US 2014/072095 A1 (FESER et al.)

Categories:

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^X :

Worldwide search of patent documents classified in the following areas of the IPC

C22B; G01N

The following online and other databases have been used in the preparation of this search report

WPI, EPODOC

International Classification:

Subclass	Subgroup	Valid From
G01N	0033/24	01/01/2006
C22B	0003/12	01/01/2006
C22B	0003/24	01/01/2006
G01N	0023/223	01/01/2006