United States Patent

[11] **3,582,221**

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[73] Assignee **The United States of America as represented by the Secretary of Agriculture**

[54] **SAMPLE HOLDER FOR INFRARED SPECTRA APPARATUS**
1 Claim, 8 Drawing Figs.

[52] U.S. Cl. **356/244,** 269/321, 350/95
[51] **Int. Cl.** **G02b 21/34**
[50] **Field of Search** 269/321; 356/244; 350/95

[56] **References Cited**
UNITED STATES PATENTS
2,265,002 12/1941 Park 356/244

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ABSTRACT: This invention relates to a sample holder useful for obtaining the infrared spectra of the short staple fibers of either a natural or synthetic fiber. The method does not require cutting, grinding, or sifting the fibers or the addition of any extraneous material. Sample preparation involves shaping a thin layer of parallel fibers under pressures comparable to those used in preparing KBr discs. The sample holder is multipurpose being operable to immobilize the fibers before, during, and after the pressing operation.

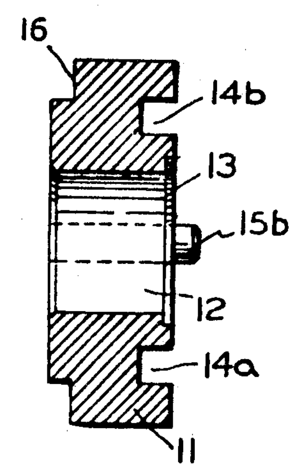

FIG. 2

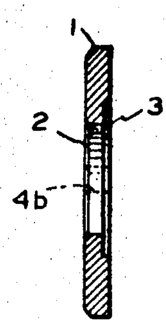

FIG. 1

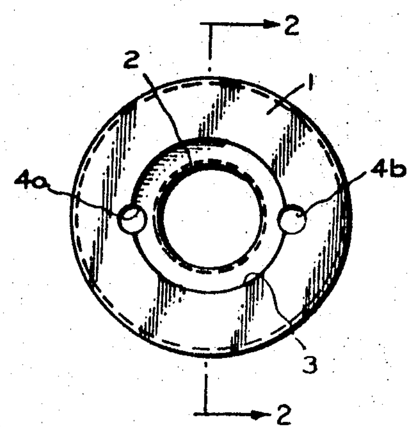

FIG. 4

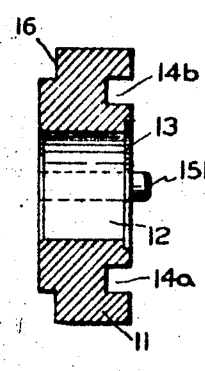

FIG. 3

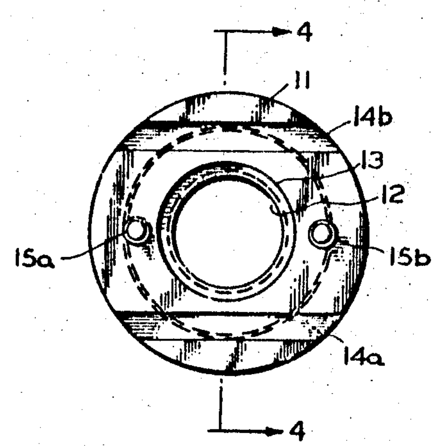

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FIG. 5

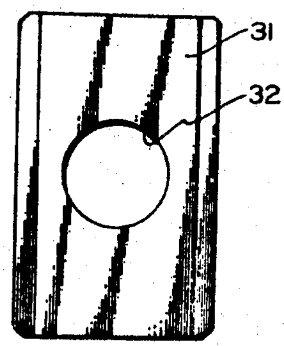


FIG. 6

FIG. 7

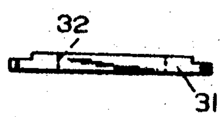
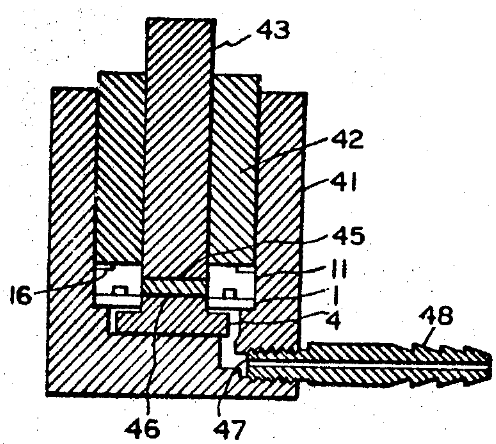


FIG. 8



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SAMPLE HOLDER FOR INFRARED SPECTRA APPARATUS

A nonexclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

Infrared spectroscopy is well recognized as a valuable analytical method in the study of both natural and synthetic fibers and of polymeric material. A source of difficulty in obtaining infrared spectra of textile fibers is the preparation of the sample for analysis. Among the most common methods used to prepare samples of natural and synthetic fibers for infrared analysis are the alkali-halide (usually potassium bromide) disc, the mull, and the thin-film techniques. In the disc technique, the sample is ground or cut finely and then, after mixing with an adequate amount of alkali halide, pressed into a disc. This method has certain disadvantages, such as grinding or cutting the fiber, which gives a random orientation in the sample, as well as the adsorption of water by the alkali halide which obscures important details of the spectrum because of the infrared absorption by the water. In the mull technique, the fibers are ground together with a suitable oil between two ground glass plates. With a mull, there is the unwanted absorption by the oil. In the preparation of a thin film from a solution, it is not always possible to find a suitable solvent, and it is usually difficult to completely remove the solvent. Also, the physical structure of the fibers is destroyed. A method of preparing samples for infrared analysis by pressing sifted fiber material between two cylindrical dies has previously been reported. Briefly, a sample of the fibers is cut into 1—2 mm. lengths, and the cut fibers are sifted through a wire mesh (200—400 mesh/in.) onto the surface of a pellet die. The layer of sifted fibers is pressed in the die as usual. This method eliminates the addition of extraneous material, but it does not eliminate grinding and shifting of the fibers. Methods have also been reported in which a single-fiber thickness grid of fibers has been prepared for infrared analysis. This latter technique requires either single filaments, 4—6 inches in length or a continuous filament which is mounted in a holder device in a parallel manner of a single-fiber thickness.

We have developed a method in which a thin pressed film of essentially parallel fibers of the short staple fibers of either a natural or synthetic material can be obtained for infrared analysis. The significance of this method is that infrared spectra of short staple fibers can be obtained and that it is not necessary to cut, grind, or sift the fibers or to add any extraneous material. In addition, the fibers are obtained in a highly oriented manner, which should be very useful for polarization studies. Excellent spectra of both natural and synthetic fibers have been obtained with this method. The press accessory and its parts are shown in FIGS. 1—8. The sample holder was constructed so that it could be used with a commercially available KBr pellet press, and it is essentially a holder for maintaining a thin, close-packed, parallel fiber arrangement before, during, and after pressing. After a sample is pressed, the holder device with the pressed sample is removed from the die press and placed on a suitable adapter plate for infrared analysis.

In order that the invention may be more readily understood, reference is made to the following description and to the accompanying drawings in which:

FIG. 1 is a face view of one of the first of 2 cooperating components of the sample holder.

FIG. 2 is a cross section taken on line 2–2 of FIG. 1.

FIG. 3 is a face view of the second of 2 cooperating components of the sample holder.

FIG. 4 is a cross section taken on line 4–4 of FIG. 3.

FIG. 5 is a face view of an adapter plate element for use primarily in fitting the assembled sample holder components of FIGS. 1 and 3 to an infrared spectrometer.

FIGS. 6 and 7 represent side and end views, respectively, of the adapter of FIG. 5.

FIG. 8 represents a cross section of an assembled press, including the sample holder of FIGS. 1 and 3, in which the fiber sample is prepared prior to transfer to the spectrometer.

Referring specifically to FIGS. 1 and 2, represents the first component 1 of the fiber sample holder, has a circular opening 2 which passes axially and concentrically through the primary component, a shallow fiber sample accommodating shoulder 3 cut into the face of the component at the circular opening intersect, a pair of index orifices 4*a* and 4*b*.

Referring to FIGS. 3 and 4, the body 11 of this component has a circular opening 12 which passes axially and concentrically through the second component, a shallow fiber sample accommodating shoulder 13 cut into the face of component 11 at the circular opening intersect (this is the counterpart of a similar item on the first component), two parallel channels 14*a* and 14*b* of rectangular cross section cut as chords entirely across the face of the second component one on either side of the circular opening, a pair of index pins 15*a* and 15*b* located intermediate the parallel channels, extended normal the face of the second component for a distance not exceeding the thickness of the first component and conforming with the counterpart index orifices 4*a* and 4*b* of the first component with respect to size and shape, and a shoulder 16 cut into the periphery of the secondary component.

Referring now to FIGS. 5, 6 and 7 adapter plate component 31 which serves incidentally as a spacer unit during assembly of the sample holder accessory and the several press components and primarily as a holder for the mated first and secondary components together with the included fiber sample after pressing, adapting, and fitting same to an infrared spectrometer. The body of adapter plate 31 is so sized and so shaped as to be accepted within the sample holder slot of the spectrometer. A circular opening 32 passes centrally through the adapter plate and is sized to accept as a slip-fit the shoulder 16 cut into the periphery of the second component 11.

The two essential components of the device are the first and the second components 1 and 11. In the final assembly the second component 11 is the upper part and the first component is the lower part of the fiber holder. In practice, the fibers, after combing into a parallel tuft, are placed on the second component 11 and the first component 1 is then located by use of pins 15*a* and 15*b* on the second component 11. After pressing, the device is placed in the adapter plate component 31 which component fits the holder on the infrared spectrophotometer.

The fiber holder is employed as described below with reference to FIG. 8. As already indicated, FIG. 8 represents the assembled press, including sample holder components 1 and 11.

Referring specifically to FIG. 8, the assembled press is seen to comprise a press casing 41, a press barrel 42, a press plunger 43, and lower and upper press dies 44 and 45, respectively. An orifice 47 is provided near the bottom for exhausting air during pressing. Nozzle 48 screws securely into orifice 47 and is provided for attaching the assembled press to a vacuum pump (not shown). FIG. 8 shows the press assembled for pressing a single layer fiber sample 46 while it is being held between the two holder components 1 and 11.

The sample for infrared absorption measurement is prepared as follows:

Barrel 42 is first placed over hole 32 of adapter plate 31. Plunger 43 is then inserted into barrel 42 so that it passes through the hole 32 in the adapter plate component 31, and the second component 11 of the fiber holder is placed atop the barrel 42. It is essential that press plunger 43 be level with the top of second component 11.

A small bundle of fibers is combed with a finetooth metal comb until an evenly distributed, thin layer of parallel fibers approximately 3 to 5 fibers thick and wide enough to cover the hole 12 in the holder is obtained.

A convenient method for restraining the combed fibers on the holder component is to prepare small rolls of gummed tape (adhesive side outward) and not shown install a roll in each of the channels 14*a* and 14*b* cut into the face of the second component 11.

One end of the combed tuft is pressed down onto the adhesive in one channel of the second component **11**. The other end is then stuck to the adhesive material in the opposite channel. The first component **1** is then placed on top of the second component.

The lower die **44** of the press is placed in the exposed hole of the first component **1** so that the mirrored surface of the die rests on the fibers which are supported by the plunger **43**. Adapter plate **31** must be removed at this point.

The KBr die casing **41** is inverted and carefully placed on the assembled barrel, holder, and lower die. The entire assembly is moved to the edge of the table so that the plunger **43** may be held from beneath. While holding the plunger, the entire assembly is inverted.

The plunger **43** is carefully removed, the upper die **45** inserted with care, and the plunger **43** replaced. Pressure is then applied to fibers **46** for 1—5 minutes at 2—12 tons.

Plunger **43** and barrel **42** are then removed from the die casing **41**. A large pair of tweezers is a convenient tool to keep the fiber holder in place, while the casing is inverted so that the upper die **45** can drop out.

A cork fitted with a handle (not shown) is inserted tightly into the hole **12** of the second component of the fiber holder and the entire fiber holder with the pressed film is removed carefully from the die casing **41**. The film **46** is fragile and may break if caution is not used in removal.

The holder with the pressed film **46** is again placed in the adapter plate **31** component for infrared analysis.

We claim:

1. In a multipurpose accessory for positionally immobilizing a plurality of aligned fibers prior to pressing, during pressing, and operable subsequent the pressing as a sample holder for the aligned and pressed fibers, the combination of:

a. a first generally disc-shaped component with a first circular face, a second circular face axially parallel thereto, and with intervening curvilinear surface jointly defining a right cylinder, said first component sized to allow a slip-fit through the barrel portion of a separate external press device, said first component having
   1. a circular opening passing axially and concentrically therethrough, said opening being provided with a thin sample accommodating shoulder cut into the first circular face at the circular opening intersect, and said circular opening sized to allow slip-fit passage for the lower die portion of a separate, external press device,
   2. index orifice means passing axially through the first component adjacent the circular opening;

b. a second component generally disc-shaped with a first circular face, a second circular face axially parallel thereto, and with the intervening curvilinear surface jointly defining a right cylinder, said second component sized to allow a slip-fit through the barrel portion of said separate external press device, said second component having
   1. a circular opening passing axially and concentrically therethrough, said opening being provided with a thin sample accommodating shoulder cut into the first circular face at the circular opening intersect, and said circular opening being sized to allow slip-fit passage for a plunger and upper die portions of said separate, external press device,
   2. two parallel channels of rectangular cross section cut as chords entirely across the first circular face on either side of the circular opening,
   3. index pin means located intermediate the channels, extended normal the first circular face for a distance not exceeding the thickness of the first component and conforming with the counterpart index orifice means of the first component with respect to number, size, and shape, and
   4. a uniform rectangular shoulder cut into the peripheral edge of the second circular face; and

c. an adapter plate component, rigid and generally rectangular in shape, sized to serve as a sample holder in a separate, external, infrared spectrometer, said adapter plate component provided with a centrally located, circular opening, said opening sized to permit a slip-fit for the shoulder cut into the periphery of said second component.