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PROPORTIONAL COUNTER HAVING A POLYCARBONATE WINDOW

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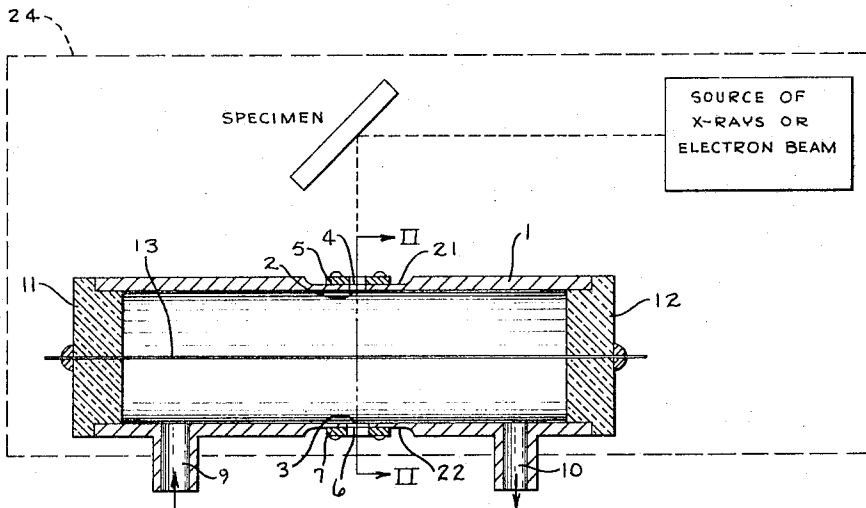


Fig. 1

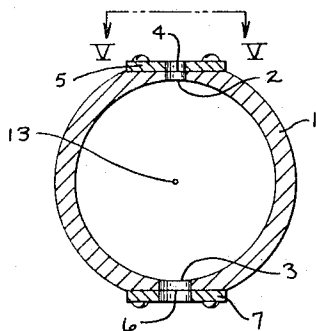


Fig. 2

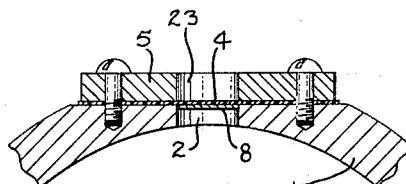


Fig. 3

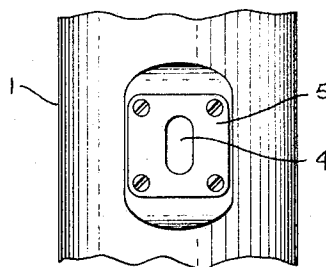


Fig. 5

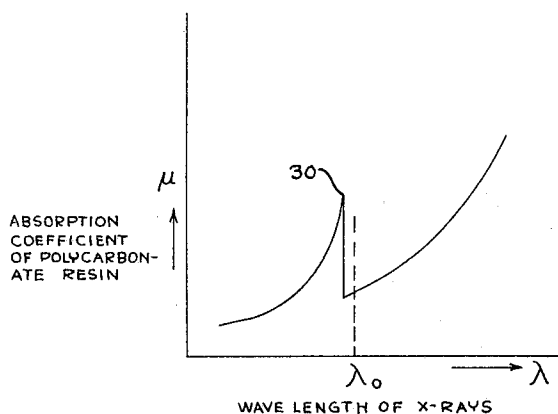


Fig. 4

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**PROPORTIONAL COUNTER HAVING A
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4 Claims. (Cl. 313-93)

This invention relates to a proportional counter and, more particularly, relates to a proportional counter for a fluorescent X-ray analyser or for an electron probe X-ray microanalyser.

In a fluorescent X-ray analyser, a sample is analysed by detecting wave lengths of characteristic X-rays, corresponding to the elements which comprise the sample, emitted by striking the sample with X-rays or an electron beam. In an electron probe X-ray microanalyser, the elements in a minute part of a sample are detected by striking the minute part with a fine electron beam produced by an electron optical system and detecting the X-rays emitted from the sample.

Different types of counters are used for detecting and measuring the intensities of the X-rays in these instruments depending on the wave lengths of the X-rays to be detected. In particular, for relatively long-wave X-rays a gas-flow-type proportional counter having an entrance window for X-rays made of mica or beryllium is used. However, mica has a comparatively low transmitting efficiency for relatively long-wave X-rays. Further, even though the transmitting efficiency of beryllium to relatively long-wave X-rays is rather high, as is understood from its atomic number, it is impossible to reduce the thickness of a beryllium foil sufficiently to minimize the absorption of X-rays by the beryllium window. In this regard, both the nature of the material of which the window is formed and its thickness determine how much of the X-rays are transmitted through the window. Moreover, it is difficult to keep the counter gas-tight against the pressure difference which exists between the outside and the inside of the counter because of the low mechanical strength of beryllium foil, which leads to the formation of pin holes. It has also been suggested to use a Mylar film as the window material. The thickness of Mylar can be reduced to some extent without losing mechanical strength but a film sufficiently thin to transmit relatively long-wave X-rays with high efficiency cannot be formed from Mylar.

Therefore, hitherto it has been impossible to detect X-rays having a wave length longer than 10 angstrom units by a conventional proportional counter.

Carbon is one of the most important elements in modern manufacturing materials, especially in steel. Therefore, it is greatly desired that a fluorescent X-ray analyser and an electron probe X-ray microanalyser be capable of detecting carbon. However, since the wave length of the characteristic X-ray of carbon is about 44 angstrom units, it has been impossible to detect carbon heretofore by fluorescent X-ray analysers and electron probe X-ray microanalysers which utilize known proportional counters.

Accordingly, it is the object of the present invention to provide a proportional counter which can detect effectively the characteristic X-rays of carbon, which counter uses a thin film of polycarbonate resin as the window material.

The invention will be more readily described and understood with reference to the accompanying drawing wherein:

FIGURE 1 is a sectional view of a preferred embodiment

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of the present invention and schematically shows additional apparatus which is associated therewith in use.

FIGURE 2 is a cross-sectional view taken along line II-II of FIGURE 1;

FIGURE 3 is an enlarged view of a fragment of FIGURE 2; and

FIGURE 4 is a characteristic absorption curve of a polycarbonate resin film plotted against wave length of X-rays;

FIGURE 5 is an enlarged plan view of a fragment of FIGURE 2 substantially as taken along the line V-V of FIGURE 2.

Referring to the figures, the gas-flow-type proportional counter in accordance with the present invention comprises a metallic cylinder 1 which, for example, may be 20 mm. in diameter. The external wall of the cylinder 1 is flattened at two diametrically opposite regions 21 and 22 approximately midway between the axial ends of the cylinder. A slit-form or elliptic opening 2 for X-rays, e.g., of 2-3 mm. in width and 5 mm. in length, is formed in the central portion of one of the flattened regions 21, and an X-ray penetrating slit 3 is formed in the central portion of the other flattened region 22. A thin film 4 of polycarbonate resin is secured to the external wall of the cylinder 1 by a suitable adhesive and extends over the opening 2. A metal ring 5 overlies the film 4 and is secured to the cylinder 1. The ring 5 has a centrally located opening 23 of substantially the same shape as the slit 2 and which is aligned therewith.

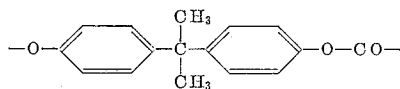
A thin film 6 of a polyethylene terephthalate resin (Mylar) is mounted on the cylinder 1 over the slit 3 therein and is secured thereto by means of a metal ring 7. If desired or necessary, an aluminum film 8 may be vacuum-coated on the inside surfaces of the films 4 and 6 in order to prevent the disturbance of the electric field around the films.

The metal cylinder 1 has a gas inlet 9 and a gas outlet 10 adjacent the respective ends thereof. Both ends of the cylinder are sealed air-tightly by end plates 11 and 12 which are made of an insulating material, such as glass. A fine metal wire 13 extends coaxially through the cylinder 1 and passes through the end plates 11 and 12.

In operation a gas mixture of, e.g., 90% argon and 10% methane is passed continuously through the cylinder 1 at about atmospheric pressure by connecting the gas inlet 9 and gas outlet 10 to gas conduits (not shown). As is well understood, argon absorbs the X-rays and methane makes the counter self-quenching whereby each current pulse through the counter is quickly extinguished. A voltage of 1500-2000 volts is applied between the wire 13 and the metallic cylinder 1 so as to make the wire 13 positive. The wire 13 and cylinder 1 are connected to any electric circuit of any suitable conventional type whereby the X-ray quanta entering the counter are counted and recorded, in conventional fashion.

X-rays to be detected are projected into the cylinder 1 through the opening 2 having the polycarbonate resin thin film 4 thereacross. The X-rays are generated by directing X-rays or an electron beam against the specimen and then directing the X-rays emitted by the specimen toward the opening, as schematically indicated in FIGURE 1. Since suitable mechanism for this purpose is well known and since the invention does not relate thereto, further description thereof is deemed unnecessary. Since the wave length of the characteristic X-ray of carbon is extremely long as mentioned above and, consequently, the scattering and absorption by air are great, the proportional counter is placed in a vacuum vessel indicated schematically at 24 to maintain the space between the X-ray source and the counter at a high vacuum.

Polycarbonate resin is a polymer in which the molecular structure is that of a chain in which each individual link comprises the following unit:



The term "polycarbonate" resins as used in this disclosure and the appended claims shall mean those resins which are derived from aromatic dihydroxy compounds and carbonic acid. The presently commercially available polycarbonate resins are manufactured essentially by reacting carbonic acid and bisphenol A (4,4' di-hydroxy diphenyl propane). The thickness of the polycarbonate resin film 4 may be below about 1 micron and is preferably about 0.5 micron. Such an extremely thin film of polycarbonate resin can be prepared as follows:

A polycarbonate resin sheet of a thickness of 0.05 mm. and an area of 1 x 5 cm.² is dissolved in 3 cc. of methylene chloride (CH₂Cl₂) and the solution is let flow down along the surface of a glass plate which is inclined at an angle of about 45°. As methylene chloride evaporates quickly, a thin film of polycarbonate resin is formed on the glass plate. The thickness of the film on the upper part of the glass plate will be thinner than that on the lower part. The film is stripped by means of tweezers and a portion having a suitable thickness is used as the film 4. The thickness of the film obtained can be adjusted by varying the concentration of the solution, the inclination of the glass plate, etc.

Since the counter is placed in vacuo and a gas mixture at about atmospheric pressure is passed through the inside of the counter as mentioned above, the film must have some thickness in order to withstand the pressure difference. However, since polycarbonate resin film is strengthened by being subjected to moderate pressure differences, even very thin films have sufficient strength for the purposes of the invention. Thus, when the dimensions of the opening 2 are those stated above, a film 4 of 0.4-0.5 micron thickness can withstand the pressure difference without rupture. If the dimensions of the opening 2 are smaller, the thickness of the film can be reduced to about 0.2 micron.

As described above, the tensile strength of the polycarbonate resin film 4 is very high and no pin holes are formed during the preparation of the film. Also, while it is impossible to form a film of thickness below several microns from Mylar, polycarbonate resin can be formed into a thin film of about 0.2-1 micron in thickness because it can be easily dissolved in methylene chloride. Further, since polycarbonate resin is a compound of carbon, oxygen and hydrogen, all of which have low atomic numbers, the transmitting power of the polycarbonate film is high to long wave X-rays. Thus, the absorption, by the film 4, of X-rays having a wave length in excess of ten angstrom units can be reduced, by reducing the thickness of the polycarbonate film below about 1 micron, to such an extent that useful results can be obtained.

As shown in FIGURE 4, the absorption coefficient (μ) of polycarbonate resin to X-rays increases in proportion to the wave length (λ) of X-rays up to a peak indicated at 30. Because the main element of polycarbonate resin is carbon, the peak 30 occurs at a point slightly below the wave length (λ_0) of the characteristic X-ray of carbon and then the absorption characteristic falls sharply. Therefore, polycarbonate resin has a high transmitting power or transparency to the characteristic X-ray of carbon. However, the characteristic X-rays of oxygen, nitrogen, etc., are absorbed by the thin film 4 of polycarbonate resin. Similarly, the characteristic X-rays of boron, beryllium, lithium, etc., which are long-

er than the characteristic X-ray of carbon, also are more completely absorbed.

In other words, since in the present invention the entrance window for X-rays is formed by a thin film 4 of polycarbonate resin that has a maximum transmitting power to the characteristic X-ray of carbon and since a thin film of polycarbonate resin below about 1 micron in thickness can be easily prepared, the characteristic X-rays of carbon can be transmitted into the counter in an effective manner so that they can be counted to give an accurate indication of the carbon in the specimen. Moreover, since the thin film 4 of polycarbonate resin has a high tensile strength the film can withstand the pressure difference so long as the opening 2 is not too large, e.g. 10-40 mm.², in area. Also, the film 4 of the present invention has a high transmitting power to the characteristic X-ray of carbon, but its transmitting power to the characteristic X-rays of other elements having different wave length than that of the characteristic X-ray of carbon decreases. Therefore, by the proportional counter of the present invention, the characteristic X-ray of carbon can be detected effectively and it is possible to analyse carbon by means of a conventional fluorescent X-ray analyser or electron probe X-ray microanalyser.

What is claimed is:

1. A proportional counter having an entrance window for X-rays which is formed of a film of polycarbonate resin of less than about 1 micron in thickness, whereby a long-wave X-ray, in particular the characteristic X-ray of carbon, can be detected effectively.

2. A proportional counter according to claim 1 in which the counter is placed in vacuo.

3. In a proportional counter which comprises wall means defining a chamber, said wall means having an opening therethrough whereby X-rays may be transmitted through said opening into said chamber, an anode wire in the chamber, and conduit means for supplying gas to and removing the gas from said chamber, the improvement which comprises a film of polycarbonate resin of less than about 1 micron in thickness affixed to said wall means and covering said opening whereby long wave X-rays, particularly the characteristic X-ray of carbon, can be efficiently transmitted into said chamber for detection therein.

4. A proportional counter comprising:

a metal cylinder having a gas inlet conduit and a gas outlet conduit connected thereto at its opposite ends, said cylinder having at least one opening therethrough between its ends, said opening being from 10-40 mm.² in area;

insulating end plates mounted on the ends of said cylinder for sealingly closing same;

an anode wire extending coaxially through the cylinder and through said end plates;

a film of polycarbonate resin of less than about 1 micron in thickness affixed to said cylinder and covering said opening whereby long wave X-rays, particularly the characteristic X-ray of carbon, can be efficiently transmitted into said chamber for detection therein.

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