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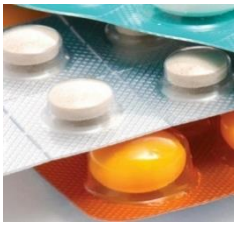
XRF

# Rapid and Reliable Zinc Determination in Liquid Using a Compact Benchtop EDXRF



Application Note

Pharmaceutical XRF24

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**Abstract:** We introduce EDXRF analysis as a rapid and reliable method for Zinc determination in liquid solution. We demonstrated Zn determination at ppm levels using the calibration curve method with commercial Zinc liquid standard samples. Our calibration curve had a good linearity ($R^2$ > 0.999) and we could accurately determine 22 ppm of Zn in 1 minute. The results demonstrated that EDXRF is an effective tool for rapid and reliable laboratory analysis.

**Keywords:** Pharmaceutical, Zinc determination, EDXRF

## Introduction

Zinc (Zn) is included in many liquid medicines due to its essential role in immune function, wound healing, enzyme activity, and cellular repair. Zinc concentration in liquid medicines must be carefully balanced: Too little may be ineffective, while too much can cause nausea, interfere with copper absorption, or lead to toxicity.[1]

Among analytical instruments to determine Zn content in liquid medicine, energy-dispersive X-ray fluorescence (EDXRF) is an effective method for laboratory usage because it requires minimal sample preparation and provides a rapid and non-destructive approach. Additionally, it can achieve ppm-level detection under appropriate conditions.

In this application note, we demonstrate Zn determination in ppm level using a HORIBA compact benchtop EDXRF system called MESA-50.
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## HORIBA MESA-50 X-ray Fluorescence Analyzer

The MESA-50 (Figure 1a) is a compact benchtop EDXRF analyzer. Its A4-paper footprint and built-in rechargeable battery enables users to conduct measurements without dependence on external power sources.

The internal optic design is represented in Figure 1c. It irradiates primary X-rays from the bottom, and detects fluorescent X-rays at a diagonal angle from the sample. In the case of powder or liquid samples, the sample can be performed by placing it in a cup for analysis.

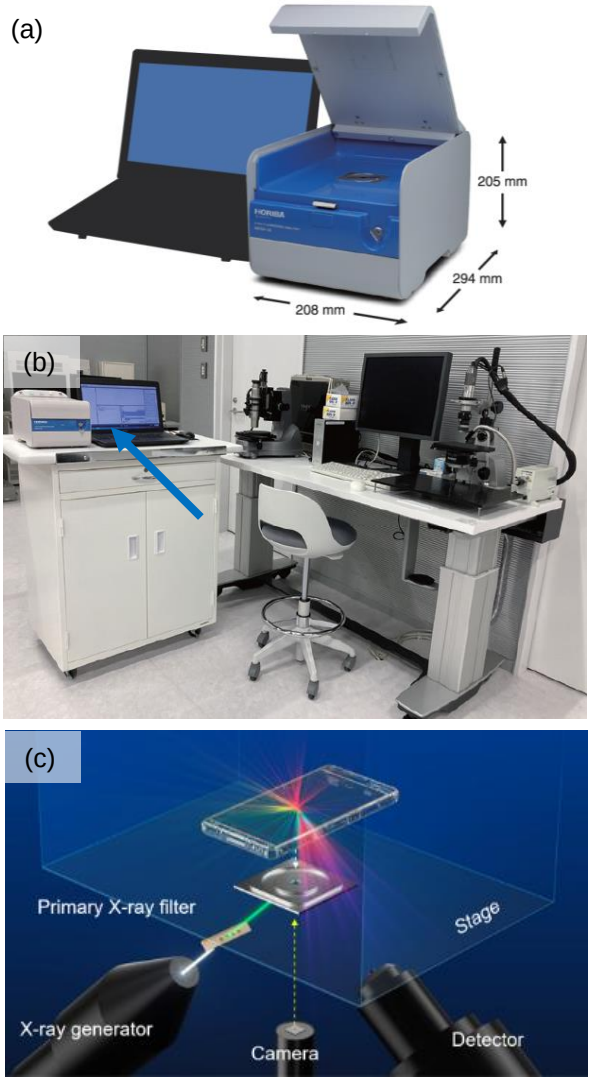



Figure 1. HORIBA MESA-50 (a) appearance (b) installation example (c) a schematic diagram of the optics inside.

## STEP 1. Calibration curve creation & linearity check

### Sample preparation

The first step consists of calibration curve creation and the linearity check. In this application note, we prepared five samples with different Zn contents (Table 1). We made the samples by diluting a commercial single-element standard for ICP-OES (Zn 1000 mg/L, HORIBA Jobin Yvon) with ultra-pure water. Each diluted solution was placed in a cup.

Table 1. The sample information for calibration curve plot.

| | a | b | c | d | e | f | g |
|---|---|---|---|---|---|---|---|
| Standard sample | 0 (blank) | 5 ppm | 10 ppm | 20 ppm | 25 ppm | 40 ppm | 50 ppm |

### Measurement

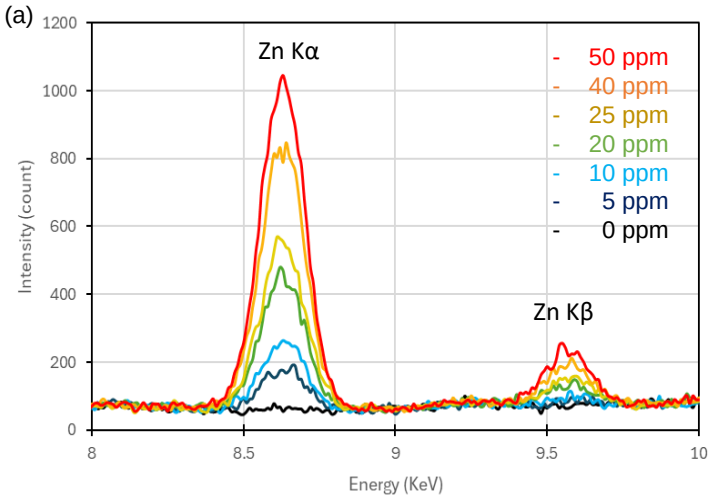
We mounted each sample in a cup onto the measurement position of the MESA-50 chamber (Figure 2), and we carried out spectrum analysis. Though the measurement condition is described in Table 2, the keynote here is that we used a primary X-ray filter to enhance the signal to noise ratio of Zn K lines by cutting the background derived from Compton scattering. .

Figure 3a shows a layered spectrum of the 7 samples' results, and we could see a trend that higher Zn concentration of Zn had higher Zn peak intensity. Using the result, we made a calibration curve of Zn concentration vs. Zn-Kα peak intensity (Figure 3b), and the result showed a good regression coefficient (0.999 > $R^2$). This is a good indication that EDXRF is a suitable method for this kind of application.


Figure 2. Sample setting inside the MESA-50 chamber.

Table 2. Measurement condition used in this application note.

| | Condition |
|---|---|
| Spot size | 7 mm |
| Voltage | 50 kV |
| Current | 100 µA |
| Measurement time | 60 s |
| Processing time | Process 3 |
| Filter | Mid |



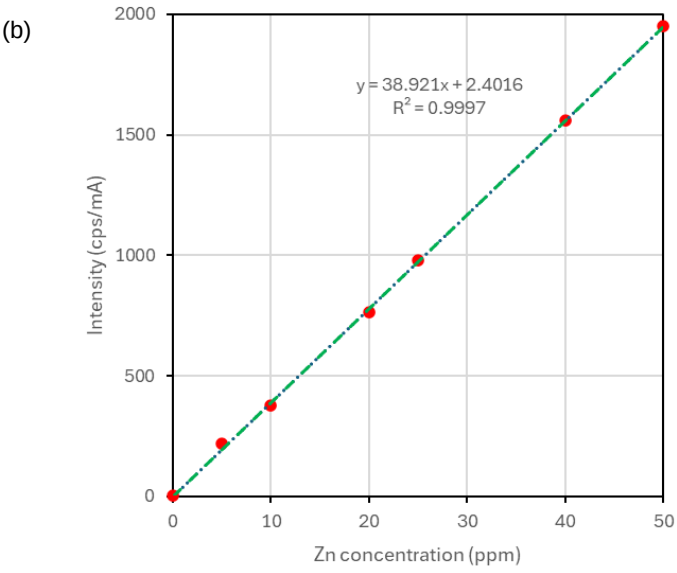




Figure 3. (a) Layered XRF spectrum of the 7 known samples (b) Calibration curve plots: Zn concentration (ppm) vs Zn-Kα ROI (cps/mA).

## STEP 2. Accuracy check of Zn determination

As the second step, we prepared another sample whose Zn concentration was known to be 22 ppm. We used it as an unknown sample for determination accuracy evaluation.

The measurement conditions were identical to those shown in Table 2. We carried out spectrum analysis 5 times ($n$ = 5) at the same position of the sample. Table 3 shows our Zn concentration results calculated using the calibration curve we made in the previous step. The calculated result showed 22.0 ppm on the average. Thus, this experiment demonstrated excellent accuracy in Zn determination at ppm levels through this experiment.

Table 3: Zn concentration results ($n$ = 5) calculated using the calibration curve we made in the previous page.

| | **Zn result (ppm)**<br>Expected value: 22 ppm |
|---|---|
| 1 | 22.0 |
| 2 | 22.3 |
| 3 | 22.0 |
| 4 | 22.0 |
| 5 | 21.8 |
| **Average** | **22.0** |
| SD | 0.16 |

## Conclusion

Based on our experiment, we demonstrated the feasibility of the HORIBA compact benchtop EDXRF analyzer, MESA-50, as a rapid and reliable laboratory analysis tool for Zn in liquid solutions at ppm levels


## Acknowledgement

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## Reference


[1] *The Pharmaceutical Journal, PJ*, March 2006; DOI: 10.1211/PJ.2023.1.181673


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