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(11) **EP 1 043 157 A2**

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
11.10.2000 Bulletin 2000/41

(51) Int Cl.7: **B41F 33/00**

(21) Application number: **00106883.2**

(22) Date of filing: **31.03.2000**

(84) Designated Contracting States:
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE**
Designated Extension States:
AL LT LV MK RO SI

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(30) Priority: **02.04.1999 IT TO990262**

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(54) **An integrated system for evaluating the residual solvent contained in packaging films during the printing process at the manufacturing plants**

(57) An integrated system for evaluating the residual solvent in packaging films during the printing process at the manufacturing plants, comprising:

- a device (17) for the automatic on-line taking of the

sample (19) to be analysed;

- a single analyser (21) that embodies the functions of three laboratory instruments: headspace or desorption unit, gas chromatograph and data system, with comparable analytical results.

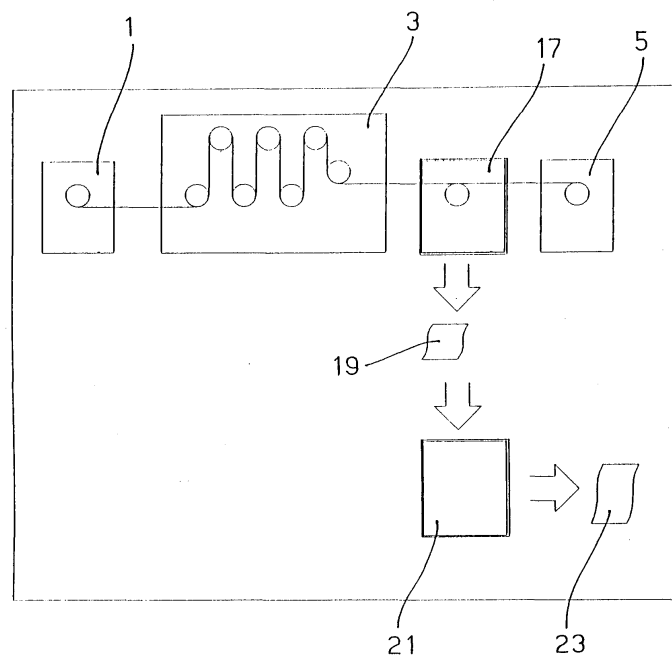


FIG. 2

Description

[0001] The present invention concerns an integrated system for evaluating the residual solvent contained in packaging films during the printing process at the manufacturing plants.

[0002] It is known that the determination of the residual solvent in packages, particularly packages used for foodstuffs, is very important for the companies operating in the field.

[0003] The prior art manufacturing plants generally comprise an unwinder 1 of the reel to be worked, a set of inking groups 3, each equipped with heated chambers for solvent vaporisation, and a rewinder 5 of the finished product (see Fig. 1).

[0004] According to the above prior art, the determination of the residual solvent is carried out in laboratories by using an analytical system generally comprising a headspace sampler or thermal desorption unit coupled with a gas chromatograph. At the end of or during film working, an operator takes a film piece 7, seals it into an envelope (usually of aluminium) and sends the piece to the laboratory. There, a sample 9 (usually, a square or round sample with an area of about 100 cm²) is obtained by means of a punch. Sample 9 thus obtained is inserted into a test tube 11 having a pierceable septum. Then test tube 11 is inserted into a headspace sampler 13 or desorption unit and incubated for about 10 to 30 minutes. During this time, solvent migration to the air within the tube takes place. The analysis of that air provides data 15 about the residual solvent concentration in the product.

[0005] The above system for determining the residual solvent is particularly laborious and generally time consuming; also, the analysis equipment demand complex calibration procedures, where error possibility is not unlikely. Indeed, it is necessary to manually prepare some vials with known concentration by injecting a quantity of sample gas dosed through a syringe. All this is not of assistance in the manufacturing process since the analysis results are known after the working has been completed. If the values are not within prescribed law limits, the finished product has to be worked again or discarded.

[0006] It is an object of the present invention to obviate the drawbacks of the prior art, by providing a system allowing a quick determination of the residual solvent amount, so as to reduce the waste to a minimum since it is possible to timely act to correct the manufacturing system.

[0007] It is another object of the present invention to allow a strong reduction of the solvent evaluation time, by setting up a sample taking system coupled with a fast analyser to be located close to the manufacturing process.

[0008] The system of the invention substantially comprises two elements, namely:

- a device for the automatic on-line taking of the sample to be analysed;
- a single analyser that embodies the functions of the three aforementioned laboratory instruments, i. e. headspace or desorption unit, gas chromatograph and data system, with comparable analytical results, and that provides for:
 - the sample incubation for the residual solvent extraction;
 - the analysis of the gaseous sample in order to obtain a chromatogram;
 - the processing of said chromatogram in order to obtain the measures.

[0009] Of course, said analyser can be coupled with a printer for printing the analysis certificate or with a host computer for data transfer.

[0010] Further characteristics of the above system will become apparent from the appended claims.

[0011] The system of the invention will now be disclosed in detail with particular reference to the accompanying drawings, given by way of non-limiting example and showing some preferred embodiments of the invention. In the drawings:

- Fig. 1 is a block diagram showing the manufacturing system according to the prior art;
- Fig. 2 is a block diagram showing the manufacturing system according to the present invention;
- Fig. 3 is a perspective view of a device for automatically taking the film sample;
- Fig. 4 is a block diagram showing the analyser according to the invention;
- Fig. 5 is a graph showing the ion current (V) due to combustion of the carbon atoms present in the solvent, versus time (t).

[0012] As mentioned before, Fig. 1 shows the way of operation according to the prior art. Said prior art has been already disclosed at the beginning of the present specification and repeating it is not necessary. Only some key features of the prior art method, present in the diagram of Fig. 1, are recalled now.

[0013] Reference 1 is the reel unwinder; reference 3 is the seat where the printing process on the film takes place, the seat housing a set of ink applying groups; and reference 5 is a film rewinder. Reference 7 is a film piece, introduced into a suitable sealed envelope; reference 9 is the film sample obtained from said piece 7; and reference 11 is the vial of the headspace sampler. From the sampler, vial 11 is sent to laboratory 13 where the aforementioned analyses (headspace or desorption unit, coupled with a gas chromatograph) take place; reference 15 denotes the results of said analyses.

[0014] Fig. 2 shows on the contrary the method according to the present invention. References 1, 3 and 5 denote the reel unwinder, the seat where the printing process takes place, and the rewinder, respectively, like

in Fig. 1. Yet, at the output of printing process seat 3, upstream rewinder 5, a device 17 is provided for automatically taking the calibrated film sample 19, which is sent to a single analyser 21, which will provide the analysis result 23, i. e. the measure of the residual solvent.

[0015] It is evident that by this system the film sample manipulations are considerably reduced, in that both the sample taking and the fast measurement thereof are made possible within the manufacturing process.

[0016] The availability in very short time of the data about the residual solvent concentration in the film has the following advantages:

- reduction of the number of operations for sample manipulation;
- possibility of a fast correction of the manufacturing process;
- possibility of increasing the number of checks on the residual solvent for a more effective manufacturing quality control;
- elimination of the solvent loss by the sample because of the waiting times for the sample transfer from the manufacturing plant to the laboratories;
- provision, in the analysis system, of a readily executable internal automatic calibration, which eliminates the present need for creating calibrated samples in vials to be analysed as references.

[0017] Fig. 3 shows an embodiment of the device for automatically taking film samples from a reel being worked.

[0018] It is evident that such device for automatically taking a film sample from reels being worked can be employed also for other applications, different from the one described here, and indeed said device is the subject matter of a copending patent application in the name of the present Applicant. Said device is disclosed only by way of non-limiting example, since it is merely one embodiment, but surely not the only one, of an automatic taking device serving the purpose.

[0019] As shown in Fig. 3, said device 17 is located in the vicinity of rewinder 5 and, more precisely, between the last printing unit and the rewinder.

[0020] The device can also be mounted on the cross-pieces or shoulders of said rewinder 5.

[0021] Device 17 consists of two side members 22, of suitable sizes, which bear two idle rollers 25 allowing an optimal winding of film 20 onto a cutting punch contrasting roller 27 coated with a synthetic material.

[0022] A cutting punch holding roller 29, carried by two levers 31 and driven by cutting punch contrasting roller 27 through driving gears 39, provides for cutting sample 19, the area of which is, in the case considered, 100 cm².

[0023] Said sample 19 is automatically ejected immediately after having been cut and is collected in a suitable basket in the reach of the operator.

[0024] Cutting is pneumatically controlled through the

action of two short-stroke compact cylinders 35, by means of a compressed air pulse determined by a valve driven by the operator. Two pneumatic cylinders 37 control the lifting of the cutting punch holding roller 29 so as to allow the guided introduction of the material to be processed, and such operation is made easier by a hand driven chain device 38 for such guided introduction.

[0025] Cutting punch 39, made of treated steel, is mounted on a removable sleeve 41, which allows locating the cutting punch along the whole film width.

[0026] A set of adjusting points (not shown) allows re-aligning cutting punch 39 onto sleeve 41 when the punch is removed for being sharpened or replaced. Lastly a set of pressure rollers 43 arranged so as to be tangent to cutting punch contrasting roller 27 ensures the adhesion of film 20 to said roller in order the drawing action of the roller is made more effective. It is to be appreciated that the whole device 17 is made to rotate by the film itself thanks to the drawing action of the rotary press.

[0027] Analyser 21 of Fig. 2 will now be disclosed with reference to Fig. 4.

[0028] Said analyser 21 essentially consists of a thermostatic chamber 49, housing incubation room 51 for sample 19, sampling "loop" or valve 53, separation column 55 and flame ionisation detector (FID) 57.

[0029] A pressure sensor 59 is connected, in said thermostatic chamber 49, to a point between the sampling loop 53 and incubation chamber 51.

[0030] Analyser 21 also comprises a set of ducts and valves, such as a duct 61 for the inlet of calibration gas with an associated electric valve 63, a duct 65 for the inlet of washing pressurisation gas with an associated electric pressurisation valve 67 and a pressure regulator 69; and a duct 71 for the inlet of carrier gas.

[0031] Duct 65 is provided, in the downstream branch, with an electric valve for communication with the atmosphere, whereas duct 61 is equipped, downstream the incubation chamber 51, with an electric valve 75 for washing said chamber.

[0032] Sample 19 of film 20 to be analysed is placed into incubation chamber 51 and hermetically sealed. During this phase, valves 63, 67, 73 are closed, thereby forming a closed environment comprising incubation chamber 51 and sampling loop 53. Incubation chamber 51 has a known volume of about 20 cm³, whereas the sampling loop has a volume of 1 cm³. It is to be appreciated that said sampling loop 53 indeed comprises a loading loop 53a and an actual sampling loop 53b.

[0033] Sample 19 of film 20, when heated, releases its residual solvent within incubation chamber 51 during a pre-set time interval, or incubation time. A sensor 59 is capable of measuring a pressure increase, if any, within incubation chamber 51 because of solvent release by film 20. Once the incubation time is over, valve 67 is opened and the environment composed of chamber 51 and loop 53a is brought to a pressure pre-set by regulator 69, which usually is not provided for in the sys-

tems at present in use. For the pressurisation phase, clean air (zero air) is fed to inlet 65. That phase is referred to as pressurisation. Clearly the pressurisation phase causes a certain dilution of the gas within the closed environment. That dilution is proportional to the difference between the pressures before and after the pressurisation phase, and is a system constant.

[0034] After the pressurisation phase, valve 67 is closed and valve 73 is opened. Thus, the pressurised gas breathes out through vent outlet 73 and fills loop 53a. This phase is referred to as loop-filling phase. After the loop-filling phase, sampling valve 77 is actuated and the content of loop 53a is brought to position 53b (sampling position). Carrier gas (zero air) fed to inlet 71 pushes the residual solvent extracted, contained in loop 53b, up to detector 57 through separation column 55.

[0035] The solvent extracted, when reaching detector 57, is burnt by the detector flame. Combustion of carbon atoms present in the solvent results in an ionic current in the flame. Said current is detected and amplified and forms the measurement signal. Said measurement signal is as shown in fig. 5, where voltage values (V) are plotted on the Y axis whereas the times of arrival of the solvent at the flame are plotted on the X axis. A number of peaks can be seen that are included within five integration windows. Each window identifies a determined family of solvents, which are separately evaluated. The peak areas are proportional to the amounts of the different solvents extracted.

[0036] Analyser 21 is calibrated through a standard gas bottle with known concentration, connected to inlet 61. During said calibration phase, valves 63 and 75 are opened, so that incubation chamber 51 is filled with said reference gas. An analysis cycle (already described) is then carried out and a signal peak is obtained, the area of which is proportional to the calibration gas concentration. That area is stored within analyser 21 together with the gas concentration in the reference bottle. Those values form the reference for all subsequent measurements.

[0037] Measurement of the residual solvent concentration may take place according to two operating modalities:

- determining the residual solvent amount released by the film at the end of a pre-set incubation time; or
- determining the solvent amount progressively released by the film during a long incubation time (e. g. 45 min), by measuring the solvent concentration within the incubation chamber at regular intervals (e. g. 3 to 5 min).

Claims

1. An integrated system for evaluating the residual solvent contained in packaging films during the printing process at manufacturing plants, characterised in

that it comprises a single analyser (21) embodying the functions of three laboratory instruments: head-space or desorption unit, gas chromatograph and data system, with comparable analytical results.

2. A system according to claim 1, characterised in that it further comprises a device (17) for the automatic on-line taking of the sample (19) to be analysed.
3. A system according to claim 2, characterised in that said automatic taking device (17) is located in the vicinity of the rewinder (5), between the last printing unit (3) and said rewinder (5).
4. A system according to claim 3, characterised in that said automatic taking device (17) is mounted on the crosspieces or shoulders of said rewinder (5).
5. A system according to claim 3, characterised in that said automatic taking device (17) comprises two side members (22), which bear two idle rollers (25) for winding the film (20) onto a cutting punch contrasting roller (27), coated with a synthetic material; wherein a cutting punch holding roller (29), mounted on suitable levers (31) and driven by the cutting punch contrasting roller (27) through driving gears (39), provides for cutting the sample (19), which is ejected after having been cut and is collected at a location in the reach of the operator.
6. A system according to claims 3 and 5, characterised in that cutting in said taking device (17) is pneumatically controlled through the action of two short-stroke compact cylinders (35), by means of a compressed air pulse determined by a valve driven by the operator; two pneumatic cylinders (37) being provided to control the lifting of the cutting punch holding roller (29) so as to allow the guided introduction of the film, said operation being made easier by a hand driven chain device (38) for guided introduction.
7. A system according to claims 3 to 6, characterised in that the cutting punch (39) is made of treated steel and is mounted on a removable sleeve (41), which allows locating the cutting punch along the whole useful film width.
8. A system according to claims 3 to 7, characterised in that a set of adjusting points allow realigning the cutting punch (39) onto the associated sleeve (41) if the cutting punch is removed for being sharpened or replaced.
9. A system according to claims 3 to 8, characterised in that a set of pressure rollers (43), arranged so as to be tangent to the cutting punch contrasting roller (27), ensure the adhesion of film (20) to said roller

in order the drawing action of the roller is made more effective.

10. A system according to any preceding claim, characterised in that the analyser (21) essentially consists of a thermostatic chamber (49), housing the incubation room (51) for the sample (19), the sampling "loop" or valve (53), the separation column (55) and the flame ionisation detector, FID (57); a pressure sensor (59) being connected, within said thermostatic chamber (49), to a point (50) between the sampling loop (53) and the incubation chamber (51); said analyser (21) being equipped with a set of ducts and valves, such as an inlet duct (61) for calibration gas with an associated electric valve (63), an inlet duct (65) for washing pressurisation gas, associated with an electric pressurisation valve (67) and a pressure regulator (69); and an inlet duct (71) for carrier gas; said duct (65) being further provided, in a downstream branch, with an electric valve (73) for communication with atmosphere, whereas said duct (61) is equipped, downstream the incubation chamber (51), with an electric valve (75) for washing said chamber (51).

11. A system according to claim 10, characterised in that the sample (19) to be analysed is placed into the incubation chamber (51) of the analyser (21) by means of an open glass support, generally in tube shape.

12. A system according to claims 10 and 11, characterised in that the pressure sensor (59) included in the analyser (21) allows execution of a multiple extraction cycle, independent of the pre-set pressurisation value.

13. A system according to any of claims 10 to 12, characterised in that the separation column (55) included in the analyser (21) allows separation of light compounds and heavy compounds, thereby permitting a differentiated evaluation of the solvent extracted.

14. A system according to any of claims 10 to 13, characterised in that the calibration of the analyser (21) comprises filling the incubation chamber (51) with a standard gas mixture and the subsequent sampling and transfer to the detector (57) without need to use syringes and/or vials.

15. A system according to any of claims 10 to 14, characterised in that the incubation chamber (51) is equipped with a pierced septum, so as to allow introducing a known amount of solvent and comparing the results with the laboratory results.

16. A system according to claims 10 to 15, character-

ised in that, to prevent pollution of the incubation chamber (51) by external gases, a purging gas is provided when the chamber cover is opened.

17. A method of evaluating the residual solvent in packaging films during the printing process at manufacturing plants, characterised in that the measurement of the residual solvent concentration in a sample (19) may take place according to two operating modalities:

- determining the residual solvent amount released by the sample at the end of a pre-set incubation time; or
- determining the solvent amount progressively released by the sample during a long incubation time (e. g. 45 min) by measuring the solvent concentration within the incubation chamber at regular intervals (e. g. 3 to 5 min).

18. A method according to claim 17, characterised in that each measurement of the concentration of gas desorbing inside the incubation chamber (51) is corrected with the data of pressure and dilution due to pressurisation, in order to obtain the incremental amount of the solvent being released by the sample (19).

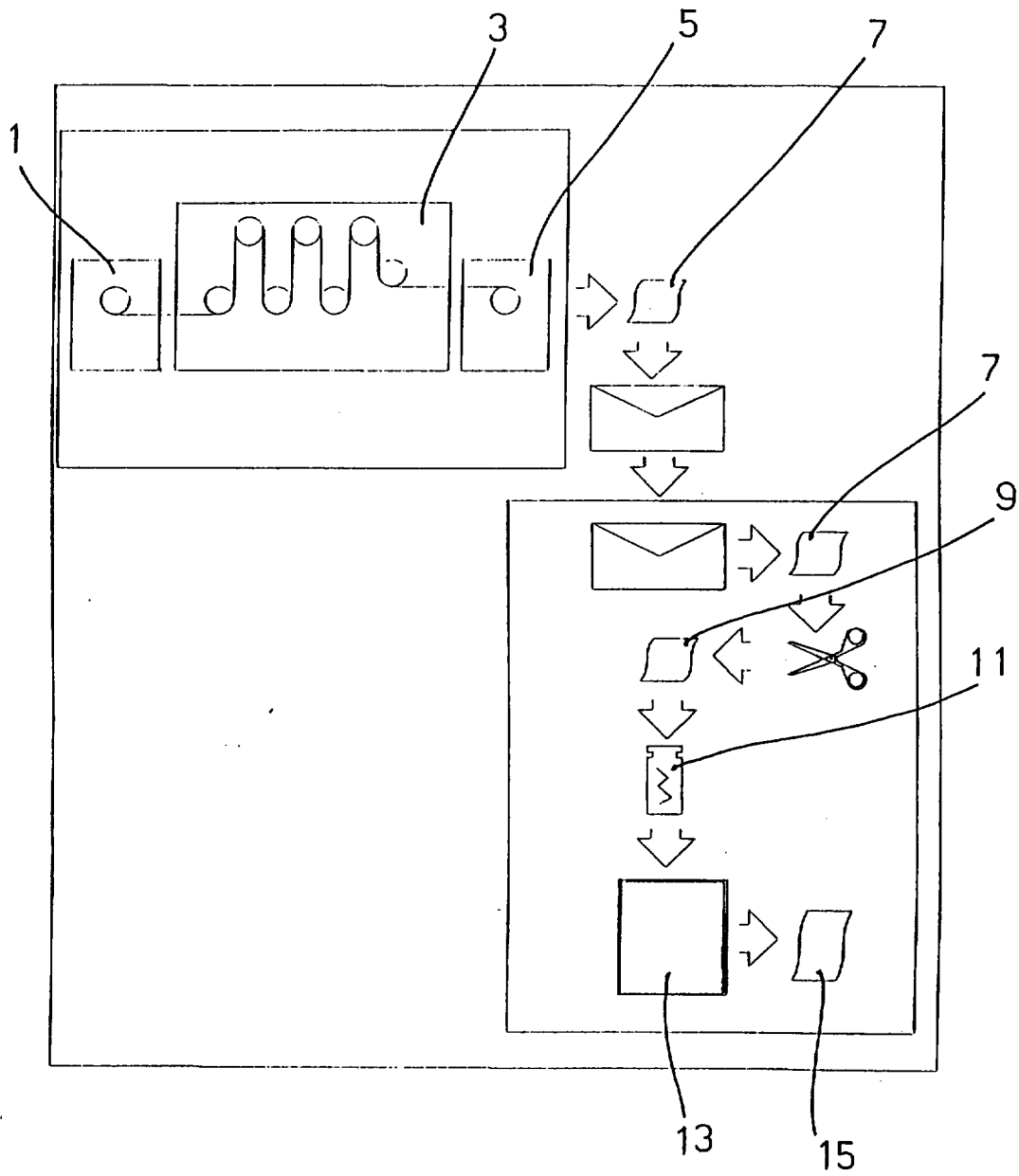


FIG. 1

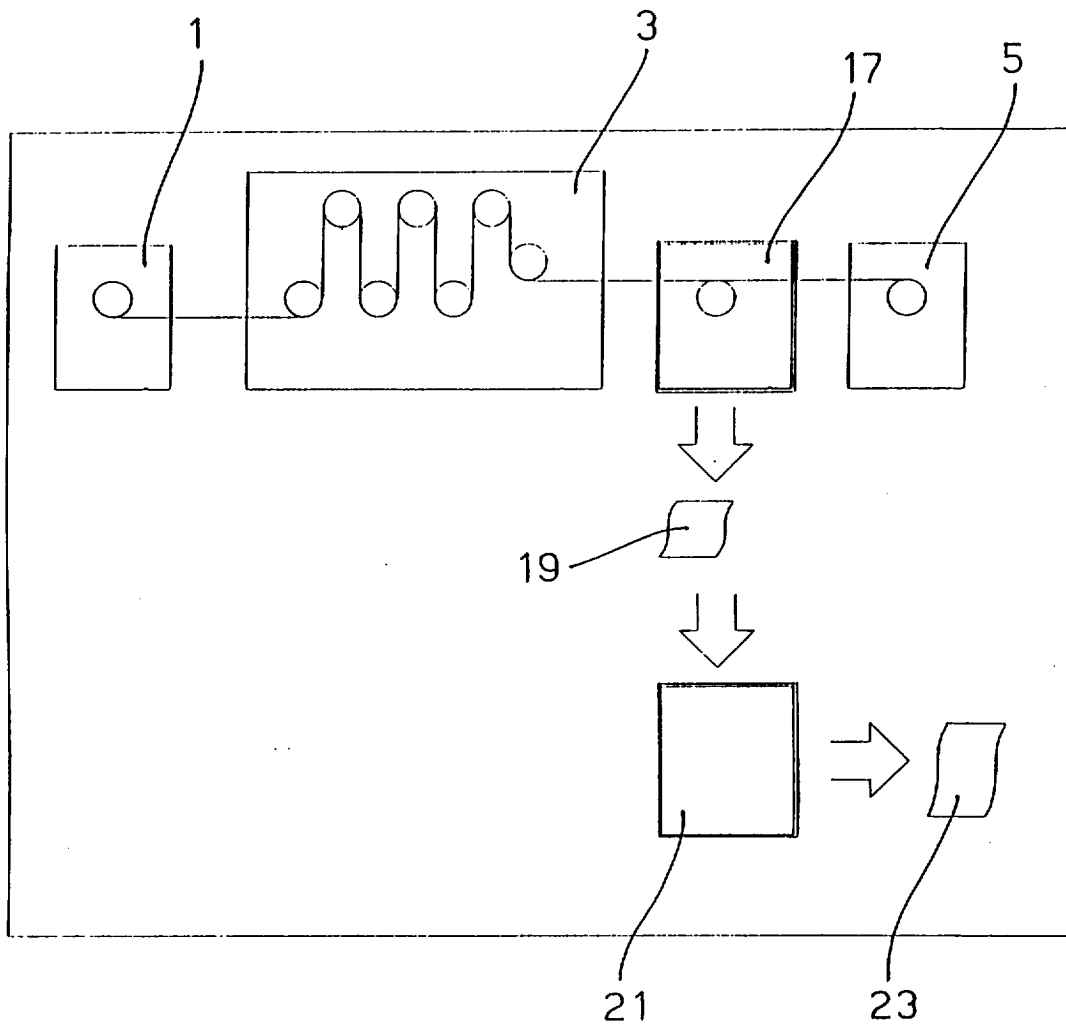
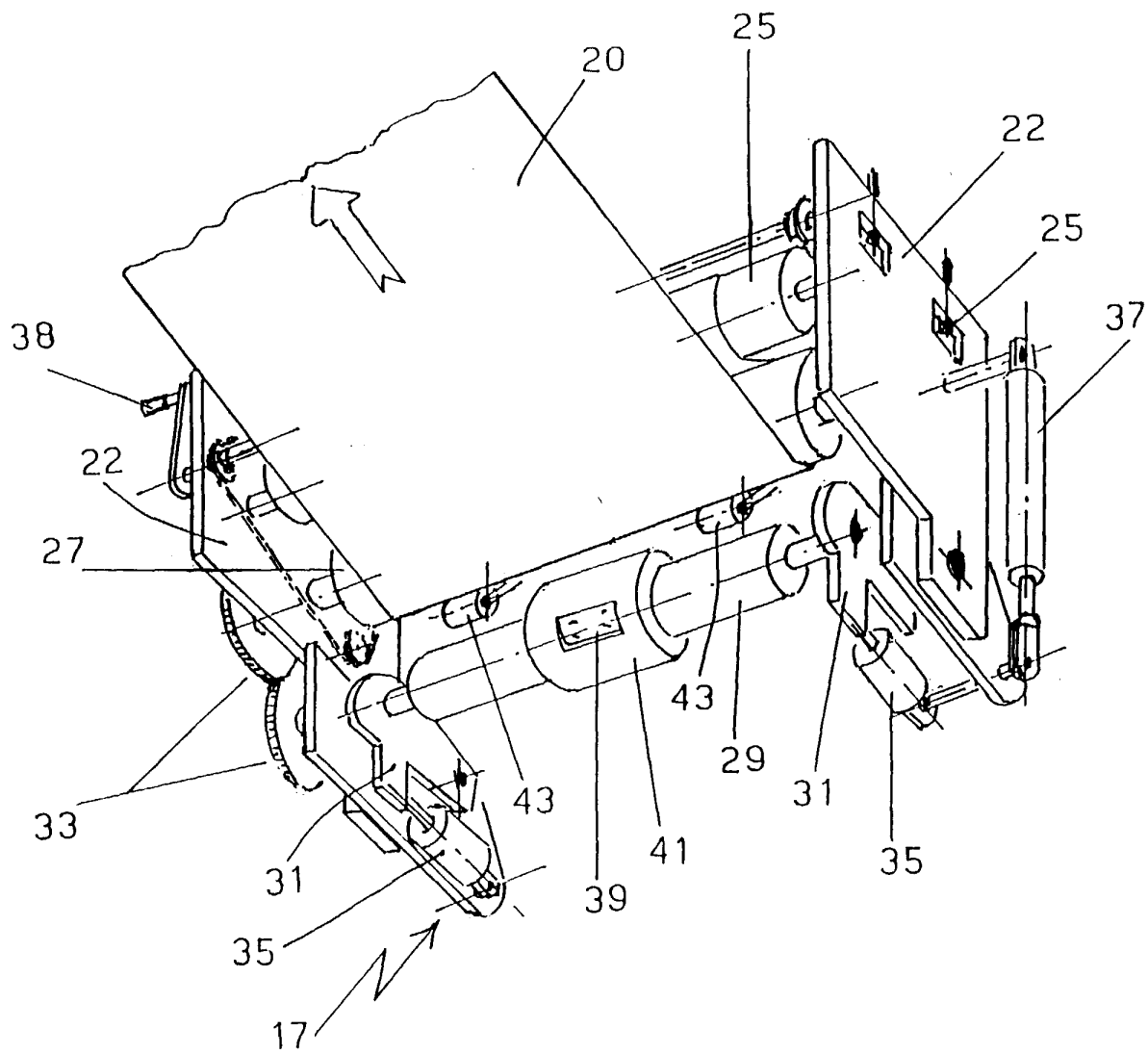


FIG. 2

FIG. 3



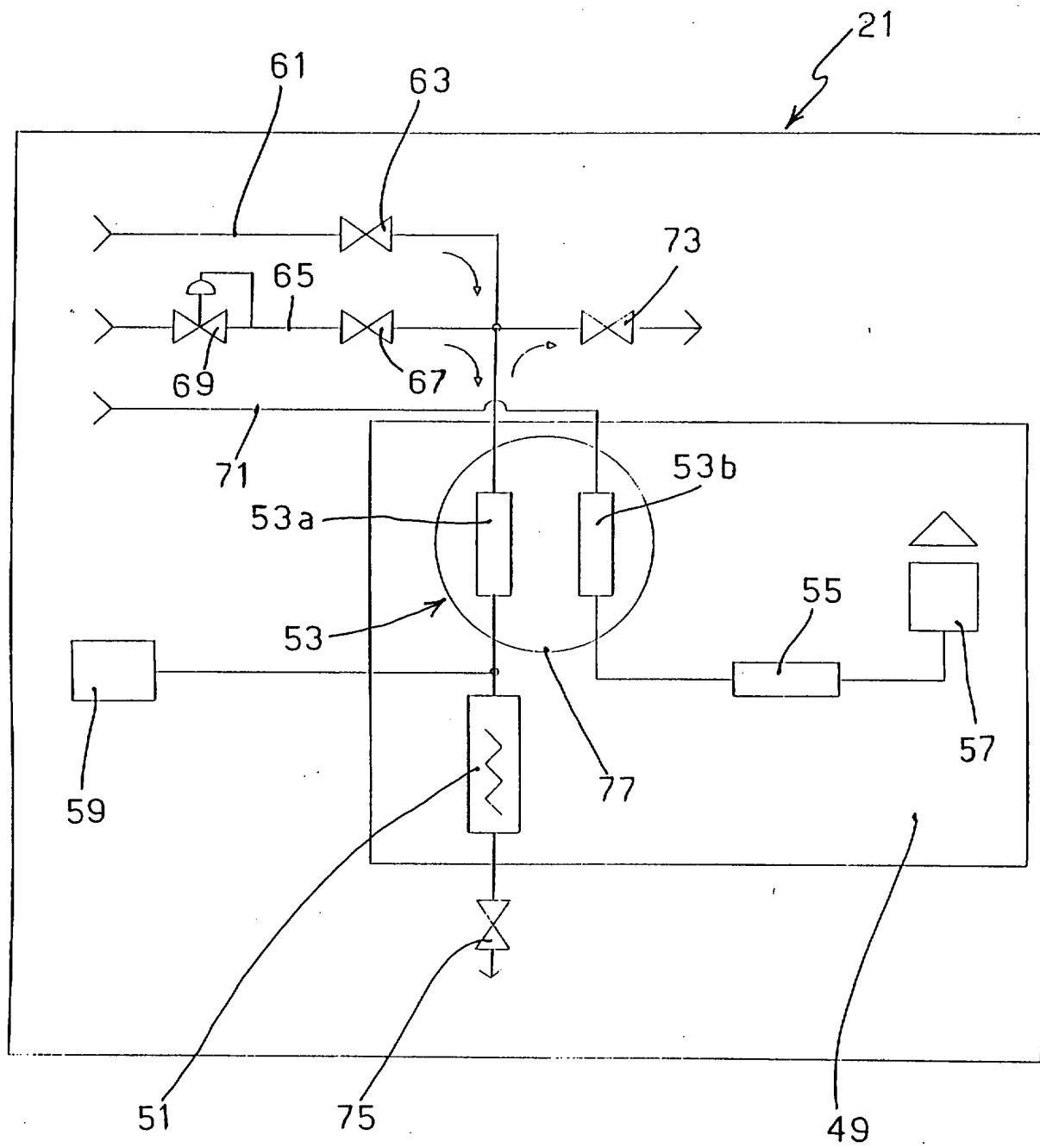


FIG. 4

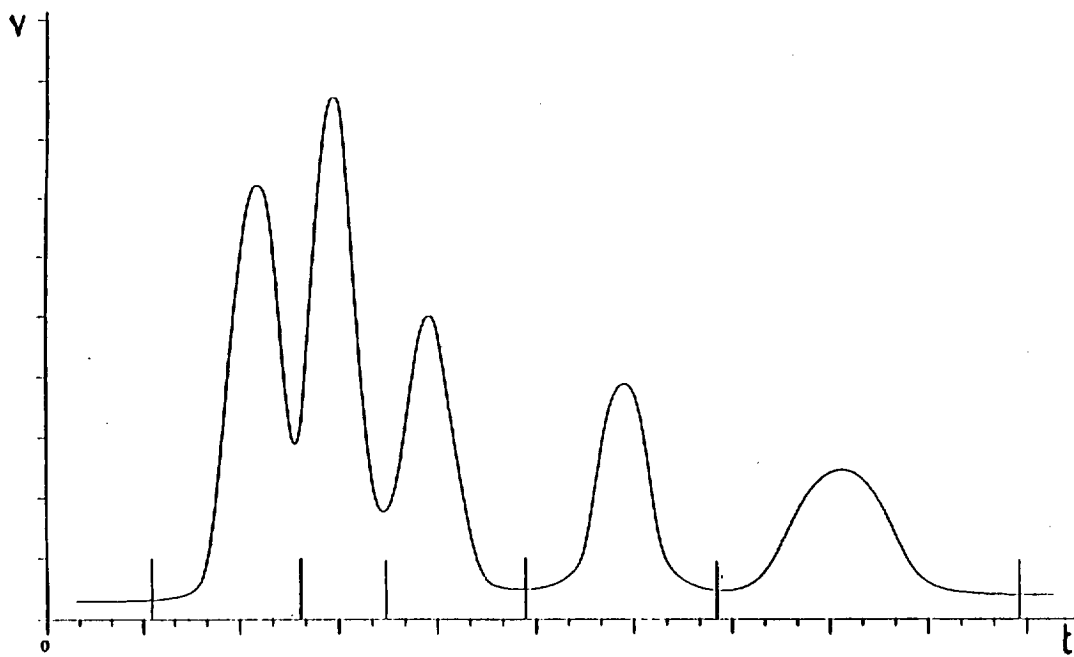


FIG. 5