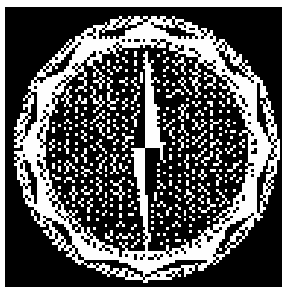


GGUN-FL

Fluorometer

Users Manual



Neuchâtel
Switzerland

	GGUN-FL Fluorometer 080811	2
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I - Specifications

- Detectable tracers : classes I / II / III¹ one of each optional IV or V
- Turbidity : 0.02 – 100 NTU (FL30: 400)
- Temperature measurement : 2°C up, 0.01°C sensitivity
- Detection threshold : $< 2 \times 10^{-11}$ g/ml (uranine)
- Saturation level : 2500 mV
- Electrical noise : 0.01 mV
- Analog to digital converter : 24 bit
- Measuring interval : 2 s up (1 lamp) to 15 min / ext
- Digital output : RS232, 9600, 8 bits, 1 start, 1 stop, no parity (3 wires).
- Power supply : 6 V (probe accepts 6-12 V)
- Battery capacity (1-2) : 12 - 24 Ah
- Consumption (stdby/normal) : 1.5 / 50 mA
- Connections : The probe connects to the data logger through a 4-wire cable with 2 waterproof connectors.
- Immersion depth : FL2X=70 m FL30 =50 m
- Head weight : FL2X=2 kg FL30=7.3 kg
- Data logger weight : 6.4 kg (with 2 batteries)

Warning : Never invert (+) and (-) battery terminals !

¹ I uranine, pyranine, eosine, chlorophyll A
 II amidorhodamine G, sulforhodamine B, rhodamine WT
 III Tinopal CBS-X, CBS-CL, amino-G-acid , photine CU
 IV duasyn brilliant yellow T
 V naphthionat

Quick start-up

1. Insert an initialised (see Appendix 1) Compact Flash card (CFC) into the data logger slot. Our cards are already initialised.
2. Attach the 6V battery terminals (red wire = "+").
3. Select one to four lamps with the rotary switches 1-4. Always select lamp #4 (turbidity), so that turbidity effects can be minimised. Always selecting the 4 lamps is recommended.
4. Select a sampling rate with the rotary switch SR (see Appendix 2 for timing). Two cases:
 - *No PC connected.*
Select SR=1 to 9. Switch power on. Remark: The settings of the 5 rotary switches are only active at power on. At completion of the tracer test, stop the data logger, move the CFC to a PC or external card reader and download the data with the programme FLUO (button Read FlashCard, see App. 3).
 - *A PC is connected to the RS232 connector through a serial cable.*
Using an optical bridge (optional – not necessary if the PC is not connected to the mains) will suppress ground loop perturbations. Select SR=0. Run the FLUO programme (see Appendix 3). Switch power on. In this case, switches 1-4 are disabled and lamp selection is done through the PC. The data are written to a PC file and on the Compact Flash card.

II - Overview

The fluorometer is employed for continuous measurements during field tracer tests using dyes such as uranine, rhodamine, Tinopal, etc. As many as three conveniently selected tracers can be used simultaneously. Water flows through the optical cell of the fluorometer (a glass tube of cylindrical cross-section). The optical system comprises four lamps and three photo detectors mounted on four perpendicular axes on two levels. Each axis is equipped with excitation and detection filters and lenses. The lamps are switched on and off in turn, measuring three independent responses and water turbidity. The offset voltage of the preamplifiers (dark signal) is also recorded at each measuring cycle (FL22 limitation: 2 lamps, 2 photodiodes, 1 tracer + turbidity).

The fluorometer sonde hosts an analog to digital converter to convert the fluorometer signals into unipolar 24-bit words (data resolution 1:16'000'000). The data flow is sent to the data logger through a 15 m-long or longer cable (4 wires). The data logger box contains one or two sealed lead batteries and the necessary circuitry for recording the data.

III - Operation

In clean and degassed water, the background noise is very stable (≈ 0.01 mV). The residual signal is produced

- by light scattering of water
- by reflections in the optical cell.

Under these ideal conditions, as little as 2×10^{-11} g/ml of dye concentration can be detected (uranine). Other dyes are a factor of 8 to 10 less sensitive. Furthermore, the detection threshold increases with turbidity.

The signal measured with an empty optical cell is not a good indicator of the level. This value is always larger than the signal of clean water.

Set-up

To avoid bubble accumulation in the optical cell, the fluorometer should be set up vertically. Air bubbles scatter the light and strongly degrade the signal quality by producing peaks.

FL30 only: Completely immerse the probe into water and secure it with a heavy brick (5 kg or more) or a nylon rope. If the flow is slow, the electrical cable is strong enough to hold the probe in place. For stronger flows, tie down the probe. Rotate it so that the water inlet (lower cylinder) is orientated upstream and the outlet downstream.

All probes: Connect the signal cable. Use only fully charged batteries. Note that the acquisition programme measures the charge of the battery and stops when the voltage drops below 5.8 V during 10 successive samples. When restarting the acquisition after a power interruption, the memory card is overwritten.

IV - Controls

Calibration

Calibration is advisable 2-3 times a year, permitting proper system operation.

FL2X calibration is done by immersing the probe. For the FL30, simply pour the liquid into the probe, after removing the 2 caps and installing a stopper.

Calibration is done by using known standard solutions:

1. clean water
2. tracers at 10^{-6} , 10^{-7} and/or 10^{-8} g/ml (Caution: use the same product for calibration and tracer test. There are significant differences between manufacturers!).
3. formazine suspension for turbidity calibration at 1, 10 and 100 NTU (see appendix).

FL30 probe: To reach the optical cell, it is necessary to remove the two cylindrical caps. Both are secured with three screws. Close the lower inlet of the glass tube with the provided rubber stopper. Gently pour in the calibration solution to prevent bubble formation (very important!). Put one of the caps on the top to prevent daylight from entering during the measurement.

All probes:

Rinse one or two times (and more for Tinopal). Copy the measured mV data into your calibration file (CALIBRAT.DAT) without modifying its format. Example:

```
-----Full calibration of tracer 1-----
2      Number of calibration lines, increasing concentration
-8  28.70      log(ppb) & signal (mV)
-7  278.05     log(ppb) & signal (mV)
-----Full calibration of tracer 2-----
2      Number of calibration lines, increasing concentration
-7   16.80     log(ppb) & signal (mV)
-6  160.81     log(ppb) & signal (mV)
-----Full calibration of tracer 3-----
2      Number of calibration lines, increasing concentration
-7   100.44    log(ppb) & signal (mV)
-6  1014.87    log(ppb) & signal (mV)
```


In this Table, tracers 1, 2 and 3 could be uranine, rhodamine and Tinopal assigned to optics 1, 2 and 3. There is a line indicating the number of concentrations used for calibration (here, 2). The line starting with [-8 28.70...] says that for a concentration of tracer 1 of 10^{-8} , lamp 1 gives a signal of 28.70 mV.

Alternatively, use the calibration utility CAL30 from the distribution software. This program creates a new file CALIBRAT.NEW. Rename it to CALIBRAT.DAT at the end.

Above 10^{-6} g/ml, signal saturation may occur. Consequently, if accurate readings are necessary at higher concentrations, calibration should be extended toward higher concentrations and calibration lines added at 10^{-6} and 10^{-5} g/ml.

Also edit the 100 ppb value in the 1st part of the table (with Notepad or Wordpad, never with Word):

```

-----Tracer #1-----
Uranine
L1  278.05
L2   3.75
L3   7.45
L4  540.20
-----Tracer #2-----
SrhomadamineB
L1  18.40
L2  16.80
L3   1.42
L4  536.50
-----Tracer #3-----
TinopalCBS-CL
L1   1.16
L2   0.46
L3  100.44
L4  543.51
-----Turbidity-----
1 NTU
L1   1.00
L2   0.42
...

```

The FLUO programme will use the polynomial interpolation only when **one tracer** is selected. If more than one is selected, then the calibration will assume a linear response and use only the response for the 100 ppb concentration given in the above Table.

V - Maintenance

To ensure optimal accuracy, the glass tube should be cleaned before each tracer test and after 2 weeks at least. There is no need for opening the fluorometer. Only remove the 2 small caps (FL30 probe). Use a Nylon tube brush. For the FL2x sonde, only use brushes that have no folded back rod, otherwise the O-ring seals could be pulled out.

Optical filter set

	Excitation	Detection
filter 1: class I	Wratten blue	Wratten orange
filter 2: class II	Wratten cyan	Wratten light red
filter 3: class III	UG11 UV filter	Wratten cyan
filter 4: (turbidity)	Wratten dark-red	Same as 2

Kodak Wratten filters are sensitive to humidity and heat and should be replaced (FL2X: at the factory) if a significant increase of the water signal relative to the original value is observed (see calibration file). This is the main reason why the probe should not be used in waters with temperature exceeding 35 °C.

FL30 probe: In case of probe opening: To close the cell the 6 main nuts must be tightened to a torque of 8 ± 0.5 Nm using a dynamometric key. Values outside this range may lead to water leakage.

The weakest part is the connector. Always insert the plug to the very end so that the O-ring seal works fine. Allow complete dry-up of the connectors before closing with the protective caps.

VI - Input /output format

Following comments are intended for designers. They are not necessary for running the fluorometer. The FL cell communicates through the 4-wire signal cable connected to the data logger or to a PC serial port. Port 1 is assumed (Germany: 2). If your PC uses port 2, then edit this entry in the file CALIBRAT.DAT. Always use non-formatting editors such as Wordpad or Notepad, not Word.

When power is applied, the cell sends the string "WELCOME" and waits for a command from the host. The command must be an upper-case character, between A and O giving the channel configuration. The Table gives the full set of the channel configuration:

Lamp	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1 Blue	x		x		X		x		x		x		x		x
2 Green		x	x			x	x			x	x			x	x
3 UV				x	X	x	x					x	x	x	x
4 Red								x	x	x	x	x	x	x	x

Remark: If a conductimeter exists, bit 16 is set to 1. In this case, codes start from P. The cell interprets the command and performs the measurement. An ASCII string is sent to the host. The first character is the given command. Then, for each lamp the message consists in 2 double numbers. The first is the photo-detector signal before lamp illumination, and the second is the signal during illumination. Following formula computes the signals by combining the 2 parts of the double number:

$$s = 2^{16} n_1 + n_2$$

The next double numbers give the battery voltage and the temperature. If there is a conductimeter, the next numbers are its voltage and current. Then comes the sample number, the time from start in seconds and finally, the check-sum. The signals can be converted into mV through multiplication

by the factor $2500 / 2^{24}$. A signal of 200 mV corresponds to a temperature of 20°C.

Examples:

without conductimeter

	baseline	signal	1	battery	temperat	sample	second	date	hour	checksum
A 001 29902	001 38112	104 48010	023 31981	00015	0000515	040902	103933	008		

with conductimeter

baseline	signal 1	battery	temperat	voltage1	current1	sample	second	date	hour
checksum									

Q 001 29737	001 38068	104 48109	023 32131	000 07621	000 07071	00016	0000525	040902	103943
007									

...

VII - Appendix

Appendix 1 - Compact Flash card (CFC)

Only the brand and capacity of the provided CFC is supported. Our CFC's come fully formatted and initialised. Use SanDisk cards (the only tested brand). A CFC must be re-formatted on your PC and initialised before data can be written to it. Use the FLUO programme to do this (Appendix 3), or better, the FLASH.EXE programme. This programme writes a special file FLASHCAR.DAT to the CFC. A CFC should contain only this file. Check with a PC that the file really exists on your CFC. For information, the file initially contains a long string of spaces (ASCII character 32).

Once a CFC has been initialised, it is not necessary to repeat this operation before a new tracer test. However, it is good practice to check the presence of the file. Do not erase the CFC between 2 recordings. The new data overwrites the old one. Use programme FLUO (Appendix 3) to read data written to the CFC. If new data have been written inadvertently, the part which has not been overwritten can be retrieved (Retrieve button). Do not initialise a CFC that contains other files. Reformat it first.

Appendix 2 – Available sampling period

The following table gives the available sampling periods selected with the rotary switch SR:

display	0	1	2	3	4	5	6	7	8	9
seconds	external	minimum	10 sec	30 sec	1 min	2 min	4 min	5 min	10 min	15 min

Setting SR=1 disables the optional conductimeter. This allows for shorter sampling periods (from 2 seconds up) useful for stream gauging (only one lamp – lamp 1, 2 or 3).

Appendix 3 – Programme FLUO

The programme FLUO has different functions controlled by a ring slide:

- **New acquisition**

Acquisition of new data. Data is not only recorded on the CFC, but also on the PC. A file is created. Its name is YYYYMMDD#UUU.MV, based on the current year, month and day. #UUU is the serial number of your fluorometer, as found in the calibration file CALIBRAT.DAT. This file with .mv extension contains data in millivolts. Data collected the same day are appended to the file. This file is also useful for real-time remote access by internet. Click STOP to exit acquisition mode.

- **Process mV**

This mode is used for opening a file of .mv extension and computing the .ppb file, i.e. the concentrations in ppb. Graphic information is displayed. Use this button to calculate responses in different ways. The .ppb file can be opened with MS Excel. In the upper-right corner of the FLUO screen there is a cursor labelled “turbidity sensitivity”. Its value can be set between 0 and 1. This is a multiplicative factor acting on the turbidity correction applied to the tracer concentration. A good setting should cancel any visible correlation between the turbidity and the breakthrough curves. For Tinopal, a value of 0 is convenient. Changing this factor will recalculate the .ppb file. Do not process .mv files with multiple tracing runs. Edit them first, with Notepad or Wordpad, keeping only one run (only one zero in column #).

The Process mV mode can be used for calculating the discharge of a stream. See full instructions at the end of the manual.

- Read CF

This utility is used for CFC initialisation and data download. Click “Extract...”. A new window opens. Look for the file FLASHCAR.DAT located on the disk unit representing the memory card (D:, E:, etc.). A second window opens. Type a new name (such as “ABC”) for the new data. The file extension is always .mv. Data are read in, and the number of records is displayed. When the red light is off, click “Quit”. To get concentration and turbidity, see the “Process MV” paragraph. Use Retrieve to retrieve previous data.

- Calibration

The calibration utility CAL30 can also be used outside of the FLUO programme. This utility allows for fluorometer calibration. Prepare 100 ppb standards for tracers and 1, 10, 100 NTU for turbidity. For tracers use also a set of concentrations between 1 ppb and 10 ppm (2 contiguous concentrations such as 10 and 100 ppb are sufficient). Simple calibration is used when several (2 or 3) tracers are present in the water. The mathematical separation is based on linear equations. In this case, the fluorometer response to tracer concentration is supposed linear, and thus, only one concentration (100 ppb) is used in the calibration. A second calibration is necessary for more accurate polynomial interpolation of the fluorometer response. However, this method works only for solutions of one tracer. Water calibration is also important. Use distilled or microfiltrated water standed a few days (to avoid air bubbles).

Calibration procedure:

Do not use tracer cocktails for calibration.

Phase 1:

The first step consists in selecting the 3 tracers with the ring boxes.

Then repeat phase 2 for water and turbidity, and phases 2 and 3 for the dye tracers.

Phase 2:

-Water: Start acquisition with data logger set on SR=0. Double click Water. Then click Calibrate. Data read from calibration file CALIBRAT.DAT appears in the "Old values" textbox. Newly acquired data are written in the yellow textbox whereas mean values appear above. After the mean values get stable (3-5 samples) click "Save to file". Note that Tinopal data decrease permanently because UV light bleaches the fluorescence. In this case do not attempt to obtain a mean. Just keep the first measurement. A CALIBRAT.NEW file is created from the existing CALIBRAT.DAT. New data replace old ones. Reset the mean (Reset mean button) if a datum looks wrong. This occurs at the beginning when a measurement of the previous concentration is still in the buffer of the serial interface of the PC.

-Tracers (uranine, rhodamine, etc): For 100 ppb concentration, repeat these steps as for water.

Phase 3:

To calibrate a second concentration (10 or 1000 ppb) click the corresponding boxes at the bottom of the screen. Optionally, before doing this, right click the concentrations to select them. They must be contiguous.

At the end of the whole process, rename CALIBRAT.NEW to CALIBRAT.DAT.

Remark: If the FLUO programme stops with an error message while using the new CALIBRAT.DAT, check in this file that the 4 data of each tracer and turbidity are at least slightly larger than those of water. Manually correct them if necessary.

Appendix 4 – Conductimeter

An optional conductimeter is installed on the fluorometer head. Each probe requires individual calibration.

Conductimeter probe set-up

The probe is waterproof (O-ring seal). If the probe is not installed, use the provided plastic cover to protect the connector on the fluorometer head. Match the red mark orientation while inserting the probe.

Calibration

At 25°C a 0.01N KCL solution displays a conductivity of 1412 $\mu\text{S}/\text{cm}$. To calibrate the probe, immerse the fluorometer head in such solution (preparation: 0.7456 g of pure (chemical grade: puriss.) and dry KCL in one litre of distilled water). The thermometer of the fluorometer head provides the calibration temperature. Run the FLUO programme. Note down the conductivity V. Calculate the correction factor with

$$c = 1412 / V$$

Stop the programme and open the calibration file (CALIBRAT.DAT) with Notepad or Wordpad (do not use Word). Look for the line “« FL Conductimeter gain, T compensation”, near the end. Replace the first number (a value in the 500-1000 range) by itself multiplied by c. Close the file. Check with the FLUO programme that the new conductivity is now within 1% of 1412 $\mu\text{S}/\text{cm}$.

Principle of the conductimeter corrections

This paragraph is indicative. Unless improved accuracy is necessary, there is no need for changing the following parameters. Two corrections are applied to the value furnished by the conductimeter:

1. Temperature correction
2. Non-linearity correction

First, the measured value is divided by a polynomial $y = 1 + ax + bx^2$ whose coefficients a and b are given in the line “« FL Conductimeter gain, T compensation” of the calibration file, in position 3 and 4. The variable is $x = T - T_{REF}$. The temperature T_{REF} (here 25°C) is the reference (position 2 of the line). The result of the division is W. The next step is to convert W into $\mu S/cm$ by multiplying it with the value in position 1 of the line.

Non-linearity is calculated in the log-log space. A polynomial $y = c + dx + ex^2$ is calculated. Its coefficients c, d and e are given in the next line of the calibration file “Non-linearity correction” in position 1, 2 and 3. The variable is $x = \log_{10}(W)$.

The final result is computed as 10^y .

Remarks

(Applies to models S: surface (FL30) D: down hole (FL2X))

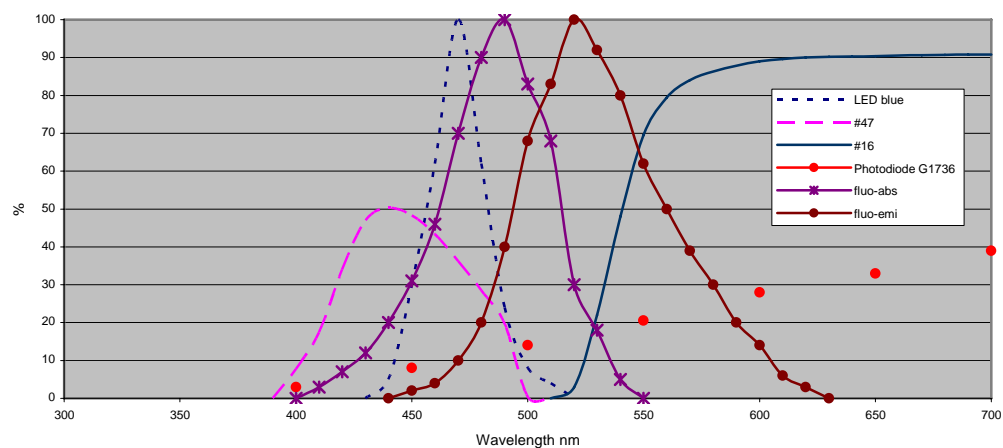
1. (S) The fluorometer works even with very slow water flow rates. In a stream, it is sufficient to set it up with the cable upwards. Azimuth is not critical. If it is not possible to fully immerse the fluorometer, connect a tube to the lower inlet. Water can be forced to flow either by gravity or with a small (<1 litre/min) peristaltic pump. Important: Air bubbles should be avoided as much as possible, as they generate noise.
2. (S) It is possible to analyse water pumped from a borehole. However, some degassing may occur. In this case, water should be passed through a degassing box prior to the analysis.
3. (S,D) Simultaneous use of 3 tracers is possible (only one tracer per class, however). Good separation will be achieved depending on the turbidity. Precise calibration is mandatory.
4. (S) When used in the laboratory, the fluorometer allows for fast tracer analysis. Fill the glass tube slowly, to avoid bubbles. Use a 50 ml plastic beaker and a syringe ended with a short tube. Rinse out two or more times (in particular for Tinopal).
5. (S,D) Warning: Using a PC connected to the mains may cause data instability due to ground loops. Use an optical coupler such as the Expert OptoBridge -----
<http://www.gude.info/index.php?lng=0§ion=products&product=optoalg>
6. (S,D) The internal clock keeps the current date and time. Its power supply comes from a battery of 10 years lifetime. After battery replacement (positive terminal up), use following procedure to set up the time:
 Connect a PC with the serial cable. Start programme FLUO (Version 23.0 or higher). On the screen, select only tracer 1 (generally, uranine). Select « Sampling period » = 10.

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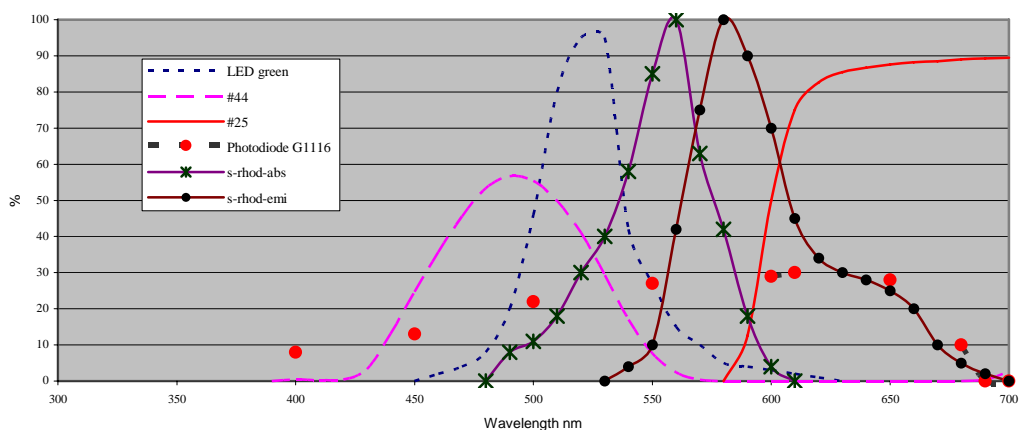
If a button « Conductimeter » appears on the screen, select Conductimeter = NO. Click « New acquisition ». On the data logger, select SR=0, then switch power on. The PC transmits the date and time to the data logger. A pop-up message appears. Click OK.

7. (S,D) In case of freezing danger, make sure the optical tube contains no water.

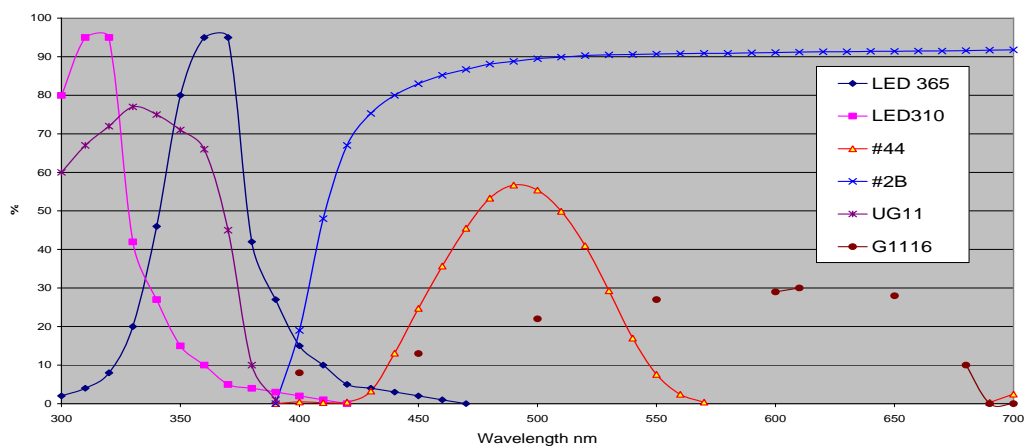
Uranine (fluorescein)



Rhodamine family

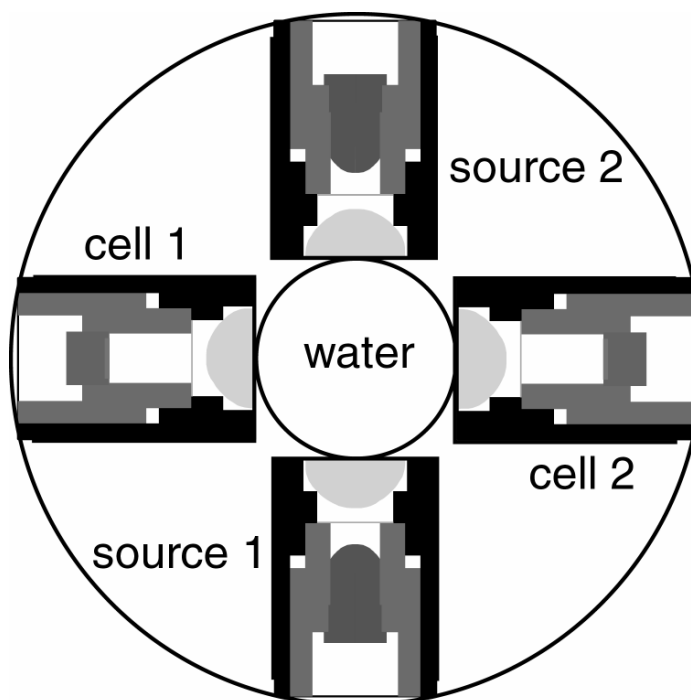


Colourless dyes



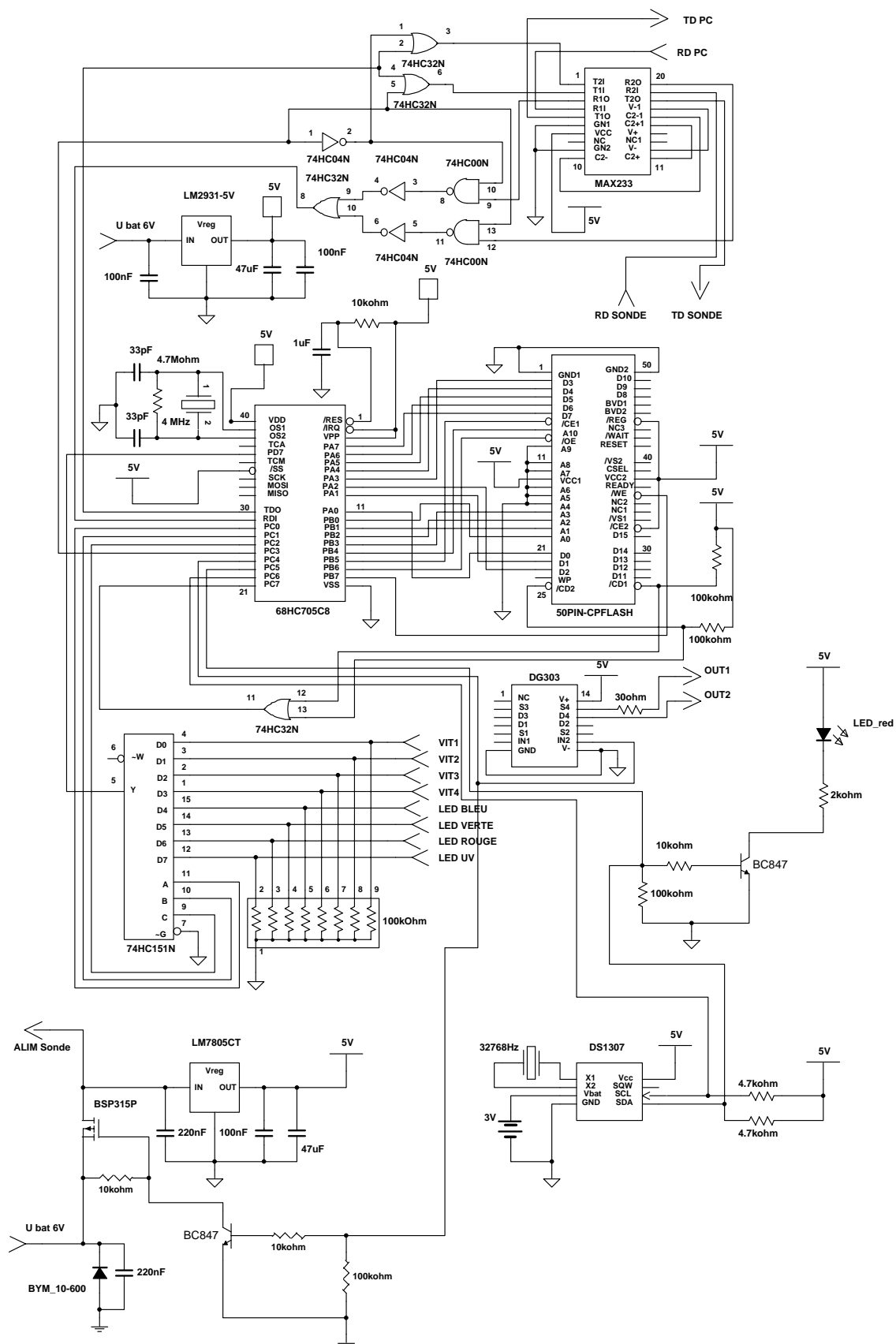
[illegible]

Nb	Pieces	with data logger	w/out data logger
1	Waterproof data logger		
1	Fluorometer head		
1	Signal cable		
1	Carry case		
2	Software for Windows 9X/NT/XP on CDrom (+ calibration file)		
2	Compact Flash card (1 GB)		
1	Compact Flash card adapter		
1	Battery charger 6 V 15 W (Europe)		
2	Sealed lead battery 6 V / 12 Ah		
1	Serial interface cable		
1	Hexagonal screwdriver #5		
1	Nylon tube brush		
1	Rubber stopper (FL30 only)		
2-3	Quick connect fittings		
1	Wratten filter blue, cyan (2x), orange, red (FL30 only)		
1	Pyrex or quartz tube (FL30 only)		
2	Fuses T 1A		
1	Service manual		
1	Adapter cable to PC		

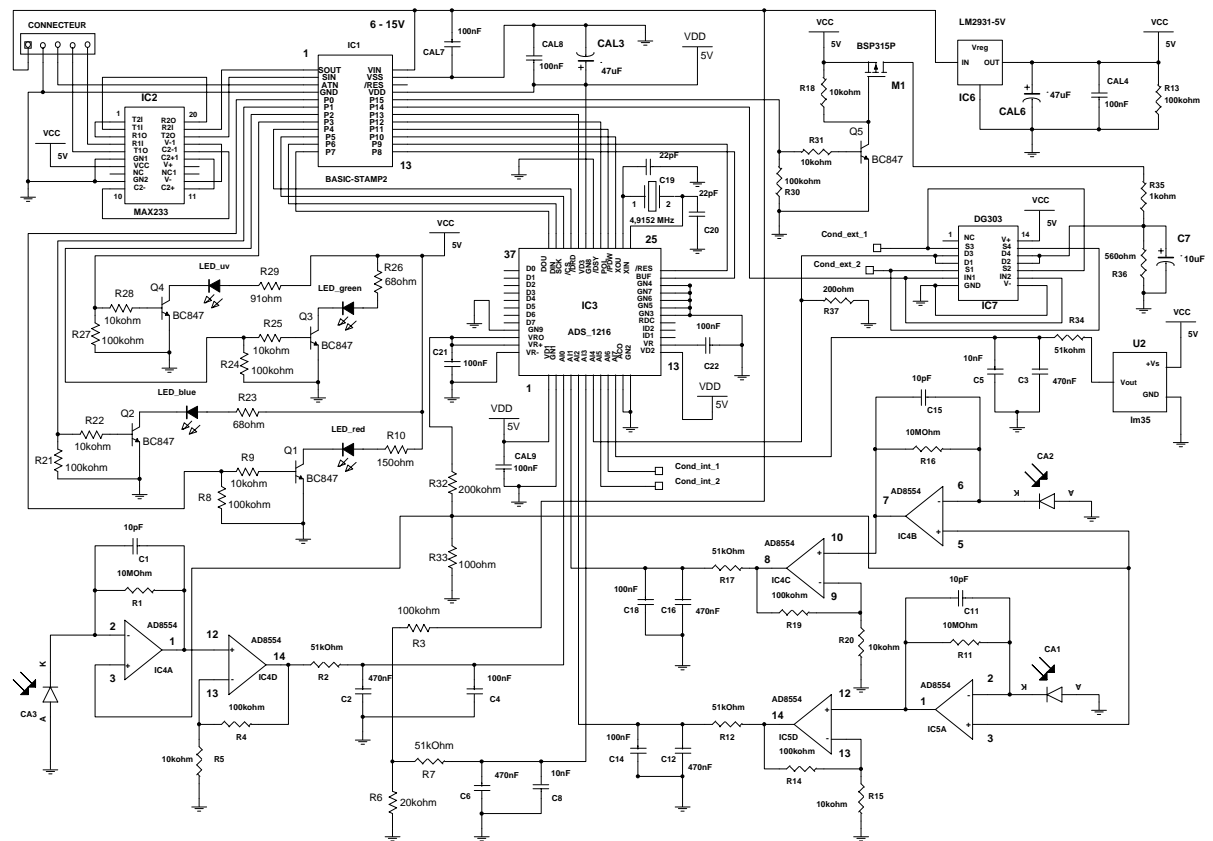


FLUOROMETER
(one level seen)

Data logger



Probe circuitry

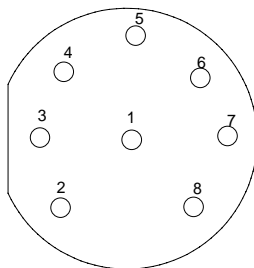


Cable and connections

Name	Wire colour	FISCHER 8 poles	HONDA 5 poles	FISCHER 11 poles
+5V	red	1,2,3	1	9,10,11
Serial out	brown	6	4	3,6
Serial in	green	7	5	2,7
GND	blue	4,5,8	2	4,5,8

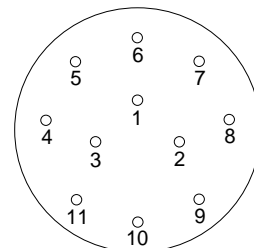
FISCHER connector
8 poles type 105

welding side



FISCHER connector
11 poles type 104

welding side



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Nephelometric method

Formazine suspension as turbidity standard

Sol 1

1 g hydrazine sulphate $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ in 100 ml water

Sol 2

10 g hexamethylene tetramine $(\text{CH}_2)_6\text{N}_4$ in 100 ml water

End solution

100 ml sol 1 plus 100 ml sol 2. Leave for 24 hours at 25 °C.

The turbidity of this solution is by definition **4000 NTU**.

The solution is stable for 1 year in the dark.

Tap water

Turbidity threshold wished: 0.02 NTU. Achieved in tap water filtered with 0.1 micron pore size filter.

Stream gauging

The mode “Process mV” can be used for calculating the discharge of a stream, by using the formula $Q = A \int_0^{\infty} c(t) dt$

where Q is the discharge, A is the tracer mass and c(t) is the concentration measured by the fluorometer. Before starting in this mode, go to the setup mode and

1. select one (and only one) tracer and deselect the turbidity,
2. click « Process mV »,
3. open the .mv file with the discharge peak,
4. move the red marker before the peak,
5. move the blue marker after the peak and double-click there,
6. click OK and enter the tracer mass (grams) followed by <enter>,
7. The discharge is displayed.
8. Restart 4 to 6 at will.

The installation folder contains the file EXAMPLE.MV that can be used to exercise this mode. A mass of 1000 grams produces a discharge of about 52245 litres/s.

For a very precise calibration and gauging scheme, see our separate Application Note: “High-precision stream gauging (Global method)”.