

Sept. 26, 1967 W. D. ASHBY ETAL 3,344,274
X-RAY ANALYSIS APPARATUS HAVING BOTH DIFFRACTION AND
SPECTROMETER TUBES MOUNTED ON A COMMON HOUSING
Filed Dec. 6, 1963 3 Sheets-Sheet 1

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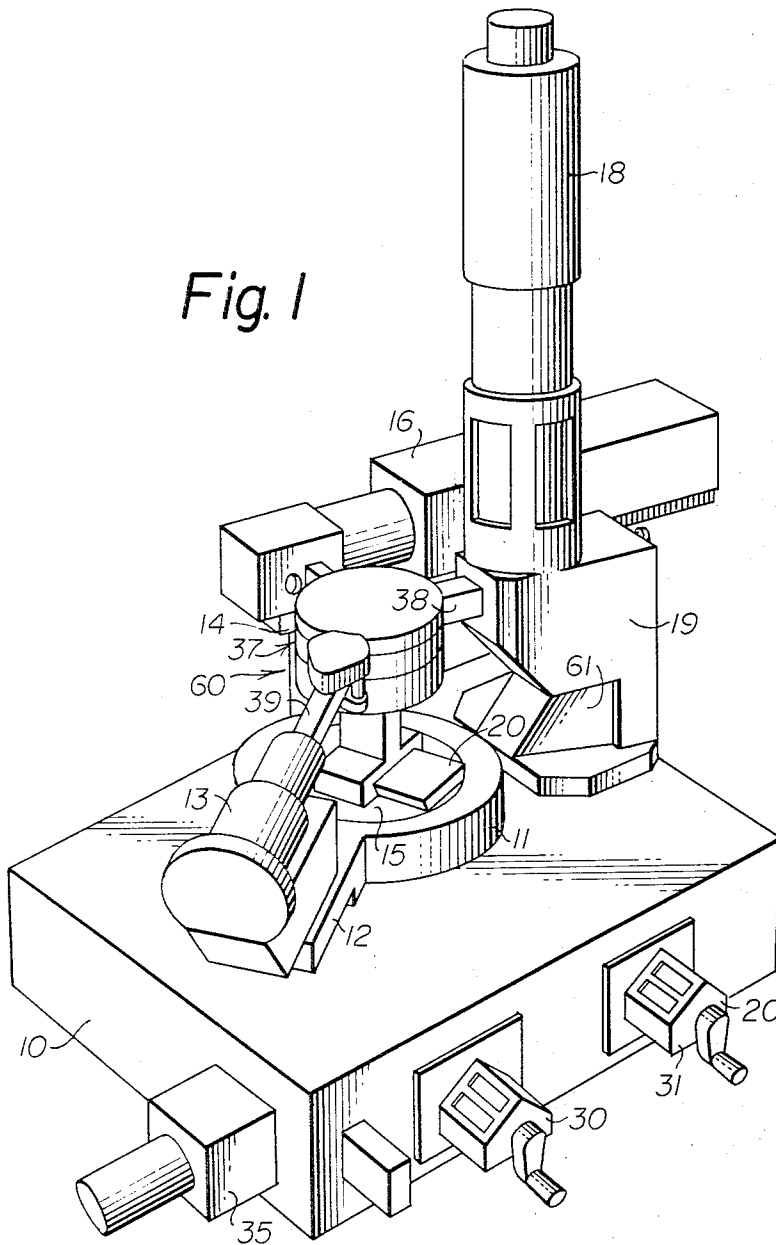
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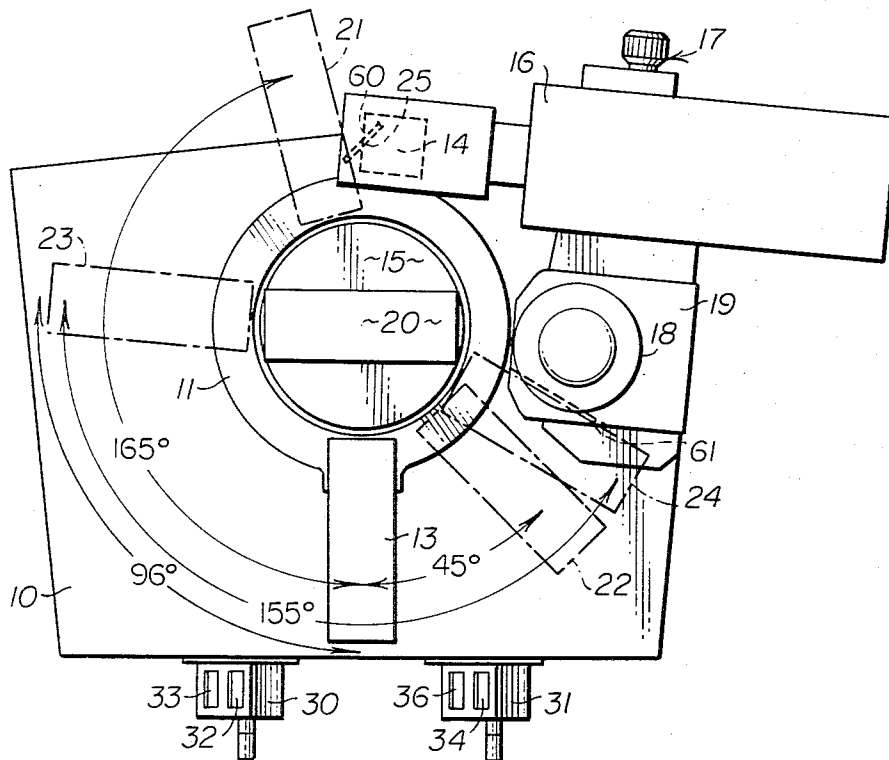


Fig. 2

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Fig. 3

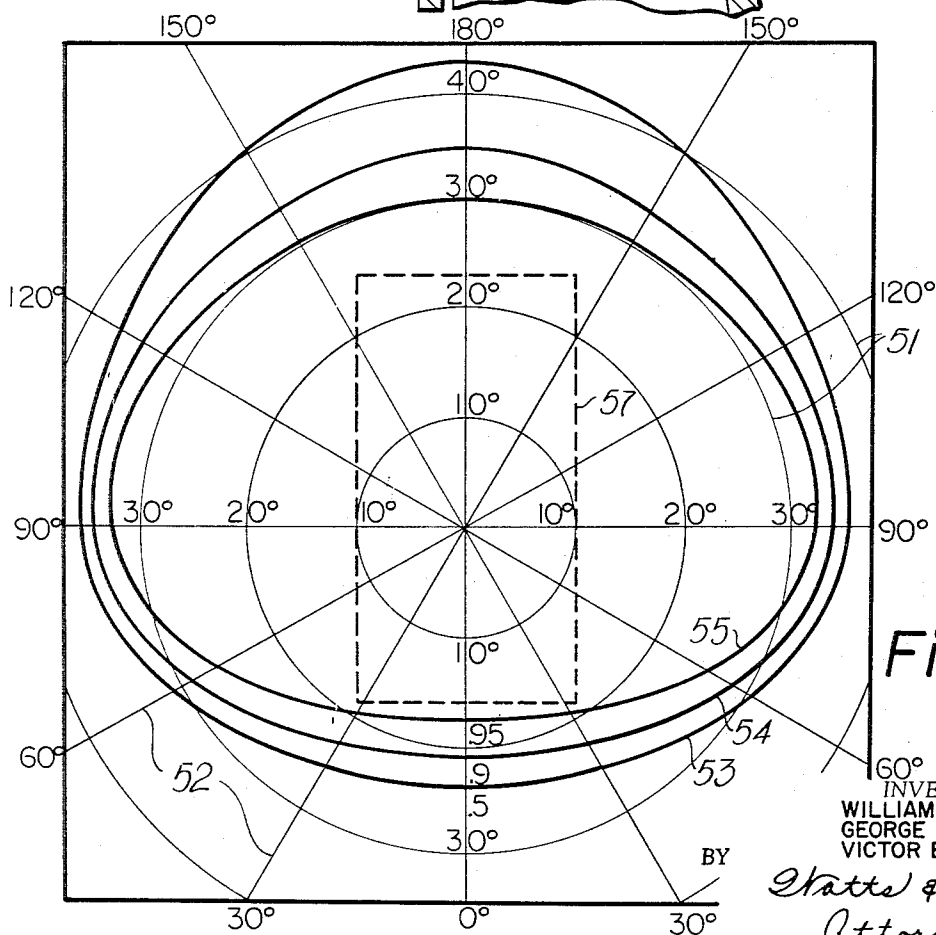
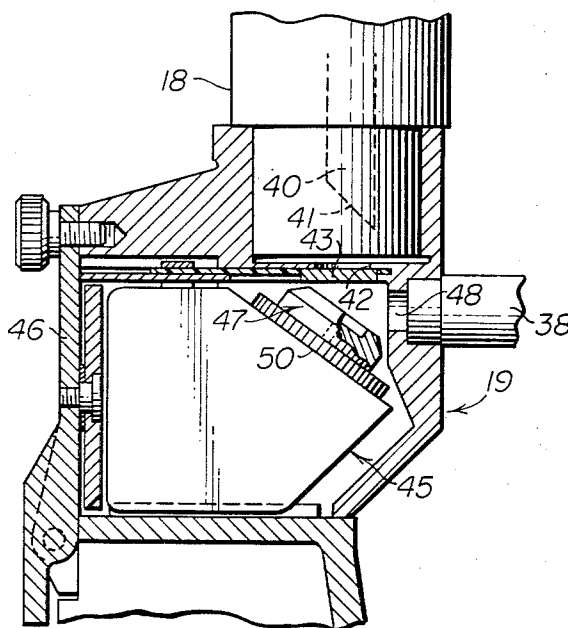


Fig. 4

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X-RAY ANALYSIS APPARATUS HAVING BOTH DIFFRACTION AND SPECTROMETER TUBES MOUNTED ON A COMMON HOUSING

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Filed Dec. 6, 1963, Ser. No. 328,539
14 Claims. (Cl. 250—51.5)

This invention relates to X-ray apparatus and more particularly to an X-ray apparatus suitable for conducting non-destructive X-ray analysis.

An X-ray diffractometer is a device in which a specimen is rotated about an axis through an angle theta or omega. To achieve this the specimen is mounted on a rotatable member known alternatively as a theta or an omega member. An X-ray tube is positioned to bombard the specimen as it is rotated. A detector is mounted on a so-called "two-theta arm" for rotation about the same axis at twice the speed of the omega member. The detector is used to detect and record X-radiation diffracted by the specimen providing information as to physical and chemical characteristics of the specimen. One X-ray diffractometer is described and claimed in co-pending application for patent, Ser. No. 236,468, filed Nov. 2, 1962, by T. C. Furnas, Jr. and entitled Diffractometer, now patent 3,218,458, issued Nov. 16, 1965.

Another type of non-destructive X-ray analysis of physical specimens is conducted through a technique known as "spectroscopy." A spectroscopic study differs from a diffraction study in that the specimen itself is excited to emit its own characteristic X-rays. Excitation of the specimen is usually accomplished by bathing the specimen in the radiation from an X-ray tube, usually of a different type than the X-ray tube used for diffraction. The apparatus used for spectroscopy is quite similar to that used for diffraction and, in fact, a spectrometer is often a modified diffractometer. The principal differences between a diffractometer and a spectrometer are (1) a single crystal known as an analyzing crystal, rather than the specimen, is positioned on the omega member for rotation; and, (2) the specimen is positioned to emit its own radiation toward the analyzing crystal and, therefore, a differently located X-ray tube is required.

In the past, spectroscopic studies have been conducted on mechanisms which are basically diffractometers. Before a spectroscopic study could be conducted on a diffractometer, it has always been necessary to remove the diffraction X-ray tube. It was also necessary to remove mounting brackets, supports, etc., and replace these with ones of different configuration and construction. With these prior devices the spectrometer specimen is positioned at the location where the focal spot of the diffraction X-ray tube was located. A spectrometer X-ray tube is then appropriately positioned to excite this specimen and cause it to emit its own characteristic X-rays in a direction which will bombard the crystal mounted on the theta or omega member.

Alignment of the X-ray tubes and specimens for both diffraction and spectroscopic studies is quite delicate and difficult. Even with the novel and improved diffraction tube support and adjustment described and claimed in the above-referenced patent of T. C. Furnas, Jr., considerable time is required to precisely align a diffraction tube for diffraction studies. Accordingly, once the diffraction tube is properly aligned, it is undesirable to move it. But, as noted above, with prior devices it was essential to move the diffraction tube and supports if a spectroscopic study was to be conducted with the same unit.

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The present invention has overcome this alignment and other problems through the provision of a mechanism which serves as either a diffractometer or spectrometer without disturbing any of the precision alignments required for either type of study. With this device, an X-ray tube for diffraction and a second X-ray tube for spectrometry are both coupled to a single housing. The coupling of the respective X-ray tubes to the housing while permitting adjustment for alignment and the like is permanent so that the unit can be moved from place to place and diffraction and spectroscopic studies can be alternately conducted without disturbing any of the precision alignments.

With this invention, precision, concentrically rotatable, theta and two-theta members are provided. The members are selectively and alternately rotatable one at a time through theta and two-theta ranges for either diffraction or spectrometry. The ranges for the diffraction and spectrometry studies overlap. Through the unique construction of this unit the ranges equal or exceed ranges previously obtained with units designed to be used only as diffractometers or diffractometers and spectrometers only by moving or removing the diffraction tube. Thus, even though one may conduct either a diffraction or a spectroscopic study with this unit without moving or removing any X-ray tube, the range of either type of study equals or exceeds that available in the past.

In both diffraction and spectroscopic studies, persons skilled in the art refer to a two-theta range. This is the angular range through which a detector can be rotated as a study is conducted. So-called "zero degree two-theta" is the position when, in the case of diffraction, the focal spot of the X-ray tube, the specimen, and the detector, are located along a straight line and the line is the axis of the beam of X-rays emitted by the diffraction tube. In spectrometry, zero degree two-theta is a position where the specimen, the crystal, and the detector are along a straight line.

In diffraction, on the so-called "positive side," pertinent diffraction effects begin anywhere from zero degrees to about thirty degrees, depending upon the information sought and the nature of the sample, and radiation. For so-called "routine studies," at least one hundred twenty degrees is required. For precision, it is desirable to approach zero error in d values; i.e., measurement of interatomic distances. To achieve this, a diffractometer should approach, as closely as possible, a one hundred eighty degree two-theta position where zero error in d values is theoretically obtainable. Thus, on the positive side, zero to thirty or forty degrees is minimum, zero to one hundred twenty degrees is required for routine studies, and zero to one hundred eighty degrees is theoretically perfect. With this invention the zero to one hundred sixty-five degree range is obtained.

On the negative side, since pertinent diffraction effects can begin to occur at about thirty degrees, it is desirable to be able to rotate the detector through a zero degree to minus thirty or forty degree two-theta range in order to check the accuracy of the location of the zero degree two-theta position. With the present invention, the diffraction range is from minus forty-five to plus one hundred sixty five degrees and, accordingly, from the foregoing discussion of desired ranges, it will be seen that a full range equal to or exceeding anything in the prior art is obtained even though the unit is also a spectrometer.

In the case of spectroscopy, there is no requirement for negative ranges. With the most efficient crystals, one would wish to have approximately one hundred fifty degrees on the positive side and must have at least eighty-five degrees to obtain usable information. Accordingly, it will be

seen that with the present invention the provided range of zero to one hundred fifty-five degrees meets or exceeds the scientist's requirements and moreover equals or exceeds that obtainable with prior art structures.

To obtain the described and desired ranges of diffraction and spectroscopy studies, the diffraction and spectroscopy tubes are positioned near one another on the housing. The tubes are positioned such that the zero degree two-theta lines for the respective studies are generally normal to one another and the two two-theta ranges overlap one another. The positive direction of two-theta rotation for each type of study is in the opposite direction from the other study. Thus, in the preferred construction, rotation from the zero degree two-theta position in a positive direction for spectroscopy is counterclockwise while the rotation from the zero degree two-theta position in a positive direction for diffraction is clockwise. With this arrangement, a single device is provided with two tubes permanently mounted and with the device an operator may selectively and one at a time conduct spectroscopic and diffraction studies.

With this invention, improved spectroscopic results are obtained through the provision of a spectroscopic X-ray tube positioned such that its target is substantially parallel to the specimen to be studied. The two are positioned as closely together as is physically possible and appropriately positioned so that a field of maximum intensity of the desired size is obtained.

As an example, with the standard specimen support of one and one quarter inches in diameter, it is desirable to have a field size of approximately one half inch square. To achieve this, the specimen is positioned at an angle with respect to the axis of crystal rotation and preferably at the minimum angle which will produce this one half inch square field size. With an X-ray tube having a target surface at forty-five degrees with the two-theta axis, this minimum angle is thirty-six degrees. At this angle, a minimum of interference between the specimen support and the X-ray tube is obtained so that the specimen can be positioned as closely as possible to the target of the X-ray tube. In the disclosed and preferred arrangement, an end window spectroscopy tube is used which has a target surface at an angle of about forty-five degrees. Thus, the plane of the target and the plane of the specimen are substantially parallel with one another so that substantially uniform X-ray of a high intensity is obtained over the entire specimen.

Another of the features of this invention is that the specimen holder is so positioned that liquid specimens can be used with the specimen holder without encapsulation of the specimen and this, in the past, has not been obtainable.

Another feature of this invention resides in odometers which provide two readings. Omega and two-theta odometers are provided each of which provides independent readings of both diffraction and spectroscopic positions. This facilitates, for example, moving the two-theta member from its diffraction zero degree position to its spectroscopic zero degree position since the respective positions are indicated on different lines of the same odometer.

Accordingly, the principal objects of this invention are to provide a novel and improved spectro-diffractometer, a novel and improved specimen support for a spectrometer, and a novel and improved apparatus for exciting a spectroscopic specimen.

Other objects and a fuller understanding of the invention may be had by referring to the following description and claims taken in conjunction with the accompanying drawings in which:

In the drawings:

FIGURE 1 is a perspective view of the spectrometer of this invention;

FIGURE 2 is a top plan view of the structure of FIGURE 1 on a slightly enlarged scale;

FIGURE 3 is a fragmentary sectional view on an enlarged scale with respect to FIGURES 1 and 2 of the spectroscopic specimen support and holder and of the spectroscopic tube; and,

FIGURE 4 is a graph showing intensity distribution of the X-rays emitted by the spectroscopic tube.

Referring now to the drawings and FIGURES 1 and 2 in particular, a main housing is shown at 10. The main housing 10 and the mechanism encased within it are preferably that described and claimed in the above-referenced patent application for a Diffractometer. A two-theta member 11 is mounted on the housing for rotation about an axis through two-theta angles. The two-theta member 11 has a detector arm 12, FIGURE 1, which supports a suitable detector 13. An omega or theta member 15 is positioned concentrically within the two-theta member 11 for rotation about the same axis. The two-theta member 11 and the omega member 15 are each suitably driven by a mechanism such as the preferred mechanism described in the above-referenced application.

A diffraction X-ray tube is carried within a diffraction tube housing 16. The diffraction tube housing 16 is mounted on a support, a portion of which is shown at 14. The support is secured to the main housing 10. The housing 16 is positioned such that the longitudinal axis of the tube is horizontal when the rotational axis is vertical. Suitable adjustment mechanism indicated at 17 is interposed between the diffraction tube housing 16 and the main housing 10 and is preferably of the type described in the previously referenced application.

An end window spectroscopic X-ray tube is positioned within a spectroscopic tube housing 18. The spectroscopic tube housing is, when the main housing is horizontal, vertically oriented. The spectroscopic tube housing 18 is carried by a spectroscopic support assembly 19 which is fixed to the main housing 10.

A specimen support member 20 is mounted on the omega member 15. The specimen support member 20 is a track which carries, in the case of a diffraction study, the specimen and in the case of a spectroscopic study, an analyzing crystal. The specimen or crystal, depending on the type of study, is positioned for rotation about the axis of the omega member 15 with that axis extending through or along the face of the crystal or specimen as the case may be.

Referring now to FIGURE 2, the detector 13 is shown in the diffraction so-called "zero degree two-theta position." That is, the detector 13, the axis of the omega member 15, and focal spot 25 of the diffraction X-ray tube are on a straight line. During a diffraction study, the detector 13 is rotatable from the solid line position clockwise in a positive direction one hundred sixty-five degrees to a phantom position indicated at 21. The detector 13 is rotatable from its solid line zero degree two-theta position counterclockwise in a negative direction forty-five degrees to a position shown in phantom at 22. Thus, the major portion of diffraction two-theta rotation is in a positive direction from the solid line zero degree position.

The detector 13 when in a zero degree two-theta position for spectroscopy is in the position shown in phantom at 23. An examination of FIGURE 2 will show the zero degree two-theta lines of diffraction and spectroscopy are substantially normal to one another. In the actual structure shown, they intersect at an angle of ninety-six degrees. The detector 13 is rotatable from the position shown at 23 counterclockwise one hundred fifty-five degrees in a positive direction to the position shown in phantom at 24. Obviously, the detector arm is also rotatable in a negative direction for spectroscopy, but this is neither required nor used during normal spectroscopic studies. The major portion of the spectroscopic two-theta range overlaps the diffraction two-theta range. In the preferred and disclosed construction, the two ranges overlap about one hundred and forty one degrees.

One of the contributing factors to this large adjustment range is the construction of the diffraction and spectroscopic tube supports 14, 19 so they are recessed at 60 and 61 respectively. This contributes to the obtaining of a large range of rotation by permitting the detector arm 12 of the two-theta member to nest under portions of the supports as demonstrated in the phantom showings at 21 and 24 in FIGURE 2.

One of the features of this invention resides in omega and two-theta odometers 30, 31. Each of the odometers is equipped with a means to indicate respectively omega and two-theta positions of both diffraction and spectroscopy. Thus, in the case of the omega odometer 30, the upper most window 32 is a diffraction window while the lower most window 33 is a spectroscopic window. Similarly, in the case of the two-theta odometer 31, diffraction and spectroscopic windows 33, 34 are provided. Angle indicating means are provided, then, for both diffraction and spectroscopic positioning. By providing separate indicia for the two kinds of study, it is possible to quickly, for example, shift the detector 13 to a zero position as indicated at the diffraction two-theta window 33 to the spectroscopic zero position shown at 23 and indicated at the spectroscopic two-theta odometer window 34. If quick movement from spectroscopic to diffraction positions is desired, preferably a high speed motor is provided at 35 and coupled to the drive shaft described and claimed in greater detail in the referenced case. When the high speed motor is not provided, the shaft is driven with a hand crank.

The device as shown in FIGURE 1 is set up for a spectroscopic study. To this end a gas tight crystal housing assembly shown generally at 37 is provided. Suitable collimating devices are provided at 38 and 39 to couple the crystal housing 37 respectively to the specimen support assembly 19 and the detector 13. These collimating devices 38, 39 may be any of the known mechanisms for collimating an X-ray beam and are devices which provide part of a gas tight X-ray chamber. The specimen support assembly 19, the crystal housing 37, the detector 13, and the collimating devices 38, 39 together define an X-ray chamber which, for many spectroscopic studies, is filled with a helium atmosphere.

Referring now to FIGURE 3, the spectroscopic X-ray tube has a target shown schematically at 40. The target 40 has a target surface 41 which, when the device is positioned with the omega axis vertical, is at forty-five degrees with the horizontal. In other words, the target surface 41 is at forty-five degrees with the two-theta axis.

The spectroscopic support 19 includes a beam aperture 42 through which X-rays emitted by the target 40 pass when an X-ray impervious shutter 43 is open. A spectroscopic specimen holder shown generally at 45 is positioned within the spectroscopic support 19. The support 19 includes a pivotal access door 46 which is selectively openable to permit the specimen holder 45 to be inserted into and removed from its space within the spectroscopic support 19.

The support 45 includes specimen holding structure shown generally at 47. The structure 47 is adapted to position a specimen within a path of beams emitted by the target 40. The structure 47 is also adapted to position the specimen such that when excited, the specimen will emit its own X-rays which will pass through a specimen beam X-ray aperture 48 and thence through the collimating structure 38. The specimen support structure 47 has a specimen supporting surface 50 which is positioned as parallel as possible to the target surface 41.

This specimen support surface 50 is positioned such that specimen X-ray beam emitted by it will have a square or rectangular field size of at least one half inch along each side when a one and one quarter inch standard size specimen is used. That is, along the axis of the collimating device 38, the specimen beam will cover a cross sectional area in a plane perpendicular to the axis of the

attenuating device 38 which is, as a minimum, a square of one half inch along each side.

Again, the specimen support surface 50 is positioned as parallel as possible to the target surface 41 to obtain maximum excitation by the beam of X-rays emitted by the target 40. At the same time, the surface is at as flat an angle with the horizontal as possible while still obtaining the desired minimum field size, so that the specimen may be positioned as closely as possible to the target 40. It has been found that the optimum angle for these purposes is an angle of thirty-six degrees.

The advantages of this arrangement are depicted in the graph which is FIGURE 4. Referring to that graph, concentric circles 51 depict the angle of a cone of X-rays projected by the target 40. The radial lines 52 show intensity distribution in various radial directions. The endless curved but non-circular lines 53, 54, 55, respectively, are the lines of five-tenths, nine-tenths, and ninety-five hundredths of maximum intensity distribution where maximum intensity is unity. The rectangular dotted line 57 depicts the projected shape of a sample beam passing through the collimating device 38.

Thus, with the preferred and disclosed structure, it is possible to obtain intensity of ninety-five to one hundred percent of maximum intensity over the entire area of the specimen as compared with intensity of the order of five-tenths to one hundred percent of maximum over the specimen area with the typical prior art structures. The result is that not only is a higher average exciting intensity achieved, but a more uniform excitation over the sample area also is obtained.

One of the outstanding advantages of the present invention is in the ability to handle liquid spectroscopic specimens. The entire assembly may be tilted until the specimen support surface 50 is horizontal. Since the diffraction and spectroscopic X-ray tubes are both rigidly connected to the main housing 10 and since the spectroscopic specimen support 45 is firmly held in place, this tipping is obtainable without effecting any of the required alignments. Spectroscopic study of non-encapsulated liquid specimens has not been possible with prior structures, but with the tiltable structure of this invention, a liquid specimen can be held by the specimen support without encapsulation.

Although the invention has been described in its preferred form with a certain degree of particularity, it is understood that the present disclosure of the preferred form has been made only by way of example and that numerous changes in the details of construction and the combination and arrangement of parts may be resorted to without departing from the spirit and the scope of the invention as hereinafter claimed.

What is claimed is:

1. In a mechanism for non-destructive X-ray analysis including a housing carrying two-theta and omega elements for rotation about a common axis, the combination of:

(a) a diffraction X-ray tube, mounting means securing said diffraction tube to said housing with the tube aligned to emit a beam of X-rays toward a point on said axis;

(b) a spectrometer X-ray tube mounted on said housing in spaced relationship with said diffraction tube;

(c) mounting means to mount a spectrometry specimen in the path of and for excitation by a beam of X-rays emitted by the spectrometer tube, said mounting means being adapted to position and align a specimen for such excitation and emission of its own rays toward said point without interference with the diffraction tube and its mounting;

(d) said two-theta member including means to support a radiation responsive mechanism thereon, means connected to said two-theta member for moving said two-theta member and said radiation responsive mechanism selectively and one at a time through

- overlapping spectrometry and diffraction two-theta ranges; and,
- (e) support means mounted on the omega member for carrying a selected one of a crystal or a specimen along said common axis. 5
2. The device of claim 1 wherein the spectrometry tube has a longitudinal axis generally paralleling the axis of rotation.
3. The device of claim 2 wherein the diffraction tube has a longitudinal axis disposed in a plane generally normal to the axis of rotation. 10
4. The device of claim 3 wherein an adjustment means is interposed between the diffraction tube and the housing for relatively adjusting the tube and housing.
5. The device of claim 1 wherein theta and omega 15 odometers are respectively connected to the theta and omega members and wherein each odometer provides independent diffraction and spectroscopic readings.
6. The device of claim 1 wherein the positive direction of two-theta rotation of the radiation responsive member for diffraction is in a direction opposite from spectroscopic rotation. 20
7. A mechanism for non-destructive X-ray analysis including a housing carrying two-theta and omega elements for rotation about a common axis, the combination of: 25
- (a) a diffraction X-ray tube, mounting means securing said diffraction tube to said housing with the tube aligned to emit a beam of X-rays toward a point on said axis;
- (b) a mounting element on said housing and defining a specimen receiving space and an X-ray pervious ray emission window adjacent the space; 30
- (c) a spectrometer X-ray tube mounted on said element in spaced relationship with said diffraction tube;
- (d) specimen mounting means to mount a spectrometry specimen in the space and in the path of and for excitation by a beam of X-rays emitted by the spectrometer tube, said mounting means being adapted to position, and align a specimen for such excitation and emission of its own rays through said window toward said point without interference with the diffraction tube and its mounting; 40
- (e) said two-theta member including means to support a radiation responsive mechanism thereon for movement selectively and one at a time through overlapping spectrometry and diffraction two-theta ranges; and, 45
- (f) support means mounted on the omega member for carrying a selected one of a crystal or a specimen along said common axis. 50
8. A non-destructive testing mechanism comprising:
- (a) a main housing;
- (b) omega and two-theta members rotatably carried by the housing for rotation about a common axis;
- (c) a diffraction tube secured to said housing and positioned to emit a beam of X-rays along a diffraction zero degree two-theta line intersecting said axis; 55
- (d) spectroscopic specimen support means secured to said housing and adapted to position a specimen to emit an X-ray beam along a spectroscopic zero degree two-theta line intersecting said axis; 60
- (e) said zero degree lines being substantially normal to one another;
- (f) a spectroscopic X-ray tube secured to the support means and positioned to excite a spectroscopic specimen carried by said support means; 65
- (g) said two-theta member being rotatable through overlapping diffraction and spectroscopic two-theta ranges; and 70
- (h) the positive direction of diffraction two-theta rotation being in one direction and the positive direction of spectroscopic two-theta rotation being in the other direction. 75

9. A mechanism for non-destructive diffraction and spectroscopic studies comprising:
- (a) a housing;
- (b) concentric omega and two-theta members rotatably carried by the housing for relative rotation about a common axis;
- (c) a diffraction tube adjustably secured to the housing, the diffraction tube having a longitudinal axis disposed in a plane generally normal to said common axis, the diffraction tube being oriented to emit a beam of X-rays toward said common axis;
- (d) a spectroscopic tube support secured to the housing and defining a spectroscopic specimen space;
- (e) an end window spectroscopic X-ray tube secured to the tube support and having a longitudinal axis generally paralleling said common axis;
- (f) means to position a spectroscopic specimen in said space and orient the specimen to be bombarded by a beam of X-rays emitted by said spectroscopic X-ray tube;
- (g) said support including an X-ray pervious window positioned to pass a beam of X-rays emitted by spectroscopic specimen toward said common axis; and,
- (h) means connected to said two-theta member for rotating said two-theta member through overlapping spectroscopic and diffraction two-theta ranges one at a time with the positive direction of diffraction two-theta rotation being in one direction and the positive direction of spectroscopic two-theta rotation being in the other direction.
10. In an X-ray spectrometer including a spectrometer housing mounting omega and theta members for rotation about a common axis the improvement which comprises:
- (a) a spectroscopy support housing mounted on the spectrometer housing;
- (b) said spectroscopy housing defining a holder space;
- (c) a specimen holder in said space;
- (d) said spectroscopy housing including a movable access door and means to maintain the door in a closed position, said door being positioned and sized to permit said holder to be moved into and out of said space;
- (e) a spectroscopy X-ray tube mounted on said spectroscopy housing and having a target at an angular relationship with said axis;
- (f) said housing including an X-ray beam aperture between said target and said holder;
- (g) specimen holding structure mounted on said holder and in the path of X-ray emitted by said target;
- (h) said spectroscopy housing defining a second X-ray beam aperture, said second aperture being positioned to receive and transmit X-rays emitted by a specimen mounted in the specimen holding structure; and,
- (i) a shutter mounted in said spectroscopy housing and adapted to selectively close one of said beam apertures.
11. The device of claim 10 wherein said shutter is positioned to close the aperture between said spectroscopy tube and said holding structure.
12. The device of claim 10 wherein said holding structure is positioned in a plane generally paralleling the plane of the surface of said target.
13. In a mechanism for non-destructive X-ray mechanism including a housing, concentric omega and two-theta elements journaled in the housing for rotation about a common axis, means for mounting selectively and one at a time a crystal or a specimen on the omega element and an X-ray detection mechanism mounted on the two-theta element such that the two-theta element and mechanism together provide a rotatable two-theta assembly, the improvement which comprises:

- (a) diffraction and spectroscopic X-ray tube mounting structures secured to the housing;
- (b) diffraction and spectroscopic X-ray tubes mounted on the respective structures in spaced relationship with one another;
- (c) said diffraction X-ray tube being in alignment with said axis such that a beam of X-rays emitted by the diffraction tube is directed to a specimen mounted on said omega element;
- (d) said spectroscopic support structure including means for positioning of a spectroscopic specimen in the path of a beam emitted by said spectroscopic X-ray tube, said spectroscopic tube and specimen support structure being positioned and aligned to emit X-rays characteristic of the specimen toward a crystal mounted on said omega member;
- (e) at least one of said tube support structures including a recessed portion therein positioned and aligned to receive and overlap at least a portion of said two-theta assembly; and,
- (f) said omega element and said two-theta assembly being selectively rotatable one at a time through a

selected one of overlapping diffraction and spectrometer ranges.

14. The mechanism of claim 13, wherein both of said X-ray tube structures include recesses adapted to receive and in part overlap a portion of said two-theta assembly during extremes of rotation of said assembly.

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