Operation Manual of CHEMOTRONIC III

Owner's Manual

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Contents:

1. Safety Precautions

1.1. Safety instructions

Read operation manual before starting to work with the instrument. Make sure, that every operator fully understands every function of the instrument and each of its components.

Working with solvents and high temperatures does not need to be dangerous when all safety recommendations are understood and obeyed by the operator

1.2. Instrument Safety

- When mineral oil is used as a solvent, make sure its boiling point is over the max. level of the temperature profile of **230°C**. If not, **decrease this level to the boiling point** of the solvent less 20°C or more.
- Put the magnetic stirring bar **gently into the glass test tube**, by tilting the tube.
- In case a glass test tube would break, **the instrument must be switched off immediately**. All the solvent in the metal jacket holding the test tube must be removed, before switching on again.
- **Never use a glass test tube twice.**
- Wear **heat protection gloves** when removing a hot test tube or cleaning a hot temperature sensor tip.
- **Do not remove any part** from the instrument during operation.
- If you have **doubts about safety** when a situation occurs that is not mentioned in the operation manual, contact the manufacturer or its representative.

Before switching on:

- Ensure that the power cable plug is connected to a main socket with an earth contact.
- Ensure that the temperature sensor is inserted into the test tube and the cable is connected to CHEMOTRONIC.

- The CHEMOTRONIC has three hot spots during a run:
  1. the visible edge of the glass test tube holder
  2. the air outlet of the cooling box at the rear
  3. the air outlet of the case at the rear

Never touch these and be sure to have a minimal distance to any object of about 10 centimetres

The instrument will automatically heat up the sample in the test tube as fast as possible (approx. 1°C per sec.). After having reached the top temperature, this temperature level is hold constant for several minutes (hold time setting) in order to dissolve all solid particles. Then the sample is cooled down as fast as possible (approx. 0.5°C per sec.). Turbidity is detected automatically and the cloudpoint temperature value is printed out via the optional data printer simultaneously.

There are 3 variable rotation speed settings for the magnetic stirring bar:

- n1 during heating up (fastest speed for optimal mixing)
- n2 during hold time and cool down (slow speed to avoid air bubbles)
- n3 below T_3 (< 80°C faster speed for optimal homogeneity)

1.2.1. Measuring devices

Temperature measurement:	Pt 100 temperature sensor (100 Ohm resistance thermometer9 Accuracy: ± 0.5°C
Rotation measurement:	Digital tachometer on motor shaft. Magnet stirring bar will follow the rotational speed during the sample is in liquid state. Accuracy: ± 1 rpm
Turbidity measurement:	Infrared transmitter and receiver devices with glass fibre extension leads. The tips of the glass fibre leads can be cleaned through the metal test tube holder with a brush. Accuracy: ± 10 NTU (nephelometric turbidity units)

All 3 values are displayed on digital indicators at the front of the instrument, by which also the setpoints can be adjusted. The min. and max. values of the turbidity range can be set by individual calibration:

P1: min. turbidity by a transparent sample

P2: max. turbidity by an intransparent sample
(for instance solidified solution)

1.3. Calibration

The following turbidity calibrations can be used:

Ac = automatic calibration, applicable to Std-program and user programs U1 – U8.

Max. sensitivity is a setting at 90 % of the max. clearness of the solution.

Min. sensitivity is a setting at 10 % of the max. clearness of the solution.

Pc = program specific calibration.
As Ac, but max. and min. of the turbidity range must be defined by the user. For this the calibration function must be used for each individual program.

Ec = external calibration:

This is a global calibration for all test programs. The turbidity range can be set by using the calibration function.

Remember:

The factory setting for the calibration mode is Ac, so that there is no need to use a standard turbidity solution for calibration.

1.3.1. Introduction

This chapter is providing information on the basic concept of the cloud point measurements of resins for applications in printing inks.

In lithography printing conventional oil-based paste inks perform best. These inks consist of resins, mineral oils, pigments and additives. Therefore the performance of these inks is highly depending on the compatibility of all these components. In order to test the compatibility of a resin and a mineral distillate, the resin must be dissolved in the mineral distillate at a high temperature first. When cooling this solution, the resin will precipitate in the liquid at a specific temperature, which is called the cloud point. The cloud point can accurately be detected by automatic turbidity measurement with the CHEMOTRONIC.

Turbidity is defined as an optical property of a liquid caused by light scattering and absorption. This state will be caused by unsolved particles, for instance fine polymers or organic material. These particles solved in a liquid lead to haze or turbidity, if the light passes through the liquid. Easily said, turbidity is the opposite of clearness.

In case of a resin dissolved in oil, the precipitation process increases rapidly within a small temperature interval until full solidification of the varnish. The temperature at which this happens is the so-called solidification point which is closely related to the cloud point. Deviations in the cloud point can lead to sedimentation in the varnish, differences in drying speed, loss of gloss and tack stability of printing inks. Therefore the cloud point is a characteristic value for the specification of printing ink resins.

In the past the cloud point detection could be determined on the manual way only. This test method had many sources of error and uncertainty due to poor reproducibility, variances in heating and cooling of the sample, inaccurate turbidity determination, etc.

The CHEMOTRONIC determines the cloud point of hard resins in solutions based on mineral distillates of analytical quality. To determine the cloud point accurately and reproducibly, the temperature of the test liquid must be cooled down under reproducible conditions until a reduction of transparency due to the effect of suspended particles, i. e. turbidity occurs. The cooling rate plays a dominant role in these local saturation and precipitation processes. However, the transparency reduction due to colour changes must be eliminated in turbidity measurements. The observed effect of the liquid on light passing through it will change in case of colour changes, but the turbidity will not change. Therefore infrared light is generally chosen for turbidity measurements to reduce the absorption effect caused by different colours. However, turbidity is not empirical. It depends on the effect of suspended particles on infrared light passing through the liquid, which in turn depends on the similarity of the process and the measurement each time.

As the turbidity measurement must be combined with the temperature measurement, a special infrared-photocell-system has been designed for the CHEMOTRONIC.

This fibreglass infrared-photocell-system measures the absorption of the infrared light beam passing through the glass test tube. The spectral response of the silicon photo diodes shows a max. sensitivity at approx. 950 nm, which is well beyond the region of visible light (380 - 780 nm), so that effect of visible colour changes is eliminated generally. This makes the photocell more sensitive for low turbidity measurements.

The output signal of the photocell detection circuit decreases linear with increasing turbidity within the relevant turbidity range. The user can adjust which turbidity point is recognized as the cloud point by executing a calibration procedure and choosing one of ten turbidity levels.

The fibreglass infrared-photocell-system for turbidity measurement in the CHEMOTRONIC III has proven to be a rugged and reliable device, designed for continuous use in factory environment, if the tips of the fibreglass are kept clean.

2. Operation modes

In accordance with the practical requirements of modern quality managment systems two operation modes were implemented into the instrument. The first mode called **Prod-Mode** includes a limited function set only and is intended for the quality control generally. The second mode called **Lab Mode** allows the access to the complete function set and is intended for the laboratory control or to adjust the **Prod Mode**.

After switching on the actual mode is displayed in temperature display fot two seconds. The following table gives an overview about each function set:

	Prod mode	Lab mode
program choose	no (standard prog)	yes
run a measurement	yes	yes
check the last 20 measurement data	yes	yes
editing user programs	no	yes
set time and date	no	yes
turbidity calibration	no	yes

LAB-mode:

This mode is providing free entry to all functions without any limitation. One can set the temperature profiles, rotation speeds and turbidity levels according to the individual demands. Up to 8 menus can be stored and retrieved so that these can be used for particular samples that need different parameters.

Prod-mode:

This mode is meant for production control applications of the CHEMOTRONIC. In the quality assurance mode, only 1 standard is active and can be started. The upper row of push buttons on the front panel is inactive in this mode.

2.1. Change the operation mode

1) Press Sel button twice for entering into edit menu
2) Press **up/down** button till the actual mode **OP Lab** or **OP Prod** is blinking.
3) Press **enter** button for enabling mode change
4) Press **up/down** button for mode change and acknowledge by **enter** button
5) Now passcode is needed. Enter passcode by setting digit by digit, use **up/down** buttons for digit setting and **enter** button for going to next digit, the first passcode after leaving factory is **444444**
6) After successful setting the buzzer sounds shortly

2.2. Change the passcode

The operation is save only with unknown passcode. That's why the passcode should changes at next supervisor's opportunity. Please note that passcode can be changed only when the instrument is in the Lab mode.

1) Press Sel button twice for entering into edit menu
2) Press **up/down** buttons till the word **code** is shown.
3) Press the **enter** button, **co000000** is shown and the first **0** is blinking now
4) Enter actual passcode by setting digit by digit, use **up/down** buttons for digit setting and **enter** button for going to next digit, the first passcode after leaving factory is **444444**
5) When accepted **cn000000** is shown and the first **0** is blinking now
6) Enter new passcode by setting digit by digit, use **up/down** buttons for digit setting and **enter** button for going to next digit
7) When successfully entered the buzzer sounds shortly and the new passcode is blinking for 2 sec.

3. Testing printing ink resins

Statistically, specifications of raw materials can at best be only as consistent as the test procedures used to determine them. Test procedures should be based on scientifically

approved principles, which means accurate test equipment for controlled test conditions such as heating and cooling temperature profiles, top temperature dwell time, agitation speed, turbidity determination, etc.

Moreover the test oil in which the resin is dissolved must have analytical quality.

3.1. Cloud point or mineral oil tolerance (MOT)

Some product specifications have been based on the MOT-value (mineral oil tolerance) in the past. The MOT-value is calculated from the ratio in weight when a hard resin dissolved in a mixture of mineral distillates is titrated with mineral distillate until the solution becomes cloudy. This method is relatively inaccurate and depends strongly on the temperature of the solution way of stirring, observation of precipitation and the experience of the person performing this test.

3.2. Automatic cloud point test method

The cloud point temperature is a characteristic value resins for printing inks. Deviations in cloud point can lead to sedimentation in the varnish, differences in drying speed, loss of gloss and tack stability.

The cloud point is the temperature at which a resin begins to precipitate in the liquid phase. The automatic cloud point test method introduced here, has been approved by a great number of resin manufacturers world-wide as a standard method for production setting and quality control of resins for printing inks.

3.3. Test oils

Resins used in printing inks today differ considerably in solubility and mineral oil tolerance. Therefore resins are tested in the liquid phase and subsequently the properties of the solution is also depending on the solvency of the test oil. Therefore a test oil must be made by appropriate processing of carefully selected oils in order to achieve a product of consistent analytical quality so that solvency deviations do not effect the measurements.

As test oils of different solvency are necessary to test various types of resins, the standard test method is based on the usage of test oils with different aromatics content. The following test oils are analytical grade test oils with the same properties as the industrial grade oils used for varnishes (manufacturer: ASCOT HALTERMANN):

- Test oil PKWF 6/9 (approx. 20 % aromatics) = high solvency
- Test oil PKWF 6/9 AF (< 1 % aromatics) = low solvency
- Test oil PKWF 6/9 AF new (< 1 % aromatics) = medium solvency

Test procedure:

A quantity of 2.0 grams of resin with 18.0 grams of test oil are precisely weighed into a glass test tube with a magnet stirring rod. This test tube is placed into the automatic cloud point tester CHEMOTRONIC. Under determined heating and stirring conditions the mixture is heated up to 230°C and remains at this temperature level for 2 minutes during which the resin is completely dissolved. After this time, an infrared photocell is activated for automatic turbidity detection at a calibrated level during the falling slope of the sample temperature.

The cloud point is determined at the temperature at which the resin precipitates in the solution. The test result is subsequently printed out by the optional data printer.

4. Features of CHEMOTRONIC III

The **CHEMOTRONIC III** is a fully automatic turbidity measuring unit for the determination of the cloud point of hard resins. The main features of the CHEMOTRONIC III are :

- Reproducible cloud point detection and documentation via optional impact printer or WinTURBI PC software
- Automatic microprocessor controlled test system for production and quality control applications in industrial production processes of printing inks
- Time-saving, fully automatic test method, independent on the analyst or circumstances
- Accurate, reliable and easy to operate precision test system as well for laboratory use as for the production of printing inks
- Cost savings by higher production yields, improved final ink product quality and less labour costs
- Different temperature profiles predefined by factory or user
- Fast calibration procedure to define the turbidity detection range by user
- User definable stirring speed control to reduce the generation of bubbles and to improve the accuracy
- Complete remote control via USB by PC with the software package **WinTurbi V2.0**
- Measurement result overview and comparison with date, time and comments of the last 20 measurements by the software package **WinTurbi V2.0** or printing via optional impact printer
- Easy to use by "switching on and pressing start" for a high measurement efficiency
- Implementation of security algorithm for a fast failure detection and to improve the device safety

5. Installation

The CHEMOTRONIC III is an automatic test instrument for table top use. It includes all required mechanic and electronic components and requires no installation experience.

When the CHEMOTRONIC III is put into operation the following two must be made:

1. *The main cable for power supply. The standard operating voltage is **230 V ± 10 % / 50 – 60 Hz**. In case of **100 – 125 V mains voltage**, an optional transformer is needed to increase the mains voltage to the standard operating voltage of the instrument. Please note, always an **earth connection** is available at the wall socket.*
2. *The Pt 100 temperature sensor with cable, protected by a spiralized metal sheath, and a five pole screw type plug must be connected to the instrument. The Pt 100 input socket is located at the rear side. Please note, there is a slot in the input socket in which the nose of the plug has to fit. Place the plug on the socket in the correct position first, before turning the knurled metal collar of the plug clockwise in order to insert the plug into the socket. **Do not use a lot of force and do not try to connect the plug in an incorrect position**. This may destroy the fine gold-plated pins of the socket, so that the instrument will need to be repaired not under guarantee.*

To ensure a save operation of the device the user has to be sure to have a minimal distance 25 of centimetres to any object at the rear side. When the connections have been made, the equipment can be switched on by pressing the power push button at the right side of front panel. If the self test proceeds without any failure the led of the **start/stop** button is illuminating after two seconds.

6. Calibration

6.1. Temperature calibration

In the Chemotronic a powerful microprocessor system is installed for the control of all instrument functions. A 12 bit A/D-converter is used to convert the analogue inputs of the Pt 100 sensor into a digital signal. The input circuit is designed for automatic zero setting (auto zero) and automatic span setting (auto cal) by internal references. Therefore manually calibration of the temperature measuring functions is eliminated.

The temperature control functions are also automatically optimised for quick temperature response and control (self tune). Therefore , manual settings of control parameters are eliminated is well.

6.2. Speed calibration

The speed of the magnetic stirring rod in the test tube is controlled by a motor with tachometer and electronic drive circuit. Therefore rotation speed settings and indications are absolutely exact. As the motor speed is controlled, the speed indication is also indicating a rotation speed when the magnet stirring rod is in a fixed position in the test tube, for instance due to molten resin in the test tube.

6.3. Time calibration

Due to the high accuracy of the built in real time clock there will be no short term deviations in the timing functions.

6.3.1. Turbidity calibration

The software controlled infrared photo cell system offers two functions to calibrate the system and to determinate the cloud point reference. The user can adjust which turbidity point is recognized as the cloud point by executing a calibration procedure and choosing one of ten turbidity levels to fix the sensitivity within the range. The output signal of the detection circuit decreases linear with increasing turbidity within the relevant turbidity range. The following picture shows the relation:

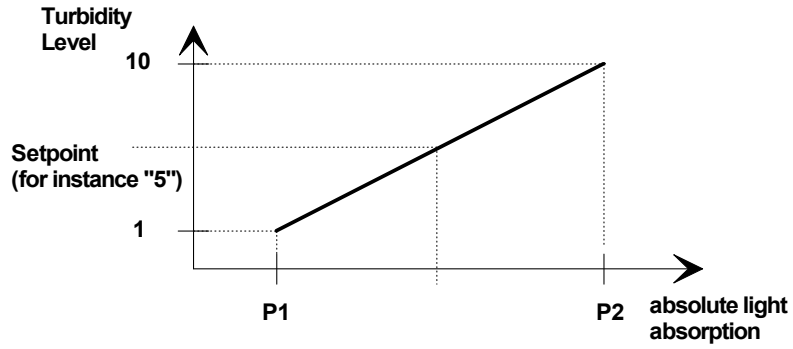


Fig.: 6.1

The absorption points P1 and P2 are determined by two calibration liquids, for instance water or test oil and diluted FORMAZIN. The turbidity within P1 and P2 is linear imaged to the turbidity levels between 1 and 10. During the measurement the user definable setpoint is compared to the sample continuously, until the light absorption exceeds this point turbidity is detected. If no measurement is running it is very simple to check the calibrated defined turbidity range. By inserting one after another of two test tubes with different calibration liquids the turbidity scale should display level "0" and level "10".

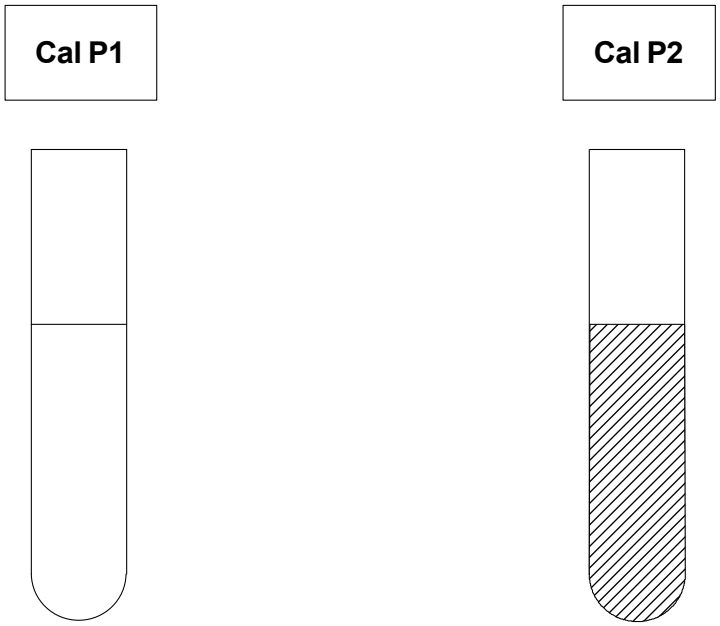
The turbidity measurement in the CHEMOTRONIC is performed by an infrared-photocell system taking both absorption and scattering infrared light passing through the sample under test into account. The measuring circuit is designed for full range measurements both of samples with a low turbidity and of samples with a high turbidity. In order to achieve a high resolution, the setpoint value can be set in 10 different steps from min. turbidity (P1) and max. turbidity (P2). These turbidity levels can accurately be calibrated by the following calibration procedure:

1) Press the **cal** button and wait until the auto cal-functions have been checked and the system is ready to accept the min. turbidity value. The display is showing "Cal P1".
2) Insert a clean test tube filled with water or mineral distillate in the CHEMOTRONIC and press the **enter** button (↵). The display is showing "Cal P2" now.
3) Insert a test tube filled with the prepared FORMAZIN turbidity calibration standard in the CHEMOTRONIC and press the **enter** button again. Both the min. and max. value of the turbidity scale are stored in the memory after this procedure.

Note: 1) The sequence can be P1/P2 or P2/P1. In both cases the correct values are stored for correct turbidity calibration

2) The Chemotronic is factory calibrated for a period of at last 12 month after delivery. See also Turbidity calibration standards.

3) Formazin is a turbidity standard liquid (see 6.5)



6.4. Program specific calibration

This additionally feature allows the user to use an individual turbidity range for the current measurement program. This option can be selected by choosing **Pc** at the end of the parameter list. By depressing of the **cal** button within this menu the calibration menu appears and it is possible to calibrate the both points P1 and P2 now.

6.5. Automatic turbidity calibration

Every program can have its individual turbidity calibration, if one select **Ac** at the end of the parameter list. In this case an automatic calibration is carried out when the photocell is activated (T_1 - 20 °C = T_2). At high temperature, the test solution is still clear, so that the turbidity is at a minimum level. This level is automatically stored as turbidity level P_1 (min. range).

In addition to this, a factor can be set for automatic turbidity detection when the solution becomes hazy. The factor is set as a percentage of clearness of the

solution measured by the automatic calibration procedure. The factor can be varied between 90 % (clear) and 10 % (hazy).

This adjustment is particularly of interest for solutions which show a slight turbidity only or those with a turbidity range. The standard test program is adjusted for automatic calibration, as it eliminates the necessity of turbidity standards. The setting of P_2 = 20 % of P_1 (Ac20).

6.6. External calibration

Ec is a global calibration for all test programs by means of turbidity standards. Both P_1 and P_2 must be calibrated by standard solutions, valid for all test programs.

6.7. Turbidity calibration standards

In turbidity measurements, primary turbidity standards are required as these are the only certified standards. By diluting a primary turbidity standard a secondary standard can be made for actual use. The derived turbidity value is traceable to the original primary standard. It is accepted that dilutions are less stable and cannot be used over a longer period of time (max. 1 week). The definition of turbidity units is based on the polymer suspension FORMAZIN. A FORMAZIN primary turbidity standard is specified for 4000 Nephelometric Turbidity Units (NTU). A wide range of secondary standards can be made by dilution with distillated water (see calibration procedure). A 4000 NTU turbidity standard is stable for long period of time (max. 1 year). Titration of FORMAZIN has proven to be the fastest and most convenient means of an accurately working turbidity standard.

For recalibration of the turbidity, the special polymer suspension FORMAZIN is recommended (available from the manufacturer/supplier). FORMAZIN primary standards are supplied in sealed dark brown 0.1 l bottles and can be used till 1 year after supply date. In order to prepare a secondary standard, one can dissolve a small portion of the primary standard in distillated water by using standard glassware. The following table is providing the ratios to be mixed.

Desired Standard	4000 NTU primary standard in 100 ml glass
1000 NTU	25 ml
800 NTU	20 ml
400 NTU	10 ml
200 NTU	5 ml
100 NTU	2.5 ml

Swirl the glass to mix the solution. Then pour the prepared secondary standard into a CHEMOTRONIC test tube.

Note:

Secondary standards are stable for less than the original stock (max. storage approx. 1 week under cool storage conditions). Therefore it is recommended to use a secondary standard just once.

On special order, we can supply more stable premixed solutions with specific turbidity in small quantities. The recommended value for P2 = 800 NTU in order to guarantee full compatibility with the CHEMOTRONIC I.

7. Function test

Chemical stability of resins, like that of many often chemicals, is highly dependent on storage conditions. Heat and light may break down the structure and the cloud point measurement will show random values. Therefore, a function test should be performed with a material that is chemical stable like stearic acid. This material dissolves in mineral oil quite well and is ideal for a function test of the CHEMOTRONIC.

Test procedure:

a) Place the test tube with stirring magnet and test tube holder on the digital balance.

b) Weigh exactly 10 ± 0.01 grams of the PRIFRAC 2981 stearic acid with 10 ± 0.01 grams of Haltermann oil (PKWF 6/9 or NC-16).

c) Place the Pt 100 temperature sensor with the head on the test tube correctly.

d) Start a test run.

e) Register printed value of the cloud point in the calibration chart of the instrument. The nominal value of the cloud point must be 59 °C.

Accuracy:

In order to check the accuracy of the cloud point measurement, the calibration procedure must be repeated 2 or times. The max. variation allowed in the cloud point measurement is ± 1 °C.

8. Cloud point measurement procedure

The CHEMOTRONIC has been developed for a small quantity of test solution, so that it is imperative to perform the weighting very carefully, otherwise large statistical errors may occur. A digital balance with 0.01 g accuracy is recommended. First the empty glass test tube with the stirring magnet, which should be 50 mm long and 7 mm in diameter, should be weighed. Please use a test tube holder, as the magnet might effect the accuracy of the digital balance.

It is recommended to crack larger resin particles to a size of approx. 2-3 mm diameter, so that these can dissolve in the test oil quicker. If necessary, one might use a sieve. After this, make a 10 % solution of your resin under test and mineral test oil as described per resin type, e. g. Haltermann PKWF 6/9. The standard weight of the test solution is 20 grams, i. e. 2 grams of resin and 18 grams of test oil.

The stirring magnet rod supplied with the instrument is 50 mm long and 8 mm in diameter. Only this type of stirring rod has to be used in order to obtain good results. The rod is placed vertically into the glass test tube and can be gently placed into the tube by putting the tube in a sloping position. Do not drop the stirring rod into the test tube. Place the magnetic stirring rod into the test tube before you fill in the resin, in order to secure that the rod is situated at the bottom of the tube.

As there are glass test tubes of inferior grades and safety on the market, it is recommended to order the standard test tubes recommended by the manufacturer. The standard test tube is a straight tube with 22 mm diameter, 200 mm length and 1.2 mm thickness of the glass wall, made of borosilicate glass. These tubes are available from NOVOMATICS.

The glass of the standard test tube is absorbing wave lengths shorter than 300 nm only so that nearly all transmission in the infrared region can pass through it without attenuation. Nevertheless, there are other glass qualities which even show

absorption at longer wave lengths and so for turbidity measurements a standard glass tube, available from your supplier, must be used.

Summary:

a) Switch on the instrument and watch the temperature display. The display is showing the mode of operation, which can be determined by the supervisor

b) Make sure the temperature sensor is plugged into its socket on the rear.

c) If the instrument is in Prod-mode, the standard menu is the only menu that can be started. If the instrument is in LAB-mode, one can make a choice of 8 user programs (U1 - 8) by the up/down buttons.

d) If necessary, you might check the menu by changing to the Edit-mode by pressing the select-button. After checking depress the **stop**-button for "escape". After correction depress the green-button for "save + quit".

e) Start the test by depressing the green-button. The LED-indicator will change from "ready" to "run".

f) At the cloud point temperature, a test report will be printed automatically when an impact printer is connected to Chemotronic III

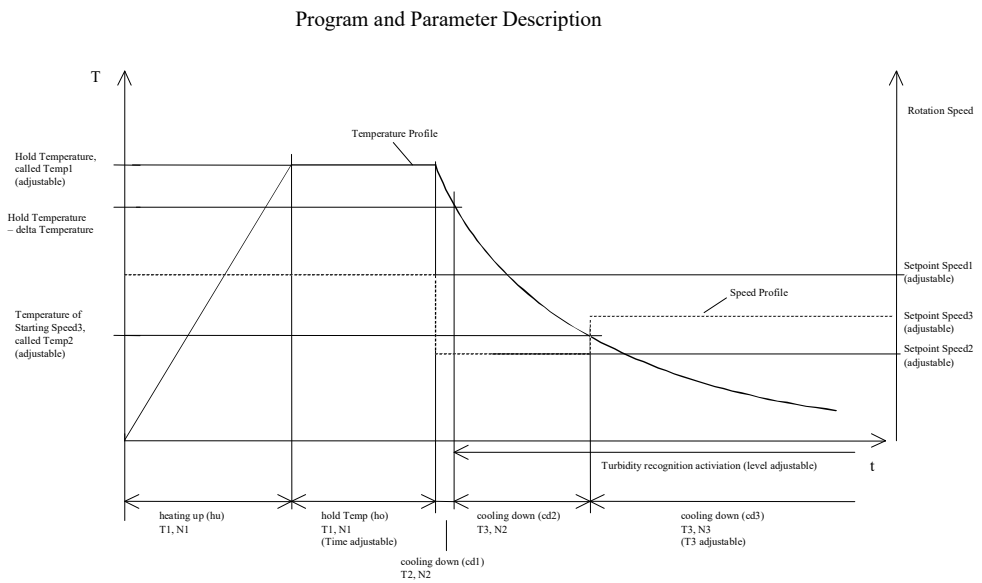
g) After this, a new test tube may be placed into the CHEMOTRONIC and the instrument is ready for the next test.

9. Principle of automatic controlled measurement

For exact cloud point determinations it is very important to dissolve the hard resins under reproducible circumstances. This means that in order to obtain reliable results, it is always necessary to granted reproducible the following criteria:

- the rate of heating up the liquid
- the rate of cooling down the liquid
- the hold time at a high temperature level
- the stirring speed during the process
- the sensitivity of the photocell system

The following figure shows a typical measurement program:



A complete cloud point measurement is divided into five segments. Every segment realises some specific functions according to the requirements of the measurement. The user can vary the following parameters to influence the measurement procedure.

1. Time (the period of the segment **hold temperature**)
2. Setpoint of rotation speed N1 for the segments **heating up** and **hold temperature**
3. Setpoint of rotation speed N2 for the segments **cooling down1** and **cooling down2**
4. Setpoint of rotation speed N3 for the **cooling down3** segment
5. Setpoint of temperature T1 of the segment **hold temperature**
6. Setpoint of temperature T3 for the start of segment **cooling down3**
7. Temperature difference between **hold temperature** and maximal temperature at which the cloud point measurement will be activiated within the cooling phase
7. Setpoint of turbidity detection
8. Selection of preferred turbidity recognition mode**: Ec, Ac, Pc**

1. Segment (heating up and mixing, hu)

The first segment serves to heating up the material under test to the programmed hold temperature T1 (for instance 230°C). The internal PID-controller uses a constant temperature gradient to achieve this temperature. Simultaneously the magnetic stirrer is mixing the substance components within the glass tube by using the programmed stirring speed N1. If process value of temperature is 5 degrees lower than the setpoint temperature segment 1 ends.

2. Segment (homogenisation and hold segment, ho)

Within this segment the homogenisation is being continued by using the programmed stirring speed at the constant high temperature level. The temperature level corresponds to the programmed hold temperature. The segment

will be finished 20 seconds before the specified hold time is turned off. The period of 20 seconds is taking the response time of the sample temperature into account.

3. Segment (cooling segment without turbidity detection, cd1)

After finishing the hold time the cooling segment starts. In general at the begin of this segment a strong cavitation of bubbles caused by a fast stirring at a high temperature of the substances can be observed. During the bubble cavitation the determination of cloud point is not possible because the bubbles produce an alternating absorption effect of the infrared beam. For this reason the stirring speed will be reduced to a lower level (speed N2). This value can be corrected by the user. In addition the turbidity detection is locked. The segment 3 is finished at the temperature T2. The temperature T2 is equal T1 reduced by the adjustable delta temperature.

4. Segment (cooling segment with turbidity detection, cd2)

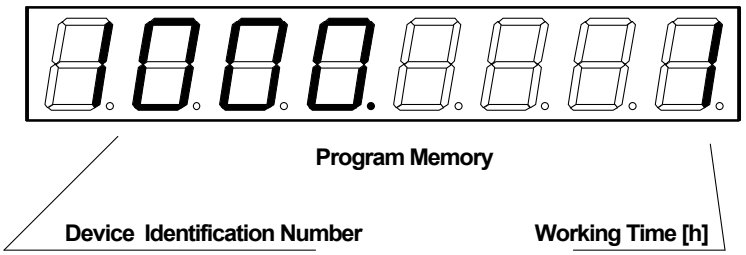
Within the segment 4 the cooling procedure is continued and the turbidity detection will be enabled. By using the turbidity setpoint a turbidity comparison will be carried out permanently. The device uses speed N2.

5. Segment (cooling segment with turbidity detection, cd3)

The speed N3 will be set at the temperature T3. Its is possible to use a higher rotation speed than speed N2 because the bubble cavitation should be finished now. After turbidity detection the measurement will be stopped and the cloud point temperature is printed out at the line printer.

9.1. The menus

After switching on the device identification number consisting of four digits, the previous working time and the current user mode are shown within the program and temperature display for two seconds. The device identification number (DIN) and the working time appear within the program display, the current user mode is shown within the temperature display.



The operating of device will be done within three global menus: the MainMenu, the DataMenu and the EditMenu. The next picture shows the overview and connection between menu and function set:

Menu Overview with the complete function set

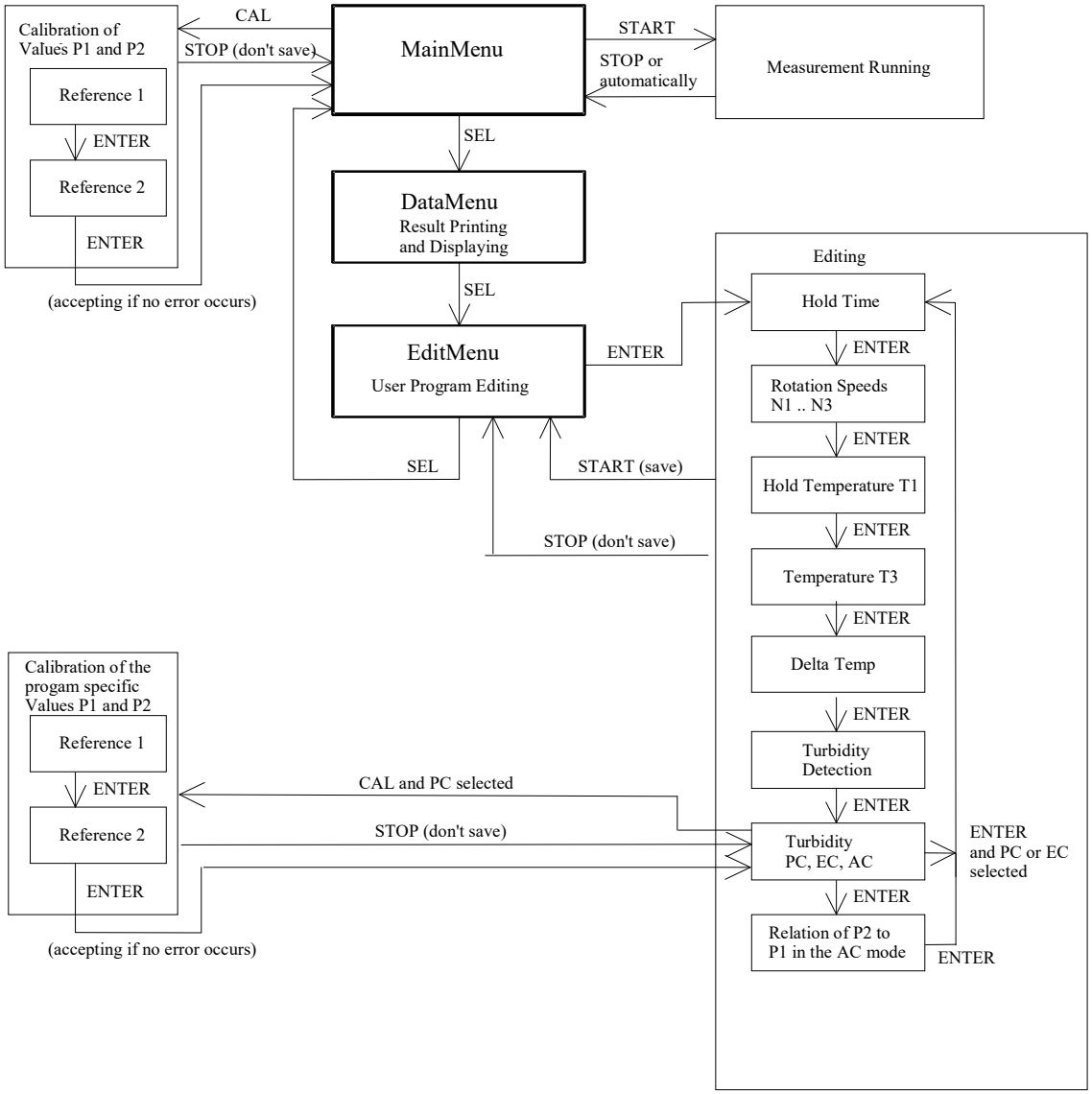


Fig.: 7.1

9.2. The main menu

Two seconds after switching on the device the main menu is available. The menu enables the user to choose and run a measurement program and allows to change to the data or calibration menu.

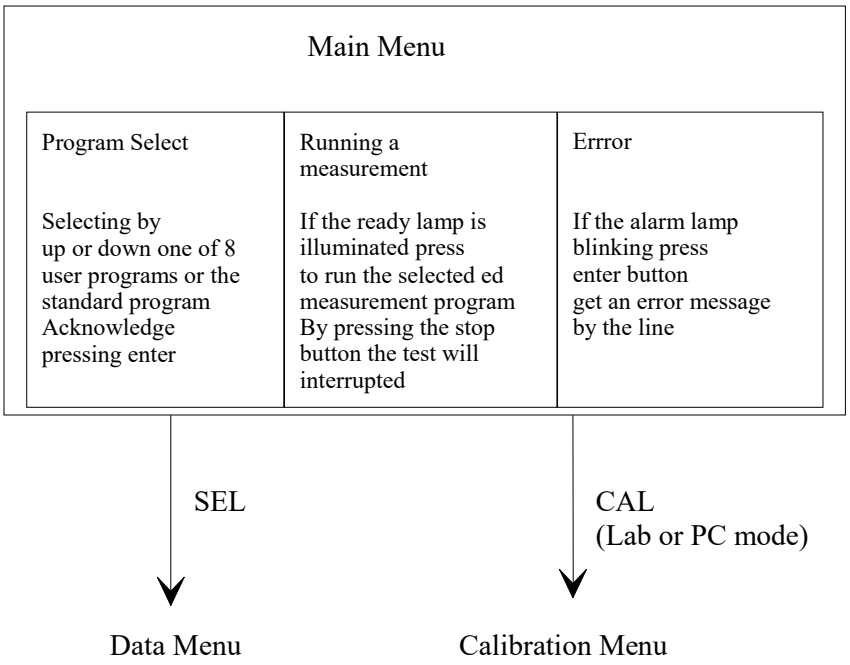
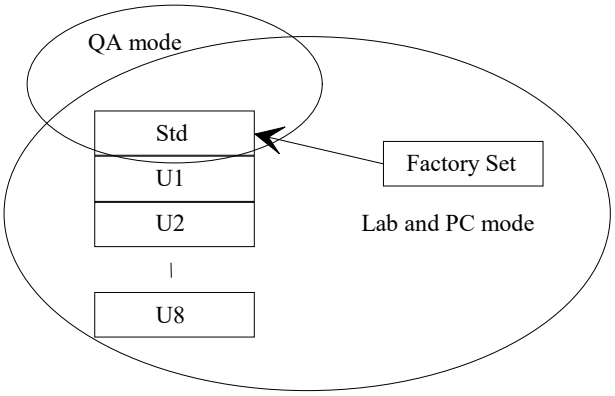


Fig.: 7.2

The adjusted day time and the current measurement program are displayed within basic status of the main display.

Program categories and selecting:

There are 9 selectable programs the standard program and the 8 user programs. The standard program designated Std is usable within all user modes and can be edited in the Lab and PC mode only. In addition it is possible to load and save fixed parameter set within the Std program. The user programs designated from U1..8 are editable and usable within the Lab and PC mode only. The following figure shows the program structure:



To select a program within the Lab mode press the **up** or **down** button. If the **ready** led illuminates the new program has been loaded. In the **Prod** mode the Std program is the only program selected. For the fast check of the several program parameter of the selected program press the **enter** button. The parameter set will be printed out.

Start and stop a measurement program:

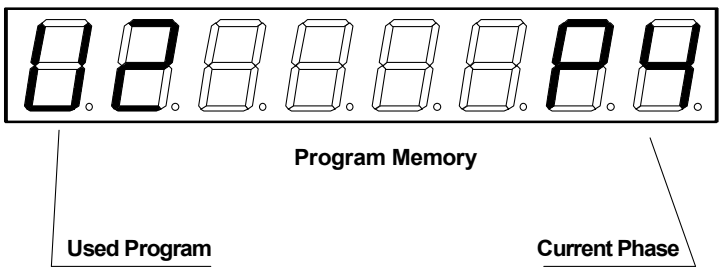
The following preparations must have been made before a run can be started:

The sample tube has been filled with precisely weighted test mixture of exactly 20 grams and has been inserted with stirring magnet, test liquid and temperature sensor into the CHEMOTRONIC III.

In order to start the run, the only manual performance required yet is pushing the **start** button indicated by the green circle. The yellow run LED will illuminate as an indication that the measurement is running and the power is supplied to the heater. The power for the electrical heater is pulse width modulated and controlled by the micro controller by using a PID algorithm. Within the on-time of the heater the right hand digit point of the program display is flashing.

The following information is displayed:

1. The time since the starting in minutes (showed within the time display)
2. The process stirring speed in rotations per minute (showed within the rotation speed display)
3. The process temperature of the sample mixture in degrees of Celsius (showed within the temperature display)
4. The used program
5. The current program segment (P1..P4, for details see chapter "Principle of temperature controlled measurement")



Print an error message:

If one or more of the following errors are detected the current measurement will be stopped and the restart will be disabled. In addition the alarm led is blinking and an error code is shown within the program display. Simply press the **enter** button to get a detailed failure report by the line printer.

The coded errors showed below will be detected:

1. Not plausible temperature gradient	E 1
2. Stirring motor is out of order	E 2
3. At least one cooling motor is out of order	E 4
4. The temperature within the heating channels is to high	E 16, E32
5. The temperature within the cooling box is to high	E 64

9.3. The calibration menu

This menus is available within the Lab or PC mode only. In this menu the two global reference points of the used turbidity range can be determined. Within the main menu press the **cal** button to **enter** into the cal menu, the first reference liquid (water or mineral distillate) is expected. For details, see "turbidity calibration".

9.1. The data menu

To simplify the report and the comparison of different measurement series a data logger was implemented within the CHEMOTRONIC III. The data logger records the relevant parameters of a successful turbidity determination in a special data sheet.

The following parameters will be saved:

1. The cloud point temperature
2. Time and date
3. Used program (Standard or U1..8)
4. A user definable comment

(only available via USB and the WinTurbi V2.0 software tool)

The data logger administrates 20 data sheets. After every successful turbidity determination the measurement parameters are saved automatically and an index counter for the next saving is incremented. If the counter value is equal 20 the next value amounts 1. Attention, in this case the first data sheet will be over written. The index of the last filled data sheet is displayed at left side of program memory display by changing into data menu.

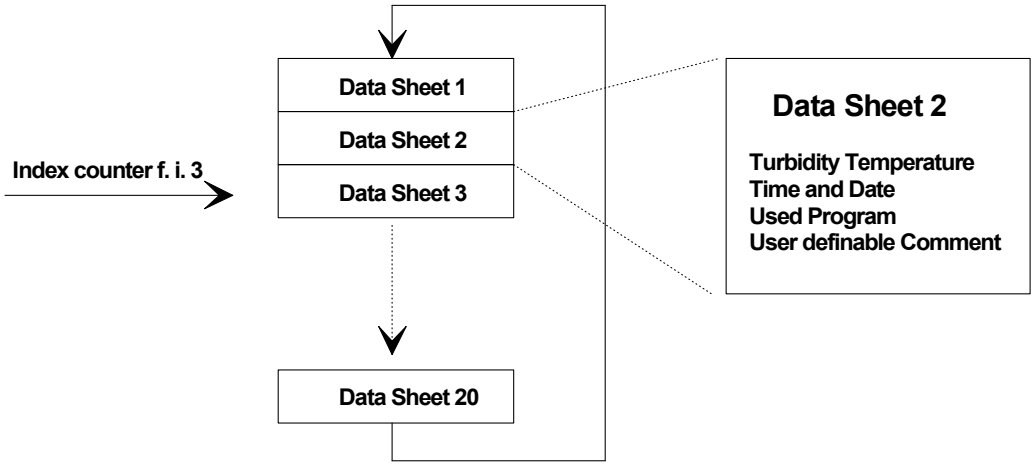


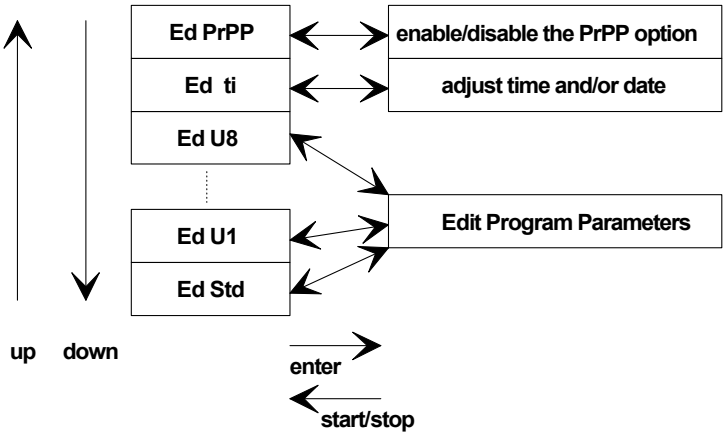
Fig.: 7.3

How can the data logger be used? Simply press the **sel** button within the main menu to change into the data menu. The last turbidity temperature, the number of the last filled data sheet and the time of the turbidity temperature determination will be shown. By pressing the **up** or **down** button select a data sheet and print out the detailed information by using the **enter** button. To print the content of all 20 data sheets press the **start** button only.

9.2. The edit menu

According to the practical requirements the menu is available within the Lab mode only. It serves to manipulate and save the four parameters of the standard program and the user programs U1..U8, to set the time and date and to change the state of the **PrPP** (**Pr**int **P**rogram **P**arameter) option. Press the **sel** button twice starting from the main menu to change in the edit menu. One of following items is displayed by using the blinking mode: **Std**, **U1..8**, **ti** or **PrPP**

Select the index by using the **up/down** button and press the **enter** button to acknowledge the choice.



Edit the program parameter:

The edit menu for the each program or time and date appears. The editing of the nine program parameters is explained the figure below.

Edit Menu Overview

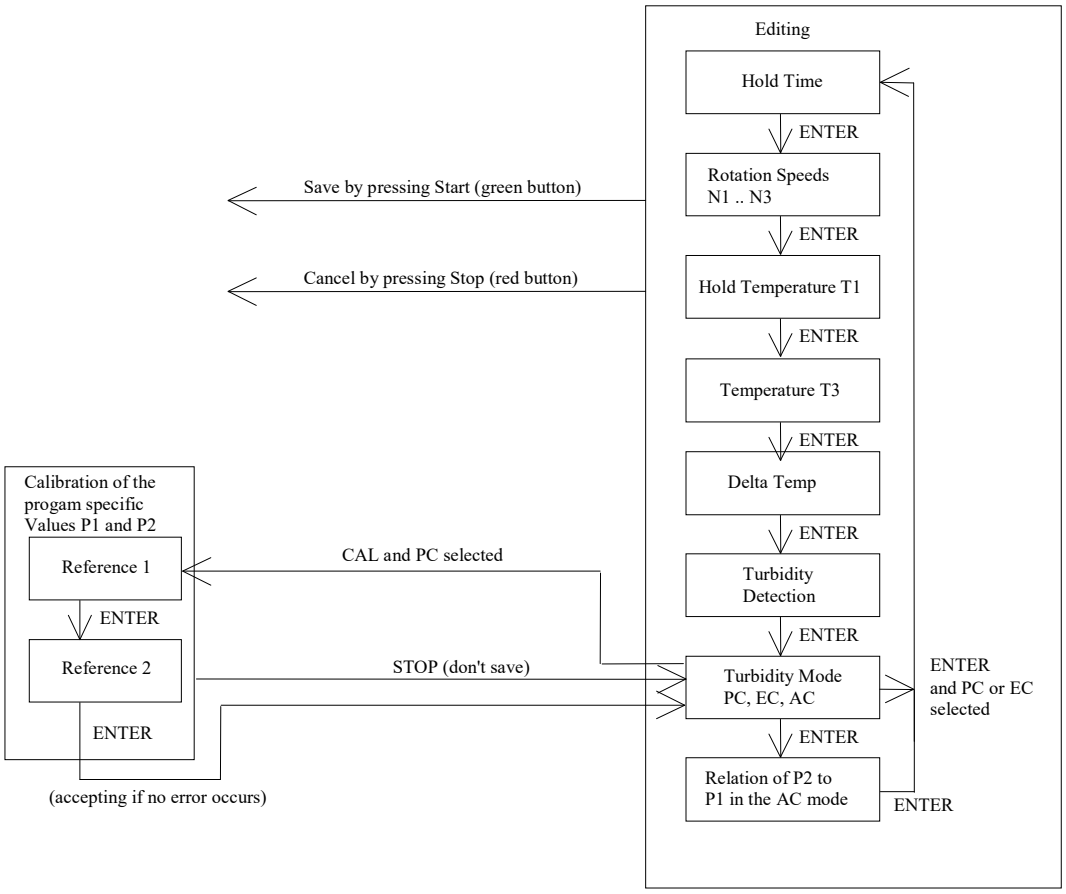


Fig.: 7.4

Press the **enter** or the **sel** button to change from parameter to parameter and use the **up** and **down** buttons to manipulate the value. All changes are cancelled by pressing the **stop** button. To save the new parameter within the permanent memory press the **start** button, a short whistle will be heard to acknowledge the saving. If the turbidity mode **Pc** is selected and the symbol **Pc** is blinking the user can change to the calibration menu of the program specific turbidity range.

Load and resave the factory parameter:

There is a possibility to set the standard program parameter to the factory predefined values. Choose the standard program within the edit menu. Press the **FF** button to load the factory values. Release the dialog by pressing the **start**

button. The new values are saved now. The factory parameters have the following values:

Hold Time	2 min
Rotation Speed N1	1200 rpm
Rotation Speed N2	800 rpm
Rotation Speed N3	1200 rpm
Hold Temperature T1	230 °C
Temperature T3	80 °C
Delta Temperature	-20 K
Turbidity	4
Calibration	Ac

9.2.1. Set time and date

The CHEMOTRONIC microcontroller uses a battery buffered real time clock as time reference. In addition to the editable user programs within the edit menu also the time and date parameter are adjustable. Select the **ti** index and acknowledge by pressing the **enter** button. The time and date parameters are adjustable now. Select the parameter with the **enter** button and correct them by using the **up** and/or **down** button. By pressing the **enter** button the dialog will be saved and closed with the new real time clock adjustment. Use the **stop** button to quit the dialog without saving.

9.3. Use the Prn Option

The setting of **Prn** determines additinional paramter which have to be printed on paper stripe.

Pr_1: prints derivation of turbidity vs. time [%/min] at cloud point

Pr_2: prints derivation of turbidity vs. temp [K/min] at cloud point

Pr_P prints program parameter

Select On/Off for activating/disactivating for the respective parameter. Use enter or select for selecting the respective parameter.

By pressing the **start** button the dialog will be saved and closed with new state. Use the **stop** button to quit the dialog without saving.

10. How to create a test parameter set

The CHEMOTRONIC is providing 8 user defined test programs (U1 - 8)and a standard test program (STD) for quality assurance applications (Prod).

All programs are identical, when the instrument is leaving the factory with one exception: The standard program **Std** uses the auto calibration function. The standard test parameter set corresponds to the recommended settings for accurate cloud point measurements of resin of good or medium solubility in mineral oils, for instance PKWF 6/9 or PKWF 6/9 new (mfr: Haltermann).

The standard test parameter set consists of the following segments:

Segment	Duration	Temp.	Speed
Heat up segment	fixed (approx. 4-5 min)	room temp. up to T_1 (230 °C standard)	N_1 (1100 rpm standard)
Hold segment	variable, normal setting 2.0 min	T_1 (230 °C standard)	N_1 (1100 rpm standard)
Cool segment 1	fixed (approx. 0.5 min)	T_2 (210 °C standard)	N_2 (800 rpm standard)
Cool segment 2	fixed (approx. 4-5 min)	T_3 (100 °C standard)	N_2 (800 rpm standard)
Cool segment 3	fixed (approx.4-5 min)	T_3 to room temp.	N_3 (1200 rpm standard)

One can create a new test program by the following procedure:

1. Depress the "select" button until "Ed(it)" is displayed and choose the number of the program by means of the up/down buttons.
2. Depress the "enter" button in order to get access to the individual parameters of this program number.
3. Enter the desired value for the **hold time**.
4. Enter the desired speed values **$N_1/N_2/N_3$**.
5. Enter the desired top temperature **T_1** and **T_3** for the speed control.
6. Enter the desired level of **turbidity**.
7. Enter the desired program **calibration**

   A_C = automatic calibration + factor

   P_C = program calibration + cal P_1/P_2

   E_C = external calibration (global values)

8. Confirm these parameters by pressing the **green button** (start button).

11. Warnings, Failure detection and hazards

11.1. Warning

A warning message will be printed out if the used calibration of the **Std**-program (EC or PC) doesn't cover the detected change of turbidity. An internal detection function checks this plausibility of the calibrated range in relation to the real change of turbidity. If the deviation is to high a message appears on the end of the result print out. This effect may be caused by unfavourable calibration references or by a pollution of the optical system. Please check both of them before you perform the next measurement to grant reasonable results.

11.2. Failure detection

The CHEMOTRONIC provides with safety precautions in case of system fault occurs. The safety functions performed by the microprocessor control unit recognise the following failures:

detected failure	effect
1. PT100 trouble	Temp. Display: "----"
2. Temperature doesn't increase	The measurement will be interrupted after 90 sec.
3. Stirring Motor Trouble	Prog. Display: "E2"
4. Cooling Motor Trouble	Prog. Display: "E4"
5. Overtemp of Channel1/2	Prog. Display: "E16"/"E32"

If any of the previous error occurs a current measurement will be interrupted and the restart disabled with exception of item 2. The device checks continuously the correct internal temperature state at critical points within a period of some milliseconds. In this way the CHEMOTRONIC reacts by switching off the heaters in a short time if any over temperature will be detected, so that the devices offers a maximum of safety.

11.2.1. PT100 trouble

In generally this error occurs if the PT100 is out of order or unplugged. Please check the correct connection of the sensor. If the failure doesn't disappears it is recommend to use an other PT100 and to switch the device off and on. In the case of failure constancy please contact your distributor.

11.2.2. Temperature doesn't increase

There are two main reasons for this detection. In the first case the PT100 with the glass test tube could be located outside of the instrument. Please check this state first. In the other case the heating system of the instrument could be out of order. If you start a measurement and you hold one hand at the rear side of the device you should feel the warm hot air of the outlet (nearly 60 °C). If the heating doesn't work please contact your distributor.

11.2.3. Stirring Motor trouble

This failure occurs if the difference between setpoint rotation speed and process value is too high for a longer time (some seconds). Restart the measurement to check the stirring motor. If the failure persists or if the failure occurs "too often" please contact your distributor.

11.2.4. Cooling Motor trouble

The micro controller system checks periodically (within some milliseconds) the value of electrical current of the two cooling motors. This check is very important for the correct cooling function of the instrument and to avoid a over temperature hazard situation. If this failure occurs switch off and on the device and restart your measurement. In the case of failure constancy please contact your distributor.

11.2.4.1. Overtemp of Channel1/2

The micro controller system checks periodically (within some milliseconds) the temperature of the two heating channels. This check is very important for the correct cooling function of the instrument and to avoid a over temperature hazard situation. The measurement of the channel temperature is redundant to the check descripted last and serves to improve the instrument safety. The temporary failure occurring is possible causes by the internal heat conduction after a measurement. If a detected over temperature interrupts periodically your measurement please contact your distributor.

11.3. Hazards

A special constructed heating system in addition with a fast failure detection grants a maximum of operation safety.

In order to avoid hazardous situations during testing, the glass test tube is inserted into a metal cylinder. At the event, that the glass tube would break, this cylinder prevents that liquid can flow into the device and the heating system. In addition the hot liquid has almost no contact to the air. In this way a fire hazard is prevented effectively.

The manufacturer doesn't recommend to use a test tube twice, but even in case of a used glass test tube, the fluid might flowing out, if there are cracks in the glass due to mechanical or thermal stresses. In addition the optical system might be affected preventing a new measurement generally.

For cooling fast the hot air with a temperature up to 300 °C a special air mixing box was been implemented also. After an effective mixing with the ambient air the outlet air jet has a temperature lower as 60 °C generally. To enable maximal air circulation between device and environment it is essential to leave a space distance with a minimum of 20 centimetres at backside of device.

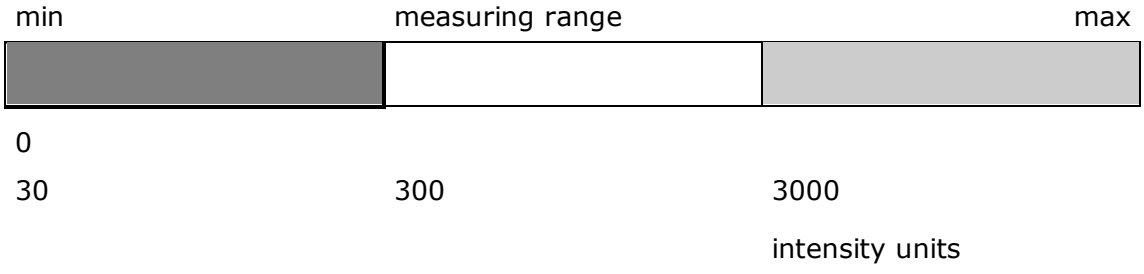
12. Photocell cleaning instructions

12.1. Inspection of photocell operation

If the instrument does not operate according to its specification, one can check the intensity of the infrared photocell system as follows:

a) Insert a clean test tube with clear water or solvent into the instrument.
b) Switch on the instrument.
c) Depress "CAL" to enter the external (global) calibration levels. "CAL-P1" will be shown in the display and EC at the temperature indicator.
d) Depress "FF" in order to check the intensity of the photocell. The value displayed on the speed indicator must be over 300 units in order to comply with the specification of the instrument.

If this value is less than 300 units, the instrument must be can be cleaned according to the next chapter.



12.2. Photocell cleaning procedure

For cleaning of photocell system it is not necessary to dismantle the instrument. By means of the cleaning brush and solvent (Isopropanol Alcohol) supplied with the instrument, direct cleaning is possible in a few minutes.

a) Check the light transmission value according to the previous chapter.

b) Wet the cleaning brush with some solvent, insert it and move it up and downwards to clean of the photosensitive areas in the test tube cylinder.

c) Insert a clean glass test tube filled with clear water or solvent into the instrument and check the intensity of the photocell again. The measured value should be over 300 units. If not, please repeat the cleaning procedure.

d) Re-calibrate the turbidity measurement according to the calibration instructions as described in the chapter 6.2.

Note:

As a precautionary measure, the cleaning may be done on a day by day basis in order to prevent that dust and dirt will stick on the sensitive tip of the optical glass fibres and might burn-in during the use of the instrument.

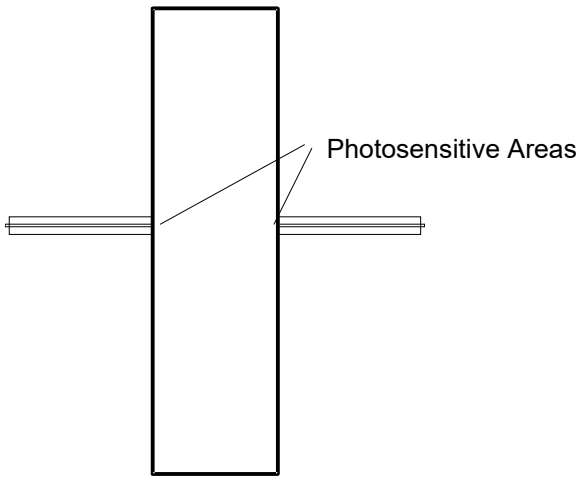


Fig.: 7.5

13. Failure check-list

13.1. Trouble shooting

In case of troubles, the user can check several points himself, before contacting the international service department. We are stating some common troubles, that can be solved easily.

Trouble	Causes and remedies
The equipment cannot be powered up:	Check fuse, cables, connectors
Temperature indicator indicates - - - :	The error message E 1 interrupts a measurement. Input connector at the rear panel or Pt 100 sensor or extension cable is defect. Replace Pt 100 temperature assembly. Make sure the connector is fitting into the socket correctly.

Cloudpoint measurements are incorrect:	Possible causes: • Wrong type of solvent: to increase the cloudpoint select a less aromatic solvent. • Glass fibre tips are dirty: clean the tips with a brush and check the intensity of the infrared light beam. • Cloudpoint is outside the calibrated range: readjust min. and max. turbidity settings. • The set delta Temp is to small. Generally near the dwell temperature the bubble generation is very strong. If the cloud point measurement is activated at this point just an apparent cloud point can be detected caused by the bubbles. The real cloud point may be much more lower. Set delta Temp to higher values f. i. 20 K.
Turbidity calibration fails:	• Check the reference liquids and grant a wide turbidity range. • The pollution of sensor system is too strong, clean up the system. Hint: Observe the variation of the absolute light absorption equivalent at the temperature display during the calibration procedure.
The time and date are lost after switching off:	The capacity of lithium cell is exhausted. Replace it, as described in chapter 10.1.
Printing troubles:	Replace ink ribbon cassette and/or paper roll. Make sure the paper roll can spin by removing some of the paper before inserting the roll. To avoid tension on the paper, always use the form feed button.

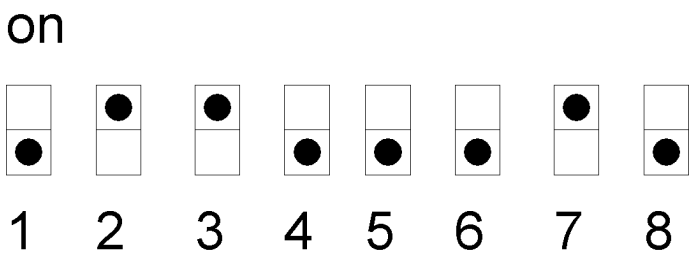
Internal hot spots:	This can lead to a shut down of the instrument and printout of the error message E64. Remove the back panel and clean the air filter at the air inlet.

14. Operation with impact printer

The Chemotronic III is equipped with a printer interface for operation with Citizen impact printer CBM-910. Cloud points, reports, program paramter and error messages are printed out via this printer interface.

For safe operation please use the compatible Chemotronic III printer cable only as listed in appendix under "Spare parts and test materials".

For correct operation with Chemotronic III the printers DIP switches has to be set as follows:



For further details regarding printer operation please follow the **User's manual** of the Citizen impact printer CBM-910.

15. WinTURBI Software

WinTURBI is a modern Windows software for complete remote control for the Chemotronic and other Novomatics turbidity instruments. Data monitoring and evaluation in graphical form makes the handling more comfortable and can give the operator much more information than the instrument operation in stand-alone form.

Main features are:

- complete recording of all measurement parameters and graphical presentation
- unlimited number of programs
- unlimited number of calibration sets
- device independent programming of all free parameter
- comprehensive 2D/3D curve plots including curve overlays
- graphical and numerical export functions to other Windows programs
- free update service for 24 month

Licence conditions:
The WinTURBI measurement and evaluation software may be handled like a freeware licensed software. Unlimited backups, further installations on other PC's or the distribution to other users are permitted. The remote control of the instruments listed within an extra licence register is possible. For Chemotronic users the WinTURBI software is a part of the measurement system.

15.1. Computer Connection of the Chemotronic III

During the starting phase of WinTURBI the program tries to communicate with the instrument via USB automatically. That's why the operator should make sure that the Chemotronic III has been switched on and the serial cable is connected to the instrument and computer before starting WinTURBI. For full function access the operator button on the rear side of the instrument should be turned to LAB-mode.

If the WinTURBI cannot communicate with the instrument, further trials are available by pressing the **Chemotronic/Connect** button into the menu bar. By pressing **Chemotronic/Disconnect** the communication will be finished and the computer port is available for other applications.

Appendix

Standard test method for cloud point measurements

Apparatus:	1)	CHEMOTRONIC automatic cloud point tester
	2)	Digital balance min. range 0..100 grams with 0.01 grams resolution
Preparation:		A 10% solution of the resin under test oil with the high-quality grade mineral test oil must be prepared as follow:
	1)	Place the glass test tube with the magnet stirrer rod into the test tube holder and place the holder on the balance.
	2)	Zero the balance.
	3)	Weigh in 2.0 grams of resin.
	4)	Zero the balance.
	5)	Weigh in 18.0 grams of test oil.

Standard test procedure:

1) Insert the filled test tube into the hole on the top of the CHEMOTRONIC instrument and place the Pt 100 sensor head on top. The display will show **Std** for standard program and the **clock time.**

2) Check the **program parameters** by pressing the **enter button to make a printout**. Please note that automatic calibration with P_2 = 20 % P_1 is used in the standard program.

3) If you wish to **change parameters** (only possible in LAB mode), press sel button and select Ed(it) Std (flashing) to check the test parameters. Step through the program by pressing the enter button instantly and change the parameter(s) by the up/down buttons. Press the start button (green point) for confirmation and to **return to the standard mode.** An audible sound must be heard.

4) Press the **start** button (green point) on the key board in order to start the test.

5) The cloud point is measured automatically and the temperature value is printed on the paper when the solution becomes hazy.

Procedure with user programs

1) Select **Ed(it)** mode by pressing the **sel** button twice.
2) Select U_1 – U_8 by pressing the up button.
3) Confirm the selected user program by pressing the enter button.
4) Set the program parameters by entering the hold time (flashing) first and all the other parameters next by pressing the enter button instantly.
5) Confirm all inputs by pressing the start button (green point). An audible sound must be heard.
6) Start the selected program by pressing the start button again.

Specification

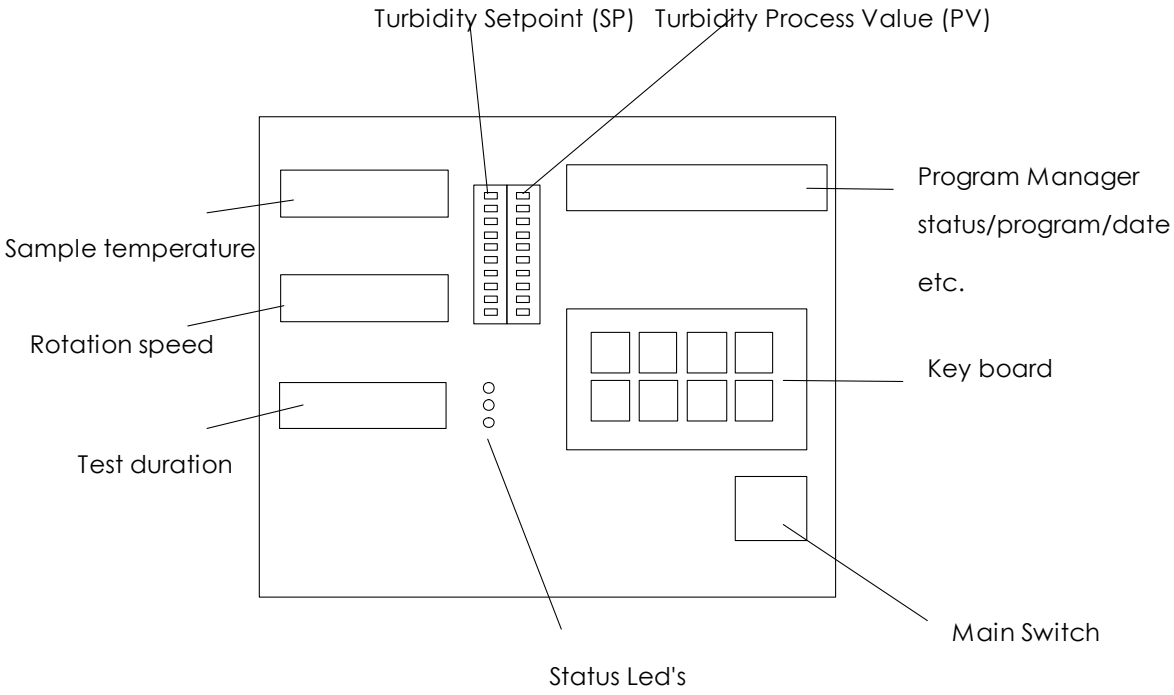
Temperature	
Range:	30 - 230 °C
Accuracy:	±0.1 °C
Stirring speed	
Range:	0 - 1100 rpm
Accuracy:	±2 rpm
Automatic calibration:	Standard tachometer function
Timing	Quarz controlled Real Time Clock
Accuracy:	+15 / -5 ppm
Turbidity	
Range:	41 - 4000 NTU
min./max. settings:	can be calibrated
Guaranteed measuring accuracy	±0.2 °C
Mains Voltage:	110 V till 240 V
Mains Frequency:	50 - 60 Hz
Power Consumption:	1200VA

Spare parts and test materials

Part number	Item
p/n 973602	Citizen printer CPM-910
p/n 97360201	Printer cable for operation with Citizen printer CPM-910
p/n 97360202	ink ribbon cassette for Citizen Printer CPM-910
p/n 973603	paper rolls 58 mm width Citizen printer CPM-910
p/n 973611	Citizen Thermo printer CT-S280
p/n 97360201	Printer cable for operation with Citizen thermo printer CT-S280
p/n 973613	Thermal paper rolls 58 mm width for Citizen thermo printer CT-S280
p/n 973604	Pt100 temperature probe with stainless steel connection head, 4-wire lead and 4-pin connector with gold-plated pins
p/n 973605	magnetic stirrer 8 x 50 mm for CHEMOTRONIC
p/n 973606	high quality glass test tube AR -glass 200 x 22 x 1.2 mm standard package : 90 ea. min. quantity: 360 ea.
p/n 973607	test tube holder ø 23 mm
p/n 973608	magnetic pick-up bar
p/n 973609	100 gr. stearic acid for checking the cloudpoint (59 °C +/- 1 °C)
p/n 973610	100 ml Formazin for turbidity calibration
p/n 973616	Cleaning Brush for the metal test tube holder
p/n 973617	Lithium cell CR 2032 for the real time clock

Part number	Item
p/n 973620	WinTURBI application software for use with CHEMOTRONIC III - user friendly application software for Windows 10 / 11 - easy remote control, monitoring, instrument calibration - multiple graphs, reports and simplified documentation

Front Side CHEMOTRONIC III



Rear Side CHEMOTRONIC III

Important: Keep 25 cm distance to other objects!

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