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Iterative method combined with HRGS for physical characterization of uranium materials in the frame of nuclear forensics investigations

High-resolution gamma spectrometry (HRGS) is a non-destructive analysis technique, that plays a dual role at the first stages of nuclear forensic examination: fast in-field categorization of radiological or nuclear material in order to identify the safety risk to first responders and to the public and more comprehensive characterization of the material in the laboratory to accurately determine the isotopic composition.

Let us imagine that the nuclear forensic scientist needs to investigate seized nuclear material, categorized as natural uranium (NU), in a sealed container. With equal probability, the container may store a piece of metal uranium or powder of uranium ore concentrate (UOC) or uranium hexafluoride (UF_6) or other uranium compounds. To perform a high-precision destructive analysis, a scientist must open the container, but the safety of the work will be greatly facilitated if the scientist could accurately estimate the risks posed by the material contained within. In this case, material's physical characterization in order to establish its shape can help.

\begin{figure}[htbp]
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\includegraphics[width=3.2in]{figure_1.eps}
\caption{Material physical categorization based on value of matrix density and uranium mass fraction\label{F1}}
\end{figure}

The characteristics of uranium materials of the nuclear fuel cycle vary over a wide range of values. The uranium mass fraction (uranium assay), ω, and the matrix density, ρ, are the physical characteristics of the material, the knowledge of which makes it possible to assess its physical form and, as a consequence, make a decision on the necessary safety measures in the process of its further characterization. Once the quantities, ω and ρ, of an unidentified material are determined, its shape can be deduced (see the diagram in Fig.1).

The proposed approach rests on processing HRGS experimental results with the iterative quasi-Newton Broyden method for finding roots of two equations in variables ρ and ω.

For investigation we used set of uranium materials in various shapes (powders, granules, metals and alloys), see Fig. 2 with enrichment from 0.4 to $19.7\,\%$. In addition, unsealed samples of pure uranium metal (see Fig. 3) and fuel elements in the form of microspheres based on UO_2 (see Fig. 4 and 5) were studied.

Using HRGS characterization supplemented with our iterative algorithm, the powders of U_3O_8 with uranium mass fraction of about $84\,\%$ can be distinguished from the powders of UO_2 with uranium mass fraction of about $87\,\%$, as well as uranium products in form of liquids or loose powders with matrix density of $0.5 - 2.0\,g/cm^3$ can be distinguished from uranium products in form of compacted fuel elements with matrix density of $6.0 - 10.0\,g/cm^3$ or from pure metal uranium and uranium alloys with matrix density of $14.0 - 19.0\,g/cm^3$.

\begin{figure}[htbp]
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\includegraphics[width=3.2in]{figure_2.eps}
\caption{A set of uranium materials in various shapes.\label{F2}}
\end{figure}
\begin{figure}[htbp]
\centering
\includegraphics[width=3.2in]{figure_3.eps}
\caption{Unsealed sample of pure uranium metal.\label{F3}}
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\begin{figure}[htbp]
\centering
\includegraphics[width=3.2in]{figure_4.eps}
\caption{Image of coated by SiC fuel microsphere slice obtained by Leica M165 optical microscope\label{F4}}
\end{figure}
\begin{figure}[htbp]
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\includegraphics[width=3.2in]{figure_5.eps}
\caption{Fuel microspheres based on UO2 (general view).\label{F5}}
\end{figure}

The values of ω and ρ obtained by HRGS are confirmed by independent analytical methods. Uranium mass fraction in the fuel microspheres based on UO_2, estimated as $88.02\,\%$ by HRGS is consistent with the results of isotope dilution mass spectrometry $87.76 \pm 0.64\,\%$. Densities of two different uranium metal samples, estimated as $18.42\,g/cm^3$ and $19.33\,g/cm^3$ by HRGS is consistent with the results of gas pycnometry technique $18.24 \pm 0.55\,g/cm^3$ and $18.86 \pm 0.59\,g/cm^3$, respectively.

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