FLASH WINE TITRATOR, QUICK GUIDE

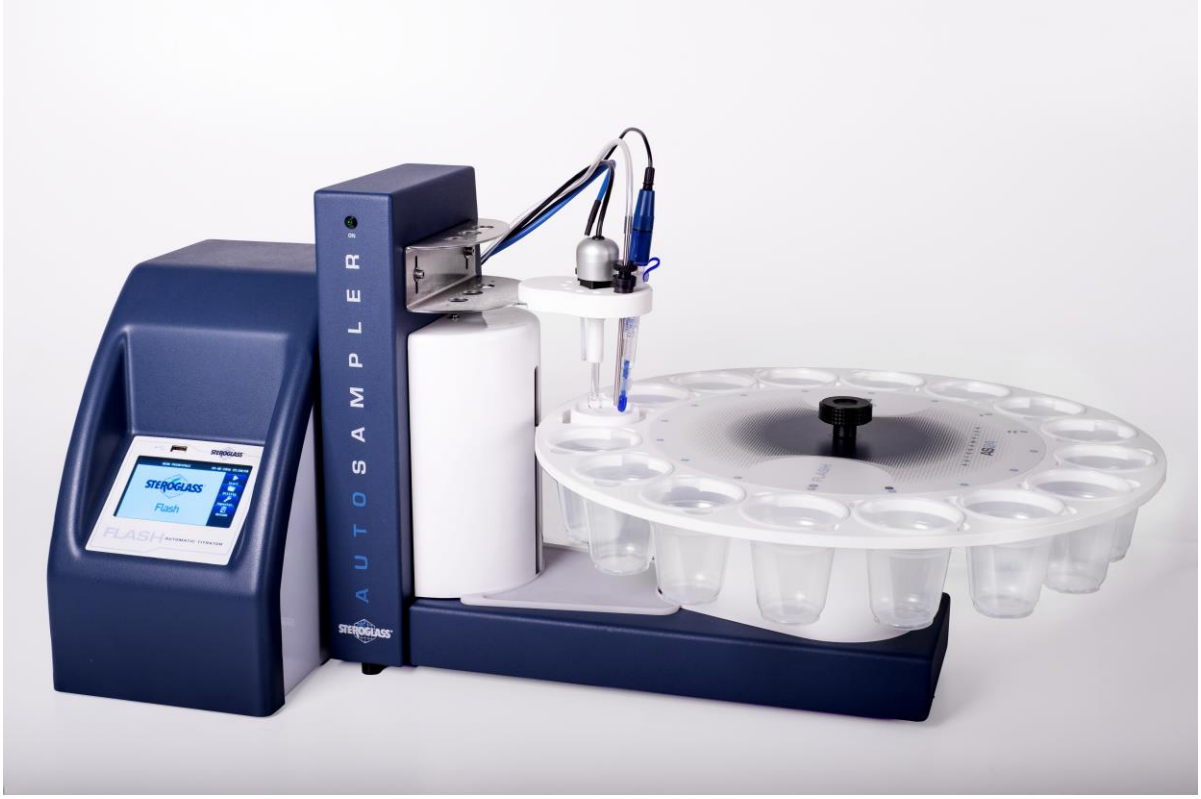


TABLE OF CONTENTS

1. FLASH CONNECTIONS

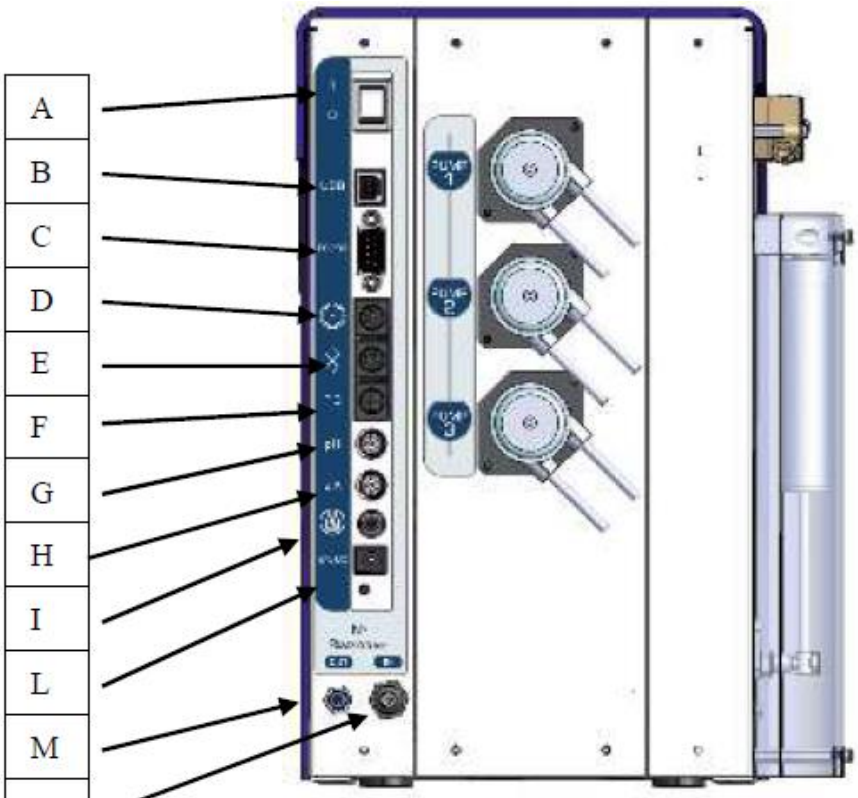


Fig. no. 1.2

Fig. no. 1.3

A	**ON/OFF Switch**
B	**USB to PC connection**
C	**RS-232 connection (Printer)**
D	**Connection with Autosampler (RS-232)**
E	**Bluetooth accessory connection**
F	**Temperature sensor connection**
G	**BNC connection for pH electrodes**
H	**BNC connection for double platinum electrode**
I	**Stirrer support connection**
L	**External power connection**
M	**Nitrogen outlet connection**
N	**Nitrogen inlet connection**
O	**USB flash drive connection**

2. SWITCHING ON

Switch on the instrument with the switch on the back.
Switch on the autosampler, too, if it is connected.
FLASH will ask to initialize the burettes; always press OK.
NEVER press STOP until the procedure (which lasts a few seconds) has been completed.
Open the EASYDATA software if the instrument is connected to an external PC.

3. MAIN MENU FUNCTIONS

START: Use this section to launch an analysis.
UTILITY: From this section you can wash or rinse the burettes; do pH calibration, check the results of analyses and standardization, use the Measurements function, etc..
SETTINGS: From this section you can change the general instrument settings (the default password entered is FLASH); check the burettes and the operation of the autosampler and peristaltic pump.
METHODS: Here you can store up to 30 different methods and select 10 of them as favorites (red star).

4. STARTING

If the burette circuit is empty or has been washed, it must be rinsed with the titrant needed for the method (for example Sodium Hydroxide, 0.25N for Total Acidity).
Go to UTILITY → RINSING → Select the Burette number, select the number of cycles (3 are sufficient), select the titrant recovery option (we suggest selecting NO).
If the instrument is connected to the autosampler, select the discharge beaker in position 15 (check that there is an empty beaker in position 15). Then press NEXT.
The instrument reminds you to check that the filling tube is inserted in the titrant reagent bottle; confirm with NEXT.
Check that the reagent comes out of the burette tubes (colored blue) into the discharge beaker.
In addition, always check that the peristaltic pump circuit is properly filled (e.g. for dosing nitric acid in the determination of chlorides).
If this is not the case:
Go to Settings → Peristaltic → Select peristaltic pump 1 and 2 to activate them; you must see the reagent coming out of the peristaltic pump tubes (colored red) into the discharge beaker.
With the AS24 autosampler, the third peristaltic pump (if present) can be used to automate the dosing of the sample: connect the suction tube to the desired height and set sufficient time to suction the excess sample, which is sent to the discharge.

5. APPLICATION NOTES

The following guidelines are intended as a brief reference for users to help them understand the analysis technique.
It is obviously impossible to provide detailed and specific information that would cover all possible cases of analyses.
It will be up to the users to optimize the analyses for their specific samples.

6. HOW TO PERFORM THE ANALYSIS OF TOTAL PH/ACIDITY IN WINE

PRINCIPLE
The total acidity (TA) of a wine or a must is the sum of all the titratable acids that bring the wine to pH 7.0 by adding a standardized sodium hydroxide solution (NaOH). It is commonly expressed in g/l of tartaric acid (or sulfuric acid).
Given that the acidity due to the CO2 content must not be included in the determination of total acidity, the wine sample must be properly degassed prior to the analysis.

INSTRUMENT AND ACCESSORIES
- Flash Titrator with at least one burette
- Single analysis stand or AS24 automatic sampler
- pH electrode (recommended with PTFE collar)
- Temperature sensor

REAGENTS

- Titrant: Sodium hydroxide solution (NaOH) 0.25 M

NOTES:

- This titrant concentration is recommended when analyzing sample volumes of 30 to 40 ml; for higher volumes, use 0.5 M NaOH.
- If the initial detection of the pH value is not required, the analysis can be performed on a 10 ml wine sample using 0.1 M NaOH. Since the PTFE collar of the electrode must be immersed in the sample, 30-40 ml of freshly distilled, CO2-free water must be added to the wine.
- NaOH solutions can be standardized by titration of a known quantity of a primary standard (hydrogen potassium phthalate).

SAMPLE PREPARATION

Samples with a considerable CO2 content must be degassed in an ultrasonic bath (for 5 minutes) or by stirring for several few minutes or by filtering through paper. The wine can also be poured into a suitable container connected to a vacuum suction system for 2-3 minutes. It is not necessary to filter turbid samples.

AUTOMATIC DEGASSER

The FLASH can be supplied with an automatic degassing function. If the degassing option is installed, the inlet must be connected to the nitrogen line or cylinders, always remembering that a pressure reducer is required. The maximum acceptable pressure for the Flash degasser input is 0.3 bar / 4.35 psi (check carefully).

1) Connection:

A tube with an external diameter of 6 mm must be used to connect the Flash inlet to the line with a maximum nitrogen pressure of 0.3 bar / 4.35 psi (always check the pressure reducer specifications).
To connect the Flash degasser to the sampler (single stand or automatic sampler), it is recommended that you use the Flash degasser outlet tube (item no. SQTR074977) and the Flash nitrogen flow regulator (item no. SQTR076887), which will help you accurately regulate the flow of nitrogen into the sample. Remember that this regulator cannot be used to reduce the pressure from the nitrogen cylinder, but only to regulate the flow from the Flash to the sample!!

2) Use:

The degasser is run directly in the TOTAL ACIDITY method and functions as a peristaltic pump (it mechanically consists of a solenoid valve). This means that the user must set the time in seconds for the opening of the solenoid valve in the method and save it (according to the degassing method).
It can be set from 0 to 999 seconds; for still wines, a time between 240 and 360 seconds is usually sufficient, and for sparkling wines a time of 480 to 600 seconds should be set. The user can change this value during installation according to the CO2 concentration of the samples.

PRELIMINARY OPERATIONS

Check that the burette and the fluid circuit are rinsed with and full of titrant.
Verify that the pH electrode is present in the titration holder. We suggest that the pH electrode be calibrated every day before starting the analyses:
1) Insert the pH electrode into the titration holder.
2) Go to the UTILITY menu and select CALIBRATION.

3) Select the type of calibration you prefer. Usually we recommend performing the “classic” “AUTO 2 BUFFER” calibration (pH 7 and 4 buffers) or the special “A and B” calibration (pH 7 and 3 buffers can be used).
The instrument will ask you to insert two buffers in sequence, follow the instructions on the display.
4) The electrode is considered good if at the end of the procedure the asymmetry is within the range of +/- 20 mV and the efficiency is between 90 and 110%.
To double-check that the calibration is good, you can:
Go to UTILITY>MEASUREMENTS> measure the buffer pH.
Readings should be between pH 4.00 +/- 0.03 and pH 7.00 +/- 0.03. If the values do not correspond to this, repeat the pH calibration.

If it is already calibrated:
1) Go to START from the main menu and select the TOTAL acidity method;
2) If connected to the autosampler, enter the number of samples present, e.g. “2”, and place the beakers in the sampler;
3) If you want to give a name to the samples, enter the name in SAMPLE DESCRIPTION;
4) Press START to start the analysis.

```
*****************************************
            PRINT METHOD
Date:   29-08-2019     Hour: 15:53:49
*****************************************
Method type             End Point
Method name             TA
Descip./Cod. sample     SAMPLE
Pump level N:           0
Pump level sec:         0
Degassing sec:          0
Agitator speed          6
Time of pre-agitation   20
Measure type            pH
Initial auto-stability(pH)0,02
Initial auto-stability time(s)5
Initial addition        0,00
Initial agitation       3
Titrant burette         1
Type addition           Progressive
Addition(ml)            0,25
Limiting volume(ml)     30,00
Polarization value      NA
Value End Point(pH):    7,00
Auto-stabil(pH)         0,05
Auto-stab time (s)      1
Auto-stab max time(s)   60
Delay end titration(s)  3
Factor                  75,0000
Concentration (mol/l)   0,2500
Sample Volume(ml)       50,0
Result units            g/l
Decimals number         2
Approaching factor      250
Back titration vol(ml)  0,00
Blank(ml)               0,00
Washing type            Washing  position
Washing time(s)         5
Reagent Standardization NO
Type equation           Default
```

Total acidity (TA) in wines and musts can be expressed in two ways:

- as g/l of tartaric acid
- as g/l of sulfuric acid

Based on the settings listed in this table, the results are expressed in g/l of tartaric acid. To obtain the results expressed in g/l of sulfuric acid, just replace the value for the "Factor" parameter with **49.00**.

NOTES:

1) The initial addition can be set from **0 to 9 ml** depending on the sample, the volume and the concentration of the reagent

2) For the United States, it is necessary to set the end point value (pH): **8.20**

3) For the analysis of grape must, we suggest setting the Sample Volume (ml) at **10.00.** Thus it is necessary to add about 40 ml of distilled water to carry out the titration. The initial pH value is not taken into account, but the TA result will be correct.

4) If using the FLASH connected to the **AS24 MICRO sampler**, set the following values:
Pre-stirring time: **10**
Initial addition: **0**
Limit volume (ml): **10.00 to 15**
Sample volume (ml): **10.00**

7. HOW TO PERFORM THE ANALYSIS OF FREE SO2 IN WINE

PRINCIPLE

Free sulfur dioxide in wine or must is understood as both that present in the gaseous state (SO2) and that present in the form of sulfuric acid H2SO3, HSO3- and $SO^{=}$.
Sulfur dioxide is determined by iodometric titration (Ripper method): the S02 is oxidized by iodine in accordance with the reaction:

$$SO_2 + I_2 + 2\ H_2O \rightarrow H_2SO_4 + 2\ HI$$

INSTRUMENT AND ACCESSORIES

- Flash Titrator with at least one burette and two peristaltic pumps;
- Single analysis stand or AS24 automatic sampler;
- Double platinum electrode.

REAGENTS

- Titrant: Iodine 0.01 M (0.02N) + 30 g/l of Potassium Iodide (KI) (For each liter of titrant add about 30 g of KI - the salt dissolves instantly)
- Sulfuric acid 25% w/v (to prepare 1 liter of solution, start with 250 grams or 140 ml of concentrated H2SO4 and bring to volume with distilled water H20).

NOTES:

- The iodine concentration is optimized for the method indicated below, but higher (maximum 0.025 M or 0.05 N) or lower (minimum 0.005 M or 0.01N) concentrations can also be used.
- The iodine solution can be standardized by titration of a known quantity of sodium thiosulfate: see Appendix.

SAMPLE PREPARATION

No preparation is necessary. Avoid long waiting times between sampling and analysis, which could cause loss of SO2 by evaporation and oxidation due to exposure to air.
It is recommended that the analysis be performed on a sample volume of not less than 25 ml and not more than 50 ml; however, it is possible to work with lower volumes, as long as the platinum electrode terminals are immersed, adding distilled water if necessary.

PRELIMINARY OPERATIONS

Check that the burette and the peristaltic pump circuit have been rinsed with titrant. Check to ensure that the double platinum electrode is in the titration holder.
1) Go to START from the main menu and select the FREE SO2 method;
2) If connected to the autosampler, enter the number of samples present, e.g. “3”, and place the beakers in the sampler;
3) If you want to give a name to the samples, enter the name in SAMPLE DESCRIPTION;
4) Press START to start the analysis;

```
*****************************************
            PRINT METHOD
Date:   29-08-2019      Hour: 15:55:29
*****************************************
Method type             SO2 Free
Method name             FREE SO2
Polarization value      200
Pump level N:           0
Pump level sec:         0
Acid pump N:            1
Acid pump sec:          2
Value End Point(uA):    1,000
Titrant burette         1
Addition(ml)            0,05
Limiting volume(ml)     20,00
Initial addition        0,00
Auto-stabil(uA)         0,100
Auto-stab time (s)      0
Auto-stab max time(s)   60
Agitator speed          6
Time of pre-agitation   0
Initial agitation       0
Delay end titration(s)  3
Factor                  64000,0000
Concentration (mol/l)   0,0100
Sample Volume(ml)       50,0
Result units            ppm
Decimals number         0
Descip./Cod. sample     Sample
Washing type            Washing  position
Washing time(s)         5
Reagent Standardization NO
```

FREE SO2 RESULTS

The program shown here gives the results directly in ppm of SO2 by applying the following formula:
ppm SO2 = (ml * C * 64000) / V

where:
- ml: ml of titrant reagent consumed
- C: concentration of the titrant reagent (mol/l)
- V: Volume (ml) of the sample taken

Note:

1) If using the FLASH connected to the AS24 MICRO sampler, set the following values:
Pre-stirring time: **10**
Initial addition: **0**
Limit volume (ml): **10.00 to 15.00**
Sample volume (ml): **5.00 to 10.00**

8. HOW TO PERFORM THE ANALYSIS OF TOTAL SO2 IN WINE

PRINCIPLE

Total sulfur dioxide in wine or must is understood as the sum of SO2 that is free and combined, or linked to the other substances in the wine (aldehydes, ketones, etc.); the latter is returned to the free state before titration by means of an alkaline attack and subsequent re-acidification.
Sulfur dioxide is determined by iodometric titration (Ripper method): in an acid environment the S02 is oxidized by iodine in accordance with the reaction:

$SO_2 + I_2 + 2\ H_2O \rightarrow H_2SO_4 + 2\ HI$

INSTRUMENT AND ACCESSORIES

- Flash Titrator with at least one burette and two peristaltic pumps
- Single analysis stand or AS24 automatic sampler
- Double platinum electrode

REAGENTS

- Titrant: Iodine 0.01 M (0.02N) + 30 g/l of Potassium Iodide (KI) (For each liter of titrant add about 30 g of KI – the salt dissolves instantly)
- Sulfuric acid 25% w/v (To prepare 1 liter of solution, start with 250 grams or 140 ml of H2SO4 concentrate and bring to volume with distilled water H20).
- Sodium Hydroxide (NaOH) 4N (To prepare 1 liter of solution weigh 160 grams of NaOH in flakes, dissolve with distilled water H2O and stir).

NOTES:

- The iodine concentration is optimized for the method indicated below, but a higher (maximum 0.025 M or 0.05 N) or lower (minimum 0.005 M or 0.01N) concentration can also be used.
- The iodine solution can be standardized by titrating a known quantity of sodium thiosulfate: see Appendix.

SAMPLE PREPARATION

No preparation is necessary. Avoid long waiting times between sampling and analysis, which could cause loss of SO2 by evaporation and oxidation due to exposure to air.
It is recommended that the analysis be performed on a sample volume of not less than 25 ml and not more than 50 ml; however, it is possible to work with lower volumes, as long as the platinum electrode terminals are immersed, adding distilled water if necessary.

PRELIMINARY OPERATIONS

Check that the burette and the peristaltic pump circuit have been rinsed with titrant. Check to ensure that the double platinum electrode is in the titration holder.
1) Go to START from the main menu and select the method TOTAL SO2;
2) If connected to the autosampler, enter the number of samples present, e.g. “3”, and place the beakers in the sampler;
3) If you want to give a name to the samples, enter the name in SAMPLE DESCRIPTION;
4) Press START to start the analysis;

```
*****************************************
            PRINT METHOD
Date:   29-08-2019     Hour: 15:59:28
*****************************************
Method type             SO2 Total
Method name             TOTAL SO2
Descip./Cod. sample     SAMPLE
Pump level N:           0
Pump level sec:         0
Alkaline pump N:        2
Alkaline pump sec:      5
Response time of alk.(min):5
Acid pump N:            1
Acid pump sec:          8
Agitator speed          6
Time of pre-agitation   2
Initial addition        0,00
Initial agitation       5
Titrant burette         1
Addition(ml)            0,05
Limiting volume(ml)     25,00
Polarization value      200
Value End Point(uA):    1,400
Auto-stabil(uA)         0,050
Auto-stab time (s)      0
Auto-stab max time(s)   60
Delay end titration(s)  5
Factor                  64000,0000
Concentration (mol/l)   0,0100
Sample Volume(ml)       50,0
Result units            ppm
Decimals number         0
Washing type            Washing  position
Washing time(s)         5
Reagent Standardization NO
```

TOTAL SO2 RESULTS

The program shown here gives the results directly in ppm of SO2 by applying the following formula:
ppm SO2 = (ml * C * 64000) / V

where:
- ml: ml of titrant reagent consumed
- C: concentration of the titrant reagent (mol/l)
- V: Volume (ml) of the sample taken

Note:

1) If using the FLASH connected to the AS24 MICRO sampler, set the following values:
Pre-stirring time: **10**
Initial addition: **0**
Limit volume (ml): **10.00 to 15.00**
Sample volume (ml): **5.00 to 10.00**

9. HOW TO PERFORM THE ANALYSIS OF DOUBLE SO2 IN WINE (FREE AND TOTAL SO2 ON THE SAME SAMPLE)

PRINCIPLE

In order to make the analysis even more automatic, FLASH allows you to perform free SO2 and total SO2 determinations on the same sample. First the free SO2 is titrated in an acid medium, then the combined SO2 (returned to the free state by an alkaline attack and subsequent re-acidification). The sum of the two values represents the total SO2. Sulfur dioxide is determined by iodometric titration (Ripper method): in an acid environment the S02 is oxidized by iodine according to the reaction:

$SO_2 + I_2 + 2\ H_2O \rightarrow H_2SO_4 + 2\ HI$

INSTRUMENT AND ACCESSORIES

- Flash Titrator with at least one burette and two peristaltic pumps
- Single analysis stand or AS24 automatic sampler
- Double platinum electrode

REAGENTS

- Titrant: Iodine 0.01 M (0.02N) + 30 g/l of Potassium Iodide (KI) (For each liter of titrant add about 30 g of KI – the salt dissolves instantly)
- Sulfuric acid 25% w/v (To prepare 1 liter of solution, start with 250 grams or 140 ml of H2SO4 concentrate and bring to volume with distilled water H20).
- - Sodium Hydroxide (NaOH) 4N (To prepare 1 liter of solution weigh 160 grams of NaOH in flakes, dissolve with distilled water H2O and stir).

NOTES:

- The iodine concentration is optimized for the method indicated below, but a higher (maximum 0.025 M or 0.05 N) or lower (minimum 0.005 M or 0.01N) concentration can also be used.
- The iodine solution can be standardized by titrating a known quantity of sodium thiosulfate: see Appendix.

SAMPLE PREPARATION

No preparation is necessary. Avoid long waiting times between sampling and analysis, which could cause loss of SO2 by evaporation and oxidation due to exposure to air.

It is recommended that the analysis be performed on a sample volume of not less than 25 ml and not more than 50 ml; however, it is possible to work with lower volumes, as long as the platinum electrode terminals are immersed, adding distilled water if necessary.

PRELIMINARY OPERATIONS

Check that the burette and the peristaltic pump circuit have been rinsed with titrant. Check to ensure that the double platinum electrode is in the titration holder.

1) Go to START from the main menu and select the method DOUBLE SO2;
2) If connected to the autosampler, enter the number of samples present, e.g. “3”, and place the beakers in the sampler;
3) If you want to give a name to the samples, enter the name in SAMPLE DESCRIPTION;
4) Press START to start the analysis;

```
*****************************************
            PRINT METHOD
Date:   29-08-2019      Hour: 15:59:56
*****************************************
Method type             SO2 Double
Method name             DOUBLE SO2
Descip./Cod. sample     SAMPLE
Pump level N:           0
Pump level sec:         0
Acid pump N:            1
Acid pump sec:          3
Alkaline pump N:        2
Alkaline pump sec:      8
Response time of alk.(min):5
Acid pump sec:          10
Agitator speed          6
Time of pre-agitation   0
Initial addition        0,00
Initial agitation       0
Titrant burette         1
Initial add. Combined   0,00
Addition(ml)            0,05
Limiting volume(ml)     20,00
Polarization value      200
Value End Point(uA):    1,400
Auto-stabil(uA)         0,100
Auto-stab time (s)      0
Auto-stab max time(s)   60
Delay end titration(s)  5
Factor                  64000,0000
Concentration (mol/l)   0,0100
Sample Volume(ml)       50,0
Result units            ppm
Decimals number         0
Washing type            Washing  position
Washing time(s)         5
```

DOUBLE SO2 RESULTS

The program shown here gives the results directly in ppm of SO2 by applying the following formula:
ppm SO2 = (ml * C * 64000) / V
where:
- ml: ml of titrant reagent consumed
- C: concentration of the titrant reagent (mol/l)
- V: Volume (ml) of the sample taken

The results will appear on the display in sequence:

1) free SO2
2) combined SO2
3) total SO2

Note:

1) If using the FLASH connected to the AS24 MICRO sampler, set the following values:
Pre-stirring time: **10**
Initial addition: **0**
Limit volume (ml): **10.00 to 15.00**
Sample volume (ml): **5.00 to 10.00**

10. APPENDIX

10.1 STANDARDIZATION OF IODINE

Iodine is photosensitive, thus we recommend that you always keep it in dark glass bottles and periodically check the titer. This can be done quickly with the FLASH using the same program dedicated to free SO2, as follows:

- Carefully pipette 1 ml of 0.1 M sodium thiosulfate (0.1 N) or 10 ml of 0.01 M sodium thiosulfate (0.01 N) into a reaction beaker. Add about 40 ml of distilled water.
- Analyze the sample using the free SO2 method (do not use the automatic auto level option).
- Take note of the equivalent volume (Ve, in milliliters) of iodine used and apply the following calculation:

$$c(I_2)\ (mol/l) = 0.1 / (V_e * 2)$$

You can use the **automatic standardization method**: modify the FREE SO2 method, save it with another number and call it "STANDARD IODINE - IODINE STD."

Modify:

Volume (ml):	1.0
Result unit:	Factor
Concentration (mol/l)	0.1
Factor:	0.5
Reagent standardization:	YES

Launch the standardization method on 3 samples with 1 ml of 0.1 M sodium thiosulfate (0.1 N) and 40 ml of distilled water.

At the end, go to UTILITY → RESULT → STANDARDIZATION to check the results found.

Click on MEDIA, confirm the titer calculated by the FLASH and save the new iodine titer found after the standardization in the FREE, TOTAL and COMBINED methods.

10.2 INTERFERING SUBSTANCES IN THE DETERMINATION OF SO2

Anomalous behavior can be seen in the titration of some wines: near the end point, the polarization current recedes several times before the titration is considered finished, with the risk of overestimating the amount of SO_2 in the sample.

This behavior occurs more frequently with red and especially deep red wines.

To attenuate this phenomenon, it helps to add potassium iodide (KI) to the wine, in one of two ways:

- A spatula-tip full of KI (salt) in each sample; or
- About 2 ml of 30% KI solution (300 g/l) in each sample.

In this way the titration has a more regular trend and one can see a slight decrease and greater repeatability of the results obtained.

10.3 ESTIMATE OF THE CONTRIBUTION OF INTERFERING SUBSTANCES

If you suspect that other reducing substances have made a significant contribution in the determination of SO2, it is advisable to evaluate the amount of iodine consumed by them in the titration. To do this, fix the SO2 with an excess of acetaldehyde before titration.

Proceed as follows:

- Dose in the reaction vessel the same quantity of wine used for the free SO2 analysis;
- Add an aliquot of 7 g/l acetaldehyde solution (approximately 1 ml per 10 ml of wine, e.g. 5 ml per 50 ml of sample);
- Wait at least 30 minutes (capping the titration beaker is recommended);

- Titrate with the same program used for free SO2.

In this way, an estimate is obtained of the interferents already expressed in ppm of SO_2; this will be the value to be subtracted from the result obtained by analyzing wine not treated with acetaldehyde. Since this contribution is considerable only in the case of wines with added ascorbic acid, this method can be used to estimate its content in mg/l.
To do this, a dedicated titration program can be used that differs from the one described above only in the factor, which will no longer be 64000 but 176130.

11 WASHING CYCLES

How to empty and wash burettes ... when should it be done?
It is strongly recommended that you wash the burette and leave it empty before the weekend or before a long period in which the titrator will not be used. This guarantees longer life for syringes and valves.
1) Go to UTILITY→WASHING→E.g.: Burette number 2, number of cycles 5, titrant recovery NO.
2) The instrument reminds you to place a discharge beaker and that the filling tube must be inserted into a container of DISTILLED WATER.
Confirm by pressing NEXT.
3) When the instrument has done about 3 washing cycles, remove the suction tube from the water container and keep it in the AIR to empty and dry out the internal circuit.
Let the procedure end.
4) Turn off the FLASH (and the autosampler, if it is connected)

12 LIST OF POSSIBLE ERRORS

Err.1 ERRORE_STAB_NON_PRESENTE // reading not stable, maximum stability time exceeded

Err.2 ERRORE_VOL_MAX_SUPERATO // maximum limit volume reached. End of analysis with error

Err.3 ERRORE_VOL_MAX_SUP_LIB // maximum limit volume reached in free SO2 analysis. End of analysis with error

Err.4 ERRORE_VOL_MAX_SUP_TOT // maximum limit volume reached in total SO2 analysis volume. End of analysis with error

Err.5 ERRORE_STAB_NON_PRES_LIB // reading not stable, maximum stability time exceeded (error in Free SO2 while executing the DOUBLE SO2 method)

Err.6 ERRORE_STAB_NON_PRES_TOT // reading not stable, maximum stability time exceeded (error in Total SO2 while executing the DOUBLE SO2 method)

Err.7 ERRORE_BIANCO_MAGG_VOLEQ //blank greater than equivalent volume
(vol Eq – Blank)<0

Err.8 ERRORE_VOL EQ_MIN_AGG_INIZ // analysis completed with the sole contribution of the initial addition.

Err.9 ERRORE_VOL EQ2_MIN_AGG_DOPP // analysis completed with the sole contribution of the initial addition (Double SO2 Method).

Err.10 ERRORE_LETT_INZIZ_LIB // initial reading (uA) above the maximum threshold before starting the dispensing of titrant in the SO2 method.

Err.11 ERRORE_LETT_INIZ_TOT // initial SO2 reading above the threshold before dispensing something (electrode conditioning or after free addition too large)

Err.12 ERRORE_VOL EQ_MAGG_BIANCO //Calculation error in the case of (Blank - VolEq) with Blank<Veq

Err.13 ERRORE_EQUAZIONE //Management error: no equation

13 TROUBLESHOOTING

The following table gives the most frequent problems and their possible solutions.

Problem	***Cause***	***Solution***
Flash does not start or it stops during operation	*- The device may be improperly connected to the external power supply* *- The switch could be turned off*	*- Reconnect correctly the plug correctly and the red ground connector.* *- Turn on the switch*
Flash does not activate the second burette	*- The second burette could be set as "absent" in the settings menu.*	*- Set burette number 2 as "present" in the settings menu*
Flash does not activate the AS24 sampler	*- The sampler is turned off* *- Cable disconnected* *- The sampler is not selected; check in the Settings menu.* *- The sampler has not completed the initialization procedure.*	*- Switch on the sampler* *- Connect the cable* *- Set the sampler present and select the right model (AS24 with 16 or 35 positions).* *- Wait for the sampler to finish its initialization procedure.*
The temperature is not displayed	*- Sensor disconnected* *- Temperature sensor not set; check in the settings menu.*	*- Connect the sensor*
Does not read the pH	*- Input connector disconnected or connected to the wrong channel (uA)*	*- Connect the input connector*
Does not read the uA or mV	*- Input connector disconnected or connected to the wrong channel (pH)* *- Double platinum electrode tips are*	*- Connect the input* *- Clean the electrode tips with paper;*

	dirty or are touching each other. *- Faulty double platinum electrode or cable.*	*if they are very dirty and darkened keep them immersed in a 1:1 water/5% bleach solution for 5 minutes, then rinse with distilled water.* *- If the tips touch each other, gently separate them with your fingers.* *- If the electrode still does not read anything, set a polarization value of 200mV from the Measurements menu, touch the two tips simultaneously with a metal object (key, paperclip) and read the measurement in uA: the reading should be high (full scale).* *If the reading is 0 uA, the cable or double platinum electrode must be replaced.*
Does not print	*- Printer turned off* *- Printer disconnected* *- Printer not enabled in the Settings menu.*	*- Turn on the printer* *- Connect the printer* *- Enable the printer in the Settings menu*
No stirrer	*- The stirrer is disconnected* *- The speed is too slow* *- The stirrer blade movement is blocked by dirt* *- The magnetic anchor is missing on the single-position stand.* *- The single-position stand connector is loose or detached.*	*- Connect the stirrer* *- Increase the speed* *- Turn off the AS24 sampler and try to move the stirrer blade with your hands. If there is resistance, spray DRY contact spray between the white plastic cylinder and the motor and continue rotating manually until the stirrer is released.*
- **The liquid titrant** is not sucked up by the burette, **but you do NOT hear unusual noises**	*- The filling tube is not inserted in the bottle.* *- The filling tube is too long and forms a kink inside the bottle.* *- "Valve reversal NO" has been set by mistake* *- Burette not initialized.*	*- Check that the titrant tube is inserted in the right bottle and that the tube is not kinked or pinched.* *- Check on the Settings menu that the "valve reversal" parameter is set to YES.* *- Turn the instrument off and then on again to initialize the burettes; do not press STOP.*
- The **liquid titrant** is not sucked up by the burette, and **you hear beeps and unusual noises**	*- Clogged antidiffuser* *- Clogged filling tube* *- Faulty solenoid valve* *- Faulty burette motor card*	*- Remove and clean the shaped antidiffuser (see section 16)* *- Unscrew the filling tube from the valve and check that it is free of obstructions.* *- Unscrew and remove the hexagonal screw that moves the syringe piston and try to start a rinse: if the unusual noises cease, the solenoid valve is*

		faulty and must be replaced. *- If the unusual noises and beeps continue even after removing the hexagonal screw, the burette motor card is faulty and must be replaced.*
Analyses are not precise, peaks in the graph	*- Ground connector disconnected* *- There are two electrodes in the titration beaker at the same time*	*- Reconnect the ground connector* *- Remove the electrode that is not being used and put it in the rest position outside the titration vessel.*
The sulfur dioxide analysis with the Double method (Free + Combined on the same sample) does not work well.	*- The soda (NaOH) is not dispensed* *- Not enough sulfuric acid (H2SO4) is dispensed* *- Check that the concentrations of the auxiliary reagents Sulfuric Acid and Soda have not changed; try replacing the reagents, rinse the pumps.*	*- Check that the peristaltic pump of the soda is dispensing and that the tube is not clogged (about 1 ml/sec)* *- Check that the peristaltic pump of the acid is dispensing and that the tube is not clogged (about 1 ml/sec)* *- Replace the bottles of the sulfuric acid and soda auxiliary reagents.*
The analyses with self-leveling are not repeatable	*- Tube or self-leveling pump are clogged.*	*- Check the self-leveling circuit and if necessary disconnect the L-fittings from the peristaltic pump to understand where the obstruction is in the circuit. Disconnect the fitting from the yellow (soft) tube to avoid breaking the plastic L-fitting.*
The analyses with self-leveling are repeatable but are underestimated or overestimated	*- Self-leveling pickup tube has moved*	*- Check that the black guide of the self-leveling tube is well inserted* *- The height of the self-leveling tube has changed and the volume must be rechecked*
The analysis results in general are not repeatable	*- The titrant reagent tubes (blue) are not inserted for picking up in the sample* *- A titrant reagent tube has lost the antidiffuser* *- Auxiliary reagents tubes (red) picking up in the sample*	*- Check that the blue titrant tubes pick up in the sample* *- Check that the antidiffusers are present and are clean* *- Check that no auxiliary reagent tubes (red) are immersed in the sample.*
Air bubbles in the circuit	*- Black screw fittings are not tight* *- Glass syringe not screwed on correctly* *- Glass syringe leaks considerably*	*- Check the tightness of the black screw fittings* *- Check that the glass syringe is screwed on correctly.* *- Replace the defective syringe*

14 SYRINGE REPLACEMENT

- Before replacing the syringe, it is necessary to make sure that the filling tube is positioned outside the titrant bottle and is empty if possible (rinse with distilled water and then with air)

Figure 8.2

- Bring the burette to the low position by going to the menu Settings > Burette > Syringe MAX position.
- Unscrew and completely remove the hexagonal screw as shown in Fig. 8.2

- Loosen by hand by one turn the black screw fitting located above the syringe, and only after this is done, unscrew by hand the glass syringe, grasping the metal part (Fig. 8.2.1).
You can use pliers to grip the syringe only on the metal part if it is not unscrewed by hand. Check that the white washer seal has remained in position inside the brown plastic and did not remain attached to the old syringe.
- Screw in the new syringe all the way, grasping it only by the metal part.
- Tighten the upper black screw by hand
- Lower the plunger until the position of the metal hole is aligned with the thread on the burette and reinsert the hexagonal screw, tightening it without forcing it.

Figure 8.2.1

- Go to the menu Settings > Burette> Burette Calibration. The instrument automatically calibrates the minimum position of the new syringe.
- Reinsert the filling tube into the titrant bottle and run a rise (3 cycles) to eliminate air bubbles.

15 SOLENOID VALVE REPLACEMENT

- Turn off the instrument and disconnect the power supply connector.

Figure 8.2.2

- The valve has 3 fluid connections: disconnect the inlet, outlet and lower fitting tubes by unscrewing the black fittings by hand.

- Unscrew the 4 screws that fasten the blue plastic chassis (circled in red in Figures 8.2.3 and 8.2.4). Be careful not to mix up the screws when you reassemble the chassis: the two upper screws are self-tapping and have a pointed tip.


Figure 8.2.3

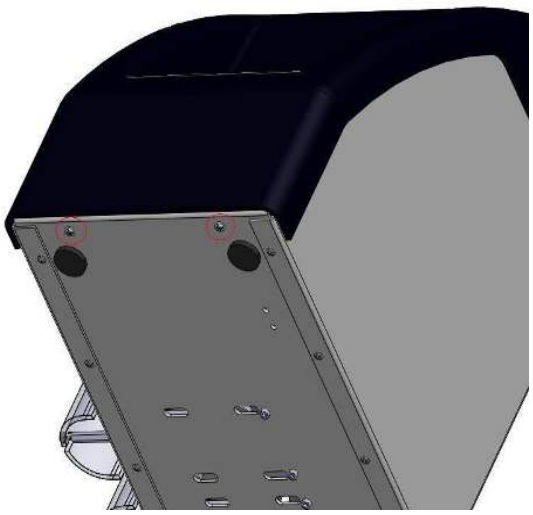


Figure 8.2.4

- Set the plastic chassis gently on one side, being careful not to force the flat gray cable.

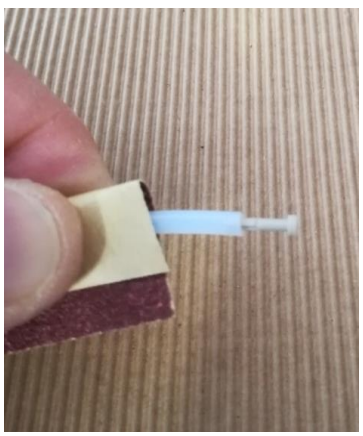
- Remove the metal clip that holds the black coil fastened to the valve as indicated by the red arrow in Fig. 8.2.5) by pulling it to the side.

- Unscrew the two Phillips head screws that fasten the valve to the chassis (Fig. 8.2.2).
- Recover the 3 white seals from the old valve by tapping it on the table or use 3 new washer seals.
- Take the new valve without fastening it to the instrument and connect the three screw fittings of the tubes after inserting the white seal in each connection.
- Fasten the solenoid valve to the structure using the two steel Phillips screws.
- Insert the new coil on the solenoid valve and secure it with the metal clip as shown in Fig.8.2.5.
- Reassemble the chassis and fasten it with its 4 screws, remembering that the 2 upper screws are the ones that have a pointed tip. Check that the gray flat cable is fully inserted in the two connectors.
- Check the tightness of the fittings by hand before turning the instrument back on.
- **After having replaced the solenoid valve, remove and clean the antidiffuser, run 3 start-up cycles without the antidiffuser and then reassemble it**. This eliminates scale, buildup or residues in the tubes that could obstruct the circuit.

Figure 8.2.5

16 CLEANING THE ANTIDIFFUSER

The titrant reagent tube ends in the titration beaker with a small grooved cap called the ANTIDIFFUSER, which prevents the sample from rising back up into the tube.
If the antidiffuser is clogged, the titrator will not dispense the titrant, and it must be removed to clean it.
Grip the PTFE tube with a piece of sandpaper, otherwise it will be slippery.
With the help of a knife, remove the cap from the white tube **without cutting the tube**.
Clean the antidiffuser by keeping it immersed in 5% bleach for a few minutes, then rinse and thoroughly clean the lateral groove. Reinsert the antidiffuser using the sandpaper.



17 PERISTALTIC PUMP HEAD REPLACEMENT

- Before replacing a pump head, suction distilled water and then air in order to empty the circuit (go to the menu Settings > Peristaltic and activate the desired pump)
- Disconnect the inlet/outlet tubes by gently grasping the L-shaped connector.
- Remove the head by grasping the clips on both sides as shown in Figure 8.2.6.
- Remove plastic residues or dirt from the motor shaft using dry paper.
- Insert the new head onto the motor shaft gently until you hear a click. Check that it is properly fastened by pulling it towards you.
- Connect the two L-shaped connectors of the filling/discharge tubes to the new head.

Figure 8.2.6

18 UPDATING THE SYSTEM

The Flash Titrator can be updated to the latest firmware release using a simple procedure. When switching on the instrument, it shows the current release for a few moments (e.g. v 1.04)
To update the system, it is necessary to have the original USB flash drive supplied with the instrument and to upload onto it the update file sent by Steroglass.
With the instrument turned off, insert the USB flash drive into the USB port located near the display.
When the instrument is turned on, the update starts.
Do not turn off the instrument during this phase.
At the end of the update, the tool asks to initialize the burettes; press OK.
The instrument can now be turned off and the USB flash drive removed.
When turning on the instrument, check the new release displayed, e.g. (1.09)
Delete all the results in memory after updating UTILITY> RESULTS> delete.
Check the factor and titrant concentration in the methods.