April 26, 1966 A. H. PITCHFORD 3,248,543

X-RAY SPECTROGRAPHIC APPARATUS HAVING A PAIR OF X-RAY TUBES WITH DIFFERENT EMISSION PROPERTIES

Filed April 18, 1963 2 Sheets-Sheet 1

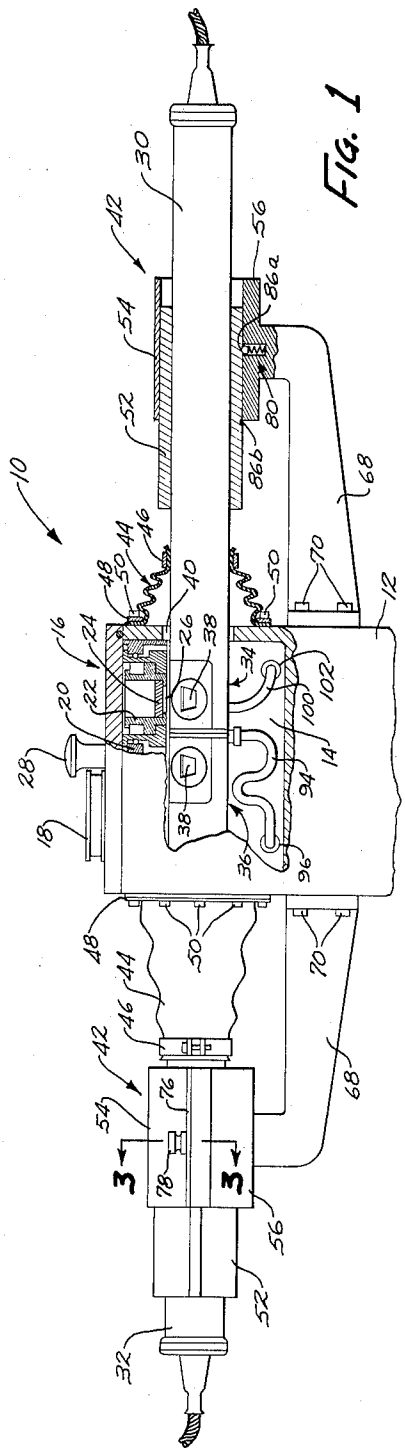


FIG. 1

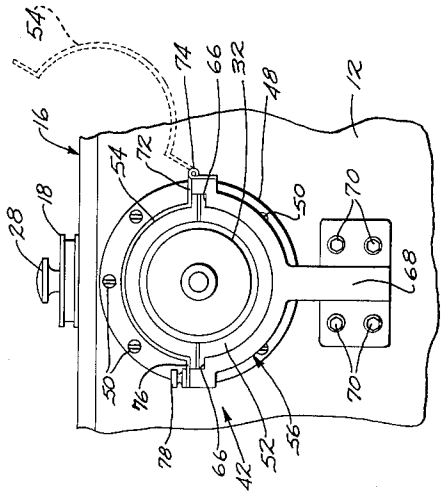


FIG. 2

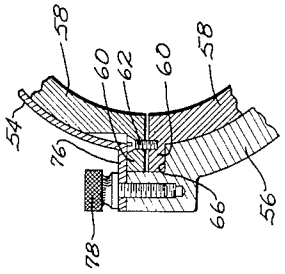


FIG. 3

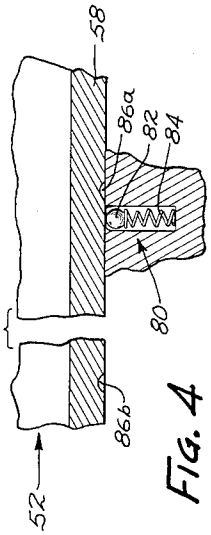


FIG. 4

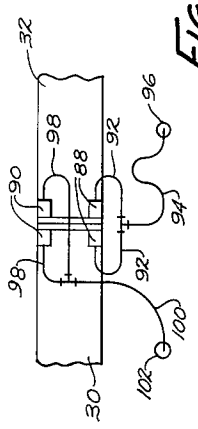


FIG. 5

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X-RAY SPECTROGRAPHIC APPARATUS HAVING A PAIR OF X-RAY TUBES WITH DIFFERENT EMISSION PROPERTIES

Filed April 18, 1963 2 Sheets-Sheet 2

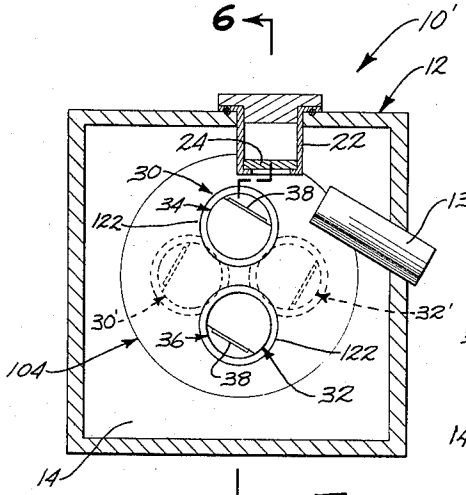


FIG. 7

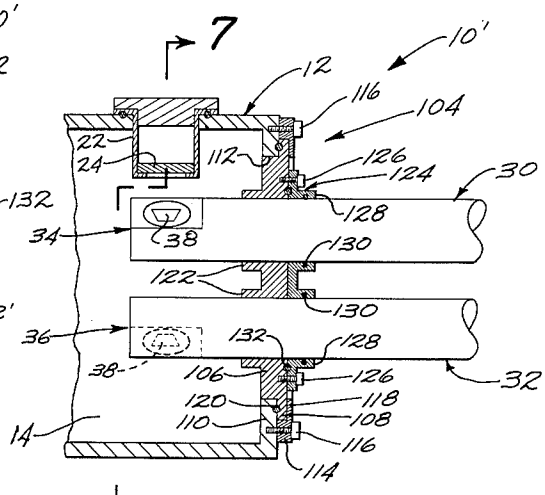


FIG. 6

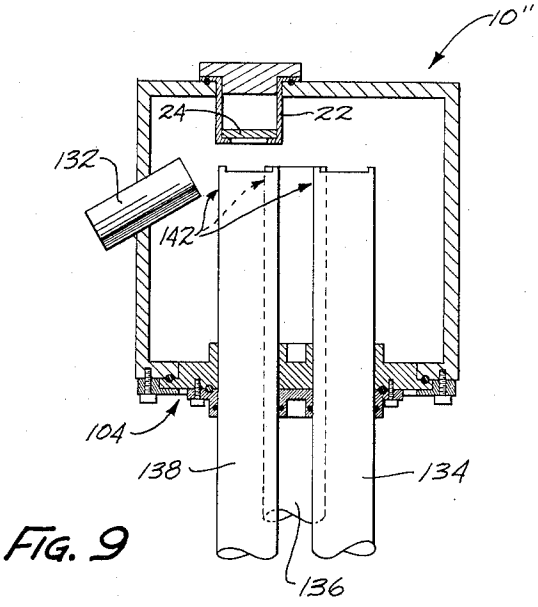


FIG. 9

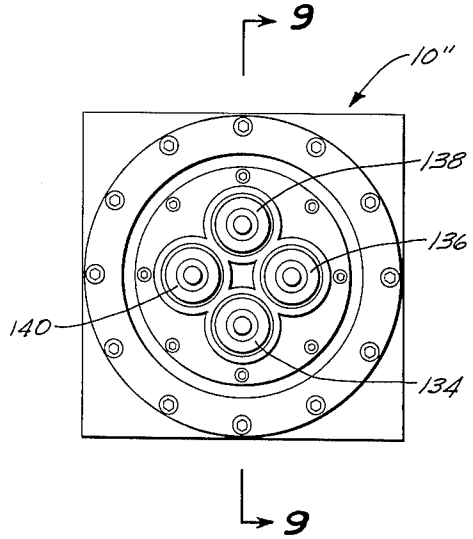


FIG. 8

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X-RAY SPECTROGRAPHIC APPARATUS HAVING A PAIR OF X-RAY TUBES WITH DIFFERENT EMISSION PROPERTIES

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Filed Apr. 18, 1963, Ser. No. 273,871
3 Claims. (Cl. 250—51.5)

This invention relates to X-ray spectrographic apparatus and more particularly to X-ray spectrographic apparatus having a plurality of X-ray emitting means for producing X-radiation of different wave lengths and wherein each of the X-ray emitting means may be positioned in radiating relation with the specimen to be analyzed.

In X-ray spectrography, an X-ray tube is employed which radiates a specimen with a primary beam of X-radiation. The primary beam of X-radiation causes the various elements present in the specimen to emit their characteristic X-ray spectra as secondary radiation. The secondary radiation is directed onto an analyzing crystal which separates the various wave lengths of the secondary radiation. The analyzing crystal may comprise, as for example, a single crystal of lithium fluoride, sodium chloride, EDDT or topaz. The sets of atomic planes of the crystal lattice are spaced at a suitable distance so that the analyzing crystal serves as a diffraction grating which will separate the various wave lengths comprising the secondary radiation emitted by the specimen.

As is well known, each element has its own characteristic X-ray spectrum. The wave lengths vary in a regular order from one element to another, i.e., the wave lengths decrease as the atomic numbers increase. Hence, each element present in a specimen may be identified by detecting radiation of each wave length at its corresponding angular position. The long wave lengths are dispersed to high angles while the shorter wave lengths are dispersed to lower angles.

A proportional or flow proportional counter, or a scintillation counter may be employed to detect the X-radiation reflected by the analyzing crystal at different angles. Hence, the analyzing crystal and the counter must be rotated to scan the desired angular region for each element. By recording the number of counts produced in the detector, a quantitative analysis may be made of the elements present in the specimen.

As stated above, the wave lengths vary in regular order from one element to another, i.e., the wave lengths decrease as the atomic number of the elements increase. The wave lengths are classified as "soft" X-rays which include the longer wave lengths and "hard" X-ray which include the shorter wave lengths. The secondary radiation emitted by each of the elements having atomic numbers from 11 to about 22 is relatively long and therefore is classified as "soft" radiation. The secondary radiation emitted by each of the elements having atomic numbers from about 22 and higher is relatively short and therefore is classified as "hard" radiation. Hence, most X-ray spectrographic apparatus employ two crystals mounted in such a manner that each may be moved into the path of the secondary radiation emitted by the specimen. As for example, an EDDT crystal, having relatively wide spaced atomic planes, may be employed in the diffraction of soft radiation and a lithium chloride crystal, having relatively closely spaced atomic planes, may be employed in the diffraction of hard radiation.

In order to maximize the intensity of the secondary radiation emitted by the elements in the specimen, it has

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been a common practice to employ one X-ray tube which emits hard radiation for the detection of the higher atomic number elements and another X-ray tube which emits soft radiation for the detection of the lower atomic number elements. As for example, an X-ray tube having a tungsten target will emit hard radiation and an X-ray tube having a chromium target will emit soft radiation.

Present day X-ray spectrographic apparatus uses only one X-ray tube at a time which is rigidly mounted and prealigned in radiating relation with the specimen. This X-ray tube normally employs a target formed from tungsten, platinum, molybdenum or the like, which emits hard radiation. Should the technician desire to employ an X-ray tube which emits soft radiation, he must remove the mounted X-ray tube and install another X-ray tube having a target formed from chromium, aluminum or the like. The removal and installation of X-ray tubes is a time consuming operation which requires disconnecting coolant conduits and the power cable as well as disconnecting the X-ray tube from its support. To install an X-ray tube, the coolant conduits must be reconnected and checked for leaks, the power connection must be made as well as physically securing the X-ray tube to its support. In vacuum systems all seals must be checked for leaks. Furthermore, since an X-ray tube is an expensive piece of equipment, the fewer number of times it is handled the lesser are the chances of damaging it.

Modern steel mills, for example, employ X-ray spectrographic apparatus for analyzing samples of the steel which are taken during its manufacture. Each heat of steel, then, must be held up until each sample is analyzed. It will be appreciated, then, that the time consuming operation of exchanging one X-ray tube for another is an undesirable feature of present day X-ray spectrographic apparatus.

Still another disadvantage of X-ray spectrography concerns the "reflection" of the X-radiation emitted by the X-ray tube. As hereinbefore stated, each specimen will emit secondary radiation which is characteristic of the elements present therein when the sample is radiated with a primary beam of X-radiation. However, a portion of the primary beam of X-radiation also will be reflected by the specimen. Hence, the record produced by the detector on a strip chart, as for example, will include an indication of the presence of a major quantity of the element from which the X-ray tube's target is made. This indication is usually ignored by the technician in his analysis of the specimen. It should be appreciated, then, that if the specimen contains a quantity of the same element from which the X-ray tube's target is made, the secondary radiation emitted by this element will be added to the reflected radiation of the primary beam and hence will be lost. This should be evident since the technican has no way of knowing just how much of the indicated quantity of the element corresponds to the quantity of the element present in the specimen. The X-ray spectrographic apparatus provided by this invention overcomes this disadvantage.

Accordingly, the primary objects of the present invention include:

To provide an X-ray spectrograph which is capable of detecting the lower atomic numbered elements as well as the higher atomic numbered elements;

To provide an X-ray spectrograph having a plurality of X-ray tubes each of which is capable of emitting a different type of X-radiation for the most optimum detection of the lower and higher atomic numbered elements;

To provide an X-ray spectrograph which is capable of detecting the elements from which the X-ray tubes' targets are made; and

To provide an X-ray spectrograph having a plurality of X-ray tubes each of which may be quickly and easily positioned in radiating relation with the specimen being analyzed.

These and other objects and advantages of the present invention will become apparent from the following detailed description by reference to the accompanying drawings, in which:

FIGURE 1 is a fragmentary elevation view, partly in cross section, of the present X-ray spectrograph;

FIG. 2 is an end elevation view of the apparatus of FIG. 1;

FIG. 3 is a fragmentary cross-sectional view, taken along the line 3—3 of FIG. 1;

FIG. 4 is a fragmentary cross-sectional view illustrating an indexing means employed in the present X-ray spectrograph;

FIG. 5 is an elevation view schematically illustrating the coolant connection of the X-ray tubes of FIG. 1;

FIG. 6 is a cross-sectional view, taken along the line 6—6 of FIG. 7, illustrating an alternative configuration of the present X-ray spectrograph;

FIG. 7 is a cross-sectional view, taken along the line 7—7 of FIG. 6, further illustrating the alternative configuration of FIG. 6;

FIG. 8 is a bottom view illustrating a further alternative configuration of the present X-ray spectrograph; and

FIG. 9 is a cross-sectional view, taken along the line 9—9 of FIG. 8, further illustrating the alternative configuration of FIG. 8.

Referring now to FIGS. 1 and 2, an X-ray spectrograph, generally indicated by the numeral **10**, comprises a main housing **12** having an open-ended chamber **14** and a cover **16** having a sample entrance port (not visible) which is covered by means of a cap member **18**. Within the cover **16** there is a turret-type specimen carriage **20** on which a plurality of specimen holders **22** (only one shown) are supported. The specimen holder **22** carries a specimen **24** at the open end **26** thereof. A handle **28** is connected to the specimen carriage **20** and serves to successively rotate each specimen **24** into the position shown in FIG. 1.

The X-ray spectrograph **10** is provided with a pair of X-ray tubes **30**, **32** having head portions **34**, **36** which reside within the chamber **14** and which are secured together in abutting relation by any suitable means. Each of the head portions **34**, **36** includes a window **38** through which a primary beam of X-radiation is directed onto the specimen **24**. In the X-ray spectrograph **10**, the X-ray tube **30** emits hard radiation and has a target which preferably is formed from tungsten. Alternatively, the target of the X-ray tube **30** could be formed from platinum or molybdenum. The X-ray tube **32**, on the other hand, emits soft radiation and has a target which preferably is formed from chromium. Alternatively, the target of the X-ray tube **32** could be formed from aluminum or copper or other lower atomic numbered elements. Hence, the X-ray spectrograph **10** is capable of detecting not only the higher atomic numbered elements through the use of the X-ray tube **30** but also is capable of detecting the lower atomic numbered elements through the use of the X-ray tube **32**. The X-ray spectrograph **10** is capable of detecting the lower atomic numbered elements with higher sensitivity particularly when these lower atomic numbered elements are present in trace quantities. It should be evident then that the present X-ray spectrograph has a greater versatility than any prior art X-ray spectrograph.

As illustrated in FIG. 1, the X-ray tube **30** has its head portion **34** disposed in radiating relation with the specimen **24**. As can be seen, the X-ray tube **30** extends outwardly through an opening **40** in one wall of the housing **12** and is slideably supported on a tube mount assembly **42**. Similarly, the X-ray tube **32** extends outwardly through an opening (not visible) in the housing **12** and is slideably supported on a similar tube mount assembly **42**.

A bellows seal **44** is provided for each X-ray tube **30**, **32** which has one of its ends secured to the body of the X-ray tube in sealed relation therewith by means of a strap clamp **46**. The other end of the bellows seal **44** is clamped between a ring member **48** and the wall of the housing **12** by means of a plurality of fasteners **50** which extend through the ring member **48** and the end of the bellows seal **44** and which are threaded into the wall of the housing **12**. The bellows seal **44** preferably is formed from resilient material such as rubber and the like. The need of a seal such as the bellows seal **44** will become apparent later in the specification.

Reference is now directed to FIGS. 1, 2 and 3, wherein the preferred construction of the tube mount assemblies **42** is illustrated. Each tube mount assembly **42** generally comprises a tube aligning member **52**, a cover strap **54**, and a cradle member **56**.

The tube aligning member **52** comprises a pair of semicircular members **58** each having a flange **60** extending from each side thereof. The semicircular members **58** are placed around the X-ray tube in opposed relation and a plurality of fasteners **62** (one shown in FIG. 3) are employed to rigidly clamp the semicircular members **58** to the X-ray tube.

The cradle member **56** comprises an arcuate portion **64** whose upper ends are provided with shoulders **66** which are adapted to receive and support the flanges **60** of the tube aligning member **52** in sliding engagement therewith. The cradle member **56** also includes a support arm **68** which is secured to the housing **12** by means of bolts **70**.

The cover strap **54** is semicircular in shape and is pivotally secured at one flanged end **72** to the cradle member **58** by hinge means **74** and is secured at the other flanged end **76** to the cradle member **58** by means of a thumbscrew **78**. The cover strap **54** is shown in a raised position in phantom outline in FIG. 2.

The cover strap **54** cooperates with the arcuate portion **64** of the cradle member **56** to permit the X-ray tube to move only in a longitudinal direction, i.e., toward or away from the chamber **14** of the housing **12**. The shoulders **66** and the flanged ends **72**, **76** of the cover strap **54** serve as guideways for the flanges **60** of the tube aligning members **52** and also prevent rotation of the X-ray tubes **30**, **32** about their longitudinal axes.

Referring now to FIGS. 1 and 4, the tube mount assembly **42** associated with the X-ray tube **30** also is provided with an indexing means **80** which limits the amount of longitudinal movement through which the X-ray tube **30** may move whereby the window **38** thereof is aligned in radiating relation with the specimen **24**. The indexing means **80** comprises a ball **82** which is biased toward the tube aligning member **52** by means of a spring member **84**. The lower semicircular member **58** of the tube aligning member **52** is provided with a pair of detents **86***a*, **86***b* into which the ball **82** may enter. The detents **86***a*, **86***b* are spaced apart by a predetermined distance which is equal to the distance between the centerlines of the windows **38** of the X-ray tubes **30**, **32**. The purpose of these detents will become apparent later in the specification. In any event, when the X-ray tube **30** is moved, a click will be heard each time the ball **82** engages one of the detents **86***a*, **86***b*.

Referring now to FIGS. 1 and 5, each of the X-ray tubes **30**, **32** requires a coolant to be circulated internally thereof in order to cool the target. As can be seen in FIG. 5, each of the X-ray tubes **30**, **32** has a coolant inlet port **88** and a coolant outlet port **90**. As schematically illustrated, rigid conduits **92** leading from the inlet ports **88** are connected to a flexible conduit **94** which extends from a supply port **96** in the housing **12**. Similarly,

rigid conduits **98** are connected to a second flexible conduit **100** which extends from a discharge port **102** in the housing **12**. Since the conduits **92** and **98** are rigid, they serve the secondary function of coupling the X-ray tubes **30** and **32** together in a rigid manner. Since the conduits **94** and **100** are flexible, the X-ray tubes **30** and **32** are free for longitudinal movement.

In operation, then, the technician places a number of the samples **24** in the specimen holders **22**. To analyze each of the specimens **24** for elements having atomic numbers from about 22 and higher, the technician employs the X-ray tube **30** in the position illustrated in FIG. 1. When this analysis is completed, the technician need only slide the X-ray tubes **30, 32** to the left until a click is heard (ball **82** engages detent **86***b*) which indicates that the window **38** of the X-ray tube **32** is in radiating relation with the specimen **24** being analyzed. The technician proceeds with his analysis of the elements having atomic numbers from 11 to about 22. It should be noted that the bellows seals **44** permit the X-ray tubes **30, 32** to be moved without breaking the seal provided thereby. Hence, the X-ray spectrograph **10** may be employed to analyze specimens either at atmospheric pressure or under a vacuum.

Alternatively, the X-ray tubes **30, 32** may be mounted on the X-ray spectrograph **10** on a rotatable carriage means whereby they may be rotated into radiating relation with the specimen **24**. This alternative arrangement is illustrated in FIGS. 6 and 7. Corresponding numerals will be employed to identify corresponding parts already described.

Referring to FIGS. 6 and 7, an X-ray spectrograph **10′** has a rotatable carriage means **104** which is employed to support the X-ray tubes **30, 32** in spaced, side-by-side, substantially parallel relation. The rotatable carriage means **104** comprises a rotatable plate **106** having a peripheral flange **108** engaged with a wall **110** of the main housing **12**. The rotatable plate **106** is positioned within an opening **112** of the wall **110** and is rotatable therein. An annular plate **114** is secured to the wall **110** preferably by means of bolts **116** and includes an inwardly extending flange **118** which engages the peripheral flange **108** of the rotatable plate **106** with a slight pressure whereby the plate **106** may be rotated. An O-ring seal **120** is provided between the peripheral flange **108** and the wall **110**.

As can be seen the X-ray tubes **30, 32** extend through and are supported by annular hub members **122**. A seal plate **124** is secured to the rotatable plate **106** by means of bolts **126** and includes annular hub member **128** through which the X-ray tubes **30, 32** extend. O-ring seals **130, 132** are provided for sealing the seal plate **124** against the entry of air. The O-ring seals **120, 130, 132** are especially important when the chamber **14** of housing **12** has been evacuated.

The rotatable carriage means **104** maintains the head portion **34** of the X-ray tube **30** in radiating relation with the specimen **24** disposed in the specimen holder **22**. When it is desired to employ the X-ray tube **32**, the technician merely rotates the X-ray tube **32** into radiating relation with the specimen **24**. Although not illustrated in FIGS. 6 and 7, an indexing means similar to the indexing means **80** hereinbefore described preferably is provided as a means for indicating the proper alignment of each of the X-ray tubes **30** or **32** with respect to the sample **24**.

In FIG. 7, a collimator **132** is secured to the housing **12** and is aligned with the specimen **24** to direct the secondary radiation onto an analyzing crystal (not shown).

The X-ray spectrograph **10′** has been illustrated supporting two X-ray tubes. Alternatively, more than two X-ray tubes may be supported by the rotatable carriage means **104**. As can be seen in FIG. 7, two other X-ray tubes **30′** and **32′** are shown in phantom outline in the position in which they would be supported by the rotatable carriage means **104**.

FIGS. 8 and 9 illustrate a further alternative configuration of the present invention. An X-ray spectrograph **10″** includes the housing **12** provided with the rotatable carriage means **104** hereinabove described. The rotatable carriage means **104** supports X-ray tubes **134, 136, 138** and **140** in vertical substantially parallel side-by-side relation. Each of the X-ray tubes **134, 136, 138** and **140** includes a head portion **142** having an end window **144** through which X-radiation is emitted in a direction which is parallel to the longitudinal axis of the X-ray tube. In this instance, the specimen holder **22** supports the specimen **24** directly above the end window **144** of the X-ray tube **138**. The collimator **132** is aligned with the specimen **24** and directs the secondary radiation onto an analyzing crystal (not shown).

In this configuration, the X-ray tubes **134** and **136** may comprise hard emitters while the X-ray tubes **138** and **140** comprise soft emitters. That is to say, the targets of the X-ray tubes **134** and **136** may be formed from tungsten and platinum respectively which are hard emitters while the targets of the X-ray tubes **138** and **140** may be formed from chromium and copper respectively. Hence, the technician may employ the X-ray tube **134** (tungsten target) for the detection of the higher atomic numbered elements present in the specimen **24** with the exception of tungsten. The technician may then rotate the X-ray tube **136** (platinum target) into radiating relation with the specimen **24** for the detection of the element tungsten. Similarly, the technician may employ the X-ray tube **138** (chromium target) by merely rotating it into radiating relation with the specimen **24**. The X-ray tube **138** may be used to detect the lower atomic numbered elements present in the specimen **24** with the exception of chromium. To complete his analysis of the specimen **24**, the technician may then rotate the X-ray tube **140** (copper target) into radiating relation with the sample **24** for the detection of the element chromium.

Although not shown in FIGS. 8 and 9, an indexing means similar to the indexing means **80** hereinabove described preferably is provided as a means for indicating the proper alignment of each of the X-ray tubes **134, 136, 138** and **140** with respect to the sample **24**.

Although the invention has been shown in connection with certain specific embodiments, it will be readily apparent to those skilled in the art that various changes in form and arrangement of parts may be made to suit requirements without departing from the spirit and scope of the invention.

I claim as my invention:

1. In X-ray spectrographic apparatus, the combination comprising a pair of separate generally tubular X-ray tubes, one of said X-ray tubes having a head portion incorporating means for emitting relatively soft X-radiation and the other of said X-ray tubes having a head portion incorporating means for emitting relatively hard X-radiation, and carriage means supporting said pair of tubular X-ray tubes in substantially coaxial head portion-to-head portion abutting relationship for movement along the axes of the tubes, whereby the X-radiation emitting means in a selected one of said tubes may be moved into radiating relation with a specimen to be analyzed.

2. In X-ray spectrographic apparatus, the combination comprising a pair of separate X-ray tubes each having a head portion from which X-radiation is emitted in a direction which is transverse to the longitudinal axis of its associated X-ray tube, one of said X-ray tubes being capable of emitting relatively soft X-rays and the other of said X-ray tubes being capable of emitting relatively hard X-rays, carriage means for movably supporting said pair of X-ray tubes in axially aligned, head portion-to-head portion, generally abutting relation, and means for rigidly connecting said pair of X-ray tubes whereby a selected one of said pair of X-ray tubes may be moved on the car-

riage means into radiating relation with a specimen to be analyzed.

3. In X-ray spectrographic apparatus, the combination comprising a pair of separate X-ray tubes each having a head portion from which X-radiation is emitted in a direction which is transverse to the longitudinal axis of the X-ray tube, one of said X-ray tubes being capable of emitting relatively soft X-rays and the other of said X-ray tubes being capable of emitting relatively hard X-rays, carriage means for movably supporting said pair of X-ray tubes in axially aligned, head portion-to-head portion, generally abutting relation, said carriage means including a main housing which encloses said generally abutting head portions, specimen supporting means in the housing, tube mount assemblies on opposite sides of said housing for supporting the respective ends of said X-ray tubes which are opposite said generally abutting head portions, and means for rigidly connecting said pair of X-ray tubes whereby either one of said pair of X-ray tubes may be moved on said tube mount assemblies into a position where

the X-radiation emitted from its head portion is directed onto a specimen supported on said specimen supporting means.

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