Dec. 16, 1958 L. V. SORG ET AL 2,864,725

FLUID AND SYSTEM FOR PREPARING HYDROGEN SULFIDE SENSITIVE TAPE

Filed Aug. 15, 1956 2 Sheets-Sheet 1

Fig. 1.

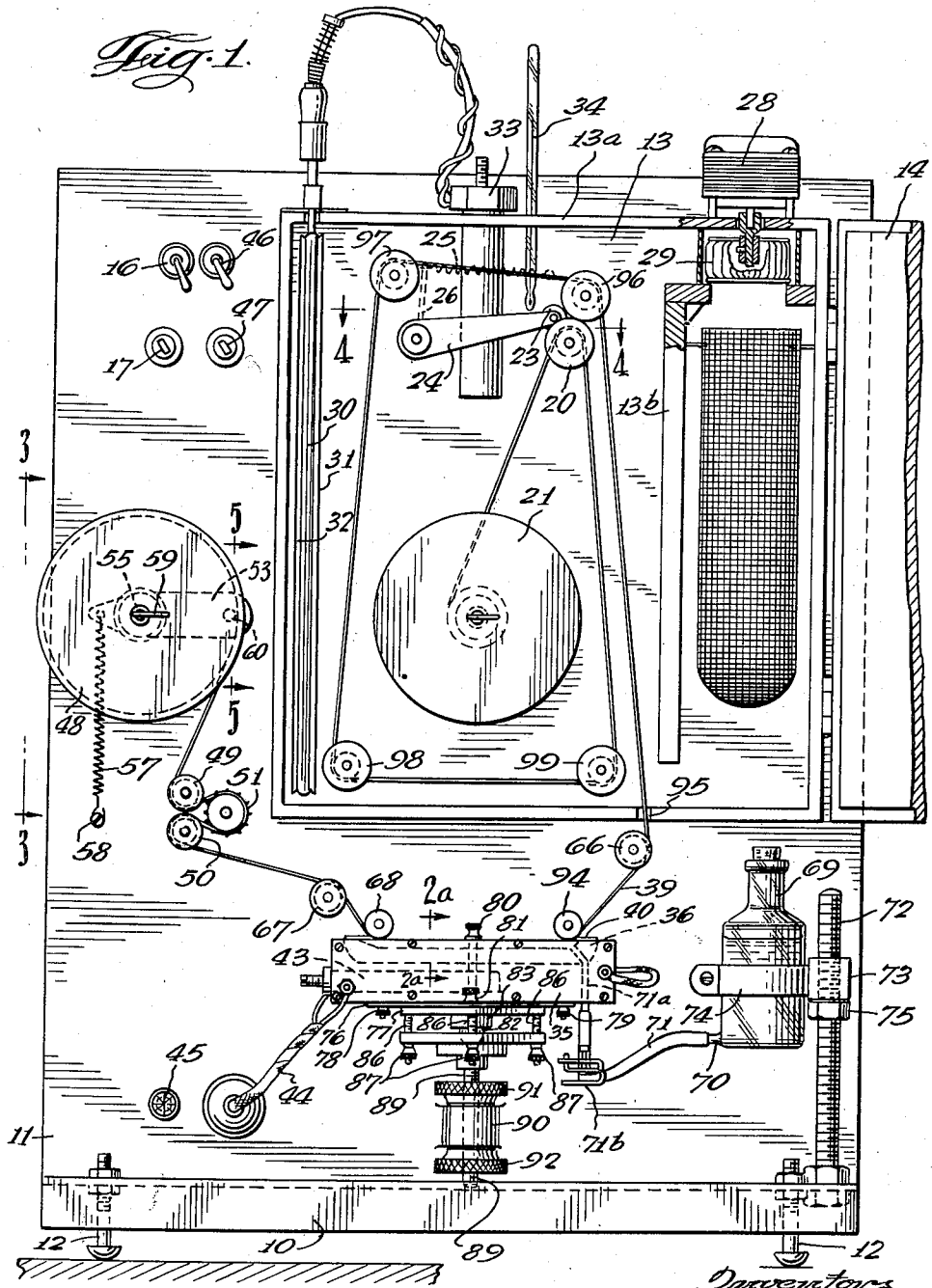

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Fig. 3.

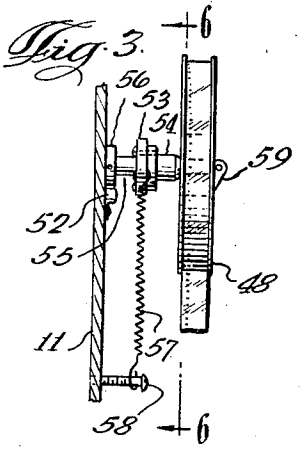


Fig. 7.

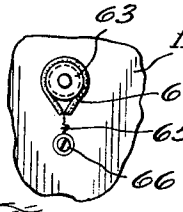


Fig. 2.

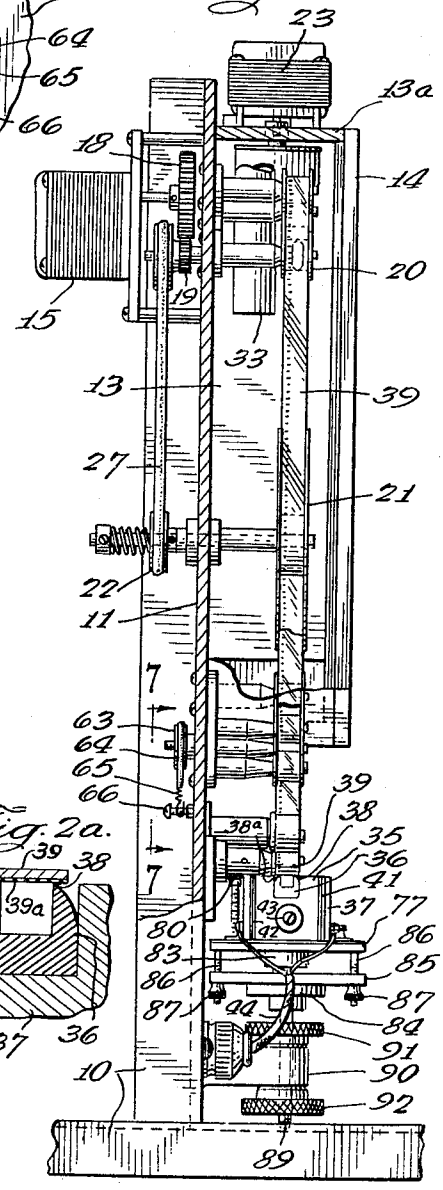


Fig. 5.

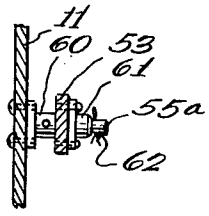


Fig. 4.

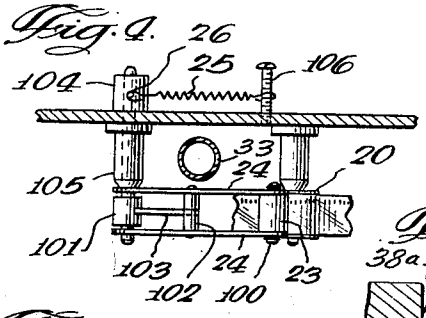
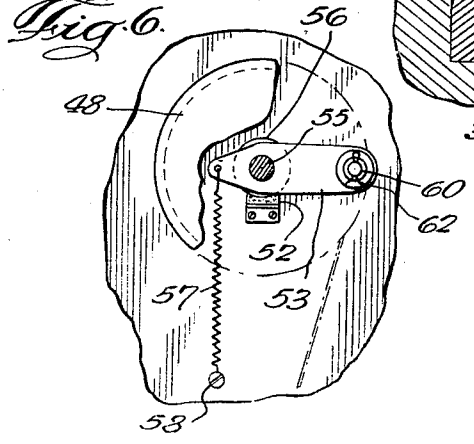


Fig. 2a.

Fig. 6.



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2,864,725

FLUID AND SYSTEM FOR PREPARING HYDROGEN SULFIDE SENSITIVE TAPE

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Application August 15, 1956, Serial No. 604,169

5 Claims. (Cl. 117—83)

This invention relates to the continuous treatment of strip material and, in particular, comprises improved apparatus for sensitizing one coated surface of tape or other strip material.

The manufacture of our sensitive film includes the steps of applying a liquid sensitive gelatin coating on a film support such as a continuous transparent and fluid-impervious tape. Subsequently, the coated tape is sensitized by impregnating the gelatin with a reactive solution.

In connection with a hydrogen sulfide analyzer-recorder described in our co-pending patent application Ser. No. 375,032, now issued as U. S. 2,800,397, we employ a sensitive-to-hydrogen-sulfide tape which comprises an acetate film base having a gelatin coating. This corresponds to motion picture film from which the photosensitive silver has been removed. By impregnation, we place a certain amount of lead acetate and sodium acetate into the gelatin coating.

An important object of the invention is to provide an improved apparatus for continuously feeding tape or other strip material over a treating solution to effect a controlled impregnation of a gelatin coating thereon. A further object of the invention is to provide an apparatus for the rapid, accurate and uniform treatment of the gelatin coating without injury or mutilation of the base strip material. A more specific object of the invention is to provide an apparatus which prevents the sensitized or gelatin side of the tape from coming in contact with the parts of the apparatus along which it moves to permit the use of relatively small amounts of treating solution, to provide means for maintaining the surface of the solution at a constant level, and to maintain the solution at the desired temperature.

Another object of the invention is to provide a system utilizing a sensitizing station and a drying chamber with means for feeding the supporting tape through the station and chamber in series so that the tape will be maintained in such a position that the sensitized gelatin coating is dried without contacting any portion of the apparatus. Still another object is to provide a system which controls the temperature and circulates air in the drying chamber whereby there is a progressive drying of the coating to produce a non-tacky sensitized strip. These and other objects of our invention will become apparent as the description thereof proceeds.

Briefly, we attain the objects of our invention by providing means for successively impregnating and drying a gelatin coating on a fluid-impermeable tape or base material which includes a temperature-controlled impregnating station which is provided with means for accurately maintaining the level of the impregnating solution such that it will contact the gelatin-coated surface of the moving tape without submerging the uncoated surface of the tape during the impregnating step. The drying chamber means includes a thermostatic heating unit paralleling a substantial span of the treated tape. Further details of construction and advantages of our apparatus will be described in conjunction with the accompanying drawings illustrating a preferred embodiment of the invention and wherein:

Figure 1 is an elevation, partly in section, showing one form of apparatus for impregnating a gelating coating on a continuous support and for drying the impregnated coating;

Figure 2 is a side elevation, partly in section, of the apparatus shown in Figure 1;

Figure 2*a* is an enlarged fragmentary section of the trough;

Figure 3 is a detail taken along the lines 3—3 in Figure 1;

Figure 4 is a detail taken along the lines 4—4 in Figure 1;

Figure 5 is a detail taken along the lines 5—5 in Figure 1;

Figure 6 is a detail taken along the lines 6—6 in Figure 3; and

Figure 7 is a detail taken along the lines 7—7 in Figure 2.

Similar reference numbers refer to corresponding parts in the several views of the drawings.

Referring to the drawings, the framework 10, comprising angle irons, supports 0.25 inch aluminum panel 11 upon which all of the components of our apparatus are mounted. Four bolts or feet 12 extending through the lateral surfaces of the angle irons 10 permit adjusting the apparatus to a level position.

A compartment 13 comprised of walls 13*a* and hinged cover 14 comprises the drying chamber.

A motor 15, controlled by switch 16 and fused by 17, provides the drive for moving the film or tape through the instrument. Gears 18 and 19 driven by motor 15 drive sprocket 20 and take-up reel 21 through a friction clutch 22 driven through belt 27. An idler 23 presses the film into engagement with sprocket 20 thereby preventing tape slippage. Tension is given to the idler 23 through pivoted arm 24 which is under tension from spring 25 attached to lever 26.

Electric heater 30 furnishes heat to control the temperature of the cabinet 13 to a constant value and motor 28 drives the squirrel cage blower 29 which circulates air through the drying compartment 13 about baffle 13*b*.

The heater 30 is shown in Figure 1, but for simplicity has been omitted from Figure 2. Radiating fins 31 and 32 are attached to heater 30 to aid in conduction of the heat. The temperature within the drying chamber 13 is maintained constant by thermal regulator 33 and the temperature is indicated by thermometer 34.

The treating or impregnating station 35 includes the trough 36 within constant temperature bath 37. The trough 36 is provided with paralliel knife edges 38 and 38*a* which act like a ruling pen (see Figure 2*a*) permitting a 0.375 inch trace of chemicals to be impregnated in the gelatin coating on the tape 39. Excess liquid is removed from the gelatin surface by transverse scraper 40.

The constant temperature bath 37 comprises an aluminum block and the temperature is maintained constant by strip heaters 41 and 42 which are controlled by thermal regulator 43, electrical power being brought to the heating elements by lead 44. Pilot light 45 indicates when the current is on. Power is furnished to motor 28 and heaters 30, 41 and 42 through switch 46, the circuit being fused through 47.

Film or tape 39 from the supply reel 48 passes over rollers 49 and 50 which maintain tension on sprocket 51. The supply reel 48 is maintained against a felt brake 52 shown in Figures 3 and 6 and the brake 52 is actuated through lever 53 to which is attached bearing 54. Attached to shaft 55 which rides in bearing 54 is drum 56 which is held against brake pad 52 by spring 57 which is anchored by pin 58. The film supply reel 48 is held onto shaft 55 by clamp 59.

Referring to Figure 5, the lever arm 53 is supported

by the panel **11**. Shaft **55***a* is held in socket **60** which is fastened to panel **11**. A bearing surface for shaft **55***a* is provided by collar **61**, the whole assembly being held onto the shaft **55***a* by pin **62**.

Sprocket **51** furnishes additional braking action against the tape **39** from pulley **63**, Figures 2 and 7, on which rides a small section of round leather belting **64** with tension applied by spring **65** which is anchored by pin **66**. All of these wheel-like mechanisms are supported to the panel **11** by individual bearing members as shown in Figure 2.

Guide rollers **66** and **67** carry the tape **39** parallel to the exposed upper surface of the impregnation trough **36** and allow the trough **36** to be vertically adjusted so that the gelatin-coated surface of the tape **39** moves over the liquid surface in the trough **36**.

The impregnating solution is retained in leveling bottle **69** provided with a tube connection **70** which permits the solution to flow through the plastic tubing **71** into trough **36**. The liquid level in trough **36** is raised or lowered by positioning bottle **69** along the threaded rod **72** by collar **73**, which supports bottle clamp **74**, the selected position being secured by nut **75**.

The coating station **35**, including the trough **36** and the constant temperature bath **37**, is adjusted by plate **76** which is fastened to platform **77** by knurled nuts **78** and **79**. Loosening the knurled nuts **78** and **79** permits plate **76** to be positioned with respect to panel **11** through slots therein.

Lateral movement of station **35**, parallel to panel **11**, is controlled by the studs and knurled nuts **80** and **81** which are fastened to the trough assembly or station **35** through slots in plate **76**.

The entire trough assembly **35**, plate **76** and platform **77** rest on steel ball **82** which in turn operates in socket **83** in platform **77** and lower socket **84** in sub-platform **85**. Stud bolts **86** and knurled nuts **87** secure the platform **77** and the sub-platform **85** together to clamp the steel ball **82** therebetween. Adjustments can be made so as to level the trough assembly **35** and to dispose the trough **36** with respect to the panel **11** and the path of travel of the tape **39** to obtain the desired contact of the gelatin coating with the liquid surface in the trough **36**.

Platform **77** and sub-platform **85** are triangular sections, the apex of the triangular facing the viewer. Depending from sub-platform **85** is a threaded shaft **89** passing through support collar **90** which is rigidly secured to panel **11**. Knurled nuts **91** and **92** on opposite sides of the collar **90**, through which the shaft **89** freely passes, permits the entire assembly of platforms **77** and **85** through assembly **35** to be raised or lowered and fixed in the desired position.

The unsensitized film is drawn from supply reel **48** through sprocket assembly **49**, **50**, **51** over roller **67** along knife edges **38** and **38***a* and under rollers **68** and **94**, the excess solution being removed from the gelatin coating by scraper **40**.

After the gelatin coating on the tape **39** has been impregnated with the desired chemicals it passes over positioning roller **66** and enters the drying chamber **13** through the port **95**. Time for drying the coating is obtained by passing the slowly moving film over positioning rollers **96**, **97**, **98**, **99** and drive roller **20**. Thence, the film **39** is collected on takeup reel **21**, the temperature-controlled air circulated by blower **29** having dried the gelatin coating on the film. It will be noted that after the tape **39** leaves the scraper **40** the gelatin side of the tape **39** does not touch any component of the apparatus or another portion of the tape **39** until it is fully dried and being accumulated on the take-up reel **21**.

A cross-section of the drive sprocket **20**, idler **23** and lever arm **24** is shown in Figure 4. The idler **23** is pinned to a pair of levers **24** by a pin **100**, the other end of the levers **24** operating against the hub and shaft **101**. Hub **101** is pinned to retainer bar **102** by rod **103**, the entire assembly being fastened to collar **104** through the hub and shaft **101** which passes through bearing **105**. The force lever **26** is fastened to pin **106** through spring **25** which furnishes the necessary tension.

The described apparatus has been used successfully for the preparation of sensitized tape for use in connection with the hydrogen sulfide analyzer-recorder described in our co-pending application Serial No. 375,032, filed August 18, 1953, now Patent No. 2,800,397 and entitled "Hydrogen Sulfide Analyzer Recorder."

The tape **39** comprises a uniformly transparent strip having a sensitized plastic absorptive coating such as of gelatin or agar agar. The strip is of non-reactive, non-porous, non-absorptive flexible material such as cellulose acetate. A suitable base strip for our hydrogen sulfide sensitive tape is 16 mm. movie film having marginal sprocket-receiving perforations for driving the tape through the sensitizing apparatus described herein and through the hydrogen sulfide analyzer-recorder referred to above.

The tape is passed along the trough **36** at a rate of about 45 feet per hour and immediately introduced into the drying chamber **13** wherein a temperature of about 113° F. is maintained, the dried film being accumulated on reel **21**. Under these conditions, a uniform impregnation of the gelatin is obtained, the desired concentration of lead acetate being between about 0.25 to 0.30 mg. per sq. cm. which corresponds to 12 to 14 mg. per foot of 16 mm. tape.

In preparing such sensitized tape the blank film **39** is fed from supply reel **48** and the gelatin coating thereof contacted at a temperature of about 113° F. in trough **36** with a solution of lead acetate and sodium acetate.

The solution for sensitizing the plastic coating is 1 normal lead acetate and 1 normal sodium acetate at a pH of about 6.7. The pH is important because if too much acidity is present, sensitivity to hydrogen sulfide is decreased whereas if the acidity is too low, the lead will precipitate from the sensitizing solution with the result that substances such as mercaptans will have greater effect on the staining of the tape. The sensitized coating obtained from the defined solution stains uniformly, has the desired optical density for a given concentration of hydrogen sulfide, and has low susceptibility to variations in optical density.

The coating is not light-sensitive and retains its transmittancy when exposed to the light beam and photoelectric cell arrangement used to monitor the moving tape as described in our co-pending application.

The film may be stored for long periods of time without impairing its sensitivity to hydrogen sulfide. Our tape is uniform with respect to its buffered lead acetate content and is light-, air-, and age-stable when prepared pursuant to our system.

A number of batches of tape produced in accordance with the above were examined with respect to the uniformity of the sensitized film coating. In making such examination, the gelatin was stripped from the base tape and treated with hydrochloric acid to produce an extract containing the dissolved lead acetate. The lead content was then determined by means of a polarographic apparatus with the results illustrated by the data shown in the following **table:**

Table I

Batch	Lead Acetate Content per Foot of 16 mm. Tape
	Mg.
A	13
B	14
C	14
D	13

The apparatus described may be used on 16-mm. or

35-mm. film when the size of the coating trough **36**, the block **37**, the pad rollers, the supply and take-up reels, and the tension and drive sprockets are appropriately provided.

This application is a continuation-in-part of our co-pending applications Serial No. 375,032, now issued as U. S. 2,800,397, and Serial No. 556,238, filed December 29, 1955, entitled "Apparatus for Treating One Surface of a Strip," issued as U. S. 2,884,333.

Although we have described our invention by reference to a preferred embodiment thereof illustrated in the drawings, it should be understood that this is by way of example only. Further, it is contemplated that modifications in the apparatus and mode of using the apparatus can be made without departing from the scope and spirit of the described invention.

What we claim is:

1. The method of preparing a gelatin-coated tape which is quantitatively sensitive to hydrogen sulfide and which is light-, air-, and age-stable which comprises the steps of sensitizing a gelatin coating supported by a transparent gas-impervious tape with 0.25 to 0.30 mg. lead acetate per sq. cm. from an aqueous solution comprising 1 normal lead acetate and 1 normal sodium acetate by exposing only the coated surface to a pool of the solution, and subsequently drying the sensitized gelatin coating.

2. The method for preparing a hydrogen sulfide sensitive fluid-impervious transparent tape, said tape supporting a water absorptive coating which contains from 0.25 to 0.30 mg. lead acetate per sq. cm. and is quantitatively sensitive to hydrogen sulfide, the steps which comprise contacting the coating with an aqueous sensitizing solution by moving said tape over an elongated body of the solution with said coating running over the surface of said body, said body comprising an aqueous solution of 1 normal lead acetate and 1 normal sodium acetate maintained at a pH of about 6.7, removing excess solution from the coating as it emerges from said body, and

drying the sensitized tape by passing it through a drying zone.

3. A fluid adapted to sensitize a gelatin coating on a transparent tape to render it quantitatively responsive to hydrogen sulfide concentrations comprising an aqueous solution of 1 normal lead acetate and 1 normal sodium acetate maintained at a pH of about 6.7.

4. The method of preparing a gelatin-coated tape which is specifically responsive to hydrogen sulfide and which is light-, air-, and age-stable which comprises the steps of impregnating a gelatin-coated transparent gas-impervious tape with 0.25 to 0.30 mg. lead acetate per sq. cm. by contacting the gelatin coating directly with an extended surface of a pool of a solution comprising 1 normal lead acetate and 1 normal sodium acetate at pH of about 6.7, and subsequently drying the treated gelatin coating.

5. The process of impregnating a supported gelatin coating with 0.25 to 0.30 mg. lead acetate per sq. cm. deposited from an aqueous solution of 1 normal sodium acetate and 1 normal lead acetate at pH about 6.7 which comprises maintaining an elongated body of liquid-impregnating solution, passing the gelatin coating in contact with the surface of said solution, removing excess solution from the gelatin coating at a point adjacent at end of said body and returning the removed excess to said body, and drying the impregnated gelatin coating by passing through a drying zone.

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