X-RAY SPECTROGRAPHY SAMPLE HOLDER CONTAINING AN ADJUSTABLY MOUNTED INTERNAL REFERENCE STANDARD


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1 Claim

ABSTRACT OF THE DISCLOSURE

An internal reference standard and sample holder for use in analysis by X-ray spectrography is disclosed herein. The holder comprises a sample cup having in its top portion an externally threaded support ring radially disposed and engaged by a support ring in which it is fixedly mounted. The reference standard holder has a reference standard mounted in a support which is threadably adjustable and lockable within the sample cup. The reference standard holder is mounted on the sample cup to set the desired distance between the cup and the standard and locked in place.

This invention relates to a device which facilitates X-ray spectrographic determinations and which particularly facilitates the use of an internal reference standard when making such determinations.

X-ray spectrography has been found to be a useful tool for qualitative analyses. Accurate quantitative analyses, however, have been complicated by absorption and enhanced effects of all elements (including the element sought) and the sample being analyzed. Among the prior art procedures that have been used in order to compensate for these matrix absorption effects are direct comparison with a standard, dilution and adjustment of internal standard procedures. The dilution technique and the technique which utilizes direct comparison with a standard, both suffer from serious deficiencies when they are employed in qualitative analyses. The internal standard procedures of the prior art include the use of background lines and the physical addition of a suitable element to the sample being analyzed. The use of background lines has not been too successful. Physical addition of a suitable element as part of the sample is subjected to X-ray spectrography and which thereby acts as an internal reference standard has been most successful in the case where a suitable element's absorption edge and emission line has been very close to that of the element sought. Most internal standard procedures, however, have not been completely compensated for matrix absorption effects because of the inability to isolate the added internal reference standard employed in the test sample and the difficulty of mechanically obtaining and retaining the required accuracies in test sample density and thickness.

Matrix absorption effects are partially compensated for by the use of an effective internal standard. In general the internal standard chosen is one of the two elements adjacent in the periodic table to the element for which spectrographic determinations are desired. The two adjacent elements most closely approximate the sought element in absorption properties and their intensities will vary in nearly the same way. Therefore the ratio of the intensities of the sought and the standard elements will not be much affected by changes in the matrix posed by the hydrocarbon sample variations. In addition, physically adding an internal standard entails additional time for sample preparation. The use of our novel device, in conjunction with an internal standard procedure, eliminates matrix absorption effects and eliminates loss of time occasioned by the methods heretofore used in sample preparation.

It is an object of our invention to provide a device to facilitate X-ray spectrographic determinations.

It is a further object of our invention to provide an internal reference standard holder which will facilitate X-ray spectrographic determinations.

It is an additional object of our invention to provide a device which will facilitate the X-ray spectrographic determination of elements in hydrocarbons and other matrices.

It is yet another object of our invention to provide an internal reference standard which will completely compensate for any matrix absorption effect encountered during X-ray spectrographic determinations.

These and other objects of this invention will become apparent from the description of the invention which follows, and from the accompanying drawing which illustrates a preferred embodiment of the invention, and in which:

The accompanying drawing is a cross sectional view of our internal reference standard holder.

The present invention contemplates in a preferred embodiment the use of a conventional sample cup suitable for use in an X-ray spectrograph, such as the Phillips inverted sample, three-position X-ray spectrograph. A sample cup is provided with a plastic film 11 which forms the bottom of the sample cup and holds the liquid sample to be analyzed. In accordance with the present invention, reference standard holding means taking the form of a pellet holder 14 is provided, which is suitable for holding an internal reference standard 13 in the form of a pellet. The pellet holder 14 is provided with a suitable opening 15 to receive reference standard adjusting means, such as an adjusting member 17, which as shown in the drawing has a lower disc portion 18 adapted to press against the upper portion of the pellet 13, and an upper portion 19 threaded for engagement with an internally threaded portion 16 of the pellet holder. The adjusting member 17 is also preferably provided on its upper end with a suitable means such as a slot 21 by which the adjusting member may be turned, thereby raising or lowering the disc portion 18 with respect to the pellet holder 14.

The lower portion of the pellet holder 14 includes a generally cylindrical inner wall 22 for receiving the pellet 13, and a tapered outer wall 23 adapted to cooperate with a tapered inner wall 24 of a locking ring 26 to engage the edges of a plastic film 27, which is preferably stretched across the bottom of pellet 13 with its edges engaged by the locking ring 26.

The upper portion of the pellet holder 14 is provided with an externally threaded portion 28 and with suitable means such as holes 29 by which the entire pellet holder may be rotated.

As will be apparent from the drawing, the sample cup includes a shoulder 31 in its upper portion. In order to suspend the reference standard holder of the present invention within the sample cup, a supporting ring 32 is provided which has a shoulder portion 33 adapted to engage the shoulder 31 of the sample cup.

The supporting ring 32 is also provided with internal threads 34 adapted to engage the externally threaded portion 28 of the pellet holder 14. In this way, the position-
ing of the pellet holder 14 and, therefore, of the internal reference standard 13 may be adjusted vertically by rotating the pellet holder 14 with respect to the supporting ring 32. This is normally accomplished by use of holes 29 in the pellet holder as previously described. The supporting ring 32 is preferably restrained from rotation with respect to the sample cup by the use of hole 36 in the supporting ring which is adapted to engage a pin 37 of the sample cup.

Locking means, such as a lock plug 38, may also be provided to lock the pellet holder 14 in place with respect to the supporting ring 32 and the sample cup 10 after the test sample has been poured into the cup 10 and if used, the lock plug 38 is provided with external threads 39, adapted to engage the threads 34 of the supporting ring. The lock plug 38 may be provided with suitable means such as a slot, not shown, so that it can be tightened with an ordinary screwdriver.

In operation, the pellet 13 is placed in pellet holder 14, the plastic film 27 is drawn tightly in place by use of locking ring 26 and the pellet is pressed tightly against the plastic film 27 by screwing the adjusting member 17 downwardly with respect to the pellet holder. The pellet holder is then placed within the sample cup 10 and the supporting ring 32 is placed on the shoulder 31 of the sample cup as shown in the drawing. The pellet holder is then screwed downwardly with respect to the supporting ring and sample cup, by means of the holes 29 until the pellet is the desired distance above the bottom of the sample cup, which is formed by the plastic film 11. Locking plug 38 is then screwed down tightly against the top of the pellet holder to hold the entire reference standard holder in position within the sample cup 10.

Although it is preferred to use the plastic film to cover the internal reference standard, you can also mix or coat the internal reference standard with an epoxy or other binder and then glue it to the internal reference standard holder.

Our novel device may be utilized in the X-ray spectrographic determination of calcium, lead, barium and zinc concentrations in lubricating oils and the lead content of gasolines. Our device may also be used in the determination of elements other than those mentioned above. Our device may also be used to determine elements in other matrices.

Obviously, many modifications and variations of the present invention are possible in light of the above teachings. It is, therefore, to be understood that within the scope of the appended claim, the invention may be practiced otherwise, than as specifically described.

Therefore, we claim:

1. An internal reference standard holder for suspending an internal reference standard within a sample cup having an X-ray transparent window in the bottom and containing a sample to be analyzed by X-ray spectography which comprises:
   an internally threaded upper supporting ring adapted to be accommodated in a fixed position in the upper portion of said sample cup;
   reference standard holding means having an externally threaded portion adapted to engage said internally threaded upper supporting ring for vertical movement within said sample cup for adjusting the distance between the bottom of said reference standard holding means and the bottom of said sample cup, said reference standard holding means also having an internally threaded portion and a generally cylindrical lower portion, the outer surface of which is tapered inwardly and downwardly;
   an internal reference standard suitable for use in X-ray spectography mounted below said reference standard holding means;
   a plastic film adapted to form the bottom of said reference standard holding means mounted between said internal reference standard and the sample contained in the sample cup whereby contamination of said internal reference standard by said sample in said sample cup may be prevented;
   reference standard adjusting means screwingly mounted in said internally threaded portion of said reference standard holding means to move vertically within said reference standard holding means, said internal reference standard being fixedly mounted to the bottom of said reference standard adjusting means, to adjust and fix the vertical position of said internal reference standard with respect to said reference standard holding means;
   an outer locking ring in a lower portion of said sample cup adapted to fit over the lower, tapered portion of said reference standard holding means and to seal the outer edge of said plastic film firmly between said reference standard holding means and said outer locking ring; and
   locking means having external threads adapted to engage said internally threaded upper supporting ring for securing said reference standard holding means against accidental rotation.

References Cited

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