AUTOMATIC DISTILLATION UNIT

MINIDIS ADXpert OPERATION MANUAL M-V1.32 SW-V3.09

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GENERAL SAFETY SUMMARY

Review the following safety precautions to avoid injury and prevent damage to this product or any products connected to it. To avoid potential hazards, use this product only as specified. Only qualified, service-trained personnel who are aware of the hazards involved should remove the cover

from the instrument.

To avoid Fire or Personal Injury:

• Use Proper Power Cord

Use only the power cord specified for this product and certified for the country of use. Avoid bending or stretching the power cord.

• Ground the Product

This product is grounded through the grounding conductor of the power cord. To avoid electric shock, the grounding conductor must be connected to earth ground.

Do Not Operate Without Covers

Do not operate this product with covers or panels removed.

• Use Proper Fuse Use only the fuse type and rating specified for this product.

• Do Not Operate With Suspected Failures

If you suspect there is damage to this product, have it inspected by qualified service personnel. Do not ignore warnings on the display or warning signals. Please read the manual or call our representative if you are not sure what to do.

- **Provide Proper Ventilation** Do not cover the ventilation slots. Place the instrument so that proper ventilation is guaranteed.
- Place the instruments so that the main power switch is accessible every time

• Do Not Operate in Wet/Damp Conditions

To avoid electric shock, do not operate this product in wet or damp conditions.

- **Do Not Operate in an Explosive Atmosphere** To avoid injury or fire hazard, do not operate this product in an explosive atmosphere.
- Keep Product Surfaces Clean and Dry
- Operate with proper protective Clothing (goggles, gloves, lab coat)

• Be careful with flammable liquids

Flammable liquids should only be used by qualified employees. These substances should be used with constant regard to the danger they pose to life and property. Under normal circumstances they should always be used in a fume-hood away from possible sources of ignition. For further information read the safety data sheet of the substance.



Symbols





1. GENERAL INFORMATION

MINIDIS ADXpert is a true atmospheric distillation analyzer which produces test results according to ASTM D7344. This instrument is suitable to determine the boiling rate characteristics of natural gasoline, light and middle distillates, engine fuels, kerosene, diesel fuels, naphtha and white spirits and many more. Based on its composition, vapor pressure, expected IBP (or EP) or a combination thereof, the sample is categorized in one of five groups $(0\rightarrow 4)$. The arrangement of the MINIDIS ADXpert, i.e. the temperature of the condenser tube and chamber and other operational variables are dependent of the group in which the sample falls, and are set according to the specific design of the MINIDIS ADXpert.

The instrument performs fully automatically with a total sample volume of approx. 20 mL. About 15 mL are used for rinsing to avoid contamination with the previous sample. The distillation curve is determined with a sample volume of approx. 6 mL.

The MINIDIS ADXpert is a stand-alone unit.

Measured data are displayed on an easy to read, illuminated liquid crystal display.

For a hard copy a printer with a serial interface can be connected directly for immediate printout of the measured data. Using the MINIDIS ADXpert in the field, the measured data are stored in the internal memory and printed later.

1.1 MINIWIN Software



Using a PC and the data management software MINIWIN devices from Grabner Instruments can be operated remotely.

Results and data also can be stored, recorded and managed. With MINIWIN instruments can be easily implemented into LIMS.

1.2 MEASURING METHOD

A boiling chip is put into a disposable sample cup (2), the cup is then weighted and placed in the instrument. The cup is pressed against a stainless steel distillation column (3). 6 ml of the sample is automatically introduced into the cup and heated with an electrical heater (1). The temperature of the evaporated vapors is measured with a thermocouple (4). The vapors condense in a condenser chamber (5) whose temperature is controlled by a thermoelectric element. The volume of condensate is monitored using a combination of a stationary optical meniscus detector (6) and a receiver cell (7) whose volume can be changed through driving a piston with a stepper motor. Once the final boiling point is reached, the sample cup can be removed and weighted. The residual is determined from the weight difference before and after the distillation.





1.3 DEFINITIONS & TERMINOLOGY

• Charge Volume

Volume of the liquid sample (6ml) transferred from the Filling System to the sample cup at the temperature specified according to its group.

• Percent Recovered

Volume of condensate observed in the Condenser chamber at any point during the distillation, expressed as a percentage of the charge volume, in connection with simultaneous temperatures.

- Percent Recovery Maximum percent recovered.
- **Percent Total Recovery** Combined Percent Recovery and Percent Residue in the sample cup.
- Corrected Percent Recovery Percent Recovery corrected for barometric pressure (101.3 kPa).



• Corrected Total Recovery

Combined Percent Recovery and Percent Residue in the sample cup corrected to 101.3 kPa barometric pressure.

• **Corrected Temperature Readings** Temperature measuring device readings which are corrected to 101.3 kPa barometric pressure.

• Percent Evaporated

Sum of the percent recovered and the percent loss.

• Evaporated Temperatures

Temperature measuring device readings at specified percentages evaporated calculated with arithmetical procedures.

- Percent Loss 100 minus the Total Recovery.
- Corrected Loss Percent Loss corrected to 101.3 kPa barometric pressure.
- Percent Residue

Volume of residue in the sample cup expressed as a percentage of the charge volume.

• Initial Boiling Point (IBP)

Corrected vapor temperature corresponding to the shift in light intensity from the diode where the initial liquid meniscus was detected to the next diode in the array.

• Vapor Temperature

Temperature of the vapors in the distillation column monitored by a NiCr/Ni thermocouple device.

• Final Boiling Point (FBP), or End Point (EP)

Barometric corrected maximum vapor temperature monitored during the test at the moment of stable volume reading of the recovered condensate on the diode array of the Condenser chamber.

1.4 Patent

The MINIDIS ADXpert is protected by following patents:

- WO 03091667
- US 7 7556 716
- US 7 820 015
- EP 1 499 879
- EP 1 645 869
- AT 412 025

You can search for the patents here:

- Espacenet http://worldwide.espacenet.com
- United States Patent and Trademark Office http://patft.uspto.gov/



2. FEATURES

- True atmospheric distillation
- Results according to ASTM D7344, excellent correlation to ASTM D 86 (ISO 3405, IP123)
- Automatic dry point detection
- Measuring sample volume 6 ml
- Disposable, cheap copper sample cups
- No fragile glass parts
- Automatic and easy-to-use stand alone unit
- Truly portable and rugged for field use (e.g. Mobile Labs)
- Automatic sample introduction
- Peltier temperature control
- Automatic cleaning program
- Built-in balance for precise residual determination
- Built-in pressure sensor for automatic barometric correction
- Fixed position of built-in NiCr/Ni thermocouple to ensure a correct vapour temp. recording
- Automatic dry point detection
- Applicable for gasoline, jet and diesel fuels, solvents and many more
- Short turnaround time due to small sample volume and fast heating
- Large memory: > 120 measurements stored, virtually unlimited data storage via USB flash device
- 10 different pre-installed programs for each group (0 to 4)
- Large memory for custom made programs
- Low energy consumption
- Serial and compact printer support

Automatic Distillation Unit				
Temperature range	ambient to 400 °C (ambient to 752 °F)			
Temperature resolution:	0.1 °C (0.2°F)			
Total required sample volume:	Appr. 20 ml			
Distilled sample volume	6 ml			
Measuring time	15 minutes (light samples)			
Environmental Specifications	Operating Temperature: +5°C to +40°C (41°F to 104°F)			
Communication language	English			
Units of temperature	Celsius or Fahrenheit			
Units of pressure	kPa, psi			
Power requirements:	100/120/230/240 V AC, 50/60 Hz, 200 W			
Optional	DC/AC power converter for vehicle battery			
Fuse	Type T2AL250V, 5 x 20 mm			
Interfaces	2 RS232 interfaces for printer and PC, 2 USB ports			
Physical dimensions:	W x H x D = 253 x 368 x 277 mm (10" x 14.5" x 10.9")			
IP-Code	IP21			
Weight:	Approximately 20 kg (44 lb)			

2.1 TECHNICAL DATA



3. UNPACKING

The instrument is originally shipped with a specially designed shock proof box. Please keep this box. You might need it some day to ship the instrument back to us for service.

Picture	Description	Order Code
	1 pc. Automatic distillation unit MINIDIS ADXpert	ADXPERT
	1 pc. filling tube with luer connector for standard instrument sample inlet	DIS/LUEREIN
	1 pc. outlet tube	VPSG-M890128.29
	1 pc. disposal container with lid	BEH-M500/80/DECK-GEB
	250 pcs. disposable sample cups	DIS-WEGWERFCUPS-CU
	1 pc. Sample cup carrier	DIS/PROBENZANGE
	1 pc. Balance	ADX-PROBENTELLER and DIS-M090924.08









Contraction of the second seco



Options:





1 pc. USB device

USB-STICK

1 pc. Instruction manual

1 pc. Test certificate

PROwatt 300 power converter 12V vehicle battery operation DC/AC-WANDLER 12V/230 DC/AC-WANDLER 12V/115

Mini - keyboard

MINIKEYBOARD-EU MINIKEYBOARD-US



3.1 MINIDIS ADXpert FRONT and SIDE PANEL





Hot surface possible when opening side inlet!

The keys on the front panel have the following functions:

STOP	Stop measurement at any time / Escape a menu or return to previous menu any time
RUN	Start measurement
ENTER	Confirm selected operation / Activate keypad for easy insertion of characters
+ -	Modify characters or values
	Change the cursor position
SHIFT	Execute special functions

Operation of the instrument is very easy due to the graphics display and the programming dialogue. The instrument always displays the possible operations on the display and the operator only has to select the desired function.

When the cursor is positioned at a function and the key is pressed, this operation will be executed. If values or letters have to be altered, shift the cursor to the desired position and alter by pressing the keys.



to return to the previous menu any time.





3.2 MINIDIS ADXpert REAR PANEL



On the rear panel of the instrument you unu a connector for a PC-keyboard (Please use an IBM PC-AT or compatible keyboard). A keyboard with protection cover can be ordered from GRABNER INSTRUMENTS directly. Connect the keyboard.



3.3 INSTALLATION



Only qualified and trained personnel should operate the analyzer!



Verify if the line voltage corresponds to the voltage setting above the power inlet on the rear of the instrument.

In case the voltage setting of the instrument doesn't correspond to mains at your location pull out the voltage selector and turn it to the appropriate voltage.





CAUTION, chemical substances

INLET: Fill only petrochemical substances or related chemicals, e.g. Gasoline or Diesel. **OUTLET:** Chemical waste could spill. Make sure waste container is safely attached!



MINIDIS ADXpert is ready to use.





When the instrument was exposed to low temperatures before powering up the unit leave it for a while for room temperature equilibrium. Condensations inside the instrument would cause a electric short cut when turning on.





3.4 FIELD / MOBILE INSTALLATION

The tester can be used in the field particularly in mobile laboratories mounted on vibration lowered mounting plates. When used in mobile laboratories mains is available. If operated in the field with a car battery a power converter available for the analyzer can be used to power up the instrument.



In case you don't have access to mains voltage, MINIDIS ADXpert can be powered from a 12V DC vehicle battery. The supply cable, is connected to a DC/AC converter which is connected to the cigarette lighter of a car. The instrument can be used normally.

3.4.1 DC/AC power converter PROwatt 300



1. Connect the instrument with the supply cable to the power converter on one of the AC outlets.

2. Connect the power converter via plug to the cigarette lighter slot.

3. Turn on the power converter by switching the On/Off switch.

4. Turn on the instrument. The instrument is ready for operation.





4. SETUP

Switch on the instrument. The display of the instrument is illuminated and shows:



A service counter on the bottom line shows the number of measurements performed since the last service.

4.1 DISPLAY CONTRAST

In order to adjust the Contrast press the 🛽 keys to increase or decrease.

4.2 SETUP

Switch on the instrument. Move the cursor to SETUP and press "ENTER".



4.2.1 LOCATION AND OPERATOR NAME

In order to identify the print-outs from the same location, a name can be programmed. This name is then printed on all hard copies.



Place the cursor next to **Location** and enter your desired identification for printouts either with an external keyboard or using the **external** keys.

To enter the operators name place the cursor next to **Operator** and enter the desired name.







When using the keypad, move the cursor to "bck" and press "ENTER" to delete characters.



4.2.2 MANUAL FILLING AND DENSITY MEASUREMENT

To activate the manual filling procedure, go to "Manual Filling" and use the two keys to switch manual filling to "1" (1 = 0N, 0 = 0FF). If manual filling is activated, the instrument will not do a rinsing and the sample has to be injected with a syringe. The instrument will require only 5.5 to 6 mL sample for a complete run. When in manual filling mode, the **DENSITY** of the sample is also measured during a test.

-		
ADXpert Setup-Menu	29.07.201	6 13:56
Location: Operator:		
Preheat:1 1: ON Man Fill:1 1: ON RS232: 9600;8;n;1;dtr USB Host: Stick re	/ 0: OFF / 0: OFF , 66 lines moved	
Info Clock Cal. Ou	tp. Progr.	End
LOCATION	OPERATOR	
Measurements since last s	service: 16	
Measurements since last s	service: 16	
Heasurements since last s	ervice: 16 DXpert	1

4.2.3 PRINTER SETTINGS

The serial interface (25pin Connector) for connection with a printer can be configured according the connected device. The serial interface (9pin Connector) for connection with a computer can't be configured.

N		
ADXpert Setup-Menu	29.07.2016	3:56
Location: Operator:		
Preheat : 1 1: ON /	/ 0: 0FF	
RS232: 9600:8:n:1:dtr USB Host: Stick rer	, 66 lines	
Info Clock Cal. Ou	tp. Progr.	End
LOCATION	OPERATOR	
Heasurements since last s	ervice: 16	
neasurements since last s	ervice: 16	
	ervice: 16	
SHIFT MINIDIS AD)Xpert	5
SHIFT MINIDIS AL	xpert	5
)Xpert	
)Xpert	
SHIFT MINIDIS AL		

In the **SETUP MENU** place the cursor next to **"RS232"** and enter your desired configuration for communication either with an external keyboard or using the **FIG** keys.

Interface parameters

baud rate



8	number of data bits
n	parity check
1	number of stop bits
dtr	protocol, response function of the connected device.
lines/page	determines the length of one page (only for prn)
USB Host	shows USB settings

Standard Settings for Communication with printer EPSON LX300+: RS232: 9600,8,n,1,dtr, 66 lines Standard Settings for Communication with printer Citizen compact printer: RS232: 4800,8,n,1,dtr, 66 lines



The EPSON printer has to be installed for graphic mode. If you choose to use the citizen compact printer, please turn the ADXpert analyzer off and on after the settings have been changed.

4.2.4 INSTRUMENT INFORMATION

In the **SETUP MENU**, place the cursor on "**INFO**" and press "**ENTER**". The analyzer shows the current instrument configuration and software version.





keys.

4.2.5 DATE AND TIME

- 1. In the SETUP MENU place the cursor on "CLOCK" and press "ENTER".
- 2. Move the cursor to the desired position and select the format with the \square

3. When date and time format have been adjusted, press "ENTER".

4. The cursor jumps to "SET" , press to store the format and to store the format





If you leave the menu without confirmation the changes are not saved. Date and time are maintained by a built-in battery backup.

	Possible format of	f date			
2010 08 31	08 31 2010	31 08 2010			
2010-08-31	08-31-2010	31-08-2010			
2010 /08/31	08/31/2010	31/08/2010			
2010.08.31	08.31. 2010	31.08.2010			
Possible format of time					
02.52p	14.52				
02:52p	14:52				



4.2.6 CALIBRATION OFFSETS

4.2.6.1 TEMPERATURE AND PRESSURE OFFSETS

- 1. In the SETUP MENU place the cursor on "CAL." and press "ENTER"
- 2. To adjust the offset for **Pressure** and/or **Tem<u>perature</u>**, <u>pla</u>ce the cursor to the right of the
- "OFFSET" field and adjust values using the keys. 3. The corrected reading is shown right next to **Pressure** and/or **Temperature**. Т Т ENTER DXpert Setup-Menu 29.07.2016 13 59 ocation Operator 1: ON / 0: OFF 1: ON / 0: OFF 1:dtr, 66 1: Preheat : 1 Man Fill: 1 9600; RS232 Stick USB Host ADXpert Calibr.Offs 29.07.2016 13 Info Clock Cal. Jutp. Progr. End (added to shown (lue) LOCATION OPERATOR Measurements since last service: 99.7 [kPa] Offset 0.0 [kPa] 16 Pressures Ι Т Ľ Tenperature: 26.7 [°C] Offset 0.0 [°C] Disable balance: 🗆 End Offsets MINIDIS ADXpert **L** LOCATION OPERATOR Measurements since last service: 16 4 RUN MINIDIS ADX RUI

4.2.6.2 DISABLE / ENABLE BALANCE

If you like to disable the balance place the cursor on to **"Disable balance**" and press **"ENTER".** The rectangle is highlighted (balance disabled) or blank (balance enabled).



4.2.6.3 D86 OFFSETS

MINIDIS ADXpert allows to easily adapt single distillation points via Offsets.

- 1. In the menu Calibration Offset move the cursor to "OFFSETS" and press "ENTER".
- 2. Move the cursor to the first line to change the **GROUP**, for which the offsets should be reset.
- 3. Move the cursor to the desired position and select the offset with the **Litural** keys.



Press "RESET" to reset all D86 offsets for the selected distillation group.

4.2.7 SET OUTPUT PARAMETERS

In this menu, the units for temperature and barometric pressure can be selected, special distillation points can be defined and the results can be configured.

In the SETUP MENU move the cursor on "OUTP." and press "ENTER".



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4.2.7.1 SET MEASURING UNITS

To change the Temperature Units, move the cursor to D to change the Pressure Units move the cursor to D to change the values by using the **FIG** keys.



4.2.7.2 SET CUSTOMER EVAPORATION POINTS

You can also define up to four distillation points (like E70, E100, ...) for each of the five groups: Adapt the output group and place the cursor next to the **"E"-Values"** (E for "Evaporated") and alter the





4.2.7.3 CONFIGURE RESULT SCREEN

In this menu you can decide how much information you want to be shown in the result screen. The following example shows how to deselect all parameters except for IBP, T10, T50, T90 and FBP:

- 1. Move the cursor to **OUTPUT SELECTION** and press **ENTER**
- 2. Move the cursor over the paramters with cursor and press ENTER to deselect or select specific parameters.
- 3. Only the selected parameters stay highlighted.
- 4. Move the cursor to **SAVE** and press **ENTER** to store the configuration of the results screen.



Selectable Parameters

Basics	Basic information about the measurement, such as program name and group, air
	pressure, recovery, residuals and loss information
ad Inf	Additional information, such as condenser temperature, time to IBO and FBP, %recov.
	at FBP, driveability index
Evap (d	Customer Evaporation Points (e.g. E70, E100, E150, E200)
1% USB	Activate this function to download distillation results for every 1% distilled
	(instead of every 5%) to the USB flash device (connect the USB before or while
	measuring!).



Per default all parameters are selected for the results screen. To deselect parameters solely affects the result shown, but does NOT DELETE the deselected parameters.

1% distillation steps can be downloaded to the USB flash device, but are not shown on the results screen. The USB flash device HAS TO BE CONNECTED during the MEASUREMENT!



4.2.8 DISTILLATION GROUPS AND HEATING PROGRAMS

4.2.8.1 Distillation groups

MINIDIS ADXpert performs a true atmospheric distillation.The distillation conditions are different for different products.5 groups are defined, depending on the sample properties as defined in Table 1.

Table 1: Group Characteristics (ASTM D7344-17)

	Group 0	Group 1	Group 2	Group 3	Group 4	
Sample characteristics						
Distillate type	Pure Comp.	Gasoline	Gasoline	Jet Fuel	Diesel	
Vapor pressure	(ASTM	1 D323, D4953,	, D5190, D5191,D5482, IP69, IP394)			
at 37,8°C, kPa			<65.5			
(at 100°F psi)				(<9.5)		
Distillation properties						
Initial Boiling Point IBP	> 20° C	≤100 °C	>100 °C (212 °F)			
	(68° F)	(212 °F)				
End Point EP	< 400° C	≤ 250 °C	(482 °F)	> 250 (482 °F)	
	(752° F)					

The distillation conditions for the respective groups are summarized in **Table 2**.

Table 2: Conditions during Test Procedure (ASTM D7344-14)

	Group 0	Group 1	Group 2	Group 3	Group 4	
Temperature of filling system ^A		10±0.2 °C	40±0.2 °C (104±0.4 °F)			
Temperature of condenser ^A		10±0.2 °C	(50±0.4 °F)		40±0.2 °C (104±0.4 °F)	
Temperature of receiver system ^A		10±0.2 °C	(50±0.4 °F)	40±0.2 °C (104±0.4 °F)		
Time from first application of heat to Initial boiling point, min	< 12 min	< 7 min	< 8 min	< 12 min		
Uniform rate of condensation at 5 % and 95 % recovered			≥ 0.15	i mL/min		
Uniform rate of condensation from 10 % to 90 % recovered			0.3 to 0	I.9 mL/min		
Time from 95 % recovered to End Point	< 4 min					
Specimen volume of sample (mL)		6±0.05 5.5±0.05				
Mean density of residual, g/ccm	0.72	0.76	0.76	0.86	0.87	

The proper temperature may be higher for samples with high wax content.

The group of a sample has to be selected before a measurement. MINIDIS ADXpert will then automatically set the corresponding temperatures for the filling system, condenser and receiver system and regulate the power so that the measurement will satisfy the conditions defined in Table 2 for the selected group.

4.2.8.2 Heating programs

The heater power is different for different products and depends on the vapor temperature. The dependence of the heater power on vapor temperature is, however, quite similar for different products and is programmed in the MINIDIS ADXpert. Therefore, it is not necessary to define a heater curve for MINIDIS ADXpert.

The controller applies small corrections to the programmed power curve and holds the rate of condensation within a specified range (between 0.3 and 0.8 ml/min).

Only two parameters have to be set for MINIDIS ADXpert: Initial heating power and final heating power. The initial heating power is initially applied to the heater and remains constant until the sample starts to evaporate and recondenses. In MINIDIS ADXpert, this is the parameter A.

The final heating power is the heater power applied to the heater after a certain percentage of the sample has condensed and is not changed until the distillation is finished. The parameter B characterizes this power.

MINIDIS ADXpert has factory settings for A and B for the different groups. If, however, it is found that for some sample these values are not working well enough (for example if a condition defined in Table 2 is not met), the parameters can be changed. For each group, 10 different programs can be defined, i.e. 10 different sets of parameters can be set if necessary.

The value for the parameter A for initial heating power should be decreased if the rate of condensation is very high at the beginning of a distillation (in excess of 0.8 ml/min at T5). On the other hand, if this rate is too low (smaller than 0.2 ml/min at T5). Please do not change the value of A by more than 10 at a time to avoid conditions that fall out of the ranges specified in Table 2 or to create flow rates that are too high to be handled well by the receiver system. Table 3 shows proposed A values, depending on the expected IBP.

If the residual is too much, the final heating power B can be increased (to not more than 230!). If the cup is completely dry and black after a distillation, the value of B may be decreased. Again, it is recommended not to change B by no more than 10 at a time.

Program	Group	A/B values	T COND / DT BRK.	Final Heat at	Other settings
Acetone	0/0	65/110	10/30°C (50/86°F)	96%	Pure Comp.
Toluene	0/1	95/130	10/30°C (50/86°F)	96%	Pure Comp.
Hexadecane	0/2	157/225	40/30°C (104/86°F)	96%	Pure Comp.
Gasoline	1/0	70/200	10/30°C (50/86°F)	96%	
Jet Fuel	3/0	120/200	10/30°C (50/86°F)	96%	
Diesel	4/0	140/225	40/40°C (104/104°F)	96%	

Factory settings for Standard Heating programs:



Hexadecane has to be heated to >18°C to stay liquid.



IBP (expected) °C	IBP (expected) °F	A-value (proposed)
50	122	60
60	140	67
70	158	74
80	176	81
90	194	88
100	212	94
110	230	100
120	248	105
130	266	110
140	284	115
150	302	120
160	320	124
170	338	129
180	356	133
190	374	137
200	392	141
210	410	145
220	428	148
230	446	152
240	464	155
250	482	159
260	500	162
270	518	165
280	536	168
290	554	172
300	572	175
310	590	178
320	608	181
330	626	183
340	644	186
350	662	189
360	680	192
370	698	195
380	716	197
390	734	200
400	752	203

Table 3: Proposed A values, depending on IBP

4.2.8.3 Adapt A-Value during testing

The initial heat (A-Value) can be changed during testing, by (holding the "Shift" key and) pressing the +/keys. A change during the actual distillation run should be done only by very experienced users. With every click on +/- the A-value is either increased or decreased by 3 steps.

Example – JET FUEL (Group 3 distillation):

- Problem: Distillation does not start within 5-6 minutes with the predefined A-Value (120)
- Solution: Increase the initial heat (A-Value) by some notches to an 8-10 higher A-Value (e.g. from 120 to 128-130)



4.2.8.4 Adjust distillation groups and heating programs

In the SETUP MENU move the cursor on "PROGR." and press "ENTER".



Configurable Parameters:

Program	Select program for adjustment of parameters
Program name	Select name for this specific program
A-Value (A/B)	Regulates the initial heater power
B-Value (A/B)	Regulates the final heater power
Final heat at	Characterizes the value after which a certain percentage of the sample has condensed. At this value the maximum heater power (B-Value) is reached.
OFFSETS	Activate calibration offsets for the selected program (if activated, the rectangle is highlighted)
DRYPOINT	Activate optional dry point measurement. If "DRYPOINT" is activated, the results of the ADXpert will show "DP" (Dry Point) instead of "FBP" (Final Boiling Point), which is in line with ASTM D86 requirements. Please note that the Dry Point can be different to the Final Boiling Point.
PURE COMP.	Select, if you measure pure compounds. A special heater regulator will control the distillation to simplify the usage of pure compounds. Do NOT ACTIVATE, when measuring mixtures - like Gasoline or Diesel.
NARROW CUT	Select, if you measure narrow cuts. A special heater regulator will control the actual flow rate during the distillation, the heating regulator behaves slightly different for those substances compared to 'standard samples'.
T COND	Receiver temperature, set according to distillation group.
DT BRK	Temperature drop of distillation vapor temperature that is the criteria to finish the distillation.
STOP AFTER: %	If the distillation should only be performed up to a certain percentage of recovered sample, it is possible to enter the break volume (i.e. 80%).
CONST. LOSS:%	Constant loss of sample, can be entered when measuring the same sample with the same loss all the time.
CONST. RESID:-%	Constant residual of a sample, can be entered when measuring the same sample with the same residual all the time.
OFFSETS	Access the D86 offsets menu for results finetuning.
END	Leave the menu and save results.





4.2.8.5 Checking Calibration, heating programs for Toluene, Hexadecane and Acetone

Three programs are configured for performing distillations with pure Toluene, pure Hexadecane or pure Acetone.

Toluene is used to check the calibration of the vapor temperature sensor. The distillation of toluene is performed according to group 1 conditions with program number 0. The temperature **T50** has to be **110.6 ±0.8°C (231.1 ±1.4°F)** if the calibration of the temperature sensor is correct.

Hexadecane is used to check the calibration of the vapor temperature sensor in the high boiling range. The distillation of Hexadecane is performed according to group 1 conditions with program number 0. The temperature **T50** has to be **287 ±1.5°C (548.6 ±2.7°F)** if the calibration of the temperature sensor is correct.

Acetone is used to verify instrument performance. The temperature T50 has to be 56.5 ±0.7°C (133.7 ±1.3°F).

4.2.8.6 Sample check programs

Sample check programs can be created to determine if the instrument settings are correct for a standard compliant distillation run. These programs ensure that critical parameters like **initial heat, time to IBP and flow rate** are accurately met in the beginning of the distillation run. A check program can also be used for **in-depth rinsing and to minimize cross contamination** between samples.

Sample check programs are not installed in the unit, but can be created easily for every measurement group. For example, a **<Check Gasoline>** program should use the same parameters as the gasoline program, with the only difference that the **distillation should stop after 20%**.

It is recommended to use such programs when

- switching between different sample types (gasoline, jet fuel or diesel)
- or after an Acetone cleaning run has been performed
- and if the user wants to **check** the **distillation settings** on a new sample



5. MEASURING MENU

5.1 SAMPLE NAME

- 1. In the MAIN MENU place the cursor on "MEASURE" and press "ENTER".
- 2. Place the cursor on "SAMPLE ID" and press "ENTER" again. The Keypad pops up.
- 3. Insert the sample id, move the cursor to **"END"** and press **"ENTER"** to return to the measurement menu.



5.2 MEASURING ROUTINE

MINIDIS ADXpert incorporates a lot of preconfigured programs, among those methods for **B100 Biodiesel** and for measuring **UNKNOWN SAMPLES**, without selecting any distillation group.



For detailed information on distillation programs see 4.2.8 DISTILLATION GROUPS AND HEATING PROGRAMS or refer to ASTM D7344 or ASTM D86 standard.



If the instrument has not been in use for a longer period, we recommend to do an "EMPTYING CYCLE" and run the "ACETONE CLEANING" prior to the measurement to make sure no residuals stay in the system. See 8. Maintenance for further information.



1. To start a measurement move the cursor onto the program you want to test and press "ENTER" to select the distillation program. The name of the distillation program and the settings are changed automatically.



2. Press **"RUN"** to start the measurement: The balance is equalized.



3. Put the sample cup with only **one boiling chip** on the balance and confirm with **"RUN":** MINIDIS ADXpert is determining the weight of the cup including the boiling chip.



4. After the cup weight determination the cup can be inserted into the heater.



Press **"RUN"** to start the measurement.

The instruments detects automatically, if the sample cup lid is closed and if the sample cup is inserted. The sample cup is pressed tightly against the distillation column.

5. For rinsing and sample introduction put the tube into the sample and press "RUN"



- 6. When all steps are finished, MINIDIS ADXpert starts the distillation. First MINIDIS ADXpert initializes the optical detection system and after that the unit powers on the heater.
- 7. The rotating, small bar indicates that the measurement is in progress



Press "STOP" to stop the measurement. Please wait, until emptying of the sample has been finished, before you open the lid.



Do not open the lid during the measurement! Some parts inside become VERY HOT and severe burns will occur if one of these parts is touched!



TVap	current vapor temperature	
TCnd	current condenser System temperature	
TFil	current filler system temperature	
Heater	current heater power in DAC unit	
	0 means no power, 230 max. power	
Flow	current rate of condensation in ml/min.	
05:00	The displayed stopwatch in the bottom right corner shows the	
	elapsed time since the heater was initially switched on.	

After the instrument has detected the final boiling point (or any other termination criteria is reached, like dry point or a certain percentage recovered) the heater is switched off and the sample will be removed from the instrument.

The display shows emptying of the system. During emptying the receiver and filler system the sample cup is cooled down. When a certain temperature limit is reached the heater moves down. If the balance is disabled the user now has the possibility to enter a cup weight manually.

If the balance is activated remove the sample cup with the delivered tongues as shown in the picture below. On the bottom side of the tongues, there is a ring with a diameter larger than the rest machined to the tool. With this ring, the rim of the sample cup can be grabbed so that it can be pulled out of the heater.

When the display shows place the sample cup on the balance and press "RUN".





If the measurement was terminated before the final boiling point was reached the sample cup will contain sample, it may even be almost completely full! In such cases use utmost care when you remove the cup in order not to spill any sample over the heater and inside the instrument.



Caution: Hot surface!



5.3. MANUAL FILLING AND DENSITY MEASUREMENT

Please make sure that in the Settings Menu "Manual Filling" is set to 1.

- 1. Press **"RUN"** to start the measurement: The balance is equalized.
- 2. Put the sample cup on the balance: The empty cup is weighted.
- 3. Fill the amount of sample requested by the instrument into a syringe, then disconnect the syringe tip.



- 4. Fill the sample into the sample cup.
- 5. Weight the cup again. Now the **DENSITY** of the sample is calculated.
- 6. Put the sample cup into the oven and press RUN.
- 7. Make sure that the tubing is still connected to the instrument and is immersed in the sample. This is required for the adjustment of the condenser.



5.4 RESULTS MENU AND PRINTOUT

The results list will be shown directly after the measurement.

- 1. Place the cursor on **"RESULT"**
- 2. The sample ID is displayed together with date and time when the measurement had been performed.
- 3. Browse through the list line by line with the and and keys. For fast scrolling press

"SHIFT" and **Second** or **Second** simultaneously.

4. Press **"ENTER"** to access the measurement details



To select actions from the botton line use the and keys. Selectable parameters on the Results screen:

Result	display the selected Result
Print	print the desired result(s)*
Del	delete the desired result(s) *
USB	download the desired result(s) to an attached USB flash device*
Mark	mark the desired result(s)
End	exit the menu

* It is possible to print/delete more than one result at once, you have to mark them first.

5.4.1 ACCESSING RESULTS LIST

There are two ways to access the MINIDIS ADXpert results list:

1. On the **MAIN MENU** move the cursor onto **"RESULT"** and press **"ENTER"**. The results list will show.



2. To access the results list directly from the measurement screen move the cursor onto **"RESULT**" and press **"ENTER"**.



To return to the measurement menu, press "STOP or END" in the results screen.



5.4.2 PRINTOUT, SAVE AND DELETE RESULTS

5.4.2.1 SINGLE RESULTS

To print, save or delete single results

- 1. Place the cursor on **"PRINT" or "USB" or "DEL"** respectively
- 2. Browse through the list line by line with the \uparrow and \downarrow keys.
- 3. Press "ENTER" for the selected result to "PRINT", "SAVE" or "DELETE" the selected result





Attach the USB flash device prior to selecting "SAVE". A double "BEEP" informs about successful data download.

5.4.2.2 MULTIPLE RESULTS

It is possible to print/delete more than one result at once. Therefore you have to mark them first.

- 1. To mark several results, move the cursor to **"MARK"**, browse through line by line with the ↑ and ↓ keys and select the desired results and press **"RUN"**.
- 2. A small asterisk appears to the left of the desired result.
- 3. To mark all results at once move the cursor to **"MARK**" and press **"SHIFT**" and **"RUN**" simultaneously.
- 4. Then select "PRINT", "USB" or "DELETE" and press "ENTER".



6. MAINTENANCE MENU

MINIDIS ADXpert is equipped with a maintenance menu where also a status report can be created. In the **MAIN MENU** place the cursor on **"MAINTENANCE"** and press **"ENTER"**.



6.1 AUTOMATIC CLEANING



CAUTION!

Do not use cleaning agents that could cause a hazard as a result of reaction with the used probe material; the manufacturer or his agent is to be consulted if there is any doubt about the compatibility of cleaning agents!

It is recommended to clean the instrument with a mixture of Acetone, Methanol and Toluene after 12 measurements under group 4 conditions or a total of 20 measurements, whatever condition is reached first. Especially group 4 samples tend to produce buildups that need to be removed regularly. A **mixture of 1/3 Acetone, 1/3 Methanol and 1/3 Toluene is an excellent solvent** for these deposits. Automatic cleaning is a fixed program that can't be changed. Please **prepare the Acetone-Methanol-Toluene mixture** and store within easy reach of the MINIDIS ADXpert.

Automatic cleaning is different to a regular distillation, a different filling cycle takes place, with a different type of sample introduction, to guarantee a proper rinsing and elution step of the vapor tube ranging from the separation column to the receiving system. For this reason, in the cleaning run the amount of cleaning sample drawn into the sample cup is not accurately detectable, and the cleaning run does NOT give you information about the various distillation points. Also, the percentage of recovered volume, at which the final heat adjustment would take place, is handled differently. To ensure, that all solids washed out of the vapor tube are collected in the sample cup, the distillation stops at the percentage of the final heat adjustment.

MINIDIS ADXpert automatically counts the number of measurements and also the number of group 4 measurements. If a measurement is started after a total of 20 distillations or 12 group 4 distillations the instrument requires to remove deposits by an Automatic cleaning measurement.

If cleaning is required prior to measurement, the following message will pop up:

Run Automatic cleaning program ! Press RUN or STOP to continue

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Attach a bottle of the prepared Acetone-Methanol-Toluene mixture and press **"RUN"** to start the cleaning program. The Automatic cleaning counter will automatically reset.

You can start the cleaning program any time between measurements:

- 1. On the MAIN MENU move the cursor to "MAINTENANCE" and press "ENTER"
- 2. Put a boiling stone into an empty sample cup and put the sample cup into the heater
- 3. Close the lid
- 4. Attach the cleaning solution (bottle with 1/3 Acetone, 1/3 Methanol, 1/3 Toluene)
- 5. Move the cursor to **"AUTOMATIC CLEANING"** and press **"ENTER"** to start the cleaning programme.
- 6. Wait appr.15 min. until the cleaning procedure is finished (DO NOT press STOP to cancel the cleaning procedure)
- 7. Before testing a new sample run a check program on a new sample, to avoid influence from remaining cleaning agent.





DO NOT press STOP during an Automatic cleaning! Cleaning fluid might spill into the instrument, if STOP is pressed during the cleaning procedure!

If the sample cup is erroneously filled with regular sample (gasoline, diesel, ...) instead of cleaning fluid, please adjust the "A-" heating value to match the expected IBP of the sample. For A-values see Table 3 in chapter 4.2.8.2 Heating programs.

6.2.1 SKIP AUTOMATIC CLEANING

Automatic cleaning requirement can be skipped, if another test run is required before cleaning, e.g. if a repeat run has to be finished before Automatic Cleaning is started.

To suppress Automatic Cleaning for another run, press **"SHIFT"** and **"RUN"** simultaneously. The cleaning run will be skipped until the next measurement.



6.2 SELF TEST

There is a self test program installed to check several mechanical and electric parts. In the **MAINTENANCE MENU** move the cursor to **"SELF TEST"** and press **"ENTER"**.

ADXpert Machin Service Automatic cle	e Care USB 2 & Mainteners eaning S	9.07.2016 14 37 Here elf test	
Empty	Stat PRN	Stat USB	ENTER
Lubricate	Rinse	End	
LOCATION Measurements s	OPERF ince last servic	NTOR be: 16	
		ert 🔐	

The instrument starts the Self test and the display shows the progress of the self tests with some informational messages:





If all tests have been performed the display shows the result:

Stepper:	OK	Filler:	OK
Lift:	OK	Peltiers:	OK
T-Sensors:	OK	p-Sensors:	OK
Heater:	OK	Balance:	(Skip)
			End

6.3 EMPTYING CYCLE

If for any reason some liquid remains either in the recovering or the filling system it is possible to manually start a complete emptying cycle.

In the MAINTENANCE MENU move the cursor to "EMPTY" and press "ENTER".



6.4 STATUS REPORT

For service/diagnosis purposes MINIDIS ADXpert can print or save a status report to USB. At this report the values of important parameters are printed together with the current values of the heating power parameters A and B for all 5 groups and all programs.

In the **MAINTENANCE MENU** move the cursor to either **"STAT PRN"** (for printing) or **"STAT USB"** (for saving to an attached USB device) and press **"ENTER"**.



6.5 LUBRICATION OF THE FILLING SYSTEM O-RING

After 100 measurements it is recommended to do the Lubrication of the Filling System O-Ring. A message will pop up and inform about lubrication. In the **MAINTENANCE MENU** move the cursor to **"LUBRICATE"** and press **"ENTER"**. The instrument will ask you to remove the FRONT of the MINIDIS ADXpert:



To remove the FRONT:

- 1. Remove the balance
- 2. Up on the back is a handle (marked red on the second picture below)



3. Pull the back handle with one hand and lift the top handle with the other hand to remove the top handle



4. Loosen the four front screws on the left and on the right side of the instrument with the screw driver delivered and flip the instrument front to the side







- 5. Press **"RUN"** again, the piston will move to lubrication position
- 6. The instrument will then ask you to put some drops of oil on the piston



7. Fill approximately 0.5 ml Piston Oil with the delivered plastic syringe



8. Inject the Oil under the black mark between the O-Rings slowly







 After injecting the oil, press "RUN" again. The piston moves up and down a few times automatically to lubricate the O-Ring.



10. Once the lubrication is done, the lubrication warning will no longer appear on the screen.



It is highly recommended to do lubrication immediately after the lubrication warning appears. A lubrication counter request to lubricate the system every 100 measurements.

- 11. Once lubrication is finished, put the top cover back in its position and tighten the screws in the manner you have loosened them
- 12. Put the receiver sealing back (pull the backside handle prior to insertion of the sealing)
- 13. Press the top handle down (pull again at the backside handle)
- 14. Push the backside handle until the top handle rests with a "CLICK".
- 15. Push down the receiver button until the button rests with a "CLICK".















6.6 RINSING THE FILLING AND RECEIVER SYSTEM

It is recommended to perform a rinsing after group 4 distillation with a sample that may have solid residuals, like samples with a high wax content.

Some materials have a freezing point higher than 10°C which is the temperature of the filling system and condenser for groups 0 to 3.



For detailed information see chapter 4.2.8 DISTILLATION GROUPS AND HEATING PROGRAMS

If residual solidifies when the system is cooled to 10°C, tubing and valves may become blocked. It is therefore recommended to perform a rinsing if the distillation group is changed from group 4 to another group. Please rinse with the group 0 to 3 sample that you want to measure right after a group 4 distillation.

Usually, it is sufficient to perform acetone-cleaning to keep the system clean and free of deposits.



For detailed information see chapter 6.1 AUTOMATIC CLEANING

Start a rinsing:

- 1. In the MAINTENANCE MENU move the cursor to "RINSE" and press "ENTER"
- 2. The system performs an emptying cycle
- 3. Put the sample tube into the sample
- 4. Press "RUN" to start the rinsing of the filling and receiver system





7. ROUTINE MAINTENANCE



CAUTION!

Do not use cleaning agents that could cause a hazard as a result of reaction with the used probe material; the manufacturer or his agent is to be consulted if there is any doubt about the compatibility of cleaning agents!

7.1 CLEANING THE DISTILLATION COLUMN

It is highly recommended to clean the conical ring on the bottom of the distillation column after EVERY measurement to avoid contamination.

To do so take a dry tissue and swap the bottom of the distillation column.

7.2 CLEANING / REPLACING THE INLET FILTER

To avoid that the tubing's and the valves of MINIDIS ADXpert are clogged the sample is filtered at Luer Inlet. Filtering is achieved with a metal filter disc with a porosity of 200 µm. This Filter disc can be either cleaned or replaced.



Therefore turn the Luer Inlet nut anticlockwise and remove it.



7.3 CLEANING THE OPTICAL DETECTION SYSTEM

To remove contaminating residual from the optical detection system it is recommended to clean it manually using a cotton swap soaked with Acetone or any other polar cleaning agent.

At the top of the instrument there's a black seal, to give access to the inner parts of the detection system.

- 1. Pull the back handle with one hand and lift the top handle with the other hand to remove the top handle. The black sealing will automatically jump out.
- 2. Remove the black sealing
- 3. Take a cotton swap, soak it with cleaning agent
- 4. Clean the optical detection system with the cleaning swap
- 5.-8. Put back the seal and attach the handle









4

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8. ERRORMESSAGES AND TROUBLE SHOOTING

8.1 Sample cup detection error

If the Sample cup detection fails the display shows:



Follow the displayed Instruction and replace the Sample Cup, in case it is deformed. If the same Error occurs several times in series contact your local supplier for help. A leak test is recommended.

8.2 TROUBLESHOOTING

Problem	Cause	Solution
Liquid in the front panel	flooding of the receiver	Lower the A value of the heater;
		Perform an Acetone cleaning
		measurement
Distillation does not start up or	too little power of the heater;	Increase the A value of the
stops shortly after IBP	heater defective	heater
TCup temperature decreases	heater defective;	check/replace heater;
slowly	power supply board defective	Replace power supply board
Recovery rate is too low	wrong distillation group;	check distillation group;
	clogged inlet filter	clean/replace Inlet filter



CUSTOMER SUPPORT AND INFORMATION





GRABNER INSTRUMENTS Messtechnik GmbH A-1220 Vienna / Austria Dr. Otto Neurath Gasse 1

 Tel:
 +43 / 1 / 282 16 27-0

 Fax:
 +43 / 1 / 282 16 27 300

 E- Mail:
 grabner.office@ametek.com

 Internet:
 www.grabner-instruments.com

For technical support write to



grabner.support@ametek.com

or call us



+43/1/282 16 27-200

Representatives and Distributors:



www.grabner-instruments.com/contact/distributors/index.aspx

