



## All-solid-state Lead(II)–Selective Potentiometric Sensor Based on 9,10-dibromo-2-(propylthio)anthracene-1,4-dione

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**Abstract** – Ion-selective sensors are widely used in routine analysis of ionic species. In this research, a highly selective potentiometric sensor was developed, which is sensitive against  $Pb^{2+}$  ions compared to other common inorganic cations. The optimum composition of the developed sensor was determined to be as 3.0% ionophore, 64.0% bis(2-ethylhexyl)adipate (DEHA), 32.0% poly (vinyl chloride) (PVC), and 1.0% anion excluder (KTpCIPB). The  $Pb^{2+}$ -selective sensor had a Nernstian response of  $28.7 \pm 2.2$  mV/decade over a wide linear range of  $1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$  M and a low detection limit of  $2.77 \times 10^{-6}$  M. The newly developed sensor worked over a wide pH range (4.0–10.0), and the response time was short (7s). The sensor, which had good reproducibility, was applied to various water samples and determined  $Pb^{2+}$  ions with very high recoveries.

**Keywords** – Sensor, 1,4-anthraquinone, lead(II), ion-selective electrodes, potentiometry

## 1. Introduction

Lead (Pb) is a harmful environmental pollutant and a naturally occurring heavy metal with high toxicity against multiple animal tissues [1]. Its particular chemical and physical characteristics, including its malleability, plasticity, low melting point, and corrosion resistance, make it amenable to a broad range of uses in the industry, leading to its ubiquity in the environment. Although this ubiquitous xenobiotic metal can be absorbed through the skin, its primary source of entry to the human body is via the respiratory and digestive systems (by polluted water, food, or air). Its high affinity for sulfhydryl groups also makes this heavy metal particularly toxic to multiple enzyme systems, including heme biosynthesis, causing a decrease in blood hemoglobin levels. Pb exposure has been shown to induce various neurological, respiratory, urinary, and cardiovascular pathologies due to its disruptive influence on immune modulation, oxidant-antioxidant system (oxidative stress), DNA damage, and inflammatory mechanisms (immunotoxicity) [2–4]. Moreover, the nervous system is the most vulnerable to children's lead exposure. Exposure to very high concentrations of lead can seriously damage the brain and central nervous system (CNS), leading to coma, convulsions, and death. Children who survive severe lead poisoning may be left with permanent intellectual disability, cognitive impairment, and behavioral disorders [5, 6]. Besides, exposure to this non-essential metal can lead to renal effects, such as chronic nephropathy, Fanconi-like syndromes, and gout [7–9]. However, many people globally still live in housing with deteriorated lead-based paint. The international concern for lead poisoning is 10  $\mu$ g/dl in the blood [10, 11].

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Ion-selective electrodes (ISEs) have seen much interest in recent years with the introduction of new sensor materials, new sensing concepts, and better theoretical understanding and modeling of the potentiometric responses of ISEs [12, 13]. Potentiometric sensors have particular advantages such as small size, quick response, ease of use and manufacturing, low cost and resistance to color and turbid interferences, and high selectivity and sensitivity, to name just a few [14, 15]. Herein, we developed a new  $\text{Pb}^{2+}$ -selective sensor using an anthracene derivative molecule (Figure 1) as an ionophore and subsequently determined the working conditions of the sensor under laboratory conditions.

## 2. Experimental

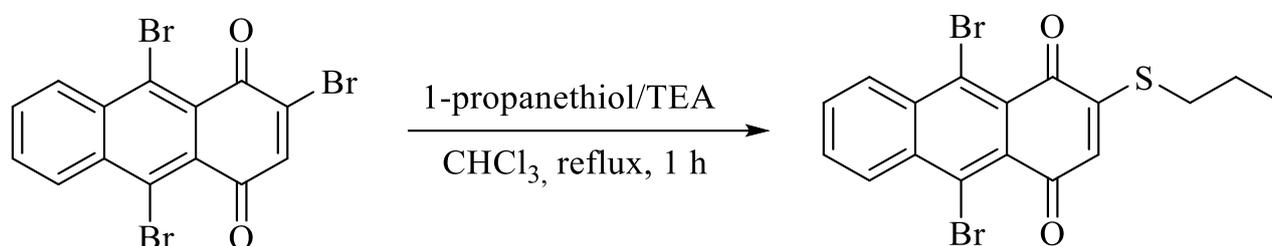
### 2.1. Chemicals and Apparatus

The solvents and chemicals used in preparing ISEs and studying all other performance parameters were of analytical grade. They were obtained from Sigma Aldrich and Merck. In ionophore characterization,  $^1\text{H-NMR}$ ,  $^{13}\text{C-NMR}$  (Bruker Avance DPX-400 spectrometer), and FTIR spectroscopy (Jasco FT/IR 430 instrument) were used. Melting points were determined with a Thomas-Hoover apparatus. Potential measurements were performed with a four-channel laboratory-made potentiometer device (Medisen Medical Ltd. Sti., Türkiye). The Ag/AgCl electrode (Thermo Scientific) was used as a reference electrode in all possible measurements.

### 2.2. Methods

#### 2.2.1. Synthesis of 9,10-dibromo-2-(propylthio)anthracene-1,4-dione

9,10-dibromo-2-(propylthio)anthracene-1,4-dione was synthesized according to a method previously reported in the literature [16] (Figure 1).



**Figure 1.** Synthesis scheme of 9,10-dibromo-2-(propylthio)anthracene-1,4-dione

*9,10-dibromo-2-(propylthio)anthracene-1,4-dione*: Yellow needle crystals (yield 80%), m.p. 147–149 °C (lit. m.p.: 149–150 °C) [16].  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.81–8.75 (m, 2H, ArH), 7.85–7.80 (m, 2H, ArH), 6.70 (s, 1H, H<sub>3</sub>), 2.86 (t,  $J=7.4$  Hz, 2H, S-CH<sub>2</sub>), 1.89–1.80 (m, 2H, CH<sub>2</sub>), 1.14 (t,  $J=7.4$  Hz, 3H, CH<sub>3</sub>),  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  181.5, 179.9, 155.9, 135.4, 134.8, 131.3, 131.1, 130.8, 130.6, 128.5 (128.51), 128.5 (128.46), 128.4, 126.9, 125.9, 32.8, 21.1, 13.7.

#### 2.2.2. Preparation of Sensors

In general, the preparation of sensors consists of two stages, as in our previous studies [17–19]. The first stage includes the preparation of the solid contact mixture that forms the substructure, and the second stage includes the preparation of the PVC membrane cocktail and coating it on the solid contact surface. Solid contact electrodes were prepared by dissolving a mixture containing 50.0% graphite, 35.0% epoxy, and 15.0% hardener in tetrahydrofuran (THF). After it became a homogeneous mixture, copper wires were dipped into this mixture several times, and their surfaces were coated. Then, polymer membrane sensors were prepared by dissolving the mixtures in the proportions given in Table 1 in THF. The mixtures were coated on the solid

contact electrode surfaces, and their potentiometric performance properties were investigated using  $\text{Pb}^{2+}$  ion solutions. The prepared sensor compositions and potentiometric performance properties prepared in this way are given in Table 1.

### 3. Results and Discussion

#### 3.1. Membrane Composition

In order to determine the most suitable composition of the proposed  $\text{Pb}^{2+}$ -selective potentiometric sensor, mixtures containing ionophore, plasticizer [DEHA, dibutyl phthalate (DBP) and bis(2-ethylhexyl)sebacate (BEHS)], PVC, and potassium tetrakis(*p*-chlorophenyl)borate ( $\text{KTpCIPB}$ ) in different proportions were prepared. The potentiometric responses of the  $\text{Pb}^{2+}$  selective sensors developed with the prepared mixtures were then investigated using  $\text{Pb}^{2+}$  solutions. The prepared sensor compositions and potentiometric performance data are given in Table 1.

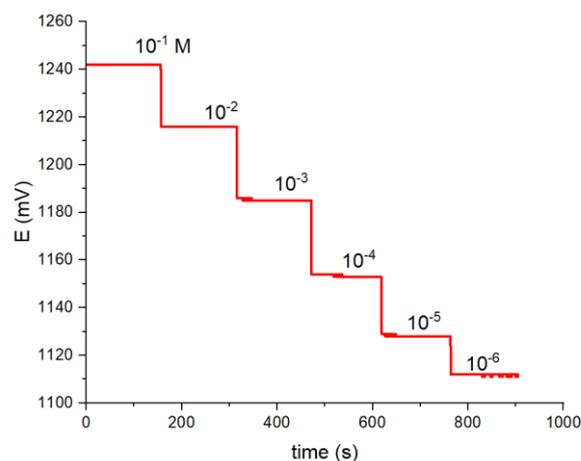
**Table 1.** The prepared potentiometric sensor compositions

No	Polymer membrane composition (w/w)						Potentiometric performance			
	PVC	Ionophore	DEHA	BEHS	DBP	KTpCIPB	Linear concentration range, M	Limit of detection, M	Slope ( $\text{mV dec}^{-1}$ )	$R^2$
1	32.0	3.0	64.0			1.0	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$2.77 \times 10^{-6}$	$28.7 \pm 2.2$	0.9990
2	32.0	3.0		64.0		1.0	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-2}$	$9.77 \times 10^{-6}$	$21.8 \pm 4.1$	0.9812
3	32.0	3.0			64.0	1.0	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$4.64 \times 10^{-6}$	$14.1 \pm 2.4$	0.9954

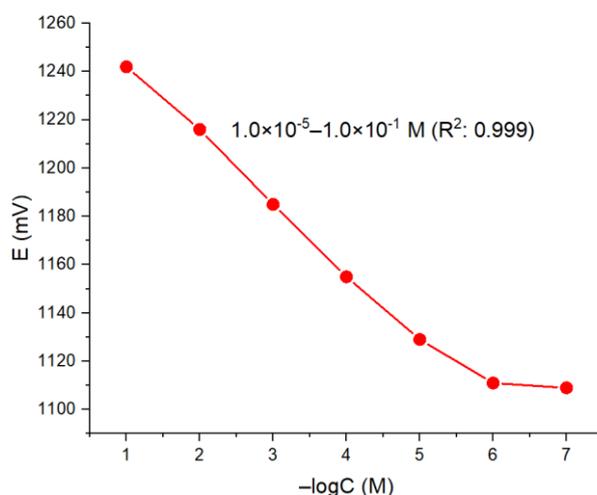
When the potentiometric performance values given in Table 2 were examined, it was determined that the most suitable sensor composition was 3.0% ionophore, 64.0% DEHA, 32.0% PVC, and 1.0%  $\text{KTpCIPB}$ . All potentiometric performance tests after this stage were performed using the sensor with this optimum composition.

#### 3.2. Potentiometric Response

The potentiometric response of the  $\text{Pb}^{2+}$  selective sensor, whose optimum composition was determined as detailed above, was then tested. This study examined the sensor's potentiometric behavior in standard  $\text{Pb}^{2+}$  solutions with different concentrations ranging from  $1.0 \times 10^{-6}$  to  $1.0 \times 10^{-1}$  M. Using the obtained data, the potential ( $E$ , mV)– $\log[\text{Pb}^{2+}]$  graph was created and the potentiometric performance properties of the  $\text{Pb}^{2+}$  selective sensor, such as linear working range, slope, and  $R^2$  values, were determined. The potentiometric response of the sensor is given in Figure 2, and the sensor's calibration curve and  $R^2$  values (0.999) are presented in Figure 3. According to Figures 2 and 3, the proposed sensor was found to work linearly in the concentration range of  $1.0 \times 10^{-1}$ – $1.0 \times 10^{-5}$  M ( $R^2=0.999$ ) and exhibit a Nernstian behavior of  $28.7 \pm 2.2$  mV/decade. The detection limit of the sensor was calculated by substituting the potential obtained from the intersection point of two linear regions of the calibration curve in Figure 3 into the linear equation according to the rules reported by IUPAC [20]. Thus, the detection limit of the sensor was calculated as  $2.77 \times 10^{-6}$  M.



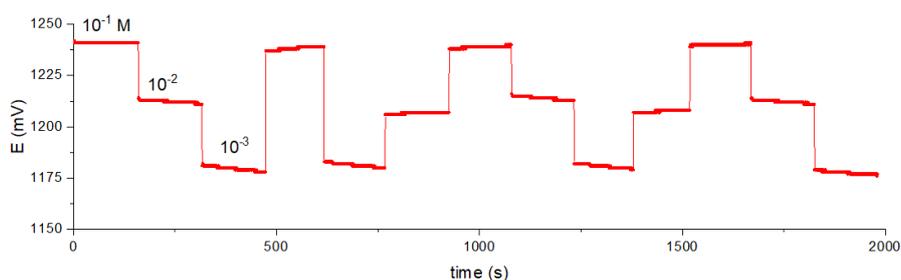
**Figure 2.** Potentiometric response of the newly developed sensor



**Figure 3.** Calibration curve of the newly developed sensor

### 3.3. Repeatability

The repeatability of the potentiometric response of the  $\text{Pb}^{2+}$ -selective sensor was tested using  $1.0 \times 10^{-1}$ ,  $1.0 \times 10^{-2}$ , and  $1.0 \times 10^{-3}$  M  $\text{Pb}^{2+}$  ion solutions. The  $\text{Pb}^{2+}$ -selective sensor was kept in standard  $\text{Pb}^{2+}$  solutions until it reached the equilibrium potential, and the observed potential data at the moment the equilibrium potential was reached were recorded. The  $\text{Pb}^{2+}$  selective sensor is given in Figure 4. The repeatability data are shown in Table 2. The standard deviation ( $\pm$ SD) values calculated for the three concentrations are  $\pm 0.82$ ,  $\pm 0.94$  and  $\pm 0.47$ , respectively. According to Figure 4 and Table 2, the developed sensor exhibited a stable and reproducible behavior.



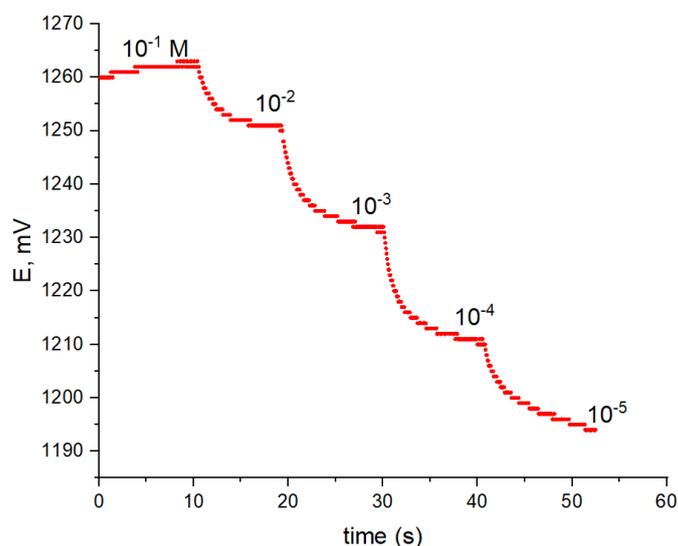
**Figure 4.** Repeatability of the proposed sensor

**Table 2.** Repeatability data of lead(II)-selective sensor

Concentration, M	Potentials			
	I	II	III	Average ( $\pm$ SD)
$1.0 \times 10^{-1}$	1241.0	1239.0	1240.0	1240.0 ( $\pm 0.82$ )
$1.0 \times 10^{-2}$	1211.0	1213.0	1211.0	1211.7 ( $\pm 0.94$ )
$1.0 \times 10^{-3}$	1181.0	1181.0	1180.0	1180.7 ( $\pm 0.47$ )

### 3.4. Response Time

In this study, to determine the response time of the  $\text{Pb}^{2+}$ -selective sensor,  $1.0 \times 10^{-1}$ ,  $1.0 \times 10^{-2}$ ,  $1.0 \times 10^{-3}$  and  $1.0 \times 10^{-4}$  M  $\text{Pb}^{2+}$  solutions were used. The  $\text{Pb}^{2+}$ -selective sensor was directly immersed in these solutions. The equilibrium time was determined during the transition from one solution to another [20]. According to Figure 5, the proposed  $\text{Pb}^{2+}$ -selective sensor was found to have a response time of 7 seconds.

**Figure 5.** Dynamic response of the newly developed sensor

### 3.5. Selectivity Coefficients

Selectivity coefficient values for potentiometric ion-selective sensors show the numerical expression of anionic or cationic species that interfere with the primary ion. In this study, the selectivity of the newly developed  $\text{Pb}^{2+}$ -selective sensor was determined by a separate solution method based on separate measurements of each ion [21]. In order to assess the selectivity coefficient values of the sensor according to this method, the potential values of ions in Table 3 at  $10^{-2}$  M concentration were calculated by substituting them in the below equation:

$$\log K_{A,B}^{\text{pot}} = \frac{(E_B - E_A)Z_A F}{RT \ln 10} + \left(1 - \frac{Z_A}{Z_B}\right) \log a_A$$

where,  $K_{A,B}^{\text{pot}}$  = selectivity coefficient,  $a_A$  = activity of lead ion,  $a_B$  = activity of interfering ion,  $Z_A$  = charge of lead ion,  $Z_B$  = charge of interfering ion;  $R$ ,  $T$ , and  $F$  have the usual meanings.

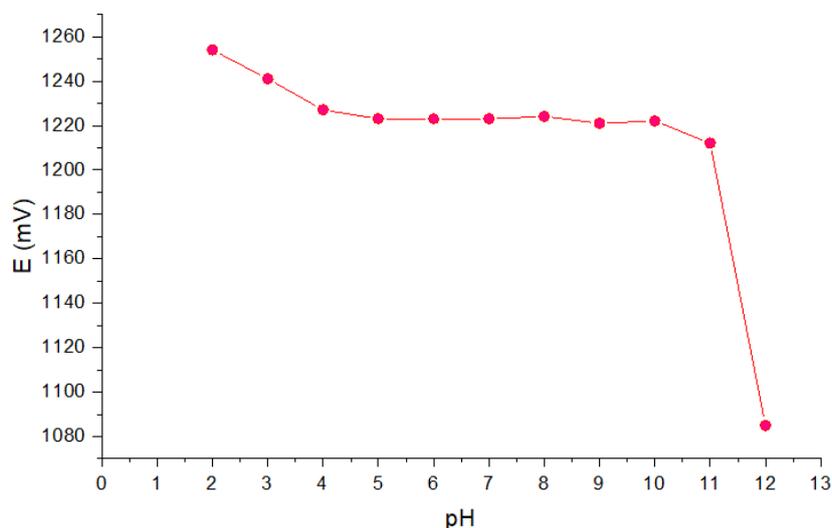
According to Table 3, this newly proposed sensor exhibited a highly selective behavior towards  $\text{Pb}^{2+}$  ions.

**Table 3.** Selectivity coefficients of Pb<sup>2+</sup>-selective sensor

Interfering ions	$\log K_{Pb(II),M^{n+}}^{pot}$	$K_{Pb(II),M^{n+}}^{pot}$	Interfering ions	$\log K_{Pb(II),M^{n+}}^{pot}$	$K_{Pb(II),M^{n+}}^{pot}$
Ba <sup>2+</sup>	-1.62	2.40×10 <sup>-2</sup>	Co <sup>2+</sup>	-3.11	7.76×10 <sup>-4</sup>
Li <sup>+</sup>	-1.76	1.74×10 <sup>-2</sup>	Cr <sup>3+</sup>	-3.15	7.08×10 <sup>-4</sup>
Na <sup>+</sup>	-2.30	5.01×10 <sup>-3</sup>	Ni <sup>2+</sup>	-3.28	5.25×10 <sup>-4</sup>
Al <sup>3+</sup>	-2.33	4.68×10 <sup>-3</sup>	Ca <sup>2+</sup>	-3.31	4.90×10 <sup>-4</sup>
Cu <sup>2+</sup>	-2.37	4.27×10 <sup>-3</sup>	Zn <sup>2+</sup>	-3.69	2.04×10 <sup>-4</sup>
Sr <sup>2+</sup>	-2.57	2.69×10 <sup>-3</sup>	Mn <sup>2+</sup>	-4.13	7.41×10 <sup>-5</sup>
Mg <sup>2+</sup>	-3.04	9.12×10 <sup>-4</sup>			

### 3.6. pH Working Range

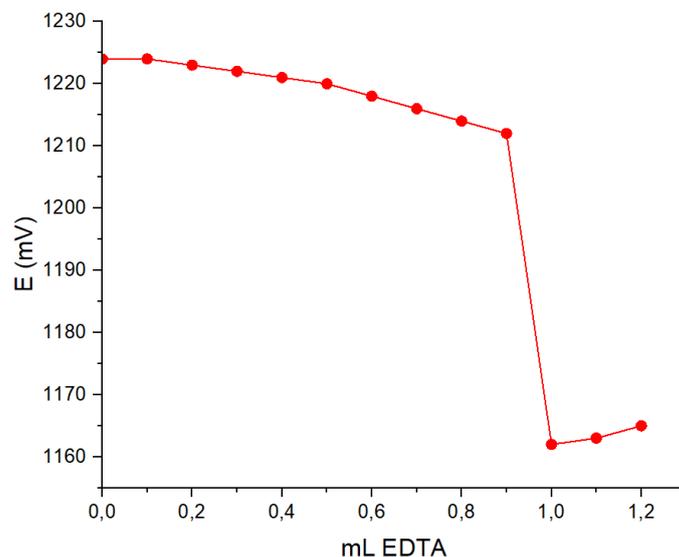
The pH working range is an essential potentiometric performance property that shows the usability of a developed ion-selective sensor in different matrix environments. In this study, to determine the pH working range of the Pb<sup>2+</sup>-selective sensor, solutions containing 1.0×10<sup>-2</sup> M Pb<sup>2+</sup> and having different pH values (pH=2.0–12.0) were prepared. NaOH solutions were used for basic regions, and HCl solutions were used for acidic regions to adjust the pH values. The pH working range of the proposed sensor is given in Figure 6. When the graph in Figure 6 is examined, it was observed that the potentiometric behavior of the Pb<sup>2+</sup>-selective sensor is not affected by the pH changes of the solutions in the pH range of 4.0–10.0. It is thought that at pH values below pH=4.0, there are increases in the observed potential values due to the protonation of the sensor membrane by hydronium ions in the solution, and at pH values above pH=10.0, there are decreases in the observed potential values due to the formation of Pb(OH)<sub>2</sub> by hydroxyl ions and Pb<sup>2+</sup> ions in the solution.

**Figure 6.** pH working range of the newly developed sensor

### 3.7. Analytical Applications

Potentiometry is also a method used in titration processes [22]. In order to determine the usability of the Pb<sup>2+</sup>-selective sensor as an indicator electrode in potentiometric titrations, the potentiometric titration of Pb<sup>2+</sup> ions with ethylenediaminetetraacetic acid (EDTA) was investigated. For this analysis, 10 mL of 1.0×10<sup>-3</sup> M Pb<sup>2+</sup> ion solution was taken, and 0.1 mL of 1.0×10<sup>-2</sup> M EDTA solution was continuously added. The potential values

observed after each EDTA addition were recorded. According to Figure 7, a very sharp peak was observed at the equivalence point. This shows that  $\text{Pb}^{2+}$  ions and EDTA were complexed in a 1:1 ratio. As a result, the proposed  $\text{Pb}^{2+}$ -selective sensor was shown to be successfully used in the potentiometric titration of  $\text{Pb}^{2+}$  ions with EDTA.



**Figure 7.** Potentiometric titration of  $\text{Pb}^{2+}$  ions with EDTA

The real sample analysis application of the  $\text{Pb}^{2+}$ -selective sensor was carried out using three different water samples. For this study,  $\text{Pb}^{2+}$  ion was added to the water samples in the volumes given in Table 4. The potential measurement of the water samples to which the standard addition was made was carried out, the read potential values were written in the linear equation created with the help of the calibration graph, and the  $\text{Pb}^{2+}$  ion concentrations of the samples were calculated. According to Table 4, it can be said that the proposed sensor can determine the  $\text{Pb}^{2+}$  ion in the water samples with very high recoveries.

**Table 4.** Real sample analyses performed with the  $\text{Pb}^{2+}$ -selective sensor

Spiked Samples	Pb <sup>2+</sup> quantity, (M)		% Recovery
	Added Pb <sup>2+</sup>	Found ( $\pm$ SD) with sensor*	
Bottled water	$1.0 \times 10^{-4}$	$9.33 (\pm 0.36) \times 10^{-5}$	93.3
Snow water	$1.0 \times 10^{-3}$	$9.55 (\pm 0.23) \times 10^{-4}$	95.5
Purified water	$1.0 \times 10^{-4}$	$9.66 (\pm 0.11) \times 10^{-5}$	96.6

\*Average value ( $n = 3$ )

### 3.8. Comparison Study

In this study, the new  $\text{Pb}^{2+}$ -selective potentiometric sensor we proposed was compared with some of its counterparts in the literature in terms of linear concentration range, limit of detection, pH working range, response time, and slope (mV/decade). As seen in Table 4, the proposed new sensor had a similar linear concentration range and response time to its counterparts. It had a lower detection limit and wider pH working range than the sensors in Table 5.

**Table 5.** The newly developed sensor is compared with other potentiometric ISE reported in the literature

Ionophore	Concentration range (M)	Limit of detection (M)	pH working range	Response time (s)	Slope (mV/decade)	Reference
Acridono-crown ether	$1.0 \times 10^{-4}$ – $1.0 \times 10^{-2}$	$7.9 \times 10^{-6}$	4.0–7.0	5	26.9	[23]
(E)-2-(2-((2-((4-methoxyphenyl)sulfonyl)hydrazineylidene)methyl)phenoxy) acetic acid	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$2.89 \times 10^{-6}$	3.0–9.0	5	$27.7 \pm 1.3$	[24]
PbS nanoparticles	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-2}$	Not reported	3.0–7.0	10	Not reported	[25]
(E)-2-((1H-pyrrol-2-yl)methylene)hydrazinecarbothioamide	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$3.96 \times 10^{-6}$	5.0–9.0	5	$29.5 \pm 1.6$	[26]
Bentonite clay	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$9.0 \times 10^{-6}$	Not reported	~5	31.0	[27]
9,10-dibromo-2-(propylthio)anthracene-1,4-dione	$1.0 \times 10^{-5}$ – $1.0 \times 10^{-1}$	$2.77 \times 10^{-6}$	4.0–10.0	7	$28.7 \pm 2.2$	This work

## 4. Conclusion

This study performed detailed potentiometric tests of the  $\text{Pb}^{2+}$ -selective sensor prepared with a 1,4-anthraquinone derivative molecule. As a result of the measurements, it was determined that the  $\text{Pb}^{2+}$ -selective sensor had a Nernstian response and a low detection limit in a wide concentration range. The proposed sensor had essential features such as good selectivity, reproducibility, easy preparation, short response time, and a wide pH range. Combined, it can be stated that the newly produced  $\text{Pb}^{2+}$ -selective potentiometric sensors can be used as indicator electrodes in  $\text{Pb}^{2+}$  ion analyses and can determine  $\text{Pb}^{2+}$  ion contents in various real samples with high recoveries. 1,4-anthraquinone derivative molecules can be used as sensor materials for selective, rapid, and low-cost determination of various ionic species. The results obtained will increase the interest of researchers working in this field in the synthesized molecules compared to commercial ionophores. The findings obtained in the study will be guiding for researchers working in this field. Our work on the synthesis and sensor properties of new ionophore molecules continues.

## Author Contributions

All the authors equally contributed to this work.

## Conflict of Interest

The authors declare no conflict of interest.

## Ethical Review and Approval

No approval from the Board of Ethics is required.

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