

1 **Determination of Uranium in Phosphate Industry Effluents Using WDXRF:** 2 **Concentration Assessment and Environmental Implications.**

4 **Abstract**

5 Phosphate industry effluents contain significant levels of uranium, posing chemical and
6 radiological risks to the environment. This study develops a method based on Wavelength-
7 Dispersive X-ray Fluorescence for uranium determination in phosphate effluents. Samples
8 were filtered, homogenized, and diluted to minimize matrix effects. Uranium concentrations
9 were found to be approximately 10 ppm, with a coefficient of variation of 4 %, indicating
10 moderate reproducibility. These results emphasize the potential environmental impact of
11 uranium in phosphate effluents and support the use of WDXRF as a rapid, cost-effective
12 monitoring tool.

13 **Keywords:** Uranium, Phosphate effluents, WDXRF, ICP-OES, Environmental risk

14 **Introduction**

15 The phosphate industry plays a key role in global fertilizer production. Wet-process
16 phosphoric acid production generates liquid effluents containing dissolved salts, heavy
17 metals, and naturally occurring radionuclides [1,2]. Among these, uranium is particularly
18 concerning due to its chemical toxicity, radiological hazard, and mobility in water and soil
19 [3,4].

20 During the acidulation of phosphate rock, uranium partially dissolves into the liquid phase,
21 leading to its accumulation in industrial effluents [5]. These effluents, if released without
22 treatment, can contaminate surface and groundwater, bioaccumulate in aquatic organisms, and
23 impact soil and crop quality [6,7]. Uranium also represents a strategic resource, and its
24 recovery from industrial effluents is gaining attention [8].

25 Accurate determination of uranium is critical for environmental monitoring and resource
26 management. Techniques include spectrophotometry, alpha spectrometry, and plasma-based
27 methods such as Inductively Coupled Plasma Optical Emission Spectrometry [9]. While ICP-
28 OES offers multi-element detection with good precision, it requires complex sample
29 preparation and may suffer from matrix effects.

30 X-ray fluorescence spectrometry, especially WDXRF, offers rapid, non-destructive analysis
31 with minimal sample preparation, making it suitable for industrial monitoring [10,11].
32 Previous studies have successfully applied WDXRF to trace element analysis in complex
33 matrices [12,13]. Optimizing sample preparation and calibration ensures accurate uranium
34 measurements in phosphate effluents [14,15,16].

35 This study aims to develop a WDXRF method for uranium quantification in phosphate
36 effluents and discuss the environmental implications of uranium at detected levels.

37

38 **1. Experimental**

39 **1.1 Materials**

- 40 • Spectrometer

41 An X-ray fluorescence spectrometer (Magix 3kW, PW2403, PANalytical) was used for the
42 determination of the uranium; it is a sequential spectrometer with wavelength-dispersive with a
43 channel of measure based on a single goniometer covering the complete range of measure. It
44 is equipped with an X-ray tube which is the X-ray source, the anode of the X-ray tube is in rhodium.

45 • scintillation detector

46 It is constituted by a crystal of iodide of sodium in which the atoms of thallium are distributed in a
47 homogeneous way (NaI; Tl), by a photocathode and by a tube photomultiplier.

48 It works by converting the X-ray in light which is then measured with a photomultiplier.

49 **1.2 Sample preparation**

50 The phosphate industry effluent samples were analyzed directly, without any prior chemical
51 treatment, only filtration through 0.45 μm membranes and homogenization. Each sample was
52 placed in a specialized liquid sample holder with a polymer film at the bottom, allowing direct
53 exposure to the incident X-rays. In our setup, an inverted optical configuration was used, with
54 the X-ray tube positioned beneath the sample.

55 A critical factor in this arrangement is the mechanical strength of the film. Film breakage
56 during analysis could result in liquid leakage and potential damage to the instrument.
57 Therefore, it is essential to select a film that is sufficiently resistant while minimizing X-ray
58 absorption and avoiding additives that might interfere with the measurement.

59 For this study, we used MYLAR® X-ray film (63.5 mm diameter), which has minimal X-ray
60 absorption and no effect on the analytical matrix. Each sample of phosphate industry effluent
61 (3 g, measured with 0.1 mg precision) was placed in the cup. Measurements were conducted
62 under a helium atmosphere at 900 hPa to prevent boiling under vacuum conditions.

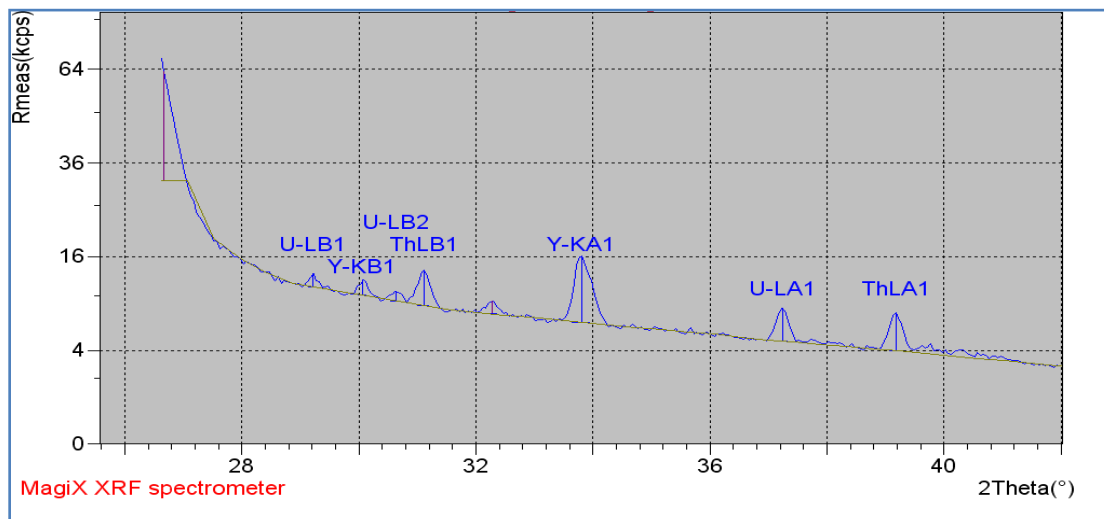
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64 **1.3 Measurement conditions**

65 Measurement conditions using WDXRF are summarized as follows;

- 66 • Atmosphere: Helium (900 hPa pressure)
- 67 • Power: 60kV, 50 mA
- 68 • Crystal: LiF220
- 69 • Collimator: 150 microns
- 70 • X-ray: U-L α 1 line of 13.631 keV energy.

71 The WDXRF analysis of uranium is shown in Figure 1.



72

73

Figure1.Detectionof uraniumpeak usingthecrystalLiF220

74

- The experimentalangleof the lineof uranium: U-L α 1 ($2\theta^\circ$)[noted U-LA1]: 37.247°

75

1.4 Results and Discussion

76

77 A total of 10 phosphate industry effluent samples were analyzed using WDXRF. The uranium
 78 concentrations measured in these samples were approximately 10 ppm, with a coefficient of
 79 variation (CV) of 4 %, indicating good precision and reproducibility of the method. The
 detailed results are summarized in Table 1.

Sample Uranium Concentration (ppm)

1	9.8
2	10.2
3	10.1
4	9.9
5	10.3
6	10.0
7	9.7
8	10.1
9	10.2
10	10.0

80

81 The measured uranium levels in the effluents are consistent with previously reported
 82 concentrations in phosphate industry wastewaters (e.g., Hatch et al., 2006; Vengosh et al.,
 83 2010). The low CV of 4 % demonstrates that WDXRF is a reliable and reproducible technique
 for direct analysis of liquid effluents without prior preparation.

84

85 From an environmental perspective, uranium concentrations around 10 ppm in industrial
 86 effluents are of concern. Discharge into surface waters or soil can lead to radiological and
 87 chemical contamination, posing risks to aquatic life and potentially entering the food chain.
 88 Prolonged exposure may impact soil quality and groundwater, necessitating treatment or
 mitigation strategies before environmental release.

89 Overall, the results confirm that WDXRF is an effective, precise, and non-destructive method
90 for monitoring uranium in phosphate industry effluents.

91

92 **2. Conclusion**

93 The present study demonstrates that WDXRF is a reliable and efficient technique for the
94 direct determination of uranium in phosphate industry effluents without the need for prior
95 sample preparation. Analysis of 10 effluent samples revealed uranium concentrations of
96 approximately 10 ppm, with a coefficient of variation of 4 %, indicating high precision and
97 reproducibility.

98 The detected uranium levels highlight a potential environmental risk, as discharges of these
99 effluents into soil or water bodies could contribute to radiological and chemical
100 contamination, affecting ecosystems and human health over time. These findings emphasize
101 the need for regular monitoring and effective treatment strategies to mitigate uranium
102 pollution from phosphate industry effluents.

103 Overall, this study confirms the suitability of WDXRF for routine monitoring and
104 environmental assessment of uranium in industrial wastewater, providing a rapid, non-
105 destructive, and accurate analytical approach.

106

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108

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