Title: CALIBRATION METHOD AND APPARATUS

Abstract: Calibration of a sample material analysis device (1), capable of emitting a penetrative energy output (4) detectable by one or more detectors (3), is performed by introducing at least a first reference material with a first attenuation response into the penetrative energy output (4) and recording a corresponding attenuation measurement. This attenuation measurement is compared with the first attenuation response and any difference calculated as a first correction factor. The correction factor is then applied to an attenuation measurement indicative of a sample material (6) composition as measured by one or more of the detectors. The accuracy of the composition measurement of the sample material is enhanced.
TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published: with international search report

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CALIBRATION METHOD AND APPARATUS

TECHNICAL FIELD

The present invention relates generally to a method of non-destructive analysis and apparatus pertaining thereto, and in particular to a means of improving the accuracy and calibration of X-ray analysis machines.

BACKGROUND ART

X-ray analysis has become a prime investigative and measuring tool for use in a variety of industries and applications, including industrial process quality control, industrial inspection systems such as airport baggage scanners and medical systems such as bone densitometers. In particular, dual energy detectors found in Dual energy X-ray Absorptiometry (DXA) machines are employed in systems seeking to distinguish between sample materials containing multiple constituents.

The objectives and information requirements naturally differ significantly between such applications. However, some attributes of the x-ray equipment such as measurement accuracy, correct calibration and awareness of potential errors and their sources are germane, albeit to varying degrees, to each application.

Composition information of a sample material may be determined by measurement of x-ray energy attenuated by a sample material in two distinct energy bands.

Photoelectric absorption and Compton scattering are the two mechanisms generally responsible for the absorption of x-rays. Moreover, the degree of absorption by each of these mechanisms is a function of x-ray energy and differs among materials of different atomic numbers. Consequently, measurements at two distinct energies can be used to distinguish between two different constituent sample materials.
DXA techniques can, for example, be used to distinguish bones from soft tissue in medical imaging, to identify hazardous materials in baggage scanning or to calculate the Chemical Lean (CL) ratio of fat to meat for meat produce processed by meat works and the like.

In order to obtain information from two energy levels, several different techniques may be utilised. As one example, in a switched mode DXA system, the voltage on the x-ray tube is periodically changed from a high to low voltage, thus shifting the energy spectrum of the resultant x-ray beam. This enables measurements of the imaged sample material at the different energy outputs to be obtained from a single set of solid state detectors illuminated by the x-ray beam.

Such arrangements require the use of x-ray detectors having broad-band sensitivity to the different energies levels outputted. However, switch-mode systems have drawbacks in terms of accuracy, sensitivity and response time.

An alternative involves the use of a broad spectrum x-ray beam produced with multiple output energies, and relies on the detectors to discriminate between x-ray energies whereby separate detectors output separate signals for high and low x-ray energies received. The various types of detectors used include those with two detector elements stacked on top of each other, i.e., a ‘stacked array’ and ‘side-by-side’ detectors which, as implied by the name, involves two rows of detectors placed ‘side-by-side’, each row having a different energy sensitivity, corresponding to the high and low energy values.

Regardless of the sensitivity and response of these detector methods, the accuracy of the results is wholly dependent on the accurate calibration of the DXA machine itself and on maintenance of that calibration.

The accuracy of DXA machines, like many physical measurement instruments, will drift, both in the short term and over time. This may be due to a variety of reasons including changes in the x-ray source spectrum, detector sensitivity, beam-hardening and so forth.
In principle, x-rays analysis devices capable of emitting an x-ray beam and measuring the absorption or attenuation of that beam by a sample material are predominately stable. However, attempts to use such devices to perform low tolerance measurements can be affected by transient changes in the x-ray environment. These transient changes, which can affect the device’s x-rays emission and/or detection, may have duration of up to several seconds.

Such changes to the x-ray environment might include:

- Large-scale changes in the electrical supply, e.g. variations in the AC current frequency or fluctuations in the supply voltage;

- Small-scale electrical changes within the machine such as performance drift in the machine components;

- Temperature changes to critical components, for example, warming of the x-ray detector module, whose performance is temperature dependant;

- Alterations to the path of the x-ray beam as components move, age or change temperature;

- Drift in the x-ray spectrum as the target point (where the electron beam impacts, creating the x-ray beam) warms/changes.

- Changes to the base, or “dark-current” response of the x-ray detectors due to a number of factors such as temperature, humidity, electrical environment, and the like. These changes result in a small shift or ‘offset’ in the detector response, in comparison to the expected result.

- Changes to the response rate or “gain” of the detectors due to a number of factors such as temperature, humidity, electrical environment, etc. Changes to the gain result in a small proportional shift in the detector response. This shift increases as the detector response increases compared to the expected result.
Known calibration techniques involve the use of defined reference material(s) or ‘phantoms’ of known x-ray absorption properties used to compare the results measured by the machine with the expected results. Such phantoms are typically comprised of materials exhibiting absorption properties at the extremes of the response spectrum (i.e. prefect reflectors or absorbers), with intermediate values being interpolated therebetween.

However, this calibration technique assumes that the response of the DXA machine and the sample material is predictable between the phantom reference points. It has been found that this assumption is not necessarily true and can result in significant errors.

While the above discussed problems related to primarily to x-ray analysis of materials, the principles involved are equally applicable to other non-destructive analysis means including magnetic resonance imaging (MRI), computed tomography (CT), ultrasound, and the like.

To illustrate the shortcomings of known composition analysis devices, the meat industry is now referred to by way of example only.

As a component of the New Zealand Beef Industry, manufactured meat represents the most significant portion of beef sold. Such meat is packed and shipped from a meat plant in a plurality of plastic bags located within large cardboard boxes.

There is a significant fiscal incentive to be accurately aware of the CL values of the meat produced. If meat is sold with a CL exceeding the customer’s defined parameters, significant financial penalties can be applied.

Inaccuracies in existing systems in monitoring the meat CL include various short and long term calibration errors which are either unquantifiable or are not correctable in real time as part of an in-line processing of the meat.

Present methods for CL measurement involve selecting a few random sample cartons from those being processed before determining their CL by any of several known alternative methods.
However, there are several fundamental problems with this methodology. Firstly, the sampling is intermittent and therefore cannot be accurately representative of the entire meat consignment. Furthermore, a consequence of the nature of the sampling is the inability to sell the sample boxes at all, or only at a discount.

There are clear advantages in being able to measure the CL of manufacturing meat as part of the continuous meat production process, for example after being chunked, diced or minced. However, the standard soxhlet test currently employed for CL measurement is unsuitable for in-line plant operations whilst the alternate methods available have significantly less precision.

It is an object of the present invention to address the foregoing problems or at least to provide the public with a useful choice.

Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

**DISCLOSURE OF INVENTION**

According to one aspect of the present invention there is provided a method of providing enhanced calibration of a sample material analysis device capable of emitting a penetrative energy output detectable by one or more detectors, said device including reference material with a known attenuation response to said penetrative energy,

wherein the sample material is passed through said penetrative energy output and a corresponding first attenuation measurement indicative of said sample material composition is measured by one or more detectors,

said method characterized by the steps of:

- introducing at least a first reference material portion with a first attenuation response into said penetrative energy output and recording a corresponding second attenuation measurement;
- comparing said second attenuation measurement to said first attenuation response and calculating any difference as a first correction factor;

- applying said first correction factor to said first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

According to a further aspect of the present invention, there is provided a penetrative energy beam sample material analysis device capable of performing the above-described method.

It can be seen therefore that by application of the above method, the accuracy of composition measurement performed is improved by correcting the initial attenuation measurement of the sample material from the effects of offset errors, i.e. the erroneous signal or 'correction factor' measured by the detectors unrelated to the incident penetrative energy beam. In particular, the above method is pertinent to measurement systems where offset is the predominant measurement error factor and the effects of gain error (i.e. the correlation variations in the detector output according changes in the penetrative energy beam incident on the detectors) are negligible. It will thus be appreciated that said correction factor may be positive or negative and may vary with respect to time.

As used herein, the term 'attenuation response' of a material denotes the variation in attenuation of an penetrative energy beam incident on the material as a function of a given parameter of the energy beam such as (but not limited to) input energy, wavelength, voltage, amplitude, and so forth.

The term 'reference material portion' refers to that portion of the reference material impinged by said penetrative energy beam during the respective attenuation measurements and includes the case where all the reference material is thus impinged.

Therefore, according to one aspect of the present invention, said reference material may be formed from one or more reference material portions.

It will be appreciated that different reference material portions may be formed with different
attenuation responses by varying the portion’s constituent materials, thicknesses or both. This
variation may be achieved by physically separate portions and or by presenting different
aspects of an asymmetrical and/or non-homogenous potion to the penetrative energy beam.

As used herein, the term ‘penetrative energy output’ includes any radiation or emission
capable of at least partial penetration of, transmission through, and/or absorption by the
sample material without direct physical contact between the sample material and the material
analysis device. Any appropriate energy mechanism or medium may be used including any
electromagnetic, sonic, nuclear and/or radioactive transmission or emission.

The term ‘sample material’ as used herein, indicates any material whose composition is
sought, typically being a material with an irregular surface and which may be of a variable
consistency and of non-uniform size.

The sample material would typically be composed of two or more constituents of differing
molecular characteristics. Dependent on the particular application, the nature of the
constituent material information to be ascertained may differ. In the case of airport baggage
scanners, the items of interest may be explosives that may appear as contaminates concealed
within other materials. In the determination of meat Chemical Lean (CL) the sample material
would be a combination of lean meat and fat, the accurate determination of which is the
information sought.

Preferably, said analysis device includes an x-ray source capable of outputting penetrative
energy in the form of an x-ray beam.

However, the invention is not necessarily limited to x-rays and associated apparatus, but is
equally applicable for use with other non-destructive/non-invasive material analysis methods
including, but not limited to ultrasonic scans, MRI, CT and the like.

Reference made to use of x-rays as the material analysis mechanism should be understood as
being for exemplary purpose only and is not limiting.
According to a further aspect of the present invention, the reference material is formed from one or more constituent materials that in combination simulate the attenuation properties of said sample material to the penetrative energy beam.

Preferably, said first attenuation response of said first reference material portion is selected to closely match that of the sample material.

Preferably, the variation the reference material x-ray attenuation as a function of incident x-ray energy has a relationship substantially similar to that of the sample material, or to constituents of interest within said sample material. In one embodiment, said reference material x-ray attenuation response is scaled to be substantially congruent with that of said sample material. Preferably, said scaling is provided by appropriate variation in the quantity of reference material matter.

Preferably, the or each said reference material portion is introduced into the x-ray beam immediately before and/or after passing the sample material through said x-ray beam. According to one embodiment, the or each reference material portion is moved linearly into and out of the penetrative energy output.

The indicative composition measurement of the sample material may be further enhanced by recording an x-ray attenuation measurement of the unimpeded x-ray beam.

According to a further aspect of the present invention, said sample material analysis device is a Dual energy X-ray Absorptiometry (DXA) device.

Preferably, said detectors include detectors configured with optimum sensitivity for low energy attenuation measurements and detectors with optimum sensitivity for high-energy attenuation measurements.

The low and high energy attenuation measurements are typically performed in energy bands of approximately 50-80 KeV and 90—140 KeV respectively. The underpinning principle behind the use of two separate energy level measurement is to ensure the utilization of the
Photoelectric effect and Compton scattering mechanisms to permit composition analysis in known manner.

According to a further aspect of the present invention, the analysis device is configured such that by being introduced into said penetrating energy output the or each said reference material portion is interposed between the penetrating energy output source and each detector.

Preferably, a said first attenuation measurement is recorded for each individual detector.

Preferably, a said first correction factor is applied to each individual first attenuation measurement of each detector.

By placing the or each reference material portion into the penetrative energy output/ x-ray beam immediately before, after or before and after taking the attenuation measurement for each sample material, both short and long term drift or calibration errors may be addressed almost instantaneously.

Most changes in the x-ray environment are relatively small, and the environment quickly reaches a new equilibrium over a few seconds. To effectively compensate for these relatively rapid changes in the x-ray environment, the attenuation of the penetrative energy output by the or each reference material portion must be measured:

- as soon as is practical before and/or after the first attenuation measurement of said penetrative energy output by the sample material;

- under conditions close as possible to those existing at the point of said first sample material penetrative energy output attenuation measurement.

In particular, the penetrative energy output beam path, detector settings and penetrative energy output emission settings of the penetrative energy output material analysis system should be identical for both the reference material and sample material attenuation measurements.
Thus, according to a further aspect of the present invention, the or each reference material portion is moved into, and out of, the penetrative energy beam output path, whilst the remainder of the material analysis device remains static. Reducing the moving parts by this mechanical configuration minimizes the number of the error inducing system variations associated with moving componentry.

According to one embodiment, said penetrative energy output and the or each detector may remain operational during and between successive analysis of different sample materials. Thus, both the practical inconvenience and the fluctuations associated with repeated power supply switching are obviated.

In circumstances where large variations in the sample material thickness or composition may be encountered and/or a significant degree of gain fluctuations, the above measurement method may be yield insufficient accuracy. To counteract the effects of gain fluctuation, a two-step correction, utilizing at least two reference material portions of different attenuation responses is required to take account of the two unknowns present.

Thus, according to a further aspect of the present invention, said method is characterized by the further steps of:

- introducing a second reference material portion with a second attenuation response into said penetrative energy output and recording a corresponding third attenuation measurement;

- applying said fist correction factor to said third attenuation measurement to obtain an offset-corrected third attenuation measurement;

- calculating a second correction factor from the ratio of any differences between the said second attenuation response and offset-corrected third attenuation measurement;

- applying said second correction factor also to first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.
According to one embodiment, said first and second reference material portions are configured to be physically distinct with differing attenuation responses.

In an alternative embodiment, said first and second reference material portions are physically connected and are defined by moving different regions and/or orientations of the reference material into the penetrative energy output.

Preferably, said reference material is formed from regions of different thickness and/or composition.

Preferably, said first reference material portion produces a total attenuation of said penetrative energy output of a factor of at least 2 less than said second reference material potion.

Preferably, said first and second attenuation responses of said first and second reference material portions respectively are chosen to bracket the range of attenuation measurement variation likely due to variations in sample material thickness and composition.

Although the first and second reference material portions need not be formed from the same constituents, it is desirable, though not essential for both the first and second attenuation response are substantially matched to the anticipated attenuation characteristics of the sample material in the operational attenuation regions of the reference material portions.

Considering the calibration correction for gain and offset from a further aspect, the present invention, said method is characterized by the further steps of:

- introducing a second reference material portion with a second attenuation response into said penetrative energy output and recording a corresponding third attenuation measurement

- comparing said second and third attenuation measurements to said second attenuation response of said second reference material portion;

- calculating corrective gain and offset by solving in known manner simultaneous
equations resulting from a comparison of the said second and third attenuation measurements to the said known first and second attenuation responses respectively to give a second corrective factor

- applying said second correction factor also to first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

In a simple linear system, the output is related to the input by the equation:

\[ \text{Output} = (\text{Gain} \times \text{Input}) + \text{offset} \quad \text{Equation 1} \]

Given that both the gain and offset can drift with time, these can be corrected in a DXA material analysis system (for example) by using at least two reference material portions of differing attenuation response to provide two set of values for equation 1 above. Solution of these two simultaneous equations can be used to yield the corrective gain and offset values to apply to the said first attenuation measurement obtained from the sample material.

Thus, in a preferred embodiment, two measured coordinate pairs:

15 - \((A_1, B_1)\) and

- \((A_2, B_2)\),

where

- \(A_1\) is said known first attenuation response corresponding to said first reference material portion; and

- \(A_2\) is said known said second attenuation response corresponding to said second reference material portion

- \(B_1\) is said second attenuation measurement for the first reference material portion

- \(B_2\) is said third attenuation measurement for the second reference material portion
are related by simultaneous equations 2 and 3;

\[ B1 = GAIN \times A1 + OFFSET \]  \hspace{1cm} \text{Equation 2}

\[ B2 = GAIN \times A2 + OFFSET \]  \hspace{1cm} \text{Equation 3}

and solving these in the normal manner, the corrective gain and offset are given by the equation 4 and 5

\[ GAIN = \frac{(B1 - B2)}{(A1 - A2)} \]  \hspace{1cm} \text{Equation 4}

\[ OFFSET = B2 - (GAIN \times A2) \]  \hspace{1cm} \text{Equation 5}

and are used to calculated said second corrective factor and applied to the first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

Measuring the attenuation of the penetrative energy beam by the or each reference material portion and that of the sample material independently increases the measurement accuracy. The detector measurement error is largely quantum and thus related to the square root of the photon count received by the detectors. Therefore, the smaller the received signal by the detectors, the larger the error.

It is thus desirable to solve for offset from equation 3 due to the higher measured response values and consequently less inherent noise due to quantum effects, as referred to below.

If the or each reference material portion and the sample material were placed in the penetrative energy beam simultaneously, this would effectively double the mass attenuating the penetrative energy output and cause the error to rise by the square root of two.

Moreover, it is only by independently measuring the penetrative energy attenuation by the reference material portions and the sample material that an assured measurement of any difference between the second attenuation measurement (and the third attenuation measurement if applicable) and the or each known reference material portion attenuation...
response can be attributed solely to a measurement error by the device.

Although it may be theoretically possible to measure the combined attenuation response of the reference material and the sample material simultaneously, in practice, it is impossible to accurately distinguish between the two responses.

In existing systems, such as in a meat production line, after the x-ray machine is initially calibrated, there is no instantaneous means of correcting the composition analysis measurements (particularly in relation to CL measurements) taken for each sample material, i.e. each meat box. As the randomly selected samples used for analysis are currently subjected to a time consuming testing process, defective samples may go undetected or require retrospective recall once a faulty batch has been determined.

Therefore, the ability to correct for drift and other calibration errors in (effectively) real time provides significant practical, performance and economic benefits.

Furthermore, maintaining a record of the corrections applied to a given DXA machine over time provides an indication of possible preventative maintenance requirements.

A relatively rapid increase in the magnitude of said first correction factor applied to said first attenuation measurement could indicate the impending failure if the x-ray source for example.

Thus, according to a further aspect of the present invention, a historical record of each said first correction factor applied to said first attenuation measurement is recorded to provide a preventative maintenance database.

**Brief Description of Drawings**

Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawings in which:

Figure 1. Shows a perspective view in partial section of a preferred embodiment of the
present invention of a DXA analysis device and housing;

Figure 2. Shows a side elevation of the DXA analysis device shown in figure 1;

Figure 3. Shows a plan view of the DXA analysis device shown in figure 1;

Figure 4. Shows a perspective view of the DXA analysis device shown in figure 1;

Figure 5. Shows an exploded perspective view of the components of the DXA analysis device shown in figure 4;

Figure 6. Shows a plan view in section of the DXA analysis device shown in figure 1 with the reference tile in the x-ray beam;

Figure 7. Shows a plan view in section of the DXA analysis device shown in figure 1 with the reference tile out of the x-ray beam, and

Figure 8 a-f Shows different configurations of reference tile in accordance further embodiments of the present invention.

**BEST MODES FOR CARRYING OUT THE INVENTION**

The figures illustrate one embodiment of the present invention of a penetrative energy beam analysis device in the form of a Dual Energy X-ray Absorptiometry (DXA) device (1). Although the DXA device (1) may be used in a variety of applications, the embodiment illustrated relates to the analysis of meat to establish the Chemical Lean (CL) of manufactured meat.

It will be understood that this is for exemplary purposes only and the present invention may be utilised, by way of disparate example, to determine the presence of explosives in airport baggage scanning, to assess the bone density of a human patient or the fat content of cheese.

Figure 1-7 illustrates a DXA (1) device (shown in figures 2-7 without a shroud or other distracting encumbrance) consisting, in essence, of an x-ray generator (2) and a plurality of
detectors (3). The x-ray generator (2) produces a fan-shaped x-ray beam (4), emitted in a plane orthogonal to the direction of movement (represented by the direction of arrow A) of a conveyor belt (5) transporting sample material in the form of meat boxes (6) through the x-ray beam (4).

The x-ray generator (2) componentry includes an enlarged substantially cuboid base unit (7), with a substantially planar inverted triangular x-ray beam guide (8) extending transversely from the upper surface. The fan shaped x-ray beam (4) is emitted from, and co-planar with, the top of the x-ray guide (8), extending vertically until intersecting the detectors (3).

One type of detector (3) which may be used is a stacked array detector (3) in which two detector elements are stacked on top of each other. Typically, a forward detector will measure total x-ray flux and a rearward detector will measure only higher energy x-ray photons not stopped by an intervening filter. Low energy photons may be deduced from these two measurements.

Alternative detectors (3) include side-by-side detector construction, whereby two rows of detector elements (3) are placed side-by-side and scanned along the imaged sample material (6) in a direction perpendicular to the rows. The detector (3) elements of the first row has a different energy sensitivity from that of the second row. Scanning the sample material (6) causes the two rows to pass over the same areas of the imaged object, each making measurements.

Figure 1 is partially sectioned to show the DXA device (1) located within a housing (9) configured to pass the sample materials to be analyzed (i.e. meat boxes (6)) along a conveyor belt (5), which runs orthogonally (vertically and horizontally) through the x-ray beam (4). The housing (9) is aperture to accept the passage of the meat boxes (6), and contains appropriate radiation shielding.

Referring to figures 2-7 a reference material and actuator mechanism are shown. The reference material is in the form of a first reference tile (10) composed of an elongated
aluminum u-shaped channel (11), into which is inserted an elongated rectangular cross-sectioned polythene bar (12).

The reference tile assembly (11, 12) is moveably located within an actuator assembly (13), in which dual solenoids (14) are attached to the actuator framework (15) and to each end of the first reference tile (10). Application of the appropriate drive signals to the solenoids (14) moves the first reference tile (10) laterally into or out of alignment with the x-ray beam (4).

Figure 5 shows an exploded view of the DXA (1) components shown in figure 4, showing the reference tile (10) and actuator assembly (13) constituents in more detail.

Figures 6 and 7 show a plan view of the DXA (1) device viewed from below the detectors (3) looking downwards to the actuator assembly (13) and x-ray generator (2). The first reference tile (10) is shown in figure 6 located directly in the x-ray beam (4), whilst in figure 7, the solenoids (14) have moved the reference tile (10) within the actuator assembly (13) to a position outside the x-ray beam (4).

The solenoids (14) may rapidly cycle the first reference tile (10) into and out of the x-ray beam (4) immediately before, immediately after, or before and after a sample meat box (6) moving along the conveyor (5) passes through the x-ray beam (4).

As a brief overview of the calibration process involved in the present invention, the following steps are performed:

A measurement of the detector (3) response to the unimpeded x-ray beam is taken, i.e., an ‘empty field’ measurement for use in calibration calculations as detailed below.

The meat box (6) is passed through the x-ray beam (4) and a corresponding first attenuation measurement is measured by the detectors (3) and recorded.

The first reference tile (10) is then moved into said penetrative energy output, i.e. the x-ray beam (4) immediately after the meat box (6) has cleared the x-ray beam (4) and a corresponding second attenuation measurement is made by said detectors (3) and recorded.
In alternative embodiments, the reference tile (10) may be moved into the x-ray beam (4) immediately before, or before and after the meat box (6) has cleared the x-ray beam (4), with corresponding second attenuation measurements being taken in each instance.

The second attenuation measurement is then compared to the known attenuation response of the first reference tile (10) and a first correction factor is calculated based on from any difference. By applying said first correction factor to said first attenuation measurement the accuracy of said indicative composition measurement of the sample material is enhanced.

In more detail;

Considering the occurrence of an unspecified event which affects either the x-ray beam (4) spectrum or detector (3) absorption response, so that the effective x-ray absorption of the sample material is altered, the present invention is directed to the removal of the effects of such a unspecified event.

The underlying relationships are as follows:

Firstly, although beam-hardening issues are still valid in this situation, for the sake of clarity they are ignored in the following discussion due to their complexity and do not in any event affect the analysis validity.

Making the assumption that de Beer's law is applicable for a composite detector (3) response, with

\[ K_S = \frac{L_0}{L} \]

and

\[ \ln(K_S) = \mu_S m_s \]

where
\[ K_S = \text{the detector (3) response ratio for the sample material (6),} \]

\[ L_0 = \text{the detector (3) response with no material in the beam path,} \]

\[ L = \text{the detector (3) response with sample material (6) in the beam path,} \]

\[ \mu_S = \text{the sample material's (6) attenuation coefficient,} \]

\[ m_S = \text{the mass of sample material (6) in the beam path.} \]

Regarding a reference tile (10) constructed in such a manner that its attenuation coefficient, \( \mu_R \), is everywhere related to the sample material (6) attenuation coefficient, \( \mu_T \), by a constant ratio \( \alpha \), the absorption coefficients of the reference tile (10) and sample material (6) may be characterised as \( \mu_R = \alpha \mu_T \).

The actual intensity ratio before the event is denoted by \( K_S \) and the intensity ratio being currently measured (i.e. during the event) for some target sample (6), measured after the event is denoted by \( Q_S \).

At the closest practical instant to measuring the sample material (6) intensity ratio, a corresponding measurement of the reference tile (10) is performed, for which the intensity ratio before the event, \( K_R \), and the current intensity ratio, \( Q_R \) are known. Given the applicability of de Beer's relationship, in the case of the reference tile (10),

\[ \ln(K_R) = \mu_R \cdot m_R \]

and

\[ \ln(Q_R) = \eta_R \cdot m_R \]

where \( \eta_R \), is the current effective attenuation coefficient for the Reference Tile. Similarly, for the sample material (6),

\[ \ln(K_S) = \mu_S \cdot m_S \]

and

\[ \ln(Q_S) = \eta_S \cdot m_S \]

the ratio of natural logs for the sample material (6) is
\[
\frac{\ln(K_s)}{\ln(Q_s)} = \frac{\mu_s m_s}{\eta_s m_s} = \frac{\mu_s}{\eta_s}
\]

whilst that for the first reference tile (10) is

\[
\frac{\ln(K_R)}{\ln(Q_R)} = \frac{\mu_R m_R}{\eta_R m_R} = \frac{\mu_R}{\eta_R}
\]

As the relationship between the absorption coefficients of the first reference tile (10) and sample material (6) were defined as \( \mu_R = \alpha \mu_T \), then it follows that

\[
\frac{\mu_s}{\eta_s} = \frac{\mu_T}{\eta_T}
\]

and therefore,

\[
\frac{\ln(K_s)}{\ln(Q_s)} = \frac{\ln(K_R)}{\ln(Q_R)}
\]

Consequently, we can estimate the unaffected sample material (6) intensity relationship as

\[
\ln(K_s) = \frac{\ln(K_R)}{\ln(Q_R)} \times \ln(Q_s)
\]

This relationship can readily be generalised to multiple detector (3) cases e.g. dual or multiple energy detection systems.

Thus the effects of variation in either the x-ray spectrum or the detector response can be correctly accounted for and eliminated, provided the attenuation curves of the sample material (6) and first reference tile (10) match.

As will be readily appreciated by one skilled in the art, the above attenuation correction calculation are repeated for different energy regions as required, corresponding to the points of maximum (3) detector sensitivity for the detector configuration of the particular system being considered.
Theoretically, this technique could account for quite large changes in the x-ray environment, but in practice the reference tile is chosen to match the attenuation curve of a particular sample material (6). Individual sample material (6) may differ to each other and, to a lesser or greater extent, to the sample material attenuation curve.

Hence, the ability to use the first reference tile (10) to account for x-ray environment changes is limited by the degree to which the reference tile (10) attenuation curve matches those of the individual sample material (6).

Consequently, if the range of attenuation curves required to encompass the likely variations in sample materials (6) is large, there are advantages in using a range of reference tiles (10). The range of different reference tiles (10) is chosen to provide an incremental range of different attenuation curves, from which the appropriate reference tile (10) is chosen to match the attenuation response characteristics of the sample material (10) being analysed.

The calibration errors affecting the accuracy of the sample material analysis device manifest themselves as two main effects, namely gain and offset errors.

Although the magnitude of both effects drifts with time, it has been found in practice that in a DXA machine system, the offset term can vary considerably in a few seconds. Conversely, although the gain term can also vary, its drift is slower and less significant.

The above embodiments using a single reference tile (10) to determine the effects of offset on the measurement system, are based on the assumption the gain error component is insignificant.

However, if large changes in sample material thickness or composition are routinely encountered, or if the gain drift is large, the effect of uncorrected gain changes may be significant in the measured result. Consequently, a two-step correction is required, to overcome the two unknowns.

In a linear system, the following equation may be applied;
Output = (Gain x Input) + Offset  \hspace{1cm} \text{Equation 1}

If a second reference tile (110) with a differing attenuation response to the first reference tile (10) is used to obtain a second set of measurement data, a set of two simultaneous equation 1 is generated and may be solved in known manner to give the offset and gain error.

This method is an extension of the above described method used for a single reference tile (10) and consists of the further steps of;

- introducing the second reference tile (110) with a known second attenuation response into the x-ray beam (4) and recording a corresponding third attenuation measurement

- comparing said second and third attenuation measurements to said second attenuation response of said second reference material portion;

- calculating the corrective gain and offset by solving the simultaneous equations resulting from a comparison of the said second and third attenuation measurements to the said known first and second attenuation responses respectively to give a second corrective factor, and

- applying said second correction factor also to first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

Thus, by applying the above method using two reference tiles (10, 110) of differing attenuation response two set of values for equation 1 are obtained. Solution of these two simultaneous equations can be used to yield the corrective gain and offset values to apply to the said first attenuation measurement obtained from the sample material.

Represented mathematically, two measured coordinate pairs:

- \((A1, B1)\) and
where

- A1 is said known first attenuation response corresponding to said first reference tile (10); and

- A2 is said known said second attenuation response corresponding to said second reference tile (110)

- B1 is said second attenuation measurement for the first reference tile (10)

- B2 is said third attenuation measurement for the second reference tile (110)

are related by simultaneous equations 2 and 3;

\[
B1 = GAIN \times A1 + OFFSET \quad \text{Equation 2}
\]

\[
B2 = GAIN \times A2 + OFFSET \quad \text{Equation 3}
\]

and solving these in the normal manner, the corrective gain and offset are given by the equation 4 and 5

\[
GAIN = \frac{(B1 - B2)}{(A1 - A2)} \quad \text{Equation 4}
\]

\[
OFFSET = B2 - (GAIN \times A2) \quad \text{Equation 5}
\]

and are used to calculated said second corrective factor and applied to the first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

The second reference tile (110) may be introduced into the x-ray beam (4) by a second actuator mechanism (not shown) substantially identical to the actuator assembly/solenoids (13, 14) used with the first reference tile (10).
Alternative actuator mechanisms are of course possible and as such fall within the scope of the invention.

In the above embodiments, the reference tiles (10, 110) have been referred to as physically distinct entities. It should be appreciated however that this need not necessarily be the case. The reference tiles may be considered as reference material portions whose key properties is their attenuation response, and in particular that the attenuation responses differ from each other.

Thus, it will be apparent that a different attenuation response may be achieved by moving an appropriately configured reference tile to alter the thickness and/or constituents seen by the path of the x-ray beam (4).

This may be achieved by numerous configurations, of which a selection is shown in figure 8 a-f.

Figure 8a shows a schematic representation where the first and second reference tiles (10, 110) are physically separate objects. The individual tiles are inserted into the x-ray beam (4) immediately before or after, or before and after the attenuation measurement of the sample material (e.g. a meat box (6)) to perform the above-described calibration correction.

In figures 8b, c, e–f, the first and second reference tiles (10, 110) are formed as single physical entity, with different attenuation responses according to the particular portion and/or orientation of the reference material inserted into the x-ray beam (4).

Figure 8b shows a stepped reference tile whereby two different attenuation responses are obtained by laterally moving the reference material in direction X to place the different thicknesses of the first and second reference tile portions (10, 110) into the x-ray beam (4). The whole reference tile (10,110) may be formed from the same constituents in this embodiment.
Figure 8c shows a similar configuration to figure 8b, with the exception that the two reference tile portions (10, 110) are of the same thickness, but formed from different constituents.

Figure 8d shows a further alternative whereby both the first and second reference tiles (10, 110) are physically distinct, though each with a stepped portion. Thus, effectively four reference tile portions with different possible attenuation responses are available. This may be beneficial for circumstances where the variation of thickness and/or constituents in the sample materials is significant.

Figure 8e shows a multi-stepped reference tile with three different thicknesses, effectively providing three different reference tile portions (10, 110, 1110).

Figure 8f shows an alternative means of moving the reference tiles (10, 110) into the x-ray beam (4). Instead of moving a stepped tiles laterally (as described above), the combined reference tile portions (10, 110) are rotated in direction Y to place the different thicknesses of the respective first and second reference tile portions (10, 110) into the x-ray beam (4).

It will be appreciated that the configurations shown represent a small exemplary range of the total configurations possible, and the present invention is not limited solely to those shown.

Known x-ray analysis devices incorporating some means of x-ray absorption measurement error correction are deficient for a variety of reasons, including:

- “Phantoms” of suitable material are measured at long time intervals, to check device calibration;

- A reference tile is brought into the x-ray beam at the same time as the sample is being measured.

- A reference material is on a different optical path to that of the target material.

- Measurements of the sample material and reference tile are not closely matched in time.
• The x-ray absorption curve of the reference tile is not matched (to within a constant ratio) to that of the target material.

The present invention addresses all these deficiencies.

To reiterate, although the present invention has been predominantly described with reference to x-ray material analysis and DXA in particular, the invention is not limited to same. Any non-invasive/non-destructive material analysis technique may be employed.

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof.
CLAIMS:

1. A method of providing enhanced calibration of a sample material analysis device capable of emitting a penetrative energy output detectable by one or more detectors, said device including reference material with a known attenuation response to said penetrative energy,

wherein the sample material is passed through said penetrative energy output and a corresponding first attenuation measurement indicative of said sample material composition is measured by one or more detectors,

said method characterized by the steps of:

   • introducing at least a first reference material portion with a first attenuation response into said penetrative energy output and recording a corresponding second attenuation measurement;

   • comparing said second attenuation measurement to said first attenuation response and calculating any difference as a first correction factor;

   • applying said first correction factor to said first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

2. A method as claimed in claim 1, wherein said reference material may be formed from one or more reference material portions.

3. A method as claimed in claim 1 or claim 2, wherein different reference material portions are formed with different attenuation responses by varying the portion’s constituent materials, thicknesses or both.

4. A method as claimed in claim 1, wherein different reference material portions are provided by presenting different aspects of an asymmetrical and/or non-homogenous
potion to the penetrative energy beam.

5. A method as claimed in any one of the preceding claims, wherein said reference material portions are physically distinct.

6. A method as claimed in any one of the preceding claims, wherein said analysis device includes an x-ray source capable of emitting a penetrative energy output in the form of an x-ray beam.

7. A method as claimed in any one of the preceding claims, wherein the reference material is formed from one or more constituent materials that in combination simulate the attenuation properties of said sample material to the penetrative energy output.

8. A method as claimed in any one of the preceding claims, wherein said first attenuation response of said first reference material portion is selected to closely match that of the sample material.

9. A method as claimed in any one of the preceding claims, wherein a variation the attenuation of the penetrative energy output by reference material x-ray as a function of incident penetrative energy output energy has a relationship substantially corresponding to said sample material.

10. A method as claimed in any one of the preceding claims, wherein a variation the attenuation of the penetrative energy output by reference material x-ray as a function of incident penetrative energy output energy has a relationship substantially corresponding to constituents of interest within said sample material.

11. A method as claimed in any one of the preceding claims, wherein the attenuation response of at least one said reference material portion is scaled to be substantially congruent with that of said sample material.

12. A method as claimed in claim 11, wherein said scaling is provided by quantity variation of reference material matter.
13. A method as claimed in any one of the preceding claims, wherein, the or each said reference material portion is introduced into the penetrative energy output beam immediately before and/or after passing the sample material through said penetrative energy output beam.

14. A method as claimed in any one of the preceding claims, wherein the or each reference material portion is moved linearly into and out of the penetrative energy output.

15. A method as claimed in any one of the preceding claims, wherein the indicative composition measurement of the sample material incorporates a recording of a penetrative energy output attenuation measurement obtained from an unimpeded penetrative energy output beam.

16. A method as claimed in any one of the preceding claims, wherein said sample material analysis device is a Dual energy X-ray Absorptiometry (DXA) device.

17. A method as claimed in any one of the preceding claims, wherein said detectors include detectors configured with optimum sensitivity for low energy attenuation measurements and detectors with optimum sensitivity for high-energy attenuation measurements.

18. A method as claimed in claim 17, wherein said high and low energy attenuation measurements are typically performed in energy bands of approximately 90—140 KeV and 50-80 KeV respectively.

19. A method as claimed in any one of the preceding claims, wherein the analysis device is configured such that by being introduced into said penetrating energy output the or each said reference material portion is interposed between the penetrating energy output source and each detector.

20. A method as claimed in any one of the preceding claims, wherein a first attenuation measurement is recorded for each individual detector.

21. A method as claimed in any one of the preceding claims, wherein a said first correction
factor is applied to each individual first attenuation measurement of each detector.

22. A method as claimed in any one of the preceding claims, wherein the or each reference material portion is moved into, and out of, the penetrative energy beam output path, whilst the remainder of the material analysis device remains static.

23. A method as claimed in any one of the preceding claims, wherein said penetrative energy output and the or each detector may remain operational during and between successive analysis of different sample materials.

24. A method as claimed in any one of the preceding claims, wherein a historical record of each said first correction factor applied to said first attenuation measurement is recorded to provide a preventative maintenance database.

25. A method as claimed in any one of the preceding claims, wherein said method is characterized by the further steps of:

   - introducing a second reference material portion with a second attenuation response into said penetrative energy output and recording a corresponding third attenuation measurement

   - applying said fist correction factor to said third attenuation measurement to obtain an offset-corrected third attenuation measurement;

   - calculating a second correction factor from the ratio of any differences between the said second attenuation response and offset-corrected third attenuation measurement

   - applying said second correction factor also to first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

26. A method as claimed in claim 25, wherein said first and second reference material
portions are configured to be physically distinct with differing attenuation responses.

27. A method as claimed in claim 25, wherein said first and second reference material portions are physically connected and are defined by moving different regions and/or orientations of the reference material into the penetrative energy output.

28. A method as claimed in any one of the preceding claims, wherein said reference material portions are formed from regions of different thickness and/or composition.

29. A method as claimed in any one of claims 25-28, wherein, said first reference material portion produces a total attenuation of said penetrative energy output of a factor of at least 2 less than said second reference material potion.

30. A method as claimed in any one of claims 25-29, wherein said first and second attenuation responses of said first and second reference material portions respectively are chosen to bracket the range of attenuation measurement variation likely due to variations in sample material thickness and composition.

31. A method as claimed in any one of the preceding claims, wherein said method is characterized by the further steps of:

- introducing a second reference material portion with a second attenuation response into said penetrative energy output and recording a corresponding third attenuation measurement

- comparing said second and third attenuation measurements to said second attenuation response of said second reference material portion;

- calculating corrective gain and offset by solving in known manner simultaneous equations resulting from a comparison of the said second and third attenuation measurements to the said known first and second attenuation responses respectively to give a second corrective factor
applying said second correction factor also to first attenuation measurement to enhance the accuracy of said indicative composition measurement of the sample material.

32. A sample material analysis device capable of performing the method as claimed in any one of the preceding claims.

33. A method substantially as hereinbefore described with reference to, and as shown in the accompanying drawings.

34. A sample material analysis device substantially as hereinbefore described with reference to, and as shown in the accompanying drawings.
Figure 5
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.\(^7\): G01N 23/083

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DWPI, JAPIO: Int. Cl.\(^7\) and keywords: G01N 23/-, H05G 1/00, 1/02, 1/04, 1/06, A61N 5/10, G03B 42/02, 42/04, 41/16, 41/18; X ray, DXA, calibrate, compensate, correct, drift, tile, plate, template, standard, gauge, reference, mimic, scan, monitor, inspect.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>1-30, 32-34</td>
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<td>X</td>
<td>US 4787098 A (SILVER) 22 November 1988 Whole document</td>
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<td>X</td>
<td>US 6148057 A (URCHUK et al) 14 November 2000 Whole document</td>
<td>1-14, 19-24, 32-34</td>
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* Further documents are listed in the continuation of Box C

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<--- Special categories of cited documents: --->

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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Date of the actual completion of the international search

26 February 2003

Date of mailing of the international search report

07 MAR 2003

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