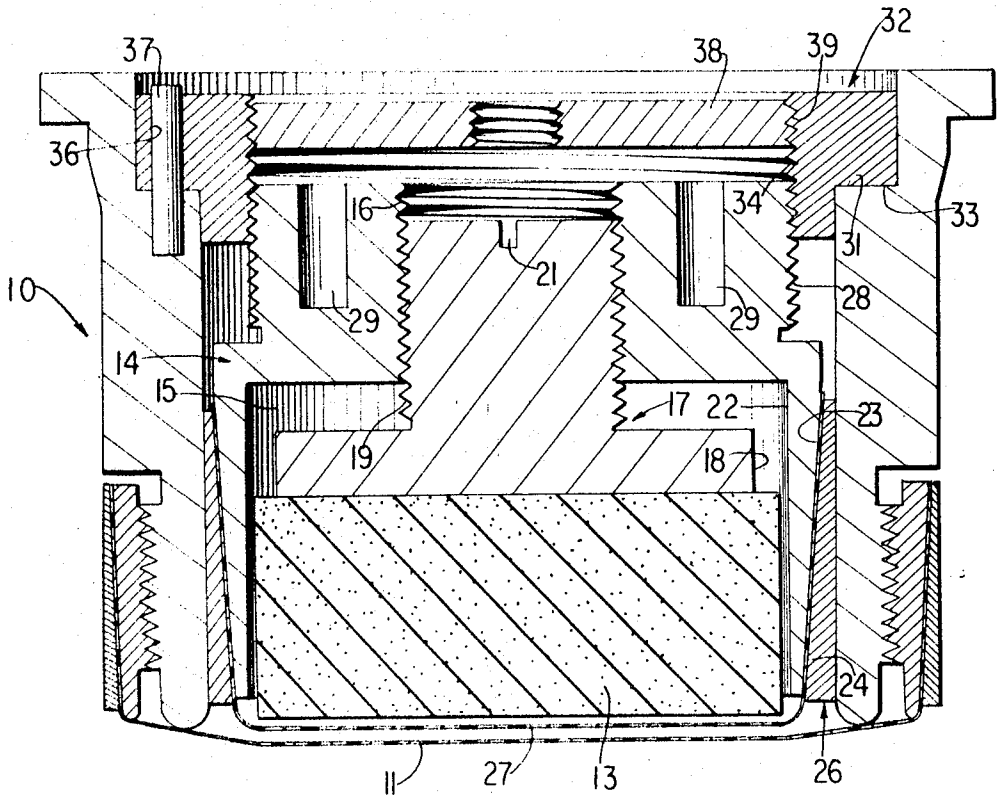
Aug. 19, 1969 W. E. BURKE ET AL 3,462,598

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

Filed Nov. 15, 1965



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United States Patent Office 3,462,598
Patented Aug. 19, 1969

3,462,598

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

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Filed Nov. 15, 1965, Ser. No. 507,918
Int. Cl. G01n 23/20
U.S. Cl. 250—51.5 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 internally threaded supporting ring in which is adjustably mounted an externally threaded reference standard holding means. The reference standard holder has a pellet or reference standard mounted in a support which is threadably adjustable and lockable within the holding means, and a plastic film fitted over the lower portion of the holding means by a locking ring so as to seal the reference standard from the sample cup for use in X-ray spectrography. The reference standard is vertically adjusted in the holding means, locked in a position and then the holding means is threadably 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 enhancement effects of all elements (including the element sought) and the sample being analyzed. Among the prior art procedures that have been utilized in order to compensate for these matrix absorption effects are direct comparison with a standard, dilution technique, and 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 quantitative analyses. The internal standard procedures of the prior art include the use of background lines as references and the physical addition of a suitably chosen element to the sample being analyzed. The use of background lines has not been too successful. Physical addition of a suitably chosen element as part of the sample subjected to X-ray spectrography and which thereby acts as an internal reference standard has been most successful when the added element's absorption edge and emission line has been very close to that of the element sought. Most internal standard procedures, however, have not completely compensated for matrix absorption effects because of the inability to isolate the added internal reference standard from physical contact and contamination by the test sample and the difficulty of mechanically obtaining and retaining the required accuracies in test sample density and thickness.

Matrix absorption affects 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

effected 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 holder which will facilitate the X-ray spectrographic determination of elements in hydrocarbons and other matrices.

It is still yet another object of our invention to provide a device employing 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 **10** suitable for use in an X-ray spectrograph, such as the Philips inverted sample, three-position X-ray spectrograph. A sample cup **10** 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 **10** 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 spectrography 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 screwably 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.

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