TECHNICAL MANUAL

ELECTROCHEMICAL QUARTZ CRYSTAL NANOBALANCE

SYSTEM EQCN-700

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1. INTRODUCTION

The Model EQCN-700 Electrochemical Quartz Crystal Nanobalance is a measurement system for monitoring extremely small variation in the mass of a metal working electrode. The standard EQCN-700 System consists of a Model EQCN-701 Nanobalance Instrument, Model EQCN-702 Faraday Cage, and Model EQCN-703 Remote Probe Unit.

The material of the working electrode is gold, unless otherwise ordered. The working electrode is in the form of a thin film, and is placed on one side of a quartz single crystal wafer which is sealed to the side opening in an electrochemical cell. The AT-cut quartz crystal oscillates in the shear mode at nominal 10 MHz frequency. Any change in the mass rigidly attached to the working electrode results in the change of the quartz crystal oscillation frequency. The frequency of the working quartz crystal is compared to the frequency of the standard reference quartz crystal. The frequency measurements are differential, i.e. the frequency of the reference crystal is subtracted from the frequency of the working crystal. The obtained frequency difference is then measured by a precision frequency counter and displayed on the front panel. The frequency difference is converted to a voltage signal, calibrated in Sauerbrey mass units (referred to as the effective mass) and output to an analog recorder, or analog-to-digital converter.

Typical processes leading to the frequency change which corresponds to the effective mass change at the working electrode are listed below:

adsorption/desorption
metal/alloy plating
surface oxidation
corrosion and corrosion protection
etching
heterogeneous polymerization
ion ingress to (or egress from) ion exchange films
oxidation/reduction of conductive polymer films
intercalation

coadsorption and competitive adsorption
moisture accumulation (from gaseous phase)
etc.

With the Model EQCN-700 you can monitor time transients of the effective electrode mass in an electrochemical or non-electrochemical cell, filled with liquid or gas. You can also perform voltammetric experiments of any type, and monitor potential or current dependence of the effective electrode mass.

The digital resolution of the EQCN-700 is 0.1 Hz which corresponds approximately to 0.1 ng of the effective mass change. The analog resolution is 0.05 ng (100 ng range). The short-term stability is mostly dependent on the state of the working electrode surface and purity of the solution. Usually, it is better than 5 Hz. The exceptional linearity of mass measurements extends up to 100 μg. The use of AT-cut quartz crystals reduces temperature coefficient to the minimum. Under normal circumstances, the effect of temperature can be neglected in the range near the room temperature. If a very high sensitivity or wide temperature range are required, it is recommended to use a thermostatted cell and Model EQCN-703B Remote Probe Unit with thermostatted reference oscillator, or Model EQCN-704 Remote Probe Unit with external reference quartz crystal.

To perform electrochemical measurements, a potentiostat may be required. We offer a line of potentiostats specially designed to work with oscillating quartz crystal electrodes. The Model PS-205 is a general purpose potentiostat/galvanostat with potential control from -8 to +8 V. The Model PS-305 is a precision potentiostat/galvanostat, and Model PS-605 offers exceptionally low noise, high speed, and high precision. Both Models PS-305 and PS-605 have an extended potential range control (-10 to +10 V) and maximum current capability of 0.3 A.

For computer controlled measurements, we recommend VOLTSCAN Real-Time Data Acquisition and Control system based on 16-bit Digital-to-Analog and 16-bit Analog-to-Digital Converters and MasterWindows Data Processing and Interactive Graphics software which is also available (Cat. # DAQ-616SC). As the computer may introduce some noise to the system, usually through the potential waveform, it is often beneficial to filter it out. This noise can be conveniently damped with tunable low-pass filters (FLT-01 single channel, or FLT-03 three channel filters), available from ELCHEMA. These filters are mounted in small boxes with BNC sockets and can be inserted in any signal cable.

2. SPECIFICATIONS

Measurement Ranges

Frequency Difference:		0 to 500 kHz (digital)
Mass Change:		
	RANGE:	**READINGS:**
	100 μg (kHz)	-100.00 to +100.00 μg (kHz),
	10 μg (kHz)	-10.000 to +10.000 μg (kHz),
	1 μg (kHz)	-1.0000 to +1.0000 μg (kHz),
	100 ng (Hz)	-100.00 to +100.00 ng (Hz)
Extended linearity		10 % over nominal range
Overload Indicator		*ca.* 2 % over nominal range

Resolution

Frequency Difference	0.1 Hz
Mass Change	0.05 ng (analog)

Recorder Output

Analog Output Voltage		-10 to +10 V
Extended linearity		10 % over nominal range
Voltage to Mass Change Ratio		10 V per nominal range
Offset Voltage:	coarse	90 μg (approx.)
	fine	800 ng (approx.)

Operating Parameters

Reference Crystal Frequency		10.000 MHz
Power Supply		110/220 V, 50 - 60 Hz
Dimensions:	Instrument	4H x 17W x 16.5D, inch
	Faraday Cage	16H x 12W x 11D, inch

EQCN-700 System Components

Model EQCN-701 Electrochemical Nanobalance Instrument
Model EQCN-702 Faraday Cage
Model EQCN-703 Remote Probe Unit (mounted on the back of the Faraday Cage)

Options

EQCN-703B	Remote Probe Unit with Thermostatted Reference Oscillator
EQCN-704	Remote Probe Unit with External Reference Crystal
RTC-100	ROTACELL Electrochemical Cell System with Piezocell
DAQ-616SC	VOLTSCAN Real-Time Data Acquisition and Control system (including VOLTSCAN software, 16-bit D/A and 16-bit A/D Converters, Break-up Box, 686/Pentium microcomputer, and MasterWindows Data Processing and Graphics Software)
PS-205B	General Purpose Potentiostat/Galvanostat
PS-605	Precision Potentiostat/Galvanostat (±10 V, 300 mA max, 0.1 pA resolution, 300 ns rise time)
FC-299	Frequency Meter/Calibrator/Generator
Electrodes	Wide selection of quartz crystal working electrodes with Ag, Au, Al, Cr, Cu, Fe, Ni, Pt, and Zn coatings

3. OPERATING INSTRUCTIONS

3.1. INSPECTION

After the instrument is unpacked, the instrument should be carefully inspected for damage received in transit. If any shipping damage is found, follow the procedure outlined in the "Claim for Damage in Shipment" section at the end of this Manual.

3.2. PRECAUTIONS

Care should be taken when making any connections to the instrument. Use the guidelines for maximum voltage at the inputs. There should be no signal applied to the inputs when the instrument is turned off. The outputs should not be loaded. They can only be connected to high input impedance devices such as plotters or oscilloscopes.

Use minimal force when putting on, or taking off, the BNC connections, otherwise they might become loose. Push the BNC forward when making a connection or a disconnection in order to relieve the rotational tension on the BNC socket.

Observe the color codes when connecting the power to the probe unit.

Operate the instrument in a cool and well ventilated environment.

Contact us in the event that any of our components do not operate properly. Our components are marked with seals. DO NOT try to open and fix anything yourself, otherwise your warranty agreement will be **nullified**.

3.3. FARADAY CAGE

The Faraday cage tips over easily. It is also possible for someone to accidently brush against it and break, or spill the contents of the cell inside. Hence, it is advised to secure the Faraday cage by mounting an **L**-shaped bracket in the back of it.

3.4. GROUNDING AND INTERFERENCES

It is very important to properly ground the Nanobalance system. The circuitry operates at a high frequency of 10 MHz and is very susceptible to electromagnetic radiation and interference present in the surroundings. Since the working oscillator can not be enclosed in a case due the nature of measurements, the use of a Faraday cage is necessary. Usually, the door of the Faraday cage does not need to be completely closed. (Do not lock the door unless you see an improvement in shielding efficiency and temperature stability of the crystal frequency.). If you find that in your application leaving the door of the Faraday cage wide open does not result in any external influences on the frequency reading, you may wish to remove the door to save space on the bench. Normally, Faraday cage does not need to be grounded with additional wires. You can connect the Faraday cage chassis to the AC ground or water pipe using a thick grounding cable, if necessary. Make this connection only if you see an improvement in shielding or reduction in noise. Avoid creating ground loops! The Nanobalance Instrument (EQCN-701) is connected to the AC ground through the HP type 3-conductor power cable. With the short banana-to-banana cable (provided), connect the chassis of your potentiostat to the chassis of the Nanobalance Instrument EQCN-701 using the CHASSIS banana socket (metal hex nut) on the back panel of the EQCN-701. You can also use this socket for reference purposes or to connect to other instruments which need to be grounded. The analog ground of the EQCN-701 is provided on the back panel (black banana socket marked GND) for reference purposes. Normally, do not connect anything to this socket. However, you can short it to the AC ground (CHASSIS) or to the analog ground of the potentiostat if necessary. Remove all unnecessary cables from the instrument before doing measurements. (Cables which are not connected on the other end may act as antennas and should also be removed. Note that some measurement instruments, e.g. oscilloscopes, have often the cable guard shorted to the AC ground. Making a connection to such an instrument is equivalent to shorting the analog ground of Nanobalance Instrument to the AC ground.).

Since the oscillating circuit is connected to the electrochemical cell through the working electrode, changes in position of wires connected to the Reference and Counter Electrodes with respect to ground planes may result in some frequency change due to the change in parasitic capacitance. Make sure that all electrodes are firmly attached to the cell top and the connecting wires are short and do not bounce during the measurements. The connections between the working quartz crystal and the Remote Probe Unit (blue and white tip banana jacks) should be as short as possible and the interwire capacitance should be kept constant during the measurements.

In microelectrogravimetric technique, we do not measure the absolute mass of the working electrode but rather the mass change that occurs during the experiment. At the beginning of the experiment, you set the initial mass to zero (or any other value you wish) using the MASS OFFSET potentiometers located on the front panel of the instrument. Due to the high sensitivity of measurements (compare it with a regular balance), it is essential that you maintain all the system parameters (including parasitic capacitances of connecting wires and cables) constant. Only then the frequency change observed during the experiment will correspond to the change in values of parameters of the equivalent circuit of the quartz crystal assembly (especially the inductance change proportional to the change in mass rigidly attached to the working electrode).

With electrochemical cells, a significant frequency change (observed as a drift) may occur even at constant potential due to surface changes, adsorption, poisoning, etc. Use only high purity chemicals. Please keep in mind that the gold substrate may undergo slow dissolution at higher potentials which may lead to the apparent mass decrease (absolute frequency increase). Very often mass changes may result as a consequence of the surface oxide formation. Sudden metal dissolution may result in oversaturation and deposition of salts on the electrode surface. Deposits on the surface may be sometimes difficult to remove and may block the surface and change the electrode activity. If you are not familiar with electrochemistry at solid electrodes, consult general textbooks and monographies (*e.g.*, A. J. Bard and L. R. Faulkner, *Electrochemical Methods*, J. Wiley and Sons, New York, 1980).

WARNING: Do not attach ground wires to a gas or heating pipe.

3.5. THERMAL SENSITIVITY

As with any electronic equipment, this instrument should be warmed up in order to achieve the greatest accuracy. Under normal circumstances, the frequency difference readout on the front panel of the instrument should be stable to 1-2 Hz after 15 minute warmup and the mass change readout should stabilize in 30 minutes.

4. INSTALLATION

The operating instructions have been made short and simple but make sure they are followed in this exact order. **Bold** letters indicate connections and controls on the instrument only.

4.1. Initial set-up

(1) Unpacking.
Carefully remove all paper and tape used in shipping. Place instrument on a convenient bench. Check the items against the packing list.

(2) The Faraday Cage (EQCN-702) should be placed on the left side of the Nanobalance Instrument (EQCN-701) to have connections as short as possible.

(3) This instrument has been set for 110 V a.c., 50-60 Hz, power supply. If necessary, you can change this setting to 220 V a.c. by changing the position of the supply voltage selector 110/220 located on the back panel of the instrument. If you have to make the change, make sure the power in the instrument, **AC**, and in all other devices is off, and nothing is connected to the instrument or the probe unit. With the power cord ***disconnected*** from the instrument, set the power supply voltage switch to the appropriate position, 110 or 220 V. Connect the power cord back to the instrument.

(4*a*) If you have a standard EQCN-700/PS-205 or EQCN-700/PS-605 system, connect the **PROBE** BNC sockets on the back panel of the Nanobalance Instrument (EQCN-701) labeled **1**, **2**, and **3** to BNC sockets with the corresponding numbers on the side panel of the Remote Probe Unit which is mounted on the back of the Faraday Cage, using the coaxial cables provided.

(4*b*) If you have a custom system EQCN-700/PS-705, connect the **PROBE** BNC socket labeled number **3** on the back panel of the Nanobalance Instrument (EQCN-701) to the BNC socket labeled **Δf** on the side panel of the Remote Probe Unit which is mounted on the back of the Faraday Cage. Use the coaxial cable provided. Do not connect anything to BNC sockets labeled number **1** and **2** on the back panel of the Nanobalance Instrument (EQCN-701).

(5) Connect the multiconductor cable marked **EQCN-700 SPLY** with 6-pin audio-type connector on one side and 8-pin audio-type connector on the other side to the corresponding sockets marked **SPLY** (SUPPLY) on the back panel of the instrument (EQCN-701) and on the side panel of the Remote Probe Unit (the EQCN section). Note that PS-705 uses a different supply cable which is marked **PS-705 SPLY** and is to connect potentiostat to the Remote Probe Unit, PS section.

(Input Gate on our system)

(6) Put the **FREQUENCY SENSITIVITY** switch to '**x 1**' and the **INT/EXT** (internal / external frequency source) toggle switch to the **INT** position.

(7) Put the **RANGE** selector to **100 μg** position (least sensitive).

(8) Connect the RECORDER **V-OUT** BNC output socket (for your convenience, there are two identical V-OUT sockets on the front panel and back panel of the instrument and you can use any one of these) to the input of the recorder. You can use a strip chart recorder, X-Y recorder, or a computerized data logger, *e.g.* VOLTSCAN Real-Time Data Acquisition and Control System DAQ-616SC available from ELCHEMA.

(9) Zero the recorder.

(10) Set the range on the recorder to 10 V.

(11) Put the mass change SIGN (+/-) switch to the + (plus) position.

(12) Put the **M/F** mode switch to the **M** position for frequency difference conversion to the Sauerbrey mass change.

(13) Put the **INT/EXT** switch selecting the frequency source for data processing to the **INT** position which is used for EQCM measurements.

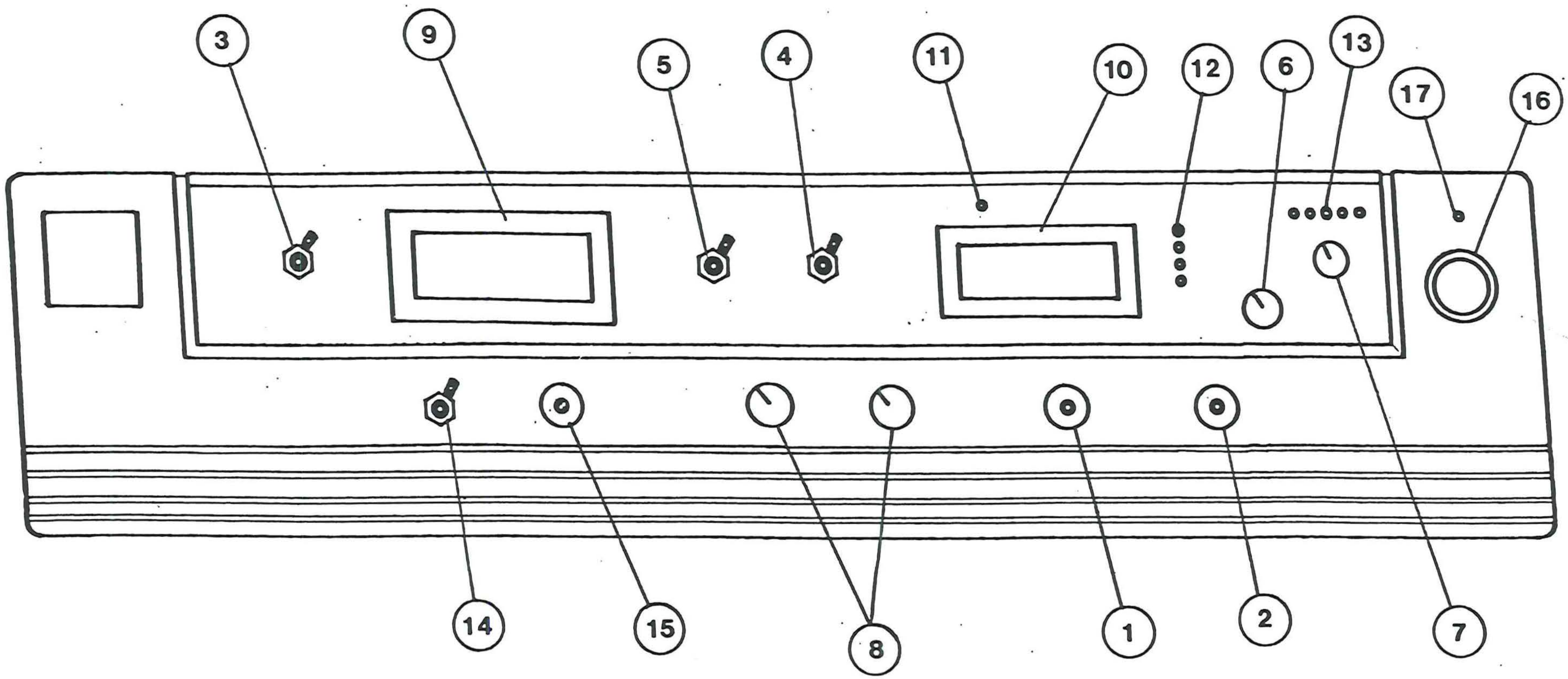

FIGURE 1. Front panel view of the Model EQCN-701 Nanobalance Instrument.
1 - **F-OUT** (frequency difference output); 2 - **M-OUT** (analog output voltage proportional to the apparent mass change); 3 - frequency resolution selector; 4 - **+/-** mass change polarity switch; 5 - **M/F** mass/frequency mode switch; 6 - **RANGE** (mass sensitivity selector); 7 - **DAMPING** (output filter selector); 8 - **COARSE** and **FINE** mass **OFFSETS**; 9 - frequency difference panel meter; 10 - apparent mass change panel meter; 11 - mass overload indicator; 12 - RANGE indicators; 13 - DAMPING indicators; 14 - **INT/EXT** (internal / external frequency source toggle switch); 15 - external frequency input socket; 16 - main **POWER** switch; 17 - power indicator.

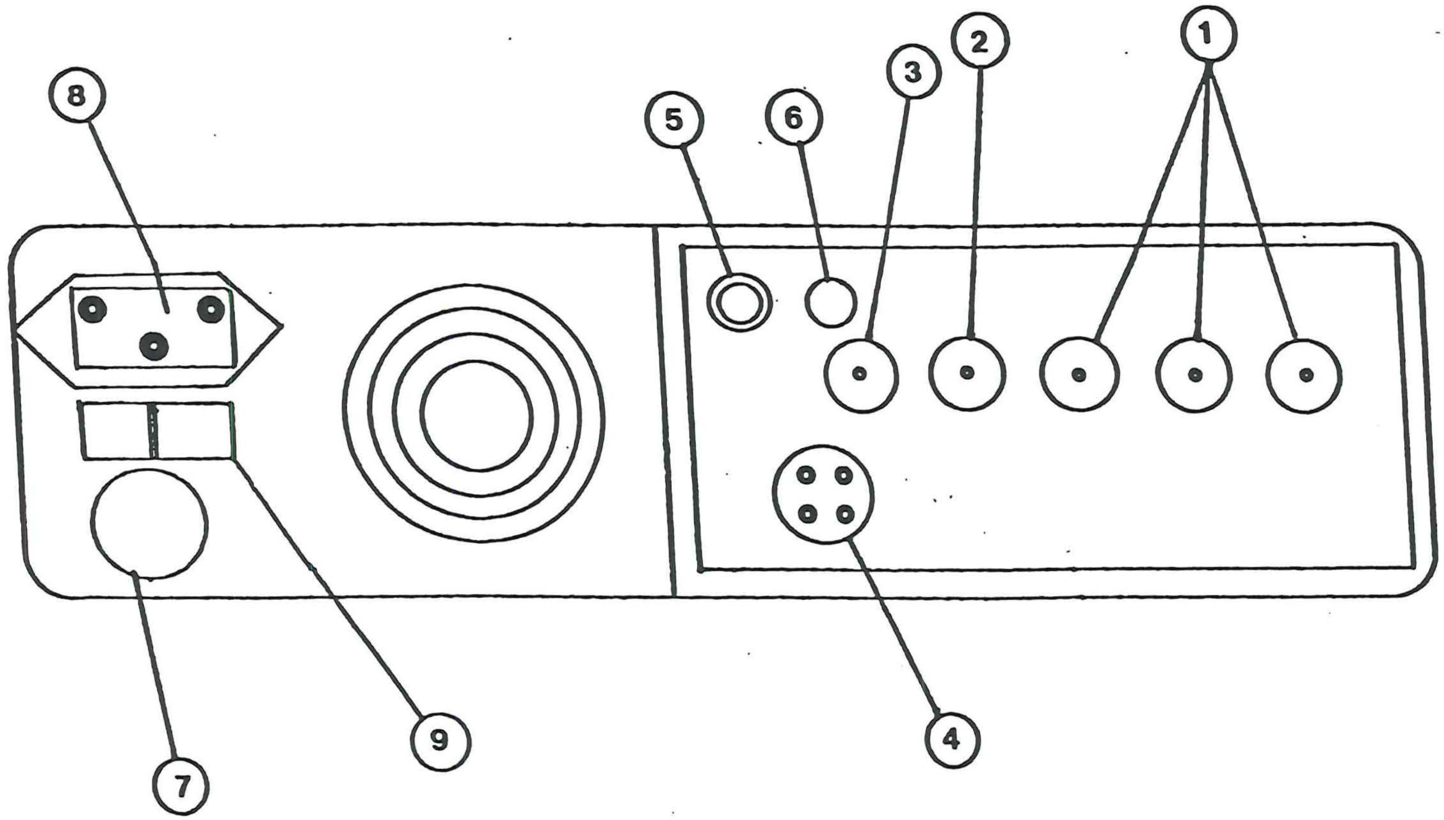

FIGURE 2. Back panel view of the Model EQCN-701 Nanobalance Instrument.
1 - **PROBE** (three BNC sockets for connections to the Remote Probe Unit); 2 - **WE** (BNC socket for connection to the working electrode output in a potentiostat); 3 - **CE** (BNC socket for connection to the counter electrode output in a potentiostat); 4 - **SPLY** (audio-type socket for connection of power supply to the Remote Probe Unit); 5 - **CHASSIS**; 6 - **GND** (analog ground); 7 - power **FUSE**; 8 - HP-type power socket; 9 - power supply voltage selector switch (110/220 V).

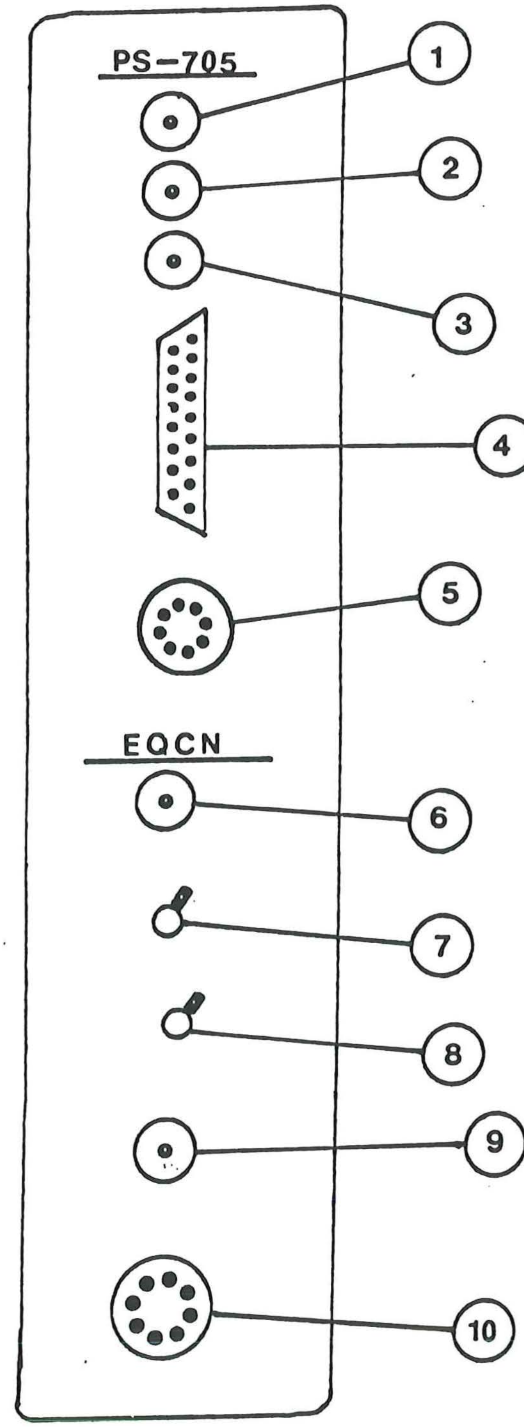

FIGURE 3. Side panel view of the custom EQCN-703 Remote Probe Unit mounted on the back of the Faraday Cage.

(A) PS-705 Potentiostat section: 1, 2, 3 - BNC sockets marked **C1**, **C2**, and **C3** for connections to corresponding BNC sockets on the back panel of the potentiostat; 4 - **I/O** socket for connection to the corresponding socket on the back panel of the potentiostat; 5 - **SPLY** (6-pin audio-type socket for connection of power supply from the back panel of the potentiostat);

(B) EQCN section: 6 - **ΔF** output to be connected to the **PROBE 3** BNC socket on the back panel of Nanobalance Instrument EQCN-701; 7 - **GND / FLOAT** toggle switch for optional grounding of the working oscillator; 8 - $\mathbf{f_{WO}}$ / $\mathbf{f_{RO}}$ toggle switch; 9 - **F-OUT** (BNC socket providing the working oscillator $\mathbf{f_{WO}}$ or reference oscillator $\mathbf{f_{RO}}$ signals for optional measurements on external frequency meter or viewing on oscilloscope).

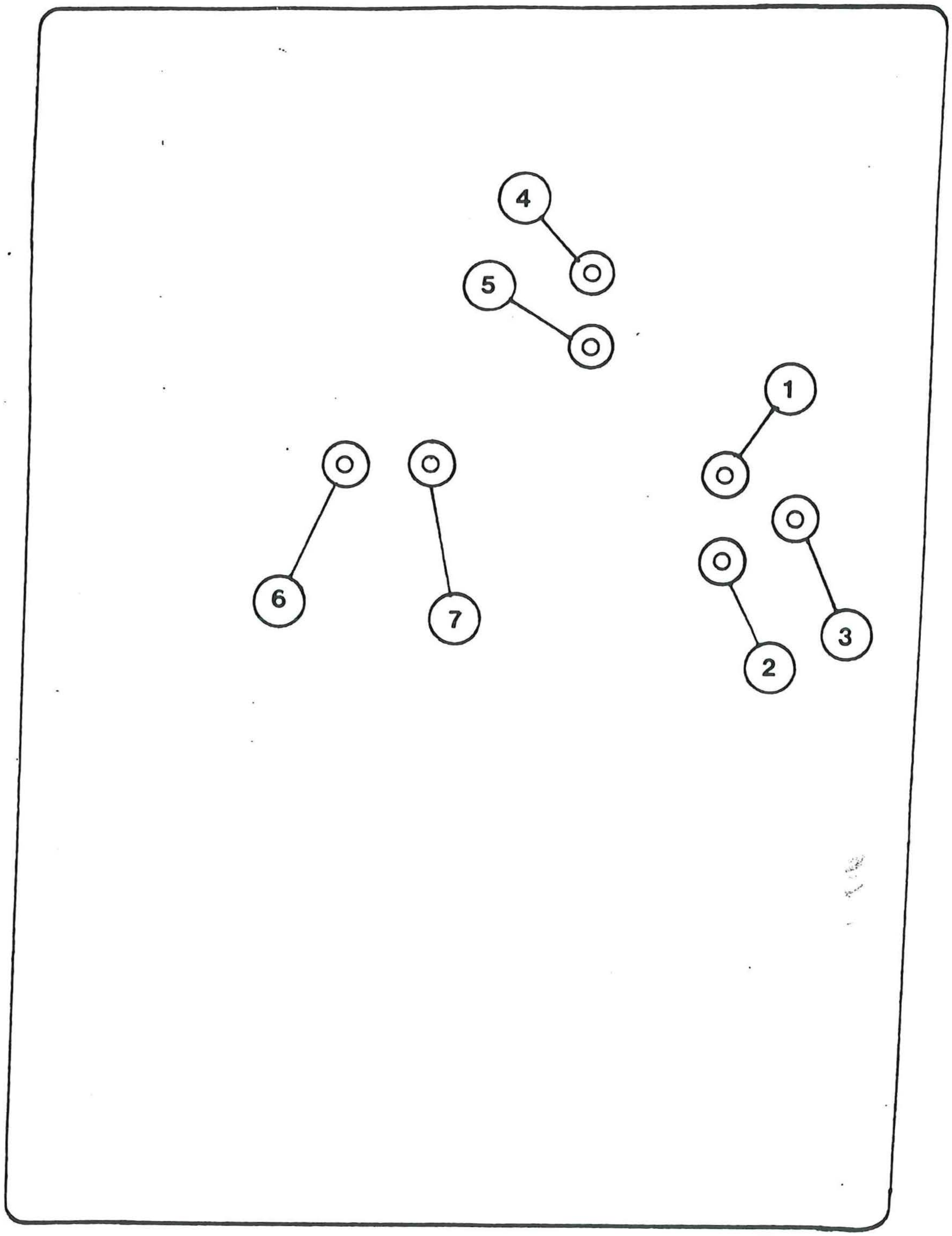

FIGURE 4. Inside panel view of the Faraday Cage.

The **'PS only'** section (1-3) is for experiments without Nanobalance, the **EQCN** section (4-7) is for combined electrochemical and nanobalance measurements, and the **CRYSTAL** section (6,7) is for measurements utilizing nanobalance only.

1,4 - **CE** (pin tip banana sockets for connection to the *counter electrode* in electrochemical cell); 2,5 - **REF** (pin tip banana sockets for connection to the *reference electrode*); 3 - **WE** (pin tip banana socket for connection to the *working electrode*); 6,7 - **CRYSTAL** (two pin tip banana sockets marked **AIR** and **LIQUID** for connections to the quartz crystal electrodes exposed to air and solution, respectively.

4.2. Power ON checks

(1) Turn the power switch **ON**.

(2) Set the **DAMPING** selector to the first position.

(3) Use the **COARSE** and **FINE OFFSET** knobs to set the MASS display and the recorder output voltage to zero (or close to zero). The recorder output voltage provided at the V-OUT BNC jack is 10 V FS (full scale) for the nominal mass RANGE, *i.e.* the usefull output voltage is ±10 V.

(4) Insert a 10 MHz quartz crystal (*e.g.* ELCHEMA Cat. # QC-10-AuPB) in the Crystal-Cell assembly (*e.g.* our ROTACELL, Model RTC-100). Connect pin tip banana plugs from the Crystal-Cell assembly to the white and blue pin tip banana jacks **CRYSTAL**, inside the Faraday Cage. These jacks are labeled **AIR** and **LIQUID** for electrodes on the quartz crystal exposed to the air and solution, respectively. (For Crystal-Cell assembly instructions refer to Chapter 5.)

(5) At this point, the frequency meter should give you some frequency difference reading. Typical values are from 500 Hz to 90 kHz. If the reading is 0, the contacts to the working crystal may not be good or crystal cannot oscillate due to a strong damping (thick film deposited on the crystal, broken crystal, *etc.*).

(6) Note the voltage at the output. The recorder output voltage is 10 V per nominal mass range (FS).

(7) Use the **COARSE** and **FINE OFFSET** knobs to set the mass reading on MASS panel meter to zero. You can now change the MASS **RANGE** to more sensitive one. Again, use the **COARSE** and **FINE OFFSET** knobs to set the mass reading on MASS panel meter to zero.

(8) Repeat the operations (7) until the MASS **RANGE** with desired sensitivity is selected.

(9) In the following testing, observe the rules:
- Start experiments with ***MASS change*** set to ***zero*** (or close to zero).
- Before disconnecting the Working Crystal, change the mass RANGE to 100 μg (least sensitive).

4.3. Connections to a potentiostat

(1) Make sure, the potentiostat CELL switch is set to OFF, or a DUMMY CELL position.

(2) Connect BNC sockets **C1**, **C2**, and **C3** on the back panel of your potentiostat to corresponding BNC sockets on the side panel of the Remote Probe Unit (Faraday Cage).

(3) Connect the audio-type 6-pin **SPLY** socket on the back panel of your potentiostat to the corresponding socket on the side panel of the Remote Probe Unit (Faraday Cage), in the PS-705 section, using a multiconductor cable marked **PS-705 SPLY**.
WARNING: *Do not attempt to connect directly the **SPLY** socket on the back panel of the Potentiostat PS-705 to the **SPLY** socket on the back panel of the Nanobalance Instrument EQCN-701 since this may result in an electric shock and serious damage to both intruments.*

(4) Connect the standard DB-25 female **I/O** socket on the back panel of the potentiostat to the corresponding DB-25 male socket on the side panel of the Remote Probe Unit (Faraday Cage).

(5) Connect the **E-OUT** and **I-OUT** BNC sockets on your potentiostat to the inputs of a recorder. For your convenience, two sets of these sockets are provided, one on the front panel and one on the back panel of the Nanobalance Instrument EQCN-701. Any recording device with input resistance higher than 500 ohm (higher than 2 kohm recommended) can be used, for instance, an XY-Recorder or a Data Logger. If you are using our VOLTSCAN Real-Time Data Acquisition and Control system, connect BNC cables marked **E** and **I** which extend from the Break-up Box to the **E-OUT** and **I-OUT** sockets, respectively.

(6) Connect the **P-IN** (PROGRAM INPUT) BNC socket on your potentiostat (use either the socket located on the front panel or the one on the back panel) to the output of a waveform generator, *e.g.* ELCHEMA's Model FG-206F, or a Digital-to-Analog Converter. If you are using our VOLTSCAN Real-Time Data Acquisition and Control system, connect the BNC cable marked **P** which extends from the Break-up Box (Model DAQ-617) to the **P-IN** input socket in potentiostat.

(7) Connect the *Counter Electrode* in the electrochemical cell to the red pin tip banana jack **CE** inside the Faraday Cage.

(8) Connect the *Reference Electrode* in the electrochemical cell to the yellow pin tip banana jack **REF** inside the Faraday Cage.

(9) Make sure the *working crystal* is connected to the white and blue pin tip banana jacks marked **CRYSTAL, Air** and **Liquid**, respectively, inside the Faraday Cage. (For Crystal-Cell assembly instructions refer to Chapter 5.)

(10) You are now ready to start your experiment. Refer to the next section to learn the details of the experimental procedure illustrated with an example of the deposition and stripping of copper.

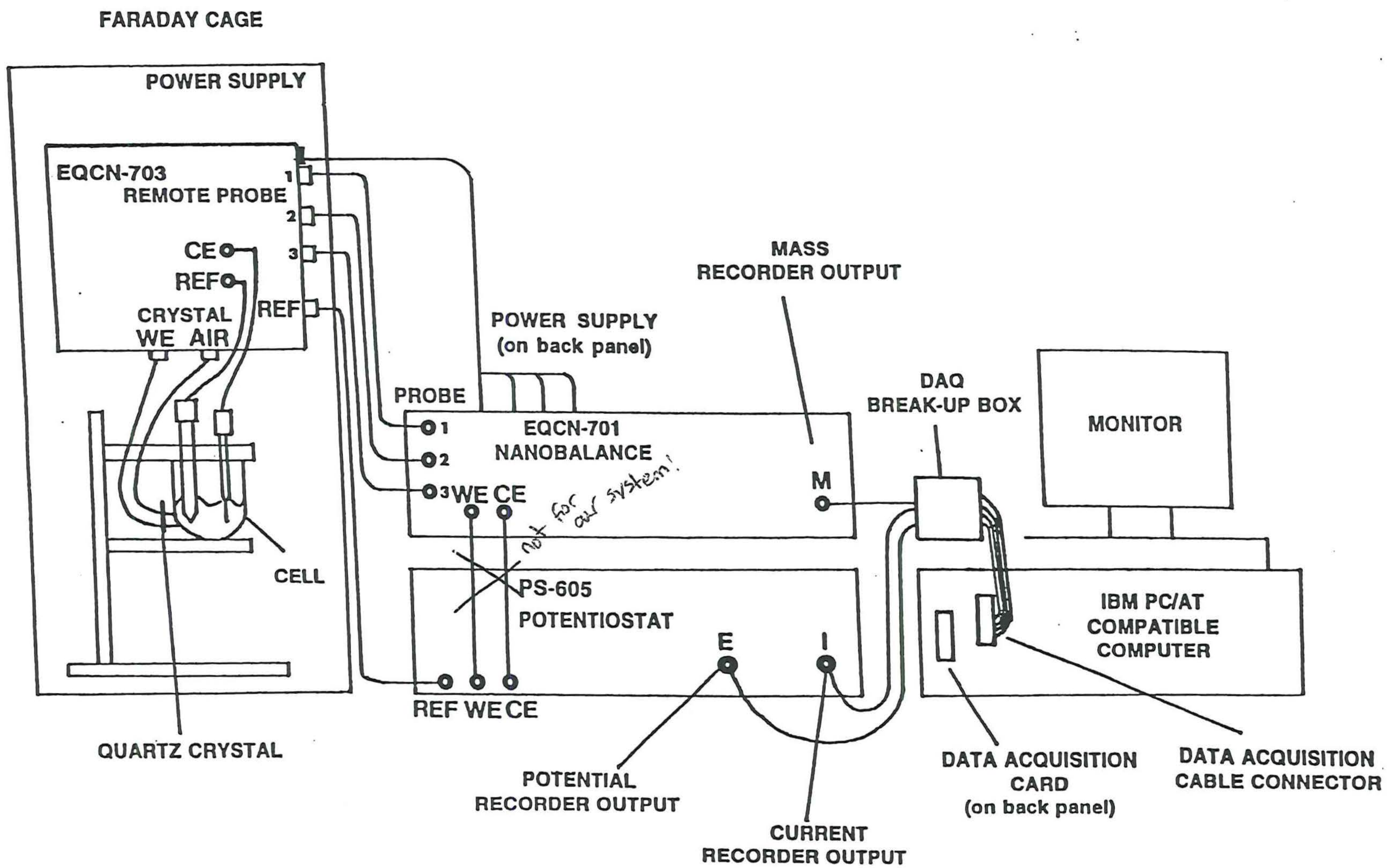

FIGURE 5. Connections of the EQCN-700 Electrochemical Quartz Crystal Nanobalance System with potentiostat and microcomputer.

4.4. Testing experiment with real cell ON

(1) You are now ready to use the instrument for measurement purposes. If you want to perform a simple checking experiment, you can use, for example, a 5 mM copper(II) solution in 0.1 M HNO_3 . Program your waveform generator for a conditioning potential E_{cond} = +500 mV *vs.* SCE, sweep from E_1 = +500 mV to E_2 = 0 mV, and back to E_3 = +500 mV; the final potential should be $E_4 = E_{fin}$ = +500 mV. If you are using the VOLTSCAN Real-Time Data Acquisition and Control System, set the data in the PARAMETERS Table as follows:

Potential mV vs. SCE	**Holding time** s	**Scan Rate** mV/s
E1 = +500	1	100
E2 = 0	0	100
E3 = +500	0	100
E4 = +500	0	

potentials assume an Ag/AgCl junction reference

(Number of) SCANS: 3

Follow the instruction manual for details of the VOLTSCAN operation.

(2) Check if the *Reference Electrode* is connected and placed in the solution.

(3) Check if the *Counter Electrode* is connected and placed in the solution.

(4) Set the MODE switch on your potentiostat to select the PS (POTENTIOSTATIC) control.

(5) Set the current range on your potentiostat to 1 mA FS (full scale).

(6) Turn the CELL switch on your potentiostat to the ON (or: EXTERNAL

CELL) position.

(7) Turn the CONTROL switch on your potentiostat to the ON position.

(8) Initiate the potential scan. Using the **OFFSET** knobs, appropriately position the mass response curve on the recorder chart, or monitor plot.

(9) Carefully change the cathodic potential limit E_2 to a more negative one. Keep changing the E_2 value until the copper deposition just begins to take place. On the voltammogram, you should be able to observe an increase in the cathodic current due to copper deposition, and an increase in the anodic peak due to the copper stripping. On the *mass-potential* curve, which can be recorded simultaneously with the *current-potential* curve, you should observe a mass histeresis with a clear mass increase in the potential region where copper is being deposited, and a mass decrease which is fastest in the region of the stripping current peak. If you observe mass changes in the opposite direction, change the **POLARITY** switch (+/-) setting. (When the working crystal frequency is lower than the reference crystal frequency, the mass increase is manifested by the increase in the measured frequency difference. When the working crystal frequency is higher than the reference crystal frequency, the mass increase is manifested by the decrease in the measured frequency difference.)
Do not deposit too much copper. During the anodic stripping process, very often a high concentration of the dissolved metal builds up in the vicinity of the electrode surface, and it may result in the formation of metal oxides on the electrode surface (the oxides may be sometimes difficult to remove).

Depending on your experiment and the range of your frequency measurements you may wish to increase the sensitivity of measurements by changing the **RANGE** selector or by increasing the sensitivity of the recorder, e.g., to 500 mV. (Be careful with whatever changes you make in instrument settings and connections because the instrument is capable of outputing ±15V at the **RECORDER V-OUT**). If you want to change the **RANGE** selector to more sensitive range, first offset the mass reading to zero (or close to zero) with **COARSE** and **FINE** offset potentiometers. The offset potentiometers will allow you to do measurements at high sensitivity on large signals.

4.5. Other utilities (Optional)

(1) The frequency difference may be viewed and measured externally by connecting a measuring device to the **ΔF-OUT** BNC output located on the front panel of the instrument. This output provides a square wave logic signal of 0 V to 5 V and frequency indicated on the **FREQUENCY** panel meter.

(2) The absolute frequency of the *working oscillator* **WO** and the *reference oscillator* **RO** may be viewed and measured externally by connecting a measuring device to the **F-OUT** BNC output located on the side panel of the Remote Probe Unit attached to the Faraday Cage. This output provides a high frequency waveform (*ca.* 10 MHz), approximately 0 to 5 V. Use only short concentric cables (2-3 feet) to connect the **F-OUT** BNC socket to the measuring device (an oscilloscope or frequency meter). The **WO** and **RO** are selected with the toggle switch **F** located above the **F-OUT** socket.

(3) This instrument may by used as a frequency meter. To do this, connect the frequency source to the BNC socket marked **EXT F-IN** (EXTERNAL frequency input) on the front panel of the Nanobalance Instrument EQCN-701. The voltage on this input *must be* a logic signal, 0 V to 5 V. Set the **INT./EXT.** (INTERNAL / EXTERNAL) toggle switch on the front panel to the **EXT**ernal position. Frequencies up to 500 kHz can be measured with a maximum resolution of 0.1 Hz.

(4) (Optional) A synchronized gate output, **GATE OUT,** from the frequency meter, outputs a logical square wave, 0 V to 5 V, at a frequency of 0.1 Hz, 1 Hz or 10 Hz depending on the frequency sensitivity switch setting. (This output is not available on all instruments.).

(5) The **DAMPING** selector switch controls a filter which damps the noise of the output voltage at the **RECORDER V-OUT**. Use the setting which works best for the particular experiment you are doing. Make sure that the damping acts only on the high frequency noise and not on the (slower) signal. The obtained curve should be more smooth (less noisy) but not more sluggish (signal should be intact). The time constants of the DAMPING are specially selected for typical scan rates used in cyclic voltammetry and microelectrogravimetry experiments. Usually, the first

or second setting, from the left, is appropriate. The filter time constants increase, from left to right, in the following order:

FILTER POSITION	TIME CONSTANT *ms*	INDICATOR NUMBER
0	none	none (all OFF)
1	10	1
2	40	2
3	80	3
4	200	4
5	1000	5

5. CRYSTAL-CELL ASSEMBLY

5.1. Mounting Quartz Crystals

(1) Remove the rubber band and the metal cover can of the quartz crystal.

(2) Carefully bend the contact spring wires close to the quartz crystal, such, that after sealing, the gold working electrode to be immersed in the solution will contact the blue wire and blue pin tip banana jack marked **Liquid**, inside the Faraday Cage. Bend w/ two pliers on wire, a little on each side at a time - it is very easy to crack crystal!

(3) Glue the crystal to the side opening in the cell.
Seal w/ lots of extra glue around outside to protect crystal

5.2. Assembling Piezocells in ROTACELL Holder

(1) Once the glue is dry, place the cell into the ROTACELL holder and adjust the height of the bottom support if necessary.

(2) Press the clip lever open and lock it in this position with the clip support by turning clockwise the black anodized aluminum knob in the bottom plate of the ROTACELL.

(3) Rotate the cell top plate (with the reference and counter electrodes) clockwise, out of the Faraday Cage, and place the cell underneath. Hold the cell and rotate it together with the cell top back to the initial position (inside the Faraday Cage).

The quartz crystal sealed to the cell should be directed toward you so that the contact pins extending from the crystal will not be damaged by the clamp fixture. After the cell top, with the cell, is rotated back into the Faraday Cage and the cell is fully supported by the bottom plate of the ROTACELL, carefully turn the cell clockwise until the crystal pins slide onto the disk shaped contacting metal pads which are hot-pressed into the clamp base block.

(3) While pressing firmly the clip lever, release the clip support by turning the black anodized aluminum knob in the bottom plate counter-clockwise. Then, by slowly releasing the clip lever, lower the clip jaw until it touches the crystal pins and secures their contact to the metal pads.

NOTE: If the crystal pins do not move unobstructed to the contact plates of the clip, remove the cell and gently pull the crystal pins to bend the spring wires. Once the pins are positioned flat on the contact plates, release the clip lever. If the crystal pins are not on the level of the contact metal pads, adjust the height of the clip base block using the spring loaded screws beneath the base plate.

(4) Check if the cell is tightly positioned in the holder. If it is loose, press the clip to free crystal pins, and remove the cell from the holder. Adjust the bottom support level and start from (2), again.

5.3. Disassembling Piezocells from ROTACELL Holder

(1) To remove the cell from the ROTACELL holder, press the clip lever open and lock it in this position with the clip support by turning clockwise the black anodized aluminum knob in the bottom plate of the ROTACELL.

(2) Very carefully rotate the cell counter-clockwise to free the crystal pins from the clip.

(3) Now, holding the cell and the top plate with the right hand, rotate them clockwise, out of the Faraday Cage, while keeping the bottom plate from moving with the left hand. Remove the cell by moving it downward.

(4) Clean the piezocell by repeatedly pouring distilled water into the cell. Do not direct water jet straight onto the crystal as it may get damaged (small cracks developed on the crystal may only be seen under a microscope). Store the EQCN cells filled with distilled water or in a dry condition. Be aware that the electrode surfaces (both the inside working electrode and the outside 'air' electrode) may

get contaminated by pollutants from the environment.
Clean the reference and counter electrodes by washing with distilled water. If you are using our REF-100 Double-Junction Reference Electrode, store it in a 1 M KNO_3 solution unless you changed the original KNO_3 electrolyte filling the intermediate chamber of the electrode to some other solution. Store the counter electrode in a dry condition.

5.4. Final checks

(1) Check the assembly in order to avoid a short circuit across the contact plates in the clip.

(2) Make sure there is no significant strain on the crystal pins.

(3) Make sure there is no solution spills on the air-side of the quartz crystal. Any mass change on the air-side of the quartz crystal will also influence the frequency of oscillation.

6. ELECTRICAL CIRCUITS

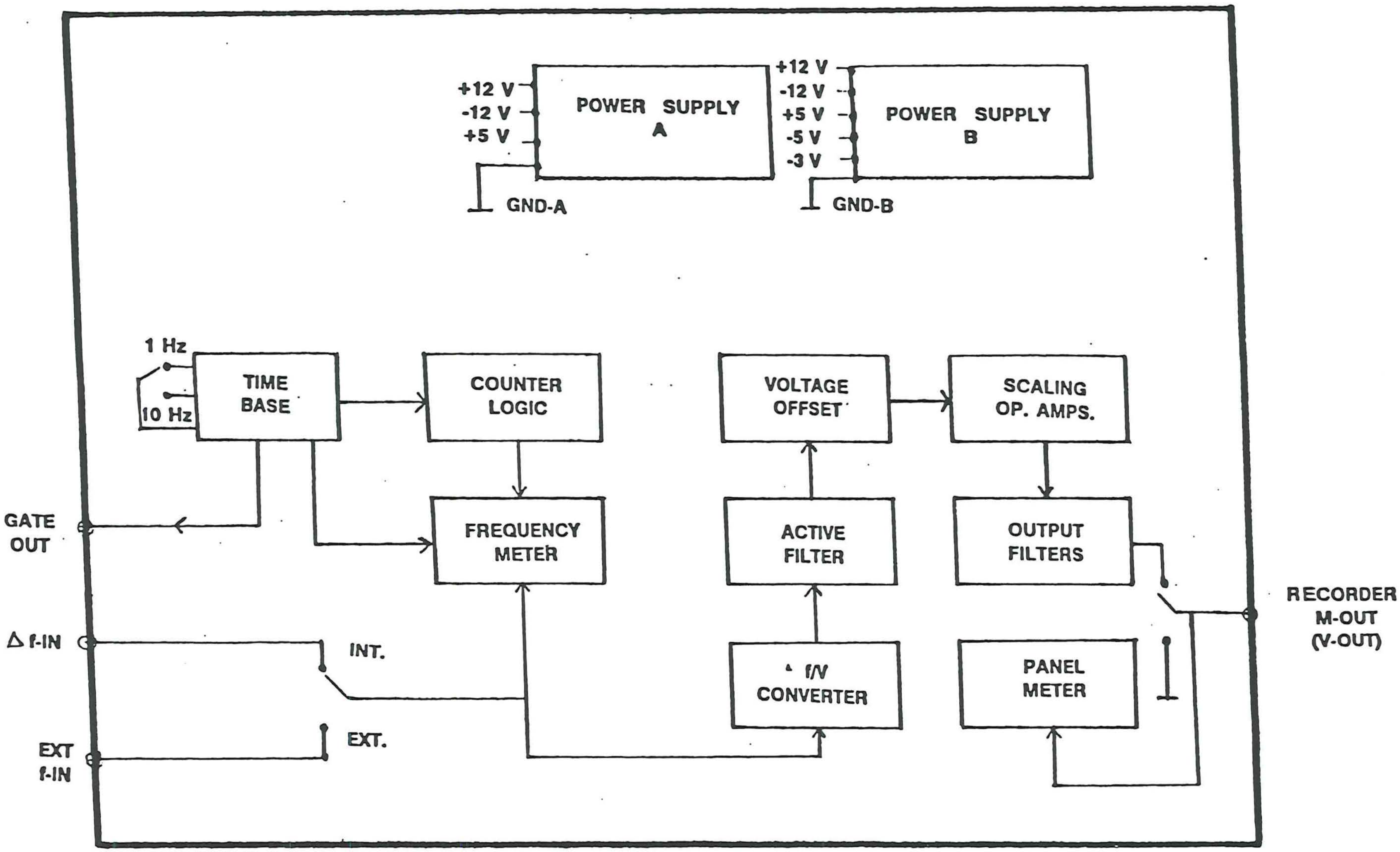

FIGURE 6. Block diagram of EQCN-701 Nanobalance Instrument.

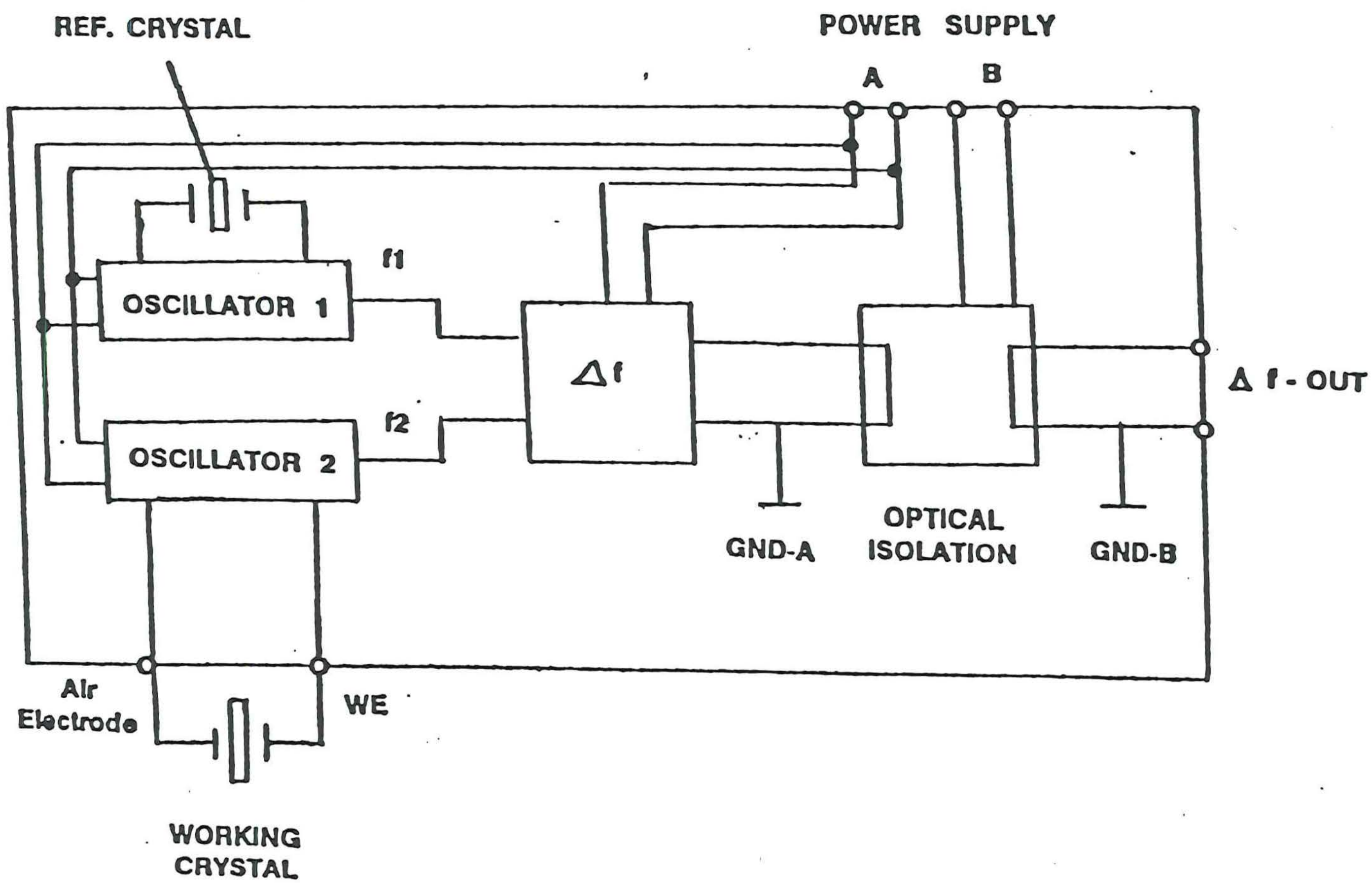

FIGURE 7. Block diagram of the Remote Probe Unit, Model EQCN-703.

7. SERVICING NOTES

In case of malfunction of the EQCN-700 instrument, the unit may be returned to the factory for service. It should be returned postpaid. Since the equipment is guaranteed for one year, no charges for repair will be made for time and materials. The guarantee does not cover misuse of the Model EQCN-700 or damage due to improper handling or service.

WARRANTY

All our products are warranted against defects in material and workmanship for one year from the date of shipment. Our obligation is limited to repairing or replacing products which prove to be defective during the warranty period. We are not liable for direct, indirect, special, incidental, consequential, or punitive damages of any kind from any cause arising out of the sale, installation, service, or use of our instrumentation.

All products manufactured by ELCHEMA Company are thoroughly tested and inspected before shipment. If ELCHEMA receives notice from the Buyer of any defects during the warranty period, ELCHEMA shall, at its option, either repair or replace hardware products which prove to be defective.

Limitation of Warranty

A. The Warranty shall not apply to defects resulting from:
   1. Improper or inadequate maintenance by Buyer;
   2. Unauthorized modification or misuse;
   3. Operation in corrosive environment (including vapors, solids, and aggressive solvents);
   4. Operation outside the environmental specification of the product;
   5. Improper site preparation and maintenance.

B. In the case of instruments not manufactured by ELCHEMA, the warranty of the original manufacturer applies.

C. Expendable items, including but not limited to: glass items, reference electrodes, valves, seals, solutions, fuses, light sources, O-rings, gaskets, and filters are excluded from warranty.

THE WARRANTY SET FORTH IS EXCLUSIVE AND NO OTHER WARRANTY, WHETHER WRITTEN OR ORAL, IS EXPRESSED OR IMPLIED. ELCHEMA SPECIFICALLY DISCLAIMS THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

For assistance of any kind, including help with instruments under warranty, contact you ELCHEMA field office of instructions. Give full details of the difficulty and include the instrument model and serial numbers. Service date and shipping instructions will be promptly sent to you. There will be no charges for repairs of instruments under warranty, except transportation charges. Estimates of charges for non-warranty or other service work will always be supplied, if requested, before work begins.

CLAIM FOR DAMAGE IN SHIPMENT

Your instrument should be inspected and tested as soon as it is received. The instrument is insured for safe delivery. If the instrument is damaged in any way or fails to operate properly, file a claim with the carrier or, if insured separately, with the insurance company.

SHIPPING THE INSTRUMENT FOR WARRANTY REPAIR

On receipt of shipping instructions, forward the instrument prepaid to the destination indicated. You may use the original shipping carton or any strong container. Wrap the instrument in heavy paper or a plastic bag and surround it with three or four inches of shock-absorbing material to cushion it firmly and prevent movement inside the container.

GENERAL

Your ELCHEMA field office is ready to assist you in any situation, and you are always welcome to get directly in touch with the ELCHEMA Service Department:

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