

## Investigations of molybdenum content in beef liver

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**Abstract:** A new solid-state molybdenum-selective electrode has been developed. A solid-state electrode was prepared using  $\text{MoS}_2$  as ion exchanger. The slope of the linear portion ( $1 \times 10^{-1}$ - $1 \times 10^{-5}$  M) was about  $45 \pm 2$  mV/10-fold concentration changes in molybdenum. It was found that pH change between 1 and 12 had no effect on the potential of the electrode. The life of the electrode was more than 2 years when used at least 4-5 times a day and the response time was about 30 seconds. This electrode was used for the determination of molybdenum ion in calf liver using the standard addition method. Validation values of the electrode were measured. The same sample was analyzed with both the electrode and DP polarography and high consistency was obtained.

**Keywords:** Molybdenum, Solid-state electrode, DP polarography, Beef liver.

### Introduction

Molybdenum (Mo) is an essential trace element for cell growth. High concentrations of this element have toxic effects on human health [1]. The importance of molybdenum for biological systems is that it plays an active role in the action of some enzymes such as xanthine oxidase, aldehyde oxidase and sulfite oxidase involved in carbohydrate metabolism [2].

Many methods were used in the analysis of molybdenum, which was found in low amounts in the selected samples. Many analytical methods such as stripping voltammetry in water and foodstuff samples [3], spectrophotometry in plant foodstuff samples [4], inductively coupled plasma–optical emission spectrometry (ICP-OES) in gasoline samples [5], electrothermal atomic absorption spectrometry (ETAAS) in milks and environmental samples [6-7], graphite furnace atomic absorption spectrometry (GF-AAS) in waters and solids [8-9], inductively coupled plasma mass spectrometry (ICP-MS) in chicken, duck, goose, and pigeon eggs [10], inductively coupled plasma-atomic emission spectrometry (ICP-AES) in environmental water samples [11], high performance liquid chromatography inductively coupled plasma mass spectrometry (HPLC-ICP-MS) in ground waters and solids [12] and flame atomic absorption spectrometry (FAAS) in foods/beverages and corn samples [13-14] in the last decade have been developed for the preconcentration and determination of the Mo in the various samples.

The aim of this study was to prepare an electrode that is easy to prepare, easy to use and has a long life for molybdenum ion. Determination of molybdenum in other methods is costly and requires pre-concentration. The most important advantage of this electrode is that it is not sensitive to other ions. Molybdenum ion in calf liver was analyzed both with the new electrode and with DP polarography. The results were compared.

### Materials and Method

Potential measurements were made with a Jenway 3040 ion meter. All potential values reported, are taken with respect to an 924036 Double Junction Ag/AgCl saturated reference electrode which was hold in a Luggin-Haber capillary. Reagent grade chemicals were used without further purification. All solutions were prepared with triple distilled water.

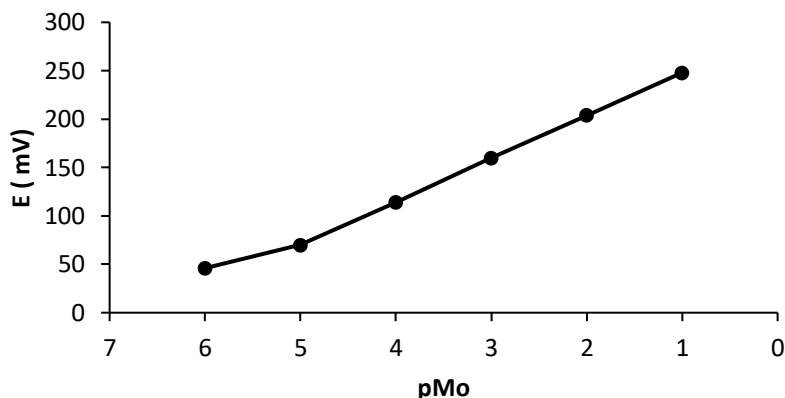
Precipitated solid salts such as  $\text{MoS}_2$ ,  $\text{Ag}_2\text{S}$  and  $\text{Cu}_2\text{S}$  were dried and then mixed in varying compositions. A certain amount of the salt or salt mixture (10-15 mg) was taken and pellets were made by holding first under a pressure of  $5000 \text{ kgcm}^{-2}$  for 5min and then under a pressure of  $8000 \text{ kgcm}^{-2}$  for 10 min using the hydraulic press of IR

instrument. The pellets of 7mmdiameter and 0.1-0.3mm thickness were sealed with epoxy resin (0.7 g epoxy and 0.9 g hardener).

A special dissolution procedure was applied to the DNA liver sample to be analyzed. 0.4 g of sample was weighed and placed in a polytetrafluoroethylene container. Then, 1 mL of concentrated nitric acid and 2 mL of hydrogen peroxide were added. The containers were closed and placed in the device. The dissolution process was carried out twice at 80 bar and 240 degrees. The temperature of the solution reached room temperature. The total volume was then completed to 25 mL with ultrapure water.

**Result**

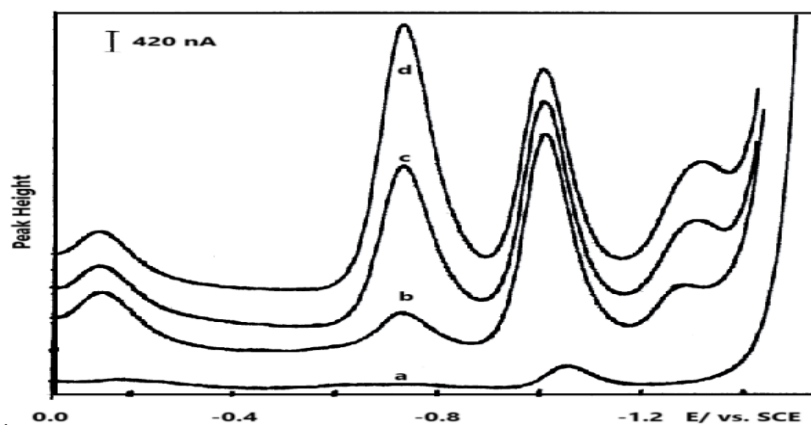
The sensitivity of the electrode to molybdenum ion concentration was investigated. For this purpose, a dilute solution of the appropriate molybdenum solution (prepared from  $\text{Na}_2\text{MoO}_4$ ) was added to the cell and the corresponding potential value was recorded. All measurements were made in 20 mL of 0.1 M  $\text{NaNO}_3$  solution for a constant ionic strength. The molybdenum concentration in the cell was increased 10 times and the potential values were measured. The calibration graph is given in Figure 1.



**Figure 1: Calibration chart of molybdenum electrode**

For the determination of molybdenum, first the potential of 20 ml of 0.1M  $\text{NaNO}_3$  is measured using molybdenum ion selective electrode. Then, appropriate volumes of liversamples were added and once more potential was measured.

The polarogram of this liver sample was taken with differential pulse polarography in pH=4 0.1 M HAc buffer. It was understood that the peak at approximately -0.8 V was the molybdenum peak. It was analyzed by adding standard molybdenum solution and is given in Figure 2.



**Figure 2: Analysis of molybdenum in DP polarography**

Good agreement was obtained between two different methods. Molybdenum sample was also determined using the newly established electrode. The results are summarized in Table 1 together with the result obtained using DP Polarography.

**Table 1: Determination of molybdenum ion in liver sample.**

| Sample | DP Polarography<br>µg /g | Molybdenum electrode<br>µg /g | $t_{\text{experimental}}$<br>$t_{\text{critical}}=2.015$ |
|--------|--------------------------|-------------------------------|--|
| liver  | 43 ± 2                   | 42 ± 2                        | 0.819  |

95% CI and N=5.

### Conclusion

Although there are many investigations about solid-state electrodes prepared from solid salts, none of these were subject for molybdenum ion determination. In this work a new molybdenum ion selective electrode using solid salts of MoS<sub>2</sub>, Ag<sub>2</sub>S and Cu<sub>2</sub>S has been prepared. The preparation of the electrode is simple, it displays very good performance in regard to reproducibility, sensitivity and lifetime. The slope of the linear portion ( $1 \times 10^{-1}$ - $1 \times 10^{-5}$  M) was  $45 \pm 2$  mV/decade change in molybdenum ion. The liver sample was analyzed with both electrode and DP polarography, and the results were found to be consistent with each other.

### Source of Funding

None.

### Conflict of Interest

No conflict of interest to be disclosed.

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