

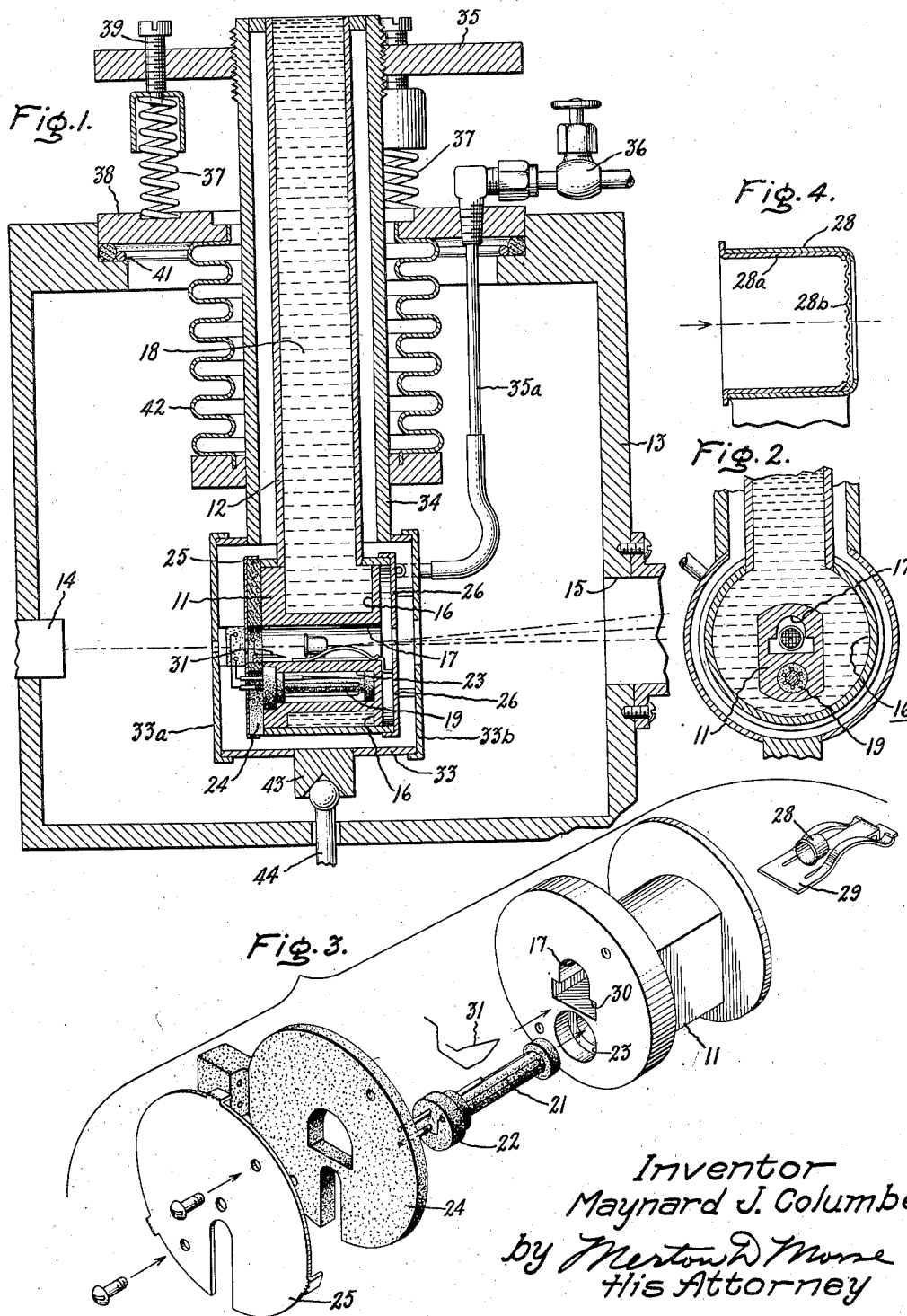
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LOW TEMPERATURE CHAMBER FOR ELECTRON-OPTICS INSTRUMENTS

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LOW TEMPERATURE CHAMBER FOR ELECTRON- OPTICS INSTRUMENTS

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The present invention relates to an attachment for electron optics instruments.

More particularly, the invention relates to a low temperature sample chamber for use in electron diffraction instruments, electron microscopes, and other electron optics instruments of a similar nature.

The continued widespread acceptance of electron optics instruments such as the electron diffraction instruments as a useful tool of science and industry, has made it desirable to continually seek new fields of application for these instruments. As a result of this activity, a specific need has already arisen in the field of low temperature studies concerning the effect of low temperatures on the molecular structure of different materials. To meet this need, the present invention was developed.

It is, therefore, one object of the present invention to provide a low temperature attachment for electron diffraction instruments and electron microscopes, and other instruments of a similar nature whereby the temperature of a sample being examined may be reduced below ambient temperature conditions for purposes of examination.

Another object of the invention is to provide a low temperature sample chamber for electron optics instruments which allows a sample to easily be inserted and removed from the chamber without requiring extraordinary safeguards to prevent the entrance of foreign matter into the analyzing region of the instrument which would impair its operation.

Still another object of the invention is to provide a low temperature sample chamber for electron optics instruments which has the characteristics set forth in the preceding paragraph, and which allows the sample supported therein to readily be manipulated from outside the chamber in order to allow control of the position of the sample with regard to the electron beam developed in the instrument.

A still further object of the invention is to provide a device having the above set forth features which is simple in construction and relatively inexpensive to manufacture.

In practicing the invention a low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration, is provided. This attachment comprises a sample chamber adapted to be supported in the electron beam path of the instrument so that the electrons impinge upon a sample supported therein, and means for reducing the temperature of the sample chamber and the sample contained therein to desired temperature value below ambient temperature conditions. In the preferred embodiment of the invention the cooling means comprises liquid coolant disposed in a leakproof region of the sample chamber, and a controllable heating means positioned adjacent the sample chamber for controlling the temperature thereof. Additionally, it is desirable that an insulating housing be disposed surrounding the chamber and liquid-tight portion thereof, and include a means for

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introducing a dry gas into a region intermediate the insulating housing and a sample chamber. It is further desirable that the insulating housing in the sample chamber be supported upon flexible bellows which allows the housing to engage a translating manipulator rod that serves to move the housing into any desired direction so as to align the sample in the sample chamber thereof with the electron beam path.

Other object, features, and many of the attendant advantages of this invention will be appreciated more readily as the same become better understood by reference to the following detailed description when considered in connection with the accompanying drawings, wherein like parts are identified by the same reference character, and wherein:

Fig. 1 is a sectional view of a portion of an electron optics instrument showing a low temperature attachment constructed in accordance with the invention, mounted therein;

Fig. 2 is a cross-sectional view of a sample chamber portion of the attachment shown in Fig. 1;

Fig. 3 is an exploded, perspective view of the sample chamber shown in Figs. 1 and 2, and illustrates the details of construction thereof; and

Fig. 4 is a cross-sectional view of a sample holder comprising an element of the sample chamber illustrated in Figs. 1 and 2.

The low temperature attachment shown in Fig. 1 comprises a sample chamber 11 which is supported at the end of a post 12 that is hollow and preferably cylindrical in shape. The sample chamber 11 is supported within a housing 13 that comprises a part of an electron optics instrument such as an electron diffraction instrument or an electron microscope of the type disclosed in U. S. Patent No. 2,606,292, issued August 5, 1952, to Maynard J. Columbe, and assigned to the same assignee as the present invention. In instruments of this type an electron beam is produced across the housing by means of an electron source, such as is indicated at 14, for examining a specimen under consideration. The electron source 14 generally comprises a source of electrons and an electron lens or a system of electron lenses for bringing the electrons to a focus through an opening 15 in the side of housing 13 upon an image forming means such as a photographic plate (not shown). By positioning a specimen to be examined in the electron beam between electron lens system and the image forming means, a diffraction pattern of the specimen to be examined may be obtained. Such diffraction patterns generally comprise predetermined arrangements of concentric circles, the relative spacings of the circles being determined by the electron diffraction which is in turn determined by the lattice spacing of the atoms of the crystals comprising the specimen material. These patterns may be correlated with known material diffraction patterns to indicate the chemical composition of the electron irradiated specimens being examined. As the methods and techniques for obtaining and using such electron diffraction patterns are relatively well-known in the art, and have thoroughly been described in literature printed on this subject, further discussion of these techniques is believed unnecessary.

In addition to the diffraction patterns, methods and techniques are known for obtaining electron shadow images or micrographs of specimens with apparatus of a similar type to that shown in Fig. 1. When the apparatus is used in such a manner, electrons are transmitted through or reflected from a portion of a specimen focused by an electron lens or system of electron lenses to provide an enlarged electron micrograph or image of the specimen on an image forming means such as a

photographic plate. Images of this type generally resemble conventional light microscope images, and the techniques and methods for obtaining such images are likewise relatively well described in the literature concerning this subject.

Heretofore, the uses of electron optics instruments such as the electron diffraction instrument and the electron microscope have been restricted to solid state materials, that is materials which are normally solid at atmospheric temperature. Because of the fact that it is desirable to study the characteristics of certain materials which are normally liquid in form at atmospheric temperature (such as petroleum products, mercury, gallium and water) in order to determine what happens to such materials at lower temperatures upon the same reaching a solid state or nearly solid state, it has been determined that some means is required for obtaining diffraction patterns and micrographs of such materials. In order to meet this need, the present invention was developed.

For the purpose of obtaining low temperatures, and for maintaining samples under examination at such low temperatures, the sample chamber 11 has a leakproof or liquid-tight region 16 formed therein. The chamber 11 may be constructed out of a solid block into which the region 16 is machined, or if desired, the chamber may be cast to include such region. The liquid-tight region 16 surrounds a central passageway 17 formed in the sample chamber in a manner best seen in Fig. 2 of the drawings, and which is designed to accommodate the sample to be analyzed. The central passageway 17 is adapted to be aligned with the electron beam produced by the source 14, and has the sample to be analyzed disposed therein so as to intercept the beam. The liquid-tight region 16 of the sample chamber, and a portion of the interior of hollow supporting post 12 are filled with a suitable liquid coolant 18, preferably a liquid gas such as liquid air, which serves to reduce the temperature of the sample chamber, and, hence, the region thereof in passageway 17 in which the sample is located to a very low value, and thereby serves to retain the temperature of the sample being analyzed at a low temperature value.

In order to control more carefully the temperature to which the sample chamber, and, hence, the sample being examined is reduced, controllable heating means indicated at 19 are provided in the sample chamber. The heating means is mounted in the portion of the sample chamber 11 immediately below the central passageway 17 in the manner best shown in Figs. 1 and 2 of the drawings so that heat provided thereby is conducted throughout the chamber. As is best seen in Fig. 3 of the drawings, the heating means 19 comprises a heating coil 21 suitably wound upon a ceramic core 22 so that the magnetic fields produced by heating current flowing through the conductors thereof are balanced out, and no distorting magnetic field is produced around the heating element. The core 22 and coil 21 are mounted in a bore 23 formed in the sample chamber immediately below the central passageway 17 in the manner best seen in Fig. 2 of the drawings, and is held in place by a ceramic end piece 24 and a metallic shield member 25 having a central aperture therein. The shield member 25 assists in holding the entire structure in assembled relation, and also acts to collimate the electron beam. A second metallic end member 26 which is readily removable and has an enlarged central aperture therein for accommodating the diffracted electron beam is mounted on the remaining side of the sample chamber. The metallic end members 25 and 26 have highly polished surfaces so that they act as radiation shields to prevent excessive loss of heat through radiation. By this construction then, it is possible to control the amount of heat supplied to the sample chamber 11, as well as the amount of heat radiation therefrom and, hence, control the temperature

of the chamber to a fine degree. Additionally, by controlling the heating current supplied to coil 21, the temperature of the sample within the central passageway of the sample chamber can be varied over a wide range of values.

Removably supported in the central passageway 17 of sample chamber 11 is a sample holder 28, best seen in Fig. 4 of the drawings, which comprises a tubular outer member 28, having a shoulder at one end, a wire grid 28b for mounting the specimen and a split collar 28a for clamping the wire grid in place. The sample holder 28 is secured to a movable slide 29 shown in Fig. 3 of the drawings having the sides thereof cut away and bent upwardly to form leaf springs. Sample holder 28 is adapted to be secured by spot welding or some other means and the slide 29 inserted into the central passageway 17 to a point where the members forming a leaf spring thereof ride over the bottom of the central passageway 17 and coast with a downwardly protruding shoulder portion 30 to clamp the slide in place within the passageway. The sample to be analyzed is disposed in a manner well-known to the art on the wire grid 28b which is then inserted in place in holder 28 by the split collar 28a.

The sample holder shown in Figs. 1 and 4 of the drawings comprises a transmission type sample holder for use with electron optics instruments; however, if desired, a reflection type sample holder such as is disclosed in copending application Serial No. 277,977, filed March 22, 1952, now Patent 2,772,073, "Specimen Heating Means," Maynard J. Columbe, may be used in its place. In the event of certain liquids having extremely low freezing points, it may be desirable to use the reflection type sample holder illustrated in the above-identified application in the place of transmission type shown in the drawings. Irrespective of which type holder is used, the sample is mounted within the passageway in the sample chamber, and the electron beam allowed to impinge thereon where it is diffracted in characteristic concentric rings, the arrangement of which is determined by the crystal structure of the sample.

If desired, a thermocouple indicated at 31 may be disposed in the central passageway 17 formed in the sample chamber for providing an indication of the temperature occurring therein. The leads to the thermocouple are brought out to a pair of terminal posts of the ceramic member 24, and provide easy accessibility to the electrical contact thereto. In this manner, the temperature at which the sample is being examined is available at any instant for purposes of correlation with recorded data obtained by a photographic member or other recording device used with the instrument. In order to insert or remove a sample from the central passageway 17 in the sample chamber, it is necessary to remove the end shield member 26 to make such a change, and as end shield member 26 is held onto the sample chamber by a snap cover arrangement, this is a relatively easy operation, and requires little or no skill.

At the time of changing the sample in the sample chamber it is necessary that the vacuum in the housing 13 be broken in order to remove or insert the new sample to be analyzed. Upon this occurrence it is quite probable that air at atmospheric pressure would rush into the chamber surrounded by the housing 13, and, hence, into the sample chamber due to the fact that this portion of the system would be under vacuum. As such air would contain moisture that might later condense upon the sample and thereby deleteriously affect the results obtained with the attachment, it was necessary that some means be provided to prevent entrance of moisture into the sample chamber. This means comprises a housing 33 that surrounds, and is spaced from the sample chamber 11, and is preferably made from metal tubing, and has centrally disposed apertures formed in the end portions 33a and 33b thereof which are aligned with the electron beam path. The housing 33 is secured to the

free end of a mounting column 34 that has the remaining end thereof threadably secured to a base plate 35. A supply line 35a is provided which has the end thereof terminating in the space intermediate the housing 33 and the sample chamber 11 through a flexible plastic tubing, and is connected to a source of supply of a dry gas through a suitable valving arrangement 36. By this construction, it is possible to introduce a dry gas into the space between the sample chamber and the housing 33 any time that it is necessary to remove a sample from the sample chamber, and by this expedient prevent moist air from entering into the sample chamber. Subsequent reduction of the temperature of the sample chamber, therefore, due to the liquid coolant in the liquid-tight section 16, will not result in frost particles within the sample chamber which would deleteriously affect the results of the analysis. To facilitate insertion and removal of samples within the chamber, the shield member 26 and end portion 33b are connected together by small spacers to form a double door arrangement. This double door is secured to housing 33 by hinges (not shown), and snaps into place when closed.

In order that the central passageway 17 in the sample chamber 11 be maintained in proper alignment with the electron beam emitted by source 14, it is necessary to provide some means for movement of the sample chamber relative to the electron beam path. To provide for such movement, the base plate 35 to which the column 34 and supporting posts 12 are secured, is floatingly supported by means of a set of supporting springs 37 that engage the mounting plate 38 and provide for freedom of movement of the housing in any desired direction. Springs 37 comprise compression springs which tend to force mounting plate 38 and the base plate 35 apart to a degree controlled by means of a set of adjusting screws 39. The mounting plate 38 is removably supported within an opening in the housing 13 on a suitable O-ring gasket arrangement 41, and is rotatable with respect to the housing 13. Because of this construction, freedom for rotation is allowed about the axis of supporting post 12. Additional freedom of movement is provided by means of a flexible bellows 42 having one end secured to the mounting plate 38, and the remaining end thereof secured to the free end of column 34 adjacent the housing 33. On the extreme end of housing 33 a socket 43 is formed which is engaged by the end of a manipulator rod 44 that can be rotated pivotally in any desired direction. Air pressure acting against plate 35 forces the end of the socket 43 into engagement with the manipulator rod so that upon movement of the rod, housing 33, and, hence, the sample chamber 11, can be translated in any desired direction so as to properly position the same with respect to the electron beam. The springs 37 can be adjusted to almost counteract the effect of air pressure on plate 35 so as to minimize the effect of friction. By this construction, the position of the sample with regard to the electron beam path can be controlled with a minimum of effort.

When operating an electron optics instrument with a low temperature attachment such as that illustrated in Fig. 1, it is first necessary to insert a sample in place within the sample chamber 11. To accomplish this, access is provided to the chamber through a suitable port in the side of housing 13. The sample is inserted into position within the chamber with the aid of a pair of forceps after removing double door comprised by the shield members 26 and 33b. After the double door has been removed the sample may be slid into place within the central passageway 17, and the sample chamber and housing subsequently closed over by the respective shield members. During this operation, it is necessary to introduce dry gas into the sample chamber through the inlet conduit 35a so as to prevent moist air from entering into the housing 13, and hence, the sample chamber. In this manner, undue frosting within the sample chamber is pre-

vented. Subsequent to placing the sample within the sample chamber, the sample is properly aligned with respect to the electron beam path by means of the manipulator rod 44, and rotation of the sample chamber by rotating the mounting plate 38. In this operation, the diffraction pattern or micrograph is observed until a sharply defined pattern is obtained on the image forming means whereupon the chamber may be left in this position and a diffraction pattern photograph or micrograph obtained.

From the foregoing description, it can be appreciated that the invention provides a low temperature sample chamber attachment for electron optics instruments whereby the temperature of the sample being examined may be reduced below ambient temperature conditions under which the instrument is operating. This attachment allows samples to easily be inserted and removed within the sample chamber without requiring extreme changes in the temperature of the chamber, or without introducing undesirable agents such as moist air into the chamber which would deleteriously effect the results of the analysis. Additionally, the invention allows a sample to readily be manipulated after once being placed in operative position within the sample chamber, and is relatively simple in construction and inexpensive to manufacture.

Obviously, other modifications and variations of the present invention can be suggested by those skilled in the art in the light of the above teachings. It is, therefore, to be understood that changes may be made herein which are within the full intended scope of the present invention and as defined by the appended claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam so that the electrons impinge upon a sample supported therein, means for reducing the temperature of the sample chamber and sample to a desired temperature value below ambient temperature conditions, and controllable heating means positioned adjacent said sample chamber for controlling the temperature thereof.

2. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam so that the electrons impinge upon a sample supported therein, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of the sample chamber and sample to a desired temperature value below ambient temperature conditions, and controllable heating means positioned adjacent said sample chamber for controlling the temperatures thereof.

3. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam so that the electrons impinge upon a sample supported therein, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of the sample chamber and sample to a desired temperature value below ambient temperature conditions, controllable heating means positioned adjacent said sample for controlling the temperature thereof, an insulating housing surrounding and spaced from said sample chamber and liquid-tight portion thereof, and means for introducing a dry gas into the region intermediate said insulating housing and sample chamber.

4. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path

of the electron beam so that the electrons impinge upon a sample supported therein, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of the sample chamber and sample to a desired temperature value below ambient temperature conditions, controllable heating means positioned adjacent said sample chamber for controlling the temperature thereof, an insulating housing surrounding and spaced from said sample chamber and liquid-tight portion thereof, means for introducing a dry gas into the region intermediate said insulating housing and sample chamber, said insulating housing being flexibly supported on one end by a retractable bellows secured to a rotatable mounting plate with said sample chamber being secured to said housing, a set of biasing springs acting between said mounting plate and said housing for forcing the housing in a desired direction, and a translatable manipulator rod engaging the free end of said housing for moving the same against the action of said biasing spring.

5. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam, said sample chamber having a passageway therethrough for accommodating the electron beam, a removable sample holder supported in said passageway for mounting a sample to be analyzed in the electron beam path, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of said sample chamber for reducing the temperature of the sample to a desired temperature value below ambient temperature conditions, and controllable heating means positioned adjacent said sample chamber for controlling the temperature thereof.

6. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam, said sample chamber having a passageway therethrough for accommodating the electron beam, a removable sample holder supported in said pas-

sageway for mounting a sample to be analyzed in the electron beam path, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of the sample chamber to a desired temperature value below ambient temperature conditions, an insulating housing surrounding and spaced from said sample chamber and liquid-tight portion thereof, and means for introducing a dry gas into the region intermediate said insulating housing and sample chamber.

7. A low temperature attachment for electron optics instruments of the type utilizing an electron beam for examining a specimen under consideration comprising a sample chamber adapted to be supported in the path of the electron beam, said sample chamber having a passageway therethrough for accommodating the electron beam, a removable sample holder supported in said passageway for mounting a sample to be analyzed in the electron beam path, a liquid coolant disposed in a liquid-tight region of said sample chamber for reducing the temperature of the sample chamber and sample to a desired temperature value below ambient temperature conditions, controllable heating means positioned adjacent said sample for controlling the temperature thereof, an insulating housing surrounding and spaced from said sample chamber and liquid-tight portion thereof, means for introducing a dry gas into the region intermediate said insulating housing and sample chamber, said insulating housing being flexibly supported on one end by a retractable bellows secured to a rotatable mounting plate with said sample chamber being secured to said housing, a set of biasing springs acting between said mounting plate and said housing for forcing the housing in a desired direction, and a translatable manipulator rod engaging the free end of said housing for moving the same against the action of said biasing spring.

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