Electrochemical Quartz Crystal Microbalance

Simultaneous electrochemistry and piezoelectric microgravimetry can be performed with the Autolab instrument and Electrochemical Quartz Crystal Microbalance (EQCM).

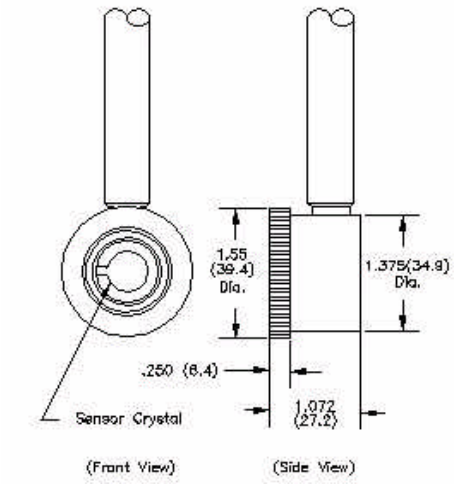
The EQCM that we recommend in combination with the Autolab instruments is the Maxtek PM-710 (MAXTEK Inc., USA) shown above. It consists of a power supply/frequency counter and a quartz crystal holder made of CPVC (MPS-550 probe), shown below.

The quartz crystal holder types can be used for quartz crystals with gold electrodes deposited on chromium adhesion layers. The technical characteristics of the crystal are shown in the following table.

Table 1: Specifications of a 5 MHz AT-cut quartz crystal.

Diameter	25.4mm
Au Area	1.37cm^2
Resolution	18.5 ng/Hz cm^2
Holder	39 mm ∅ 10 – 50 cm length

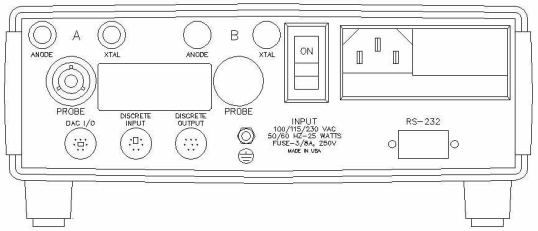
The quartz crystal is mounted vertically in the holder. The crystal can easily be exchanged from the topside of the holder. Due to the O-ring seal, no adhesive is required for crystal mounting.



The GPES software supports the EQCM, for the techniques:

- staircase cyclic voltammetry
- staircase linear sweep voltammetry
- chrono-amperometry
- chrono-coulometry
- chrono-potentiometry

The EQCM can be connected to ADC 1 or 2 (ADC 3 or 4 for the old housing of the Autolab) of the Autolab via a 7-pin mini-DIN (DAC I/O) connection on the back of the PM-710. This gives the possibility to measure thickness or deposition rate simultaneously with the current or potential signal.



The performance of an Autolab instrument equipped with the EQCM (5 MHz) is illustrated with simultaneous measurements of cyclic voltammetry and the change in thickness of a deposited layer (Figure 1).

It can clearly be seen from the curves in Figure 1, that from the onset of the Cu deposition (at 0.3 V going in the negative direction), the thickness of the deposited layer (or the amount of Cu deposited) starts to increase. This amount keeps increasing even when the potential is

scanned in the positive direction until the Cu layer starts to oxidise off (at –0.05 V going positive point "X"), then the thickness starts decreasing again, finally reaching the starting value at ca. 0.4 V (point "Y").

In a measurement like this, all the current that flows is connected with the deposition of Cu. This means that the curve of charge vs. potential and thickness vs. potential should have the same shape, both representing the amount of Cu that has been deposited (see figure 2).

Figure 1: EQCM measurement of Cu deposition on Au from a 50 mM Cu^{2+} solution. Curve B shows the cyclic voltammogram and curve A the simultaneously measured thickness of the deposited Cu layer. Scan rate 0.01 V/s. Potentials were measured against Ag/AgCl reference electrode.

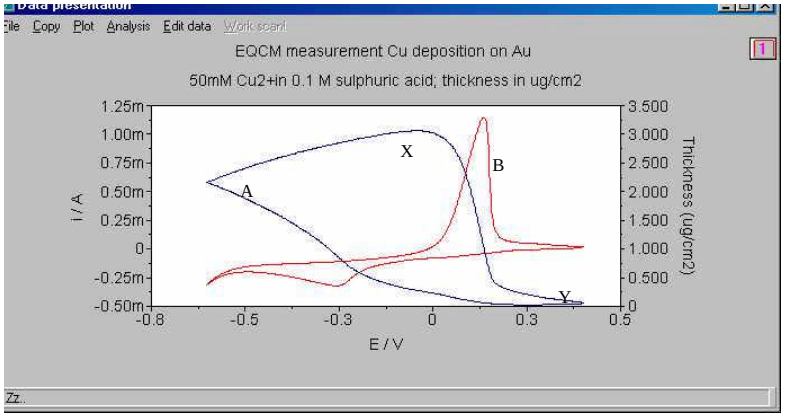


Figure 2: Comparison of the charge of the cyclic voltammogram (red or A) with the thickness of the deposited Cu layer (blue or B), both as function of potential.