

# **Developing a Non-Glass pH Meter**

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PCSE 498  
Physics Capstone Project Fall 2014 - Spring 2015

## Abstract

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I intended to develop and test a non-glass pH meter using an ion-selective field effect transistor (ISFET) for this project. The meter would have consisted of the ISFET, a reference electrode, and a voltmeter. The measurements made by this meter should agree with the Nernst equation which relates pH to potential difference.

Unfortunately, materials for this project were very hard to come by, most notably, the ISFET itself. In lieu of this situation, I built a glass pH electrode instead. I used a test tube, potassium chloride, and silver wire to make the glass electrode. Upon building this meter, I found that the readings produced were very random and gave no conclusive results on how well the pH meter worked.

# Introduction

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I am interested in measuring the pH of a solution over a prolonged period of time. I decided that a non-glass pH meter would be appropriate because it does not contain a liquid solution within it. After some research about pH meters, I discovered that all potentiometric pH meters have a reference electrode, and generally these electrodes contain a reference solution, so my non-glass meter might still require this reference solution. Because I did not want to have an internal solution in my pH meter, I thought it would be interesting to develop a simulated reference electrode along with the non-glass electrode.

The non-glass electrode to be used would be an ion-selective field effect transistor (ISFET) electrode. This is a transistor that is sensitive to the ion concentration in a solution, working similarly to a MOSFET. The current passing through the ISFET is proportional to the pH of the solution.

The purpose of the reference electrode is to maintain a constant voltage between the gate and the voltmeter of the pH meter. I planned to use a small voltage source or RLC circuit to provide a constant voltage in place of the reference electrode. I never continued with this thought of a simulated reference voltage, because I had problems even obtaining the ISFET as explained later.

Because the non-glass pH meter could not have been completed, I resorted to making a glass electrode just to continue with some of the same research I had already done. This electrode is simply dependent on the difference in ion concentration inside a glass bulb to that outside the bulb. This does require an internal solution and made my original plan more or less useless.

# Theory

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Measuring pH is determining the concentration of positive hydrogen ions ( $H^+$ ) in a solution. The pH scale is the logarithmic scale ranging from 1 through 14 describing the concentration in powers of ten. For example, a solution with a pH of 7 has a concentration of  $10^{-7}$  moles per liter.

This project focuses on measuring pH using potentiometric means using the Nernst equation to relate a potential difference to pH. The Nernst equation relates the potential,  $E$ , to the pH linearly. This linear relationship has a slope proportional to the temperature,  $T$ , by a factor of a ratio of the ideal gas constant,  $R$ , and Faraday's constant,  $F$ . Ideally,  $E_0$  is a number such that  $E=0$  at  $pH=7$ , but can vary from sensor to sensor, and is calibrated accordingly. All pH meters have two electrodes: one that is somehow sensitive to the ion concentration and another reference electrode that creates a bias and completes the circuit.

$$E = E_0 - \ln 10 \cdot \frac{R \cdot T}{F} \cdot pH$$

Or more colloquially:

$$E = E_0 - kT \cdot pH$$

For a glass electrode, on which this project focuses, the potential difference occurs due to a difference in  $H^+$  concentrations in two solutions. A glass electrode is made of a glass tube, a reference solution, and silver chloride wire. The key element of this system is a hydrated gel layer formed around the glass tube. The gel layer has the property to allow  $H^+$  ions to permeate and surround the tube, but not larger  $OH^-$  ions. There is another gel layer on the inside of the tube with the reference solution. For instance, when the tube is submerged in a solution with a  $H^+$  concentration higher than that of the reference solution, there is a positive potential difference from the measured solution to the reference solution.

For a non-glass electrode, which was the original purpose of this project, the potential difference comes from the current crossing the field effect transistor. The key element of this system is the ion-selective (thus ISFET) gate of the transistor. The oxide surface of the gate attracts  $H^+$  ions in the solution, which in turn attracts electrons in the semiconductor beneath the gate. When the gate is exposed to a solution and there is a source-drain voltage, a current, whose magnitude is determined by the concentration of  $H^+$  ions, flows from source to drain. For this to occur, an external voltage source would need to be applied across the reference and the drain. This is because the source-drain potential difference relies on the resistance of the ISFET with a particular current. Being able to adjust that external voltage would allow one to maintain a constant resistance, but this is usually embedded on a chip containing the ISFET with an amplifier with feedback.

In both cases, a reference electrode is necessary to contain the reference solution so that each electrodes' wires are in the same solution. Even more importantly, the reference electrode does this while maintaining electrical connection with the measured solution to complete the circuit. These two electrodes are then submerged in the same solution to measure and a voltmeter can be used to determine the potential across them.

## Methods

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### Construction

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The early plans for this project called for a non-glass solution. A design was developed using the MO-PSF02 ISFET from Micropto. The device would have included this chip encased in some body with just the ion-selective gate exposed, electrically connected to the reference electrode

and the voltmeter. I would have also used a temperature correcting chip that would have provided further bias and accuracy in the meter.

The ISFET electrode could not be built because I was unable to purchase one, so I began to develop a glass electrode instead. I created two electrodes and two reference electrodes to account for damages or just faulty parts. The materials for each glass electrode included a glass test tube, jewelers' silver wire, a saturated solution of potassium chloride, and water-resistant caulk. The reference electrodes were made of a plastic water hose, the same wire, the same potassium chloride solution, water-resistant caulk, and a membrane that will allow current to flow without mixing the reference solution and the solution to be measured.

The first and longest processes were creating the hydrated gel layers on the test tubes and chloridizing the silver wire. The gel was formed by allowing the test tubes to sit in a bath of vinegar, a fairly strong acid, gently rinsed, followed by allowing to sit in a bath of bleach, a strong base, followed by another gentle rinse and keeping them in distilled water to keep them hydrated. They were allowed to remain in each bath for about three to four hours. Meanwhile, the silver wire was cut into lengths a few inches longer than the tubes and then laid out in straight sticks, and submerged in bleach for eight hours to allow silver chloride to form on the surface. This includes the wires for the reference electrodes. The chlorine ions that bond with the silver make it easier for the wire to interact with other ions in the reference solution in the tube creating a better voltage reading.

While the wires were chloridizing, the reference electrode body could start to be built to allow the caulk used to be set. The plastic tubing was cut into pieces about the length of the glass electrode. Then, a small amount of caulk was applied to one end of each tube and adhered to some semipermeable plastic film.

The assembly thereafter required that all the tubes, both glass and reference, be stabilized with the opening on the top. They were all filled with the reference solution and then sealed with caulk. After the caulk has had time to set, a wire was used to pierce the caulk and insert a chloridized silver wire so that the wire nearly touched the bottom of the tube and still significant length coming from the top. More caulk was applied to reseal the piercing.

After an initial failure of that design, it was modified to produce a better gel layer and make the wire more secure. To make the wire more secure, a stopper was custom fitted to the tube and had a hole in it for the wire. This stopper was less flexible than the caulk, which makes the wire shift less in the tube, if that caused reading errors. It also allows access to the solution, should it need to be refilled.

## Testing

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The first round of tests for the project included a lineup of pH buffers from 3 to 11 ranging along only odd pH values. The electrodes were connected to a voltmeter, glass electrode on the positive and reference electrode on the negative. Then they were fastened together and held as to eliminate as much movement as possible and maintain a constant distance from each other. The temperature will also be recorded as the measurements are being taken.

These voltage readings will be plotted and compared to the expected Nernst function for the temperature recorded. This curve will be fitted as linear and can then be used to calibrate the meter itself for all pH's thereafter. Upon determining a successful model for the meter, it will be tested over a range of fluids against a commercial pH meter.

## Data

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The following table of data was taken using the two electrodes made previously, using the same reference electrode each time. A few glasses were filled with the same level of each buffer, and then each was measured using one electrode and then the other. The expected value assumes  $E_0$  of the Nernst equation is such a value where the line would intersect the x-axis (should there have been one) given the temperature. The temperature was 22.7 degrees Celsius, which means the slope ( $kT$ ) was  $-0.0596\text{mV/pH}$ . That means  $E_0$  was 0.4186, but I didn't expect this to be very accurate compared to the measured results. These were used to calculate the expected results.

pH buffer	First Electrode (mV)	Second Electrode (mV)	Expected
3	4	-18	0.238
5	-130	-83	0.119
7	70	120	0
9	-65	-44	-0.119
11	139	18	-0.238

## Observations

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These readings were incredibly inaccurate and imprecise. There were some small problems about the wires moving by tension from the multimeter, which were evidently causing spastic values. More effort was put in to maintain some stillness in the electrodes, which resulted in equally

random values, but much more consistent for each reading. In general, the readings were far from useful, and obviously should be considered inconclusive.

## Discussion and Conclusion

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This project has deviated significantly from its original intent. Originally, this project was supposed to be a research project on ISFETs as pH meters. I wanted to obtain an ion selective field effect transistor and do as many tests on it as I could and compare it to the glass electrode approach. Unfortunately, after much research on purchasing ISFETs, it became clear that they are not readily available for consumer use. The common uses for these transistors appear to be strictly oriented toward expensive commercial pH meters and catheters. Beyond that, there is only some research being done.

A significant portion of my time, between research, was contacting people and waiting for responses regarding the sales of ISFETs. It started becoming hopeless until Dr Gore managed to get someone's attention, and eventually gained an estimate sheet from Micropto. To our dismay, the prices were a little bit more than my budget allowed, which put this project in yet another state of delay.

Since then, I have tried to adjust the project to developing a glass electrode and testing it. The results of this made it apparent that the theory was much harder to implement than expected. Upon taking measurements, I had expected to see some clear (whether accurate or not) difference between the readings of a strong acid against those of a strong base. The data shows a wide range of approximately  $\pm 150\text{mV}$  in no particular order. I can attribute this to several potential factors, but three most strongly. First, the glass of the test tube created too much resistance. Second, the wire I used contained far too little silver to be useful. The quality of the build could have been improved.

The glass of the test tubes were most likely too thick and possibly made of the wrong material. A typical glass electrode membrane is about 0.1mm and contain sodium ions ( $\text{Na}^+$ ) which can flow slowly though the glass medium as charge carriers. With these specifications, the membrane usually has a resistance with an order of magnitude close to  $10^8$  Ohms. The test tube I used had a thickness of 1mm and was most likely made from a borosilicate glass. The thickness itself increases the resistance. Furthermore, borosilicate glass actually resists the flow of  $\text{Na}^+$  through the glass. It could be possible that the current was far too low to measure any voltage drop with the multimeter used.

The initial wire obtained for this project claimed to be "silver coated." It was quite obvious that the interior was a copper or brass, but the outside was not initially doubtful. However, when I set the wire in the bleach, I was unable to see much chloridizing. If the wire did not have a silver chloride coating, means that there cannot be a redox reaction between the silver and silver

chloride which improves current flow from reference solution to wire. In the event that the wire contained no silver at all, it is even possible that the wire was not very conductive at all.

The tubes built were still very much in prototype state upon testing. Upon more research about the problems with the data, it became clear that the tube itself was the largest issue, so they were never improved upon. A few structural changes could have been made to better the product however. The wires were held in place by a caulk that also simply acted a water-tight seal, but it did not keep the wire still inside the solution and allowed for more wiggling that desired. A better solution would have been to use a thick stopper that the wire could have been fed through, which could have been water-tight and held the wire much tighter and prevented more movement. Also, the glass and reference electrodes were not a fixed distance apart. This factor should not have been too significant a factor, but it should certainly be noted and a better solution would have fixed their relative positions to each other.



## Bibliography

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PRELIMINARY



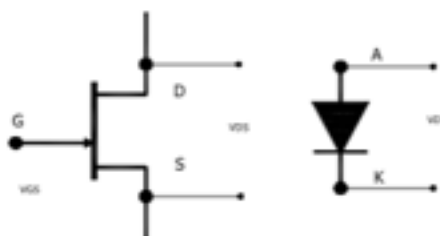
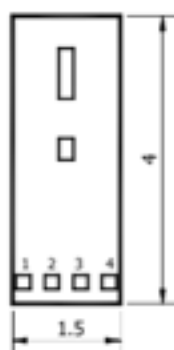
**MICROPTO**

**MO-PSF02**

**SPECIFICHE TECNICHE ISFET  
ISFET TECHNICAL SPECIFICATIONS**

*Bozza – Draft Copy (vers. 2.1)*

- Sensore di pH in silicio con incorporato sensore di temperatura per compensazione
- ISFET canale-n
- Dimensioni del chip: 1,5 mm x 4 mm
- *Silicon pH chip with embedded temperature sensor for compensation*
- *ISFETs n-channel device*
- *Chip size: 1,5 mm x 4 mm*



PARAMETER	SYMBOL	UNIT	MIN	TYP	MAX	NOTES
Sensitivity	S	mV/dec		59		@ 25° Celsius
pH Range		pH	0*		14*	
Hysteresys		mV		± 10		
Drift		mV/day			0.2	To define
Leakage current		nA		10	20	@ Vgs= Vi - 1 V (where Vi is the threshold voltage)
Drain Source Voltage				n.a		
Threshold Voltage				n.a		
Temperature diode dependance		mV / K		2		

(\* main restriction due to the package)

Gate material : Ta<sub>2</sub>O<sub>5</sub>

Gate dimensions: 20 µm x 700 µm

ISFET gate type: N

Stock and Operating Temperature: -20; +130°C (\*)

External reference: AgCl or SSR

I<sub>Drain</sub> = I<sub>diode</sub> = constant = 100 µA

V<sub>DS</sub> = constant = 500 mV

Devices with Ta<sub>2</sub>O<sub>5</sub> (new) gate oxide have a slope of 59 mV at 25 C, and a linear region of at least 1-13 (tested). Light sensitivity is less than 2 mV in laboratory conditions, and the final drift less than 2 mV per day. For our testing, we drive the devices at V<sub>ds</sub>= 0.5 V, I<sub>d</sub>= 100 microA. The working point in pH 7 vs Ag/AgCl will vary a bit, the Al<sub>2</sub>O<sub>3</sub> (old) chips will be between -2 and +2 V, the Ta<sub>2</sub>O<sub>5</sub> devices have a working point shifted in negative direction, between -4 and 0 V.

Dispositivo sensibile alle scariche elettrostatiche, luce e temperatura / *Device is sensitive to ESD, light and temperature.*

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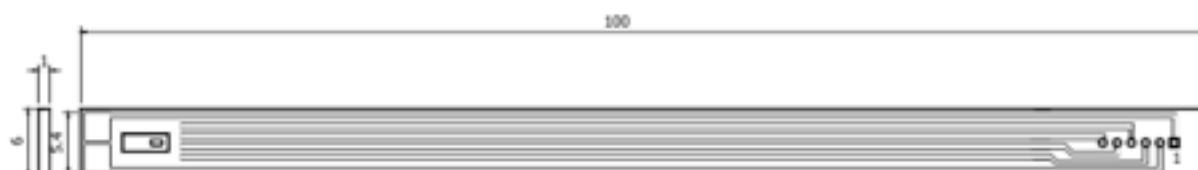
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Standard package (MO-P34 vers. 2)

Unit : mm

Esempio di PCB per sonda elettrodo (chiedere se disponibile) / *Sample of probes for electrodes PCB (ask if available)*

USCITA/ PIN OUT	DESCRIZIONE/ DESCRIPTION	USCITA/ PIN OUT	DESCRIZIONE/ DESCRIPTION	USCITA/ PIN OUT	DESCRIZIONE/ DESCRIPTION
1 (squared pad)	Aux 1	3	Drain	5	n <sup>+</sup> Temperature Diode
2	Aux 2	4	Source/P Well	6	P Temperature Diode



USCITA	DESCRIZIONE	USCITA	DESCRIZIONE
1	Drain	3	P Temperature Diode
2	N+ Temperature Diode	4	Source/P Well

Chip / *Present microchip*

**Note:** COB e altri packages su richiesta OEM / *Other packages OEM on request*

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**PC-FacSimile Transmission/PDF**

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<b>Page(s):</b> 4	
<b>FAX-μ02165</b>	Milano, lunedì 1 dicembre 2014

**Subject: Quotation of ISFET sensors and references**

Dear Dr. Gore,

thank you for the interest in our activity, here I write the prices about the products required.

Item	Model	Draw/ study (Euro)	Master/ toolings (Euro)	10 pcs. (Euro/pc.)	100 pcs. (Euro/pc.)	500 pcs. (Euro/pc.)	1000 pcs. (Euro/pc.)	10K pcs. (Euro/pc.)	Notes
A	ISFET chip on PCB (MO-P143)	no	no	46,8	35,6	29,5	24,2	--	Small package standard MO-P143 with assembled one ISFET chip+ globe top. See specs sent by email.
		--	--	3 weeks	3 weeks	4 w	6 w	8 w	
B	ISFET chip on <u>new</u> PCB base	350	420	46,8	35,6	29,5	24,2	--	Specific customized package or small probe /OEM
		1 week	3 weeks	3 weeks	3 weeks	4 w	6 w	8 w	
C	ISFET chip on PCB 100x6 mm <sup>2</sup>	no	no	46,8	35,6	29,5	24,2	--	Strip with ISFET die and globe top. Standard MO-P34
		--	--	1 week	1 week	4 w	6 w	8 w	
D	Solid state reference	no	890	0,9	0,9	0,9	0,8	0,7	Solid state reference (resin) on customer PCB. Small drop on PCB
		--	2 weeks	1 week	1 week	2 w	3 w	4 w	
E	Standard glass reference	no	no	155	ask	ask	ask	ask	Standard glass AgCl electrode of reference 12mm x 120mm others on requests
		--	--	2 week	--	--	--	--	
F	Evaluation board	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	--	Developing

**Descriptions:** "no" not required; "n.a.": not available; "--" to define/ask.

**NOTE**

- ❖ ISFET die is a critical device, it's sensitive to ESD (ElectroStatic Discharges), light, temperature and time drift. It needs to be calibrated and handled with care. Typically the electrode or the probe that guest ISFET microchips are provided on proper shields for the chip.



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- ❖ ISFET chip packaged needs external chemical AgCl reference as depicted in Micropto ISFET datasheets.
- ❖ Micropto is able to design new electronics and new packaging on customer requests.
- ❖ Package proposed in this table is one of the standard available in Micropto, other packages could be defined by the customer (OEM). Ask for the package of your interest if available as standard in Micropto otherwise we can manufacture it.
- ❖ Discounts available on quantity
- ❖ We can adopt several kind of globe top resin as required by the customer but we need a minimal quantity if not ready at stock.
- ❖ Time to delivery are depicted in the table. May change in some conditions.
- ❖ Microchips may change or be improved in the time (without revolution of technical characteristics) without prior notice. We are still working to improve this product and reduce the prices.

#### TECHNICAL SPECIFICATION AgCl reference electrode:

Standard reference AgCl with following characteristics



Elettrodo di riferimento Ag/AgCl, elettrolita a gel di KCl. Non richiede alcun rabbocco di elettrolita. /

*Electrode for references Ag/AgCl, KCl gel electrolyte. It doesn't need refilling electrolyte.*

Corpo in vetro Ø 12 mm lunghezza 120 mm / *Glass structure, diameter: 12mm; length 120mm.*

Limiti temperatura di impiego: 0-40° C / *Operative temperature: 0-40° Celsius.*

Limiti pressione operativa: 1 Bar / *Operative pressure: 1Bar*

Cavo integrale da 1 metro senza spina / *1 meter standard cable without plugs.*

Altre dimensioni su richiesta / *Other versions on request.OEM*

Alternatives to Standard Glass reference electrode: Ag pasta (SSR: solid state reference).

#### GENERAL CONDITIONS

Shipment:	EXW Micropto Milan (Italy) - by your usual courier account (FedEx, DHL, UPS, ...) If required we can charge the costs for the shipping with our courier in the invoice.
Delivery:	after receipt of your write order and remittance, pls. see the table
Term of payment:	T/T in advance on Micropto's proforma invoice by bank (or by Paypal).
Currency:	Euro.
Handling and package charge:	included (standard plastic bags; ask for tape&reel or others).
Others:	these prices and conditions are valid within 30 days from today. This quotation substitute and update older offers.
MOQ (Minimum order quantity):	5 pcs.

#### NOTE

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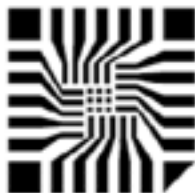
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Best regards

Fabio Carini

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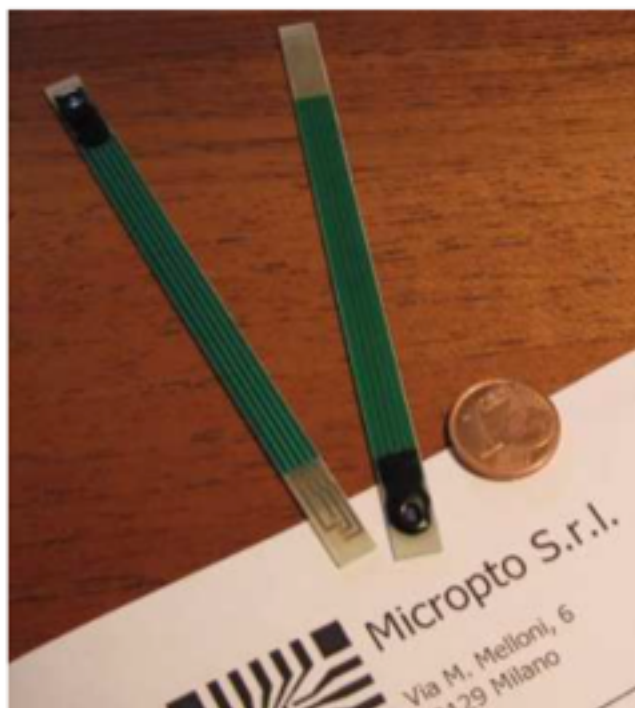
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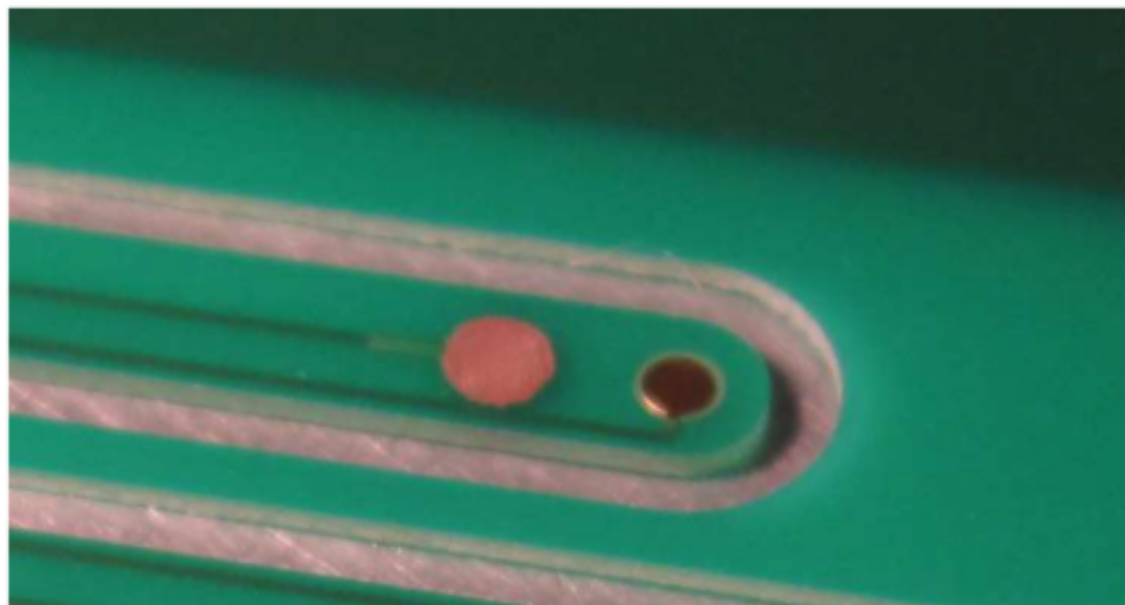
Some pictures about Micropto's ISFET sensors packages:



ISFET chip on PCB (old MO-P34): 100x6 mm<sup>2</sup>  
(D+ S+ T1 + T2 + Aux1 + Aux2 ref.)



ISFET chip on PCB (MO-P143)  
(D+ S+ T1 + T2)



SSR: Solid State Reference, the grey dot  
Pictures are a rough description, other models are available.