Nov. 11, 1969 R. GOERKE ET AL 3,478,207

SAMPLE CARRIER FOR X-RAY SPECTROMETERS AND THE LIKE

Filed Aug. 15, 1966 3 Sheets-Sheet 1

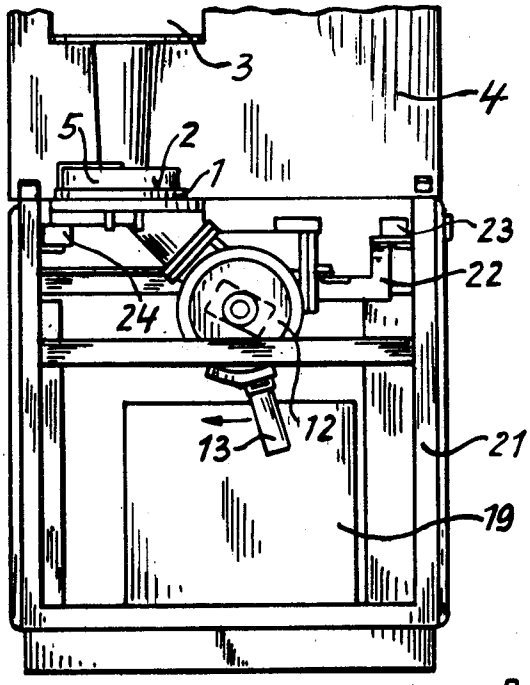

Fig 1

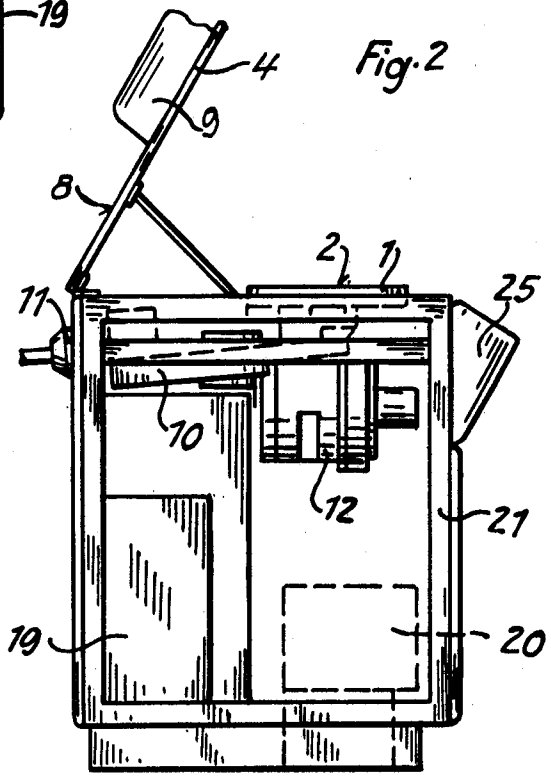

Fig. 2

INVENTORS
Rolf Goerke, Helmut Bisch,
Kurt Tögel & Werner Speck
BY
Lee & Lee
ATTYS.

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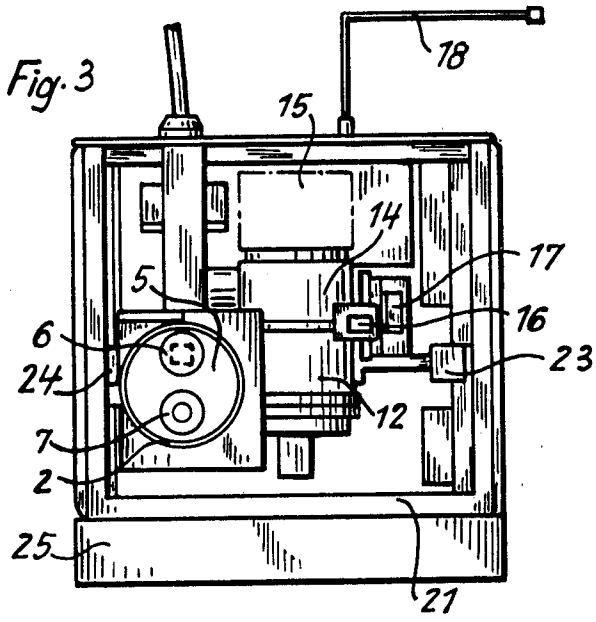
Fig. 3



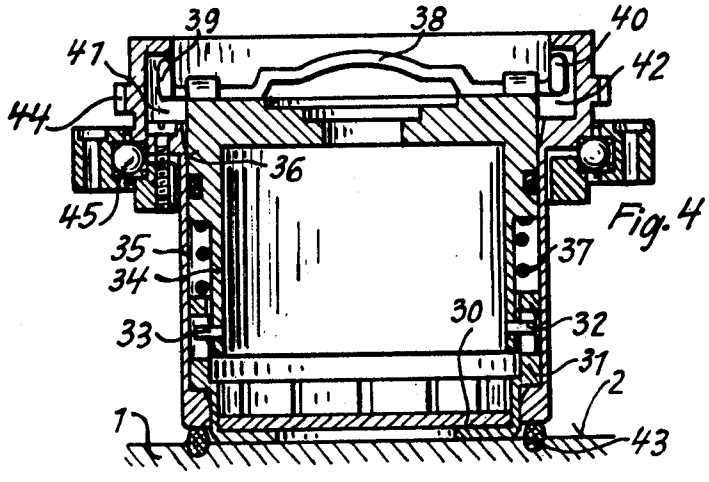


Fig. 4

INVENTORS
Rolf Goerke, Helmut Bisch,
Kurt Tögel & Werner Speck
BY Lee & Lee
ATTYS.

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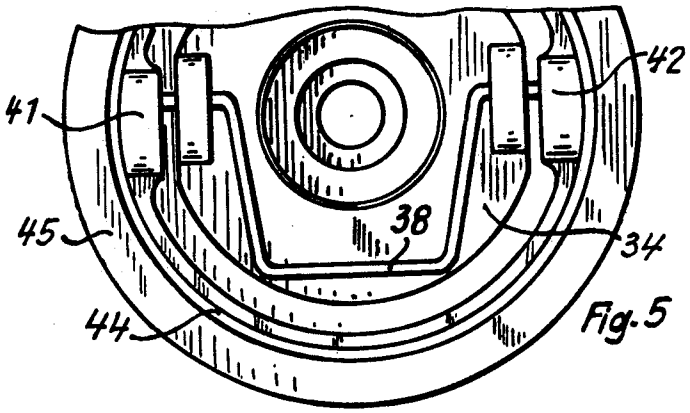


Fig. 5

Fig. 6

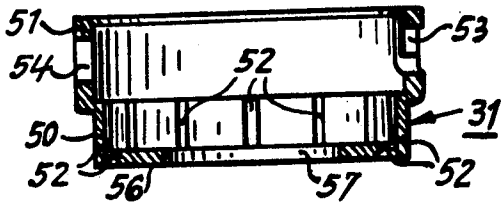


Fig. 7

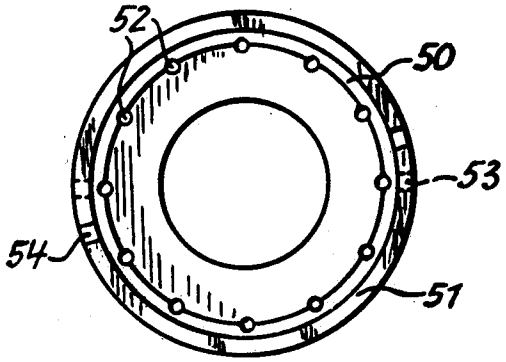
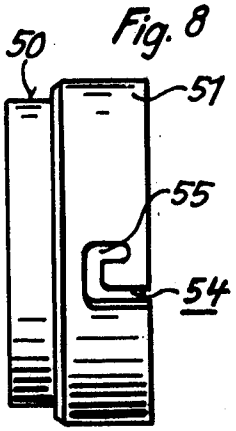


Fig. 8



INVENTORS
Rolf Goerke, Helmut Bisch,
Kurt Tögel & Werner Speck
BY Lee & Lee
ATTYS.

United States Patent Office 3,478,207 Patented Nov. 11, 1969

3,478,207

SAMPLE CARRIER FOR X-RAY SPECTROMETERS AND THE LIKE

Rolf Goerke, Langensteinbach, and Helmut Bisch and Kurt Tögel, Karlsruhe, and Werner Speck, Bietigheim, Germany, assignors to Siemens Aktiengesellschaft, a corporation of Germany

Filed Aug. 15, 1966, Ser. No. 572,289
Claims priority, application Germany, Sept. 16, 1965, S 99,455
Int. Cl. H01j 37/20
U.S. Cl. 250—51.5 2 Claims

ABSTRACT OF THE DISCLOSURE

The invention relates to a sample carrier for an X-ray spectrometer accommodated in a housing. The cover of the housing is designed as a table top whereby the surface of the top is aligned with a sample adherence surface of a sample adherence plate arranged in a recess in the table top. Sample holders for solid and/or pulverulent and/or liquid samples are fastened to the support surface of the sample adherence plate. Each of the sample holders is provided with a cup-like part to hold the sample which is pressed against the adherence surface of the sample adherence plate, the bottom of the cup-like part being as thin as possible.

The invention relates to a sample carrier for X-ray spectrometers containing an X-ray tube as well as X-ray radiation detectors. Known apparatuses of this type are, in view of the usual construction of the sample carrier, restricted to the investigation of relatively small samples, which are arranged in special sample holders. Solid samples, pulverized samples or liquids may be so involved. Frequently there also is the desire to examine larger objects by X-ray spectrometry, i.e., parts which cannot be directly placed into the sample holders referred to. In the known spectrometer constructions it is then necessary to remove a small amount from the part to be investigated, to put it in a sample holder and thereupon to investigate it by X-ray spectrometry in the usual manner. Aside from the additional expenditure of time which this procedure requires, such a process of investigation has the fundamental drawback that, strictly speaking it permits no destruction-free X-ray spectrometric examination of large samples.

There exists, therefore, a need for a sample carrier for X-ray spectrometers which is universally usable both for small and for large samples, in particular, so large that they cannot be placed in the conventional sample holder. The further requirement may here arise that, in view of the form of the large sample present, movement of the essential parts of the spectrometer must be provided, for example, of the X-ray tube and of the detectors, whereby in each case the area of interest of the sample can be examined by X-ray spectrometry regardless of the shape of the same.

A sample carrier possessing such a universal application for X-ray spectrometers containing an X-ray tube and X-ray radiation detectors is characterized according to the invention by the feature that a sample supporting plate, provided with openings for the passage of the X-ray radiation, is arranged in an opening of the cover member of the spectrometer, which is constructed as a table plate. The sample supporting surface of said sample plate is disposed in the plane of the table surface and the X-ray tube focused approximately on this plane with the radiation detectors disposed on the side of the sample supporting plate facing away from the supporting surface.

A sample carrier according to the invention, therefore, is not arranged somewhere inside the X-ray spectrometer, but forms the uppermost part of the same, and in particular, in such an arrangement that the sample supporting plate forming an essential part of the sample carrier, together with the table surface aligned with the sample supporting surface of the sample supporting plate, provide a common support for large samples. In those cases in which relatively small samples are to be examined in the ordinary manner, the sample can be placed in a sample holder. For such investigations the sample carrier according to the invention receives sample holders which are mounted on the supporting surface of the sample supporting plate for solid, pulverized or liquid samples, with the samples being so supported that their impact surface for the exciting X-ray radiation extends parallel to the supporting surface of the sample supporting plate a distance as small as possible from the supporting surface. Thereby it is assured that in all cases the samples lie practically in the same plane.

In a preferred form of construction of the sample carrier according to the invention, the sample supporting plate as well as the parts of the spectrometer mechanical inter-connected therewith, for example the X-ray tube, are carried in a pivotally supported frame or carrier. This form of construction offers the advantage that a matching of the apparatus to the form of the particular sample involved is possible in a simple manner, in particular, the position of the X-ray tube and of the detectors. The frame or carrier can be suspended by means of two pivot bearing pins whose common axis extends below the supporting surface of the sample supporting plate, within the housing of the spectrometer.

There is thus achieved a pivotability of the supporting surface with the passage openings contained therein for the primary and secondary X-ray radiation. The swinging of the frame or carrier, with the elements carried thereby, expediently is effected by means of an electric motor drive, which is controlled from a suitable switchboard, which may carry all the switches utilized in the operation of the spectrometer.

It has already been pointed out that the sample carrier according to the invention permits not only the investigation of samples so large that they cannot be placed in the usual type of sample carrier, but that it is also possible to carry out the familiar examination processes making use of sample holders. It is necessary in this case to care that the impact surfaces of the sample disposed in the sample holder for the primary X-ray radiation is, insofar as possible, identical with the plane that is formed by the surface of the supporting plate of the sample carrier and the table surface, since the measured intensity of the X-ray radiation depends upon the position of this plane with respect to the X-ray tube. These points must be taken into account in the construction of a cooperable sample carrier. In a preferred form of construction of the sample holder used with the sample carrier according to the invention, such holder has a cup-shaped part for the reception of the sample, which part has a surface with as little thickness as possible, which is pressed against the supporting surface of the sample supporting plate. The distance between the surface to be investigated of the particular sample, on the one hand, and the supporting surface of the supporting plate of the sample carrier, on the other hand, is thus determined solely by the defined thickness of the base surface of the cup-shaped part of the sample holder. It is important, therefore, that this surface have in all sample holders the same defined thickness, this thickness being as small as possible in order to bring the planes mentioned as close as possible to one another.

The base surface of the cup-shaped part understandably will be made of a material with such long wave excited secondary X-ray radiation that such secondary radiation lies outside the measuring range of the spectrometer. It thereby is assured that no alien lines derived from the material of the base surface of the cup-shaped part can falsify the analysis. As a material for the base surface of the cup-shaped part, graphite of highest purity has proved successful. Theoretically one could also employ beryllium, but in many cases this material may be excluded for reasons of cost.

It is especially expedient that the base surface of the cup-shaped part be pressed against the supporting surface under spring action, because there is then assured a full and firm support of the base surface of the cup-shaped part on the surface of the supporting plate and thereby also maintain a good parallelism of the surface of the sample charged with the primary radiation relative to the supporting surface. It is therefore particularly desirable that the cup-shaped part be pressed against the supporting surface, insofar as possible, solely under the action of the spring. For this purpose the cup-shaped part can be suitably suspended in a retaining part with sufficient play that its base surface is permitted to lie on the supporting surface. Further, the retaining part can be removably mounted in a cylinder which also loosely surrounds the cup-shaped part. The cup-shaped part is therefore loosely suspended in the cylinder below the inserted retaining part and under the action of a spring, which acts, for example, as a compression spring between the retaining part and the suspended cup-like part, urges the base surface of the latter against the surface of the supporting plate.

To take into account inhomogeneities of the sample in the measuring, or to produce a mean value, it is desirable to provide for the possibility of turning the sample. For this purpose, in a preferred form of construction of the invention, the cylinder is provided with rotating drive means which can contain an electric motor and corresponding gearing. The retaining part, expediently, is in the form of a cover for the cup-like part and carries externally extending means by which the cup-like part may be inserted in the cylinder. It is, therefore, also constructed for insertion into the cylinder. If the sample involved is an active liquid, care must be taken that in the removal of the cup-shaped part from the cylinder no splashing of the liquid takes place In order to avoid a jerky release of the retaining part from the cylinder, the latter expediently has a conical surface for the insertion of the holding part. For the same purpose a further devleopment of the invention also proves favorable, in which the retaining part carries a bail with eccentrically disposed portions, preferably end portions, which so interact with projections on the cylinder that, depending on the position of the bail, they lock the retaining part in the cylinder or push it slightly out of the cylinder. The locking action is effective, for example, when the bail is shifted to a final position, while the pressing out of the retaining part from the cylinder proceeds automatically when the bail is pivoted for the purpose of removing or changing the sample.

The sample carrier according to the invention also can be constructed with the retention of the essential feature of the supporting surface of the supporting plate being aligned with the table surface of the X-ray spectrometer in such a way that several sample holders can be brought, at will, into examination position. In a corresponding example of construction several sample holders are installed in a drum, preferably constructed as a sample chamber or lock, which is so rotatable on an axis that the samples in the individual sample holders can be swung, at will, into the range of the passage apertures for the X-ray radiation in the sample supporting surface. If the sample holders have cup-like parts as described, then the base surfaces of such parts of all the sample

holders, in the swinging movement, slide on the surface of the sample supporting plate. There, in the supporting surface of the sample supporting plate at the points in which the sample holders are located during the sample change, seals can be utilized, so that in the sample change only the part of the vacuum present in the particular sample holder is destroyed.

The opening in the table plate serving for the reception of the sample supporting plate is covered with a radiation protective cap, which can be hingedly attached to the table plate, in order to permit simple access to the supporting surface and to the drum, if any, carrying the various sample holders.

The invention is explained in the following, with the aid of the examples of construction illustrated in the drawings, wherein like reference characters indicate like or corresponding parts, and in which:

FIG. 1 is a front elevation of an X-ray spectrometer embodying the invention;

FIG. 2 is a side elevation of the spectrometer illustrated in FIG. 1;

FIG. 3 is a plan view of the spectrometer illustrated in FIGS. 2 and 3, with the cover removed;

FIG. 4 is a transverse sectional view of a sample holder;

FIG. 5 is a top plan view of the sample holder of FIG. 4, a portion of which is broken away;

FIG. 6 is a transverse sectional view of a cup-like part of the sample holder, in which the sample is disposed;

FIG. 7 is a plan view of the cup-like part; and

FIG. 8 is an elevational view of the cup-like part, illustrating details of a bayonet slot therein.

Referring to FIGS. 1 to 3, the X-ray spectrometer illustrated is equipped with a sample carrier according to the invention, the housing walls of the spectrometer being removed and the table plate illustrated in raised position. According to the invention a sample supporting plate **1** having a supporting surface **2** for the samples is arranged in an opening **3** in the cover **4** of the apparatus, which cover is so constructed as a table plate that, from the drum **5** disposed on it and provided in this example of construction with two sample holders **6** and **7**, it forms the uppermost part of the entire spectrometer. The construction is such that the supporting surface **2** lies in a common plane with the table surface **8** of the cover **4** of the apparatus. It is thereby achieved that large samples can be examined by simply placing them on the apparatus, utilizing both the supporting surface **2** and the table surface **8** of the apparatus.

As illustrated in FIG. 2, for reasons of radiation protection, a cover cap **9** may be provided, which is so shaped that, with drum **5** in position, it covers the opening **3** in the table plate **4**.

The drum **5** is detachably fastened to the receiving plate **1**, so that as illustrated in FIG. 2, it can be removed or be replaced by individual sample holders.

On the side of supporting plate **1**, facing away from the supporting surface **2**, there are located the essential parts of the spectrometer, such as the X-ray tube **11**, mounted in the tube clamp **10**, the crystal changer **12** and the scintillation counter tube **13**, which, as is evident from the arrow in FIG. 1, is pivotally arranged under the crystal changer **12**. In FIG. 3 reference numeral **14** designates a motor with suitable gearing, which is provided for the entire counter tube drive. The counting tube pulses are amplified in the amplifier **15**, and the angle reading takes place at the reading device **16**, while reference numeral **17** designates a coupling provided for manual drvie. The high voltage cable bears the reference number **18**.

The vacuum apparatus **19** and a device **20** for pressure regulation and possibly also for temperature regulation are not illustrated in detail, since the details thereof form no part of the invention.

The apparatus thus offers the possibility of examining, at will, samples which, in consequence of their small size,

can be arranged in sample holders or else, in consequence of their magnitude and form, must be examined without the use of a sample holder. The configuration of the large samples may frequently make it necessary to swing the sample supporting plate **1**, with the passage openings therein for the primary and secondary X-ray radiation, out of the plane of the table surface **8**. For this purpose the primary parts of the spectrometer are not mounted rigidly in the frame **21** of the apparatus, but are supported on the carrier **22**, which in turn, by means of pivot bearings **23** and **24**, is swingably mounted in the frame **21**. It thereby becomes possible to adapt the position of the supporting surface **2** to the sample to be investigated in each case without difficulties arising with respect to the mechanical connections between the sample supporting plate **1**, on the one hand, and the primary parts of the spectrometer, on the other hand, namely the X-ray tube **11**, the crystal changer **12** and the detector **13**.

The drive of the drum **5**, equipped with several sample holders **6**, **7**, can be accomplished in a manner in itself known to those skilled in the art, with use of an electric motor drive, including suitable gearing. The control of this drive may be accomplished from the switchboard **25**, on which there are expediently arranged all the operating elements for the X-ray spectrometer, including the necessary control lamps.

In changing the particular sample located in the examination position by swinging of drum **5**, the parts of the sample holders directly holding the sample slide on the supporting surface **2** of the sample supporting plate **1**, utilizing a construction of sample holder, as hereinafter explained in connection with FIGS. 4 and 5. It will be assumed that a disk-shaped sample **30** is involved, which is held in the cup-like part **31**. As is subsequently explained in connection with FIGS. 6 to 8, the cup-like part **31** is provided with recesses into which pins **32** and **33** of the retaining part **34**, designed in the manner of a cover, so engage that the cup-like part **31** depends from the retaining part **34**. The retaining part **34**, in turn, is inserted in the cylinder **35** at the upper end **36** thereof, which also surrounds the suspended cup-like part **31**. The compression spring **37**, with utilization of suitable play in the suspension, including the pivots **32** and **33**, the cup-like part **31** may be disposed with its base surface bearing upon the supporting surface **2** of the sample supporting plate **1**. It is thereby assured that the surface of the sample **10**, facing the supporting surface **2**, is disposed virtually in the same plane as the surface **2**, and at least extends parallel thereto. The base surface of the cup-like part **31** is therefore constructed with a minimum thickness. It consists of a material which emits such mildly excited X-ray radiation that the analysis of the sample **30** is not influenced.

On its upper end the retaining part **34** carries a bail **38** having eccentric portions **39** and **40**, which are cooperable with the lugs **41** and **42** on cylinder **35** in such a way that in the thrown-over position illustrated of bail **38** they assure a locking of the retaining part **34** and thereby retention of the sample **30** within the cylinder **35**, while upon upward pivotal movement of the bail for the purpose of removing the retaining part **34** and thereby also of sample **30** from the interior of the cylinder, by an abutting action on the lower surfaces of the lugs **41** and **42** they effect a gentle upward movement of the retaining part **34** outwardly with respect to the cylinder **35**. To facilitate insertion of the part **34** and associated part **31**, the cylinder preferably is conically constructed on its upper inner surface in the manner illustrated. This construction of the bail **38**, in conjunction with the lugs **41** and **42** on the cylinder **35**, not only assures a tight vacuum closure, but also prevents the removal of the sample **30** from the interior of the cylinder, i.e., from the sample holder, from taking place jerkily, which can be particularly dangerous with highly active liquids or powders as samples.

So that the vacuum of the apparatus is not disturbed or is disturbed as little as possible in changing of the sample, there can be provided a packing seal **43** at the location on the supporting surface **2** of the supporting plate **1** at which the sample holders, i.e., the cylinders **35** are disposed in the changing of sample **30**. It is sufficiently recessed into the plate **1** that it permits the swinging of the drum **5** (not illustrated in FIGS. 4 and 5) for the purpose of changing the particular sample disposed in examining position.

In the example of construction illustrated in FIGS. 4 and 5 there is further provided means for enabling rotation of the sample for the purpose of taking into account any inhomogeneity of the same in the examination. For this purpose, the cylinder **35** carries, adjacent its projections **40** and **41**, a toothed rim **44** which is to be connected with a gear driven by an electric motor (not illustrated). For the facilitation of the rotary movement, i.e., for the reduction of friction, the sample holder is supported in the drum **5** (not illustrated in FIGS. 4 and 5) by means of a ball bearing **45**.

FIGS. 6, 7 and 8 illustrate details of the cup-like part **31** which receives the sample **30**. It is composed of two portions **50** and **51**, of which the portion **50** is provided internally with ventilation bores **52** and the portion **51**, the upper portion in the figure, provides for the suspension of the cup-like part **31** on the pins **32** and **33** of the retaining part **34** (FIG. 4). For this purpose the upper portion **51** has two recesses **53** and **54** disposed diametrically opposite one another, which are designed, for example, in hook-like form, so that with the cooperable pins **32** and **33** on the retaining part **34** they form an interlocking bayonet-type connection. By appropriate dimensioning of the closed end **55** of the two recesses **53** and **54** sufficient play is provided in the suspension whereby the cup-like part **31** is firmly pressed, under the influence of the compression spring **37** (see FIG. 4) against the supporting surface **2** of the plate **1**. During the measuring operation, therefore, this suspension is effective to limit movement of the part **31** to the plane of the supporting surface **2**, while the cup-like part **31** remains perpendicularly thereto exclusively under the influence of the compression spring **37** and the supporting surface **2**.

As is apparent from FIG. 6, the base surface **56** of the cup-like part **31** bearing on the supporting surface **2** is not a closed surface, but has an opening **57** in its central region for passage of the primary and secondary X-ray radiation. For the improvement of the precision of the analysis it is appropriate, irrespective thereof, to form the base surface **56** of a material whose excited secondary X-ray radiation lies outside the measuring range of the X-ray spectrometer.

By means of the invention there is produced a sample carrier for X-ray spectrometers which makes it possible to examine samples of any size and any shape. By means of the sample receiving plate with the sample receiving surface, arranged in the particular manner above the other parts of the apparatus, all samples can be brought into a reproducible position, independently of their size and form. The sample carrier is universally usable inasmuch as it permits both the examination of large samples and also the spectroscopy of smaller samples which are disposed in sample holders. Many modifications of the examples of construction described are possible. For example, the sample holders can be constructed in a different manner. In those cases in which samples of liquid materials are to be investigated, the cup-like parts may be provided with a continuous base and thereby comprise actual cups. For the investigation of powders or liquids it is possible to lay a plastic foil in the cup-shaped part represented in the figures, which is supported by a metal foil with a perforation of high permeability to radiation and consists of a material which will not disturb the analysis. The difference between the position of samples which are investigated without use of a holder, and of those samples which are supported in a holder, as deter-

mined by the selected thickness of the base surface of the cup-like part, is of no importance, as there is always present a constant measuring tolerance in consequence of the selection of a defined thickness of the base surface of the cup-like part. It is not necessary that the sample reception surface and table surface lie horizontal.

Changes may be made within the scope and spirit of the appended claims which define what is believed to be new and desired to have protected by Letters Patent.

We claim:

1. A sample carrier for an X-ray spectrometer and the like comprising: a substantially tubular housing having a circumferential inturned lip around the bottom thereof, a cup-shaped sample receiving member receivable in said housing having a circumferential outturned lip around the top thereof adapted to mate with the lip on the housing to retain the said member in the said housing with the bottom of the said member extending beyond the bottom of the said housing, a retaining part adapted to retain samples in the said cup-shaped part dimensioned to be received within the said housing, the said retaining part having a reduced diameter section adapted to be received within the top of the said cup-shaped member and a larger diameter portion spaced from the bottom of the said retaining part adapted to mate with the inner diameter of the said housing, lugs extending radially outwardly adjacent the bottom of the said retaining part, bayonet slot means in the said cup-shaped member adapted to mate with the said lugs to depend the said cup-shaped

member from the said retaining part, spring means between the top of the said cup-shaped part and the said increased diameter portion of the said retaining part, bail means operatively associated with the said retaining part and the said housing to urge the said retaining part towards the bottom of the said housing compressing the said spring means, said spring means effective to urge the outturned lip of the said cup-shaped member against the inturned lip of the said housing thereby projecting the bottom of the said cup-shaped member beyond the bottom of the said housing, the bottom of the said cup-shaped member being thin.

2. The sample carrier of claim 1 wherein means are provided for rotating the said housing.

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WILLIAM F. LINDQUIST, Primary Examiner

U.S. Cl. X.R.

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