Koehler
INSTRUMENT COMPANY, INC.

K905XX
Automatic Potentiometic Titrator

Operation and Instruction Manual

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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing

CERTIFICATE OF CONFORMANCE

Automatic Potentiometric Titrator
K905XX

This certificate verifies that part number K905XX, Automatic Potentiometric Titrator, was manufactured in conformance with the applicable standards set forth in this certification.

Specifications:

ASTM D664
ASTM D2896
ASTM D3227
ASTM D4739

This unit is tested before it leaves the factory, to ensure total functionality and compliance to the above specifications and ASTM standards. Test and inspection records are on file for verification.

Jesse Kelly
Application Engineer
Koehler Instrument Company

CONTENTS

THE CONTENTS OF THIS MANUAL ARE SUBJECT TO CHANGE WITHOUT NOTICE.

SECTION 1

- **Introduction**
- **Specifications**
- **Features**

K905XX- AUTOMATIC POTENTIOMETRIC TITRATOR

INTRODUCTION

An Automatic Titrator is a combination of electrochemical potential measurement through electrode with microprocessor driven burette, thus increasing the accuracy of measurement. The instrument is capable of performing almost all basic titrametric functions and cater to all types such as 1) Aqueous 2) Non-aqueous 3) Redox 4) Precipitation / Argentometric 5) Complexometric / EDTA 6) Voltametric 7) Back titration with simplicity. User can define particular method for a specific titration.

SPECIFICATIONS :

Control	**:**	Micro-controller based.
Burette	**:**	1ml, 5 ml or 10 ml capacity, interchangeable. 25 ml Optional.
Burette resolution	**:**	1/10000 for 10 ml , 1/5000 for 5 ml and 1/1000 for 1 ml.
Filling time	**:**	< 20 seconds.
Principle	**:**	Volume determination by equivalence point, end point or pH STAT.
Modes of operation	**:**	a)Incremental b)Equilibrium c)Cut-off by pH d)pH STAT titration.
Methods	**:**	a)Acid-base b)Non-aqueous c)Redox d)Precipitation e)Complexometric f)Back g)Photometric h) Thermometric.
mV range	**:**	± 3200 mV
Accuracy	**:**	± 0.1 mV (± 0.0016 pH).
Amplifier Input Impedance	**:**	> 10^{12} ohms.
End Point detection	**:**	a)Potentiometric b)Voltametric c)Thermometric and d)Photometric.
Cut-off criteria	**:**	a) Volume b)End point c)mV/pH.
Results	**:**	Molarity, % Assay (wt), % volume (ml) , ppm , mg/l, mg/g, g/l, meq/l, mol/kg, TAN and TBN for oil samples.
Sensors	**:**	1)Electrodes for Potentiometric titration - pH, Ion, Redox, Precipitation. a)Any combination electrode with BNC connector, b)Differential System comprising Sensing (Indicator) with BNC and Reference (4mm banana connector). 2) Electrodes for KF/Voltametric titration with TNC connector. 3) Temperature (PRT/PT100) for ATC with 5-pin shell connector
Calibration	**:**	3-point Calibration with user entered buffer values and standardization (7pH)
Titration Head	**:**	Manual stand with swiveling arm for electrode, dispensing tip & stirrer is provided as standards
Stirring System	**:**	Micro-controller based variable speed, high torque stirrer with indication.
Data Storage	**:**	Non-volatile memory.
Method Storage	:	50 methods with parameters.
Titrant Molarity storage	**:**	within Method data.
Input/Output Peripheral Interface :		a) Parallel Port : 1 No. for printer. b) Serial Port : 2 Nos. for Balance & PC.
Report Format	**:**	Selection of various formats - a) Method parameters b) Titration analysis report c) Titration analysis condensed report d) Titration data table e) Titration graphic report - 1) µl v/s mV 2) µl v/s First derivative 3) µl v/s Second derivative 4) µl v/s time. f) Statistics report g) Calibration report.
Keyboard	**:**	Alphanumeric splash water proof polyester soft keys.
Display	**:**	40 x 2 line back lighted LCD display.
Convertibility	**:**	Can be used KF titrator for Moisture estimation with KF burette system (Optional)

Environmental Operating Conditions :

a) Operation : Indoor.
b) Temperature : Ambient to 45 °C
c) Operating Temperature and humidity : +5 to 45°C Max, 5 to 90% non condensing.
d) Storage Temperature and humidity : +5 to 45°C Max, 5 to 90% non condensing.
e) Altitude in meters : up to 2000m

Power : 110V/220V AC ±10%, 60/50Hz. 40W.

Weight and dimensions :

Control Unit : 12 inch x 12 inch x 6 inch (W X D X H)
Titration unit with Vortex Stirrer : 12 x 16 x 16 (W X D X H)
Magnetic Stirrer (For KF Titration - Optional) : 6 x 10 x 10 (W X D X H)
System Weight : 9 Kg.

FEATURES:

- Advanced Micro-controller based user-friendly state-of-the-art product design with alphanumeric splash waterproof polyester soft keys for keyboard and User interactive software in dialogue mode for ease of operation.
- Quick interchangeable burette assemblies with intelligent recognition for its volume size. Burette factor for dispensing correction is available for true end point volume.
- Alphanumeric entry of Sample Name, Titrant Name, Identification Number, Date & Time with type of electrode used for authentication. Daily Auto Incremented Run number and Factory entered CUSTOMER NAME & Instrument Sr.No. on report printouts make the system foolproof and GLP compliant.
- Protection against invalid parameter entries during method generation.
- Storage of 40 User methods with parameters. (Plus10 not editable – Default methods)
- Two Tier - ADMIN & USER Password Protection for Method data during ADD/EDIT, DELETE, COPY, and for Calibration.
- Three modes of titration - incremental, equilibrium and cut-off by pH mode to perform almost all types of titration. By selecting titration method, instrument prints the type of appropriate electrode.
- Automatic selection of Sensor Input as per titration method selected.
- During titration, the measured variable i.e. electrode potential (mV) or pH value is shown on display together with dispensed volume and number of End Point (EP) detected.
- User selectable End Point (EP) evaluation up to 9 EP during the run, and calculation by first, last, largest, all or selected EP with display of results and printout.
- Composite Differential Electrode Amplifier unit for potentiometric and voltametric / KF titration, having BNC connectivity to various electrodes and temperature sensor for indication.
- Capability to use as simple pH meter with display of pH, mV values & Temperature display.
- Facility to use as a dispenser for fixed volume dosing or dilution allows to perform manual titration with user defined dose and mV indication.
- User Programmable selectivity for report format, complying with GLP requirements: a) Report giving titration parameter and result. b) Data table giving mV, pH, δmV, mV/ml, 2^{nd} Deriv. and volume. c) Graphics report giving mV v/s μl titration curve. d) Graphics report 1^{st} deriv. graph v/s μl titration curve e) Graphics report of 2^{nd} deriv curve. f) Report of method parameters for 50 methods. g) Condensed report of titration parameters and results. h) Auto evaluation report for multi EP samples - EP1, EP2-EP1 & EP3-EP2 etc. available. The reports can be obtained even after resetting/power off / power fail conditions or Run Aborted.

FEATURES: **contd…**

- Result calculation facility to obtain printout in different units such as molarity, factor, % assay (wt), % volume (ml), ppm, mg/l, mg/g, ml/g, g/l, mEq/l, mol/kg, TAN & TBN.
- Reprocessing of threshold and recalculation of EP without performing the new run.
- Display and printout of daily Auto Incremented Run (AIR) number which makes system foolproof and GLP compliant.
- Statistic function with run selectivity for finding Mean, SD, RSD & CV of last 10 repeat run results could be viewed or printed.
- Real Time Clock (RTC) for time display and report printout with run time indication.
- Special Method Compliant to ASTM D664, D2896 & D4739 for TAN and TBN analysis for oil samples.
- Automatic evaluation of molarity determination of titrant.
- Quick Method for Trial analysis RUN having only titration parameter.
- Error indication on Display, helps user to trace the problem.
- Titration run can be started with last run parameters.
- Display of last run or default parameters while entering or running method.
- Display of stir time after pre-dispensing (with selectable dose and time).
- Escape and Back keys for better user interaction.
- Vortex stirrer for homogeneous stirring with glass propeller (provides chemical inertness).
- Digital Speed control of vortex and magnetic stirrer with indication.
- Availability of optional in situ accessory for Titration Vessel heating to perform titration from ambient+10°C to 80 °C. Cooling can be achieved with external chiller.
- PC Compatibility – RS232 interface for Data Downloading to Result Data Capturing Software (Ethernet based) or Windows Hyper Terminal Software (Optional).
- Balance interface for automatic sample weight transfer.
- K905XX can be converted to perform Karl Fischer titration by just changing burette assembly.

SECTION 2

- **Familiarization with Instrument**
- **Front Panel – Keyboard Layout and Functions**

K905XX - Automatic Potentiometric Titrator

Familiarization with the Instrument : -

1) Titration Controller: System Control unit for controlling the Titration Unit.

2) Titration Unit : Consists of Burette Assembly having Gas tight syringe with teflon tipped plunger with luer fitting. The miniature 3-way valve is used to control the titrant flow. Two Teflon tubings are fitted with finger tight fittings. The left side tubing is for titrant aspiration and the right side tubing is for dispensing the titrant in reaction vessel.

3) Stirrer Assembly : Consists of Vortex stirrer with glass propeller with digital controller for variable speed regulation.

4) Printer Output : 25 Pin D type connector – Centronic Parallel port interface

5) Display : 40 X 2, Back lighted, LCD display.

6) Key Board : Multi-function tactile keyboard as shown below.

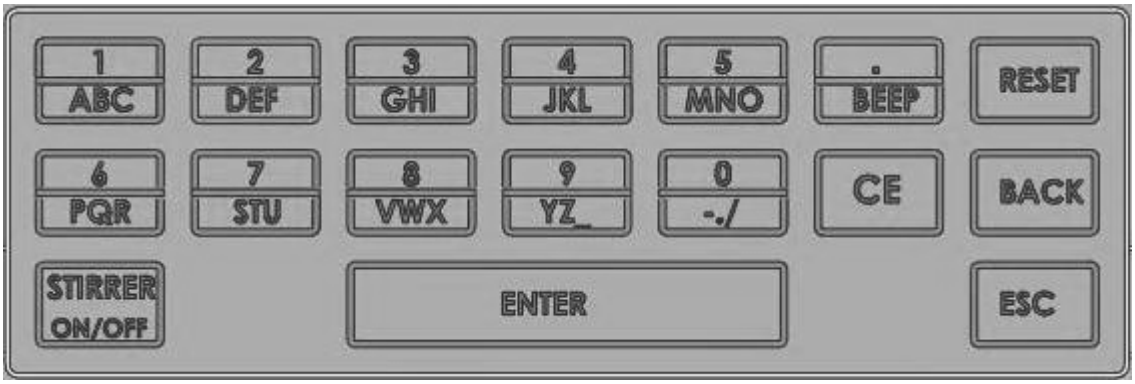
7) Electrode Connection: Differential input with BNC and 4mm Banana Connector.

8) Temperature sensor : 5 pin Shell connector

9) Balance connectivity : RS232C – DB9.

10) PC Connectivity : RS232C – DB9 for data downloading to Result Data Capturing Software (Ethernet based) (Optional) or Windows HyperTerminal or any other terminal software.

FRONT PANEL KEYBOARD LAYOUT :



Key functions:

L.H.S. Keys: Alphanumeric Dual Function (A~Z, 1~9), Zero (0) with Dash (-), Dot (.), Slash (/), Decimal point (.) and Clear Entry(CE) Key.

R.H.S. Keys: Functional Keys and RESET key.

Description of Functional Keys:

1] RESET : To reset the instrument or re-initialize before RUN.

2] BACK : To go back to previous entry.

3] ESC : To Return to Main menu from any operation and to abort the titration during run.

4] ENTER : a) To acknowledge functions.
b) To enter titration parameters.
c) To stop continuous monitoring of mV read by electrode in Scan mode.

5] CE : To cancel the wrong entries.

6] • / BEEP : To enter decimal point during numeric entries. To Add / Remove the Beep during run.

7] STIRRER ON/OFF : To Start / Stop Stirrer Manually.

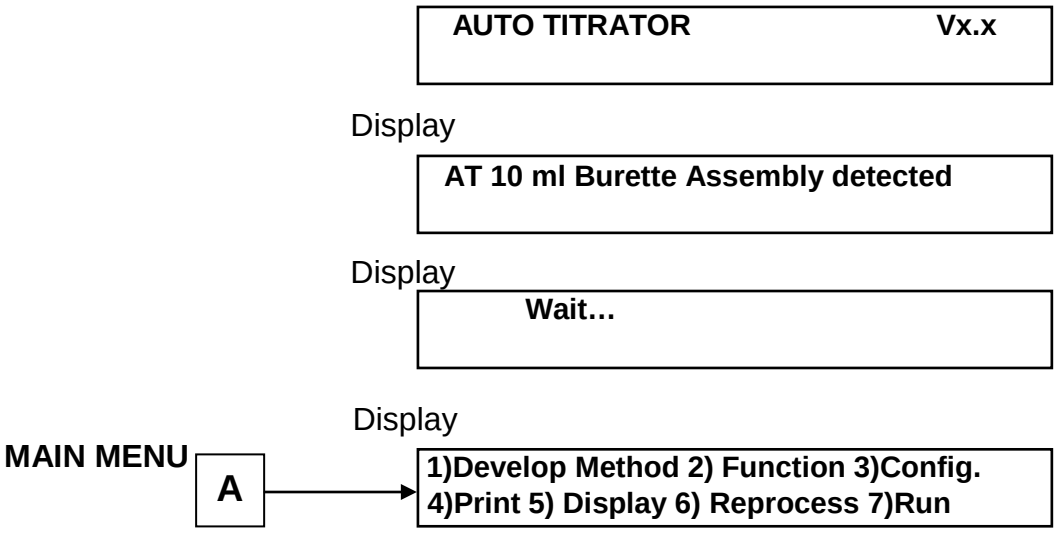
SECTION 3

- **Instrument Operation**
- **Titration Method Development**
- **Function**
- **Methods**

OPERATION :

- **<u>Switching 'ON' the instrument and connecting the accessories:</u>**

a) Connect interface cable between Controller Unit & Titration Unit.
b) Connect the DC Adapter power cord to 230V AC/50 Hz, 5A socket of stabilized power supply with proper earth connection & insert the DC jack in the Controller Unit socket.
c) Connect pH Electrode on the back side of the instrument.
d) Connect printer through appropriate cable to the backside of the instrument.
e) Connect the Vortex stirrer to the backside of Stirrer control unit.
f) Turn the power switch 'ON', with beep sound the LCD display shows the Model & the firmware version with :

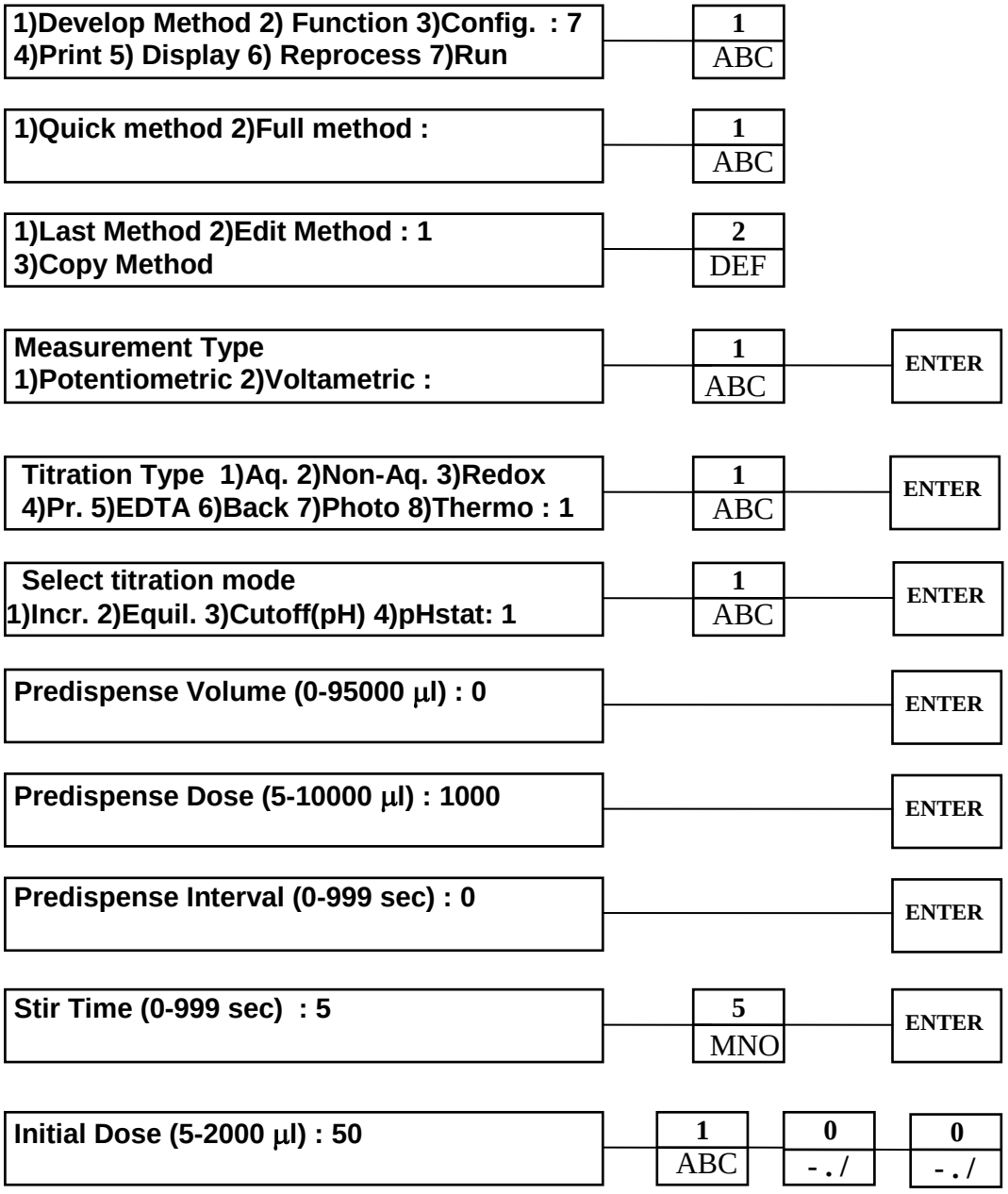


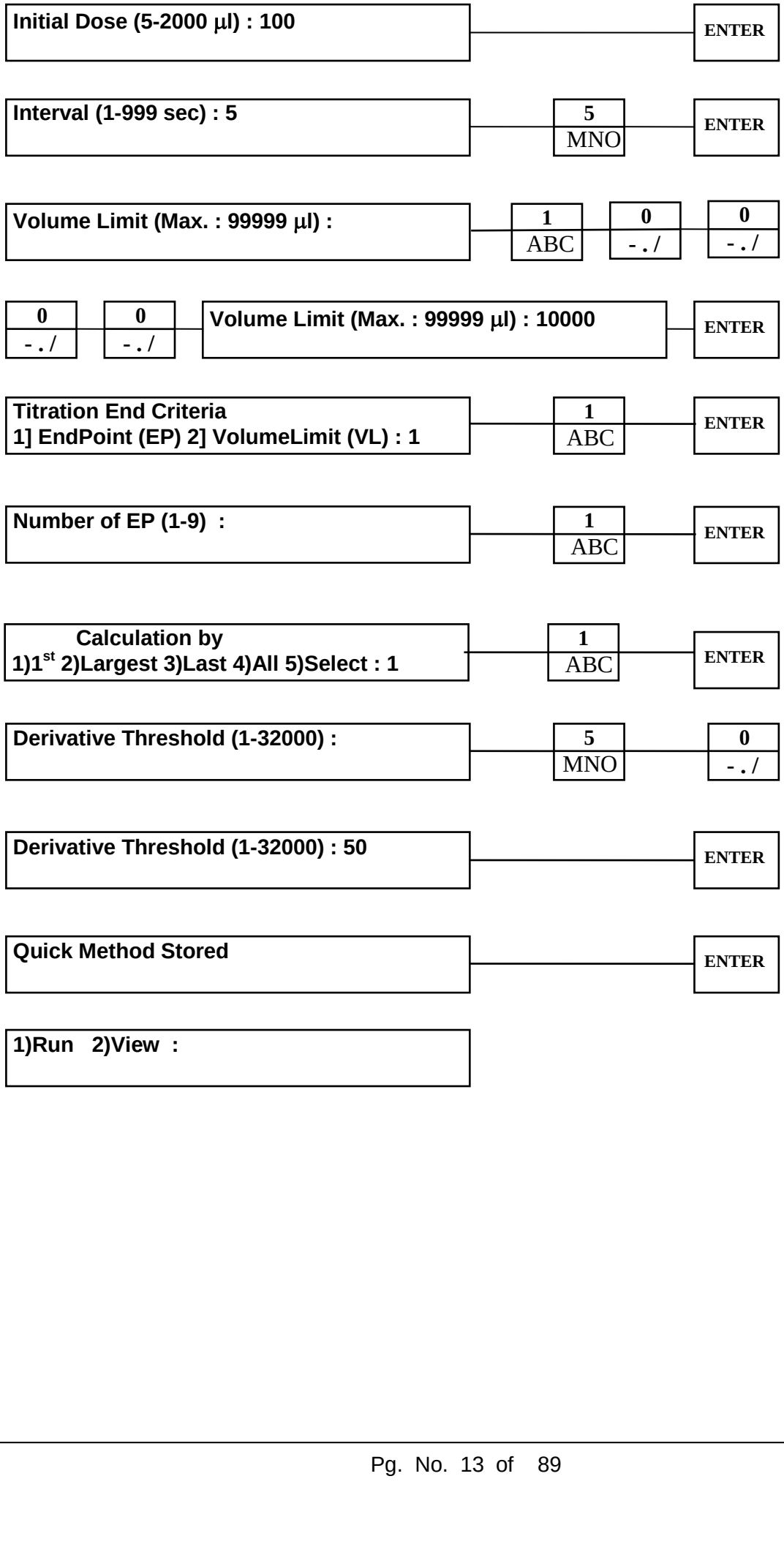
1) Develop Method :- To generate(add) Edit , View Titration Method parameter, Delete, Copy Method. (Refer pages 13)
2) Function :- To perform pH Calibration, mV scan, Burette Priming, Burette change or Manual titration. (Refer pages 45)
3) Config :- System Settings – Clock/date, Balance, Temperature compensation, Printer, Password setting, Titrant temperature factor., Data Transfer, Resolution (Refer pages 50)
4) Print :- Titration report, Statistics, Method parameter, pH Calibration report. (Refer pages 54)
5) Display :- Titration result, Statistics, pH Calibration data. (Refer pages 57)
6) Reprocess :- Data Reprocessing (Refer pages 58)
7) Run :- To Perform titration with desired method (Refer pages 59).

1. Titration Method Development

- **Developing the Quick Method :** Follow the below mentioned instructions

MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Acid-Base Aqueous
TITR. MODE	:	Incremental	P.D.VOLUME	:	0 µl
STIR TIME	:	5 sec	INITIAL DOSE	:	100 µl
INTERVAL	:	5 sec	VOLUME LIMIT	:	10000 µl
TITR. END CRIT.	:	End Point	NO. OF EP	:	1
CAL. BY	:	1 st EP	DERIV.THRESH.:		50




Initial Dose (5-2000 µl) : 100
ENTER
Interval (1-999 sec) : 5
5 MNO
ENTER
Volume Limit (Max. : 99999 µl) :
1 ABC
0 - . /
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 10000
ENTER
Titration End Criteria
1] EndPoint (EP) 2] VolumeLimit (VL) : 1
1 ABC
ENTER
Number of EP (1-9) :
1 ABC
ENTER
Calculation by
1)1st 2)Largest 3)Last 4)All 5)Select : 1
1 ABC
ENTER
Derivative Threshold (1-32000) :
5 MNO
0 - . /
Derivative Threshold (1-32000) : 50
ENTER
Quick Method Stored
ENTER
1)Run 2)View :

- **<u>Viewing & Running the Last Quick Method :</u>** Follow the below mentioned steps . Details of last quick method to be viewed & run :

MEAS. TYPE	:	Potentiometric	TYPE	:	Acid-Base Aqueous
MODE	:	Incremental	PREDISPENSE VOLUME	:	0 µl
PREDISPENSE DOSE	:	1000	PREDISPENSE INTERVAL	:	0
STIR TIME	:	5 sec	INITIAL DOSE	:	100 µl
INTERVAL	:	5 sec	VOLUME LIMIT	:	10000 µl
TITR. END CRIT.	:	End Point	NO. OF EP.	:	1
CAL. BY	:	1 st EP	DERIV.THRESH.	:	50

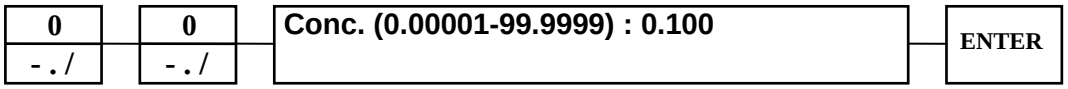
Display	Press
1)Develop Method 2) Function 3)Config. : 7 4)Print 5) Display 6) Reprocess 7)Run	**1** ABC
1)Quick method 2)Full method :	**1** ABC
1)Last Method 2)Edit Method : 1 3)Copy Method	**1** ABC
1)Run 2)View :	**1** ABC
1)Sample Weight 2)Sample Volume :	**2** DEF
Sample Volume (0.0001-999.99999 ml) :	**1** ABC → **ENTER**
Running Quick Method ...	**ENTER**
Wait ..... 00 : 00 : 24	**Quick Method RUN starts with the mentioned para.**
Disp.Vol. : XXX µl # RN BV 10 TITRA+ 250° mV : 00 : 00 : 39	
Vol. Reqd : 0.9425 ml 1)Repeat Run 2)Result 3)Print :	

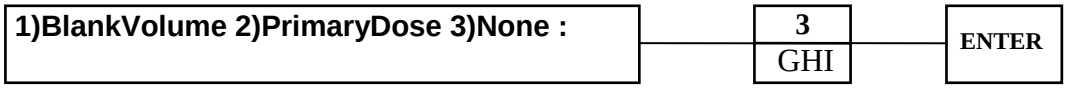
a) To repeat the same quick method with same parameters, select ‘1’ option.

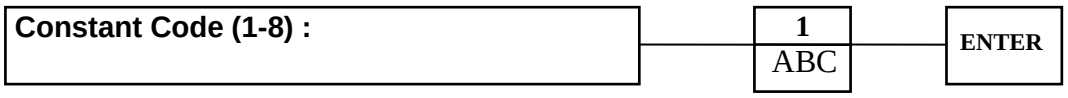
b) To print the data table report for quick method, select ‘3’ option.

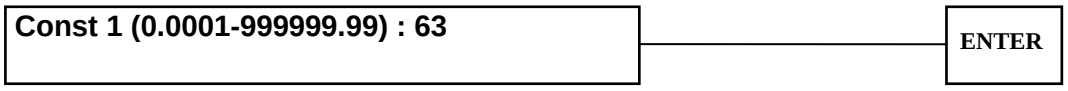
c) To get result for the quick method enter sample analysis parameters as following by selecting ‘2’ option.

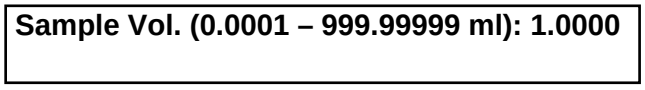
Calculation by 1)1st 2)Largest 3)Last 4)All 5)Select : 1	**ENTER**
Conc. (0.0001-99.9999) :	**0** - . / → • → **1** ABC

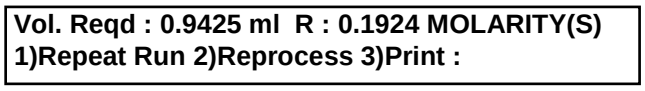








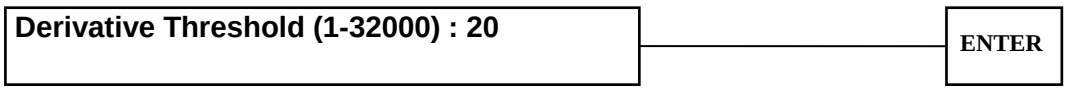


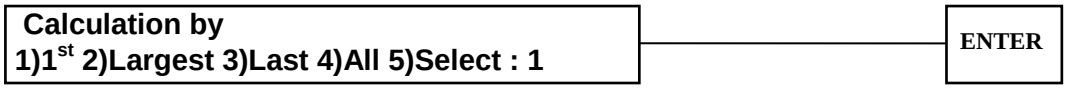


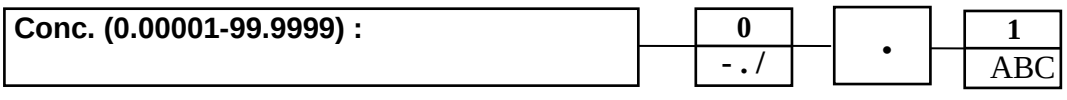
a) To repeat the same quick method with same parameters, select '1' option.

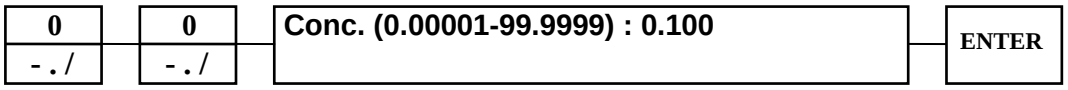
b) To print the data table report for quick method, select '3' option.

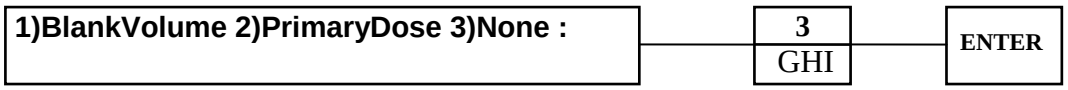
c) To reprocess/ recalculate result in different result unit or to evaluate endpoint by changing the threshold , select '2' option.

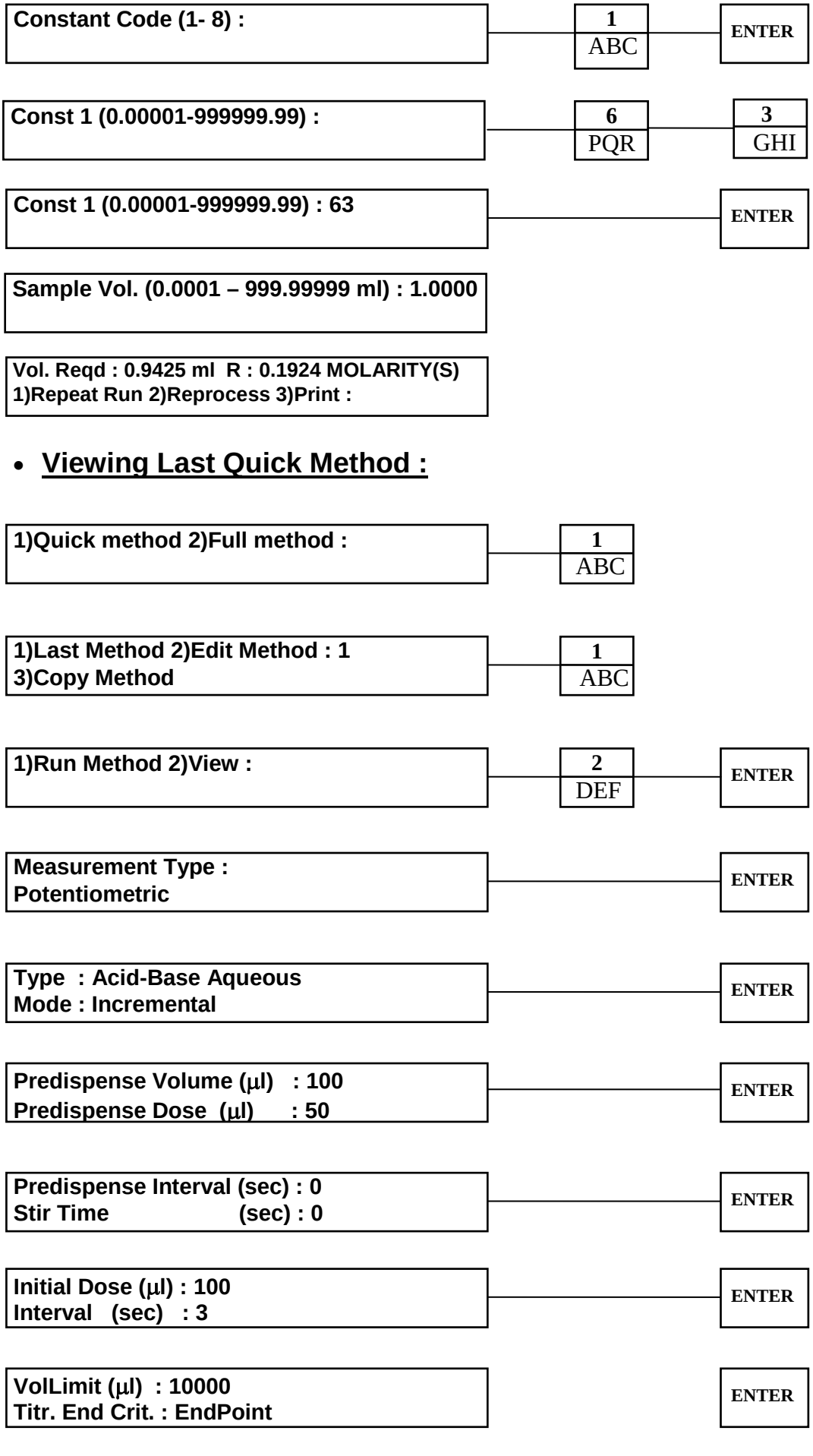




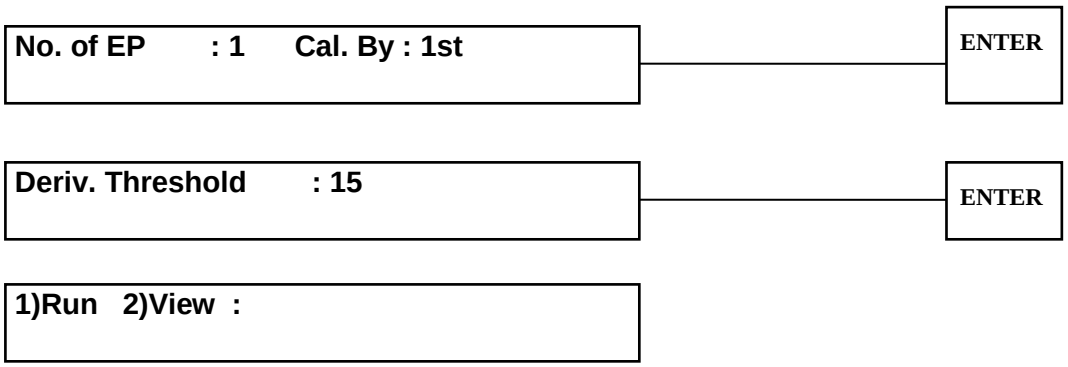




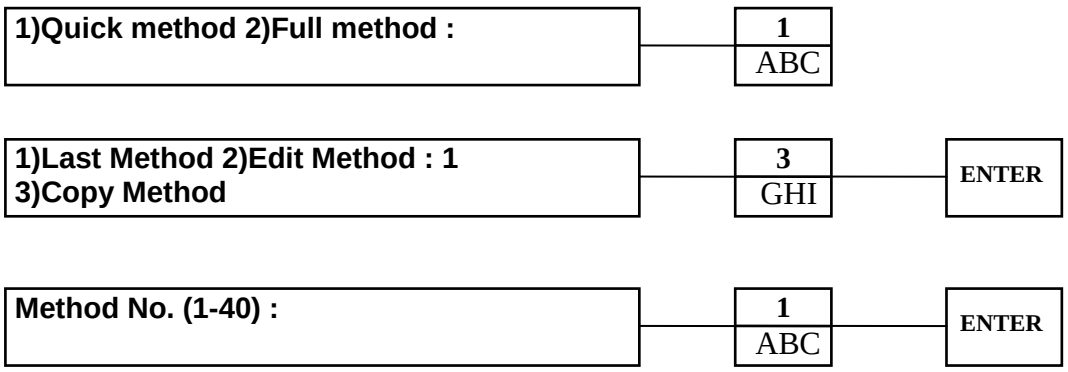


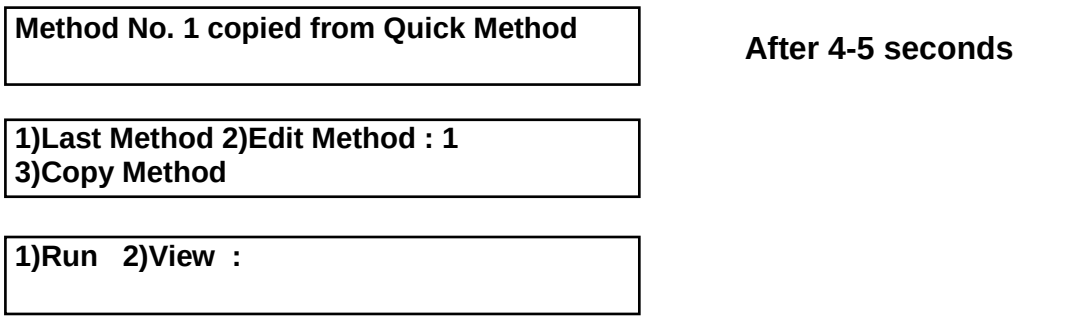


- **Viewing Last Quick Method :**



- **<u>Copying Last Quick Method :</u>** Follow the below mentioned instructions





- **Developing the Full Method :** Follow the below mentioned steps

Examples: TITRATION BY INCREMENTAL METHOD

METHOD NO.	**:**	**1**			
SAMPLE NAME	:	KHP	TITRANT NAME	:	NAOH
MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Acid-Base Aqueous
TITR. MODE	:	Incremental	P.D.CRITERIA	:	0 mV
P.D.VOLUME	:	500 µl	P.D.DOSE	:	100 µl
P.D.INTERVAL	:	5 sec	STIR TIME	:	5 sec
INITIAL DOSE	:	100 µl	INTERVAL	:	5 sec
VOLUME LIMIT	:	10000 µl	TITR. END CRIT.	:	End Point
NO. OF EP	:	1	CALCULATION BY	:	1 st
DERIV.THRESH.	:	50			
CONC. (mol/l)	:	0.100	BV/PD/None	:	None
CONSTANT CODE	:	1	CONSTANT	:	1
PRINT REPORT	:	None			

1)Develop Method 2) Function 3)Config. : 7
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : — **1** ABC — **ENTER**

If method is already present , Display reads :

Method exists !! Edit 1)Y 2)N : — **1** ABC

SAMPLE NAME : — **ENTER**

TITRANT NAME : — **ENTER**

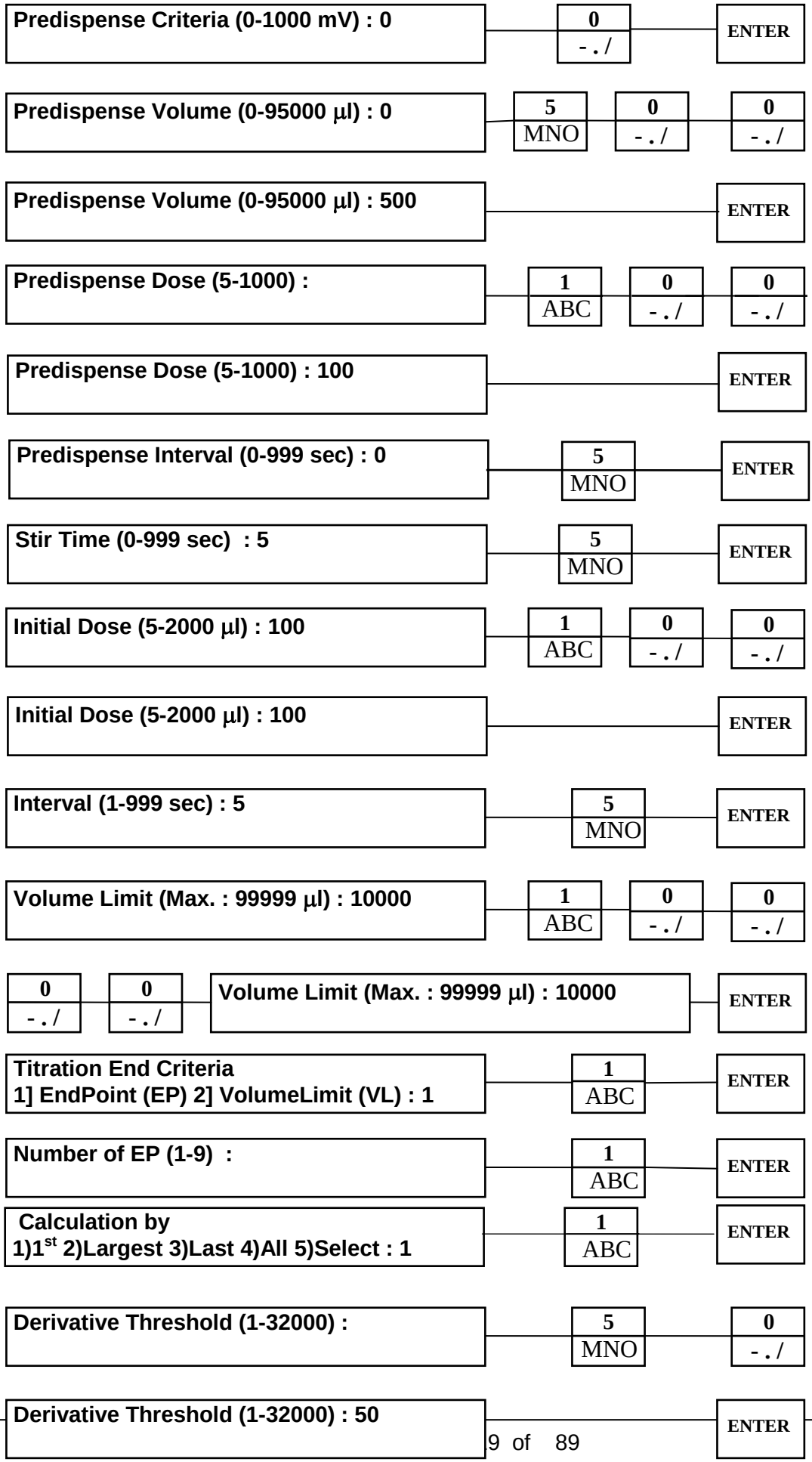
Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

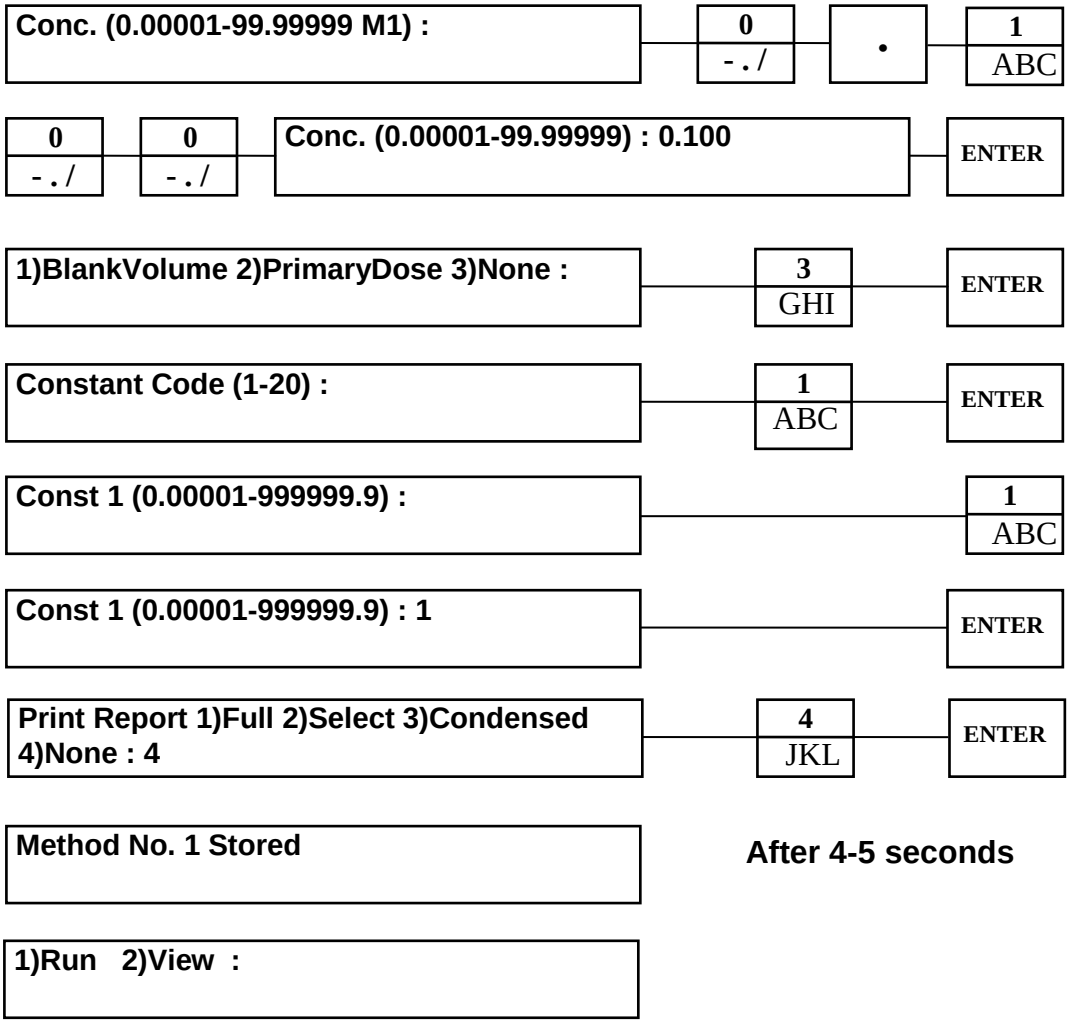
Measurement Type
1)Potentiometric 2)Voltametric : — **1** ABC — **ENTER**

Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 — **1** ABC — **ENTER**

Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 2 — **1** ABC — **ENTER**

<


Predispense Criteria (0-1000 mV) : 0
0
- . /
ENTER
Predispense Volume (0-95000 µl) : 0
5
MNO
0
- . /
0
- . /
Predispense Volume (0-95000 µl) : 500
ENTER
Predispense Dose (5-1000) :
1
ABC
0
- . /
0
- . /
Predispense Dose (5-1000) : 100
ENTER
Predispense Interval (0-999 sec) : 0
5
MNO
ENTER
Stir Time (0-999 sec) : 5
5
MNO
ENTER
Initial Dose (5-2000 µl) : 100
1
ABC
0
- . /
0
- . /
Initial Dose (5-2000 µl) : 100
ENTER
Interval (1-999 sec) : 5
5
MNO
ENTER
Volume Limit (Max. : 99999 µl) : 10000
1
ABC
0
- . /
0
- . /
0
- . /
0
- . /
Volume Limit (Max. : 99999 µl) : 10000
ENTER
Titration End Criteria
1] EndPoint (EP) 2] VolumeLimit (VL) : 1
1
ABC
ENTER
Number of EP (1-9) :
1
ABC
ENTER
Calculation by
1)1st 2)Largest 3)Last 4)All 5)Select : 1
1
ABC
ENTER
Derivative Threshold (1-32000) :
5
MNO
0
- . /
Derivative Threshold (1-32000) : 50
ENTER


Conc. (0.00001-99.99999 M1) :
0
- . /
•
1
ABC
0
- . /
0
- . /
Conc. (0.00001-99.99999) : 0.100
ENTER
1)BlankVolume 2)PrimaryDose 3)None :
3
GHI
ENTER
Constant Code (1-20) :
1
ABC
ENTER
Const 1 (0.00001-999999.9) :
1
ABC
Const 1 (0.00001-999999.9) : 1
ENTER
Print Report 1)Full 2)Select 3)Condensed
4)None : 4
4
JKL
ENTER
Method No. 1 Stored
After 4-5 seconds
1)Run 2)View :

- **TITRATION BY EQUILIBRIUM METHOD:**

METHOD NO.	**:**	**2**			
SAMPLE NAME	:	KHP	TITRANT NAME	:	NAOH
MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Acid-Base Aqueous
TITR. MODE	:	Equilibrium	P.D.CRITERIA	:	0 mV
P.D.VOLUME	:	500 µl	P.D.DOSE	:	100 µl
P.D.INTERVAL	:	5 sec	STIR TIME	:	5 sec
INITIAL DOSE	:	100 µl	EQ. FACTOR	:	5
VOLUME LIMIT	:	10000 µl	TITR. END CRIT.	:	Volume Limit
MIN.CTRL.DOSE	:	5 µl	CALCULATION BY	:	1 st
DERIV.THRESH.	:	500			
CONC. (mol/l)	:	0.100	BV/PD/None	:	None
CONSTANT CODE	:	1	CONSTANT	:	1
TITR. REPORT	:	1 (Yes)	T. DATA TABLE	:	2 (No)
mV/µl GRAPH	:	1 (Yes)	DERIV. GRAPH	:	2 (No)
Time/µl GRAPH	:	2 (No)	2nd DERIV. GRAPH	:	2 (No)

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : — **2** DEF — **ENTER**

SAMPLE NAME : — **ENTER**

TITRANT NAME : — **ENTER**

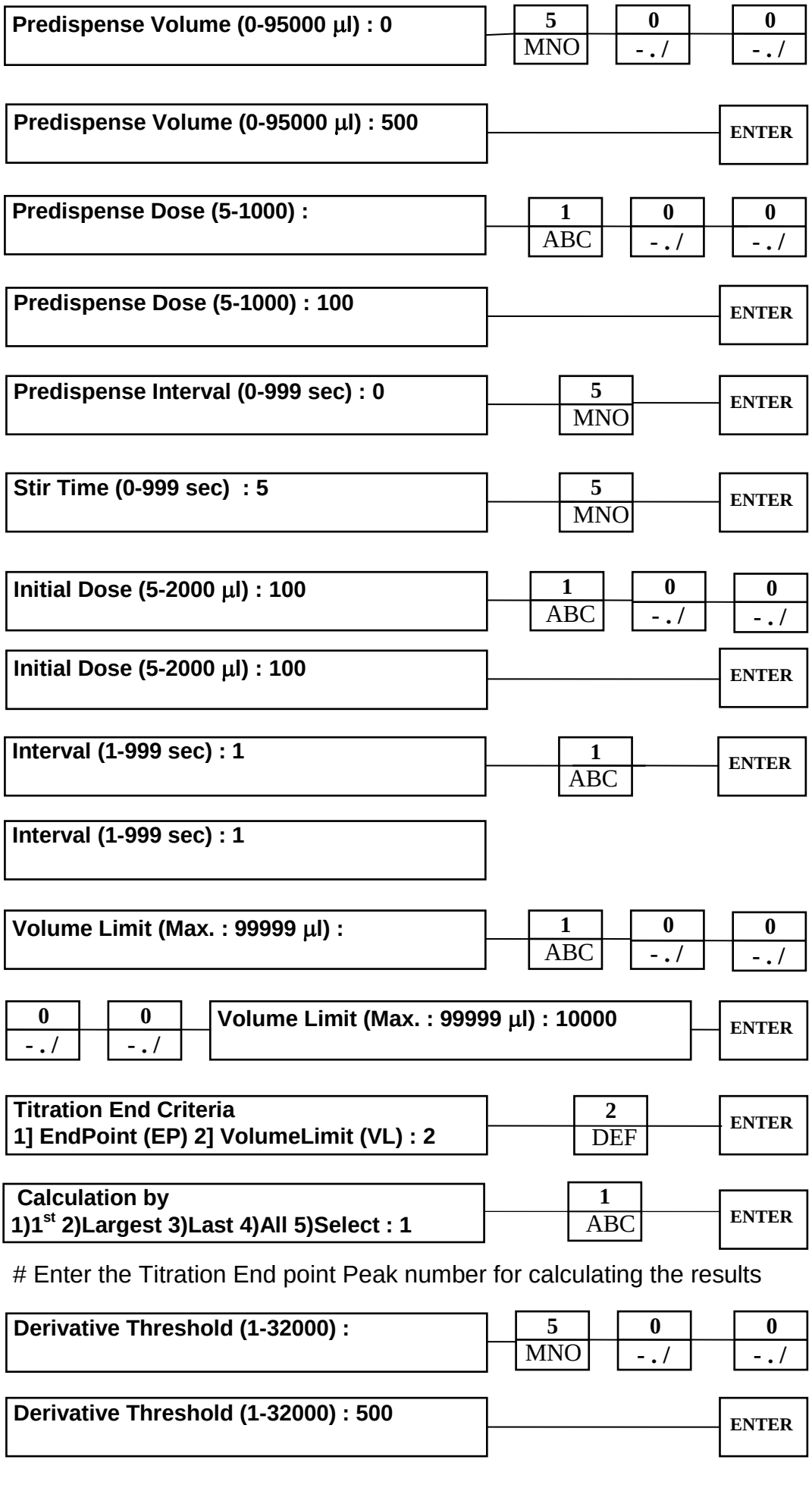
Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

Measurement Type
1)Potentiometric 2)Voltametric : — **1** ABC — **ENTER**

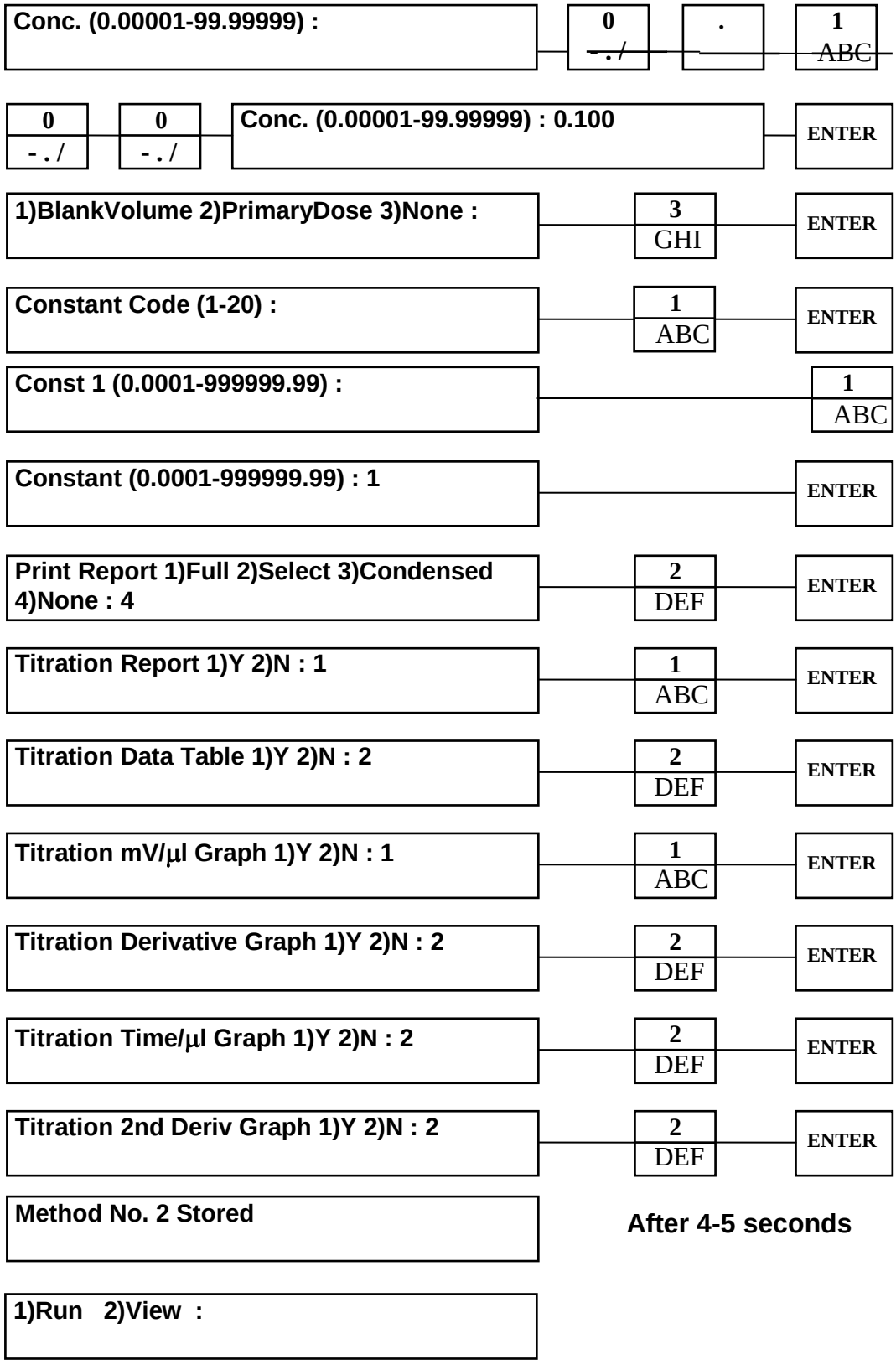
Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 — **1** ABC — **ENTER**

Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 2 — **2** DEF — **ENTER**

Predispense Criteria (0-1000 mV) : 0 — **0** - . / — **ENTER**



Enter the Titration End point Peak number for calculating the results


Conc. (0.00001-99.99999) :
0 - . /
.
1 ABC
0 - . /
0 - . /
Conc. (0.00001-99.99999) : 0.100
ENTER
1)BlankVolume 2)PrimaryDose 3)None :
3 GHI
ENTER
Constant Code (1-20) :
1 ABC
ENTER
Const 1 (0.0001-999999.99) :
1 ABC
Constant (0.0001-999999.99) : 1
ENTER
Print Report 1)Full 2)Select 3)Condensed 4)None : 4
2 DEF
ENTER
Titration Report 1)Y 2)N : 1
1 ABC
ENTER
Titration Data Table 1)Y 2)N : 2
2 DEF
ENTER
Titration mV/µl Graph 1)Y 2)N : 1
1 ABC
ENTER
Titration Derivative Graph 1)Y 2)N : 2
2 DEF
ENTER
Titration Time/µl Graph 1)Y 2)N : 2
2 DEF
ENTER
Titration 2nd Deriv Graph 1)Y 2)N : 2
2 DEF
ENTER
Method No. 2 Stored
After 4-5 seconds
1)Run 2)View :

- **TITRATION BY pH CUTOFF (END) VALUE.:-**

METHOD NO.	**: 3**		
SAMPLE NAME	: HCl	TITRANT NAME	: NAOH
MEAS. TYPE	: Potentiometric	TITR. TYPE	: Acid-Base Aqueous
TITR. MODE	: Cutoff pH	P.D.CRITERIA	: 0 mV
P.D.VOLUME	: 500 µl	P.D.DOSE	: 100 µl
P.D.INTERVAL	: 5 sec	STIR TIME	: 5 sec
INITIAL DOSE	: 100 µl	EQ. FACTOR	: 5
VOLUME LIMIT	: 10000 µl	CTRL. LIMIT	: 4.00 pH
END LIMIT	: 9.00 pH	MIN.CTRL.DOSE	: 10 µl
DERIV.THRESH.	: 500		
CONC. (mol/l)	: 0.100	BV/PD/None	: None
CONSTANT CODE	: 1	CONSTANT	: 1
TITR. REPORT	: 1 (Yes)	T. DATA TABLE	: 2 (No)
mV/µl GRAPH	: 1 (Yes)	DERIV. GRAPH	: 2 (No)
Time/µl GRAPH	: 2 (No)	2nd DERIV. GRAPH	: 2 (No)

1)Develop Method 2) Function 3)Config. : 7
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : — **3** GHI — **ENTER**

SAMPLE NAME : — **ENTER**

TITRANT NAME : — **ENTER**

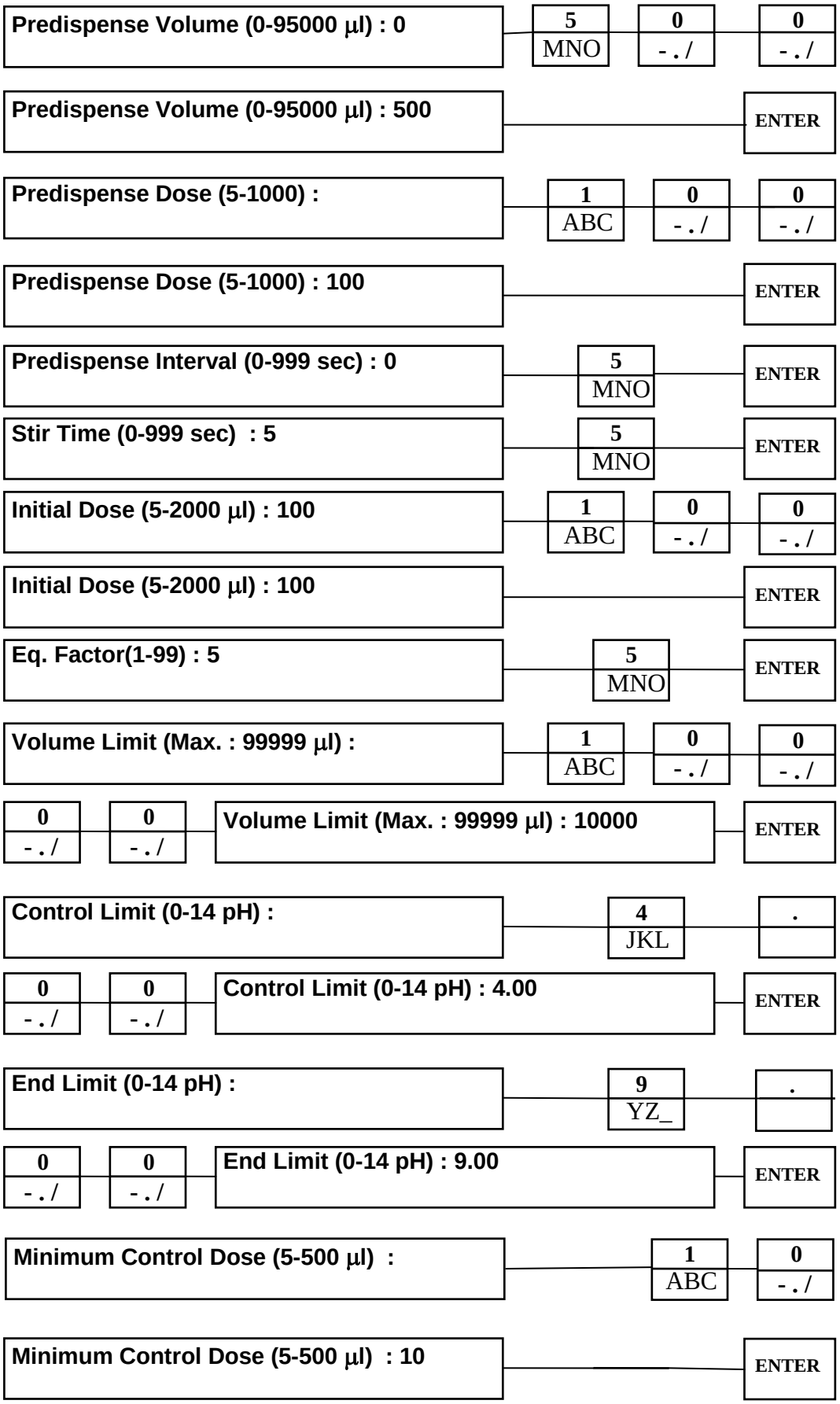
Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

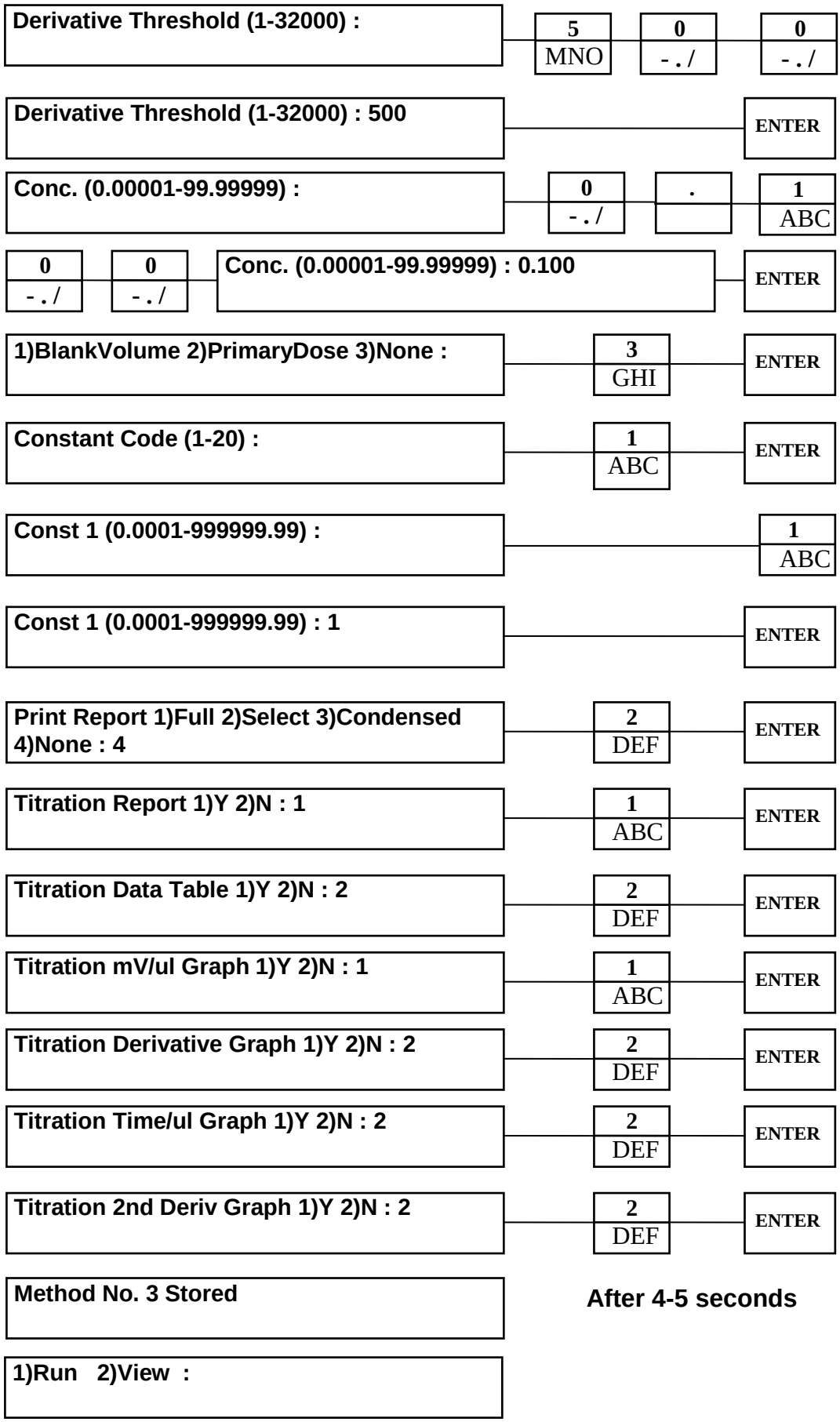
Measurement Type
1)Potentiometric 2)Voltametric : — **1** ABC — **ENTER**

Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 — **1** ABC — **ENTER**

Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 1 — **3** GHI — **ENTER**

Predispense Criteria (0-1000 mV) : 0 — **0** - . / — **ENTER**


Predispense Volume (0-95000 µl) : 0
5 MNO
0 - . /
0 - . /
Predispense Volume (0-95000 µl) : 500
ENTER
Predispense Dose (5-1000) :
1 ABC
0 - . /
0 - . /
Predispense Dose (5-1000) : 100
ENTER
Predispense Interval (0-999 sec) : 0
5 MNO
ENTER
Stir Time (0-999 sec) : 5
5 MNO
ENTER
Initial Dose (5-2000 µl) : 100
1 ABC
0 - . /
0 - . /
Initial Dose (5-2000 µl) : 100
ENTER
Eq. Factor(1-99) : 5
5 MNO
ENTER
Volume Limit (Max. : 99999 µl) :
1 ABC
0 - . /
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 10000
ENTER
Control Limit (0-14 pH) :
4 JKL
.
0 - . /
0 - . /
Control Limit (0-14 pH) : 4.00
ENTER
End Limit (0-14 pH) :
9 YZ_
.
0 - . /
0 - . /
End Limit (0-14 pH) : 9.00
ENTER
Minimum Control Dose (5-500 µl) :
1 ABC
0 - . /
Minimum Control Dose (5-500 µl) : 10
ENTER

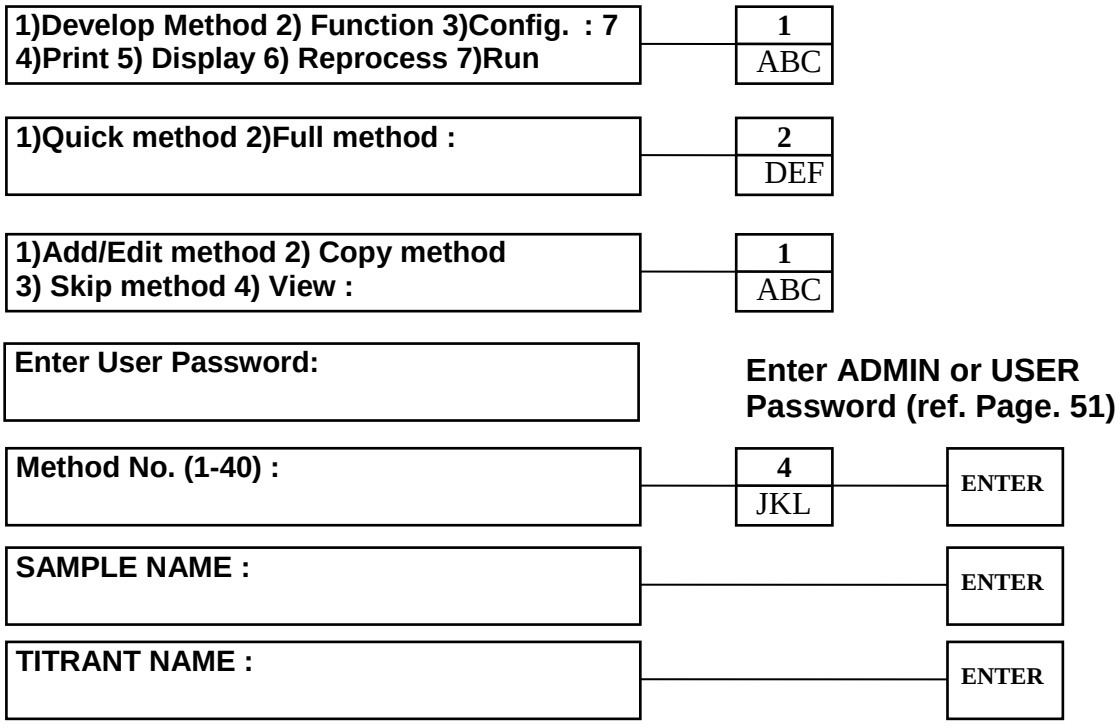

Derivative Threshold (1-32000) :
5 MNO
0 - . /
0 - . /
Derivative Threshold (1-32000) : 500
ENTER
Conc. (0.00001-99.99999) :
0 - . /
.
1 ABC
0 - . /
0 - . /
Conc. (0.00001-99.99999) : 0.100
ENTER
1)BlankVolume 2)PrimaryDose 3)None :
3 GHI
ENTER
Constant Code (1-20) :
1 ABC
ENTER
Const 1 (0.0001-999999.99) :
1 ABC
Const 1 (0.0001-999999.99) : 1
ENTER
Print Report 1)Full 2)Select 3)Condensed 4)None : 4
2 DEF
ENTER
Titration Report 1)Y 2)N : 1
1 ABC
ENTER
Titration Data Table 1)Y 2)N : 2
2 DEF
ENTER
Titration mV/ul Graph 1)Y 2)N : 1
1 ABC
ENTER
Titration Derivative Graph 1)Y 2)N : 2
2 DEF
ENTER
Titration Time/ul Graph 1)Y 2)N : 2
2 DEF
ENTER
Titration 2nd Deriv Graph 1)Y 2)N : 2
2 DEF
ENTER
Method No. 3 Stored
After 4-5 seconds
1)Run 2)View :

- **<u>TITRATION BY PH STAT METHOD: FOR ENZYME KINETIC STUDY.</u>**

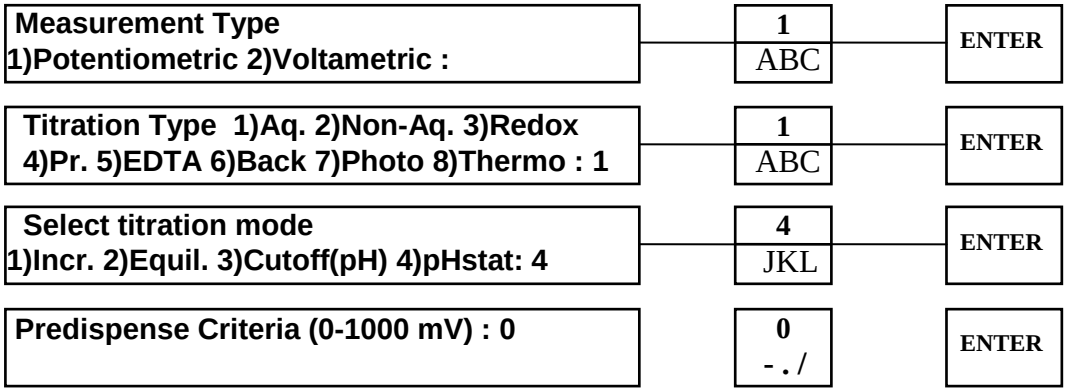
e.g. say pH to be maintained is 8 pH.±0.02pH

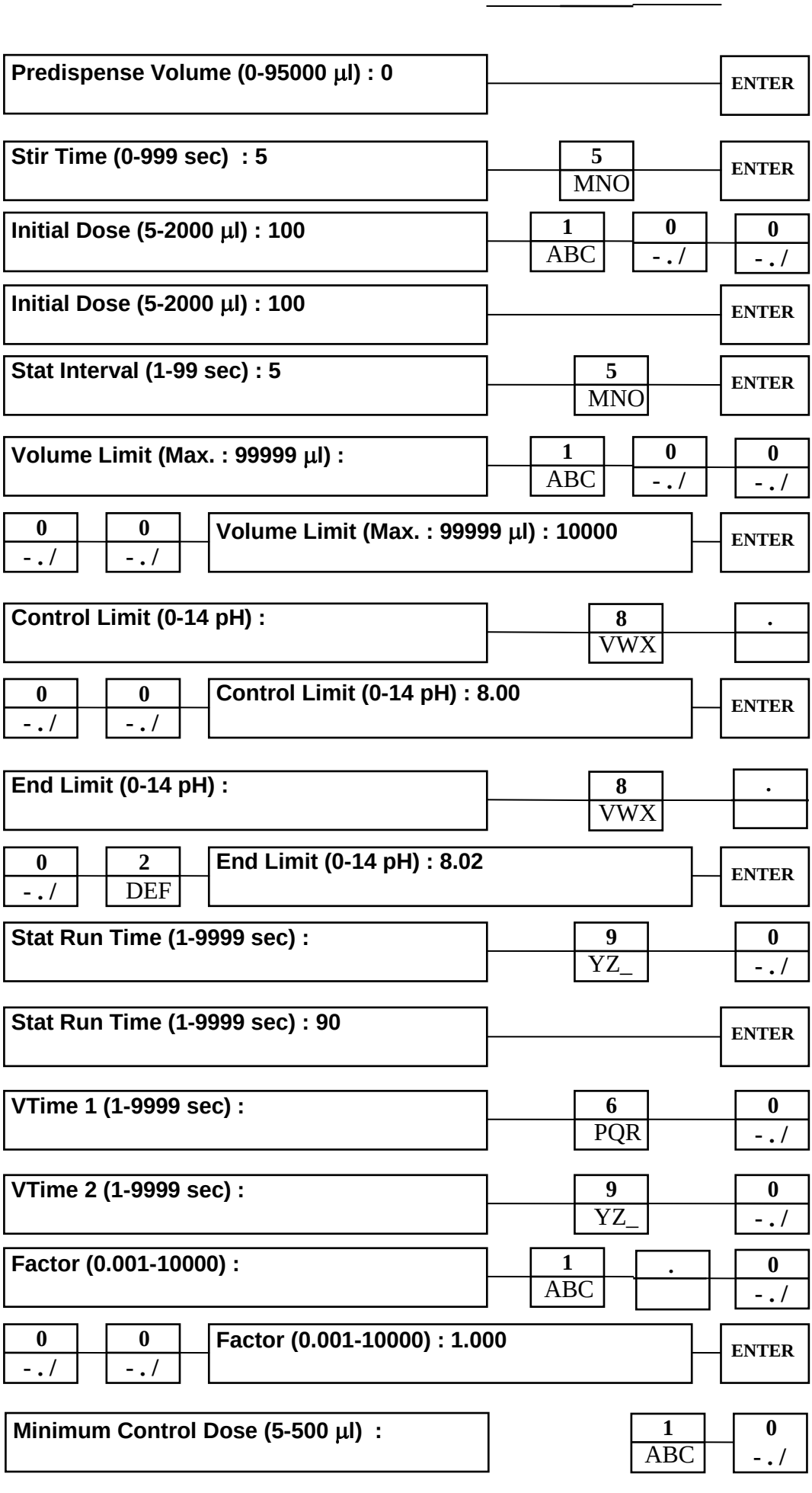
METHOD NO.	**:**	**4**			
SAMPLE NAME	:	ENZYME	TITRANT NAME	:	NAOH
MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Acid-Base Aqueous
TITR. MODE	:	pH stat	P.D.CRITERIA	:	0 mV
P.D.VOLUME	:	0 µl	STIR TIME	:	5 sec
INITIAL DOSE	:	100 µl	STAT INTV.	:	5 sec
VOLUME LIMIT	:	10000 µl	CTRL. LIMIT	:	8.00 pH
END LIMIT	:	8.02 pH	STAT RUN TIME	:	90 sec
VTIME 1	:	30 sec	VTIME 2	:	90 sec
FACTOR	:	1.000	MIN.CTRL DOSE	:	5 µl
DERIV.THRESH.	:	50			
CONC. (mol/l)	:	0.100	BV/PD/None	:	None
CONSTANT CODE	:	8	CONSTANT	:	1
PRINT REPORT	:	Full			

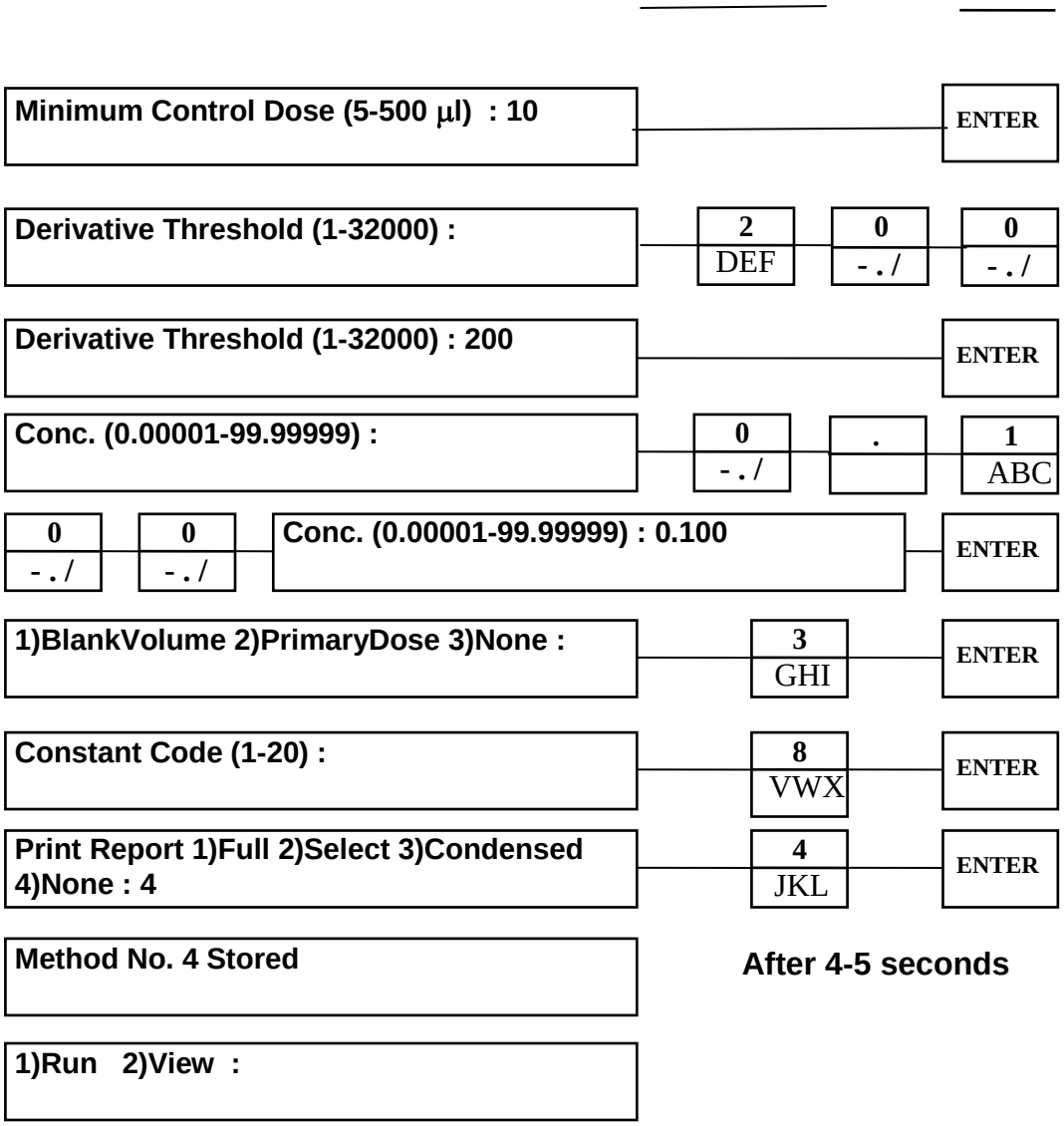
Note : To understand the meaning of each parameter, please refer Appendix F-H.



Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]




Predispense Volume (0-95000 µl) : 0
ENTER
Stir Time (0-999 sec) : 5
5 MNO
ENTER
Initial Dose (5-2000 µl) : 100
1 ABC
0 - . /
0 - . /
Initial Dose (5-2000 µl) : 100
ENTER
Stat Interval (1-99 sec) : 5
5 MNO
ENTER
Volume Limit (Max. : 99999 µl) :
1 ABC
0 - . /
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 10000
ENTER
Control Limit (0-14 pH) :
8 VWX
.
0 - . /
0 - . /
Control Limit (0-14 pH) : 8.00
ENTER
End Limit (0-14 pH) :
8 VWX
.
0 - . /
2 DEF
End Limit (0-14 pH) : 8.02
ENTER
Stat Run Time (1-9999 sec) :
9 YZ_
0 - . /
Stat Run Time (1-9999 sec) : 90
ENTER
VTime 1 (1-9999 sec) :
6 PQR
0 - . /
VTime 2 (1-9999 sec) :
9 YZ_
0 - . /
Factor (0.001-10000) :
1 ABC
.
0 - . /
0 - . /
0 - . /
Factor (0.001-10000) : 1.000
ENTER
Minimum Control Dose (5-500 µl) :
1 ABC
0 - . /


Minimum Control Dose (5-500 µl) : 10
ENTER
Derivative Threshold (1-32000) :
2 DEF
0 - . /
0 - . /
Derivative Threshold (1-32000) : 200
ENTER
Conc. (0.00001-99.99999) :
0 - . /
.
1 ABC
0 - . /
0 - . /
Conc. (0.00001-99.99999) : 0.100
ENTER
1)BlankVolume 2)PrimaryDose 3)None :
3 GHI
ENTER
Constant Code (1-20) :
8 VWX
ENTER
Print Report 1)Full 2)Select 3)Condensed 4)None : 4
4 JKL
ENTER
Method No. 4 Stored
After 4-5 seconds
1)Run 2)View :

- **TITRATION BY MULTIPLE END POINT METHOD:**

METHOD NO.	**: 5**		
SAMPLE NAME	: HALIDES	TITRANT NAME	: $AgNO_3$
MEAS. TYPE	: Potentiometric	TITR. TYPE	: Precipitation
TITR. MODE	: Incrimental	P.D.CRITERIA	: 0 mV
P.D.VOLUME	: 0 µl	STIR TIME	: 5 sec
INITIAL DOSE	: 100 µl	INTERVAL	: 5 sec
VOLUME LIMIT	: 5000 µl	TITR. END CRIT.	: End Point
NO. OF EP	: 3	CALCULATION BY	: ALL
DERIV.THRESH.	: 20		
CONC. (mol/l)	: 0.100	BV/PD/None	: None
CONSTANT CODE	: 13	CONSTANT 1	: 16.9
PRINT REPORT	: None	CONSTANT 2	: 11.9
		CONSTANT 3	: 3.55

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : — **5** MNO — **ENTER**

If method is already present , Display reads :

Method exists !! Edit 1)Y 2)N : — **1** ABC

SAMPLE NAME : — **ENTER**

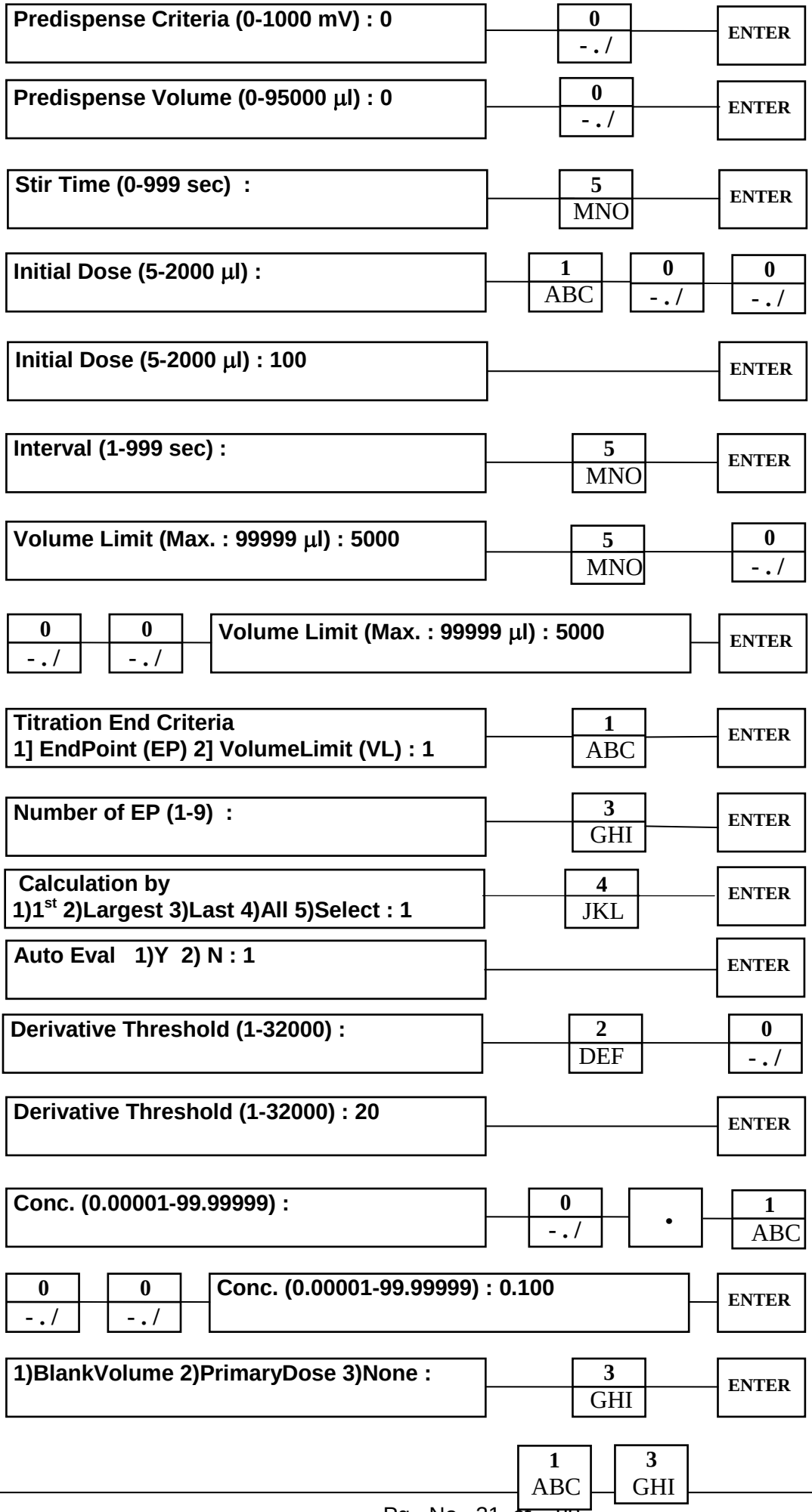
TITRANT NAME : — **ENTER**

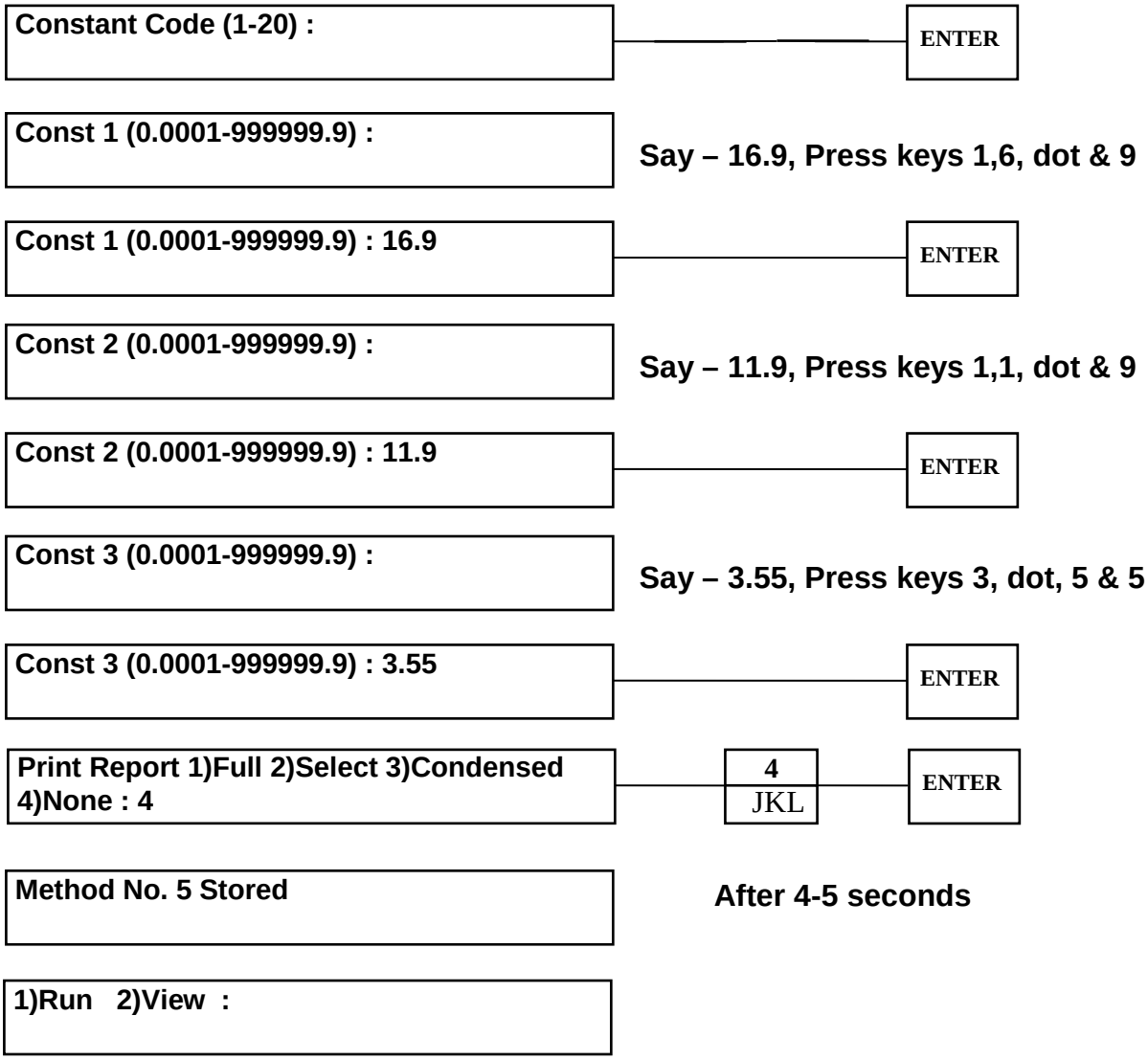
Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

Measurement Type
1)Potentiometric 2)Voltametric : — **1** ABC — **ENTER**

Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 — **4** JKL — **ENTER**

Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 2 — **1** ABC — **ENTER**


Predispense Criteria (0-1000 mV) : 0
0 - . /
ENTER
Predispense Volume (0-95000 µl) : 0
0 - . /
ENTER
Stir Time (0-999 sec) :
5 MNO
ENTER
Initial Dose (5-2000 µl) :
1 ABC
0 - . /
0 - . /
Initial Dose (5-2000 µl) : 100
ENTER
Interval (1-999 sec) :
5 MNO
ENTER
Volume Limit (Max. : 99999 µl) : 5000
5 MNO
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 5000
ENTER
Titration End Criteria
1] EndPoint (EP) 2] VolumeLimit (VL) : 1
1 ABC
ENTER
Number of EP (1-9) :
3 GHI
ENTER
Calculation by
1)1st 2)Largest 3)Last 4)All 5)Select : 1
4 JKL
ENTER
Auto Eval 1)Y 2) N : 1
ENTER
Derivative Threshold (1-32000) :
2 DEF
0 - . /
Derivative Threshold (1-32000) : 20
ENTER
Conc. (0.00001-99.99999) :
0 - . /
•
1 ABC
0 - . /
0 - . /
Conc. (0.00001-99.99999) : 0.100
ENTER
1)BlankVolume 2)PrimaryDose 3)None :
3 GHI
ENTER
1 ABC
3 GHI


Constant Code (1-20) :
ENTER
Const 1 (0.0001-999999.9) :
Say – 16.9, Press keys 1,6, dot & 9
Const 1 (0.0001-999999.9) : 16.9
ENTER
Const 2 (0.0001-999999.9) :
Say – 11.9, Press keys 1,1, dot & 9
Const 2 (0.0001-999999.9) : 11.9
ENTER
Const 3 (0.0001-999999.9) :
Say – 3.55, Press keys 3, dot, 5 & 5
Const 3 (0.0001-999999.9) : 3.55
ENTER
Print Report 1)Full 2)Select 3)Condensed 4)None : 4
4
JKL
ENTER
Method No. 5 Stored
After 4-5 seconds
1)Run 2)View :

TITRATION METHOD FOR BLANK & BACK TITRATION:-

Back-titrations may be used in volumetric analysis where direct titrations cannot be used for technical reasons such as Sample solubility, Trace of impurities, and slow reaction. For example, when a compound is not soluble in water, or where it contains impurities, which interfere with direct titration, an excess of the titrant may be added, and the excess then determined by titration (i.e. by back titration). The volume required for sample is determined by the difference between the two titrants endpoint volume.
Firstly Perform determine the Blank Volume for Excess Titrant say 20mL of 0.1M NaOH by titration with 0.1M HCL. The volume required for endpoint (EP volume) is entered as Blank Volume in the Back titration method during the actual BACK titration

METHOD NO.	**:**	**6**	**(To estimate Blank Volume)**		
SAMPLE NAME	:	NaOH	TITRANT NAME	:	HCl
MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Acid-Base Aqueous
TITR. MODE	:	Equllibrium	P.D.CRITERIA	:	0 mV
P.D.VOLUME	:	18000 µl	P.D.DOSE	:	500 µl
P.D.INTERVAL	:	5 sec	STIR TIME	:	30 sec
INITIAL DOSE	:	200 µl	EQ. FACTOR	:	6 sec
VOLUME LIMIT	:	22000 µl	TITR. END CRIT.	:	End Point
NO. OF EP	:	1	CALCULATION BY	:	1 st
MIN.CONT.DOSE	:	10	DERIV.THRESH.	:	300
			CONC. (mol/l)	:	0.100
BV/PD/None	:	None	CONSTANT CODE	:	8
PRINT REPORT	:	None			

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run → **1** ABC

1)Quick method 2)Full method : → **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : → **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : → **6** PQR → **ENTER**

If method is already present , Display reads :

Method exists !! Edit 1)Y 2)N : → **1** ABC

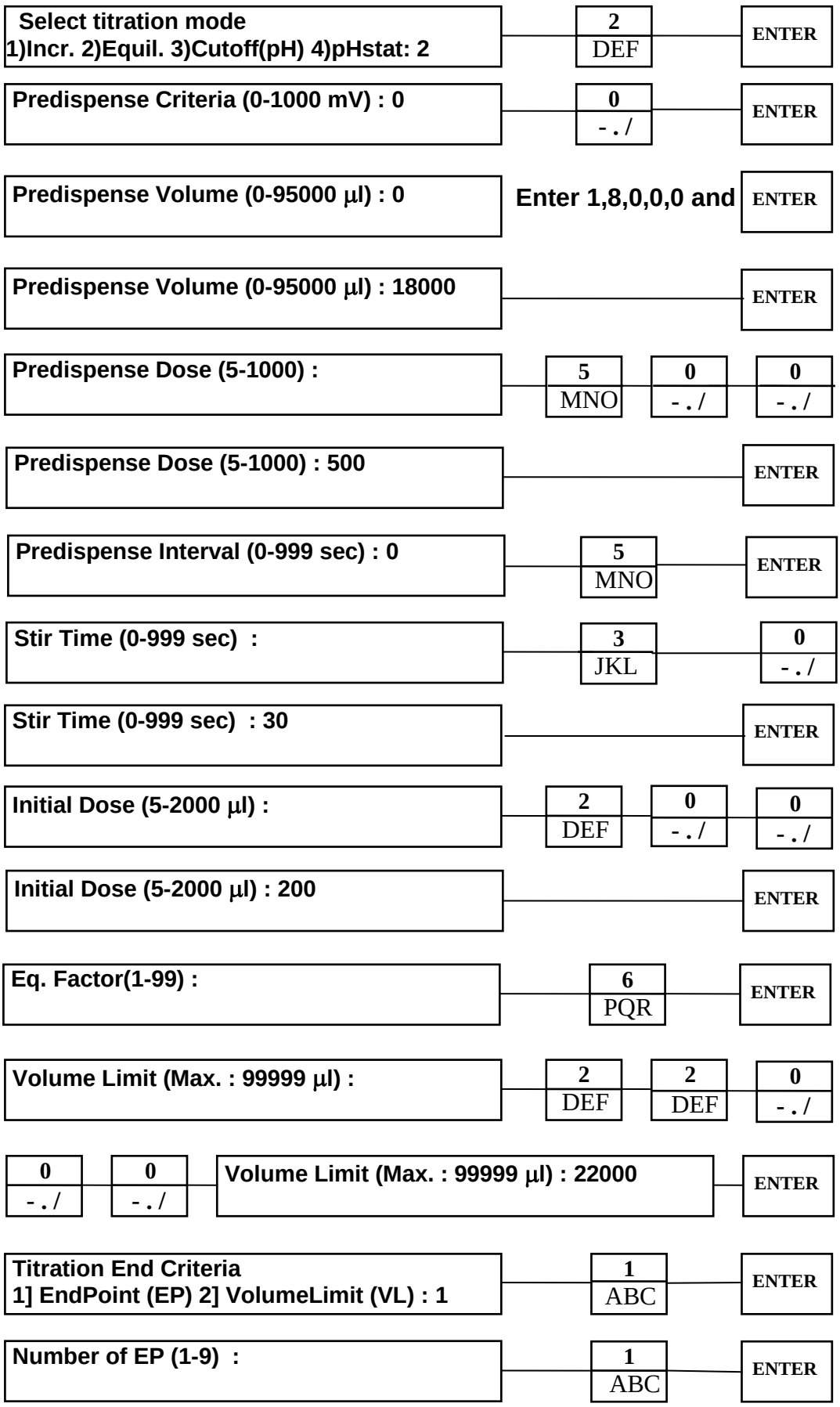
SAMPLE NAME : → **ENTER**

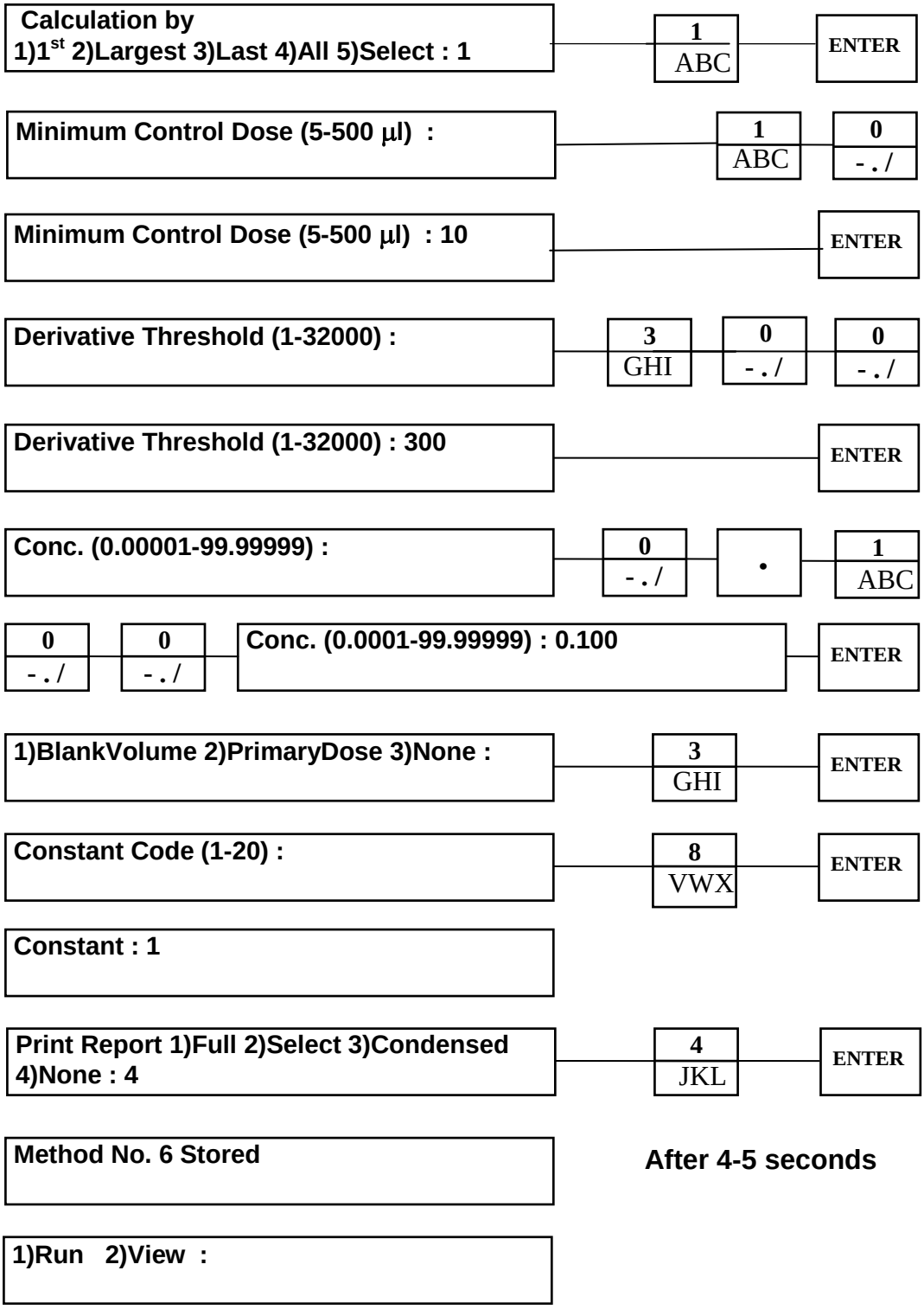
TITRANT NAME : → **ENTER**

Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

Measurement Type
1)Potentiometric 2)Voltametric : → **1** ABC → **ENTER**

Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 → **1** ABC → **ENTER**


Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 2
2 DEF
ENTER
Predispense Criteria (0-1000 mV) : 0
0 - . /
ENTER
Predispense Volume (0-95000 µl) : 0
Enter 1,8,0,0,0 and
ENTER
Predispense Volume (0-95000 µl) : 18000
ENTER
Predispense Dose (5-1000) :
5 MNO
0 - . /
0 - . /
Predispense Dose (5-1000) : 500
ENTER
Predispense Interval (0-999 sec) : 0
5 MNO
ENTER
Stir Time (0-999 sec) :
3 JKL
0 - . /
Stir Time (0-999 sec) : 30
ENTER
Initial Dose (5-2000 µl) :
2 DEF
0 - . /
0 - . /
Initial Dose (5-2000 µl) : 200
ENTER
Eq. Factor(1-99) :
6 PQR
ENTER
Volume Limit (Max. : 99999 µl) :
2 DEF
2 DEF
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 22000
ENTER
Titration End Criteria
1] EndPoint (EP) 2] VolumeLimit (VL) : 1
1 ABC
ENTER
Number of EP (1-9) :
1 ABC
ENTER



Run this Method to obtain the volume required for the titrant to be added as excess in the Back Titration. (Refer to RUN part in this manual)

- **METHOD FOR BACK TITRATION:-**

METHOD NO.	**:**	**7 (BACK Titration)**			
SAMPLE NAME	:	Sample + NaOH	TITRANT NAME	:	HCl
MEAS. TYPE	:	Potentiometric	TITR. TYPE	:	Back
TITR. MODE	:	Equllibrium	P.D.CRITERIA	:	0 mV
P.D.VOLUME	:	10000	P.D.DOSE	:	500 µl
P.D.INTERVAL	:	5 sec	STIR TIME	:	30 sec
INITIAL DOSE	:	200 µl	EQ. FACTOR	:	6 sec
VOLUME LIMIT	:	22,000	TITR. END CRIT.	:	End Point
NO. OF EP	:	1	CALCULATION BY	:	1 st
MIN.CONT.DOSE	:	10	DERIV.THRESH.	:	300
			CONC. (mol/l)	:	0.100
BV/PD/None	:	19.5	CONSTANT CODE	:	13
PRINT REPORT	:	None			

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **1** ABC

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

Method No. (1-40) : — **7** STU — **ENTER**

If method is already present, Display reads :

Method exists !! Edit 1)Y 2)N : — **1** ABC

SAMPLE NAME : — **ENTER**

TITRANT NAME : — **ENTER**

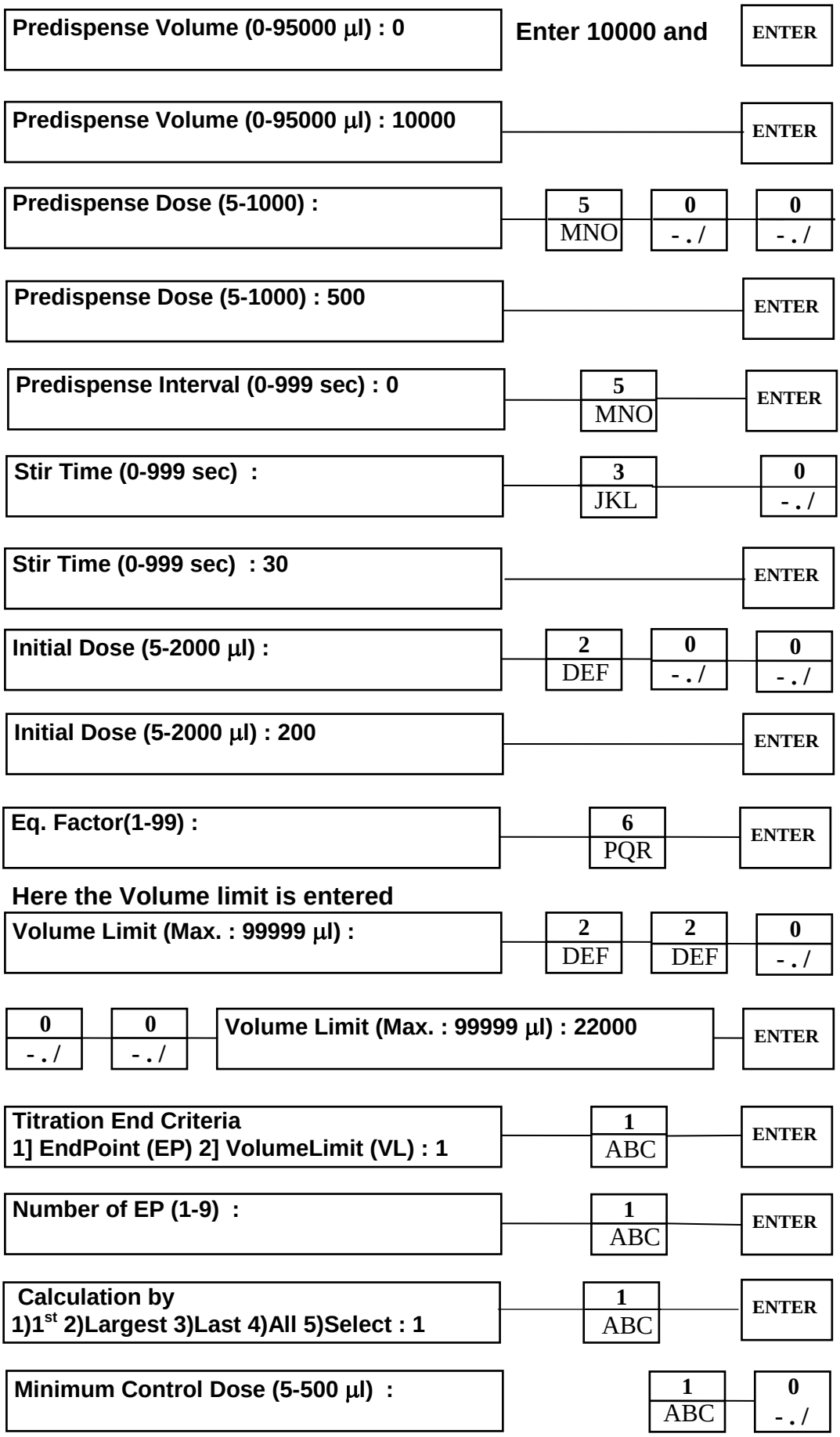
Enter Sample and Titrant Name [refer Appendix-A for alphanumeric entries]

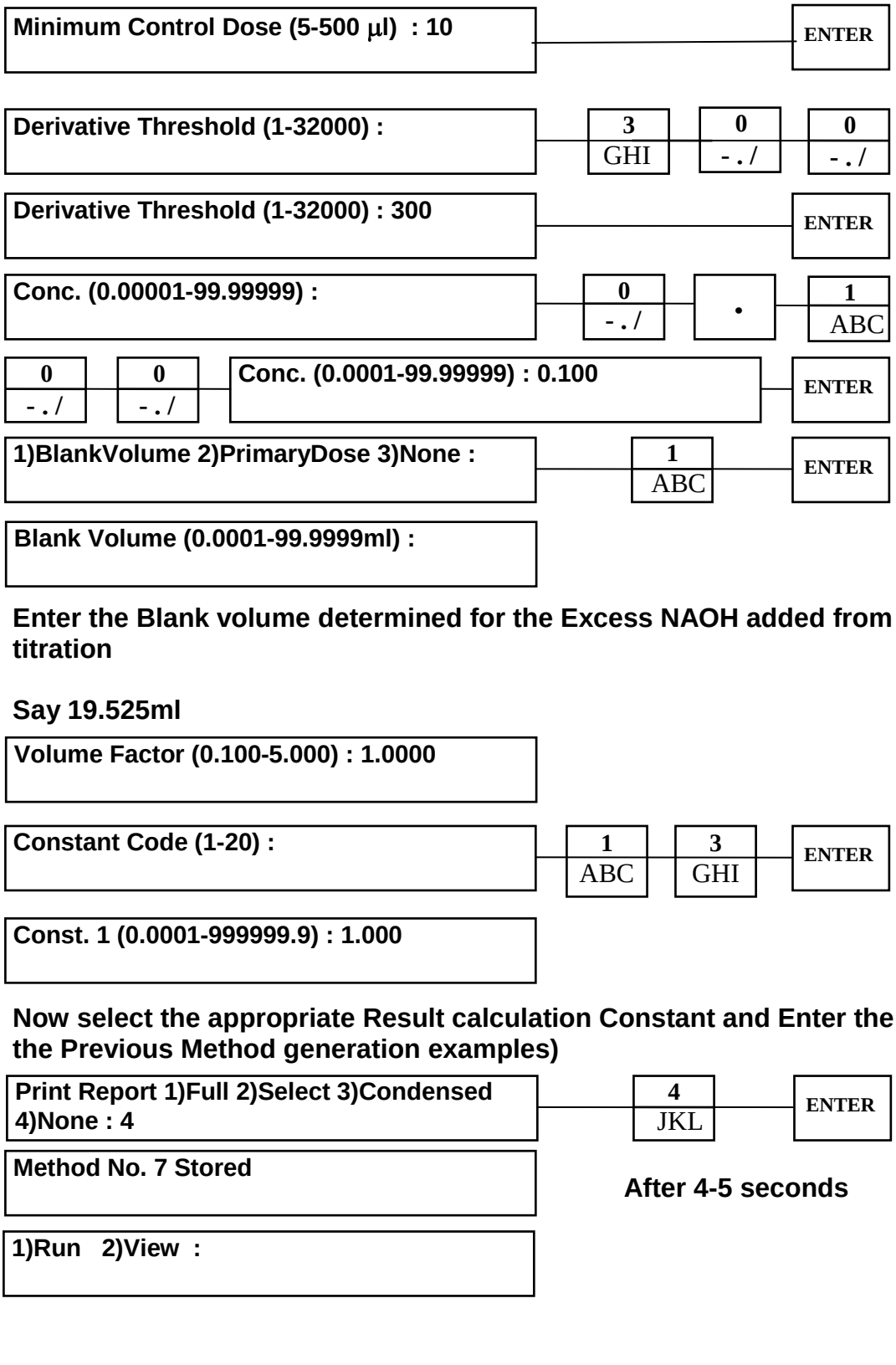
Measurement Type
1)Potentiometric 2)Voltametric : — **1** ABC — **ENTER**

Titration Type 1)Aq. 2)Non-Aq. 3)Redox
4)Pr. 5)EDTA 6)Back 7)Photo 8)Thermo : 1 — **6** PQR — **ENTER**

Select titration mode
1)Incr. 2)Equil. 3)Cutoff(pH) 4)pHstat: 2 — **2** DEF — **ENTER**

Predispense Criteria (0-1000 mV) : 0 — **0** - . / — **ENTER**


Predispense Volume (0-95000 µl) : 0
Enter 10000 and
ENTER
Predispense Volume (0-95000 µl) : 10000
ENTER
Predispense Dose (5-1000) :
5 MNO
0 - . /
0 - . /
Predispense Dose (5-1000) : 500
ENTER
Predispense Interval (0-999 sec) : 0
5 MNO
ENTER
Stir Time (0-999 sec) :
3 JKL
0 - . /
Stir Time (0-999 sec) : 30
ENTER
Initial Dose (5-2000 µl) :
2 DEF
0 - . /
0 - . /
Initial Dose (5-2000 µl) : 200
ENTER
Eq. Factor(1-99) :
6 PQR
ENTER
Here the Volume limit is entered
Volume Limit (Max. : 99999 µl) :
2 DEF
2 DEF
0 - . /
0 - . /
0 - . /
Volume Limit (Max. : 99999 µl) : 22000
ENTER
Titration End Criteria
1] EndPoint (EP) 2] VolumeLimit (VL) : 1
1 ABC
ENTER
Number of EP (1-9) :
1 ABC
ENTER
Calculation by
1)1st 2)Largest 3)Last 4)All 5)Select : 1
1 ABC
ENTER
Minimum Control Dose (5-500 µl) :
1 ABC
0 - . /



Enter the Blank volume determined for the Excess NAOH added from the 1st titration

Say 19.525ml

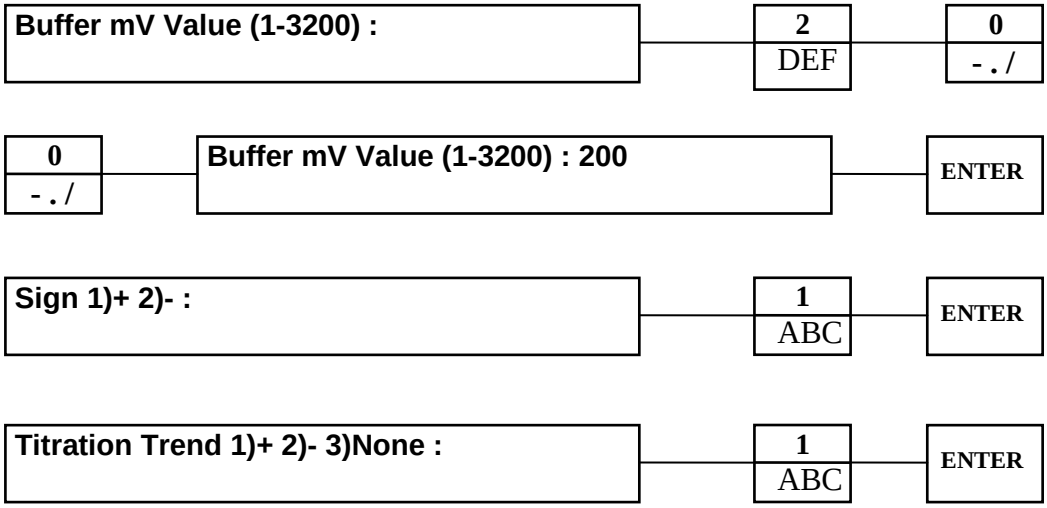
Now select the appropriate Result calculation Constant and Enter the Value (refer the Previous Method generation examples)

After 4-5 seconds

Entering Method for TAN & TBN (ASTM D664, ASTM D2896 & ASTM D4739)

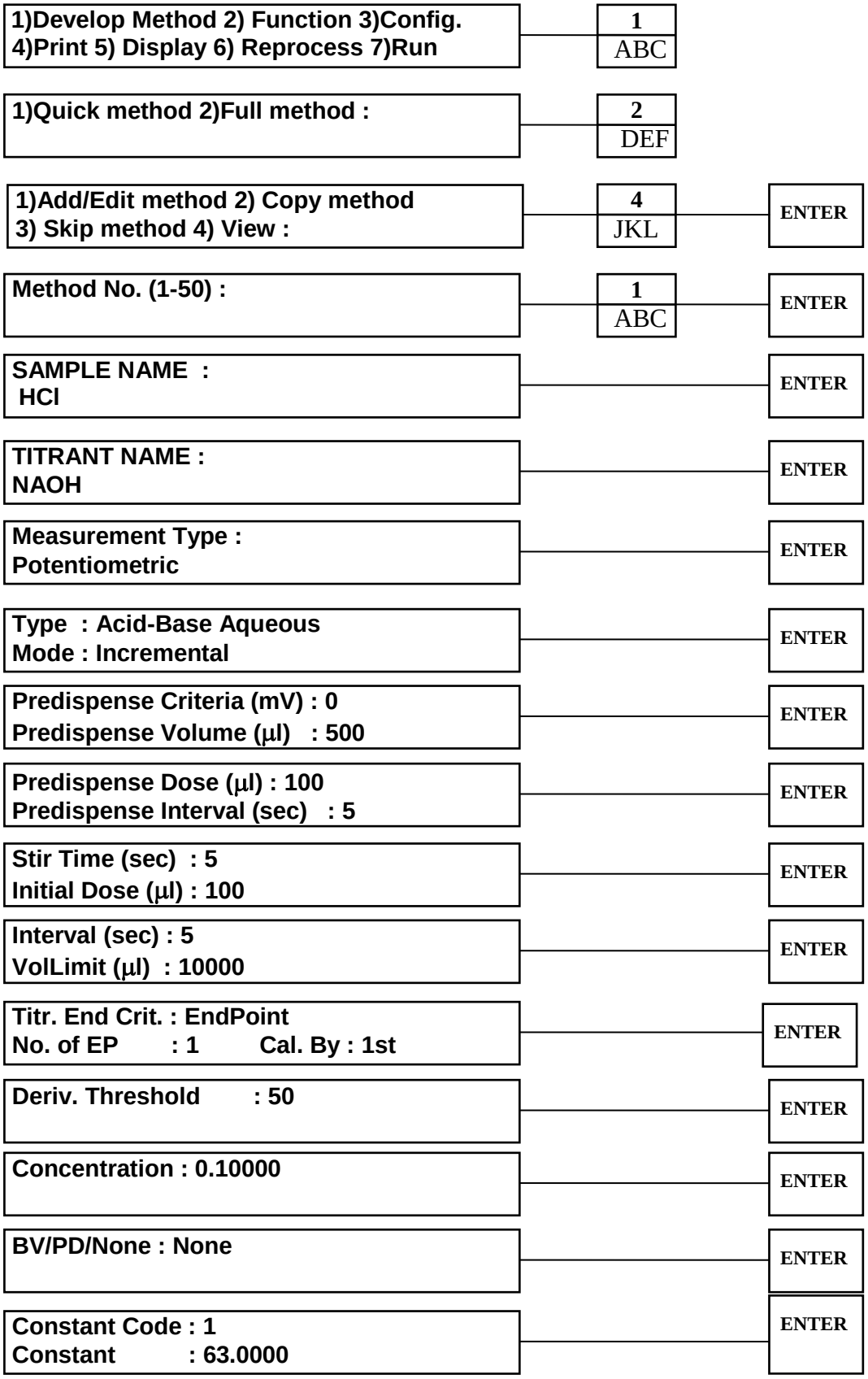
Note : Select following Titration methods numbers for TAN & TBN

- **Method No. 39** for **ASTM D2896**, which is indicated on printout.
- **Method No. 38 & 40** are respectively for **ASTM D664** and **D4739** for which buffer values should be entered as following, the same is indicated on printout.



- **<u>Viewing the Titration Methods :</u> All the 50 methods can be scanned for checking or modifying the parameters individually.**

Say Method No.1 has to be viewed before performing analysis.



Print Report : None

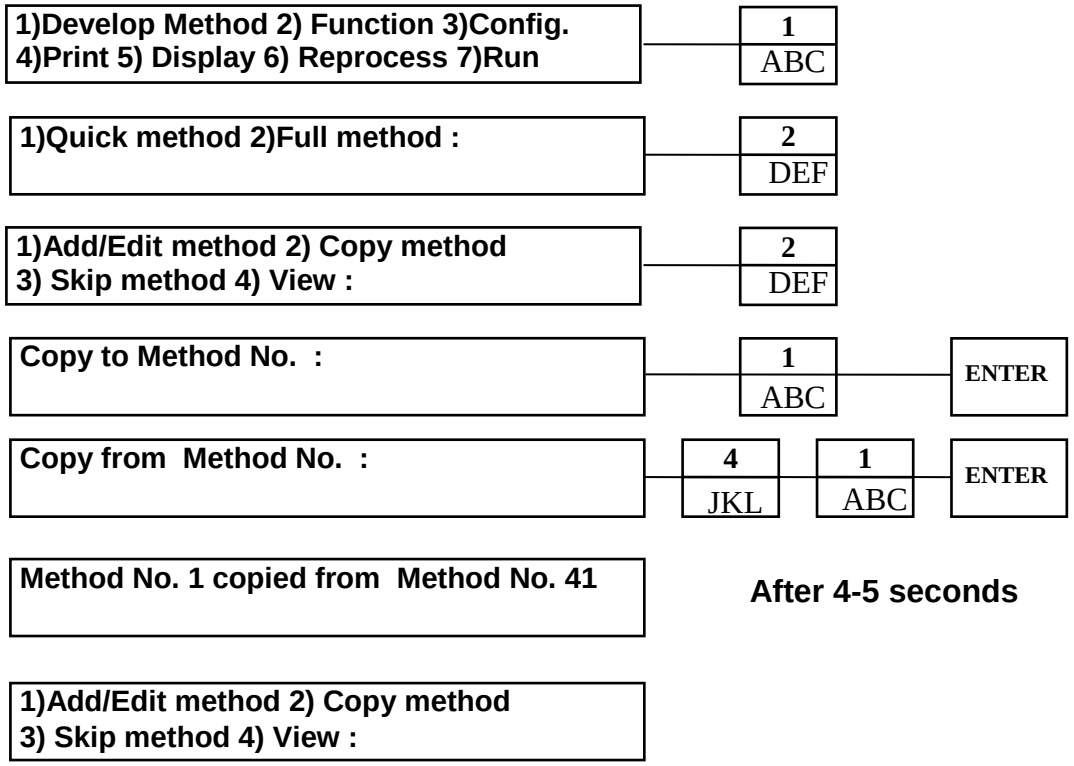
ENTER

1)Add/Edit method 2) Copy method
3) Skip method 4) View :

NOTE : 50 titration methods can be scanned independently. If the method scanned is not present or earlier entered, display shows :

Method does not exist !!

- **<u>Copying the Titration Method :</u>** Say Method No.1 has to be copied from Method No. 41. Follow the below mentioned instructions



- **Skipping the Titration Method :**

Say Method No.1 has to be skipped so memory can be made free to add new method.

a) To skip individual method :

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC

1)Quick method 2)Full method : — **2** DEF

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **3** GHI

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

1)Individual 2)All : — **1** ABC — **ENTER**

Method No. (1-50) : — **1** ABC — **ENTER**

Method No. 1 Skipped

After 4-5 seconds

1)Add/Edit method 2) Copy method
3) Skip method 4) View :

b) To skip all methods (except standard methods 41-50) :

1)Develop Method 2) Function 3)Config. : 7
4)Print 5) Display 6) Reprocess 7)Run — **1** ABC — **ENTER**

1)Quick method 2)Full method : — **2** DEF — **ENTER**

1)Add/Edit method 2) Copy method
3) Skip method 4) View : — **3** GHI

Enter User Password:

Enter ADMIN or USER Password (ref. Page. 51)

1)Individual 2)All : — **2** DEF — **ENTER**

Method No. 1-40 Skipped

After 4-5 seconds

1)Add/Edit method 2) Copy method
3) Skip method 4) View :

FUNCTION PARAMETERS

1) pH Calibration :- for pH measurement or pH titrations.

Standardization is done at 7 pH and Calibration can be done for acid and base buffer. Calibration can be performed for 3 point with 2 buffer (one on acidic side and other on basic side). If the calibration is not to be done, Press "**Escape"** key to quit.

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	2 DEF

a) Calibration of pH electrode :- Say 7 pH , 4 pH and 9pH buffer

Remove the pH electrode from its storage buffer and rinse it with distilled water.
Clean the electrode soak dry with tissue paper and put the electrode in 7 pH buffer.

1)pH Calibration 2)mV scan 3)Prime 4)Burette Change 5)Manual Titra :	1 ABC	ENTER

Enter User Password:	Enter ADMIN or USER Password (ref. Page. 51)

1)Calibrate 2)Clear Calibration	1 ABC	ENTER

Dip the electrode in 7.00 pH buffer for standardization,

Enter if stable mV (std) mV = 0.00	ENTER

System checks the Iso-potential value in ±30mV limit and display reads,

Offset : 1.2 mV Standardisation Over ...	After 4-5 seconds

Note : 1) The offset shown here is now memorized for future measurements/analysis.
2) If the offset is out of the range (more than +/- 30 mV), the display shows

Incorrect Buffer or Faulty Electrode

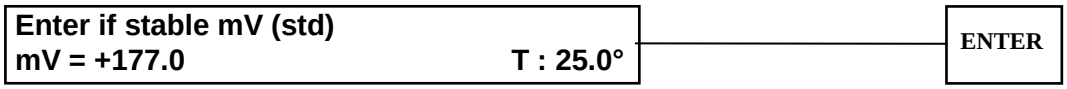
Check the buffer or change the electrode and proceed further.

To complete the calibration process use buffer of acid side first and then basic side

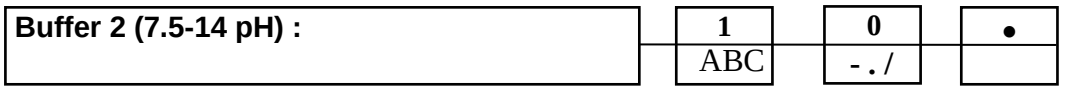
Remove the pH electrode from 7 pH buffer and rinse it with distilled water.
Sock dry the electrode with tissue paper to remove the excess DI water and put the electrode in Acidic buffer (say 4 pH).

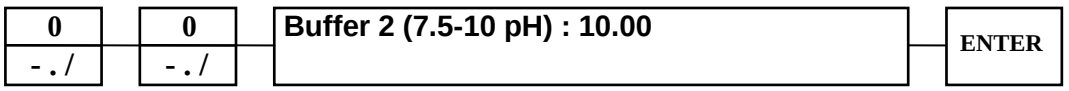
Enter pH Value (0-6.5 pH) :	4 JKL	.

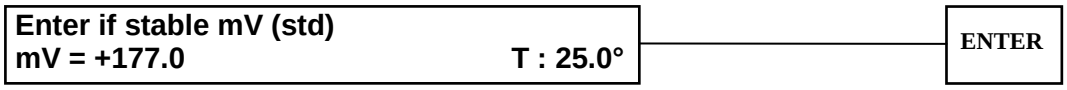
0 - . /	0 - . /	Buffer 1 (0-6.5 pH) : 4.00	ENTER

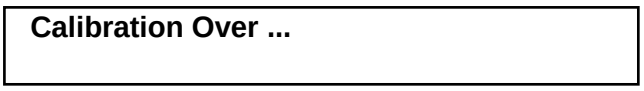


Remove the pH electrode from 4 pH buffer and rinse it thoroughly with distilled water. Sock dry the electrode with tissue paper to remove the excess DI water and put the electrode in Base buffer (say 9 pH).

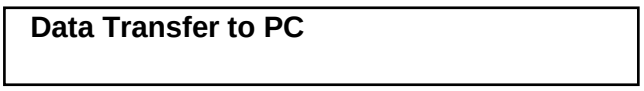








After 4-5 seconds

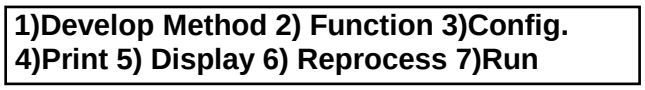


After 4-5 seconds screen returns to main menu.

The electrode parameters (offset, slope1, slope2) are calculated, automatically stored and printed in **'**Calibration report' along with Calibration date and time (if printer is connected). The slope limit provided in the instrument is 80% to 120%)

c) Resetting the Calibration data to Default Values

If instrument calibration done by buffers is not required and needs to be resetted press '2' , the system takes default values with slope (100%) and offset correction (0.00mV).

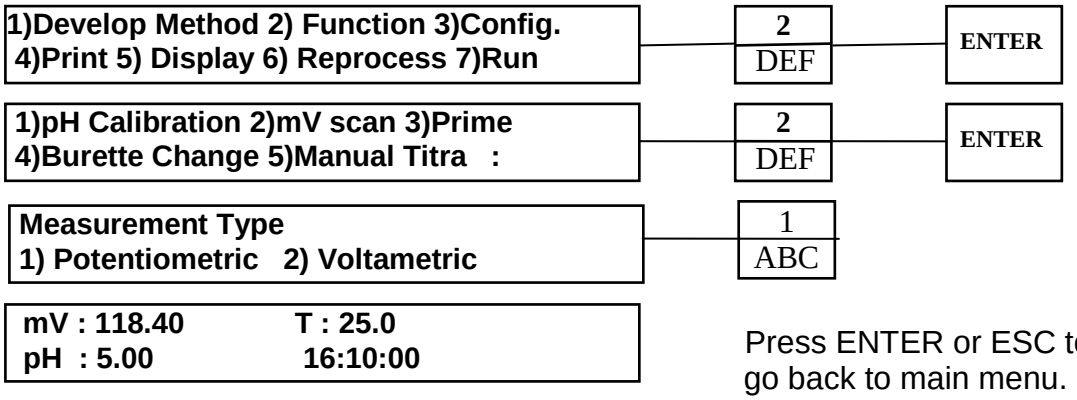


After 4-5 seconds screen returns to main menu.

' Now the instrument is ready for pH measurement use.'

2) Using instrument as pH / mV indicator (mV scan)

For pH measurement ensure that the instrument is properly calibrated before measurement.
Place the beaker containing sample whose pH is to be measured. Insert the clean and dried electrode such that it gets immersed in the sample. Now start the stirrer and proceed as following

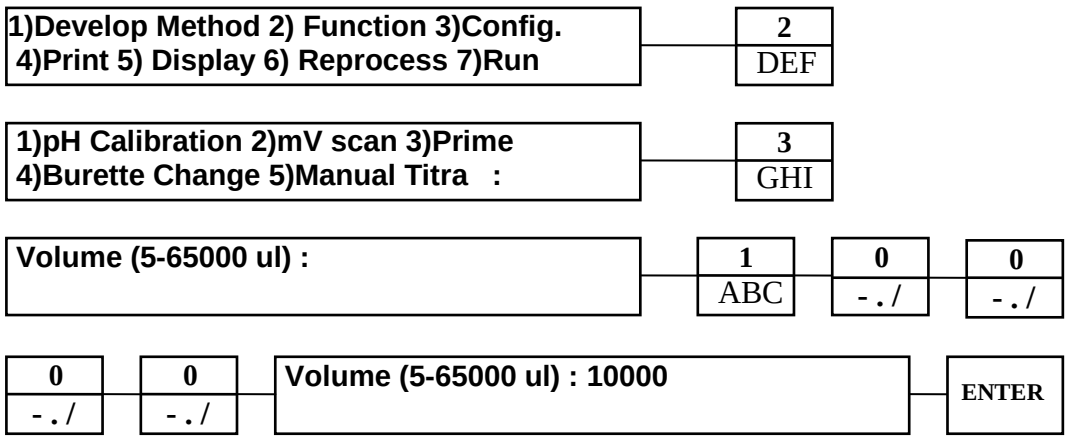


Press ENTER or ESC to go back to main menu.

3) Priming To rinse the complete liquid path by titrant and to remove air/air bubbles trapped from tubings .

In this process burette will aspire entered volume from titrant bottle and dispense it in reaction vessel or beaker. In case air bubbles in burette are noticed, tighten teflon tubing connectors. Initially PRIME function can be repeated 2 to 3 times to clean system.

The PRIME is also used for adding fixed volume as per program parameter = Primary Dose (**P.Dose)** is added.



Wait .....

After 4-5 seconds & Priming

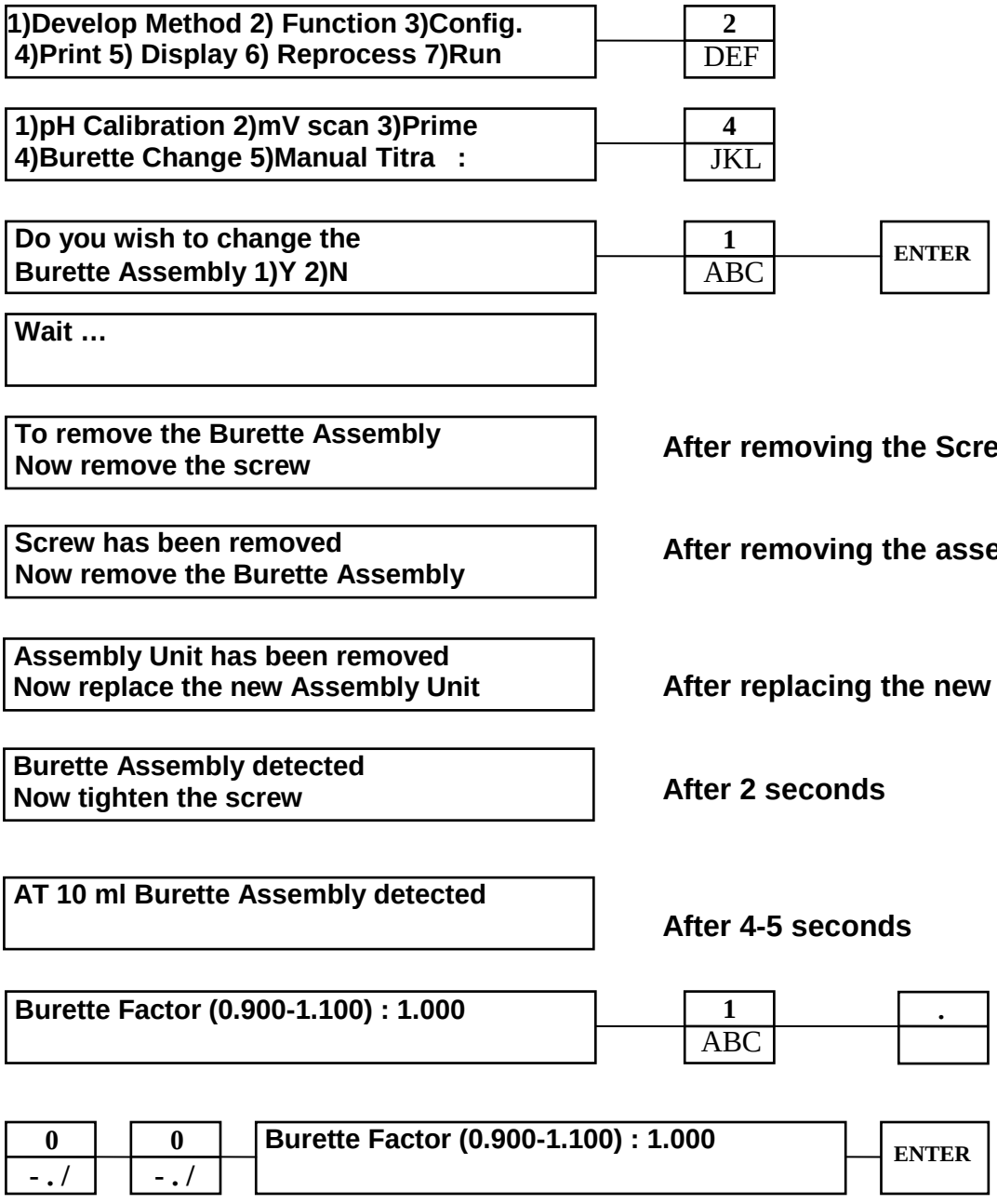
Priming Over.

Screen returns to main menu.

NOTE : Max.volume that can be primed is 65 ml. Min.volume is 1/1000th burette.

The Priming Volume should always be in multiple of 1/1000th of the burette volume.

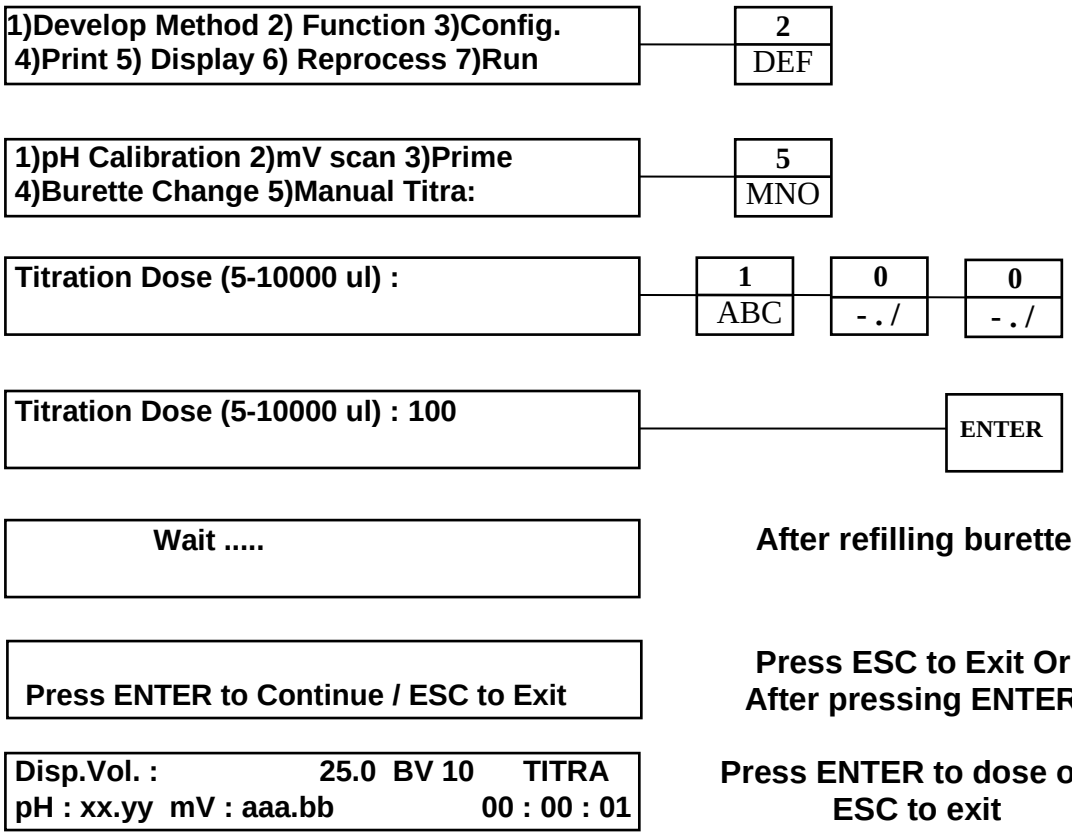
4) Replacing the burette assembly



Note: Burette Factor is for volume correction related to dispensing. The factor is multiplied to the End point/equivalence point Volume obtained for correcting the results.

The instruments automatically restarts and confirms the detection and returns to Main Menu.

5) Performing Manual Titration



pH, mV values and Volume dispensed are updated after every dose is added by pressing the “ENTER” key.
After “ESC” key is pressed, system initializes and data table report from main menu can be printed.

CONFIG PARAMETERS

1) Setting RTC (Clock)

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI
1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	1 ABC
Set Clock and Date 1)Y 2)N :	1 ABC
Min : Hour : Day : Month : Year 16 : 17 : 12 : 02 : 01	5 MNO → ENTER

After entering the values (new time, date are stored), screen returns to main menu.

2) Balance Connectivity : for automatic sample weight transfer to instrument.

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI
1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	2 DEF

a) For Manual Sample weight entry using Keys

Weight Transfer 1)Manual 2)Auto :	1 ABC

Screen returns to main menu.

b) For Sample weight entry direct from Balance (Weighing Scale) RS232

Weight Transfer 1)Manual 2)Auto :	2 DEF → ENTER

Screen returns to main menu.

Balance Setting :-
For Sartorious Balance : Set the Comm Port Properties, Baud – 1200, Data bits – 8, Parity – Odd, Stop bit – 1.

For functional details refer the titration Run Display

3) Temperature Compensation

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI
1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	3 GHI

a) For Manual Temperature compensation

Temp. Compensation 1)Manual 2)Auto :	1 ABC	ENTER

Enter the sample temperature after measuring it with external thermometer (25)

Temp.compensation 1)Manual 2)Auto Temperature (20-120) : 25	2 DEF	5 MNO	ENTER

Screen returns to main menu.

b) For Automatic Temperature compensation

Temp. Compensation 1)Manual 2)Auto :	2 DEF	ENTER

Screen returns to main menu.

4) Printer Selection

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI

1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	4 JKL

Printer 1)LX800 2)HP

To select LX800

Press | 1 ABC | → | 1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run |

5) Password Entry

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI

1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	5 MNO

1)Admin P/W 2)User P/W :	To change 1 ABC

Old Password :

New Password :

Min. 4 digit Max. 8 digit

Repeat Password :

Admin Password saved

To enter User Password

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run

→ **3** GHI

1)Clock/Date 2)Balance 3)Temp. 4)Printer
5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.

→ **5** MNO

1)Admin P/W 2)User P/W :

To change → **2** DEF

Enter Admin Password :

Set User Password :

Min. 4 digit Max. 8 digit

Repeat Password :

User Password saved

6) DATA TRANSFER

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run

→ **3** GHI

1)Clock/Date 2)Balance 3)Temp. 4)Printer
5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.

→ **6** PQR

1) Printer 2) PC

→ **1** ABC **For Data Transfer to Printer**

OR

→ **2** DEF **For Data Transfer to PC (HyperTerminal)**

After the data is printed / transferred to PC, Display returns to

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run

7) Resolution selection

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI
1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	7 STU

Result Resolution : 1)1 2)2 3)3 4)4

Enter the required result resolution, Say 4

Press 4 JKL → **1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run**

8) Titrant Temp Factor

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	3 GHI
1)Clock/Date 2)Balance 3)Temp. 4)Printer 5)PW 6)Data Xfer 7) Resol. 8) Tit. Temp. Fct.	6 PQR

Titrant Temp Factor (0.900-1.100): → 1 ABC → ENTER

This factor will get considered in calculations.

PRINT

1) Printing Report

After the titration is over, the result is stored in non-volatile memory, which can be printed for report generation. The Hardcopy of titration can be taken for : i) Result
ii) Data iii) Graph (mV v/s μl or 1st derivative of mV v/s μl) iv) Condensed Report

Care has been taken for printing in DRAFT mode. Result gives method and mode used, titration number, sample name, titrant name, run identification number and formula constants. It gives result report as end point ml with desired result units depending on the constant selection. Data gives stepwise μl reading with corresponding mV, S.No. and change in mV with each step of dosing. Graphical representation if data table is graph. User can call any one of these reports as per his requirement. Data can be transferred and stored in PC for future reference.

If the second element is 0 (i.e. this condition occurs only after resetting the system), graph cannot be printed, error message is displayed.

ERROR 2

 # If number of doses exceeds 199, during any titration run, graph cannot be printed, error message is displayed.

ERROR 2

 # If first derivative value is out of range (-32000 to 32000), during any titration run, graph cannot be printed, error message is displayed.

ERROR 3

NOTE : Auto Incremented Run number is printed on the right side of every printout. If the volume reqd. is negative (blank/back volume exceeds titration volume) the result is printed and displayed **NEGATIVE.**

- ## Printing Last Titration Result

Ensure that the calculation parameters are entered before taking hardcopy. If any of calculation parameter is altered after 1st titration run report, '(•)' is printed near Run No.

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**4** JKL

The Print function performs its operation as per configuration done in Data Xfer.
For Data Transfer mode selected to 'Printer' display shows,

1)Result 2)Data 3)Condensed 4) Statistics **5) Cal. Repo 6) Method 7) Graph**	Press	**1** ABC	To take Result Printout

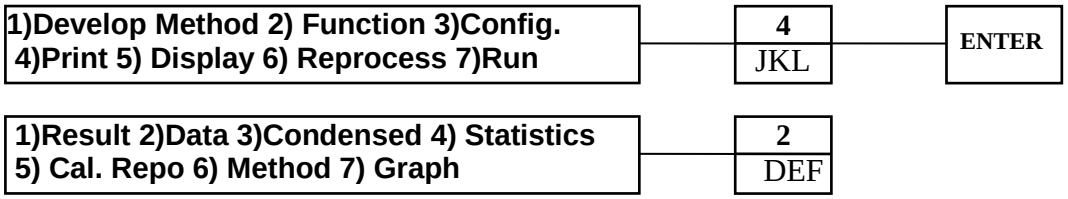
Titration Report is printed and screen returns to main menu.

For Data Transfer mode selected to 'PC' display shows,

Downloading to PC

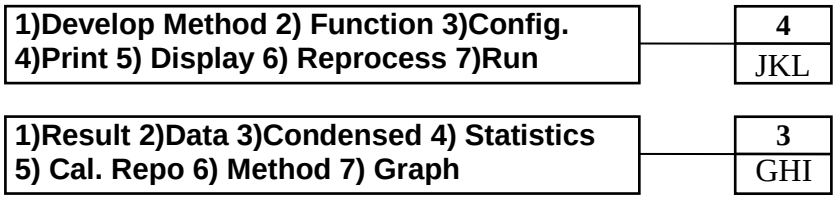
Titration Data is transferred to PC and screen returns to main menu.

- **Printing Data Table**



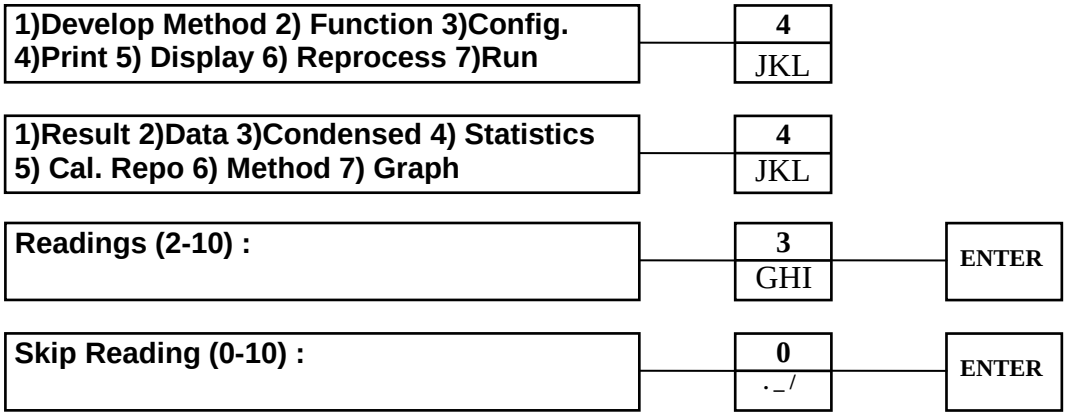
Titration Data Table is printed and screen returns to main menu.

- **Printing the Titration Analysis Condensed Report** Follow the below mentioned instructions :



Titration Analysis Condensed Report is printed and screen returns to main menu.

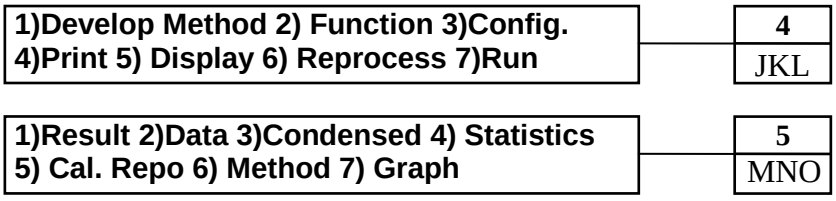
- **Printing the Titration Statistics Report :** Follow the below mentioned instructions to print statistics report for 3 readings without skipping any of them.



Titration Statistics Report is printed and screen returns to main menu.

For No data it will display

- **Printing pH Calibration Report :** Follow below mentioned instructions :



pH Calibration Report with Buffer values and Slope is printed along with the last calibration date is printed and screen returns to main menu.

- **Printing Method Parameters** Follow below mentioned instructions

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	**4** JKL	**ENTER**
1)Result 2)Data 3)Condensed 4) Statistics 5) Cal. Repo 6) Method 7) Graph	**6** PQR	**ENTER**

Parameter Report is printed along with empty methods & screen returns to main menu.

1)Individual 2)All

Select Prog. No. Say 1

Press **1** ABC — **Enter Method No. :** — **1** ABC — **ENTER**

To print all stored Prog. Parameter **2** DEF

1)Develop Method 2) Function 3)Config.
4)Print 5) Display 6) Reprocess 7)Run

If printer is not connected / Ready

Printer not ready
Want to continue? 1)Y 2)N

To continue printing, **Press** **1** ABC

- **Printing the mV v/s μl Graph Report**

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	**4** JKL	
1)Result 2)Data 3)Condensed 4) Statistics 5) Cal. Repo 6) Method 7) Graph	**7** STU	**ENTER**
1)mV/ul 2)deriv 3)time/ul 4)2nd deriv :	**1** ABC	

Titration Graph Report is printed and screen returns to main menu

Similarly other graphs can be print.

Note : Printouts of Graphs are not available if Data Transfer mode selected to PC.

DISPLAY

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**5** MNO

1) Displaying Titration Result Follow below mentioned instructions

1) Result 2) Statistics 3) Cal. Data	**1** ABC

Vol. Reqd : 0.9425 ml **Result : 0.1924 Molarity (T)**

After 4-5 sec screen

Result - 1)Print 2)Reprocess 3)Display **4)Display Stat. 5)Run Last Method :**

2) <u>Displaying the Titration Statistics Report :</u> Follow the below mentioned instructions to display statistics report for 3 readings without skipping any of them.

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**5** MNO	
1) Result 2) Statistics 3) Cal. Data	**2** DEF	
Readings (2-10) :	**3** GHI	**ENTER**
Skip Reading (0-10) :	**0** ·_/	**ENTER**

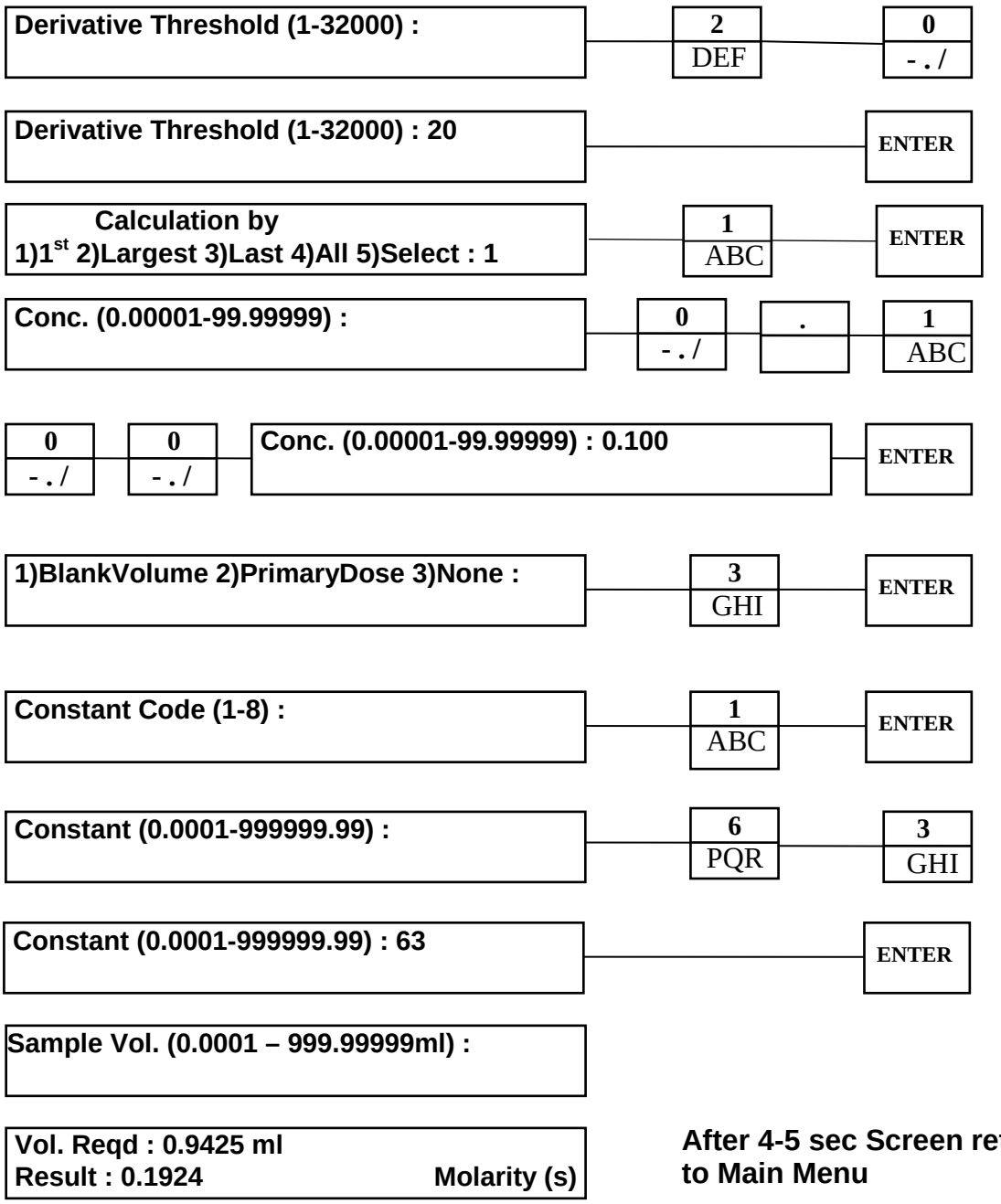
Titration Statistics are displayed and screen returns to main menu.

3) <u>Displaying the Cal. Data :</u>

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**5** MNO
1) Result 2) Statistics 3) Cal. Data	**3** DEF

Offset (mV): X	DD/MM/YY
Slope 1 : ABC%	Slope 2 : DEF%

REPROCESSING THE RESULT FOR DIFFERENT RESULT UNITS :



After 4-5 sec Screen returns to Main Menu

RUNNING METHOD : Follow below mentioned instructions :

1)Develop Method 2) Function 3)Config. 4)Print 5) Display 6) Reprocess 7)Run	7 STU

a) Run last method

1)Last Method 2)New Method :	1 ABC

If last method is quick method, Press ENTER to acknowledge :

Run Quick Method	ENTER

If last method is full method, Press ENTER to acknowledge :

Run Method 1 Press ENTER to continue / ESC to exit.	ENTER

IDENTIFICATION NO :	ENTER

Enter Identification No. [refer Appendix-A for alphanumeric entries]

Sample Volume (0.0001-99.9999 ml) :	1 ABC	ENTER

NOTE :
If constant (1-20) for the method is related to weight and balance mode is MANUAL, screen shows :

Sample Weight (0.0001-99.9999 gms) :	1 ABC	ENTER

If constant (1-20) for the method is related to weight and balance mode is AUTO, screen shows :

To Transfer Weight Press Print key of Balance

After pressing Print key of balance, screen changes to :

Weight : 0.10345 gm Press ENTER to Continue / ESC to Exit

After pressing ENTER key to acknowledge on instrument screen changes to :

Running Method No. 1

Wait ..... **00 : 00 : 18**

Method No. 1 starts with the mentioned para.

Disp.Vol. : XXX ul # RN BV 10 TITRA **25.0° mV : 00 : 00 :**

The instrument displays volume and corresponding mV after every dosing. After detecting the Titration end criteria, the exact volume required for endpoint and result is displayed :

Wait .....

Vol. Reqd : 0.9425 ml **Result : 1.0025 Molarity (T)**

After displaying the result for 4-5 seconds, the selected report (Titration Analysis or Data Table or Graph Report) printout in method are printed and the screen changes to :

Result - 1)Print 2)Reprocess 3)Display **4)Display Stat. 5)Run Last Method :**

b) Run new method

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**7** **STU**

1)Last Method 2)New Method :	**2** DEF

Method No. (1-50) :	**2** DEF	**ENTER**

Press ENTER to acknowledge and contine the run or ESC to exit to main menu.

IDENTIFICATION NO :	**ENTER**

Enter Identification No. [refer Appendix-A for alphanumeric entries]

Sample Volume (0.0001-99.9999 ml) :	**1** ABC	**ENTER**

Running Method No. 2

Wait ..... **00 : 00 : 24**

Method No. 2 starts with the mentioned para.

Disp.Vol. : XXX ul # RN BV 10 TITRA **25.0° mV : 00 : 00 : 38**

The instrument displays volume and corresponding mV after every dosing. After detecting the Titration end criteria, the exact volume required for endpoint and result is displayed :

Vol. Reqd : 0.9425 ml **Result : 1.0025 Molarity (T)**

After displaying the result for 4-5 seconds, the selected report (Titration Analysis or Data Table or Graph Report) printout in method are printed and the screen changes to :

Result - 1)Print 2)Reprocess 3)Display **4)Display Stat. 5)Run Last Method :**

c) Run last quick method

1)Develop Method 2) Function 3)Config. **4)Print 5) Display 6) Reprocess 7)Run**	**7** **STU**

1)Last Method 2)New Method :	**1** ABC

If last method is quick method, Press ENTER to acknowledge, ESC to exit :

Run Quick Method **Press ENTER to continue / ESC to exit.**	**ENTER**

1)Sample Weight 2)Sample Volume :	**2** DEF

Sample Volume (0.0001-99.9999 ml) :	**1** ABC	**ENTER**

Running Quick Method ...

Wait ..... **00 : 00 : 18**

Quick Method starts with the mentioned para.

Disp.Vol. : XXX ul # RN BV 10 TITRA + **25.0° mV : 00 : 00 :**

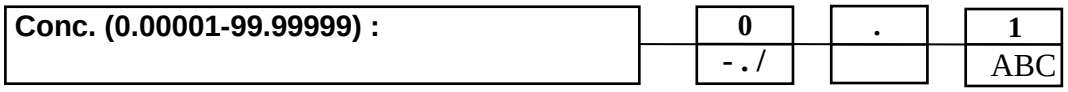
Vol. Reqd : 0.9425 ml
1)Repeat Run 2)Result 3)Print :

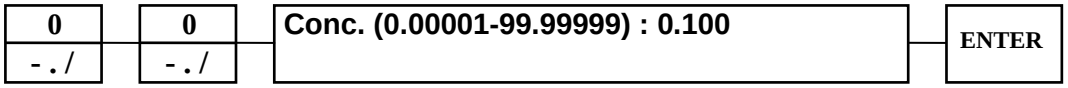
a) To repeat the same quick method with same parameters, select ‘1’ option.

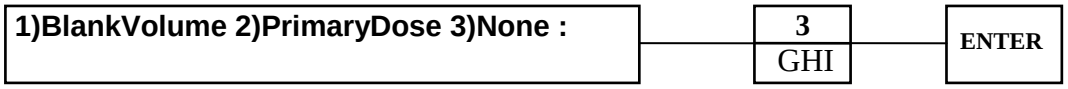
b) To print the data table report for quick method, select ‘3’ option.

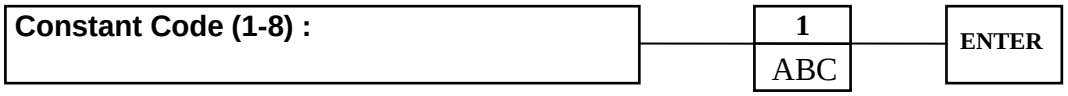
c) To get result for the quick method enter sample analysis parameters as following by selecting ‘2’ option.

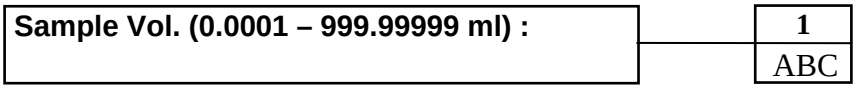
Calculation By:
1) 1st 2) Largest 3) Last 4) All 5) Select : 1











Vol.Reqd : 0.9425ml R : 0.1924 MOLARITY(S)
1)Repeat Run 2)Reprocess 3)Print :

 # If the number of doses exceeds 200 or titration run volume exceeds 99.99 ml or after 2 doses Ctrl. Lt. for pH titration exceeds sample pH, error message is displayed, the titration run is aborted and suitably printed.

ERROR 1

SECTION 4

- **Result Calculation**
- **Entering Blank Volume / Primary Dose**
- **Entering Constant Related to Sample**
- **Automatic Determining and Storing of Molarity**
- **Maintenance and Cleaning of Burette**
- **Maintenance and Cleaning of Electrode**
- **Appendix A — K**

- **Result Calculation**

a) Direct Titration :-

The result is calculated according to the following formula :

Result = M1 * EP1 * C1 / S1

where,

EP1 = Titrant consumption up to the equivalence point in ml.
C1 = Concentration of titrant in mol./lit .
M1 = Calculation constant to convert the raw result into the desired unit. Refer Pg. No. 70
S1 = Weight (or Volume) of the sample in ml / gm.

b) Direct Titration with Blank or Primary dose:-

The result is calculated according to the following formula :

Result = M1 * (EP1+E0) * C1 / S1

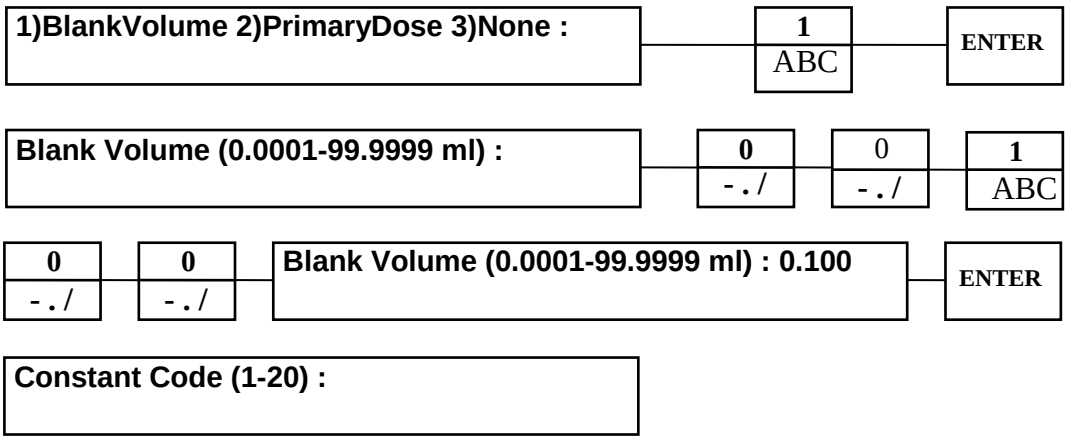
where,

EP1 = Titrant consumption up to the equivalence point in ml.
E0 = Blank volume (subtracted) OR Primary dose (added) in ml.
C1 = Concentration of titrant in mol./lit.
M1 = Calculation constant to convert the raw result into the desired unit. It can be determined in accordance with Pg. No. 69
S1 = Weight (or Volume) of the sample in ml / gm.

- **Entering Blank Volume / Primary Dose**

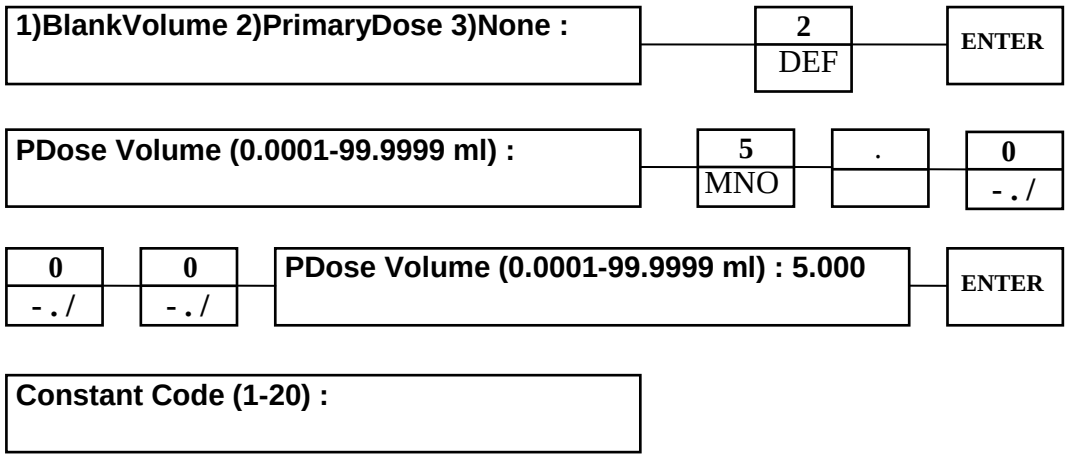
a) Blank Volume : Blank Volume is the titrant volume required for neutralizing the known amount of solvent/distilled water taken for dissolving the sample. In certain titrations a small part of titrant is consumed by the solvent before actual sample endpoint is obtained. In such cases exact result requires correction by blank volume.

The blank volume gets subtracted from titrated volume.
e.g. say the 50 ml. Solvent / distilled water got neutralized by 100 µl (0.1 ml) of titrant.



b) Primary Dose (PDose Volume) : Primary Dose is the fixed Titrant volume added in the titration beaker when i) sample volume/weight taken in large size which will require end point volume more than the burette volume and ii) to reduce the titration time.

e.g. if the titrant volume required for neutralization is about 7 ml and the installed burette assembly is of 5ml capacity, then 5ml titrant can be directly dispensed using PRIME function (refer Page 50) and the same value should be entered in P.Dose and run the titration method to determine the end-point.



NOTE : Last entered Blank Volume and Primary Dose are taken for result evaluation. Range for entering Blank Volume & Primary Dose is from 0.0001 to 99.9999 ml.

- **Entering CONSTANT related to Sample**

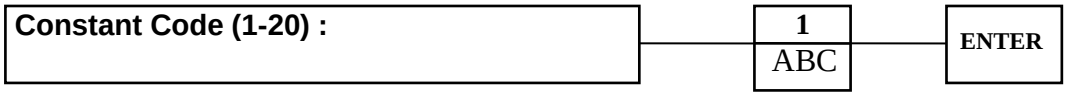
Enter Constant value by selecting appropriate code no [refer Appendix-B(Pg.75), J(Pg.86)] for automatic calculation of results in units (%, factor, molarity, ppm etc).

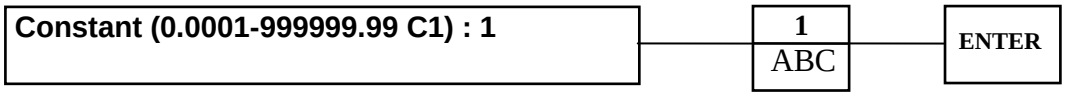
Code no 1 - 7 : are reserved and used for Liquid samples. Sample taken by volume (ml).

Code no 8 & 20 : has Constant value '1' and is to be used for End point titration when only titration end point volume is to be determined for liquid & solid samples respectively .

Code no 12-19 : are reserved and used for solid samples. Sample is taken by weight (gm).

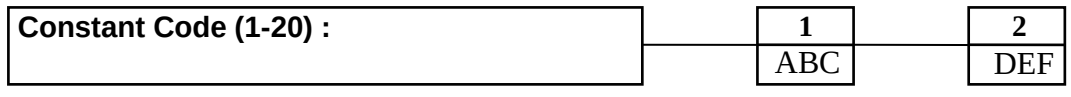
e.g. Entering Constant value - 1 in Code no. 1 for Sample Volume of 1 ml. Constant code number is displayed on the bottom corner of the screen.

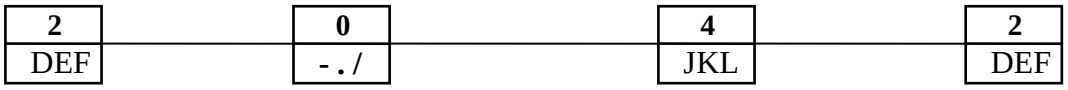


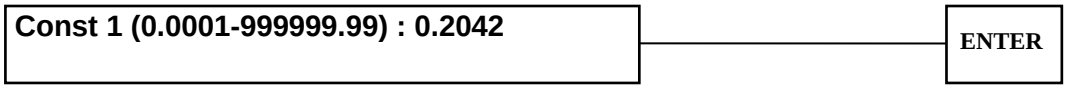


OR

e.g. Entering constant value - 0.2042 in Code no. 12 for 50.15 mg of Potassium Hydrogen Pthalate (KHP).







CONSTANT CODES

Suitable selection of constant code no. allows printout of the associated unit with result.

1) Constants (1-8) for sample taken by Volume (ml.)

CODE NO.	UNITS	FORMULA
1.	MOLARITY(S)	Z1 / Z2
2.	% VOL.	(M*Z1) / (Z2*10 *δ)
3.	MG / L	(M*1000 *Z1) / Z2
4.	PPM	(M*1000 *Z1) / (Z2*δ)
5.	MEQ / L	1000
6.	G / L	(M * Z1) / Z2
7.	MOLARITY(T)	Z1 / (C*Z2)
8.	—	1

2) Constants (12-20) for sample by weighing (mg.)

CODE NO.	UNITS	FORMULA
12.	MOLARITY(T)	(M*Z1) / (1000 *Z2)
13.	% ASSAY	(M*Z1) / (Z2*10)
14.	MG / G	(M*Z1) / Z2
15.	PPM	(M*1000*Z1) / Z2
16.	MOL / KG	Z1 / Z2
17.	ML / G	1
18.	TAN	56.1
19.	TBN	56.1
20.	—	1

Z1 : Valency of the titrant. (Mol. wt. / Eq. Wt.)
Z2 : Valency of the sample. Eq. Wt. depends upon the nature of titration reaction.
M : Molar Mass (gms.).
C : Concentration of sample (mol / L).
TAN : Milligrams of KOH required to neutralize 1 gm. of sample.
TBN : Basicity equivalent to no. of mg. of KOH per gram of sample.
δ : Density of sample

Note : Constant 8 is for getting no result unit printed for liquid sample and Constant 20 is for getting no result unit printed for solid sample.(Only volume is printed)

NOTE : Auto Incremented Run number is printed on the right side of every printout. If the volume reqd. is negative (blank/back volume exceeds titration volume) the result is printed and displayed **NEGATIVE.**

- **Determination of Molarity:**

  Standardizing 0.1 M Sodium Hydroxide (NaOH) by Potassium Hydrogen Pthalate (KHP). Enter Constant related to Standard taken volumetrically in Code 7 or enter in Code 12 if Standard is taken by weight (Refer Page 71)

  Constant Value = (M • Z1) / (1000 • Z2) = (204.22 • 1)/(1000• 1) = **0.2042** in Code 12. Z1 is valency of the titrant and Z2 is valency of sample. Also enter the weight of sample taken for analysis.

  - Select method for standardization.
  - Perform titration with Reference Standard. (Refer Run Method Page).
  - After titration Display reads the result.

Vol. Reqd : 0.9425 ml **Result : 1.0025 Molarity (T)**

Result – 1) Print 2) Reprocess 3) Display **4) Display Stat. 5) Run Last Method:**

After repeat runs take statistics report , the mean value is displayed / printed. The mean Molarity (T) can be put in the required prog. no. manually. (for manual entry of molarity see below).

NOTE : Range for entering Molarity value is from 0.0001 to 99.99999 mol./lit.

MAINTENANCE AND CLEANING OF BURETTE/SYRINGE

- Periodic cleaning of the burette, plunger adapter and tubing with suitable solvent is recommended.
- Do not dry the Non glass parts in oven.
- Never place O-rings or rubber washers in organic solvents like chloroform, carbon tetra chloride.

1. Select the Burette option in Function Menu to remove the Assembly from the titrator unit.
2. Follow the Display Screen instructions until the remove burette instruction is read on the display.
3. Clean the syringe with suitable solvent. Be careful while handling plunger teflon tip against damaging as it may result in leakage.
4. To mount it again, first locate the plunger of burette in the plunger drive arm and slide the assembly until click sound is heard with display message for type of assembly.
5. Follow the Display Screen instructions until the system is restarted with correct burette assembly display message
6. After Initialization and self-check is over perform rinsing of the entire liquid path – tubing, valve and burette.

To eliminate air bubbles from the burette, carry out at least 2-3 syringe strokes by PRIME.

MAINTENANCE AND CLEANING OF ELECTRODES

The glass electrode should be cleaned occasionally in moderately concentrated acid and kept in suitable buffer when not in use. See also the maintenance instructions packed with the electrode as set forth by the makers. The following procedure should be adopted for cleaning the glass electrode:

1) Rinse the electrode in pure water by changing water for more than 3-4 times, and wipe off water with clear filter paper or absorbent cotton.

2) If the glass electrode is extremely dirty, clean it with 0.1 N hydrochloric acid, soapsuds or detergent as necessary for a short time, and clean it thoroughly with pure water.

3) The electrode, which has been left dry for a long time, should be put in 4.00pH buffer to reach equilibrium before use.

4) If any foreign material is contained in porous frit (containing solution) of the reference electrode abnormal potential response may occur. Clean the solution area thoroughly with pure water.

5) After rinsing the electrode, clean their tips with pure water and wipe off water with clean filter paper or absorbent cotton. DO NOT RUB THE SENSING PART.

6) Now connect the electrode and observe the response of electrode as mentioned.

APPENDIX A

ALPHANUMERIC ENTRY

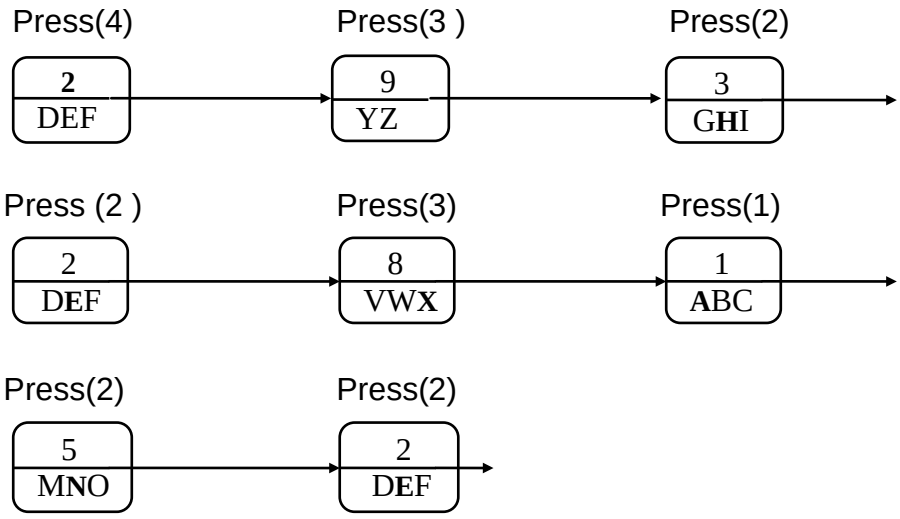
Alphanumeric entries are done to enter a Sample name, Titrant name (ENTER key) and Identification no. (RUN mode). Maximum 20 alphanumeric characters can be entered for any alphanumeric entry.

e.g. To enter Sample Name : say 2 HEXANE.

Please follow sequence of operations

SAMPLE NAME :

NOTE: Press(2) means press the button twice.



SAMPLE NAME :

Similarly enter Identification Number and Titrant Name.

APPENDIX B

ERROR CODES

# If the number of doses exceeds 200, during any titration run, titration run is aborted, error message is displayed and 'Run aborted' is printed on report.	**ERROR 1**
# If the titration run volume exceeds 99.99 ml, during any titration run, titration run is aborted, error message is displayed and 'Run aborted' is printed on report.	**ERROR 1**
# If after 2 doses Ctrl. Lt. for pH titration exceeds sample pH, error message is displayed and the titration run is aborted and 'Run aborted' is printed on report.	**ERROR 1**
# If the second element is 0 (i.e. this condition occurs only after resetting the system), graph cannot be printed, error message is displayed.	**ERROR 2**
# If number of doses exceeds 199, during any titration run, graph cannot be printed, error message is displayed.	**ERROR 2**
# If 1^{ST} deriv value is out of range (-32000 to 32000), during any run, graph cannot be printed, error message is displayed.	**ERROR 3**
# If the printer is not connected while printing data report or method parameter report, error message is displayed.	**Printer not Ready ....**
# If the method to be scanned or run is not already entered, error message is displayed.	**Method does not exist**

APPENDIX B

ERROR CODES (Contd.)

# If the electrode's offset or buffer values are not within specified range, error message is displayed.	**Incorrect Buffer or Faulty Electrode**
# If the method parameters are not suitable for running the method by changing burette, error message is displayed.	**Check Method Parameter**
# If the entered parameter is not within the specified range, error message is displayed.	**Invalid Entry**
# If the proper electrode is not connected, or BNC is open (i.e. electrode is not connected), error message is displayed.	**Connect Proper Electrode**

APPENDIX C

PRINTER DIP SWITCH SETTINGS

The printer has twelve DIP switches grouped in two groups, mounted on the back panel. DIP switch 1-1 is the switch at the far left side, and the one at the far right is DIP switch 2-4.

The following table summarizes the settings of switches for WIPRO LX - 800 printer.

Switch Number	Position
SW 1 – 1	DOWN (OFF)
SW 1 – 2	UP (ON)
SW 1 – 3	UP (ON)
SW 1 – 4	DOWN (OFF)
SW 1 – 5	DOWN (OFF)
SW 1 – 6	UP (ON)
SW 1 – 7	UP (ON)
SW 1 – 8	UP (ON)
SW 2 – 1	UP (ON)
SW 2 – 2	DOWN (OFF)
SW 2 – 3	DOWN (OFF)
SW 2 – 4	DOWN (OFF)

Note : When a change is made in DIP switch setting, turn off the power, reset the switch or switches, then turn on the power again. The printer checks and recognizes new settings only at the time power is turned on.

APPENDIX D
PARAMETER SELECTION TABLE

PARAMETER	DESCRIPTION	DEFAULT	LIMITS
Incremental Mode			
PREDISPENSE VOLUME	Initial Predispense Volume	**0**	Maximum 95000 µl.
PREDISPENSE DOSE	Dosing size after each pre-dispense interval	**OFF**	Factor of PDV & In steps of 1/1000th of burette.
PREDISPENSE INTERVAL	Time between two doses during predispense	**OFF**	0 to 999 sec in step of 1 sec
STIRTIME	Time between predispense end and actual run start	**'5'**	0 to 999 sec in step of 1 sec
INITIAL DOSE	Dosing size after each interval	**OFF**	1/1000th of burette to 1/5th of burette in steps of 1/1000th of burette.
INTERVAL	Time between two doses	**OFF**	1 to 999 sec in step of 1 sec
VOLUME LIMIT	Maximum volume of titration	**'10000'**	> than initial dose and > than predispense volume Maximum 99.999 ml
END CRIT.	By VolLimit or EP	**'EP'**	9 EP or VL max.
Equilibrium Mode			
PREDISPENSE VOLUME	Initial Predispense Volume	**0**	Maximum 95000 µl.
PREDISPENSE DOSE	Dosing size after each pre-dispense interval	**OFF**	Factor of PDV & In steps of 1/1000th of burette.
PREDISPENSE INTERVAL	Time between two doses during predispense	**OFF**	0 to 999 sec in step of 1 sec
STIRTIME	Time between predispense end and actual run start	**'5'**	0 to 999 sec in step of 1 sec
INITIAL DOSE	Dosing size after each interval	**OFF**	1/1000th of burette to 1/5th of burette in steps of 1/1000th of burette.
EQ.FACTOR	Speed of titration	**'5'**	'1' to '99' in steps of 1
VOLUME LIMIT	Maximum volume of titration	**'10000'**	> than initial dose and > than predispense volume Maximum 99.999 ml.
END CRIT.	By VolLimit or EP	**'EP'**	9 EP or VL max.

APPENDIX D

PARAMETER SELECTION TABLE (Contd..)

PARAMETER	DESCRIPTION	DEFAULT	LIMITS
Cutoff pH Mode			
PREDISPENSE VOLUME	Initial Predispense Volume	**0**	Maximum 95000 µl.
PREDISPENSE DOSE	Dosing size after each pre-dispense interval	**OFF**	Factor of PDV & In steps of 1/1000th of burette.
PREDISPENSEINTERVAL	Time between two doses during predispense	**OFF**	0 to 999 sec in step of 1 sec
STIRTIME	Time between predispense end and actual run start	**'5'**	0 to 999 sec in step of 1 sec
INITIAL DOSE	Dosing size after each interval	**OFF**	1/1000th of burette to 1/5th of burette in steps of 1/1000th of burette.
INTERVAL	Time between two doses	**OFF**	1 to 999 sec in step of 1 sec
VOLUME LIMIT	Maximum volume of titration	**'10000'**	> than initial dose and > than predispense volume Maximum 99.999 ml.
END CRIT.	By VolLimit or EP	**'EP'**	9 EP or VL max.
CTRL. LIMIT	pH at which dose control starts	**OFF**	0-14 pH in steps of 0.01
END LIMIT	pH at which titration stops	**OFF**	0-14 pH in steps of 0.01
pH stat			
INITIAL DOSE	Dosing size after each interval	**OFF**	1/1000th of burette to 1/5th of burette in steps of 1/1000th of burette.
EQ.FACTOR	Speed of titration	**'5'**	'1' to '99' in steps of 1
VOLUME LIMIT	Maximum volume of titration	**'10000'**	> than initial dose and > than predispense volume Maximum 99.999 ml.
END CRIT.	By VolLimit or EP	**'EP'**	9 EP or VL max.
CTRL. LIMIT	pH at which dose control starts	**OFF**	0-14 pH in steps of 0.01
END LIMIT	pH at which titration stops	**OFF**	0-14 pH in steps of 0.01
RUN TIME	Maintaining pH within control band	**OFF**	1-9999 sec in steps of 1 sec.

APPENDIX E

TITRATION TYPE

TYPE 1 : **ACID-BASE** In acid-base titration, an acid is determined by titration with a base, or vice-versa. The essential reaction is between H^+ and OH^-, giving water. The pH at the end-point depends on the dissociation constants of the reactants and products. It includes strong acid - strong base (e.g. HCl - NaOH), weak acid - weak base (e.g. KHP-Na_2CO_3), strong acid-weak base, weak acid-strong base and polybasic titrations.

TYPE 2 : **NON-AQUEOUS** This includes the weak acids or bases which give more satisfactory end-points when titrations are carried out in non-aqueous media (organic solvents) than aqueous solutions. Because many of the solvents used are aggressive, volatile and obnoxious, non-aqueous titrations are carried out in closed environment. Compounds that may be determined by nonaqueous titrimetry include amines, amino acids, phenols, carbonyl compounds by oxidation and Schiff's bases.

TYPE 3 : **REDOX** In these titrations a reducing agent is titrated with an oxidising agent and vice-versa. The common oxidising titrants are potassium permagnate ($KMnO_4$), potassium-di-chromate ($K_2Cr_20_7$), iodine (I_2), potassium iodide (KIO_3), potassium bromate ($KBrO_3$) and sodium hypochlorite (NaClO). The most important reducing agents are ferrous ammonium sulphate ($(NH_4)_2Fe(SO_4)_2.6H_20$), sodium thiosulphate ($Na_2S_2O_3.H_2O$) and arsenic oxide(As_2O_3).

TYPE 4 : **PRECIPITATION** These are titrations in which the analyte and titrant react to form a precipitate. The only common titrant used is silver nitrate (argentimetric titrations) and its use is mainly restricted to the determination of chloride, bromide, iodide, cyanide and thiocyanate, which precipitates with silver ion.

TYPE 5 : **COMPLEXIMETRIC/EDTA** Complexiметric titrations are used mainly to determine metal ions by use of complex forming reactions. In practise the titrants are compounds having iminodiacetic acid functional group, such as EDTA (ethylenediamine tetraacetic acid).

TYPE 6 : **BACK TITRATION** : When the titration time is long and typical samples which do not react directly with titrant, i.e. need a intermediate are done by this method.

TYPE 7 : **PHOTOMETRIC TITRATION** : Using Phototrodes this titration can be performed using optical principles.

TYPE 8 : **THERMOMETRIC TITRATION** : Using temperature sensing principle this titration can be performed.

APPENDIX F

TITRATION MODES

MODE 1: **INCREMENTAL** In this mode of titration fixed dose can be given at fixed interval of time up to the number of selected end point. This is used for determining sequential endpoints in multi-component sample. The titration is performable up to maximum 9 endpoints evaluation. The titration is stopped when volume limit is reached or number of number of EP set in method is reached.

MODE 2: **EQUILIBRIUM** This is the most convenient and widely used mode. Dose as well as time gets adjusted depending on the trend of reaction and the rate of change of mV. The maximum dose during titration is the dose entered. Large mV change increases the time interval between the doses and the dose size reduces rapidly to minimum towards the end point. For slow reactions equilibrium factor should be raised. A smaller end dose is preferred for higher reproducibility and accuracy. This mode minimizes titration time while maintaining high precision. The data of titration curve are checked for the presence of an equivalence point. If an equivalence point is found and EP cutoff is selected the titration is stopped and the result is calculated. Only equivalence points with the first derivative greater than threshold are detected. Maximum 9 endpoints can be evaluated.

MODE 3: **CUT-OFF BY pH** This is the same as equilibrium titration, but is based on pH values. In this mode selection of control and end limit is important, because it decides when the dose control begins during titration and up to what pH value the titration should be performed.

MODE 4: **pH STAT** This is same as Cutoff by pH titration, since after attaining the set control pH, it starts giving controlled doses at regular intervals to maintain the pH for defined run time (Stat Run Time). The titration ends when set run time is reached. Volume consumed at particular time during the run can be calculated and used for result evaluation.

APPENDIX G

METHOD PARAMETERS

a) **INITIAL DOSE (μL)** :
This is dispense volume of titrant in μl. This volume gets added periodically during sample run. Dose selection should be such that significant change of mV value occurs after addition of every dose. Minimum dose that can be set is 5 μl. 1/5th of the burette volume is the maximum dose that can be entered in all modes of titration. The data storage can accommodate information for maximum 99 dosed increments; equivalence points can therefore only be recognized and evaluated if they lie within 99 increments.

b) **EQUILIBRIUM FACTOR** :
This factor changes the titration time for equilibrium and cutoff titration. The equilibrium titration time increases with increased factor value. Default value '2' is suitable for most of the titrations. However one can enter these values from 1-99 depending upon nature of reaction etc. for Equilibrium and Cut-off modes. For slow/weaker reactions increase the equilibrium factor value suitably.

c) **INTERVAL (sec)** :
This is the delay time between dose for Incremental end point titrations (1-999 sec). It is important for following reasons:
1) To take care of Response time of the electrode.
2) To ensure that dispensed reagent is sufficiently mixed before dispensing next dose.

d) **STAT INTERVAL (sec)** :
This is the time between recordings of the pH values during pH stat run. Start Interval for pH stat titration can be varied from 1 to 99 sec.

e) **CONTROL LIMIT (pH)** :
This is pH value at which the dose control occurs in Cut-off by pH titration. This should be chosen such that at least 5-6 doses are given before the end point detection. Control limit ranges from 0-14 pH for cutoff mode.

f) **END LIMIT (pH)** :
This is the pH value up to which cutoff by pH titration should be performed. If the end point is detected before the end limit, titration gets over not by the end limit but by the endpoint detection. End limit ranges from 0-14 pH for cutoff mode.

APPENDIX H

SAMPLE ANALYSIS PARAMETERS

a) **PRE-DISPENSE CRITERION (mV)** :
This is the mV value difference after which predispense will automatically stop and programmed doses will be given (0-1000). The value '0' allows the use of Pre dispense Volume function in which programmed doses are given at fixed minimum time of the system.

b) **PREDISPENSE VOLUME (μL)** :
In general, small predispensing is advisable since the electrode potential at the start of the titration changes drastically or is poorly defined. Predispense volume addition start the reaction without over-shooting equilibrium point and saving time. Pre-dispense should be such that at least 4-5 doses are given before endpoint. Max. predispense volume is 32 ml.

c) **PREDISPENSE DOSE (μL)** :
This is dispense volume of titrant in μl. This volume gets added periodically during predispense at the set predispense interval. Predispense dose should be multiple of 1/1000 th of burette and should add upto predispense volume. Burette volume is the maximum predispense dose that can be entered.

d) **PREDISPENSE INTERVAL (sec)**:
This is the delay time between predispense doses given in predispensing for all modes of titrations (0-999 sec).

e) **THRESHOLD** :
The threshold is principally used to prevent relatively small disturbances in the curve being mistaken for an equivalence point. The first derivative must be larger than threshold to ensure recognition of equivalence point. Default value '50' is suitable for most of the titrations. However one can enter these values from 1-32000 depending upon nature of titration for all modes. Additionally threshold can also be used to bypass smaller peaks.

f) **STIR TIME (sec)** :
Before measurement of the initial potential and start of the titration, a stirring time can be selected which allows homogenization and dissolution of the sample. Stir time ranges from 0-999 sec.

g) **VOLUME LIMIT (μL)** :
This is the volume up to, which the titration should be done, even if end point is reached and is applicable for all titration. Max. volume limit is 65 ml.

h) **MINIMUM CONTROL DOSE (μL)** :
This is the minimum dose that can be given at endpoint for equilibrium and cutoff by pH titration mode. Values enterable from 5-500μL

APPENDIX H

SAMPLE ANALYSIS PARAMETERS (contd.)

i) **STAT RUN TIME (sec)** :
This is the time for which pH-Stat titration should be continued after attaining set pH value. Run time can be entered for the range of 1-9999 sec.

j) **VTIME 1 (sec)** :
This is the time at which one wants to calculate the volume consumed during pH stat titrations. VTime 1 can be entered in the range of 1-9999 sec. The value of VTime 1 should be multiple of stat interval time.

k) **VTIME 2 (sec)** :
This is the time at which one wants to calculate the volume consumed during pH stat titrations. Vtime 2 can be entered in the range of 1-9999 sec. The value should be higher than VTime 1 and should be multiple of stat interval time.

l) **FACTOR** :
This is a factor used for calculation of result (enzymatic activity) for pH stat titration. The value can be entered in the range 0.0001 to 10000.

m) **BUFFER VALUE (mV)** :
This is the buffer value entered for Method 38 and 40 i.e. for ASTM D664 & D4739 respectively. The endpoint can be calculated for the entered buffer value, the titration ends at 100 mV above the set buffer value. Value can be entered in the range 1-3200 mV.

n) **BUFFER SIGN** :
This is the polarity for the buffer value entered for Method 38 and 40 i.e. for ASTM D664 & D4739 respectively as mentioned above. The endpoint can be calculated for the entered buffer value, the titration ends at 100 mV above the set buffer value. + or - sign can be assigned to the entered buffer value.

o) **TITRATION TREND** :
This is the decided by the titration data table. If mV rises with every dose given i.e. (more positive with every dosing or negative to positive with every dosing) the trend is + (positive). If mV falls or drops with every dose given i.e. (more negative with every dosing or positive to negative with every dosing) the trend is - (negative). This is again related to ASTM D664 & D4739 i.e. method 38 & 40 run.

APPENDIX I

CALCULATION PARAMETERS

a) **CONCENTRATION** :
This is the molarity of titrant to be considered for result calculation. For standardization of titrant, before run the value entered is 1 and then evaluated value gets transferred in respective code. Concentration value ranges from 0.0001-99.9999 mol/lit.

b) **PRIMARY DOSE** :
This is volume in ml, which gets added in final volume required for titration. Prime the required volume, enter the prime volume as primary dose before running the required program. After running the program primary dose entered will get added to total volume. Primary dose ranges from 0.0001-99.9999 ml.

c) **BLANK VOLUME** :
This is the volume in ml, which gets subtracted in final volume required for titration. This is determination of titrant consumed for neutralization by equivalent volume of solvent to be used along with the sample. Blank volume ranges from 0.0001-99.9999 ml.

d) **CONSTANT** :
This is the constant related to the sample to be entered for result calculation. Constant code 1-10 are for liquid samples and 11-20 are for solid samples. Constant value ranges from 0.0001-999999.99.

e) **SAMPLE VOLUME** :
The amount of sample taken for run to be considered for result calculation for liquid samples. Sample volume ranges from 0.0001-99.9999 ml.

f) **SAMPLE WEIGHT** :
The amount of sample taken for run to be considered for result calculation for solid samples. Sample weight ranges from 0.0001-99.9999 gms.

g) **VOLUME FACTOR** :
This is factor which gets multiplied to the blank volume entered for back titration. It is used for calculation purpose. Volume factor can be entered in the range 0.1000-5.0000.

APPENDIX J

Standard Methods (41-50) :

Method 41 : Standardizing 0.1 M NaOH against KHP

METHOD NO	:	41	TITRANT CONC	:	0.1 M (appx.)
SAMPLE NAME	:	KHP	TITRANT NAME	:	NAOH
MEASURE. TYPE	:	Potentiometric	SAMPLE WEIGHT	:	100 mg (apx)
TITRATION TYPE	:	Acid-Base Aq.	TITRATION MODE	:	Equilibrium
PRE.DISP.CRIT	:	0 mV	PRE.DISP.VOLUME	:	3600 µl
PRE.DISP.DOSE	:	200 µl	PRE.DISP.INTV.	:	2 sec
STIR TIME	:	30 sec	INITIAL DOSE	:	100 µl
EQ. FACTOR	:	6			
VOLUME LIMIT	:	8000 µl	END CRITERIA	:	EndPoint (1)
THRESHOLD	:	300	MIN. CONTROL DOSE	:	10 µl
MOLARITY (mol/lit.)	:	1.0000			
BV/PD/NONE	:	None			
CONSTANT	:	0.2042	CONSTANT CODE	:	12
REPORT	:	None			

Method 42 : Standardizing 0.1 M HCl against

METHOD NO	:	42	TITRANT CONC	:	0.1 M (appx.)
SAMPLE NAME	:	Na2CO3	TITRANT NAME	:	HCL
MEASURE. TYPE	:	Potentiometric	SAMPLE WEIGHT	:	35-40mg (ap)
TITRATION TYPE	:	Acid-Base Aq.	TITRATION MODE	:	Equilibrium
PRE.DISP.CRIT	:	0 mV	PRE.DISP.VOLUME	:	5000 µl
PRE.DISP.DOSE	:	200 µl	PRE.DISP.INTV.	:	1 sec
STIR TIME	:	30 sec			
INITIAL DOSE	:	100 µl	EQ. FACTOR	:	6
VOLUME LIMIT	:	10000 µl	END CRITERIA	:	EndPoint (1)
THRESHOLD	:	100	MIN. CONTROL DOSE	:	5 µl
MOLARITY (mol/lit.)	:	1.0000			
BV/PD/NONE	:	None			
CONSTANT	:	0.0530	CONSTANT CODE	:	12
REPORT	:	None			

APPENDIX J

Standard Methods (41-50) contd ... :

Method 43 : Standardizing 0.1 M Alc. KOH against KHP

METHOD NO	:	43	TITRANT CONC	:	0.1 M (appx.)
SAMPLE NAME	:	KHP	TITRANT NAME	:	Alc. KOH
MEASURE. TYPE	:	Potentiometric	SAMPLE WEIGHT	:	100 mg (apx)
TITRATION TYPE	:	Acid-Base Aq.	TITRATION MODE	:	Equilibrium
PRE.DISP.CRIT	:	0 mV	PRE.DISP.VOLUME	:	3600 µl
PRE.DISP.DOSE	:	200 µl	PRE.DISP.INTV	:	1 sec
STIR TIME	:	30 sec			
INITIAL DOSE	:	100 µl	EQ. FACTOR	:	6
VOLUME LIMIT	:	8000 µl	END CRITERIA	:	EndPoint (1)
THRESHOLD	:	100	MIN. CONTROL DOSE	:	10 µl
MOLARITY (mol/lit.)	:	1.0000			
BV/PD/NONE	:	None			
CONSTANT	:	0.2042	CONSTANT CODE	:	12
REPORT	:	None			

Method 44 : Standardizing 0.1 M HClO4 against KHP

METHOD NO	:	44	TITRANT CONC	:	0.1 M (appx.)
SAMPLE NAME	:	KHP	TITRANT NAME	:	HCLO4
MEASURE. TYPE	:	Potentiometric	SAMPLE WEIGHT	:	100 mg (app)
TITRATION TYPE	:	Non Aqueous	TITRATION MODE	:	Equilibrium
PRE.DISP.CRIT	:	0 mV	PRE.DISP.VOLUME	:	3600 µl
PRE.DISP.DOSE	:	200 µl	PRE.DISP.INTV.	:	2 sec
STIR TIME	:	30 sec			
INITIAL DOSE	:	100 µl	EQ. FACTOR	:	6
VOLUME LIMIT	:	8000 µl	END CRITERIA	:	EndPoint (1)
THRESHOLD	:	300	MIN. CONTROL DOSE	:	10 µl
MOLARITY (mol/lit.)	:	1.0000			
BV/PD/NONE	:	None			
CONSTANT	:	0.2042	CONSTANT CODE	:	12
REPORT	:	None			

Standard Methods (41-50) contd .... :

Method 45 : Standardizing 0.1 M NaNO2 against Sulphanilic Acid

METHOD NO	: 45	TITRANT CONC	: 0.1 M (appx.)
SAMPLE NAME	: SULPHANILIC ACID	TITRANT NAME	: NANO2
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 100 mg (apx)
TITRATION TYPE	: Redox	TITRATION MODE	: Equilibrium
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 4000 µl
PRE.DISP.DOSE	: 200 µl	PRE.DISP.INTV.	: 5 sec
STIR TIME	: 30 sec		
INITIAL DOSE	: 100 µl	EQ. FACTOR	: 10
VOLUME LIMIT	: 7000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 300	MIN. CONTROL DOSE	: 10 µl
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.1732	CONSTANT CODE	: 12
REPORT	: None		

Method 46 : Standardizing 0.02 M KMnO4 against Na Oxalate

METHOD NO	: 46	TITRANT CONC	: 0.02M(appx.)
SAMPLE NAME	: NA OXALATE	TITRANT NAME	: KMNO4
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 45-50mg (ap)
TITRATION TYPE	: Redox	TITRATION MODE	: Equilibrium
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 6000 µl
PRE.DISP.DOSE	: 100 µl	PRE.DISP.INTV.	: 5 sec
STIR TIME	: 10 sec		
INITIAL DOSE	: 100 µl	EQ. FACTOR	: 15
VOLUME LIMIT	: 10000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 500	MIN. CONTROL DOSE	: 10 µl
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.3350	CONSTANT CODE	: 12
REPORT	: None		

APPENDIX J

Standard Methods (41-50) contd ... :

Method 47 : Standardizing 0.1 M Na2S2O3 against KIO3

METHOD NO	: 47	TITRANT CONC	: 0.1 M (appx.)
SAMPLE NAME	: KIO3	TITRANT NAME	: NA2S2O3
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 25-30 mg (ap)
TITRATION TYPE	: Redox	TITRATION MODE	: Equilibrium
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 7000 µl
PRE.DISP.DOSE	: 200 µl	PRE.DISP.INTV.	: 1 sec
STIR TIME	: 30 sec		
INITIAL DOSE	: 100 µl	EQ. FACTOR	: 5
VOLUME LIMIT	: 10000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 300	MIN. CONTROL DOSE	: 5 µl
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.0356	CONSTANT CODE	: 12
REPORT	: None		

Method 48 : Standardizing 0.02 M Hg(NO3)2 against NaCl

METHOD NO	: 48	TITRANT CONC	: 0.02M(appx.)
SAMPLE NAME	: NACL	TITRANT NAME	: HG(NO3)2
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 15mg (ap)
TITRATION TYPE	: Redox	TITRATION MODE	: Incremental
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 5000 µl
PRE.DISP.DOSE	: 500 µl	PRE.DISP.INTV.	: 2 sec
STIR TIME	: 30 sec		
INITIAL DOSE	: 50 µl	INTERVAL	: 5 sec
VOLUME LIMIT	: 8000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 10		
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.1167	CONSTANT CODE	: 12
REPORT	: None		

APPENDIX J

Standard Methods (41-50) contd ..... :

Method 49 : Standardizing 0.1 M AgNO3 against NaCl

METHOD NO	: 49	TITRANT CONC	: 0.1 M (appx.)
SAMPLE NAME	: NACL	TITRANT NAME	: AGNO3
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 40 mg (apx)
TITRATION TYPE	: Precipitation	TITRATION MODE	: Equilibrium
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 5000 µl
PRE.DISP.DOSE	: 100 µl	PRE.DISP.INTV.	: 1 sec
STIR TIME	: 30 sec		
INITIAL DOSE	: 100 µl	EQ. FACTOR	: 6
VOLUME LIMIT	: 10000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 100	MIN. CONTROL DOSE	: 10 µl
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.0584	CONSTANT CODE	: 12
REPORT	: None		

Method 50 : Standardizing 0.1 M EDTA against ZnSO4

METHOD NO	: 50	TITRANT CONC	: 0.1 M (appx.)
SAMPLE NAME	: ZNSO4	TITRANT NAME	: EDTA
MEASURE. TYPE	: Potentiometric	SAMPLE WEIGHT	: 100 mg (app)
TITRATION TYPE	: Comp./EDTA	TITRATION MODE	: Equilibrium
PRE.DISP.CRIT	: 0 mV	PRE.DISP.VOLUME	: 0 µl
PRE.DISP.DOSE	: 0 µl	PRE.DISP.INTV.	: 0 sec
STIR TIME	: 0 sec		
INITIAL DOSE	: 200 µl	EQ. FACTOR	: 5
VOLUME LIMIT	: 5000 µl	END CRITERIA	: EndPoint (1)
THRESHOLD	: 10	MIN. CONTROL DOSE	: 10 µl
MOLARITY (mol/lit.)	: 1.0000		
BV/PD/NONE	: None		
CONSTANT	: 0.2875	CONSTANT CODE	: 12
REPORT	: None		

APPENDIX K

K905XX – Data Downloading Capability to Personal Computer

Installation/setup Instructions:

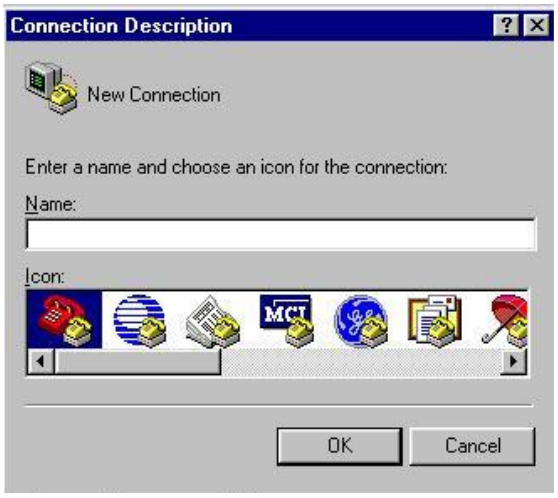
The instrument software has been incorporated with RS232C protocol interface for transferring the printable data to PC having standard data terminal software Windows - **Hyper terminal**.

Features of PC data transfer,

1. Data transfer selectivity in function menu : Printer or Personal computer.
2. All reports - a) Result Report b) Data c) Condensed Report d) Statistical Data e) Calibration Report of pH f) Methods which are available for printing will be transfer to PC (needs Hyper terminal active in the PC).
3. The data is captured as ASCII character via Hyper terminal software and then can be saved by the user as MS word doc or text file by Note Pad with suitable name by using Cut Paste utility.

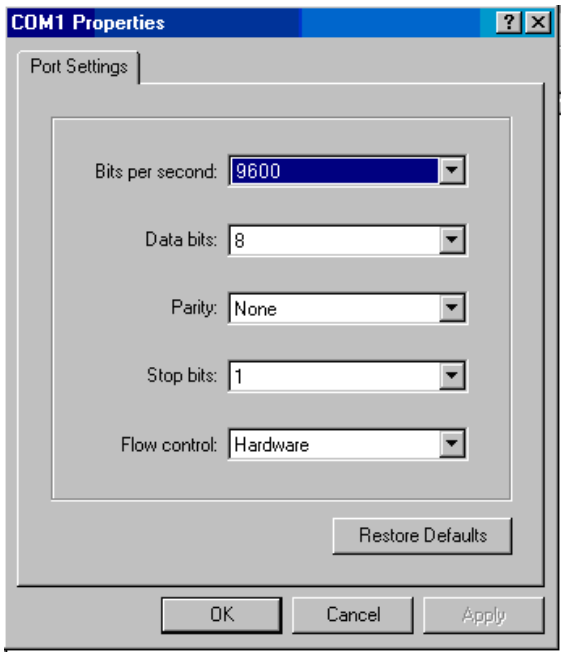
COM Port Configuration/Settings:-

Setting HYPERACCESS NEW CONNECTION and COM port selection:-

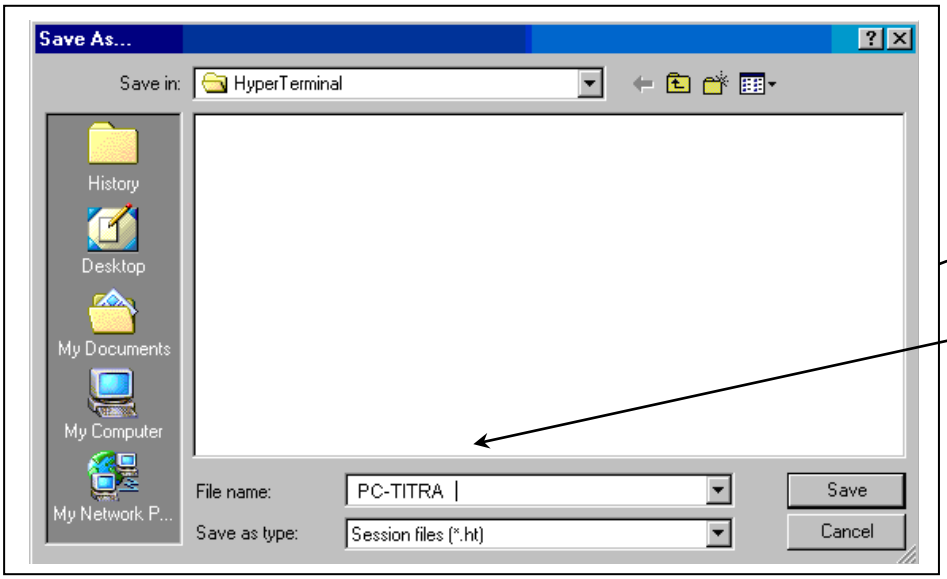


If the COM port chosen is not free or being used by another application a pop up screen will be displayed with message “Unable to Open COM”.

When a free Com port is selected The COM port Properties Pop up will be displayed on the screen.

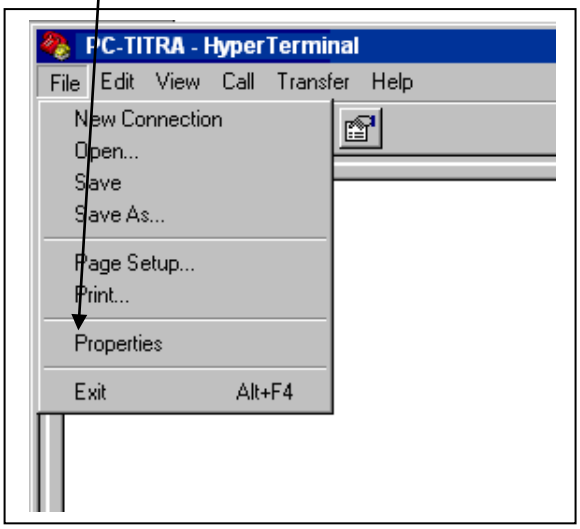


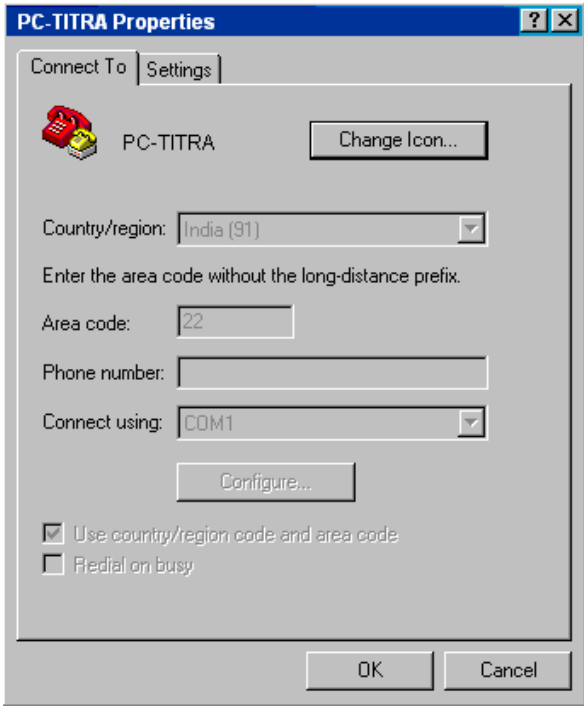
Set the Comm Port Properties, Baud-9600, Data bits 8, Parity none, Stop bit 1, Flow Control- None or Xon/Xoff.

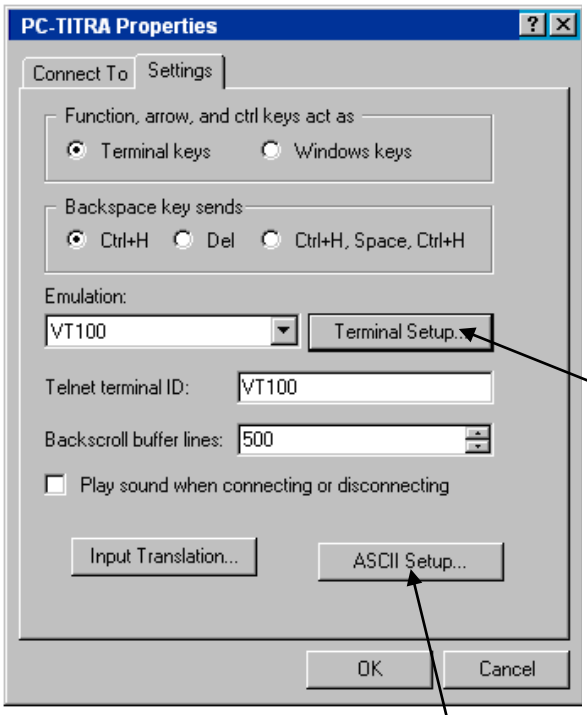


➢ Once the setting are done Save the Session so as the Session file is generated and can be run again to download the data from **PC-TITRA.**

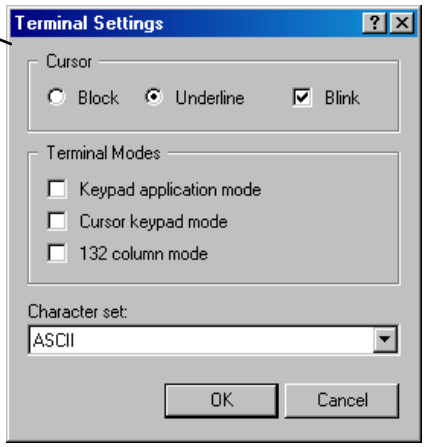
Now From the terminal window FILE menu select the Properties tool to set the Terminal Emulation mode and Data Line
Once You click on the Properties menu - Properties window will appear.





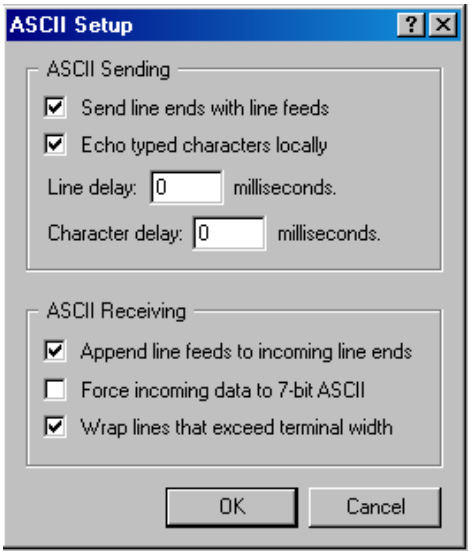


Select Settings option to set the Emulation mode and function setting.
Function:- Arrow & Ctrl Key as Terminal key.
Backspace key sends: Ctrl+H
Emulation :- VT100
Telnet id:- VT100.
Terminal Setup:- Character Set = ASCII.



Now Setup the ASCII parameters

Click the ASCII setup icon to enter the setting window.



Click the Boxes as shown in the graphics for

- Line end with line feed,
- Echo typed characters,
- Append line feed to incoming line ends,
- wrap lines that exceed terminal width,
- Then Click OK to return to Properties window

Click OK Icon on the Properties window to complete the settings.

Now once again Save the Session to store the settings.

To Generate the Icon for the TITRA connectivity with PC, right click on the file name being in the Hyper terminal Window – select Create Shortcut to desk top.

Click on the Icon and start the application. An Terminal Window will open automatically and now the PC is ready to receive the data from K905XX - Automatic Potentiometric Titrator.