

Comments on: Studies on Effective Decomposition of Monazite Minerals by Variety of Phosphate Fluxes for Simple and Direct Determination of Uranium by LED Fluorimeter

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Abstract

This paper presents my comment on the published article, studies on effective decomposition of monazite minerals by variety of phosphate fluxes for simple and direct determination of uranium by LED Fluorimeter. Monazite is a refractory REE-mineral. These REE-bearing minerals contain inclusions of monazite/xenotime. The findings have previously published elsewhere without proper cross referencing /justification in the cited manuscript. It constitutes citation manipulation including plagiarism as well as scientific misconduct. A separate publication and claim by authors dealing with uranium determination in monazite by pulsed fluorimetry (LED fluorimeter) has no novelty/originality/improvements in the manuscript. Monazite sample decomposition using concentrated sulphuric will be the best option for uranium measurement in such matrices using laser/LED fluorimetry, as it eliminates matrix matching of calibration standards.

Keywords: Uranium; REE minerals; REE bearing minerals; Phosphate flux; Pulsed fluorimetry

Comments

I have read the cited paper [1], all the cited references therein and available published literature on the above subject very carefully. I would like to present my comments on the published article, studies on effective decomposition of monazite minerals by variety of phosphate fluxes for simple and direct determination of uranium by LED Fluorimeter.

The conventional pellet fluorimetry is age old measurement procedure for the routine determination of uranium in diverse matrices [2,3]. The laser based instrumental techniques [4], such as, pulsed fluorimeter/ spectrofluorimeters/ phosphorimeters were evolved during the past three decades, utilizing the half-life of phosphorescent uranyl compounds and its decay as a diagnostic method for the low cost, simple and direct determination of uranium at μgL^{-1} levels in natural water samples for hydro-geochemical reconnaissance surveys for uranium.

In view of the rapidity and simplicity of measurement by pulsed fluorimetry, the main emphasis was either on developing new fluorescence-enhancing reagents or on methods of measurements for different types of sample matrices. Several workers have developed methods for the determination of uranium in matrices, like rocks, soils and sediments, mineralized rocks, concentrates and other U-rich materials [5-8], in which sample matrix effects are either eliminated by dilution of sample solution to such an extent that quenching by impurities no longer influences the analysis, or the separation of the uranium from the quenchers by extraction, compensation of matrix effects by use of an internal standards (standard addition), or other methods.

Sample decomposition and dissolution of refractory, nonsilicate minerals, like ilmenite, rutile, columbite, tantalite and xenotime [9], tin slag [10] monazite [11], Th and REE-rich matrix [12] have been the subject of many studies, followed by determination of REE and other accompanying elements by ICP-OES, and uranium by pulsed fluorimetry/fluorimetry. The mineral chemistry of REE-minerals and

REE-bearing minerals are well documented in the literature. Monazite and Xenotime are REE-minerals while REE-bearing minerals are Apatite, Zircon, Fluorite, Rutile and Iron oxides [13]. As claimed by the authors, the paper describes, quote "A simple, rapid, effective sample decomposition method is developed for the determination of uranium (U) in monazite minerals by fluorimetric (Light Emitting Diodes (LED) based) technique". The salts of sodium dihydrogen phosphate (NaH_2PO_4), disodium hydrogen phosphate (Na_2HPO_4) and tetrasodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$) were used to conduct studies on effective decomposition and dissolution of monazite minerals unquote. These REE-bearing minerals contain inclusions of monazite/xenotime.

The author, Chakrapani has earlier reported vide his publications (Development of Novel Flux for effective sample decomposition of Refractory samples and Field Oriented Pre-concentration methods for the determination of Twenty Nine Trace and Ultra Trace Elements in a variety of Geological Samples by Flame and Plasma Techniques for application to Geochemical Exploration of Uranium by Chakrapani [11] and A Rapid Fusion Technique for Chemical Characterization of Monazite by ICP-OES by Kumar and Chakrapani, Journal of Applied Geochemistry [11] of the publication quote "Novel sample decomposition procedure for refractory samples for direct determination of uranium" unquote and also vide his earlier publication cited as reference no. 5 in the present publication in J Indian Chem Soc [1]. These two publications also highlighting novel sample decomposition and dissolution of refractory minerals have not been included by them in the above cited manuscript. It constitutes citation

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manipulation including plagiarism as well as scientific misconduct.

As stated in the manuscript, on Page 1935, third para quote “Radhamani et al. [5] adopted a fusion method, using a mixture of tetrasodium pyrophosphate and monosodium dihydrogen phosphate (1:1) and prepared the solution in 3% HCl to solubilise the insoluble Ce^{IV} polyphosphate. The presence of high amount of chloride quenches the fluorescence” unquote. This statement quoted is not based on reference [5] in the above cited publication. The authors have suppressed the already reported and published information related with the determination of uranium in refractory minerals (a publication by Chakrapani, titled “Novel fusion method for direct determination of uranium in ilmenite, rutile, columbite, tantalite and xenotime minerals by laser induced fluorimetry by Radhamani et al. published in *Journal of Radioanal Nucl Chem* [9].

At the same time, the statement quoted in the manuscript on Page 1938, section, High sample throughput: quote “Radhamani et al. [9] adopted a fusion method, using a mixture of tetra sodium pyrophosphate and monosodium dihydrogen phosphate (1:1) and prepared the solution in 3% HCl to solubilise the insoluble Ce^{IV} polyphosphate” unquote. This is the same statement as on Page 1935, third para of the cited manuscript. There is no discussion /comment about their earlier publication cited as reference [5] in the above cited manuscript and other publications. The findings have previously published elsewhere without proper cross referencing /justification in the cited manuscript.

In my opinion, monazite sample decomposition using concentrated sulphuric acid [14] (with continuous stirring at 300°C, leached in ice, filtered off and washed for more than ten times with distilled water. The filtrate, forming dissolving monazite, is then completed up to volume and uranium is then analyzed) will be the best option for uranium measurement in such matrices using laser/LED fluorimetry. Alternatively, the procedure adopted in the application of differential technique in laser induced fluorimetry [6] will be ideal for the determination of uranium in such matrices using phosphate flux for sample decomposition as described by the authors, as it will eliminate matrix matching of calibration standards.

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