

# Potentiometric Determination of Trace Amounts of Mercury (II) in Water Sample Using a New Modified Palm Shell Activated Carbon Paste Electrode Based on Kryptofix<sup>®</sup>5\*

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## ABSTRACT

A new modified palm shell activated carbon paste electrode based on 1,13-Bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane, 8,8-(1,4,7,10,13-Pentaoxatridecylene)-diquinoline (Kryptofix<sup>®</sup>5) and plasticizing agent was prepared and studied as Hg<sup>2+</sup> selective electrode. The best performance was observed with the electrode composition having the ionophore-palm shell activated carbon-plasticizer composition 10%:50%:40% with Nernstian response over the concentration range of  $1.0 \times 10^{-8}$  -  $1.0 \times 10^{-2}$  M with a slope of  $42 \pm 1.5$  mV per decade of concentration. The detection limit as determined from the calibration plot is  $1.0 \times 10^{-7}$  M. The proposed electrode shows good selectivity for Hg(II) with interfering ions. The response time of the electrode is fast ( $\leq 10$  s), and can be used in the pH range of 3 - 11. The electrode was used to determine mercury in drinking water.

**Keywords:** Palm Shell Activated Carbon; Kryptofix<sup>®</sup>5; Ion Selective Electrode; Mercury

## 1. Introduction

Contaminate water recourses by heavy metal is a serious worldwide environmental problem. Many metals such as mercury, cadmium, chromium and lead are known to be significantly toxic [1].

The importance of mercury to the environment cannot be overemphasized. From an environmental point of view, it is highly toxic, causing severe damage to the human central nervous system. Mercury concentrations in drinking, river or sea water are typically below  $5 \mu\text{g}\cdot\text{L}^{-1}$ , making it extremely difficult to measure using many techniques.

Therefore, measurement of free Hg(II) is important in assessing mercury toxicity and environmental monitoring. A number of methods, such as atomic absorption spectrometry (AAS) [2,3], inductively coupled plasma [4], X-ray fluorescence spectrometry [5], anodic stripping voltammetry [6,7] and potentiometry [8,9] have been used for determination of mercury in the analytical samples. Many sensors for potentiometric determination of mercury(II) based on carbon paste CPE [10-16], polymeric membranes PVC [17-25] and coated wire electrodes [26-30] have been reported. However, these elec-

trodes are not very fruitful as they have either one, two, or in some cases, all the following problems: 1) a high detection limit; 2) a narrow working concentration rang; 3) a long response time; 4) serious interferences from various cations.

Potentiometric sensors based on ion-selective electrodes are especially suited for determination of several chemical species because they offer advantages such as selectivity, sensitivity, good precision, simplicity, and low cost.

Carbon-paste electrodes are considered a category of ion-selective electrodes that is composed of a carbon powder with a pasting liquid (an organic binder). The advantages of carbon paste electrodes such as ease of preparation and use, renewal of surface, chemical inertness, robustness, stability of response, low ohmic resistance, no need of internal solution and suitability for a variety of sensing and detection application drew the attention of researchers in recent years where these advantages were exploited for various measurements, including potentiometric measurements.

Palm shell activated carbon is a waste produced during palm kernel oil production, which is an important sector of Malaysia's economy. This material is easily available at low cost and has good electrochemical properties, such

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as good electrical conductivity, chemical and electrochemical inertness, favorable electrochemical behavior with analytes, low background current, and high surface area [31,32].

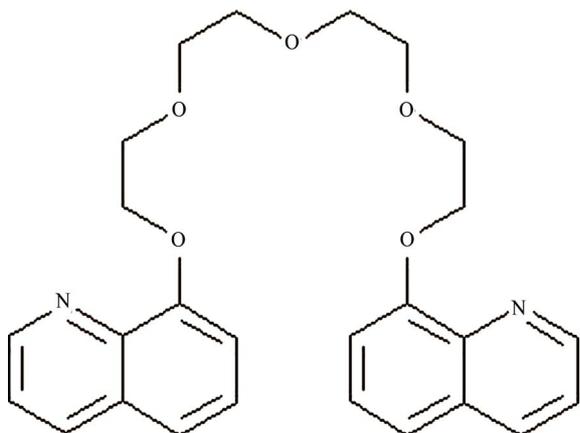
In this work, the application of Kryptofix<sup>®</sup>5 as an ionophore shown in **Figure 1** have been discussed for the detection of Hg(II) ions in drinking water samples. The sensor responds to Hg(II) ions. The recognition of small molecules in binding with heavy metals has gained importance in the field of research. The Kryptofix<sup>®</sup>5 has two donating nitrogen atoms and five oxygen atoms, low molecular weight and flexible structure were expected to act as a suitable ionophore in the preparation of carbon paste sensors for mercury ions of proper size and charge.

Structure of 1,13-Bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane,8,8-(1,4,7,10,13-Pentaoxatridecylene)-di-quinoline (Kryptofix<sup>®</sup>5).

## 2. Materials and Method

### 2.1. Materials

All analytical reagent grade chemicals and distilled, de-ionized water were used for preparing all aqueous solutions. Commercial granular palm shell activated carbon (PSAC) was provided by Pacific Activated Carbon a local manufacturer in Johor Bahru, Malaysia. Activated carbon granules with particle size <45  $\mu\text{m}$  were used throughout the experiments. PSAC was washed with distilled water and was dried in an oven at 100°C for 24 h. Metal salts as well as the plasticizers, bis(2-ethylhexyl)-adipate (DOA), dioctyl phthalate (DOP), tris(2-ethylhexyl) phosphate (DOPh), dioctyl sebacate (DOS), butyl phosphate (BPh), dibutyl phosphate (DBPh), and tributyl phosphate (TBPh) were purchased from Merck. The ionophore Kryptofix<sup>®</sup>5 was purchased from Sigma-Aldrich.



**Figure 1.** Structure of 1,13-Bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane,8,8-(1,4,7,10,13-Pentaoxatridecylene)-di-quinoline (Kryptofix<sup>®</sup>5).

### 2.2. Experimental Equipments

All potentiometric measurements were made with a pH/Ion meter (Metrohm-781, Germany) and pH Module (Metrohm-867) using proposed sensor in conjunction with a double junction Ag/AgCl reference electrode. The temperature of the cell holder was maintained at 25°C. The electrochemical cell used for this study as follow:

Ag(s), AgCl(s), KCl(3M sat.) sample solution modified palm shell activated carbon paste electrode.

Scanning electron micrographs (SEM) of the electrode surfaces were obtained by using scanning electron microscopy (AMETEK, advanced microanalysis solutions) at an accelerating voltage of 25 kV.

Hg(II) samples were analysed by Inductivity Couple Plasma ICP (PerkinElmer, model ICP optima 7000DV).

### 2.3. Palm Shell Activated Carbon Paste Electrode Preparations and Potential Measurements

Modified palm shell activated carbon paste was prepared by mixing of specified amount, as shown in **Table 1**, of palm shell activated carbon powder, Kryptofix<sup>®</sup>5 and plasticizer. The constituents were thoroughly hand mixed in a 50 mm petridish to produce a paste according to optimum value of ingredients, then the paste poured and packed into empty glassy carbon electrode (5 mm diameter), connected with the pH/Ion meter by a thin copper wire to produce electrical contact. The composite surface was smoothed on a weighing paper until the surface showed shiny appearance and rinsed carefully with double-distilled water prior to each experiment.

The potentiometric measurements were conducted as follows. The modified carbon paste electrode was placed in a stirred 50 ml 0.1 M Hg<sup>2+</sup> solution for a given period of time until the potential reading became constant. The addition method was used for investigating the electrode response characteristics. Mercury salts standard solutions were added, so that the mercury concentration ranged between 10<sup>-8</sup> and 10<sup>-1</sup> M. Potential readings were recorded after each addition, when stable values had been obtained (usually after 30 - 60 s). Potentiometric selectivity of this electrode towards different cations were calculated by using the matched potential method (MPM) [33]. In this method, the activity of Hg(II) was increased from  $a_A = 1.0 \times 10^{-5}$  M (primary ion) to  $\acute{a}_A = 5.0 \times 10^{-5}$  M, and the corresponding potential change ( $\Delta E$ ) were measured. Then a solution of an interfering ion ( $a_B$ ) in the range  $1.0 \times 10^{-1}$  -  $1.0 \times 10^{-2}$  M was added to a new primary ion ( $\acute{a}_A$ ) until the same potential change ( $\Delta E$ ) was recorded. The selectivity factor,  $K^{\text{pot}}_{A,B}$  for each interferent was calculated using the following:  $K^{\text{pot}}_{A,B} = (\acute{a}_A - a_A)/a_B$ .

**Table 1. Palm shell activated carbon paste electrode compositions and general performance characteristics of mercury (II) ion selective electrode.**

Electrode No.	Electrode composition (wt%)			Electrode characteristics			
	Ionophore	PSAC	DOP	Slope (mV)	Linear range (M)	Detection limit (M)	Response time (s)
1	0	55	45	55.52	$10^{-2} - 10^{-5}$	$1 \times 10^{-4}$	15 - 20
2	0.2	55	44.8	61.86	$10^{-2} - 10^{-6}$	$1 \times 10^{-6}$	15 - 20
3	0.5	55	44.5	59.55	$10^{-2} - 10^{-6}$	$1 \times 10^{-6}$	~15
4	1	54	45	54.07	$10^{-2} - 10^{-5}$	$1 \times 10^{-5}$	~15
5	2	54	44	53.73	$10^{-2} - 10^{-6}$	$1 \times 10^{-6}$	~15
6	4	54	42	62.08	$10^{-2} - 10^{-7}$	$1 \times 10^{-6}$	~15
7	5	50	45	61.46	$10^{-2} - 10^{-7}$	$1 \times 10^{-7}$	~10
8	6	52	42	64.46	$10^{-2} - 10^{-6}$	$1 \times 10^{-7}$	~15
9	8	50	42	55.03	$10^{-2} - 10^{-7}$	$1 \times 10^{-7}$	~15
10	9	47	44	36.12	$10^{-2} - 10^{-5}$	$1 \times 10^{-6}$	~10
11	10	50	40	42.41	$10^{-2} - 10^{-8}$	$1 \times 10^{-7}$	< 10
12	15	35	50	47.23	$10^{-2} - 10^{-6}$	$1 \times 10^{-6}$	~15
13	20	30	50	56.90	$10^{-2} - 10^{-6}$	$1 \times 10^{-4}$	15 - 20
14	20	40	40	48.60	$10^{-2} - 10^{-5}$	$1 \times 10^{-4}$	15 - 20

### 3. Results and Discussions

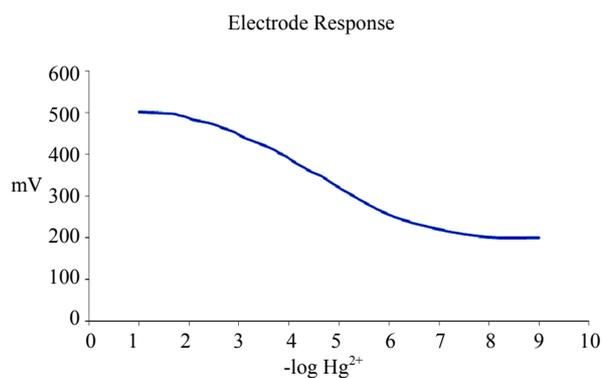
#### 3.1. Response of the Electrode

The calibration for the  $\text{Hg}^{2+}$ -selective electrode over a wide range of solution  $\text{Hg}^{2+}$  activities is shown in **Figure 2**. The slope of the calibration curve (42.41 mV/dec) closes to that predicted theoretically (58.5 mV/dec) by the Nernst equation, indicating that the electrode was sensitive to  $\text{Hg}^{2+}$  over a wide range of  $\text{Hg}^{2+}$  activities ( $10^{-7}$  fold).

In addition, the electrode showed a linear response over this range of activities, showing departure from linearity (*i.e.* loss of sensitivity) at activities lower than  $10^{-7}$  M  $\text{Hg}^{2+}$ .

#### 3.2. Effect of Plasticizer

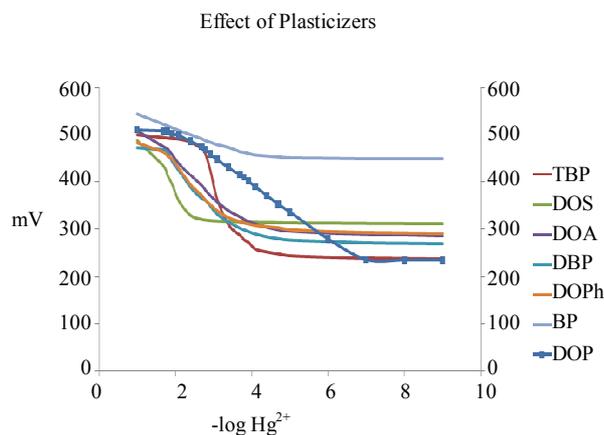
The performance of an ISE electrode depends on its composition, especially, the plasticizer [34,35]. A plasticizer is added to ensure a non-interruption mobility of the ions in the paste. The proportion of plasticizer used must be optimized in order to minimize the electrical asymmetry of the paste, to keep the sensor as clean as possible, and to stop leaching to the aqueous phase [36]. It also determines the polarity as well as provides mechanical strength to the electrode. It is well established that the polarity and chemical structure of the plasticizer can have a significant influence on the sensitivity, stability,



**Figure 2. The calibration curve for a modified palm shell activated carbon paste electrode over a wide range of solution  $\text{Hg}^{2+}$  activities.**

the selectivity and dynamic response range of ISEs [37]. **Figure 3** shows the effect of various plasticizers on the performance of the mercury ion selective electrode. DOP was found to have the widest linear range of the electrode and the best detection limit among those tested.

The good performances are exhibited in Nernstian slope of 42.41 mV/decade, linearity of concentration ranges between  $1.0 \times 10^{-2}$  and  $1.0 \times 10^{-8}$  M  $\text{Hg}^{2+}$ , LOD at  $1.0 \times 10^{-7}$  M. Average time required for every successive measurement to reach stable readings with  $\pm 2$  mV is less than 10s. There is no significant change of slope of Nernst plot over a period of 30 days. Other plasticizers,



**Figure 3.** Effect of various plasticizers on the performance of the proposed mercury ion selective electrode.

*i.e.* DOA, DBP, DOS, TBP, BP and DOPh are unsuitable as they give sub-Nernstian slopes, slower and narrow linear concentration ranges.

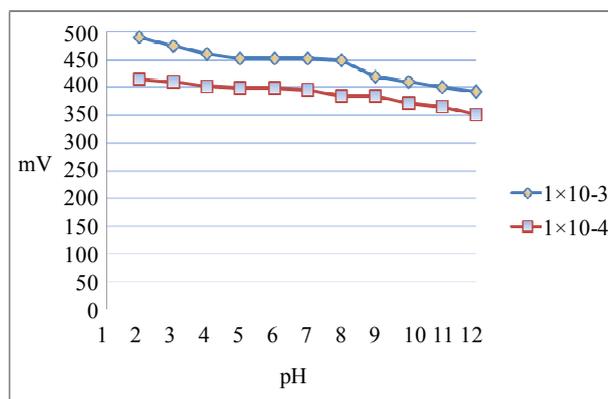
### 3.3. Effect of pH on Electrode Response

The effect of pH of the test solutions ( $1.0 \times 10^{-4}$  M and  $1.0 \times 10^{-3}$  M  $\text{Hg}^{2+}$ ) on the electrode potential was investigated by following the potential variation of the electrode over a pH range of 1 - 12. The pH was adjusted by introducing small drops of hydrochloric acid (1 M) and/or sodium hydroxide (2 M) to the sample solution. **Figure 4** shows the influence of pH on the potential response of the electrode in the sample solution on the potential response of the developed palm shell activated carbon paste electrode toward  $1.0 \times 10^{-4}$  M and  $1.0 \times 10^{-3}$  M  $\text{Hg}^{2+}$ . The experimental results show that the potential remained constant in the pH range of 3 - 11, which can be used as the working pH range of the proposed electrode. However, outside this range, the electrode responses changed significantly. The diminished potential at  $\text{pH} > 11$  was due to the interference of  $\text{OH}^-$  on the plain. The response at  $\text{pH} < 3$  seemed ascribable to the competitive blinding of protons to the ligands on the electrode surface.

Under more acidic conditions, the ligand may be protonated and thereby losing its capacity to form a complex with the metal ions. When the pH is nearly neutral, the fundamental cation is  $[\text{HgOH}]^+$ , which reacts with the ligand. The drift of potential values at pH is attributed to the formation of mercury(II) hydroxide [38,39].

### 3.4. Potentiometric Selectivity Coefficients

The influence of interfering ions on the response behavior of an ion-selective electrode has usually been described in terms of selectivity coefficient. Potentiometric



**Figure 4.** Effect of pH on the potential response of  $\text{Hg}^{2+}$  palm shell activated carbon paste electrode.

selectivity factors of the proposed mercury ion selective electrode over other ions was very important. The values of the selectivity coefficients  $K^{\text{pot}}_{A,B}$  of the proposed electrodes towards different species (B) **Table 2**, were determined by the matched potential method.

As shown in **Table 2**, it can be observed, that the proposed electrodes exhibited better selectivity for  $\text{Hg}(\text{II})$  ions.

Alkaline and alkaline earth metal ions did not interfere with the potentiometric response of the proposed electrodes. Some transition metal ions exhibited a light interference effect on the  $\text{Hg}^{2+}$  electrodes, which was more elevated in the proposed electrode.

### 3.5. Response Time and Life Time

The response time, defined as the time elapsed from the dipping of the electrode in the solution until the equilibrium potential was reached, varied from 5 to 10 s depending on the analyte concentration. The response time of the electrode was faster when the concentration was higher. The actual potential vs. time traces are shown in **Figure 5**. The sensing behavior of the membrane electrode did not depend on whether the potentials were recorded from low to high concentrations or vice versa.

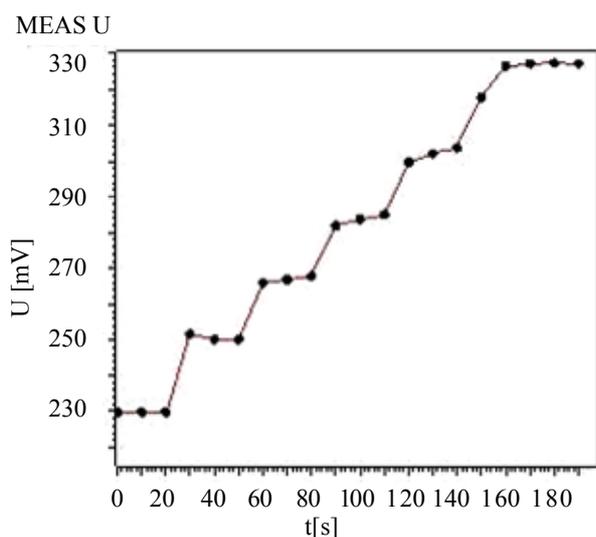
The proposed electrode can be used for one or more months without any deterioration or change in the response of the electrode.

### 3.6. Scanning Electron Microscopy Results

**Figure 6** compares the typical morphological features of palm shell activated carbon (PSAC) and palm shell activated carbon paste electrode based on Kryptofix<sup>®</sup>5 as ionophore (PSACPE) using FESEM. Pure palm shell activated carbon PSAC surface (**Figure 6(a)**) have porosity properties, smooth surfaces with long linear ridges and rough surfaces with oval ridges and micropores. **Figure 6(b)** shows a SEM image of PSACPE with more

**Table 2.** Selectivity coefficients of various ions B with Hg<sup>2+</sup> activated carbon paste ion selective electrode based on Kryptofix® 5 using matched potential method (MPM).

Interferent, B	$-\log K^{\text{pot}}_{A,B}$	Interferent, B	$-\log K^{\text{pot}}_{A,B}$
Cu <sup>2+</sup>	0.86	Na <sup>+</sup>	4.55
Cd <sup>2+</sup>	3.81	K <sup>+</sup>	4.45
Ca <sup>2+</sup>	4.50	Ni <sup>2+</sup>	3.50
Mg <sup>2+</sup>	4.15	Cr <sup>3+</sup>	3.83
Zn <sup>2+</sup>	3.55	Co <sup>2+</sup>	3.48
Al <sup>3+</sup>	2.80	A <sup>+g</sup>	1.89
Fe <sup>3+</sup>	2.45	Pb <sup>2+</sup>	2.55



**Figure 5.** Response time of the palm shell activated carbon paste electrode for Hg<sup>2+</sup> with changes of concentration from  $1.0 \times 10^{-7} \text{ mol}\cdot\text{L}^{-1}$  to  $1.0 \times 10^{-4} \text{ mol}\cdot\text{L}^{-1}$ .

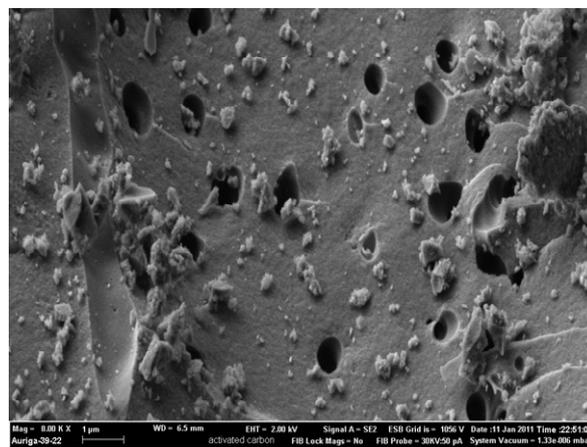
uniform surface topography and unique structure. Significant differences in the surface structure of PSACPE and PSACPE surface after it was dipped in  $1 \times 10^{-4} \text{ M Hg}^{2+}$  solution for 30 min (**Figure 6(c)**) are observed. Scanning electron microscopy images indicate significant improvement in the microstructure of the proposed electrode.

Aggregates of white needles were observed in the surface (**Figure 6(c)**), which could be presumed to be complexes formed between the Kryptofix® 5 and Hg<sup>2+</sup> ions.

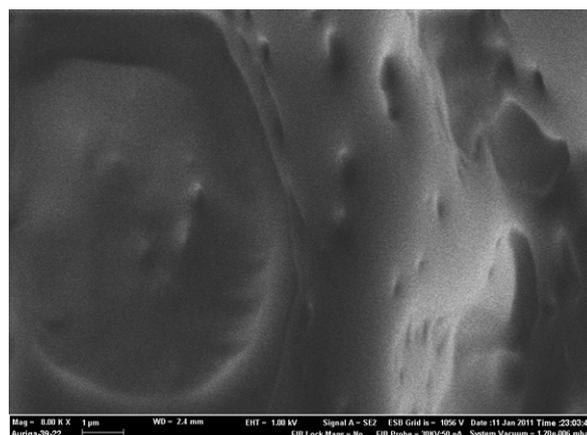
SEM images of (a) Pure PSAC, (b) PSAC paste electrode and (c) PSAC paste electrode surface after it was dipped in  $1 \times 10^{-4} \text{ M Hg}^{2+}$ .

### 3.7. Analytical Applications

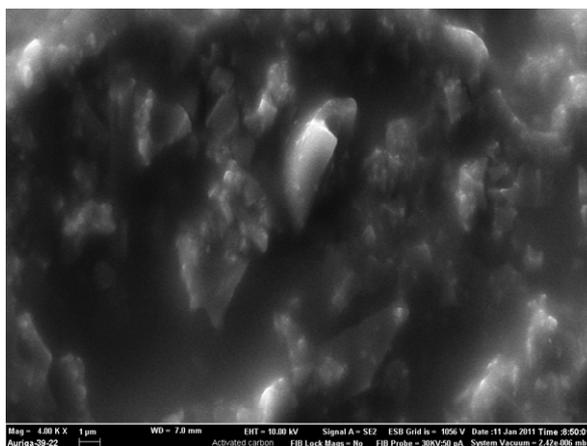
The proposed Hg-PSACPE was successfully applied for



(a)



(b)



(c)

**Figure 6.** SEM images of (a) Pure PSAC; (b) PSAC paste electrode and (c) PSAC paste electrode surface after it was dipped in  $1 \times 10^{-4} \text{ M Hg}^{2+}$ .

determination of Hg<sup>2+</sup> in groundwater samples. The results were compared with data obtained by ICP (**Table 3**). The water samples were collected from Gaza ground water well (Palestine). The results indicate that the concentration of Hg(II) in groundwater samples are in good

**Table 3. Tpotentiometric determination of mercury(II) in some groundwater samples using Hg(II) electrode and ICP.**

Sample <sup>a</sup>	Hg (II) (mg·L <sup>-1</sup> ) <sup>b</sup>			
	PSACPE	ICP	RSD%	Recovery%
(1)	1.363	2.038	3.36	95.4
(2)	1.089	1.443	1.10	98.5
(3)	1.151	1.499	4.81	93.4
(4)	1.108	1.404	2.27	96.8

<sup>a</sup>From some ground water wells in Gaza Strip. <sup>b</sup>Mean data for three replicate measurements.

agreement with those obtained by ICP method. Thus the proposed electrode was highly accurate, precise, and reproducible and can be employed for quantification of Hg(II) in real samples

#### 4. Conclusion

A new chemically modified palm shell activated carbon paste electrode (PSACPE) based on Kryptofix<sup>®</sup>5 as ionophore with simple preparation had fast response for detection of mercury ions. The electrode had wider working concentration range ( $1 \times 10^{-8}$  to  $1.0 \times 10^{-2}$  mol L<sup>-1</sup>, low detection limit ( $1 \times 10^{-7}$ ) and low response time (<10 s).

#### 5. Acknowledgements

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