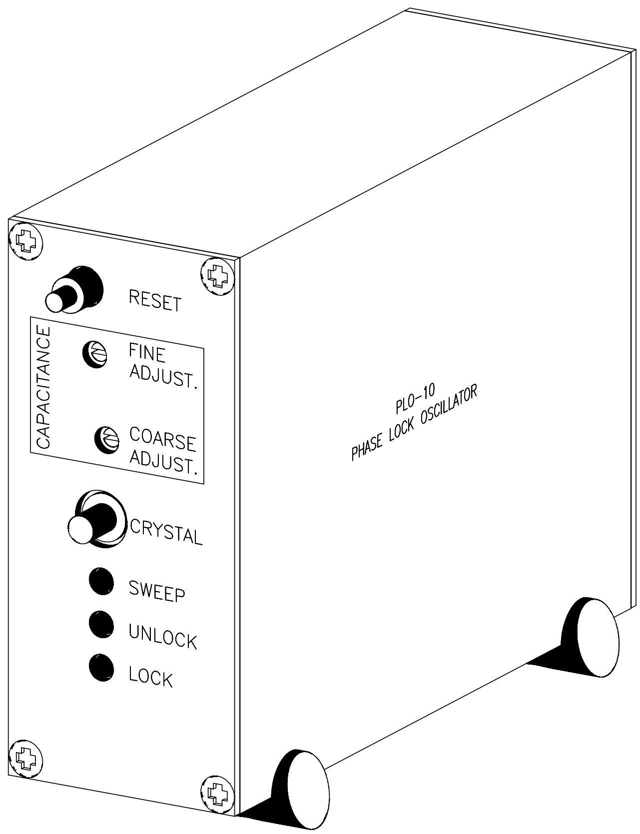
OPERATION AND SERVICE MANUAL



PLO-10 Series

Phase Lock Oscillator

IPN 605800 Rev. G

INFICON

OPERATION AND SERVICE MANUAL

PLO-10 Series

Phase Lock Oscillator

IPN 605800 Rev. G

INFICON Instruments for Intelligent Control®

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WARNING

All standard safety procedures associated with the safe handling of electrical equipment must be observed. Always disconnect power when working inside the controller. Only properly trained personnel should attempt to service the instrument.

Warranty

INFICON warrants the product to be free of functional defects in material and workmanship and that it will perform in accordance with its published specification for a period of (twenty-four) 24 months.

The foregoing warranty is subject to the condition that the product be properly operated in accordance with instructions provided by INFICON or has not been subjected to improper installation or abuse, misuse, negligence, accident, corrosion, or damage during shipment.

Purchaser's sole and exclusive remedy under the above warranty is limited to, at INFICON's option, repair or replacement of defective equipment or return to purchaser of the original purchase price. Transportation charges must be prepaid and upon examination by INFICON the equipment must be found not to comply with the above warranty. In the event that INFICON elects to refund the purchase price, the equipment shall be the property of INFICON.

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1 GENERAL DESCRIPTION

The INFICON Phase Lock Oscillator was developed specifically to support the use of the quartz crystal microbalance in the measurement of lossy films and in liquid applications. In addition to accurately tracking the frequency of heavily damped crystals, the PLO-10 provides a dc voltage that is proportional to the crystal's conductance (1/resistance). This provides additional information in the study of lossy films and viscous solutions.

The PLO utilizes an internal oscillator referred to as a Voltage Controlled Oscillator (VCO) to drive the crystal. The crystal current is monitored and the frequency of the oscillator is adjusted until there is zero phase between the crystal voltage and current. Assuming that the crystal's electrode capacitance has been effectively cancelled, this point of zero phase between the crystal current and voltage is the exact series resonant point of the crystal. The magnitude of the current at this point is directly proportional to the crystal's conductance. This current is converted to a voltage, demodulated and amplified to create a dc voltage proportional to crystal conductance.

The PLO contains a phase detector that continuously monitors the phase difference between the crystal's current and voltage. At frequencies below the crystal's resonant frequency the current leads the voltage and the phase goes to 90 degrees as the frequency separation continues to increase, see Figure 20. Above the resonant point the current lags the voltage and the phase go to minus 90 degrees. As the frequency increases through the resonant frequency, the phase goes from plus 90 through 0 to minus 90. It is interesting to note that the phase angle is 45 degrees when the VCO frequency is one half of the crystal's bandwidth above or below the crystal's resonant frequency.

The output of the phase detector is fed into an integrator. The integrator accumulates the phase error such that any positive phase error causes the integrator output to climb; a negative phase causes the integrator output to fall. With zero phase error the Integrator output holds steady.

The integrator output is connected to the VCO. Thus, if the VCO frequency is initially below the crystal resonant frequency, the phase will be positive, producing a positive output at the phase detector. This causes the Integrator output to climb, which causes the VCO frequency to increase. When the VCO frequency matches the resonant frequency of the crystal the phase will decrease to zero, the phase detector output will go to zero, the Integrator output will hold steady and the VCO frequency will be "locked" to the crystal's resonant frequency.

If the crystal's resonant frequency moves up or down, a phase difference between the crystal voltage and current will develop, producing a phase detector output. The non-zero phase detector output will drive the Integrator output up or down until the phase is zero once again, thus keeping the VCO frequency locked to the crystal's resonant frequency.

Once the frequency of the VCO is locked to the series resonant frequency of the crystal, the in-phase component (at zero phase error, there is no out of phase component) of the crystal current is demodulated to a DC voltage. The amplified output of the demodulator is provided at the Conductance output.

1.1 FEATURES

1.1.1 VERY WIDE FREQUENCY RANGE

The PLO-10 supports a wide frequency range from 3.8 to over 6 MHz. It will support both 5 and 6 MHz crystals; and with a low limit of 3.8 MHz it will support 1.2 MHz of frequency shift on a 5MHz crystal. Also, available is the PLO-10-2 to support higher frequency crystals. Its frequency range is 5.1 to over 10 MHz.

1.1.2 SUPPORT FOR VERY LOW Q, HIGHLY DAMPED, CRYSTALS

The PLO-10 will reliably lock to crystals with resistance of 5 KΩ or less. In most cases it will maintain lock up to a resistance of 10 KΩ. It will support crystal oscillation in highly viscous solutions of more that 88% glycol in water.

1.1.3 DIRECT REAL TIME MEASUREMENT OF CRYSTAL RESISTANCE

The PLO-10 provides a dc voltage output that is proportional to the crystal’s conductance. Conductance is the inverse of resistance. Based on the measured conductance output voltage, the crystal resistance is easily calculated.

1.1.4 ELECTRODE CAPACITANCE CANCELLATION

The total quartz crystal impedance includes a shunt capacitance (due to the capacitance of the crystal electrodes and holder) in parallel with the series resonant arm. The total current through the crystal is the sum of the current through the shunt capacitance plus the current through the series resonant arm. The physical motion of the crystal is reflected in the values of the L, R and C in the series arm of the crystal only, and therefore we want to subtract out or otherwise cancel the current through the shunt electrode capacitance. The INFICON PLO includes a method of canceling the electrode capacitance insuring that the measured crystal current does not include the current through the electrode capacitance and therefore is essentially the current through the series resonant arm of the crystal only.

1.1.5 “AUTOLOCK”

When the PLO-10 loses lock, the VCO is ramped up to the maximum frequency at which time it is automatically reset to the minimum frequency and a new scan is initiated.

To insure that the VCO ramps up in frequency, a small amount of quadrature current is injected into the current to voltage buffer whenever the PLO is unlocked. This current is equivalent to a shunt capacitance of about 1.5 pfd. As soon as lock is detected, the quadrature current is turned off.

1.1.6 CRYSTAL FACE ISOLATION (PLO-10i Models only)

The PLO-10i Models provide transformer isolation of the crystal front face electrode. This feature allows user to connect the crystal face to an electrochemical instrument such as a potentiostat.

1.2 CHARACTERIZING THE PLO

1.2.1 FREQUENCY ERRORS

The first thing we want to know regarding the performance of the PLO, is "What is the magnitude of the frequency error we can expect from the PLO-10?"

In any oscillator and sensing crystal system, the error in the frequency measurement, is a function of both the oscillator and the sensing crystal. The same is true for phase locked loops. Any phase error will introduce a frequency error and this frequency error will be inversely proportional to the sensing crystal's Q. These errors are over and above any change in crystal frequency due to temperature changes.

There are four important parameters that determine the frequency error of the PLO and sensing crystal system or indeed, any oscillator and sensing crystal system. The first two, the zero phase error and the electrode capacitance cancellation errors, are characteristics of the PLO. The second two are characteristics of the crystal, the Q of the crystal and the conductance (1/resistance) of the crystal.

1.2.2 FREQUENCY ERROR DUE TO PHASE ERROR

Given some finite zero phase error, the resulting frequency error depends on the sensing crystal's Q, the higher the Q, the lower the error. For phase errors below 10 degrees the frequency error is 0.087 PPM per degree for crystals with a Q of 100,000. Thus a one degree phase error in the PLO results in a 0.44 Hz frequency error for a 5MHz crystal with a Q of 100,000. For a 5 MHz crystal with a Q of 10,000, the error is 10 time greater or 4.4 Hz per degree.

$$\text{Frequency Error/degree} = df/f = PI/(360*Q)$$

1.2.3 FREQUENCY ERROR DUE TO IMPERFECT CAPACITANCE CANCELLATION

The effect of imperfect electrode capacitance cancellation can also be viewed as an equivalent phase error. This error is directly proportional to crystal resistance. The equivalent phase error due to a non-zero shunt capacitance equal to 1 pfd is one degree for a crystal with a series resistance of 556 Ω. Since the equivalent phase error is proportional to the crystal resistance, a 1-pfd residual capacitance error will result in a 10-degree equivalent error for a sensing crystal with a resistance of 5.56 KΩ.

Polar Plot of Crystal Conductance

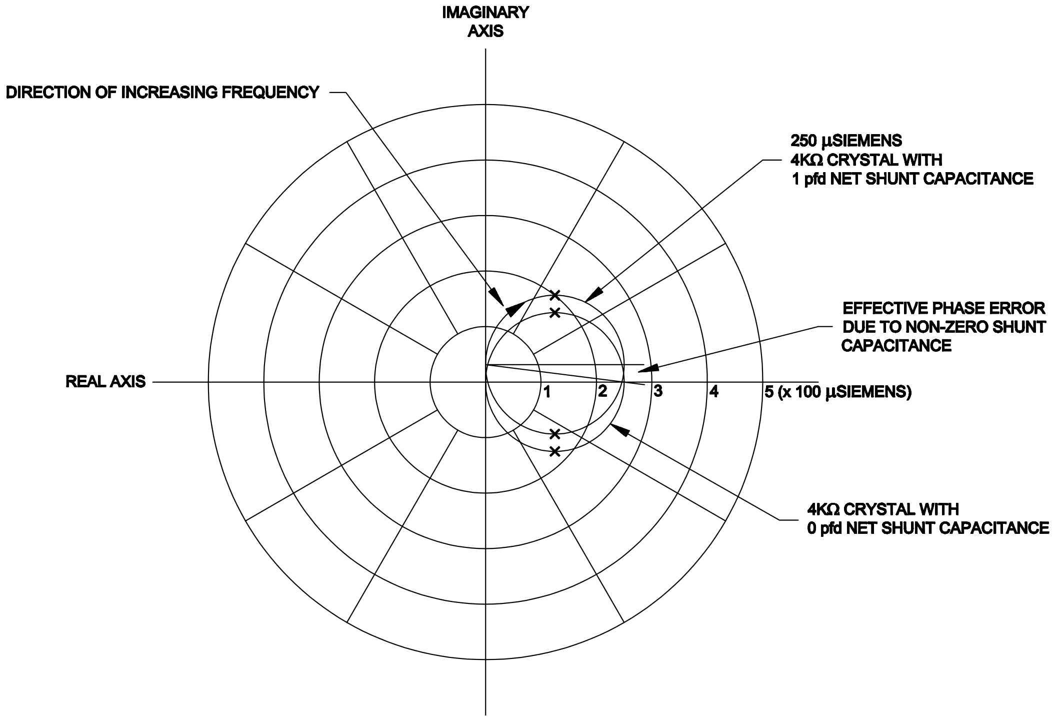


Figure 1 Equivalent Phase Error Due to Imperfect Capacitance Cancellation

1.2.4 CONDUCTANCE ERRORS

Conductance measurements are meaningful over the range of 0.0001 to 0.04 siemens (a crystal resistance of 10 KΩ to 5 Ω). Two characteristics of the PLO limit the range of the conductance measurement. The first is the zero drift of the demodulator and amplifier and determines the minimum measure-able conductance. This drift can amount to 0.00005 siemens. The second characteristic is the non-zero source impedance of the crystal drive voltage. This source impedance, 20 Ω, appears in series with the crystal resistance and the conductance output is proportional to the conductance of the crystal and source combination. The equation for crystal resistance is:

Rcry = (100/Vcond) -20

1.3 SPECIFICATIONS

Frequency range:	3.8 to 6.06 MHz, or 5.1 to 10 MHz
Capacitance compensation range:	40 to 200 pfd
Achievable capacitance cancellation:	± 0.3 pfd
Crystal conductance range:	0.2 down to 0.0002 siemen
Crystal resistance range:	5 Ω to 5.0 KΩ
Phase angle accuracy:	± 2 degrees
Phase angle stability:	± 0.5 degrees
Frequency error vs. phase error and crystal Q:	Q= 100,000 0.087 ppm per degree Q=10,000 0.87 ppm per degree Q=1,000 8.7 ppm per degree
Conductance output range:	0 to 40 millisiemen
Conductance output scaling:	100 volt/siemen
Conductance output accuracy:	± 5 % ±50 microsiemen.
Operating temperature range:	0 to 50°C
Operating temperature range for stated stability:	20 ± 10°C
Controls:	Reset Switch Capacitance Adjustment Trimmer, Course and Fine
Indicators:	Green “Lock” LED Red “Unlock” LED Yellow “Sweep Rate” LED
Crystal Drive Voltage, open circuit:	125 mV rms
Crystal Drive Source Impedance:	20 Ω ± 1%
Crystal Power:	200 µW, maximum
Crystal Isolation (PLO-10i):	Transformer, 25 Vdc maximum
Frequency Output Level:	4 Vp-p
Frequency Output Source Impedance:	50 Ω
Conductance Output Level:	0 to 4 Vdc
Conductance Output Source Impedance:	1 KΩ
Power:	12 to 15 Vdc @ 150 mA
Size:	1.6” W x 3.2” H x 4.8” D
Weight (shipping):	3 lbs.

1.4 ACCESSORIES

Part Number	Description
172205	CHT-100 Crystal Holder, Teflon®, SMB Connector
173205	CHC-100 Crystal Holder, CPVC, BNC Connector
184204	CHK-100 Crystal Holder, Kynar®, SMB Connector
828007	Cable, SMB Plug-SMB Plug,1' length, RG174A/U coax
888023	Adapter, BNC Male to SMB Jack
888026	Adapter, BNC male to binding posts
803081	Power Cord
803312	Capacitance Tuning Tool
900037	Power Supply, 100-250VAC to 12VDC

Refer to INFICON Price List for more accessories and other products.

2 UNDERSTANDING AND SETTING UP THE INFICON PLO-10

There are several LED's on the PLO-10 to indicate its operation.

The green, Lock LED is **on** when the frequency is locked to a connected crystal's resonant frequency.

The Red, Unlock LED will be **on** whenever the frequency is not locked.

The Yellow, Sweep Rate LED flashes each time the frequency ramp is reset to its low starting point.

The Reset switch allows you to force the VCO to its lowest frequency independently of the Integrator output. The Reset switch also forces the Lock LED on, thus turning off the quadrature current injection. The Quadrature current injection must be off to properly adjust the capacitance cancellation. To insure that the VCO ramps up in frequency, a small amount of quadrature current is injected into the current to voltage buffer whenever the PLO is unlocked. This current is equivalent to a shunt capacitance of about 1.5 pfd. As soon as lock is detected, the quadrature current is turned off.

2.1 NORMAL OPERATION

The PLO-10 comes set up for operation with a INFICON cable and crystal holder. If a INFICON cable and crystal holder is being used, then no initial adjustments should be needed.

During normal operation with a crystal installed and connected to the oscillator, the green Lock LED will be on and the frequency output will reflect the crystal resonance. The red Unlock LED will be off.

If the Unlock LED is on, the Sweep Rate LED should slowly flash. Continuous sweeping of the frequency range indicates that the crystal's resonant frequency is outside of the PLO's frequency range or the crystal's conductance is below the conductance threshold.

No flashing of the Sweep Rate LED when the Unlock LED is on can mean one of two things. First, if the VCO frequency is sitting at its low limit, it means the electrode capacitance is over compensated. Second, in some cases, even though the crystal conductance has fallen below the threshold necessary to indicate lock, the internal signals are still sufficient to keep the VCO locked to the crystal. In that case, the PLO really is locked and the VCO frequency will be sitting at the crystal frequency somewhere between its minimum and maximum frequencies.

If the VCO frequency is sitting at its low limit, press and hold the Reset switch and adjust the fine capacitance trimmer a few degrees clockwise (not more than ten) until the Reset LED begins to flash.

2.2 CHECKOUT

Make sure the wall mount power supply is specified for the voltage in your lab (120/240 volts).

Connect a frequency counter to the Frequency Output.

Connect a voltmeter to the Conductance output. The center conductor on the BNC connector is positive with respect to ground.

Connect the crystal holder, with a crystal installed, to the PLO by means of the 12-inch coax cable.

Plug the wall mount power supply into the wall and plug the power plug into the PLO-10.

Refer to Figure 2, Figure 3, and Figure 4 for a complete system connections.

The green, Lock, LED should come on, the frequency should indicate the correct crystal frequency and the voltmeter should indicate something between 5 millivolts and 4 volts.

Check the capacitance cancellation by pressing and holding the Reset switch. The green, Lock LED should light. Keeping the Reset switch pressed, adjust the fine capacitance trimmer clockwise by about 5 degrees. The yellow, Reset LED should flash. Back the trimmer counterclockwise to the point where the Reset LED just stops flashing. The capacitance cancellation should be checked and readjusted every time the environment of the crystal and holder is changed. For example, if the crystal and holder are moved from air to liquid or liquid to air, the capacitance cancellation should be checked and readjusted.

Remove the crystal. The red, Unlock, LED should light. The green, Unlock, LED should go off. The Sweep Rate LED should not flash. If the Sweep Rate LED flashes the capacitance is under compensated.

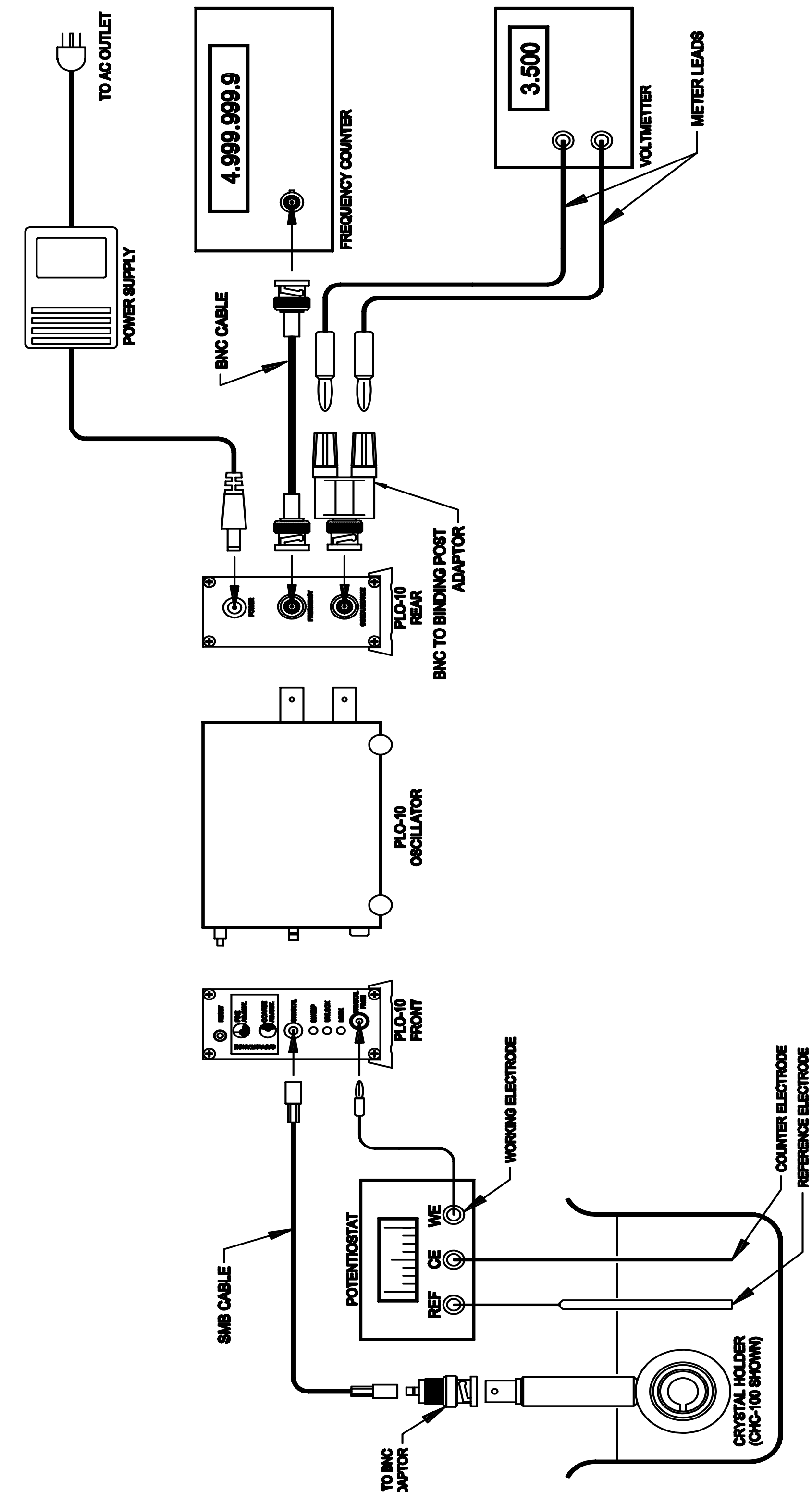


Figure 2 System Connections

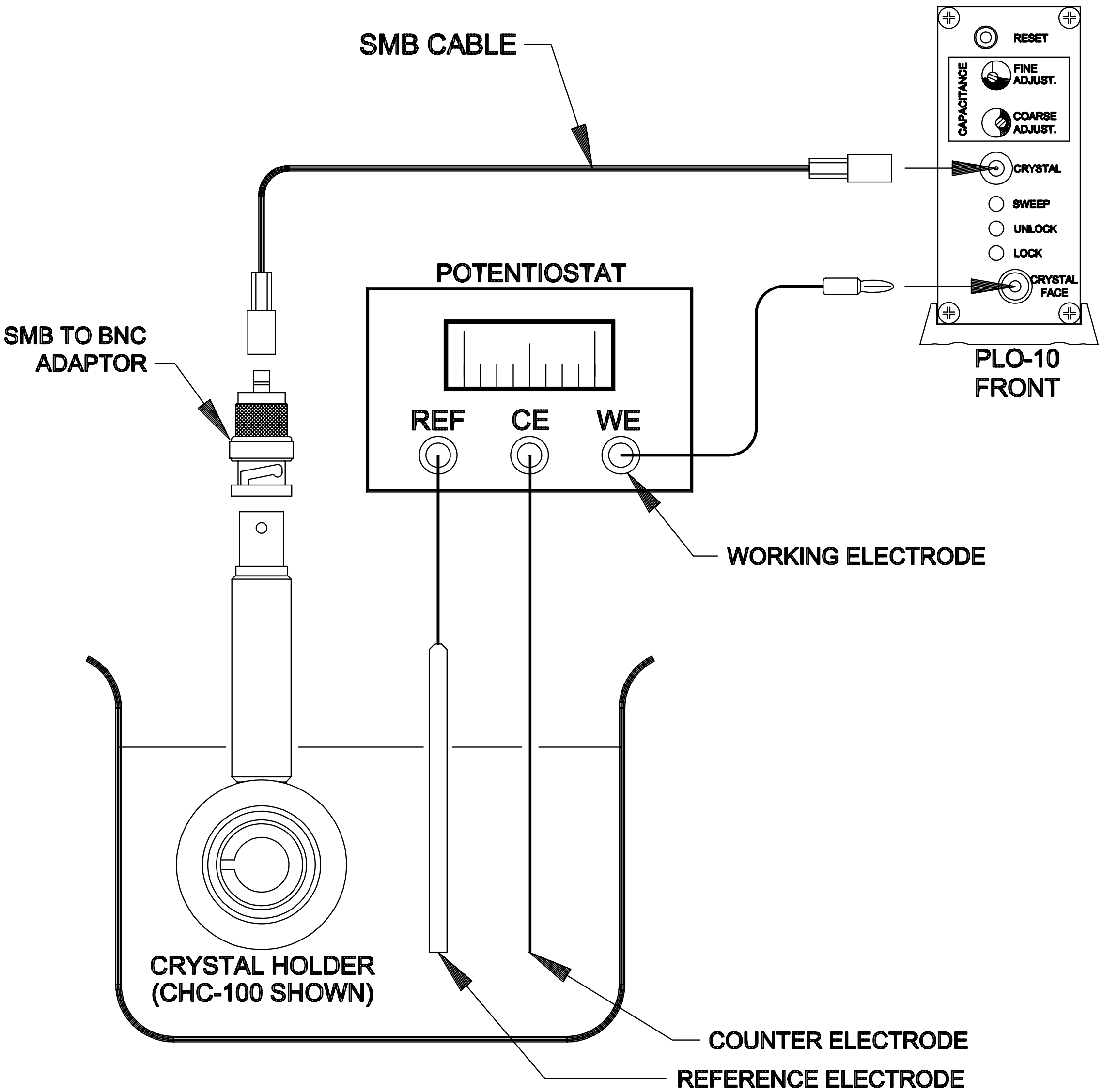


Figure 3 Front Connections

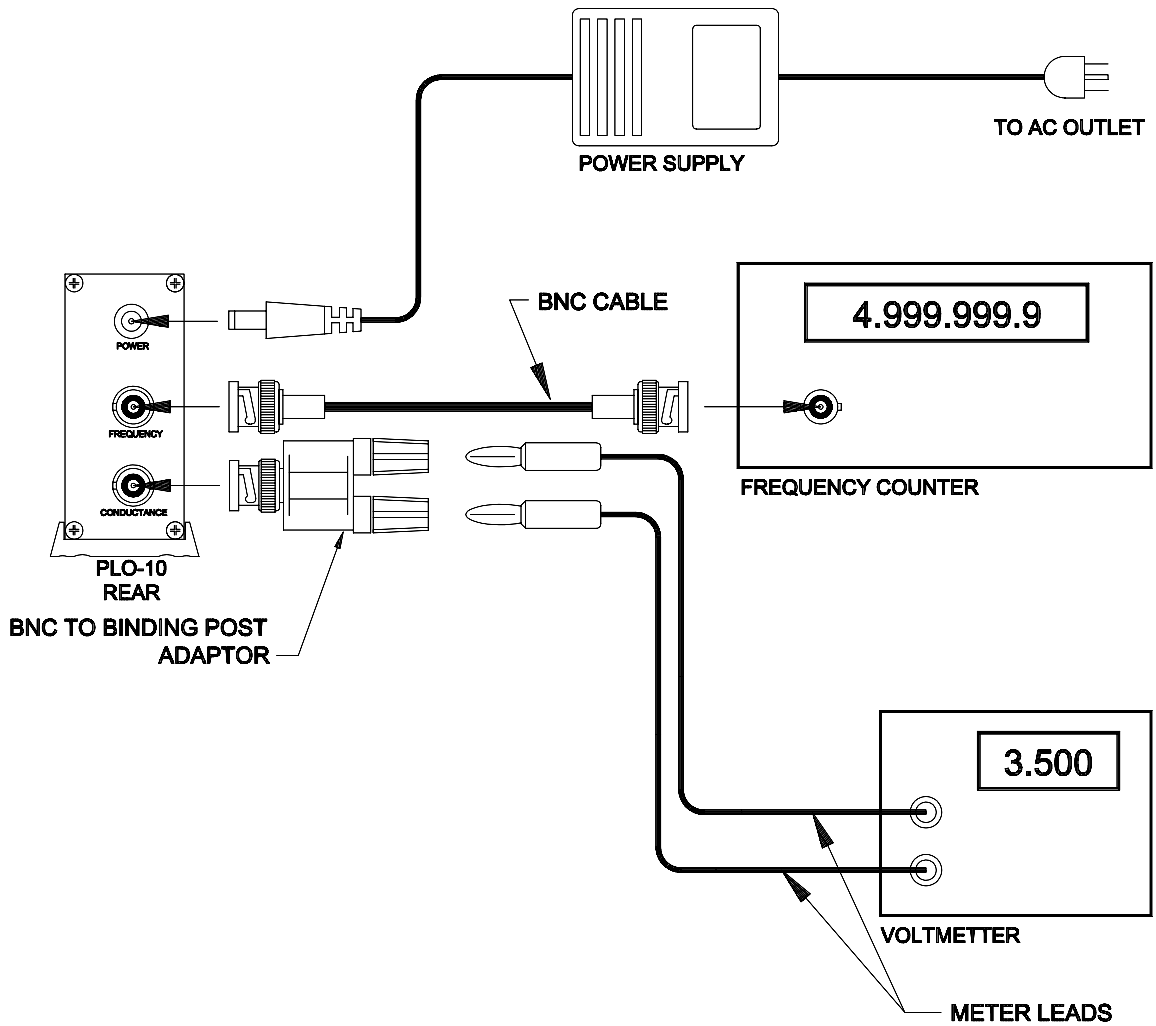


Figure 4 Rear Connections

3 CALCULATING CRYSTAL RESISTANCE

The PLO-10 provides a dc output voltage proportional to conductance. Conductance is the inverse of Resistance. Thus, Conductance = 1/Resistance or Resistance = 1/Conductance. The units of resistance are ohms (volts per ampere) and the units of conductance are siemens (amperes per volt). The PLO-10 Conductance Output is inversely proportional to the sum of the crystal resistance plus the Crystal Drive Voltage source resistance. Thus,

$$\text{Conductance Output} = 100/(R_{cry} + R_{source}).$$

The Conductance Output scaling is 100 volts per siemen. Solving for R_{cry},

$$R_{cry} = (100/\text{Conductance in volts}) - R_{source}$$

The Crystal Drive Source Resistance is 20 Ω, so

$$R_{cry}\text{, in ohms} = (100/\text{Conductance, in volts}) – 20\ \Omega$$

Examples:

1. Conductance output voltage = 1.000 volt.

   R_{cry}, in ohms = (100/1.000) – 20 = 80 Ω

2. Conductance output voltage = 0.015 volts.

   R_{cry}, in ohms = (100/0.015) – 20 = 6667 – 20 = 6647 Ω = 6.647 KΩ

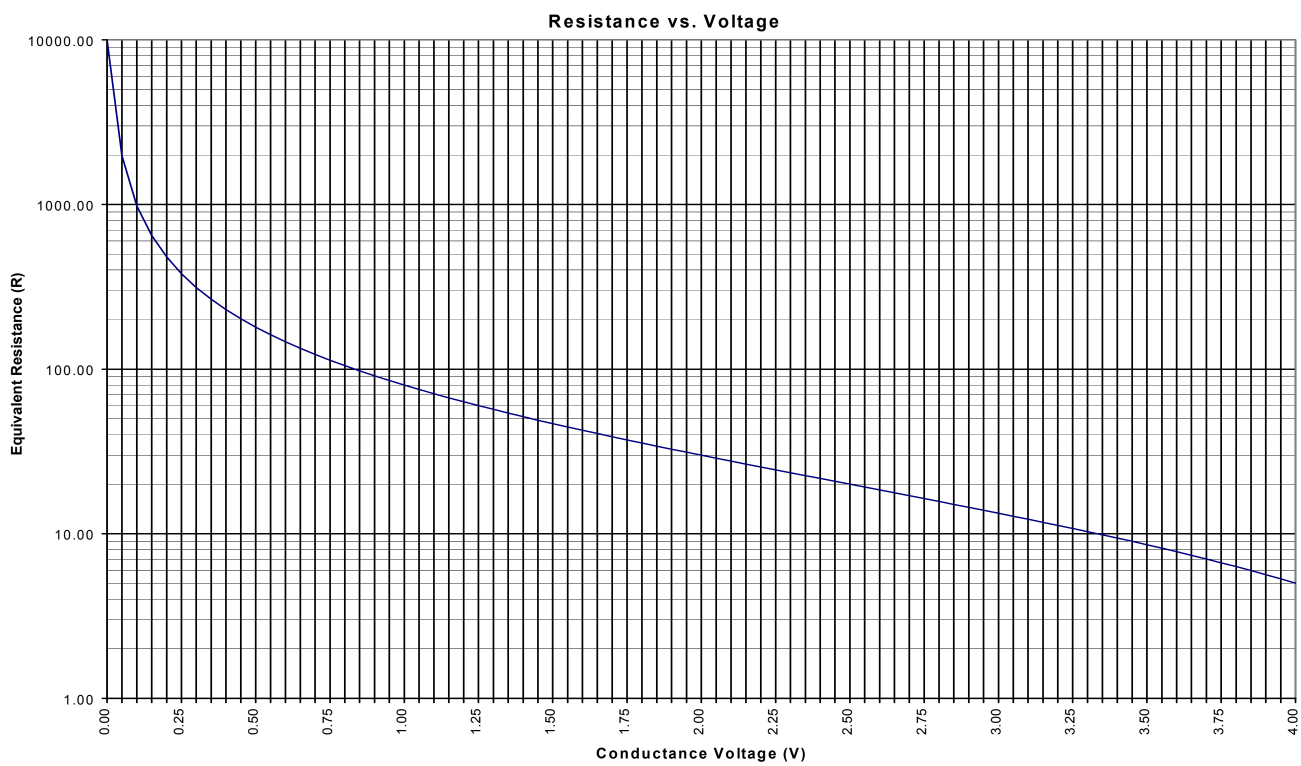


Figure 5 Resistance vs. Conductance Voltage

4 ADJUSTING THE CAPACITANCE CANCELLATION

Proper adjustment of the Capacitance Cancellation is critical in obtaining accurate results with high resistance crystals. See Section 1.2.3 FREQUENCY ERROR DUE TO IMPERFECT CAPACITANCE CANCELLATION. The cancellation adjustment should be performed with the crystal holder and crystal in the measurement environment. For instance, if liquid measurements are to be made insert the crystal and its holder into the liquid where the measurement will be made.

With the crystal and holder in the measurement environment, press and hold the Reset switch. Pressing and holding the Reset switch forces the VCO to its minimum frequency, turns on the Lock LED, and turns off the quadrature current injector. Forcing the VCO to its minimum frequency insures that the crystal is being driven at a frequency far from its resonant frequency where its impedance is essentially due only to the shunt electrode capacitance. With the quadrature current injector turned off, the measured current is due only to the net shunt capacitance. The measured net shunt capacitance is the capacitance of the cable, holder and crystal electrodes minus the compensation capacitance. If the capacitance is under compensated, the phase of the measured current leads the voltage, (a phase angle of plus 90 degrees). If the capacitance is over compensated, it lags the voltage, (a phase angle of minus 90 degrees).

The Yellow Sweep LED is used to determine whether the crystal capacitance is over compensated or under compensated. The Sweep LED flashes whenever the crystal capacitance in under compensated.

If the Sweep LED is not flashing, turn the fine compensation clockwise until it begins to flash then back up until it just stops. If it is flashing, turn the fine adjustment counter clockwise until it just stops flashing. This is a very fine adjustment. Go back and forth until you are sure you are right on the edge. The sensitivity of the fine adjustment is approximately 0.05 pfd per degree. In situations where the crystal resistance is very high (over 1 KΩ) a net capacitance of over 0.5 pfd can result in a significant frequency error so try to get this adjustment to within a couple of degrees. Remember to keep the Reset switch depressed while making this adjustment.

4.1 ADJUSTING CAPACITANCE CANCELLATION TRIMMER CAPACITORS

Setting up the capacitance cancellation is fairly straightforward. The thing to remember is that there are two variable capacitors, a course and a fine with the total compensation capacitance being the sum of the two. These trim capacitors have no stops so it’s not obvious when they are at their minimum or their maximum.

The capacitors have circular, rotor plates that mesh into fixed stator plates. The capacitance is at a maximum when the plates are fully meshed and a minimum when rotor plates are above the stator plates and not meshed. As the capacitors are rotated clockwise they go through a full cycle from maximum to minimum and back to maximum. Or, depending on where you start they may go first toward a minimum, then to a maximum and then back toward a minimum. To avoid confusion, we always want to be turning clockwise as we approach the desired capacitance and we want the capacitance to be decreasing.

If you are using a crystal holder and cable supplied with your PLO-10 then you should not have to change the course adjustment. Connect the cable and crystal holder to the PLO but don't install a crystal.

If the Sweep LED is flashing, press and hold the Reset button and then turn the fine trimmer counter clockwise until it just stops flashing. Go back and forth a few times to get a feel for the point where the Sweep LED just stops flashing. Release the Reset button and the Sweep LED should begin to flash again.

Install a crystal. The PLO should lock. Even so, press and hold the Reset button and again adjust the fine trimmer to the point where the flashing just stops. The capacitance cancellation adjustment is now perfect. Remember to check this adjustment whenever the crystal holder is moved or changed to a new environment.

If you could not find the proper zero capacitance point using the fine trimmer alone, then we have found the following approach which is best for adjusting the coarse trimmer.

First adjust the fine trimmer so that it is 50% meshed and the rotor plates are below the shaft with the oscillator upright. You can see these plates through the oversize adjustment hole. See Figure 6. Next connect a cable and crystal holder, if you haven't already done so. Don't install the crystal at this point.

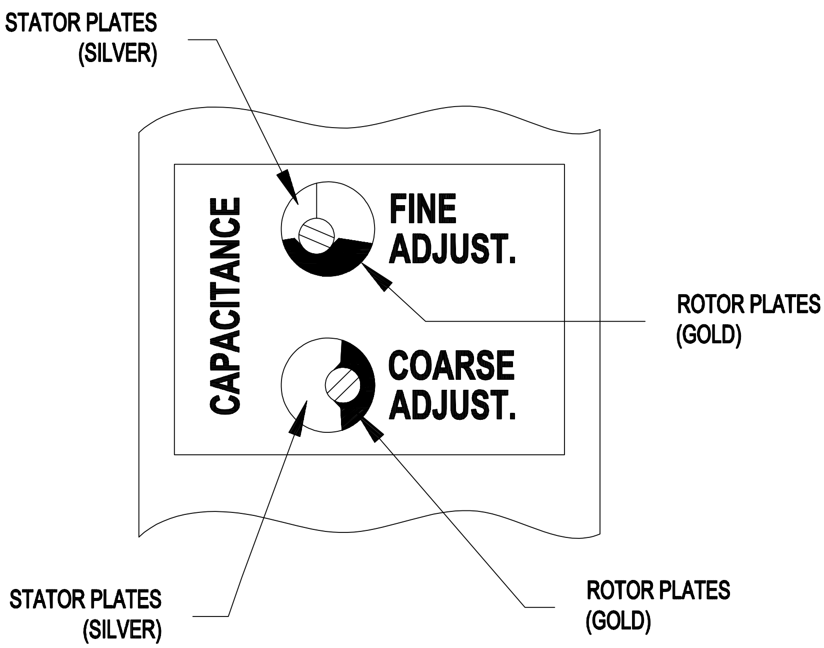


Figure 6 Capacitance Adjustments

Do not press the reset button, now slowly turn the course trimmer clockwise while watching the Lock and Unlock LED's. The green, Lock, LED will come on when the capacitance is grossly out of adjustment. Continue turning the course trimmer clockwise until the Unlock LED comes on. The adjustment is getting close. Press and hold the reset button, *Slowly* continue to turn the trimmer clockwise until the yellow, Sweep, LED begins to flash. If you continue to turn clockwise the Sweep LED will cease flashing, but this is not the point you want. Back off the course adjustment until the flashing begins again, then continue to the point where the flashing just stops. The course adjustment is now complete.

Install a crystal into the holder. Now depress and hold the Reset button. Slowly adjust the fine trimmer clockwise until the flashing of the Sweep LED begins again and then back off until it just stops. The capacitance compensation adjustment is now complete. Release the Reset button and assuming the crystal is not dead or out of range, the PLO-10 will lock on it.

4.2 WORKING WITH VERY LOW Q CRYSTALS

Very low Q crystals require very close adjustment of the compensating capacitance to insure a successful lock. To adjust the compensation capacitance one pushes the Reset button and adjusts the capacitance to the point where the Sweep LED just ceases to flash. With very low Q crystals, the PLO may not lock upon release of the Reset button. The Unlock LED will be on and the Sweep LED will be flashing. This is normal. Even so it may be possible to lock on the crystal by slowly adjusting the fine capacitance counterclockwise until the Sweep LED again ceases to flash. Lock is evidenced by the Lock LED turning on or by a value of greater than 8 millivolts at the Conductance output.

Once lock is achieved the true series resonant point can be found by adjusting the capacitance for maximum conductance. The limits of the crystal bandwidth can be determined by adjusting the capacitance and reading the maximum frequency and the minimum frequency just before the PLO loses lock.

5 FREQUENCY ERRORS DUE TO IMPERFECT CAPACITANCE CANCELLATION

There are two reasons that proper capacitance cancellation is so important with high resistance crystals.

The first is that to a first approximation, the frequency error resulting from a given phase error is proportional to the bandwidth of the crystal. The bandwidth of the crystal is proportional to the crystal's resistance. A ten-ohm crystal might typically have a bandwidth of 42 Hz, while a one thousand-ohm crystal will have a bandwidth of 4,200 Hz. A five thousand-ohm crystal will have a bandwidth of 21,000 Hz. Since the frequency error for a given phase error is proportional to the bandwidth, a phase error that would result in a 0.5 Hz frequency error in a ten ohm crystal will cause a 50 Hz error in a one thousand ohm crystal and 250 Hz error in a five thousand ohm crystal.

The second reason is that the effective phase error caused by a non-zero net quadrature current is inversely proportional to the real current, which is inversely proportional to the crystal resistance. In other words, the effective phase error is proportional to the crystal resistance. For instance, a net unbalance of 1 pfd leads to an effective phase error of 0.02 degrees for a ten ohm crystal, but it leads to a 2 degree error for a one thousand ohm crystal and a 10 degree error for a five thousand ohm crystal.

Examples:

A ten-ohm, 5 MHz crystal will have a Q (Quality Factor) of about 120,000. The bandwidth is equal to the crystal frequency divided by Q. Thus, the bandwidth of this crystal would be about 42 Hz. To a first approximation, near zero phase, the frequency error per degree of phase error is given by the following formula,

Frequency Error = -½(Phase Error, in radians)(Bandwidth)

Or,

Frequency Error = -(1/(2*57.3))(Phase Error, in degrees)(Bandwidth)

For the above ten-ohm crystal, the frequency error caused by a one-degree phase error is 42/114.6 or approximately 0.37 Hz. For a one thousand-ohm crystal, one degree of phase error results in a 37 Hz error and for a ten thousand-ohm crystal the frequency error is 370 Hz per degree of phase error.

Now, the effective phase error caused by a non-zero quadrature (imaginary) current is given by the following formula,

Effective Phase error = arctangent (imaginary current/real current)

And since current is proportional to conductance,

Effective Phase error = arctangent (imaginary conductance/real conductance)

The conductance of a one picofarad capacitor at 5 MHz is 31.4 microsiemens. The conductance of a ten-ohm crystal at resonance is 100 millisiemens.

Effective Phase error = arctangent ((31.4e-6)/(100e-3)) = 0.018 degrees

In other words a one picofarad capacitance unbalance will result in an effective phase error of only 0.018 degrees when measuring a ten-ohm crystal. However, when measuring a one thousand-ohm crystal the effective phase error will increase to 1.8 degrees and it will increase to 9 degrees when measuring a five thousand-ohm crystal.

Combining these two errors we can get an idea of the magnitude of the frequency error caused by imperfect capacitance cancellation.

For a 10 Ω crystal a one picofarad capacitance imbalance results in a 0.018 degree phase error and a 0.0067 Hz frequency error.

For a 100 Ω crystal, the phase error is 0.18 degrees and the frequency error is 0.67 Hz. For a 1000 Ω crystal, the phase error is 1.8 degrees and the frequency error is 67 Hz. For a 5000 Ω crystal, the phase error is 9 degrees and the frequency error is 1,635 Hz.

A two picofarad capacitance imbalance will result in approximately twice the above error.

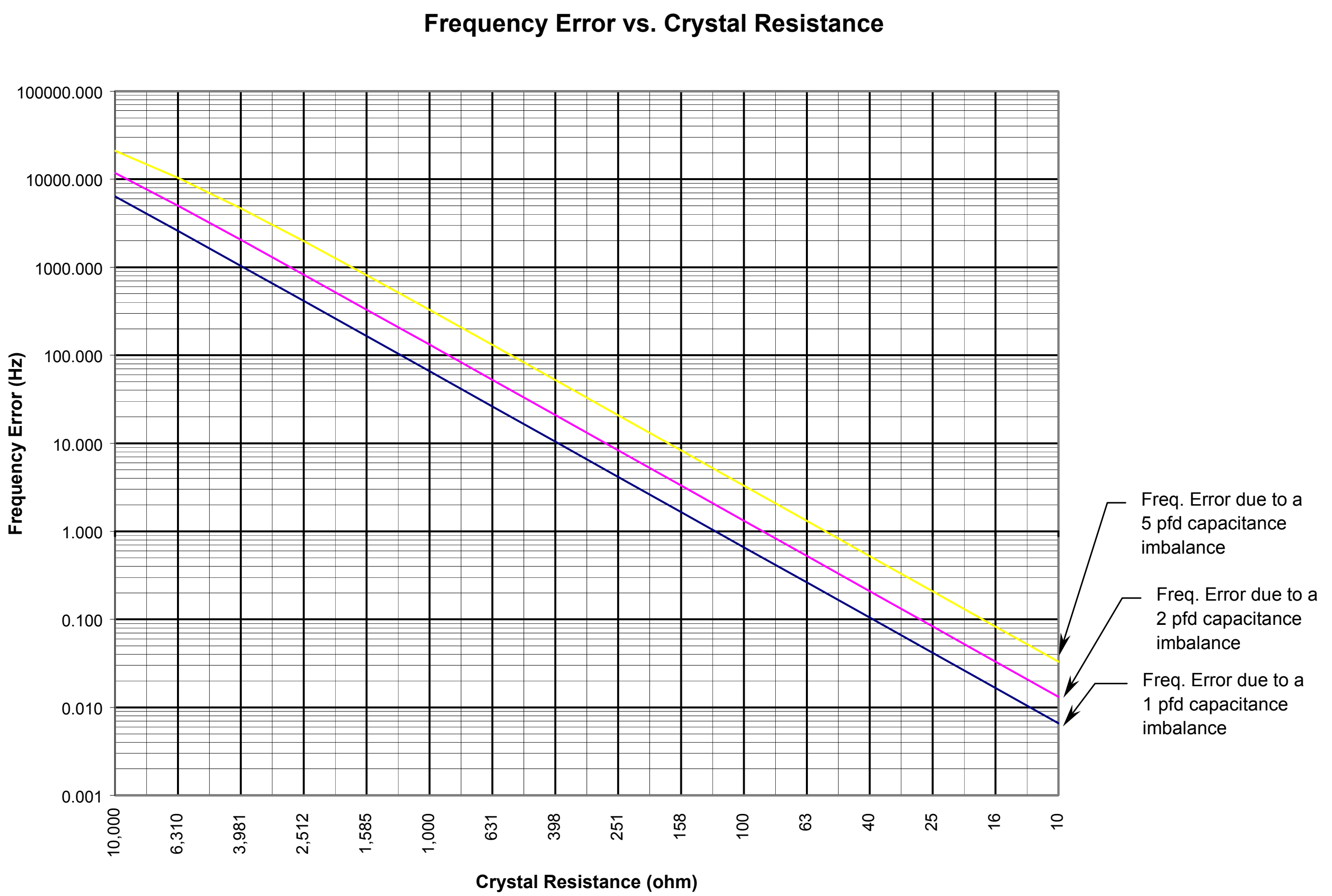


Figure 7 Frequency Error Due to Imperfect Capacitance Cancellation

6 CALCULATING CRYSTAL POWER

Using the PLO conductance voltage output, crystal power can be calculated. Refer to Section 3 CALCULATING CRYSTAL RESISTANCE, crystal resistance, $R_{cry,}$ is given as $(100/ V_{cond})-20$, where V_{cond} = Conductance Voltage.

Crystal power, $P_{cry} = i^2 * R_{cry}$

Since $i_{cry} = V_{soc}/(20 + R_{cry})$ or $(V_{cond} /100) * V_{soc}$

Hence, $P_{cry} = i^2 * R_{cry} = [(V_{cond} /100) * V_{soc}]^2 * [(100/ V_{cond})-20]$

$$P_{cry} = V_{soc}^2 * (V_{cond} /100) * [1-20(V_{cond} /100)]$$

V_{soc} = Voltage of source open circuit = 125 mV (open circuit source voltage crystal drive)

Then, $P_{cry} = 0.125^2 * (V_{cond} /100) * [1-20(V_{cond} /100)]$

Examples:

1. Conductance output voltage = 1.000 volt

   $P_{cry,\ in\ watts} = 0.125^2 * (1/100) * [1-20(1/100)] = 1.25E^{-4}$ watts or 125 μW

2. Conductance output voltage = 0.015 volts

   $P_{cry,\ in\ watts} = 0.125^2 * (0.015/100) * [1-20(0.015/100)] = 2.3E^{-6}$ watts or 2.3 μW

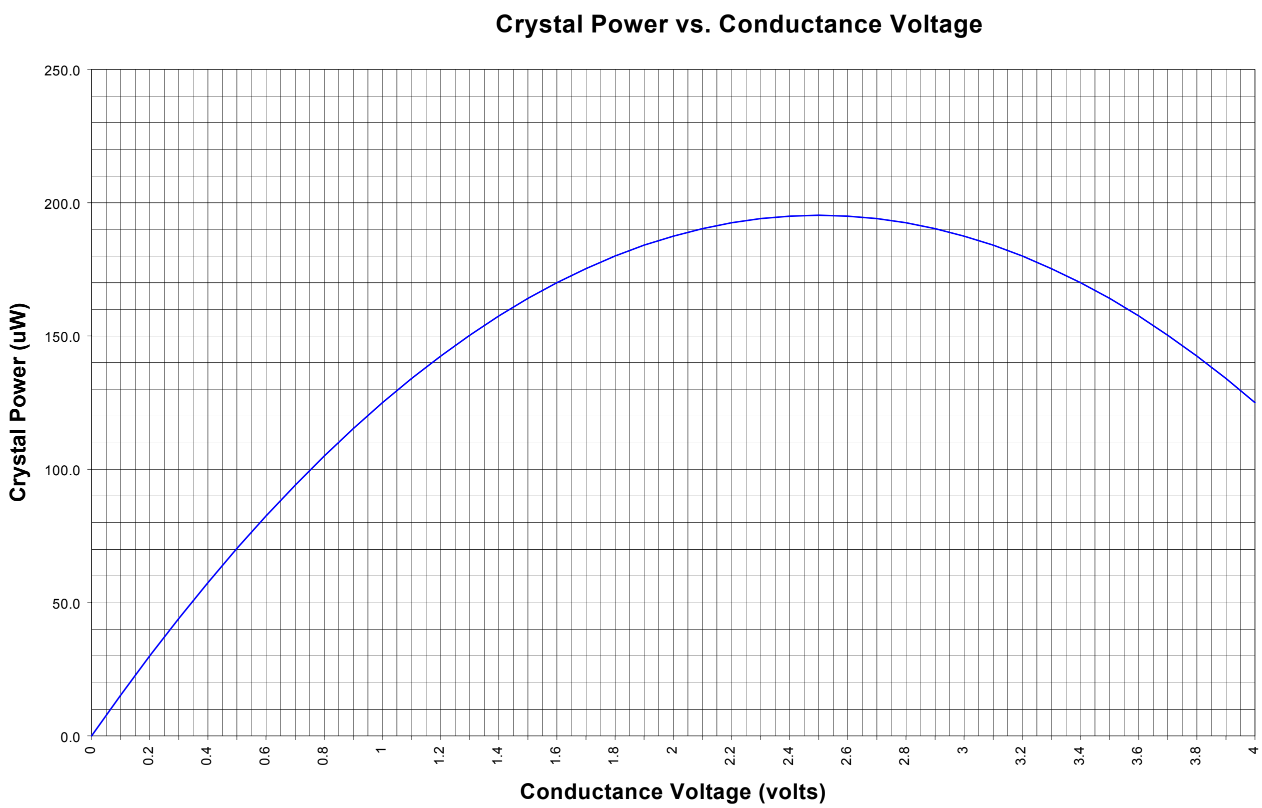


Figure 8 Crystal Power Dissipation vs. Conductance Voltage

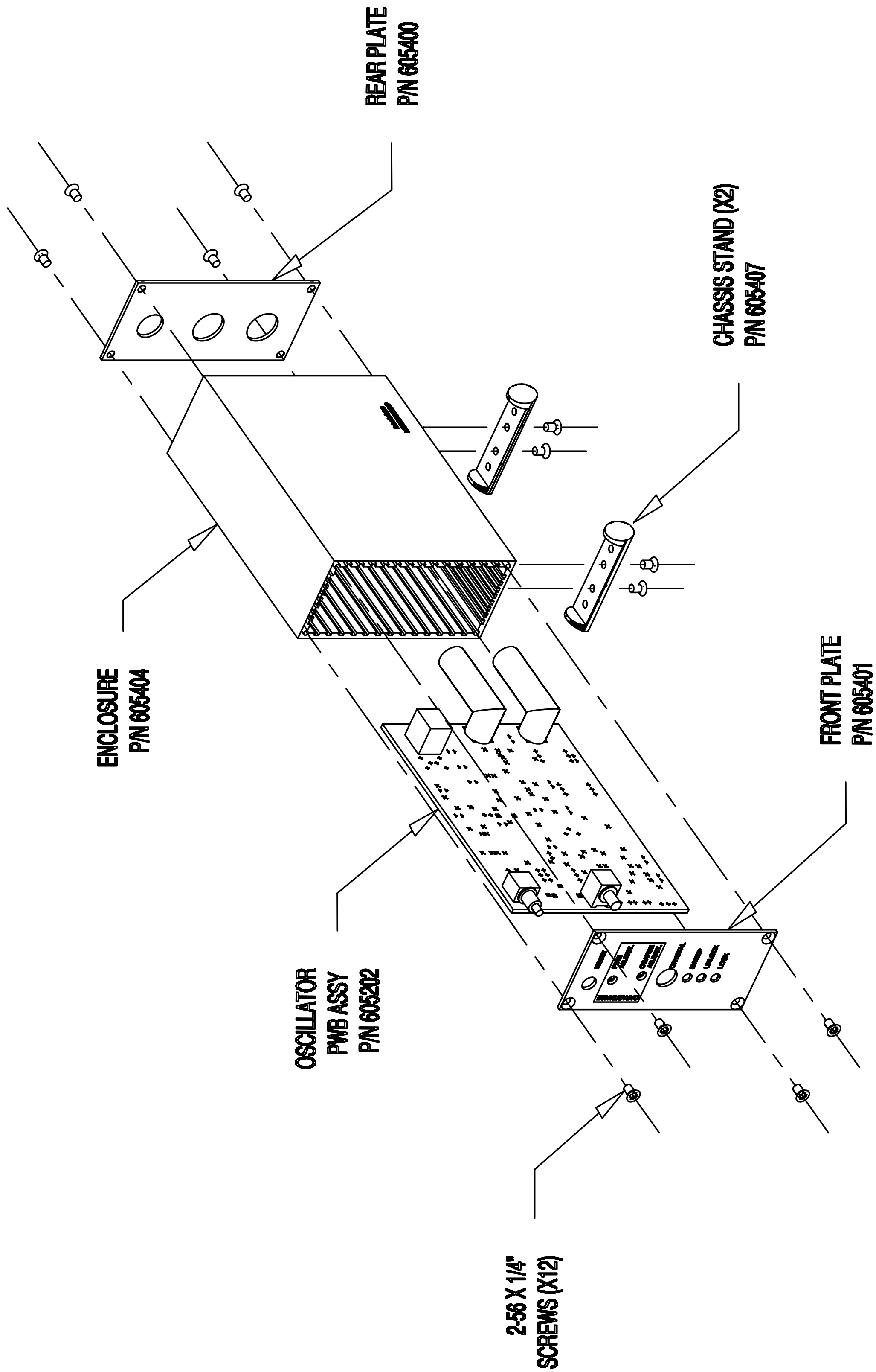


Figure 9 PLO-10 Assembly

7 CRYSTALS, HOLDERS AND FLOW CELL

An essential part of the PLO-10 Oscillator is the sensing crystal. Careful handling of both the crystal and the crystal holder must be observed to ensure proper and reproducible measurements. Furthermore, the sensing crystal, the crystal holder and the connecting cable must be orientated and connected correctly in order for the PLO-10 to work properly. This is especially true if you design your own crystal or holder.

If you have purchased a INFICON crystal, holder and cable, the installation is simple. Follow the instructions below.

If you plan to build your own crystal or holder or cable, see Section 7.3.2 HOLDER CARE AND HANDLING.

7.1 1 INCH DIAMETER CRYSTALS

INFICON pioneered the standard AT-cut, 5 MHz, 1-inch diameter crystals for use in liquid applications. The AT-cut quartz is chosen for its superior mechanical and piezoelectric properties, and the angle of cut can be adjusted to obtain a zero temperature coefficient at a desired operating temperature. The 1 inch diameter was chosen to allow enough distance between the active area of the crystal and the mounting o-ring. This improves the overall stability of the crystal by reducing the frequency changes due to mounting stress.

7.1.1 ELECTRODE CONFIGURATION

Figure 10 below shows INFICON's 1" crystal electrode patterns. The left figure shows the ½ inch diameter front electrode (also called sensing electrode) with an extended electrode that wraps around the edge of the crystal and extends into a semicircle shown in the top half of the right figure. The lower half of the right figure shows the ¼ inch diameter rear electrode (also called contact electrode).

This configuration enables both electrical contacts to be made on the backside of the crystal allowing measurement in conductive liquids.

The oversized front electrode (½ inch in diameter as oppose to the ¼ inch diameter rear electrode) was chosen to ensure a more consistence deposition across the active area of the crystal. The exposed area of the front electrode is 0.212 in^2 (137 mm^2), but the active oscillation region (displacement area) is limited to the overlapping area of the front and rear electrodes (0.053 in^2 or 34.19 mm^2).

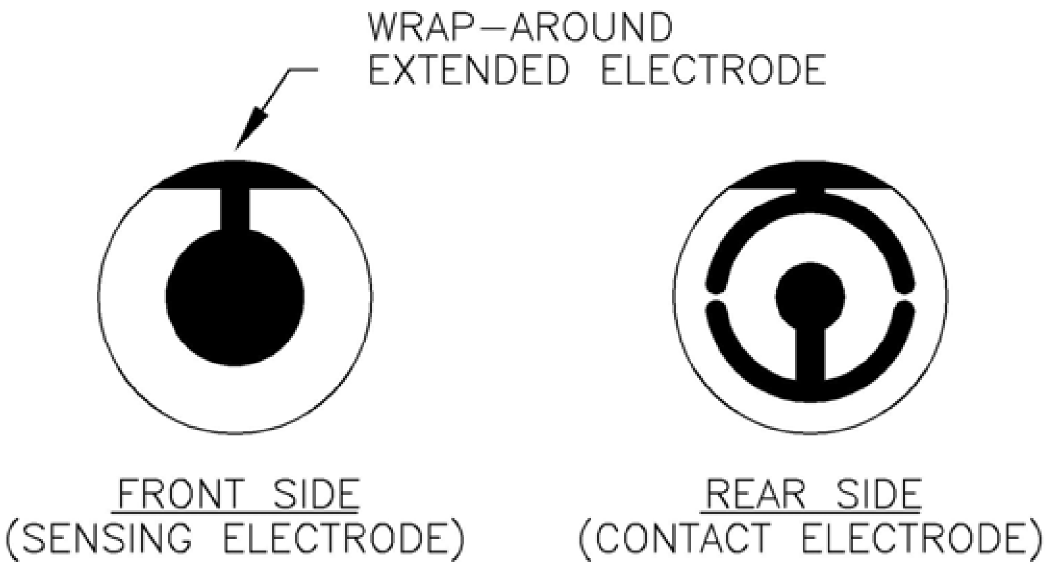


Figure 10 INFICON 1" Crystal – Electrode Configuration
The figure below shows a INFICON 1” diameter as seen from the front side.

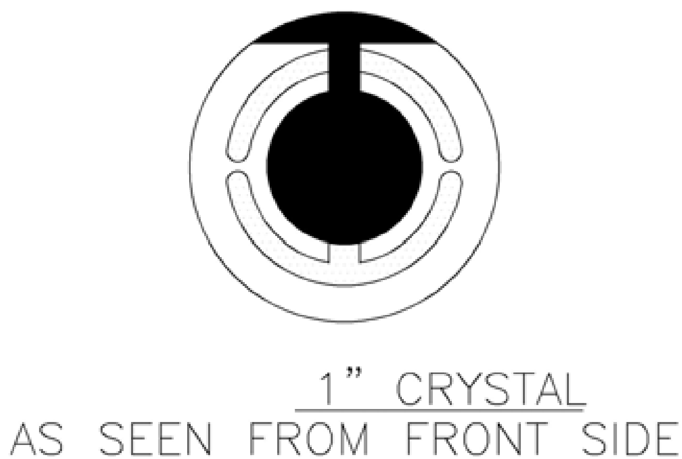


Figure 11 INFICON 1" Crystal - as Seen From The Front Side

7.1.2 CRYSTAL PARAMETERS

Polished one-inch diameter crystals that are commonly available for liquid work have the typical values as listed below.

Type	Frequency Range (MHz)	Electrode Material	Resistance (ohms)	Q Factor
5 MHz	4.976 – 5.020	Gold	~10	120,000
9 MHz	8.976 – 9.036	Gold	~7	55,000

7.1.3 CRYSTAL SURFACE FINISH

Studies have shown that electrode surface roughness can cause large apparent mass loadings due to the liquid that is trapped within pores at the crystal surface [1]. INFICON's crystals are optically polished to 50 Å average surface roughness to minimize this effect. Polished crystals are required to obtain good agreement between theory and measurement during liquid immersion experiments. Polished crystals are also required to obtain measurements reproducibility from crystal to crystal[2].

Non-polished crystals (R_a=1.8 microns) are also available at reduced costs for applications that do not require the accuracy and reproducibility of the polished crystals.

7.1.4 CRYSTAL ELECTRODE MATERIALS

INFICON's crystals are available in a variety of electrode materials including Gold, Platinum, Aluminum, Silver, Titanium, etc. INFICON also offers Gold electrode crystals with an additional SiO2 outer layer to create a hydrophilic surface needed for some biological applications.

7.1.5 CRYSTAL THICKNESS

INFICON AT cut, 1-inch diameter crystals are plano-plano. Their physical thickness is determined by a frequency constant and their final frequency. The frequency constant for an AT cut crystal is 1.668E5 Hz × cm or 65.5 kHz × in. Therefore, the crystal thicknesses for various frequencies are as follows.

5 MHz AT cut thickness = 333 microns (0.013 inch)

6 MHz AT cut thickness = 227 microns (0.0109 inch)

9 MHz AT cut thickness = 185 microns (0.007 inch)

7.1.6 MASS SENSITIVITY

The quartz crystal microbalance is an extremely sensitive sensor capable of measuring mass changes in the nanogram/cm^2 range with a wide dynamic range extending into the 100 µg/cm^2 range.

Sauerbrey was the first to recognize the potential usefulness of the technology and demonstrate the extremely sensitive nature of these piezoelectric devices towards mass changes at the surface of the QCM electrodes[3]. The results of his work are embodied in the Sauerbrey equation, which relates the mass change per unit area at the QCM electrode surface to the observed change in oscillation frequency of the crystal:

$$\Delta f = - C_f \times \Delta m$$

where

Δf = the observed frequency change in Hz,

C_f = the sensitivity factor of the crystal in Hz/ng/cm^2

(0.056 Hz/ng/cm^2 for a 5 MHz crystal @ 20° C)

(0.081 Hz/ng/cm^2 for a 6 MHz crystal @ 20° C)

(0.181 Hz/ng/cm^2 for a 9 MHz crystal @ 20° C)

Δm = the change in mass per unit area, in g/cm^2

The minimum detectable mass change is typically a few ng/cm^2 and limited by the noise specifications of the crystal oscillator and the resolution of the equipment used to measure frequency shifts. For example, the INFICON RQCM has a frequency resolution of 0.03 Hz @ 6 MHz, therefore, its minimum detectable mass change is 0.37 ng/cm^2.

The Sauerbrey equation relies on a sensitivity factor, C_f, which is a fundamental property of the QCM crystal. Thus, in theory, the QCM mass sensor does not require calibration. This ability to calculate the mass sensitivity from first principles is obviously a very attractive feature of these devices. However, it is very important to notice, that the Sauerbrey equation is only strictly applicable to uniform, thin-film deposits originating from a low pressure (i.e. vacuum) gas environment[21]. Thick deposits and operation in liquid environments or in contact with lossy films, relies on the use of more complex equations relating the frequency shifts to mass loading, and often requires calibration of the setup for accurate results. Several articles have been published on simple ways to calibrate the mass sensitivity of QCMs for electrochemical applications[4] and for vacuum thin-film deposition processes[5 6 7], and some useful calibration guidelines are also described herein.

Many studies have shown that the crystal's sensitivity is approximately Gaussian. The maximum sensitivity is in the center of the crystal and it tapers off towards the edge of the active area[8 9 10]. The mass sensitivity distribution has also been shown to become slightly more confined to the electrode region as the mass loading is increased.

7.1.7 STABILITY

A sensor crystal cannot distinguish the difference between a frequency shift due to deposited material or that due to other disturbances. Thus any extraneous factors, other than the deposited mass, which may cause the quartz crystal to change its resonant frequency, must be properly controlled. Factors that can influence the stability of a sensor crystal are categorized as follows[11]:

- The crystal itself: Improper design, localized stress, damage to the crystal
- The crystal holder: Improper seating of the crystal, large mechanical coupling between the crystal and the holder
- Thermal input: Radiation from evaporation source, radiation from substrate heater, bombardment by charge particles, energy released by condensates
- Stress: Thermal stress, stress release in the deposited materials
- Temperature: See section 7.1.9 for data on frequency versus temperature for INFICON's crystals.

Other factors that can affect stability are humidity, shock, vibration and change in pressure. Controlling those conditions is a must to insure accurate measurements of small mass changes over long periods of time.

7.1.8 CRYSTAL LIFE EXPECTANCY

It is difficult to predict the useful life of a crystal since it depends on many factors. Some of these factors are[11]:

- The quality of the quartz
- The amount of deposited material
- The stress generated in the crystal due to deposited material
- The acoustic losses in the deposited material
- The design of the oscillator circuitry

Other aspects that affect the crystal life include the type of the deposited material, splitting of source material resulting in non- uniform films, film flakes that landed on the crystal's active area, and of course, physical damage to the crystal such as chipping, cracking, or peeling of the electrode, etc.

In general, a sensor crystal can be used until its frequency drops below 50% of its uncoated value. However, for the reasons stated above, crystal failures often occur well before a 40% shift in frequency is reached.

The sensor crystals are considered expendable. However, a crystal may be reused up to 20 times on average in experiments that don't physically alter the crystal electrode. In experiments where a film is deposited, the crystal can be stripped using a chemical etchant . Care must be taken so only the deposited material is stripped and not the crystal electrodes. The amount of times that a crystal can be reused greatly depends on its condition after each experiment or stripping. Needless to say, careful handling and cleaning of the crystal is required to maximize its re-usability.

Noisy or erratic measurement indicates that the crystal is about to fail. It might even be difficult to obtain a stable baseline. Spurious signals might become evident in electrochemical QCM experiments. Visually, traces of consumption and wear can often be seen on the crystal surface. Edges of the sensor crystal might become cracked and the deposited film, even the electrode, starts to show scratches and tears.

The crystal motional resistance R does reflect the influence of deposited material on the performance of a crystal. This resistance is associated with the damping of acoustic waves by the electrodes, deposited materials, and the supporting structure. This resistance increases as more material is being deposited onto the crystal[11]. This resistance value can be used to determine when a crystal reaches a maximum loading.

7.1.9 TEMPERATURE COEFFICIENT

The temperature coefficient of quartz crystals is normally specified in units of parts per million per degree of temperature change. A one part per million change in frequency of the sensing crystal corresponds to an indicated thickness change of approximately 7.4 Å for a material with a density of 1.0 gm/cm^3. For Aluminum with a density of 2.7 gm/cm^3, this is equivalent to approximately 2.7Å. This intrinsic dependence of resonance frequency of a sensor crystal on temperature is generally small in experiments in gas phase when operating at or near its “turn-around-point”. The “turn-around-point” is where the temperature coefficient of the crystal is zero. That is, there is no change in resonance frequency due to a change in the temperature of the crystal at the turnaround point. INFICON 1 inch crystals are optimized for two operating temperatures namely 90ºC and 25ºC. These crystals have very good temperature stability when operating close to their specified temperature.

Even though AT cut crystals are designed to minimize the change in frequency due to temperature, the effect of temperature can be significant when attempting to resolve small mass (frequency) changes over long periods of time. This frequency change due to temperature is magnified when the sensor crystal is submerged in liquids. This is due to the coupling of the shear mode oscillation with the temperature dependent viscosity and density of the fluid. For experiments in liquid phase in which the frequency is to be monitored over long periods of time, the temperature must be controlled to at least 0.1°C, and preferably better. In electrochemical experiments this is often achieved with temperature controlled baths and jacketed cells. It is always good practice to wait at least 30 minutes before performing any accurate measurements after the crystal comes in contact with a new medium. This allows the crystal to come to equilibrium with the medium. If temperature control is not possible or practical, attempts should be made to measure the temperature of the solution around the crystal during the experiments and perform temperature compensation.

In short, each PLO-10 user must determine the effect of temperature on the experiments being performed and either control the temperature accordingly, or measure the temperature and compensate for it.

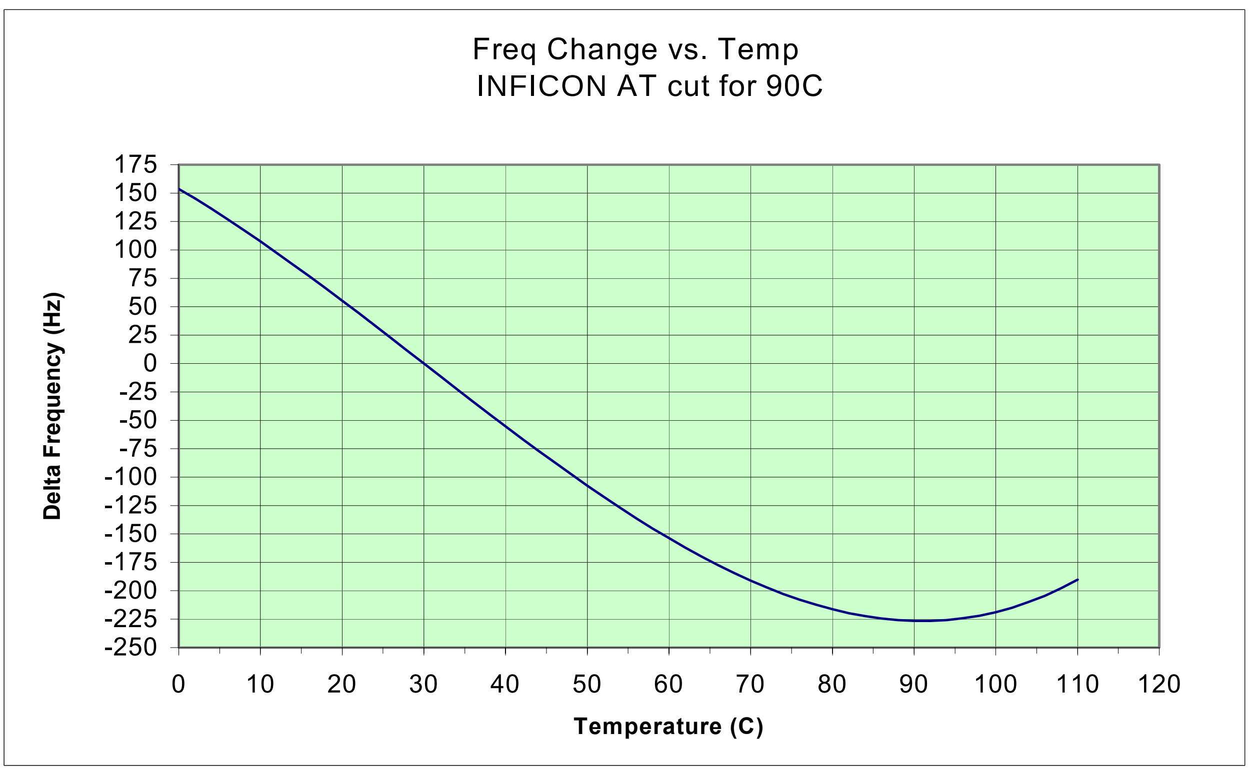


Figure 12 Frequency vs. Temperature of INFICON 1" AT-Cut Crystal for 90 C

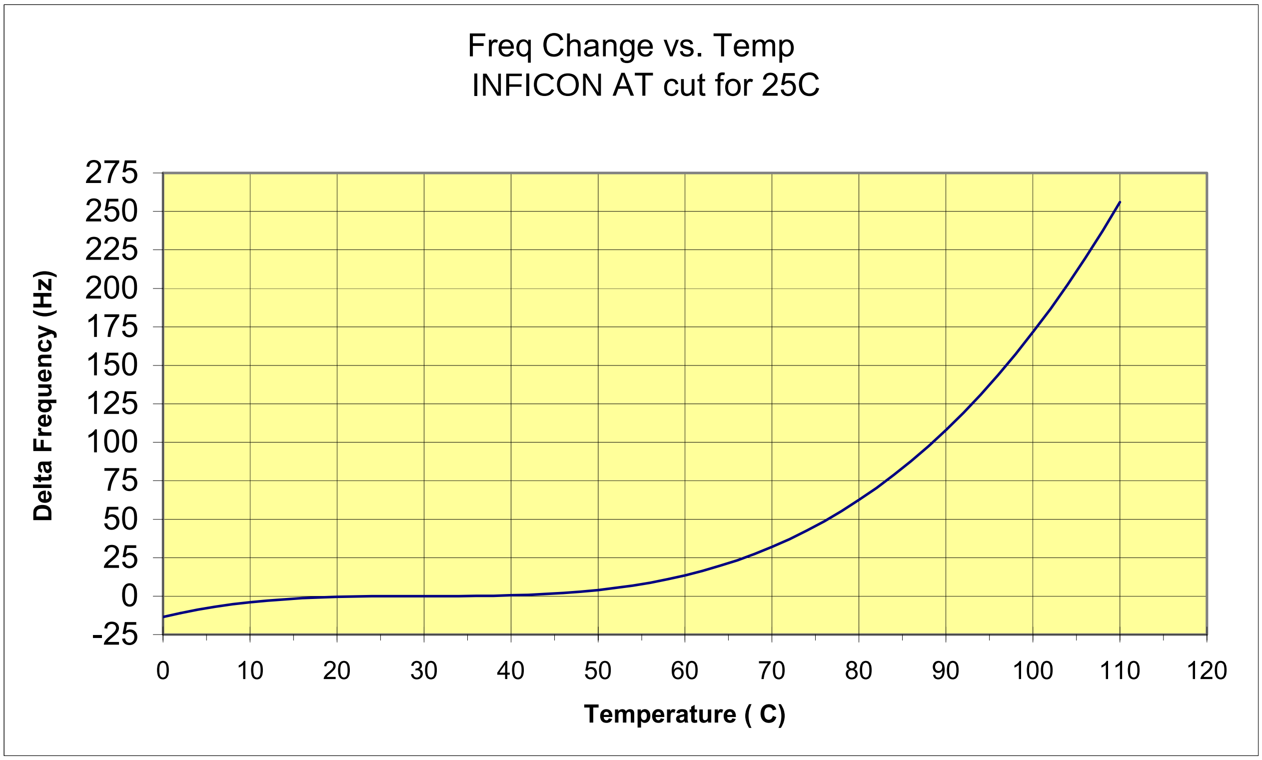


Figure 13 Frequency vs. Temperature of INFICON 1" AT-Cut Crystal for 25 C

7.2 CRYSTAL CARE AND HANDLING

It is essential that a sensor crystal is clean and free of foreign matter that may react with the experiment inducing errors in the measurements. The following guidelines are recommended for general handling of the sensor crystals.

- ♦ Keep the crystals in a clean environment. Store them in their original package until use.
- ♦ Never handle the crystals with bare hands.
- ♦ Always use plastic tweezers around the edge of the crystal during handling.
- ♦ Do not touch the center of a sensor crystal, as any oil, dirt, dust, or scratches will quickly degrade the quality of the crystal.
- ♦ When using a chemical agent to clean the crystal, ensure that the crystal electrode material(s) will not be damaged by the chemical.
- ♦ Never use cleaner that will etch the quartz surface.
- ♦ Always rinse with deionized water, or another appropriate pure liquid, before drying the crystal.
- ♦ Always use a flow of dry, oil-free, non-reactive gas (e.g. filtered nitrogen) to blow-

dry the crystal. It is better to chase liquid off the crystal than to let it evaporate off the crystal.

- ♦ Never wipe the crystal – even soft, lint-free cloth will scratch the crystal.

7.2.1 CRYSTAL CLEANING

The surface properties of the sensor crystal determine the interaction of sample material with the surface. Therefore, the developments of proper procedures for cleaning are required to obtain meaningful and reproducible measurements. This section provides the basic information you need to develop a cleaning procedure suited to your sample/surface preparation.

> **CAUTION** – When developing a cleaning procedure, always perform a test run on a crystal before committing to a larger batch cleaning. Follow the crystal handling guidelines throughout the cleaning process to protect the crystal quality. Avoid using high pH cleaners since they will etch the crystal surface.

7.2.1.1 General Cleaning

For general purpose applications such as electrochemistry and liquid or viscoelastic film experiments, it is usually sufficient to use ultrasonic cleaning method to clean the crystals in a solution of non-basic detergent in deionized water. Immediately rinse liberally with deionized water and dry in a gentle flow of filtered nitrogen gas.

7.2.1.2 Organic (hydrocarbon contaminants)

UV/ozone treatment[12] is a powerful tool for removing hydrocarbon impurities which have been adsorbed from the ambient air. This method utilizes irradiation with ultra violet light that breaks up the organics on the surface of the sample being cleaned. A flow of air or a weak vacuum carries off the organics. This method does not affect the quartz surface; it is low cost and is very efficient.

Oxygen plasma cleaning is another effective method that will remove organic matters. In this method, the plasma reaction breaks up organic matters at the surface of the sample being cleaned into smaller molecules and a vacuum pump removes them from the surface of the sample.

7.2.1.3 Biomaterials (lipids, proteins and similar biomolecules)

Start by treating the crystal in an UV/ozone chamber for 10 minutes, then immerse it into a 1:1:5 solution of hydrogen peroxide (30%), ammonia (25%) and deionized water heated to a temperature of about 75°C for 5 minutes [22]. Immediately rinse liberally with deionized water and dry in a gentle flow of nitrogen gas. Immediately before measurement, treat the crystal with UV/ozone for 10 minutes.

7.2.1.4 Lipid vesicles on SiO_2 surfaces

Treat the crystal in an UV/ozone chamber for 10 minutes, then immerse it into water with 2% of sodium dodecyl sulfate (SDS) at room temperature for 30 minutes [23]. Rinse generously with deionized water and blow dry with filtered nitrogen gas. Immediately before measurement, treat the crystal with UV/ozone for 10 minutes.

7.2.1.5 Polystyrene removal

To clean polystyrene (PS) off a crystal, immerse the crystal into a 1:1 solution of hexane and deionized water and treat it in an ultrasonic bath for 1 minute. Rinse thoroughly with deionized water and blow dry with filtered nitrogen gas.

7.2.2 ELECTRODE SURFACE MODIFICATIONS

A QCM will response to *anything* that has mass. Thus, it is imperative for the QCM user to develop a "condition" where the QCM will only responsed to the substance of interest. This usually involves a chemically or biologically sensitive layer applied to the surface of the crystal[13].

INFICON offers a wide variety of standard electrode materials for you to choose from. Contact us if you don't see one that fits your needs. If you choose to do your own crystal surface modification, use the following guidelines.

7.2.2.1 SPIN COATING

Thin films (nm to microns) of polymers and other materials can be applied by spin coating[14]. Polystyrene is a common material spin-coated on QCM sensor crystals. UV/Ozone treatment can be used to change the hydrophobicity of organic polymeric coatings[15] [16].

7.2.2.2 SELF-ASSEMBLED MONOLAYERS (SAM)

Self-assembling monolayers can be laid down on gold or silver surface by thiolization[17] [18] [19] or on SiO_2 by silanization[20] to control surface properties.

7.2.2.3 PHYSICAL VACUUM DEPOSITION (PVD)

Thin films of metals or metal oxides can be applied by sputtering or thermal evaporation in a vacuum chamber. To ensure quality and reproducible films, careful attention to cleanliness must be observed, both in the vacuum chamber and in the preparation of the crystals prior to coating. INFICON is an expert in PVD. Consult us for any special needs.

7.3 CRYSTAL HOLDERS

Figure 14 shows a INFICON CHC-100 Crystal Holder (without a crystal, the crystal retainer or the retainer cover). It has a cavity for a 1-inch diameter crystal. Inside the cavity there are two Pogo® pins providing connections to the crystal's front and rear electrodes. Note the locations of the Pogo® pins. These pins are internally connected to the BNC connector (SMB Jack for CHT-100 and CHK-100 holders) via an internal coaxial cable.

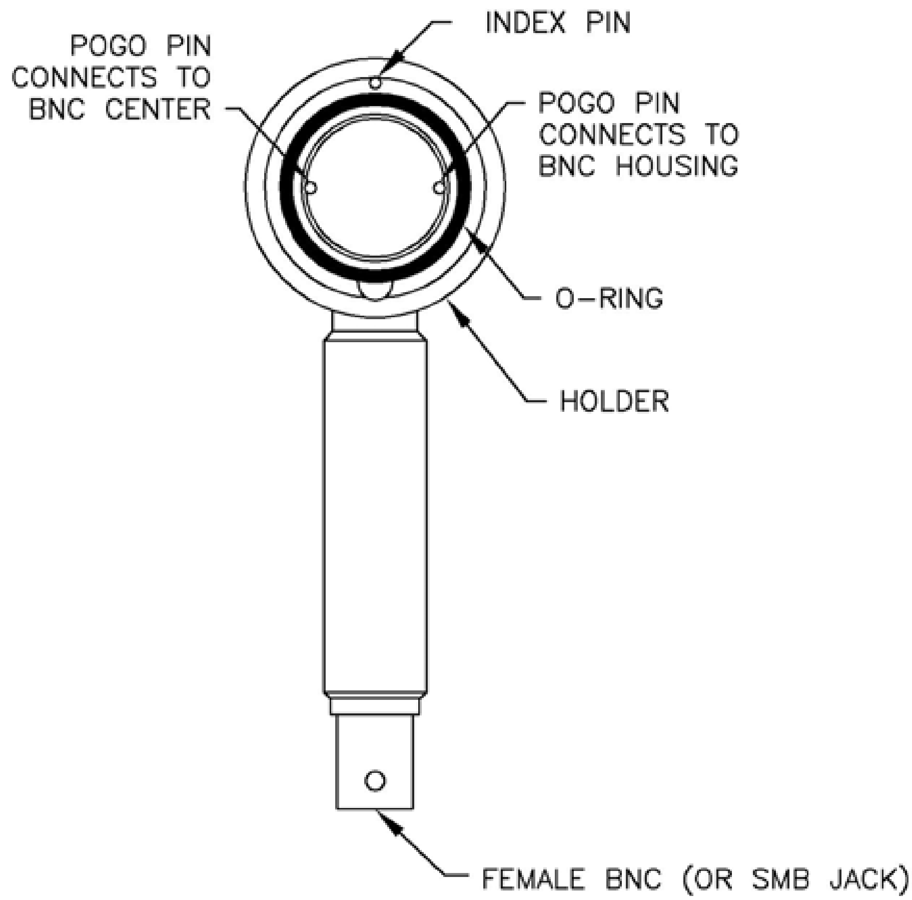


Figure 14 CHC-100 Crystal Holder

7.3.1 HOW TO INSTALL A CRYSTAL IN A INFICON CRYSTAL HOLDER

1. Identify the Front and Rear Sides of the crystal. See Section 7.1.1.

2. Clean & Dry the Crystal Holder cavity, then insert the Crystal with the Front Side (Sensing Electrode) exposed. The "Wrap-Around Extended Electrode" MUST be in the 60º region as in Figure 15 below.

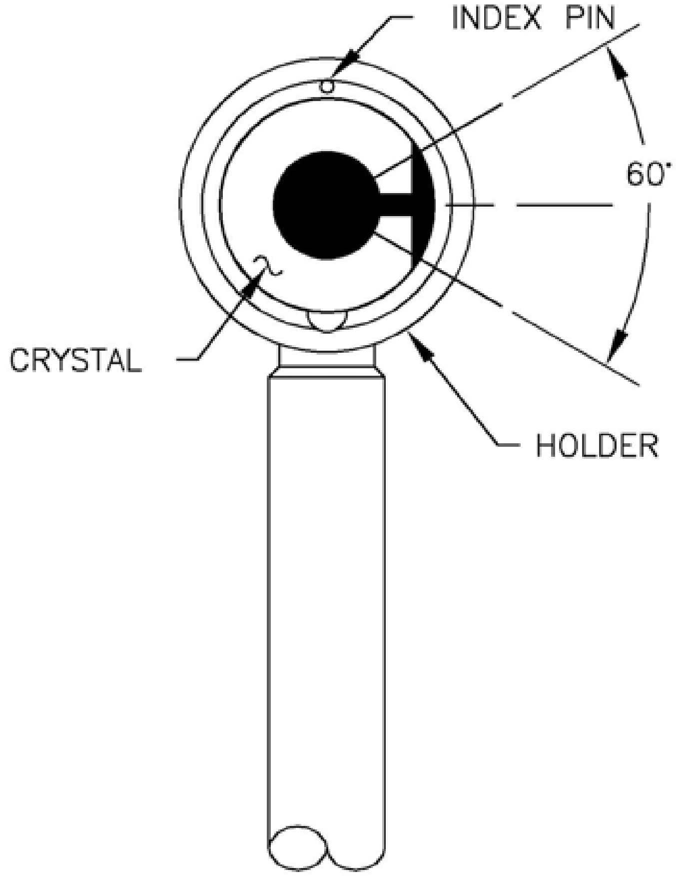
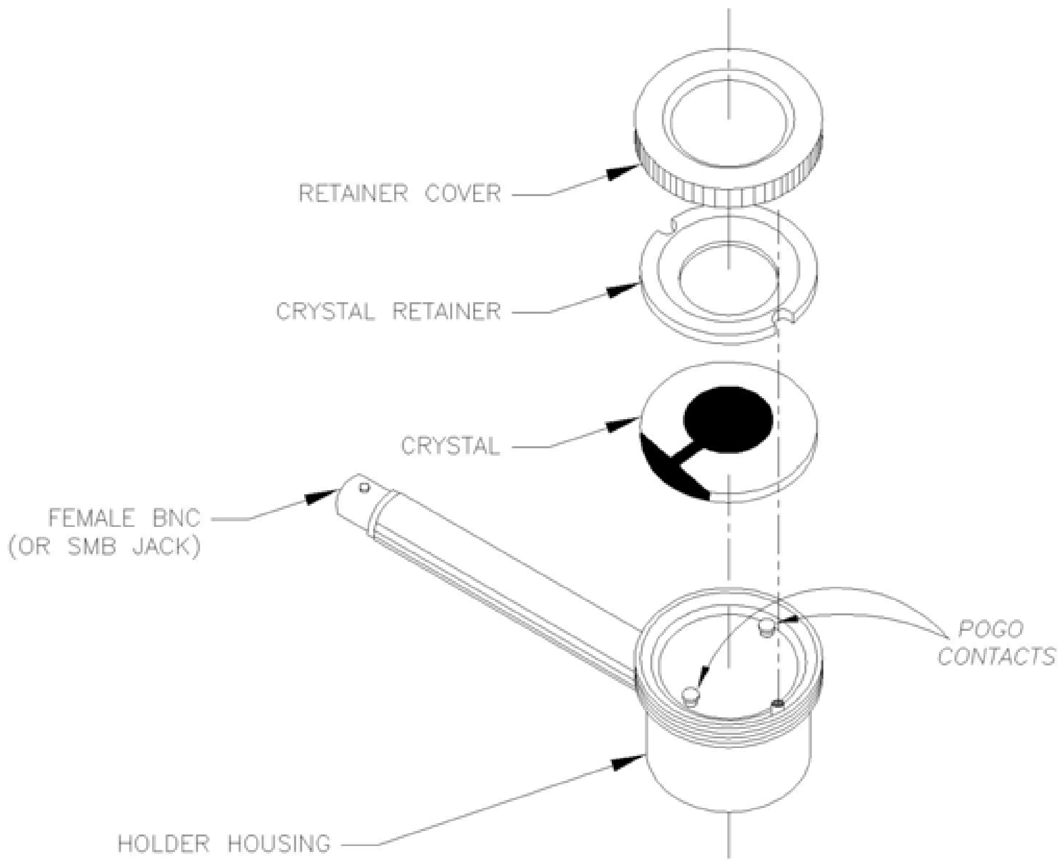


Figure 15 Crystal Installation

3. Place the Retainer Ring over the Crystal, with the Notch mating to the Index Pin.

4. Mount and turn the Retainer Cover approximately ¼ turn. Then with a gloved-finger or cotton swab *gently* press the Retainer Ring down at the Notch to make sure that it stays mated to the Index Pin. Finish tightening the Cover until it's snug.



7.3.2 HOLDER CARE AND HANDLING

With a robust design, INFICON crystal holders require little care. However, the crystal holder is in direct contact with the sensor crystal and your experiment environment. Thus, care must be taken to ensure its cleanliness eliminating any contaminants that may react with the crystal or the experiment media. The following guidelines are recommended for general handling of the holders.

- Always keep the holder clean and dry when not in use.
- Always use clean room grade gloves while handling the holder and its components.
- Never handle the holder with bare hands as human skin oils may deposit on it and react with your experiment.
- Always ensure that your holder is compatible with your experiment environment.
- Never submerge the holder unassembled or without a crystal.
- Never submerge the holder pass its terminal connector at the end of the rod.
- Always rinse the holder generously with deionized water and thoroughly blow dry using filtered air after each experiment. This is especially important if the holder has been exposed to oxidizing acids.

- Always act fast in the event that liquids or chemicals have entered the crystal cavity in the holder. Immediately clean the holder using the following procedure.
    - Remove the crystal to expose the crystal cavity.
    - Remove both Pogo® contact pins from their sockets. Use a pair of tweezers (or gloved fingernail), grab the Pogo® head firmly and pull it straight out of its socket.
    - Rinse the holder, the crystal cavity and the Pogo® sockets generously with deionized water to remove all traces of chemicals and thoroughly blow-dry the whole holder using filtered air. Ensure all liquids that may have been trapped inside the sockets are removed.
    - Generously rinse the Pogo® contact pins with deionized water, occasionally squeeze the pins to push out any liquids that may have been trapped inside the pins. Thoroughly blow-dry the pins using filtered air.
    - Install the Pogo® pins back into their sockets. Use the tip of a pair of tweezers and push down on each Pogo® pins to verify their deflection.

7.3.3 CONSIDERATIONS FOR BUILDING YOUR OWN HOLDER

You MUST consider the following aspects when building your own crystal holder.

The holder must be designed so that when a crystal is installed, its front electrode (sensing electrode) is connected to the housing (shell) of the SMB Crystal Connector on the PLO- 10; and the rear electrode is connected to the center pin of the SMB Crystal Connector.

The crystal should be clamped, as close as possible, to the edge of the crystal to avoid damping of the crystal's oscillations.

The holder clamping mechanism should have a positive stop to avoid excessive clamping force on the crystal.

If the crystal is to be used in a conductive fluid or conductive gas, the rear electrode must be sealed from the conductive environment to avoid an electrical short between the electrodes. The electrodes should be designed so the rear electrode and the electrodes contacts can be sealed. Only the front electrode should be exposed.

The connecting cable must be coaxial all the way, from SMB on the PLO-10, on up to the crystal. The shield of the coaxial must connect to the front electrode and the center conductor must connect to the rear electrode of the crystal. In addition, the coaxial cable must be free of kinks, knots, etc. to avoid unbalanced capacitance in the cable. Note that a one-foot of well balance RG174A/U coaxial cable has approximately 29 picofarads.

The total capacitance of the crystal, the crystal holder and the cable must be within the PLO-10's capacitance compensation limits (between 40 and 200 pfd).

7.4 FLOW CELL

The FC-550 Flow Cell is designed to be used with any of INFICON's 100 series crystal holders. The FC-550 is made from Kynar®. The cell has two stainless steel inlet and outlet tubes with a .047" I.D. x .062" O.D., compatible with 0.062" I.D. tubing. A Viton® O-ring provides sealing between the cell and the face of the sensor crystal. The cell is used in place of the Crystal Retainer Ring. Once installed in a probe, it creates a flow chamber of approximately 0.1 mL.

8 THEORY OF OPERATION

Sauerbrey was the first to recognize the ability of the Quartz Crystal Microbalance (QCM) to measure very small mass changes on the crystal surface. His seemingly simple equations have been used for many years and in many different applications.

8.1 SAUERBREY EQUATION

Equation 1

$$\Delta f = -C_f \times \Delta m$$

Where:

Δf = Frequency change in Hz.

C_f = Sensitivity factor of the crystal in Hz/ng/cm^2

(0.0566 Hz/ng/cm^2 for a 5 MHz crystal @ 20° C)

(0.0815 Hz/ng/cm^2 for a 6 MHz crystal @ 20° C)

(0.1834 Hz/ng/cm^2 for a 9 MHz crystal @ 20° C)

Δm = Change in mass per unit area in g/cm^2.

The Sauerbrey equations assumed that the additional mass or film deposited on the crystal has the same acousto-elastic properties as quartz. This assumption resulted in a sensitivity factor, C_f, which is a fundamental property of the QCM crystal as shown in equation 2.

Equation 2

$$C_f = \frac{2n \times f^2}{\sqrt{\rho_q \times \mu_q}}$$

Where:

n = Number of the harmonic at which the crystal is driven.

f = Resonant frequency of the fundamental mode of the crystal in Hz.

ρ_q = Density of quartz = 2.648 g · cm^{-3}

μ_q = Effective piezoelectrically stiffened shear modulus of quartz = 2.947 · 10^{11} g · cm^{-1} · sec^{-2}.

Solving these equations for Δm yields

Equation 3

$$\Delta m = \frac{-\Delta f}{C_f} = \frac{(f_q - f)\sqrt{\rho_q \times \mu_q}}{2n \times f^2}$$

Where:

f_q = Resonant frequency of unloaded crystal in Hz.

f = Resonant frequency of loaded crystal in Hz.

It is important to note that under these assumptions, the change in frequency is a function of mass per unit area. Therefore, in theory, the QCM mass sensor does not require calibration. However, keep in mind that the Sauerbrey equation is only strictly applicable to uniform, rigid, thin-film deposits [21]. Vacuum and gas phase thin-film depositions which fail to fulfill any of these conditions actually exhibit more complicated frequency-mass correlations and often require some calibration to yield accurate results.

8.2 Z-MATCH EQUATION

Sauerbrey's original assumptions were of course, questionable and indeed work with crystals heavily loaded with certain materials showed significant and predictable deviations between the measured mass and that predicted by Equation 3. Lu and Lewis[22] analyzed the loaded crystal as a one-dimensional composite resonator of quartz and the deposited film which led to the equation shown below which is also referred to as the Z-Match equation.

Equation 4

$$\Delta m = \left(\frac{N_q \cdot \rho_q}{\pi \cdot R_z \cdot f} \right) \cdot \tan^{-1}\left[R_z \cdot \tan\left[\pi \cdot \left(\frac{f_q - f}{f} \right) \right] \right]$$

where:

Δm = change in mass per unit area in g/cm^2,

N_q = Frequency Constant for AT-cut quartz crystal = 1.668 x 10^5 Hz x cm = $\frac{\sqrt{\rho_q \mu_q}}{2\rho_q}$

ρ_q = Density of quartz = 2.648 g/cm^3.

f_q = Resonant frequency of unloaded crystal in Hz.

f = Resonant frequency of loaded crystal in Hz.

R_z = Z-Factor of film material = $\sqrt{\frac{\rho_q \cdot \mu_q}{\rho_f \cdot \mu_f}}$ = Acoustic Impedance Ratio

ρ_f = Density of material g/cm^3

μ_q = shear modulus of quartz = 2.947×10^{11} g · cm^{-1} · s^{-2}

μ_f = shear modulus of film material in g · cm^{-1} · s^{-2}.

This equation introduces another term into the relationship which is the ratio of the acoustic impedance of quartz to the acoustic impedance of the deposited film. The acoustic impedance is associated with the transmission of a shear wave in the deposited mass. Notice that the units of the frequency constant for quartz is length/time or velocity. Also note that if the acoustic impedance ratio is equal to one, quartz on quartz, then Equation 4 reduces to Equation 3.

8.3 THICKNESS CALCULATION

Film thickness is often the parameter of interest in many QCM applications. Thickness can be derived from Equation 4 as follows:

Equation 5

$$TK_f = \frac{\Delta m}{\rho_f} = \left(\frac{N_q \cdot \rho_q}{\pi \cdot R_z \cdot f \cdot \rho_f} \right) \cdot \tan^{-1}\left[R_z \cdot \tan\left[\pi \cdot \left(\frac{f_q - f}{f} \right) \right] \right]$$

where:

TK_f = thickness of the film in cm.

Δm = change in mass per unit area in g/cm^2 (calculated from the Lu and Lewis equation).

ρ_f = density of film material in g/cm^3

If the period of oscillation is measured rather than the frequency, 1/period can be substituted for frequency resulting in the following equation. (See INFICON TechNote RTK-101 for details discussion).

Equation 6

$$TK_f = \left(\frac{\rho_q}{\rho_f} \right) \cdot N_q \cdot \left(\frac{\tau}{\pi R_z} \right) \cdot \tan^{-1}\left[R_z \cdot \tan \pi \cdot \left(\frac{\tau - \tau_q}{\tau} \right) \right]$$

where:

τ_q = Period of unloaded crystal in seconds

τ = Period of loaded crystal in seconds

Although the above equation still involves a number of simplifying assumptions, its ability to accurately predict the film thickness of most commonly deposited materials has been demonstrated.

The basic measurement is period, which can be thought of as a measurement of equivalent quartz mass. The actual film mass on the crystal is then found by applying the acoustic impedance correction factor.

The Lu and Lewis equation is generally considered to be a good match to the experimental results[5] [6] for frequency changes up to 40% (relative to the unloaded crystal). Keep in mind that the Z-match equation strictly applies to elastic (lossless) films. Films which behave viscoelastically, such as some organic polymer films with large thickness or viscosity, will exhibit significant deviations from both Equation 3 and Equation 6.

8.4 LIQUID MEASUREMENTS

QCMs have been used as gas-phase mass detectors with lossless films for many years. However, only recently has their applications been extended to liquids and with viscoelastic deposits. In these cases, both frequency and series resonance resistance* of the quartz crystal are important to completely characterize the material and/or the liquid in contact with the crystal electrode. The development of QCM systems for use in liquids opened a new world of applications, including electrochemistry and micro-rheology. More recent developments have focused on tailoring electrode surface chemistry (i.e. specialized polymer coatings) so that these devices can be applied as discriminating mass detectors for many applications including: specific gas detection, environmental monitoring, biosensing and basic surface-molecule interaction studies.

When the QCM comes in contact with a liquid, there is a decrease in frequency that is dependent upon the viscosity and density of the liquid. Kanazawa's solution for the change in resonant frequency of the crystal due to liquid loading is shown in Equation 7.

Equation 7

$$\Delta f = -f_q^{3/2}\sqrt{\frac{\eta_L \cdot \rho_L}{\pi \cdot \mu_q \cdot \rho_q}}$$

Where:

f_q = Resonant frequency of unloaded crystal in Hz.

ρ_q = Density of quartz = 2.648×103 kg/m^3.

μ_q = shear modulus of quartz = 2.947×10^{10} Pa

ρ_L = density of the liquid in contact with the electrode in kg/m^3,

η_L = viscosity of the liquid in contact with the electrode in N · Sec/m^2

Liquid loading also dampens the resonant oscillation of the crystal causing an increase in series resonance resistance, R, of the crystal. Δf and ΔR measurements are both routinely used as independent indicators of mass loading and viscosity at the crystal-liquid interface of the QCM resonator during chemical and electrochemical depositions in solution[23].

A Butterworth-Van Dyke equivalent circuit model (Figure 18) was applied to derive a linear relationship between the change in series resonance resistance, ΔR, of the crystal and $\sqrt{\eta_L \cdot \rho_L}$ under liquid loading. Using the relations in this study the change in resistance, ΔR, can be put in the form:

Equation 8

$$\Delta R = \Delta f \cdot \frac{2\pi \cdot (\rho_q \cdot \mu_q)^{3/2}}{f^3 \cdot 32 \cdot A_r \cdot (\rho_q \cdot e_{26})^2}$$

* See Section 8.6 for a detail discussion.

Where:

ΔR = change in series resonance resistance in Ω,

A_r = active area of INFICON 1-inch crystal = 3.419×10^{-5} m^2

e_{26} = piezoelectric constant for an AT cut quartz = 0.095 $kg/sec^2/V$

For example, moving the crystal from air to pure water @ 20°C, Equation 7 and Equation 8 predict a decrease in f of 714 Hz and an increase in R of 357.4 Ω, respectively. Note that at pure water @ 20°C has a density (ρ_L) of 998.2 kg/m^3, and a viscosity (η_L) of 1.002×10^{-3} $N \cdot sec/m^2$.

Excellent agreement between the frequency and resistance equations and the experimental results has been proved[24 25 26], making the QCM an excellent tool for the evaluation of fluid properties. Application examples include *in-situ* monitoring of lubricant and petroleum properties[27]. The tight correspondence between theory (Equation 7 and Equation 8) and the PLO is clearly illustrated by Figure 16 and Figure 17, respectively. Note that some of the discrepancy in the resistance curve could arise from an error in estimating the active electrode area.

The PLO-10 utilizes the PLO technology which allows the sensor crystal to operate under heavy viscous loading. INFICON Crystal Holders support operation in gas and liquid environments and provide single-electrode exposure to liquids as required for compatibility with electrochemical QCM measurements. The PLO-10 will maintain oscillation up to a series resonance resistance of about 5 kΩ. It will support crystal operation in highly viscous solutions up to 88% weight percentage of glycerol[28].

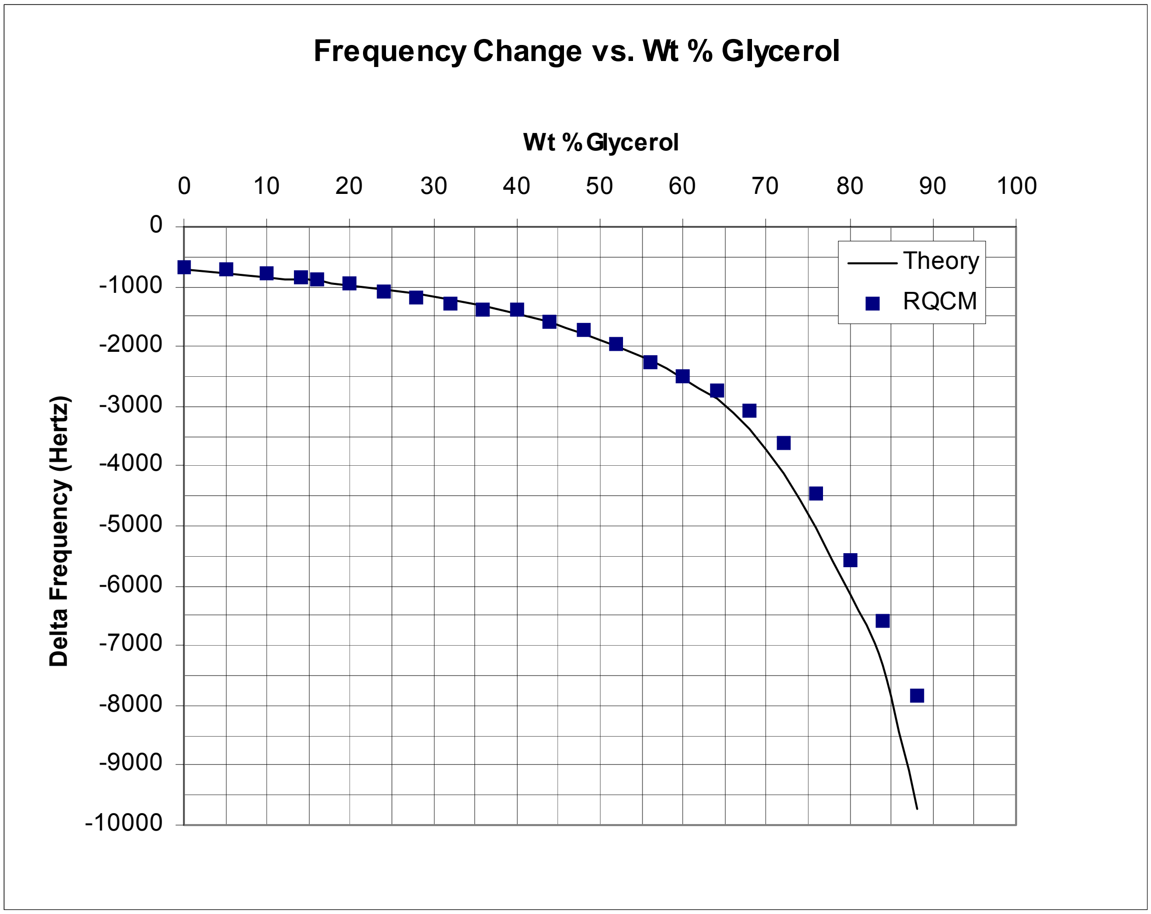


Figure 16 Frequency Change vs. Wt % Glycerol

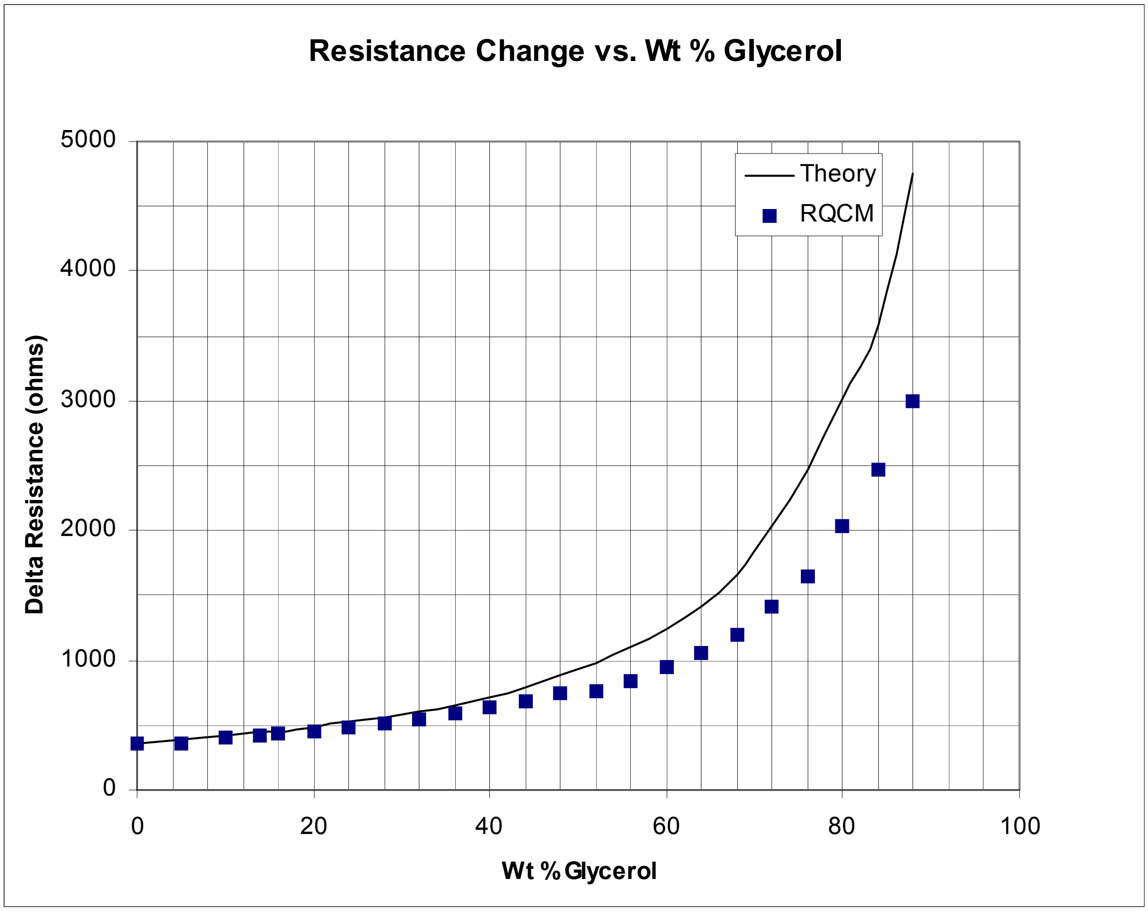


Figure 17 Resistance Change vs. Wt % Glycerol

8.4.1 DECAY LENGTH OF SHEAR WAVE IN LIQUID

As mentioned in the section above, when an oscillating crystal is in contact with a liquid, there will be a decrease in the resonant frequency and an increase in the motional resistance. The decrease in the resonant frequency is caused by the additional mass of the vibrating liquid. The increase in motional resistance is caused by the power dissipation of the shear wave that radiates into the liquid. The decay length of the shear wave into the liquid is defined by

Equation 9

$$L_D = \sqrt{\frac{2\eta_L}{\rho_L \cdot \omega_S}}$$

Where:

L_D = Decay length in m

ρ_L = Density of the liquid in contact with the electrode in kg/m^3

η_L = Viscosity of the liquid in contact with the electrode in kg/m/sec

ω_S = Angular frequency at series resonance ($2\pi f$)

For example, the decay length for a 5 Mhz crystal in water at 20°C is 2.5×10^{-7} m = 0.25 microns.

8.5 DISSIPATION METHOD

The *Dissipation Method* is an alternate way of measuring the crystal to determine the properties of the film and/or the liquid. In this method, the crystal is driven at its resonant frequency by an oscillator then the crystal shorted and both the resonant frequency and the oscillation decay time are measured. The crystal dissipation is related to Q and R as follows:

Equation 10

$$D = \frac{1}{Q} = \frac{R}{\omega_S \cdot L}$$

Where:

D = Dissipation

Q = Quality Factor

R = resistance in Ω

L = inductance in H

D can be determined from R if L is known. It has been shown that L will remain constant unless there is an acoustic resonance in the film on the crystal. Independent studies [25] have shown that as long as the effect of the parasitic capacitance (C_S) is properly cancelled, the results provided by the PLO-10 System are in good agreement with those obtained by the Dissipation Method.

8.6 ELECTRICAL DESCRIPTION OF THE QUARTZ CRYSTAL

Figure 18 shows the equivalent circuit of a quartz crystal. The circuit has two branches. The motional branch, which contains the L, R & C, is the branch that is modified by mass and viscous loading of the crystal. The shunt branch, which contains the lone Cs element, represents the shunt capacitance of the crystal electrodes and any cable and fixture capacitance.

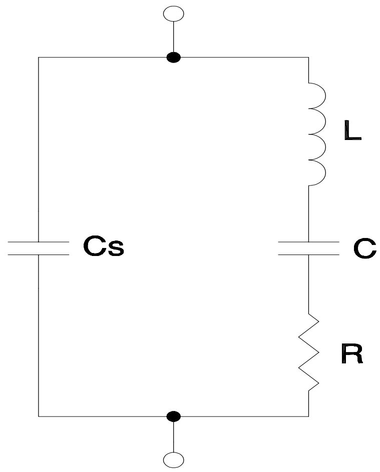


Figure 18 Crystal Equivalent Circuit

Because a crystal's impedance is minimum at resonance it is convenient to characterize a crystal in terms of admittance. Admittance is the inverse of impedance, (Y = 1/Z), thus the admittance reaches a maximum at resonance. While impedance is proportional to the voltage developed across a device when it is subjected to a current, the admittance is proportional to the current through the device when it is subjected to a voltage.

At any frequency the admittance of a quartz crystal is a complex value that can be expressed in terms of magnitude and phase or in terms of a real and imaginary value. The relationship of these two representations is shown in Figure 19.

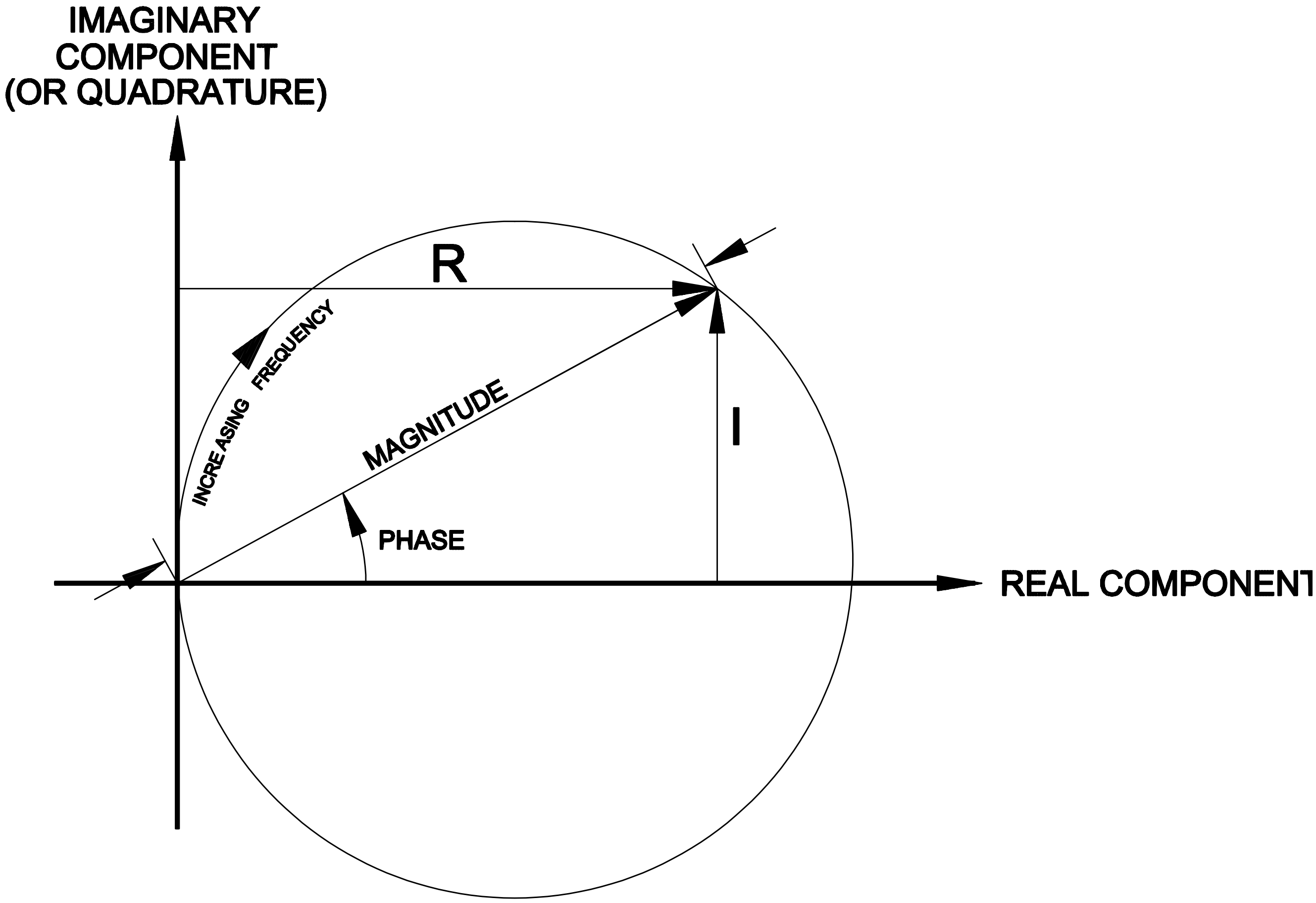


Figure 19 Polar Plot of Crystal Admittance

Figure 20 shows the conductance in terms of magnitude and phase, while shows the same information in terms of the imaginary and real part of the conductance.

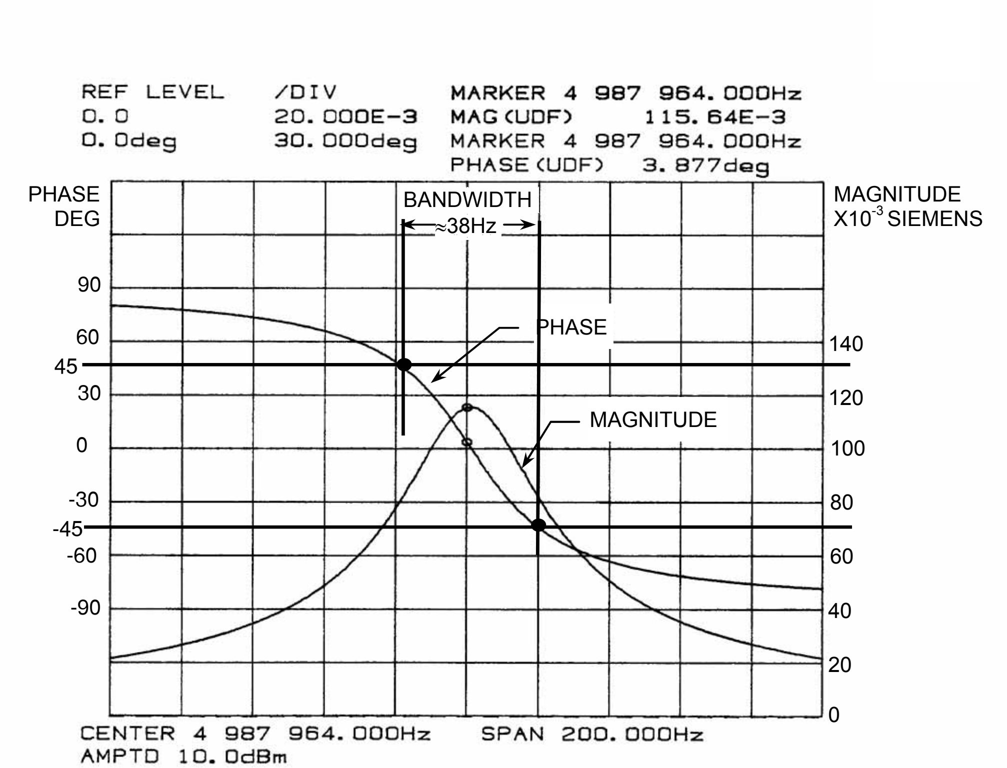


Figure 20 Admittance vs. Frequency, Magnitude and Phase of High Q Crystal

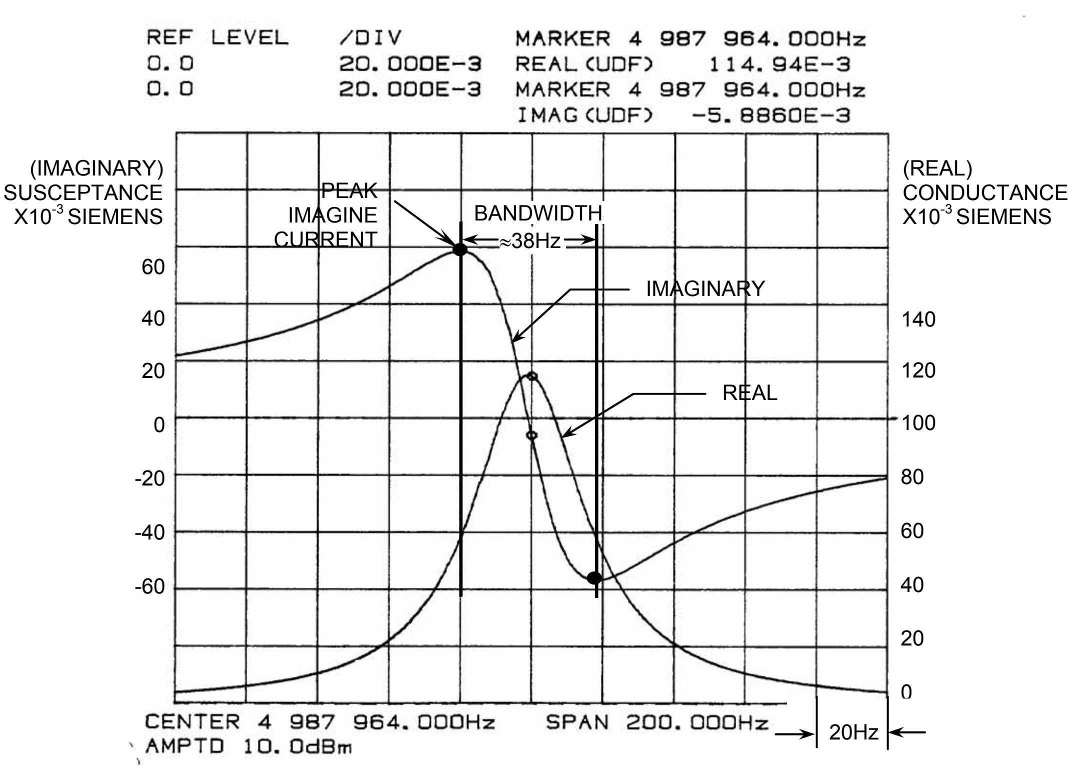


Figure 21 Admittance vs. Frequency, Real and Imaginary Components of High Q Crystal

When the above complex conductance is plotted in polar coordinates, one obtains a circle as shown in Figure 22. The vector V indicates the magnitude and phase of the crystal current divided by the applied voltage. The real part of the conductance is indicated by the vector R and the imaginary part is indicated by the vector I.

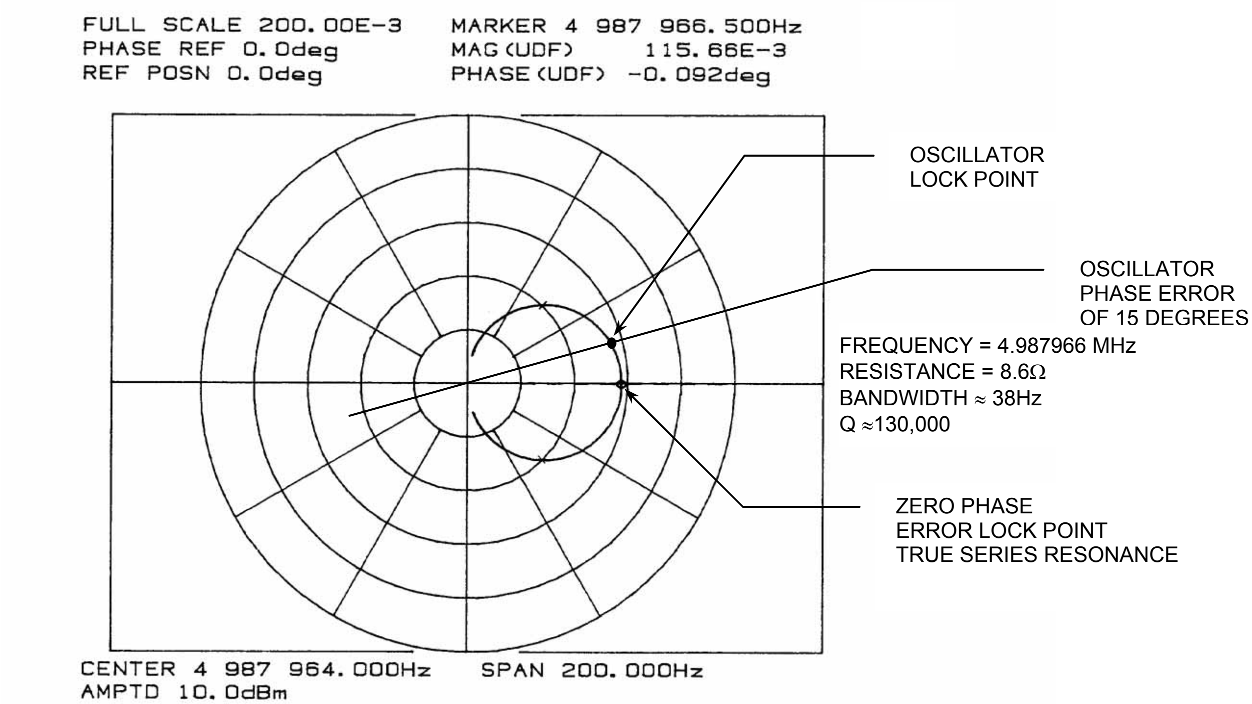


Figure 22 Polar Admittance Plot of High Q Crystal

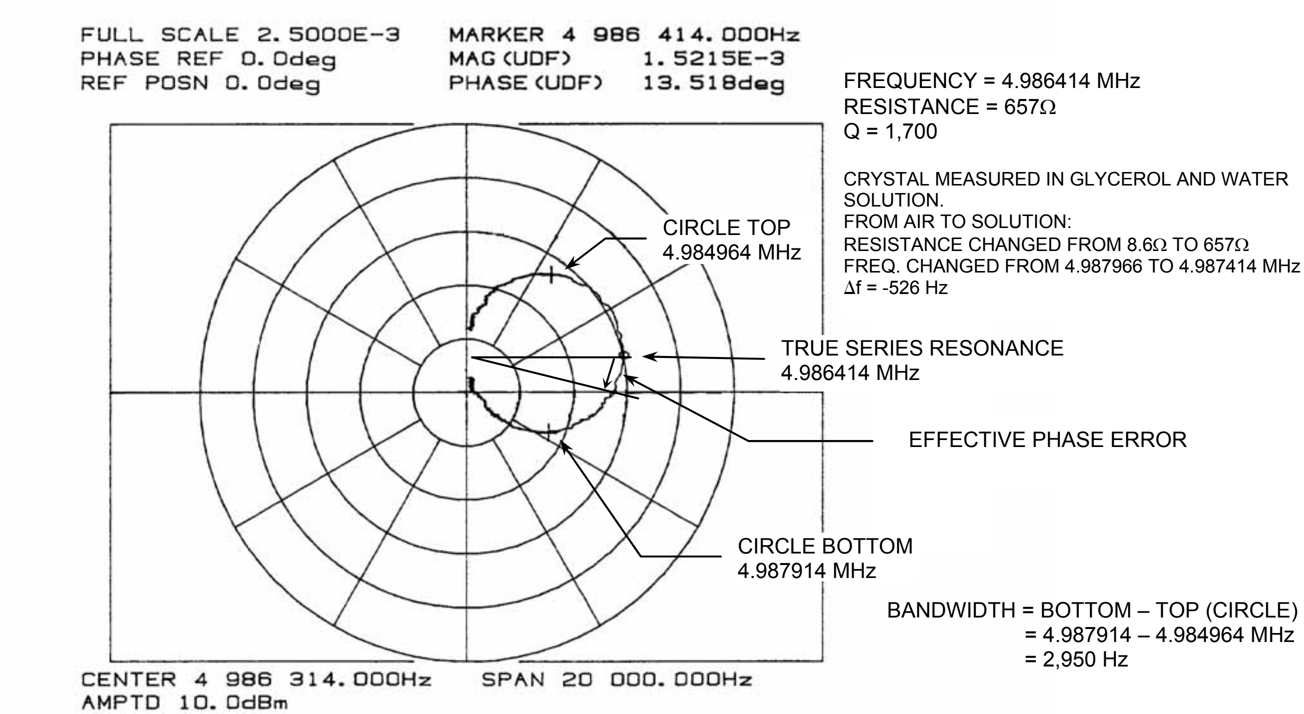


Figure 23 Polar Admittance Plot of Low Q Crystal

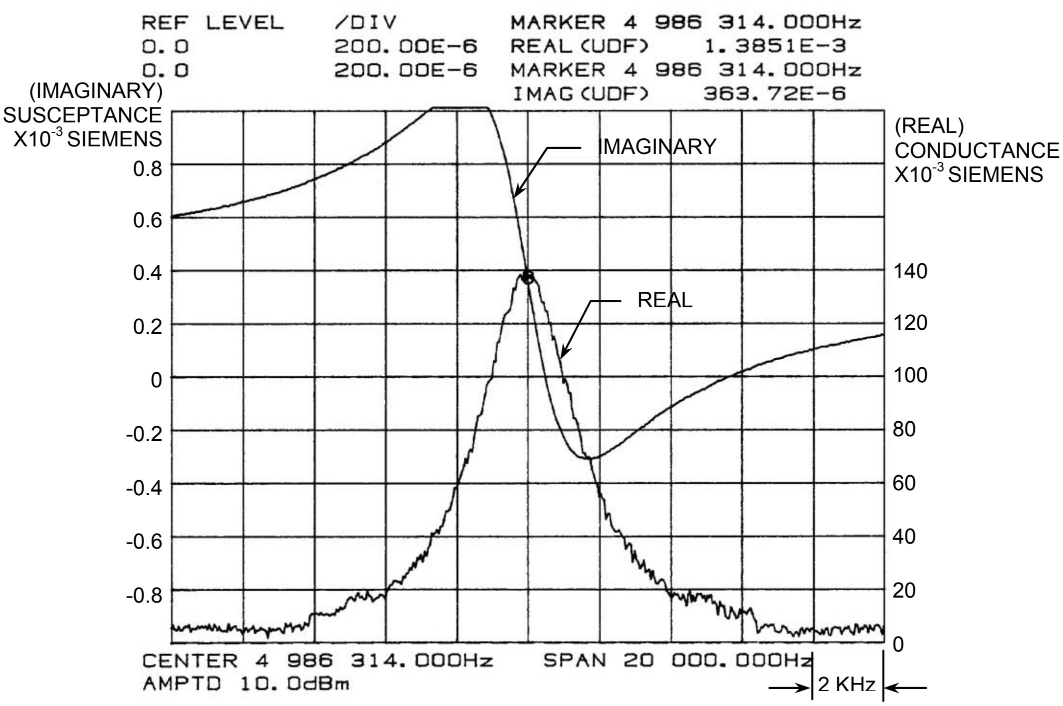


Figure 24 Admittance vs. Frequency, Real and Imaginary Components of Low Q Crystal

The conductance of the L, R & C series arm creates the circle in the polar plot with its center on the real axis. The effect of the shunt capacitance conductance is to offset the circle vertically. Figure 23 shows a heavily loaded crystal in which the offset is obvious. It is the imaginary (quadrature) current through the shunt capacitance that creates the offset. The PLO-10 provides a mechanism for canceling out the imaginary current effectively putting the center of the crystal back on the real axis. The true series resonant frequency of the crystal is then the point where the conductance circle crosses the real axis. This is the frequency at which the inductive and capacitive impedance's in the L, R & C branch cancel out and the crystal looks like a pure resistance of value R.

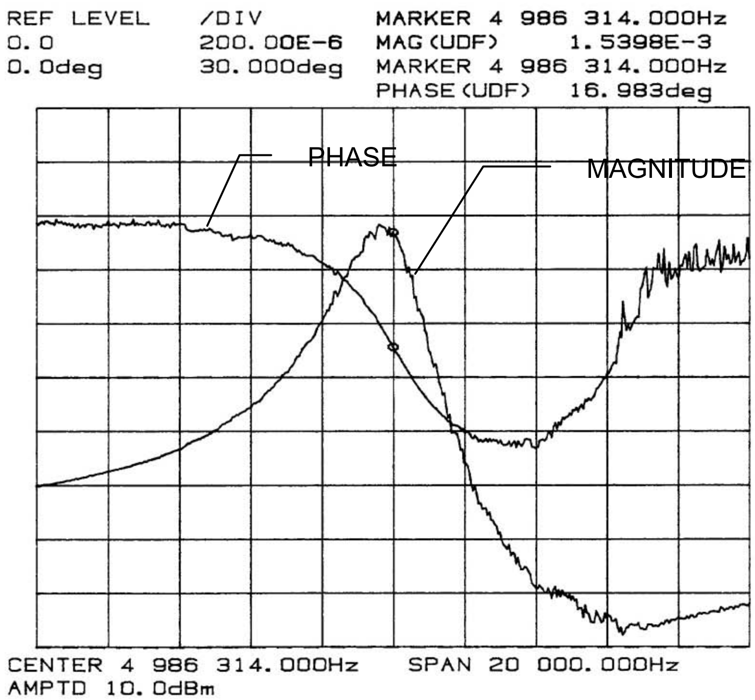


Figure 25 Admittance vs. Frequency, Magnitude and Phase of Low Q Crystal

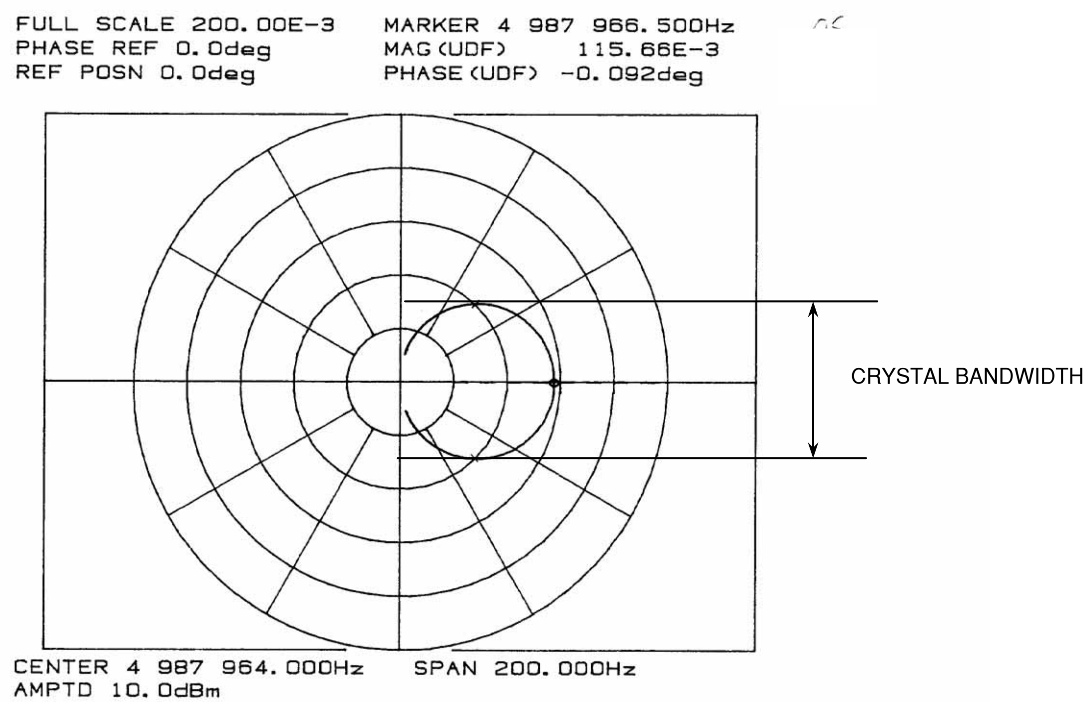


Figure 26 Non-zero Phase Lock

Figure 26 shows the result of a non-zero phase lock. Note that the frequency difference between the top of the conductance circle and the bottom is equal to the bandwidth of the crystal. For a high Q, (high conductance, low resistance) crystal, the bandwidth is very narrow and small errors in phase lock angle are insignificant. For a low Q crystal the bandwidth can be quite large and small phase errors can result in significant frequency errors. See the equations in the error discussion section.

8.7 CHARACTERIZING THE CRYSTAL MEASUREMENT

The INFICON Phase Lock Oscillator (used on the Crystal Measurement Card) was developed specifically to support the use of the quartz crystal microbalance in the measurement of lossy films and in liquid applications. In addition to accurately tracking the frequency of heavily damped crystals, the PLO-10 also tracks the crystal's resistance. This provides additional information in the study of lossy films and/or viscous solutions.

The PLO utilizes an internal oscillator referred to as a Voltage Controlled Oscillator (VCO) to drive the crystal. The crystal current is monitored and the frequency of the oscillator is adjusted until there is zero phase between the crystal voltage and current. Assuming that the crystal's electrode capacitance has been effectively cancelled, this point of zero phase between the crystal current and voltage is the exact series resonant point of the crystal. The magnitude of the current at this point is directly proportional to the crystal's conductance. This current is monitored by the PLO-10 and displayed as crystal resistance. The PLO contains a phase detector that continuously monitors the phase difference between the crystal's current and voltage. At frequencies below the crystal's resonant frequency the current leads the voltage and the phase goes to 90 degrees as the frequency separation continues to increase, see Figure 21. Above the resonant point the current lags the voltage and the phase go to minus 90 degrees. As the frequency increases through the resonant frequency, the phase goes from plus 90 through 0 to minus 90. It is interesting to note that the phase angle is 45 degrees when the VCO frequency is one half of the crystal's bandwidth above or below the crystal's resonant frequency.

The output of the phase detector is fed into an integrator. The integrator accumulates the phase error such that any positive phase error causes the integrator output to climb; a negative phase causes the integrator output to fall. With zero phase error the Integrator output holds steady.

The integrator output is connected to the VCO. Thus, if the VCO frequency is initially below the crystal resonant frequency, the phase will be positive, producing a positive output at the phase detector. This causes the Integrator output to climb, which causes the VCO frequency to increase. When the VCO frequency matches the resonant frequency of the crystal the phase will decrease to zero, the phase detector output will go to zero, the Integrator output will hold steady and the VCO frequency will be "locked" to the crystal's resonant frequency.

If the crystal's resonant frequency moves up or down, a phase difference between the crystal voltage and current will develop, producing a phase detector output. The non-zero phase detector output will drive the Integrator output up or down until the phase is zero once again, thus keeping the VCO frequency locked to the crystal's resonant frequency.

Once the frequency of the VCO is locked to the series resonant frequency of the crystal, the in-phase component (at zero phase error, there is no out of phase component) of the crystal current is demodulated to a DC voltage.

8.7.1 FREQUENCY ERRORS

The first thing we want to know regarding the performance of the crystal measurement is "What is the magnitude of the frequency error we can expect from the crystal measurement portion of the PLO-10?"

In any oscillator and sensing crystal system, the error in the frequency measurement, is a function of both the oscillator and the sensing crystal. The same is true for phase locked loops. Any phase error will introduce a frequency error and this frequency error will be inversely proportional to the sensing crystal's Q. These errors are over and above any change in crystal frequency due to stress, temperature, adsorption, and humidity changes.

There are four important parameters that determine the frequency error of the PLO and sensing crystal system or indeed, any oscillator and sensing crystal system. The first two, the zero phase error and the electrode capacitance cancellation errors, are characteristics of the PLO. The second two are characteristics of the crystal, the Q of the crystal and the conductance (1/resistance) of the crystal.

8.7.2 FREQUENCY ERROR DUE TO PHASE ERROR

Given some finite zero phase error, the resulting frequency error depends on the sensing crystal's Q, the higher the Q, the lower the error. For phase errors below 10 degrees the frequency error is 0.087 PPM per degree for crystals with a Q of 100,000. Thus a one degree phase error in the PLO results in a 0.44 Hz frequency error for a 5MHz crystal with a Q of 100,000. For a 5 MHz crystal with a Q of 10,000, the error is 10 time greater or 4.4 Hz per degree.

$$\text{Frequency Error/deg} = df/f = \pi/(360^*Q)$$

8.7.3 FREQUENCY ERROR DUE TO IMPERFECT CAPACITANCE CANCELLATION

The effect of imperfect electrode capacitance cancellation can also be viewed as an equivalent phase error. This error is directly proportional to crystal resistance. The equivalent phase error due to a non-zero shunt capacitance equal to 1 pfd is one degree for a crystal with a series resistance of 556 Ω. Since the equivalent phase error is proportional to the crystal resistance, a 1-pfd residual capacitance error will result in a 10-degree equivalent error for a sensing crystal with a resistance of 5.56 KΩ.

8.8 FREQUENCY ERRORS DUE TO IMPERFECT CAPACITANCE CANCELLATION

There are two reasons that proper capacitance cancellation is so important with high resistance crystals.

The first is that to a first approximation, the frequency error resulting from a given phase error is proportional to the bandwidth of the crystal. The bandwidth of the crystal is proportional to the crystal's resistance. A ten-ohm crystal might typically have a bandwidth of 42 Hz, while a one thousand-ohm crystal will have a bandwidth of 4,200 Hz. A five thousand-ohm crystal will have a bandwidth of 21,000 Hz. Since the frequency error for a given phase error is proportional to the bandwidth, a phase error that would result in a 0.5 Hz frequency error in a ten ohm crystal will cause a 50 Hz error in a one thousand ohm crystal and 250 Hz error in a five thousand ohm crystal.

The second reason is that the effective phase error caused by a non-zero net quadrature current is inversely proportional to the real current, which is inversely proportional to the crystal resistance. In other words, the effective phase error is proportional to the crystal resistance. For instance, a net unbalance of 1 pfd leads to an effective phase error of 0.02 degrees for a ten ohm crystal, but it leads to a 2 degree error for a one thousand ohm crystal and a 10 degree error for a five thousand ohm crystal.

Examples:

A ten-ohm, 5 MHz crystal will have a Q (Quality Factor) of about 120,000. The bandwidth is equal to the crystal frequency divided by Q. Thus, the bandwidth of this crystal would be about 42 Hz. To a first approximation, near zero phase, the frequency error per degree of phase error is given by the following formula,

$$\text{Frequency Error} = -\tfrac{1}{2}(\text{Phase Error, in radians})(\text{Bandwidth})$$

Or,

Frequency Error = -(1/(2*57.3))(Phase Error, in degrees)(Bandwidth)

For the above ten-ohm crystal, the frequency error caused by a one-degree phase error is 42/114.6 or approximately 0.37 Hz. For a one thousand-ohm crystal, one degree of phase error results in a 37 Hz error and for a ten thousand-ohm crystal the frequency error is 370 Hz per degree of phase error.

Now, the effective phase error caused by a non-zero quadrature (imaginary) current is given by the following formula,

Effective Phase error = arctangent (imaginary current/real current)

And since current is proportional to conductance,

Effective Phase error = arctangent (imaginary conductance/real conductance)

The conductance of a one picofarad capacitor at 5 MHz is 31.4 microsiemens. The conductance of a ten-ohm crystal at resonance is 100 millisiemens.

Effective Phase error = arctangent ((31.4e-6)/(100e-3)) = 0.018 degrees

In other words a one picofarad capacitance unbalance will result in an effective phase error of only 0.018 degrees when measuring a ten-ohm crystal. However, when measuring a one thousand-ohm crystal the effective phase error will increase to 1.8 degrees and it will increase to 9 degrees when measuring a five thousand-ohm crystal.

Combining these two errors we can get an idea of the magnitude of the frequency error caused by imperfect capacitance cancellation.

For a 10 Ω crystal a one picofarad capacitance imbalance results in a 0.018 degree phase error and a 0.0067 Hz frequency error.

For a 100 Ω crystal, the phase error is 0.18 degrees and the frequency error is 0.67 Hz. For a 1000 Ω crystal, the phase error is 1.8 degrees and the frequency error is 67 Hz. For a 5000 Ω crystal, the phase error is 9 degrees and the frequency error is 1,635 Hz.

A two picofarad capacitance imbalance will result in approximately twice the above error.

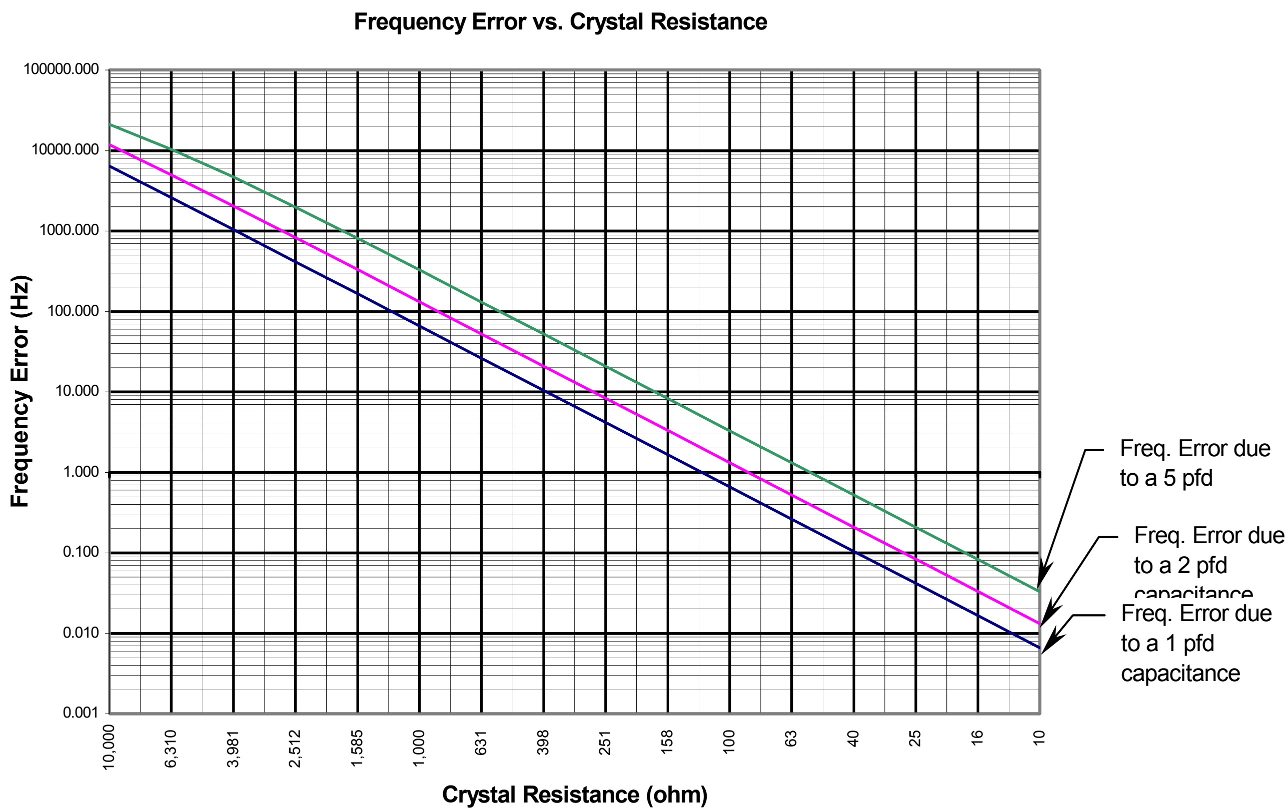


Figure 27 Frequency Error Due to Imperfect Capacitance Cancellation

8.9 CALCULATING CRYSTAL POWER

Crystal power can be calculated as follows:

Crystal power, $P_{cry} = i^2 * R_{cry}$

Crystal current, $i = V_{oc}/(R_s + R_{cry})$

Hence, $P_{cry} = i^2 * R_{cry} = [V_{oc}/(R_s + R_{cry})]^2 * R_{cry}$

Where:

V_{oc} = Open Circuit crystal drive voltage = 125 mV

R_s = Crystal drive source resistance = 20 ohms

R_{cry} = Crystal resistance value in ohms

Examples:

1. Crystal Resistance = 80 ohms

   $P_{cry,\ in\ watts} = [0.125/(20 + 80)]^2 * 80 = 1.25E^{-4}$ watts or 125 μW

3. Crystal Resistance = 4000 Ω

   $P_{cry,\ in\ watts} = [0.125/(20 + 4000)]^2 * 4000 = 3.87E^{-6}$ watts or 3.87 μW

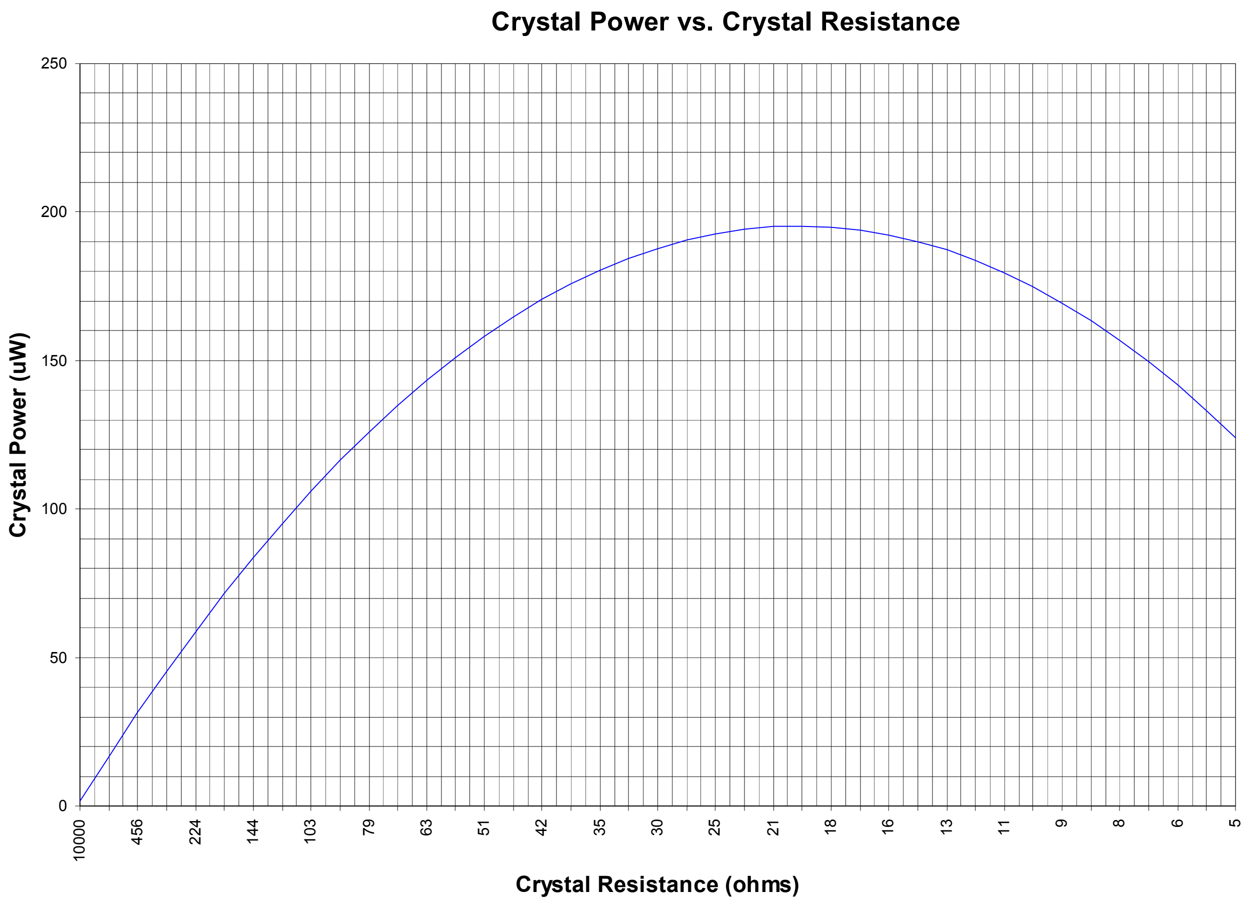


Figure 28 Crystal Power Dissipation vs. Crystal Resistance

9 GLOSSARY

Å	Symbol for angstrom, a unit of length equal to 10^{-10} meter.
Conductance	The ability to conduct. Conductance is the inverse of resistance. Conductance = 1/Resistance, or Resistance = 1/Conductance. The units of resistance are Ohms [Ω = V/A] and the units of conductance are Siemens (S = A/V].
CPVC	Abbreviation for chlorinated polyvinyl chloride, a resin patented by Goodrich, it has excellent mechanical strength and stability over temperature, and offers good resistance over a selective range of chemicals.
Crystal Bandwidth	Refers to the crystal's frequency response range, bounced by the frequency values cross at half the resonance frequency's magnitude. It is defined as f_o/Q (resonance frequency/crystal Q).
Crystal Holder	A device that houses the crystal and provides connections to the crystal's electrodes via a coaxial connector.
Crystal Q	A figure of merit used in describing the "sharpness" of the crystal response. (It is also called crystal quality factor.)
Elastic	Flexible or springy - the property of immediately returning to its original size, shape, or position after being stretched, squeezed, flexed, etc.
Hydrocarbon	A compound containing only the elements carbon and hydrogen.
Hydrophilic	Water-loving; attracted to water molecules and polar molecules.
Hydrophobic	Water-hating; not attracted to water molecules or polar molecules.
I.D.	Abbreviation for Inside Diameter. Usually use in specifying a tube size in the form *inch* I.D. x *inch* O.D., where *inch* are the dimensions.
Kynar®	Pennwalt's registered trademark of Polyvinilidene Fluoride (PVDF), a homopolymer of 1,1-di-fluoro-ethene, is a tough thermoplastic that offers unique properties including: high chemical inertness, low permeability to gases and liquids, resistance to radiation and excellent mechanical strength and toughness. Visit www.atofinachemicals.com for more detailed information.
Lipid	An organic compound found in tissue and that is soluble in nonpolar solvents.
Molar mass	The mass of a mole of substance; the same as molecular weight for molecular substances.
Mole	That amount of a substance containing the same number of units as 12 g of carbon-12.
nonpolar	Describing a molecule having no separation of centers of positive and negative electrical charge that would make the molecule assume certain orientations more than others in an electric field.

O.D.	Abbreviation for Outside Diameter. Usually use in specifying a tube size in the form *inch* I.D. x *inch* O.D., where *inch* are the dimensions.
Organic	In chemistry, organic refers to a species containing carbon. Certain small ions and compounds containing carbon (such as carbon dioxide) are usually not considered to be organic, but rather are classed as inorganic.
Picofarad	10^{-12} farads. A common unit of capacitance (abbreviated as pfd). By definition, 1 farad will store a 1 Coulomb charge when connected across a 1 volt potential.
Plasma Cleaning	A method that utilizes plasma reaction at the surface of the sample and volatile by-products are removed by the vacuum pump. The basic instrumentation required this process includes a reaction chamber, a power supply, and a vacuum source. The sample being cleaned is put into the chamber which is evacuated by the vacuum pump. Gas (oxygen) is introduced into the chamber and converted to reactive plasma by the power supply. For more information visit http://www.marchplasma.com/
PLO	Phase Lock Oscillator. A type of electronic circuit in which the frequency and the phase of the Voltage Controlled Oscillator (VCO) is locked to the frequency and the phase of a reference signal (in our case the signal from the sensing crystal).
Quadrature (current)	Refers to the imaginary component of the current through the shunt capacitance, C_S.
Redox	An oxidation-reduction reaction; the term "redox" is obtained from the first few letters of "reduction" and "oxidation."
RTD	Resistance Temperature Detector. A device that changes its resistance as a function of temperature.
Shunt Capacitance	Effective capacitance due to the electrodes on the crystal. This is the "unwanted" capacitance we try to cancel out (along with the capacitance in the cable and the holder of course) while adjusting the Fine & Coarse capacitance cancellation on the PLO-10.CM.
Silanization	The chemical conversion of hydroxyl (OH) groups, which often act as adsorption sites on silica or glass stationary chromatographic phases, with silane coupling agents to give the inactive -O-SiR3 grouping. Silanization can neutralize surface charges, thus eliminating non-specific binding.
Teflon®	DuPont Company's registered trademark of Perfluoroalkoxy Fluorocarbon Resin, a class of Teflon that offers excellent inertness to aqueous acid and aqueous alkaline, superior resistance over a wide range of pH. Visit www.dupont-dow.com for more information.
Ultrasonic cleaning	This method utilizes high frequency (ultrasonic) and high intensity sound waves into a liquid producing cavitations (rapid formation and collapse of minute cavities in a cleaning liquid. For more information, visit http://www.aqueoustech.com/images/UltrasonicPrimer.PDF
UVO cleaning	Ultra-Violet/Ozone. The UVO cleaning method is a photo-sensitized oxidation process in which the contaminant molecules are excited and/or dissociated by the absorption of short-wavelength UV radiation. Near atomically clean surfaces can be achieved using this method. The basic instrumentation required this process includes a UVO chamber, a gas (oxygen) supply, or an exhaust system. For more information, visit http://www.jelight.com/uvo-ozone-cleaning.htm

VCO	Voltage Controlled Oscillator. An oscillator circuit designed so that the output frequency can be controlled by applying a voltage to its control or tuning port.
Viscoelastic	Having or exhibiting both viscous *and* elastic properties.
Viscosity	The internal friction of a fluid, caused by molecular attraction, which makes it resist a tendency to flow.
Viton®	DuPont Dow Elastomers' registered trademark of Fluoroelastomer, offers superior mechanical properties and resistance to aggressive fuels and chemicals, well known for its excellent heat resistance. Visit www.dupont-dow.com/viton for more detailed information.

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