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Dye and ink refining system

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LONDON THE PATENT OFFICE

(73) Proprietors
Canon Kabushiki Kaisha

(Incorporated in Japan)

30-23-chome Shimomaruko
Ohta-ku
Tokyo
Japan

(72) Inventors
Masahiro Haruto
Kunitaka Ozawa
Takashi Hamamoto

(74) Agent and/or
Address for Service
R. G. C. Jenkins & Co.,
12-15 Fetter Lane,
London EC4A 1PL

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C4P

FIG. 1

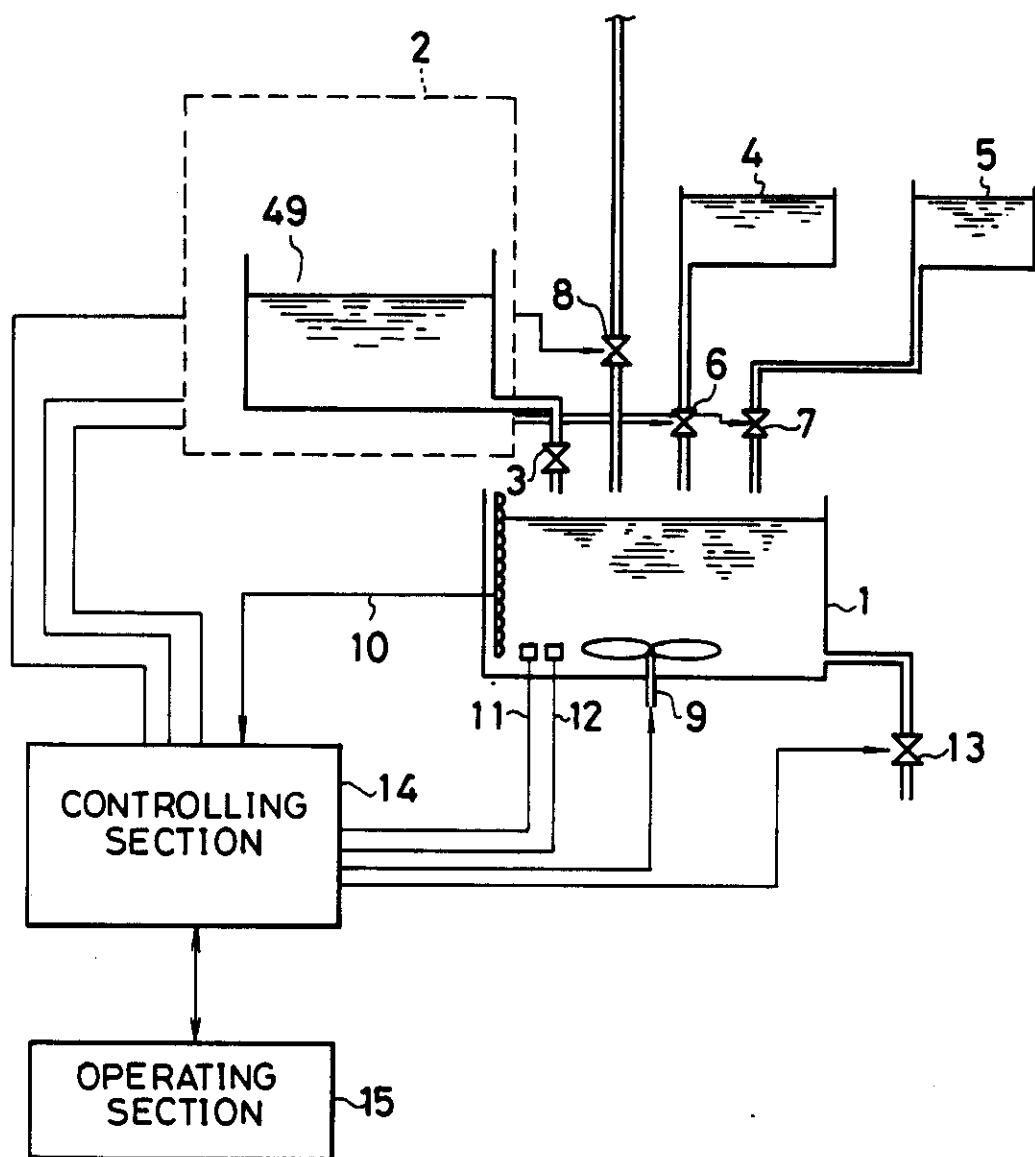


FIG. 2

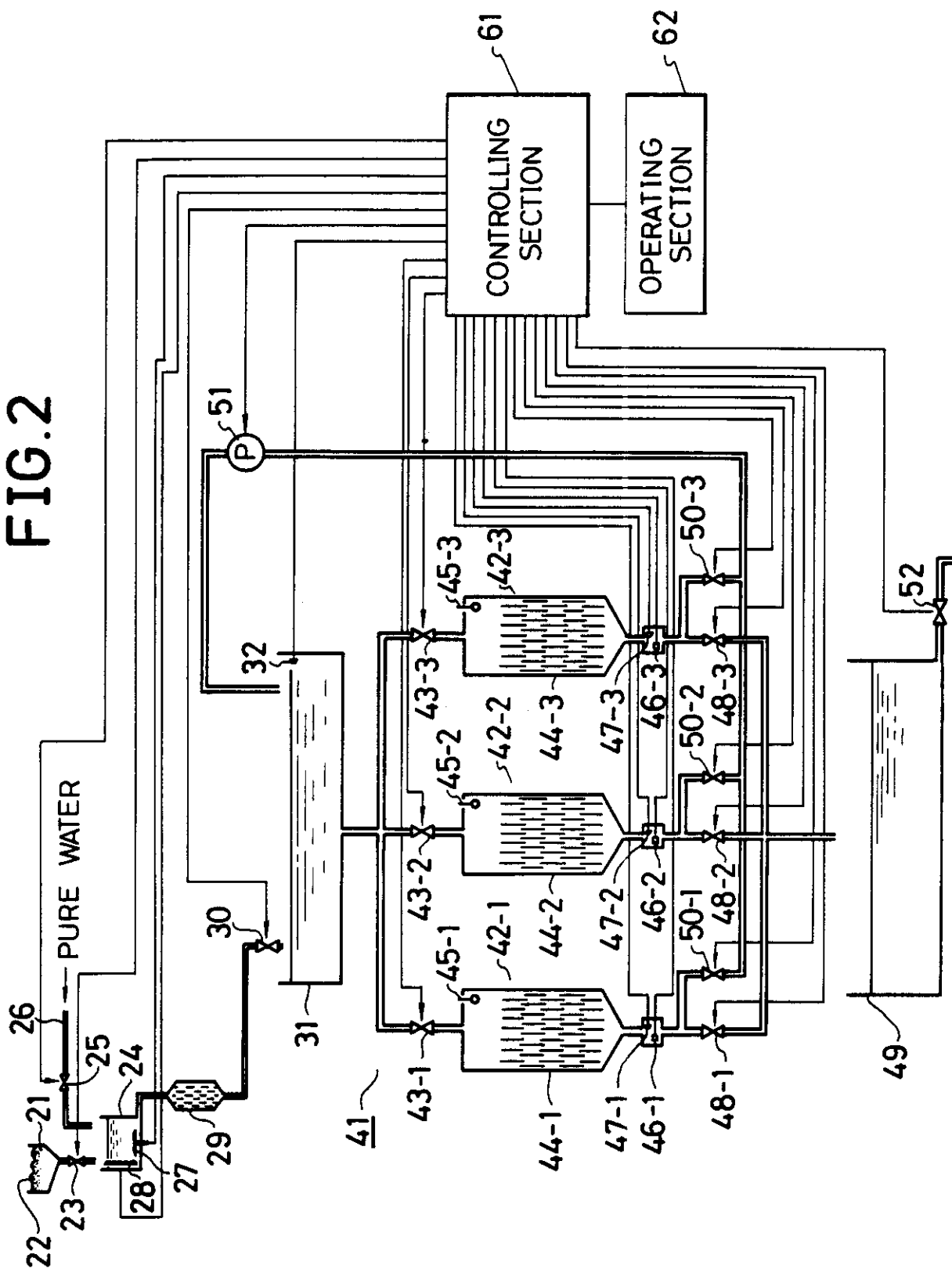
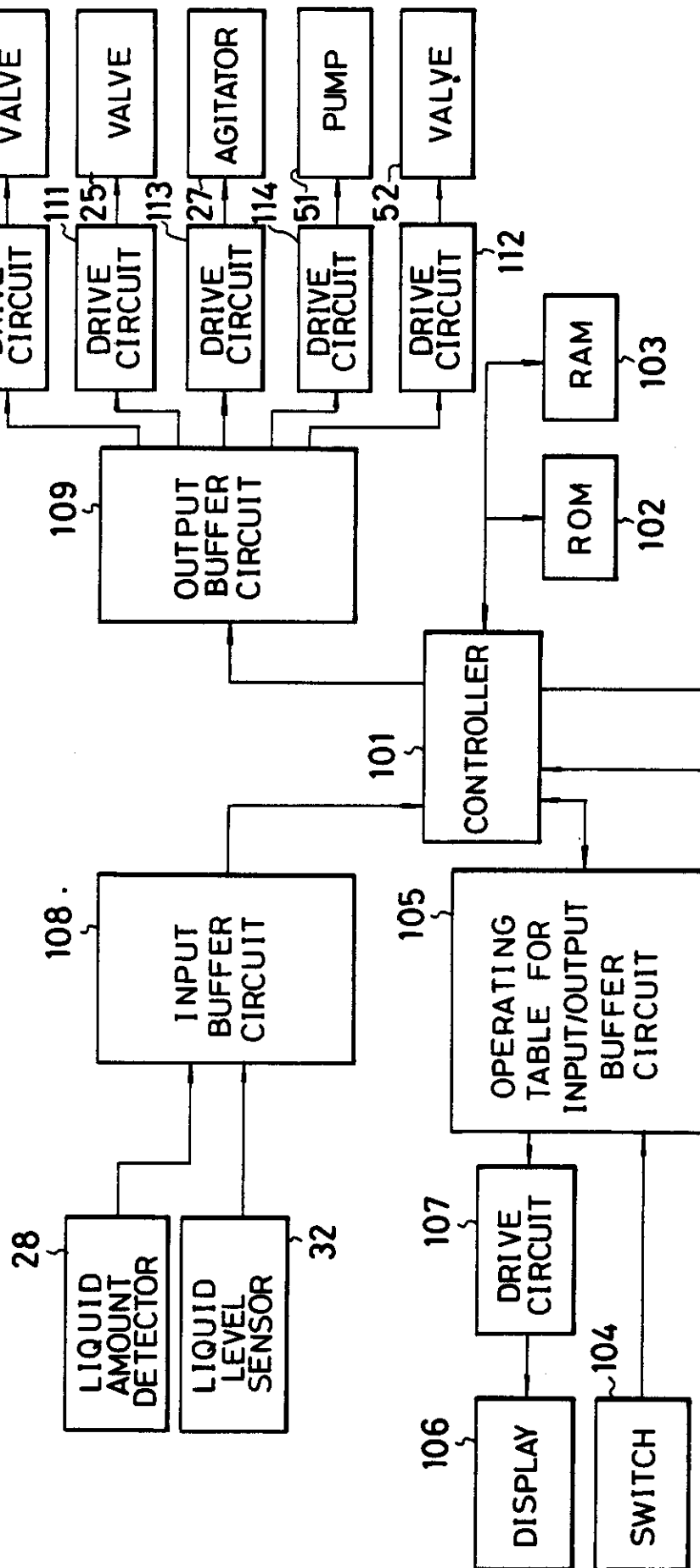


FIG. 3

FIG.3-a
FIG.3-b

FIG.3-a



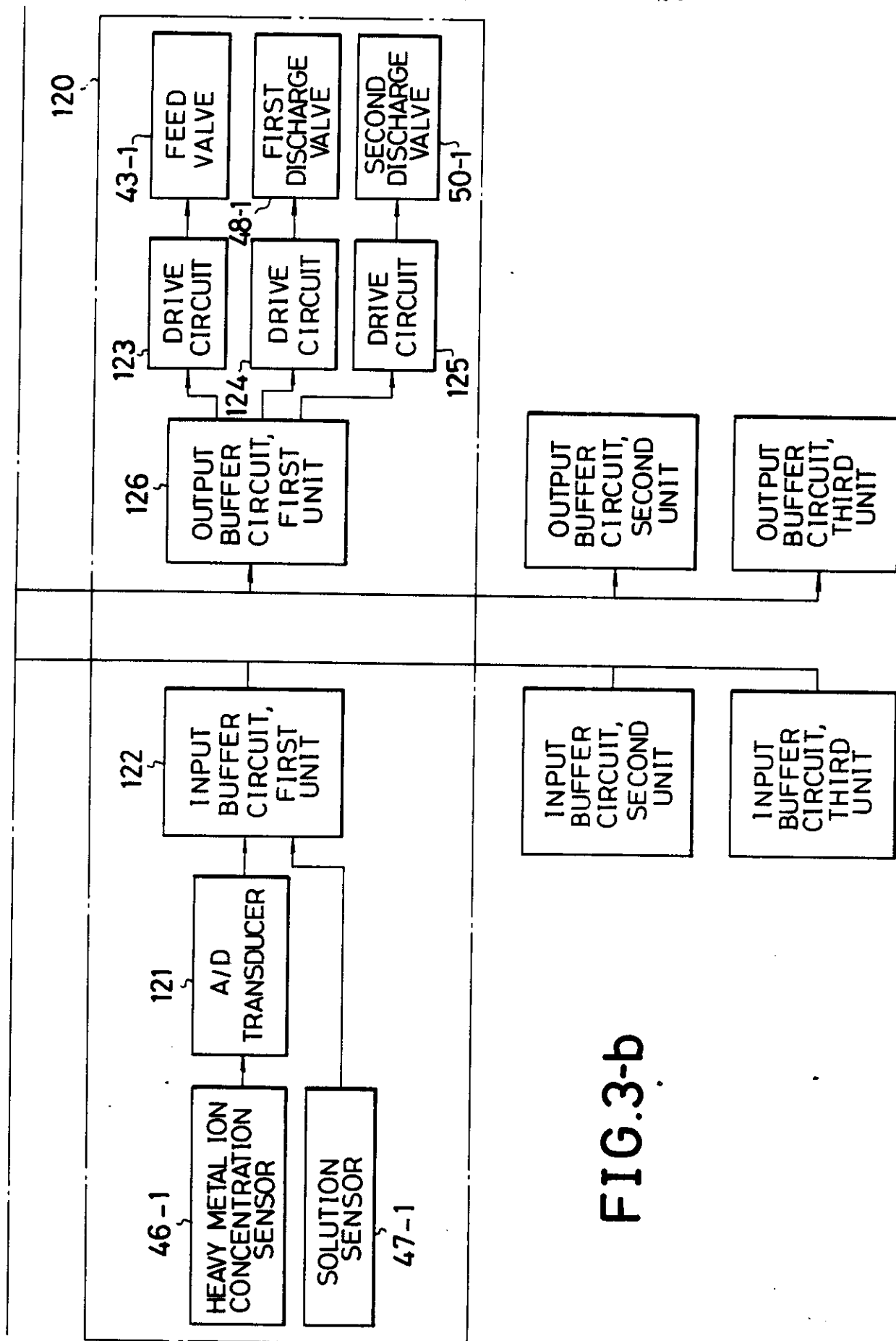
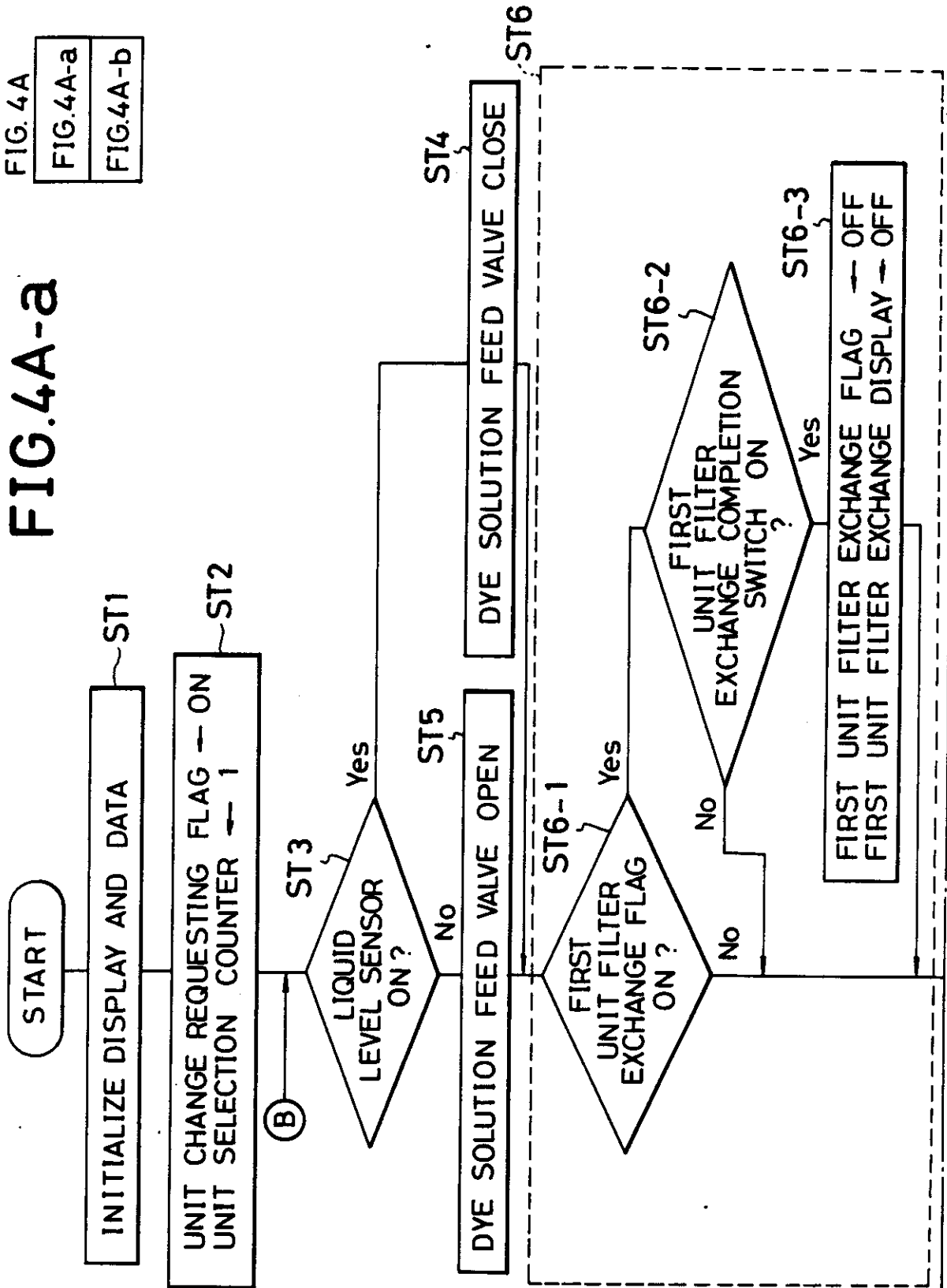


FIG.4A-a

FIG. 4A
FIG.4A-a
FIG.4A-b



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FIG. 4A-b

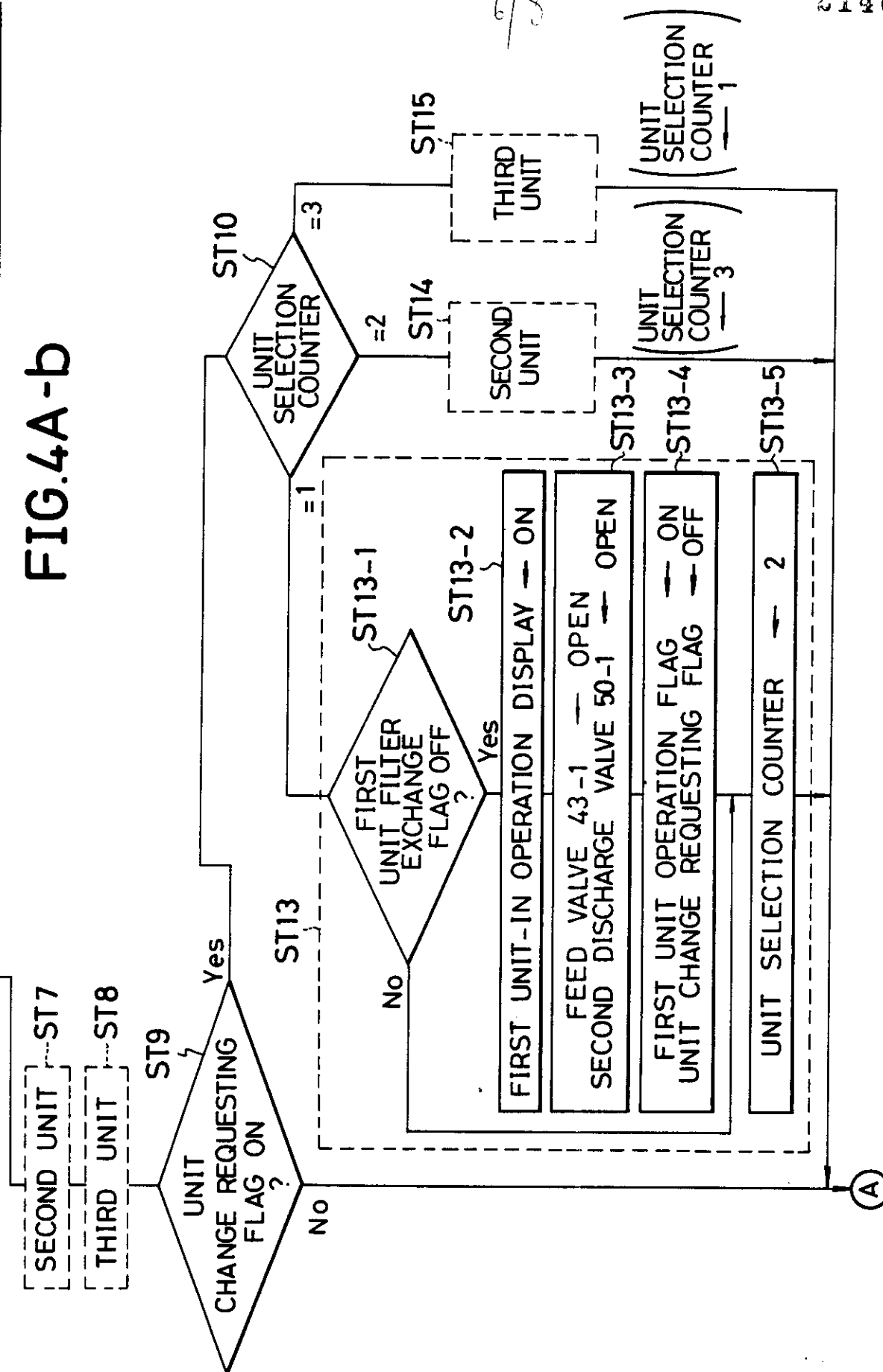
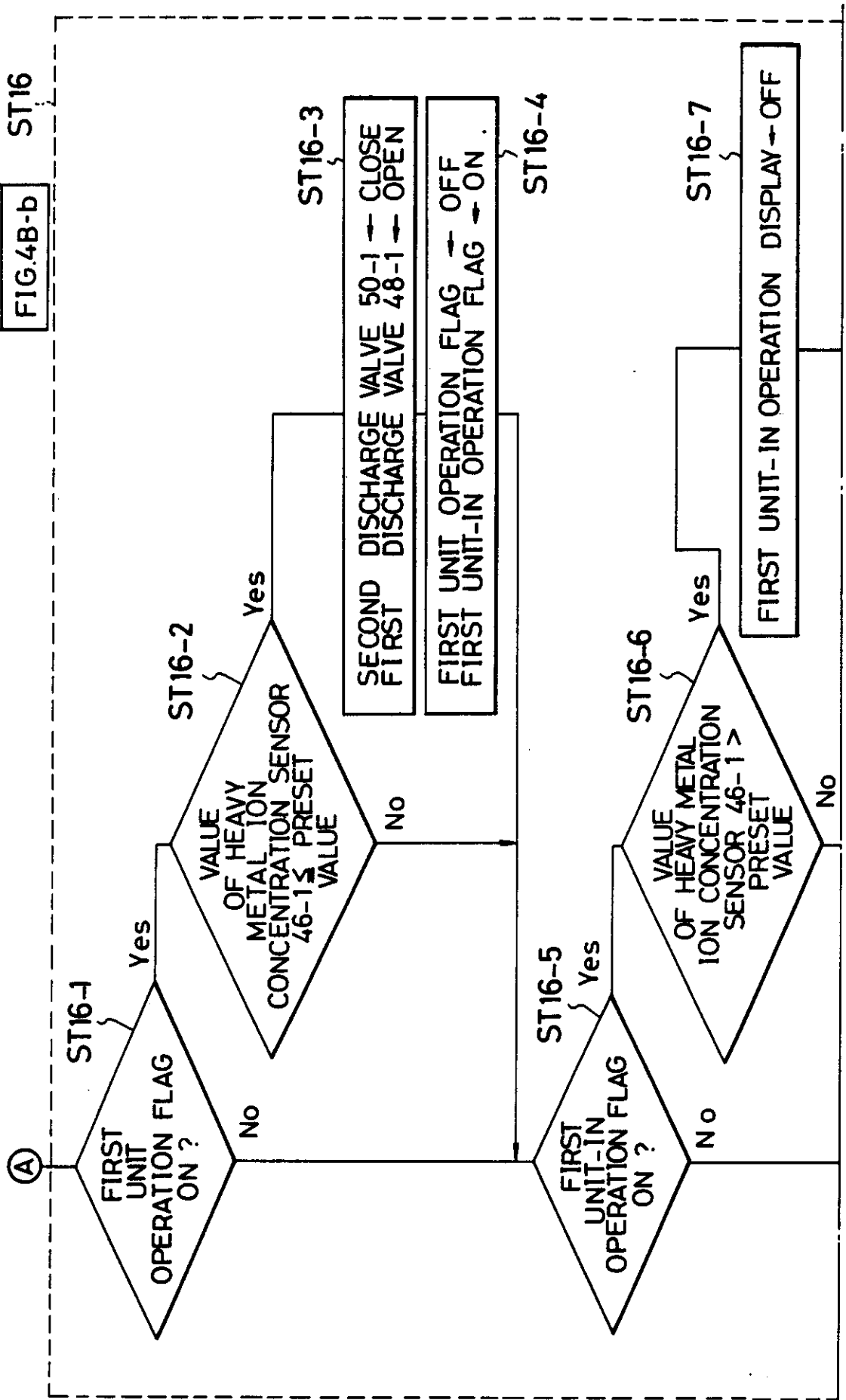
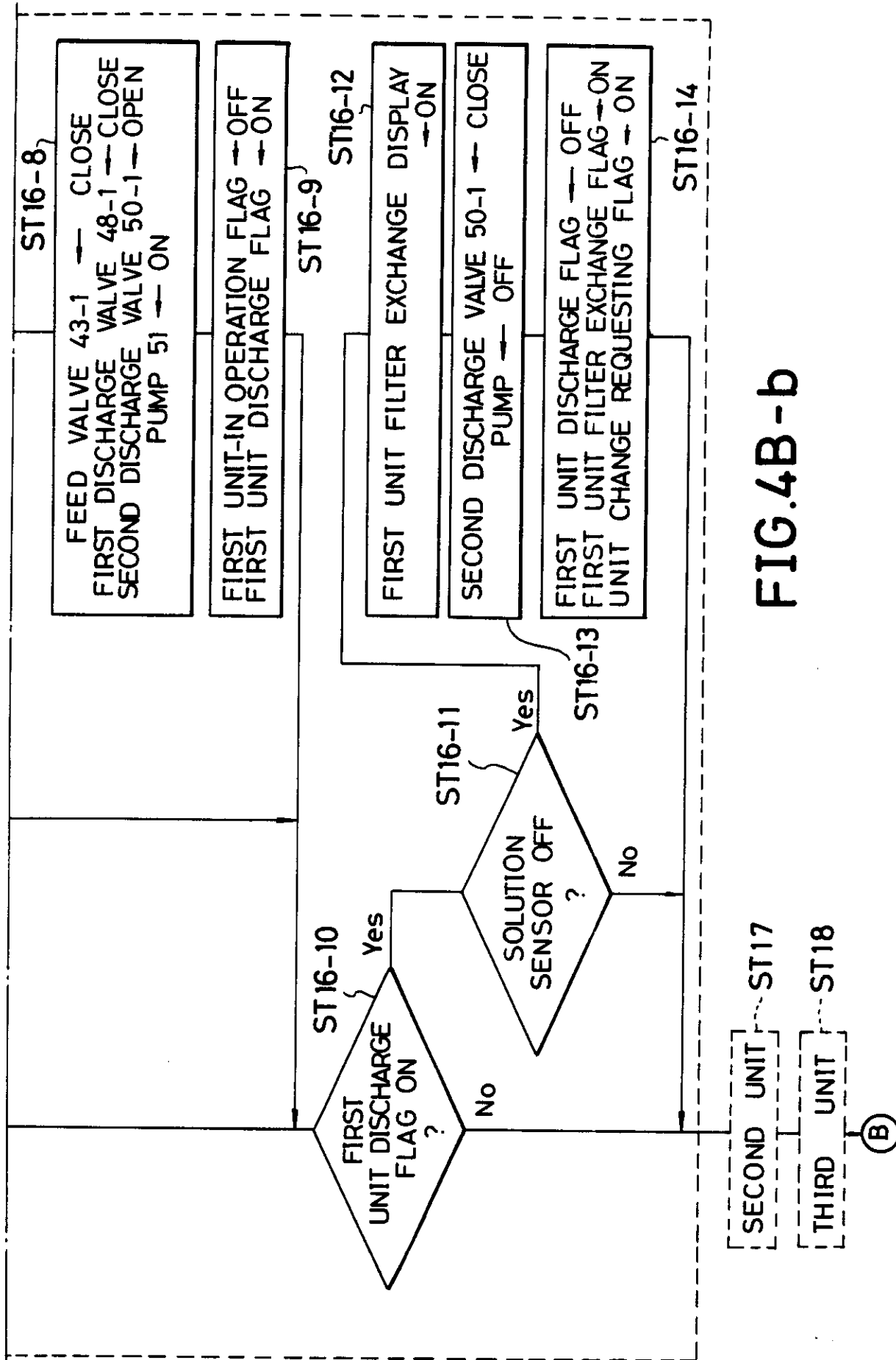


FIG. 4B-a

FIG. 4B

FIG. 4B-a
FIG. 4B-b





Dye and Ink Refining System

This invention relates to a dyestuff refining system, and, more particularly, it is concerned with a dyestuff refining system capable of
5 continuously supplying refined dyestuff which is suitable for preparing recording liquid (generally called "ink") adapted to use in ink jet recording, writing implements, and other uses.

Ink to be used for an ink jet recording
10 system which implements recording of information by ejecting ink within a recording head through an ejection orifice by means of vibrations transmitted from a piezo-electric vibrator or other expedients, employs various dyestuffs and pigments which are
15 dissolved or dispersed in a liquid medium such as water or other (e.g. organic) solvent. Similar ink is used in writing implements such as felt pens, fountain pens, etc.

Such ink may comprise the following three chief components: water soluble dyestuff, water as the solvent for the dyestuff, and glycols as a desiccation-preventive agent.

5 The water-soluble dyestuff can contain a large amount of inorganic salts such as sodium chloride and sodium sulfate either as by-products of the dye-synthesis, or as added salting-out agents, diluents, or level-dyeing agents.

10 We have discovered that when recording ink is prepared with use of dyestuff containing such inorganic salts, problems can arise: the inorganic salts can lower the solution stability of the dyestuff in the ink to bring about agglomeration and
15 sedimentation of the dyestuff. Further, in the ink jet recording heads and writing implements, if and when the ink is evaporated in the vicinity of the ejection orifice causing the liquid composition to change, deposition of the inorganic salts can be
20 induced. These phenomena cause clogging to take place at the discharge orifice which is highly undesirable.

 We have found that by controlling the inorganic salt concentration within a predetermined
25 maximum when producing the ink (in general, limited

to 0.5 wt.% or less with respect to the total ink composition) these problems can be avoided. We have found such control to be indispensable when the dye-stuff available on the general market, which
5 contains therein inorganic salts as impurities, is used for preparation of ink for the ink jet recording as well as for use in writing implements.

An object of the present invention is to provide a system for purifying a dye used for
10 producing ink which is capable of producing ink of high quality suitable for ink jet recording or writing implements, reliably and simply and capable of continuous production under automatic control.

According to the invention there is
15 provided a dye purification system comprising means for producing a solution of the dye and separation means including an ion-exchange resin for capturing heavy metal ions in said dye solution, said separation means including valve means for separating
20 dyestuff solution in which the concentration of heavy metal ions is at or below a predetermined value from dyestuff solution in which the heavy metal ion concentration is above the predetermined value.

25 The invention includes an ink production system comprising a dye purification system as described above, and an ink preparing section which

prepares ink with the purified dye solution discharged from said system.

The invention also includes a method of manufacturing ink in which, prior to the addition of further ingredients, a dye solution is purified by the reduction of heavy metal ions by ion exchange on an ion exchange resin to maintain the heavy metal ion concentration below a predetermined value.

In the drawings:-

Fig. 1 is a schematic flow chart showing an example of the device according to the present invention;

Fig. 2 is a schematic flow chart of the means for purifying dye in the device shown in Fig. 1;

Fig. 3 composed of Figs. 3-a and 3-b is a block diagram showing the controlling section in the device shown in Fig. 1; and

Fig. 4A composed of Figs. 4A-a and 4A-b and Fig. 4B composed of Figs. 4B-a and 4B-b are flow charts showing the purifying operation in the treating units in the device shown in Fig. 1.

Fig. 1 shows an example of the device of the present invention. In this Figure, numeral 1 is an ink formulating tank, and from a dye purifying means 2, an aqueous dye solution purified as

hereinafter described is supplied through a valve 3. On the other hand, from a reservoir section 4 and a reservoir section 5 are supplied through valves 6 and 7 respectively, a water-soluble organic solvent and an additive into the formulating tank 1. Further, pure water is supplied similarly through a valve 8. These materials supplied are stirred by means of a stirrer 9 to formulate an ink.

The amount of the ink prepared in the formulating tank 1 is detected by a liquid amount detector 10. The dye concentration and the heavy metal ion concentration in the ink prepared in the formulating tank 1 are also detected by a dye

concentration detector 11 and a heavy metal ion concentration detector 12, respectively. Based on the outputs from both of these detectors, the final property management of the ink (lot management) is performed. The ink prepared with desired values of respective components is discharged through a discharging valve 13, if necessary.

Numerals 14 and 15 are controlling sections which perform driving control of the respective sections, and 15 is an operating section which is provided with various kinds of display sections and driving switches, etc. (14, 15 are indicated 61, 62 in Fig. 2).

1 Fig. 2 shows the constitution of the dye
purifying means in the device shown in Fig. 1.

 In this Figure, numeral 21 is a dye feeding
section containing dye powder 22 and the dye powder
5 22 is fed through a dye valve 23 into a formulating
tank 24. Also, into the formulating tank 24, pure
water is fed through a pure water feed pipe 26
having a pure water valve 25 interposed therein.

 In the formulating tank 24, the dye powder
10 22 and pure water are mixed and dissolved by means
of a formulating tank agitator 27 to prepare an
aqueous dye solution. The amount of the aqueous dye
solution residing in the formulating tank 24 is
detected by a formulating tank liquid amount detector
15 28. In the aqueous dye solution obtained in the
formulating tank, there remain particles of the dye
powder not dissolved, and these are removed by
filtration with a filter 29. For the filter 29, a
conventional filter paper or Fluoropore (trade name),
20 etc. may be available. The dye solution removed of
particles by passing through the filter 29 is fed to
a feed tank 31 through a dye solution feed valve 30.

 The feed tank 31 is provided with a feed tank
liquid level sensor 32 and the liquid level in the
25 feed tank is controlled by opening-closing control
of the dye solution feed valve 30 based on the output
from the sensor.

1 Next, 41 is a purifying section for removing
heavy metal ions from an aqueous dye solution,
comprising a plurality of treating units, three units
of the first to the third treating units 42-1, 42-2
5 and 42-3 in this example. In each treating unit 42
(42-1 - 42-3), the aqueous dye solution in the feed
tank 31 is fed through the feed valve 43 (43-1 - 43-3)
to the ion-exchange resin column 44 (44-1 - 44-3).
The ion-exchange column 44 separates heavy metal
10 ions from the aqueous dye solution through the
so called ion-exchange separation. More specifically,
through ion exchange in the column, heavy metal ions
higher in absorption contained in the aqueous dye
solution are captured by the ion-exchange resin and
15 remain within the column. Here, within the column,
a valve 45 (45-1 - 45-3) for control of the liquid
level is provided to control the amount of the
aqueous dye solution fed into the column. The column
44 is also mounted detachably from the treating unit,
20 and whether the column is mounted or not is detected
by the filter exchange completion switch which is
not shown.

 Next, heavy metal ion concentration sensor
46 (46-1 - 46-3) are provided in the effluent
25 solution passage at the lower end portion of the
columns 44 to detect the heavy metal ion concentra-
tion in the effluent solution from the column 44.

1 As the method for detecting heavy metal ion con-
centration, there may be employed the measurement
method with the use of metal ion electrodes or the
measurement method by atomic absorption spectroscopy.
5 Solution sensors 47 (47-1 - 47-3) are similarly
provided in the effluent solution passage to detect
presence of the effluent solution in the effluent
solution passage.

The effluent solution from the column 44 is
10 discharged through a first discharge valve 48 (48-1
- 48-3) to a reservoir 49. Also, through a second
discharge valve 50 (50-1 - 50-3), by the action of
a circulating pump 51, the solution is recycled to
the feed tank 31. Opening-closing control of both
15 valves 48 and 50 is performed based on the detection
result of the heavy metal ion concentration sensor
46 as described below.

The aqueous dye solution removed of heavy
metal ions obtained in the reservoir 49 is taken out
20 through the feed valve 52, (3 in Fig. 1) and
provided for use in preparation of ink.

Fig. 3 shows each parts concerning the dye
purifying means in the control system in the device
as shown in Fig. 1. In this Figure, 101 is a
25 controller and has the function of driving control
of the respective sections. 102 is a read-only
memory (ROM) and memories the control program with

1 the operational procedure as shown in Fig. 4.
103 is a random access memory (RAM) and performs
temporary memories of various kinds of data.
104 shows various kinds of switches arranged at the
5 operational section 15, and sends various kinds of
instruction signals through the input-output buffer
circuits 105 to the controller. 106 is a display
arranged at the operational section 15 and, as
hereinafter described, has the first to the third
10 unit filter exchange displays representing that
column exchange in the respective treating units
is necessary and the first to the third in operation
displays representing that the respective units are
in operation of purifying the aqueous dye solution.
15 107 is a drive circuit for performing display control
of the display 106 based on the drive signal from
the controller 101.

The detection outputs from the liquid amount
detector 28 of the formulating tank and the liquid
20 level sensor 32 of the supplying tank are supplied
to the controller 101 through the input buffer
circuit 108. The drive circuits 110 to 114 are used
for on-off control of respective loads (valves, 23,
25, 52, agitator 27, pump 51) based on the drive
25 control signals supplied through the output buffer
circuit 109 from the controller 101.

1 Next, 120 is the control system for the
treating unit 42-1. Since the constitutions for the
respective treating units are same, other units 42-2
and 42-3 are omitted in the drawing. The detection
5 signal from the heavy metal ion concentration sensor
46-1 is converted to digital signal through the A/D
converter 121 and then supplied through the input
buffer circuit 122 to the controller 101. On the
other hand, from the liquid sensor 47-1 is outputted
10 a digital detecting signal and supplied through the
input buffer circuit 122 to the controller 101.
123, 124 and 125 are drive circuits for opening or
closing the respective valves 43-1, 48-1 and 50-1,
respectively, and they are placed under on-off
15 control by the drive signals supplied through the
output buffer circuit 126 from the controller 101.

 In the dye purifying means in this Example
as constituted above, based on the detection signals
from the heavy metal ion concentration sensor 46,
20 treatment for removal of heavy metal ions is con-
ducted while successively changing the respective
treating units 44-1 - 44-3. This is because, there
is limit in capacity of the ion-exchange resin
within the ion-exchange column 44 of capturing heavy
25 metal ions and, after absorption of a certain amount
of heavy metal ions, no more ion-exchange separation
is effected. As the result, the dye solution

1 containing heavy metal ions is discharged as such
and the heavy metal ion concentration in the effluent
solution from the column 44 will be increased. By
detection of the elevation of such concentration,
5 use of the ion-exchange column is made no longer
possible, and purification of the dye solution is
conducted thereafter by either one of the two columns.
The ion-exchange resin column which has been made no
longer useful is exchanged with a new column or the
10 ion-exchange resin is subjected to regeneration
treatment so as to be capable of purification in
the same treating unit again.

Fig. 4 shows the operations in each of such
treating units, wherein the respective flags perform
15 the following judgements:

Unit filter exchange flag: flag which indicates
exchange of filter, namely exchange of the ion-
exchange resin column, and, when this flag is on,
the corresponding unit filter exchange display
20 gives a display that filter exchange is necessary;

Unit change demanding flag: flag demanding change
of the treating unit, and, when this flag is on,
the treating unit designated by the unit selection
counter is selected, and the dye purifying opera-
25 tion is transferred to the treating unit selected;

Unit operating flag: flag which indicates that the
corresponding treating unit is selected;

1 Unit-in operation flag: flag which indicates that
the corresponding treating unit is in operation;
Unit discharging flag: flag which becomes on when
the heavy metal ion concentration in the effluent
5 solution from the column in the corresponding
treating unit exceeds a setting value, and
indicates start of recycle operation of the
effluent solution to the feed tank 31 by the
pump 51.

10 Next, the respective steps in Fig. 4 are
to be explained.

In Fig. 4 (A), in the step ST1, the respec-
tive displays and data are initialized. In the step
ST2, the treating unit change demanding flag is
15 turned on, and the content of the unit selecting
counter is set on "1". In the step ST3, from the
detection output from the feed tank liquid level
sensor 32, it is judged whether a predetermined
amount of dye solution is stored in the feed tank 31
20 or not. When the desired amount of dye solution is
stored, after closing the dye solution feed valve 30
in the step ST4, the operation proceeds to the step
ST6. Whereas, when the desired amount is not
attained, the feed valve 30 is opened in the step
25 ST5 to feed the dye solution.

The step ST6 is the treating step when there
is filter exchange demand in the first treating unit

1 42-1, and consists of the steps ST6-1 through ST6-3.
First, in the step ST6-1, when it is judged that the
first unit filter exchange flag is on, the operation
proceeds to the step ST6-2, and judgement is made
5 about whether the first unit filter exchange comple-
tion switch is on or not. When it is on, the
operation proceeds to the step S6-3, where the first
unit filter exchange flag is turned off, simultane-
ously with extinction of the display on the first
10 unit filter exchange display that exchange is
necessary. Then, the operation proceeds to the step
ST7. In the step ST6-1, if the filter exchange flag
is off and the filter exchange completion switch is
off in the step ST6-2, the operation should proceed
15 to the step ST7 along the flow of "NO".

The step ST7 and the step ST8 are treating
steps, respectively, when there are filter exchange
demands for the second treating unit 42-2 and the
third treating unit 42-3, and they have the same
20 content as the step ST6 and therefore their explana-
tion are omitted.

In the step ST9, it is judged whether the
unit change demanding flag is on or not, and the
operation proceeds to the step ST10 if the judgement
25 is affirmative, while to the step ST16 in Fig. 4 (B),
if it is negative. In the step ST10, judgement is
made about the content of the unit selection counter,

1 and the operation proceeds to the step ST13, if the
content is "1", to the step ST14 when it is "2",
and to the step ST15 when it is "3", respectively.

5 The steps ST13 - ST15 are treating steps
when the respective treating units 42-1 - 42-3 are
selected. Since the content of each of the steps
ST13 - ST15 is the same, only the content of the
step ST13 when the first treating unit 42-1 is
selected is to be explained. First, in the step
10 ST13-1, it is judged whether the first unit filter
exchange flag is off or not. If the judgement is
negative, the operation proceeds to the step ST13-5,
while to the step ST13-2, when it is affirmative.
In the step ST13-2, the first unit-in operation
15 display is turned on to indicate that the first
treating unit 42-1 is in operation. In the step
ST13-3, the feed valve 43-1 and the second discharge
valve 50-1 are opened. Subsequently, in the step
ST13-4, the first unit operation flag is turned on,
20 simultaneously with turning off the unit change
demanding flag. Next, in the step ST13-5, the
content of unit selection counter is changed to "2"
and then the operation proceeds to the step ST16.
Here, the unit selection counter is a ring counter
25 in which the content is changed in the order of
"2", "3", "1", "2"....., and its content changes to
"3" by performing the step ST14, and its content

1 changes to "1" by performing the step ST15.

Next, the steps ST16, ST17 and ST18 in
Fig. 4 (B) are steps for controlling discharge of
the effluent solution from the ion-exchange columns
5 in the respective treating units. Since the content
of each step is the same, only the content of the
step ST16 for exhibiting control in the first
treating unit 42-1 is to be explained.

First, in the step ST16-1, it is judged
10 whether the first unit operation flag is on or not.
In the case of negative judgement, the operation
jumps to step ST16-5, while it proceeds to the step
ST16-2 in the case of affirmative judgement. In the
step ST16-2, it is judged whether the heavy metal
15 ion concentration in the effluent solution from the
column detected by the heavy metal ion sensor 46-1
is at a setting value or lower. If negatively judged,
the operation proceeds to the step ST16-5, while to
the step ST16-3 if affirmatively judged. In the step
20 ST16-3, the second discharge valve 50-1 is closed
and the first discharge valve 48-1 opened. As the
result, in the ion-exchange resin column 44-1, the
aqueous dye solution from which the heavy metal
ions are removed to a value not higher than the
25 setting value is discharged into the reservoir tank
49 through the first discharge valve 48-1. Next,
in the step ST16-4, the first unit operation flag is

1 turned off and the first unit-in operation flag
turned on before proceeding to the step ST16-5.

In the step ST16-5, it is judged whether the
first unit-in operation flag is on or not. In the
5 case of off-state, the operation proceeds to the
step ST16-10, while to the step ST16-6 in the case
of on-state. In the step ST16-6, it is judged
whether the concentration detected by the heavy metal
ion concentration sensor 46-1 is a value in excess
10 of the setting value or not. When it does not
exceed the setting value, the operation proceeds to
the step ST16-10, while to the step ST16-7, when it
is excessive. In the step ST16-7, the first unit-in
operation display is turned off and then the opera-
15 tion proceeds to the step ST16-8, where the feed
valve 43-1 and the first discharge valve 48-1 are
closed. Further, simultaneously with opening of the
second discharge valve 50-1, the circulating pump 51
is driven to commence recycle operation which recycles
20 the effluent solution from the column 44-1 through
the second discharge valve 50-1 to the feed tank 31.
Then, in the step ST9, after the first unit-in
operation flag is turned off and the first unit
discharge flag is turned on, the operation proceeds
25 to the step ST16-10.

Next, in the step ST16-10, it is judged
whether the first unit discharge flag is on or not,

1 and the operation proceeds to the step ST17 if it is
off, or to the step ST16-11 when it is on. In the
step ST16-11, it is judged whether the solution
sensor 47-1 arranged in the passage of effluent
5 solution from the column 44-1 is off or not, namely
whether there remains the effluent solution in the
passage of effluent solution. In the case when it
is not in off-state, that is, when the solution is
detected, the operation proceeds to the step ST17,
10 or to the step ST16-12, when it is under off-state.
The off-state means that all the aqueous dye solution
having higher value of heavy metal ion concentration
than the setting value discharged from the column
44-1 has been completely recycled to the feed tank
15 31. In the step ST16-12, the first unit filter
exchange display is turned on to indicate that
exchange of the column of the first treating unit is
necessary. Next, in the step ST16-13, while closing
the second discharge valve 50-1, driving of the pump
20 51 is stopped to stop the recycle operation. Next,
in the step ST16-14, the first unit discharge flag
is turned off, and while turning on the first unit
filter exchange flag, the unit change demanding flag
is also turned on. Thereafter, the operation
25 proceeds to the step ST17.

Subsequently, after following the steps ST17
and ST18, the operation returns again to the step

1 ST3 shown in Fig. 4 (A).

By practicing the respective steps as described above, purification of the aqueous dye solution is effected in the respective treating
5 units 42-1 - 42-3, whereby purified aqueous dye solution reduced in heavy metal ion concentration to lower than the setting value can be obtained in the reservoir 49.

As described above, according to the first
10 aspect of the present invention, by capturing heavy metal ions in a dye solution by practice of ion-exchange separation, heavy metal ions contained in the dye solution can be excluded and an ink can be prepared with the use of the dye solution from which
15 the heavy metal ions have been removed, and therefore it is possible to produce an ink of good quality suitable for use in ink jet recording or writing implements. Further, according to the present invention, such an ink can be produced continuously and
20 automatically.

According to the second aspect of the present invention, heavy metal ions in a dye solution are captured by practice of ion-exchange separation, only the dye solution from which the heavy metal
25 ions are removed is extracted by fractionation by practice of such ion-exchange separation, and by doing so, an ink can be prepared with the use of such a

1 purified dye solution. Therefore, it is possible
to produce an ink of good quality suitable for use
in ink jet recording or writing implements. Further,
according to the present invention, such an ink can
5 be produced continuously and automatically.

According to the third aspect of the present
invention, in preparation of ink, the heavy metal
ions in a dye solution are removed by providing a
plurality of treating sections for capturing heavy
10 metal ions by practice of ion-exchange separation
and the dye solution from which heavy metal ions
are removed with these treating sections is used for
preparation ink, whereby large scale production of
ink of good quality suitable for ink jet recording
15 or writing implements is rendered possible continu-
ously and automatically.

Further, according to the fourth aspect of
the present invention, in preparation of ink, the
heavy metal ions in a dye solution are removed by
20 providing a plurality of treating sections for
capturing heavy metal ions by practice of ion-
exchange separation and operating these treating
sections at the same time or selectively to produce
a dye solution removed of the heavy metal ions, and
25 the dye solution thus produced can be used for prepara-
tion ink, whereby a desired amount of ink suitable
for ink jet recording or writing implements can be

produced automatically and at good efficiency.

Further, by providing a plural number of treating means for capturing metal ions in a dye solution by practice of ion-exchange separation and displaying which one of these means is in state of operation, production of a purified dye solution suitable for preparation of an ink for ink jet recording, etc. can be performed simply and reliably.

Also, by providing a treating means for capturing metal ions in a dye solution by practice of ion-exchange separation and controlling the operation of the treating means based on the metal ion concentration in the dye solution after practice of such ion-exchange separation, removal of metal ions from the dye solution can be done very reliably.

Further, by providing a treating means for capturing metal ions in a dye solution by practice of ion-exchange separation, judging whether renewal of the treating means is necessary or not based on the metal ion concentration in the dye solution after practice of such ion-exchange separation, and displaying so when it is necessary, removal of metal ions from the dye solution can be done very reliably.

CLAIMS:

1. A dye purification system comprising means for producing a solution of the dye and separation means including an ion-exchange resin for capturing heavy metal ions in said dye solution, said separation means including valve means for separating dyestuff solution in which the concentration of heavy metal ions is at or below a predetermined value from dyestuff solution in which the heavy metal ion concentration is above the predetermined value.

2. A system according to claim 1 including means for recycling dyestuff solution where the heavy metal ion concentration is above said predetermined value.

3. A system according to any preceding claim including a plurality of ion-exchange separation units.

4. A system according to claim 3 including an operation control means for selectively operating any one of said units.

5. A system according to claim 3 or claim 4 including a display means which displays which one of said units is operating.

5 6. A dye purification system substantially as described herein with reference to Figs. 2 to 4 of the accompanying drawings.

10 7. An ink production system comprising a dye purification system according to any preceding claim, and an ink preparing section which prepares ink with the purified dye solution discharged from said system.

15 8. An ink production system substantially as described herein with reference to the accompanying drawings.

20 9. A method of manufacturing ink in which, prior to the addition of further ingredients, a dye solution is purified by the reduction of heavy metal ions by ion exchange on an ion exchange resin to maintain the heavy metal ion concentration below a predetermined value.

10. A method according to claim ⁹~~15~~ in which a plurality of ion exchange units are utilised in succession to allow continuous production and replenishment of spent units, under computer control.

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28.07.1983 in Japan - doc: 58136808

Title DYE AND INK REFINING SYSTEM

Applicant/Proprietor

CANON KABUSHIKI KAISHA, Incorporated in Japan, 3-30-2 Shimomaruko,
Ohta-ku, Tokyo, Japan [ADP No. 003631190271]

Inventors

MASAHIRO HARUTA, 18-8 Miyamoto 4-chome, Funabashi-shi, Chiba-ken, Japan
[ADP No. 030485920011]

KUNITAKA OZAWA, 20-6 Azumabashi 2-chome, Sumida-ku, Tokyo, Japan
[ADP No. 028557160011]

TAKASHI HAMAMOTO, 4-1104 Wakabadai 1-chome, Asahi-ku, Yokohama-shi,
Kanagawa-ken, Japan [ADP No. 030486000011]

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Canon Kabushiki Kaisha, Incorporated in Japan, 3-30-2 Shimomaruko,
Ohta-ku, Tokyo, Japan

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