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# Determination of $^{99}\text{Tc}$ in $\text{UO}_2$ sample using liquid extraction separation and LSC measurement

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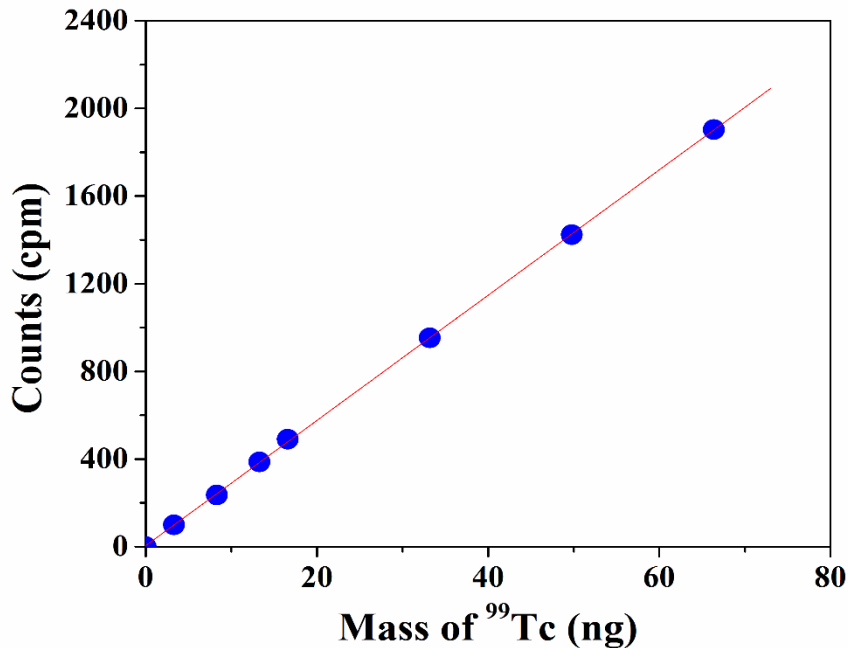
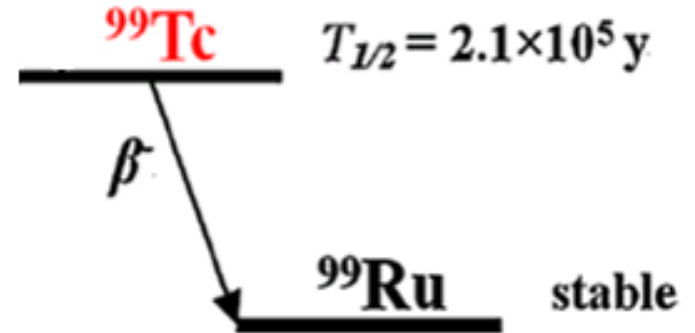






## For $^{99}\text{Tc}$ measurement

- A purity  $\beta$  emitter;
- Maximum  $\beta$  energy of 0.294 MeV;
- Can be measured by liquid scintillation counting





## Determination of $^{99}\text{Tc}$ in $\text{UO}_2$ sample

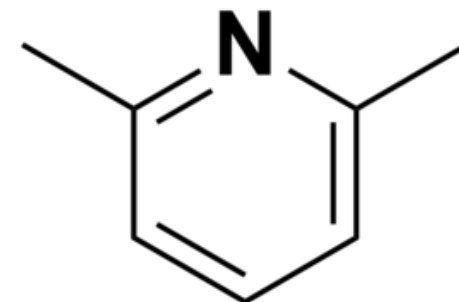
- Low level of  $^{99}\text{Tc}$ ;
- High concentration of uranium;
- Efficient decontamination of uranium interference is necessary

Sample number	Concentration of $^{99}\text{Tc}$ ( $\mu\text{g/mL}$ )	Concentration of U(VI) ( $\text{g/mL}$ )	$\beta$ counting rate (cpm)
1	0.0332	0	980
2	0.0332	0.004	67297
3	0.0332	0.006	99628
4	0.0332	0.008	128990
5	0.0332	0.01	157160
6	0.0332	0.012	183974
7	0.0332	0.016	232437



## Solvent extraction method was applied for $^{99}\text{Tc}$ purification

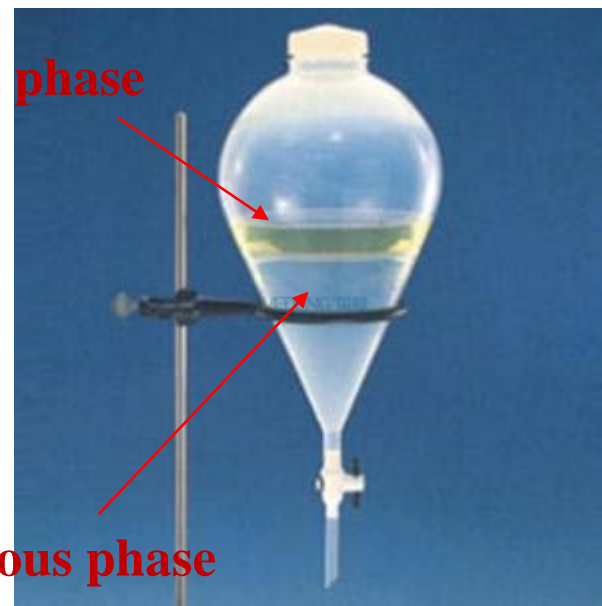
- Extractant: 2,6-dimethylpyridine;
- Aqueous: NaOH medium;
- Optimized parameters
  - Contact time;
  - Concentration of  $\text{NO}_3^-$  ;
  - Concentration of NaOH;
  - Volume ratio of organic to aqueous



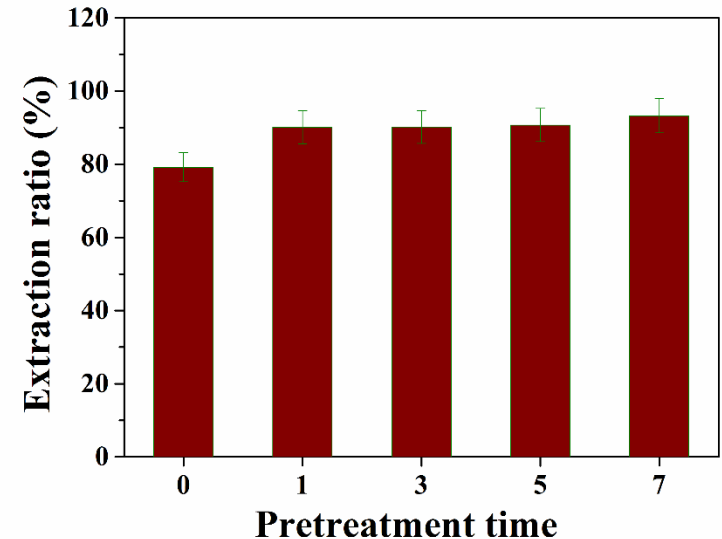
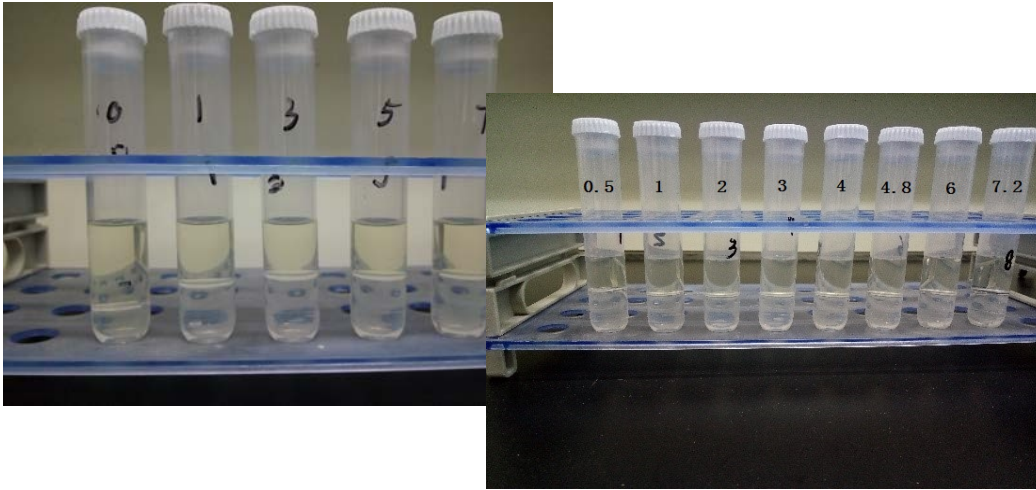
2,6-dimethylpyridine

$^{99}\text{Tc}$  in organic phase

U(VI) in aqueous phase



## □ Pretreatment of extractant with NaOH solution

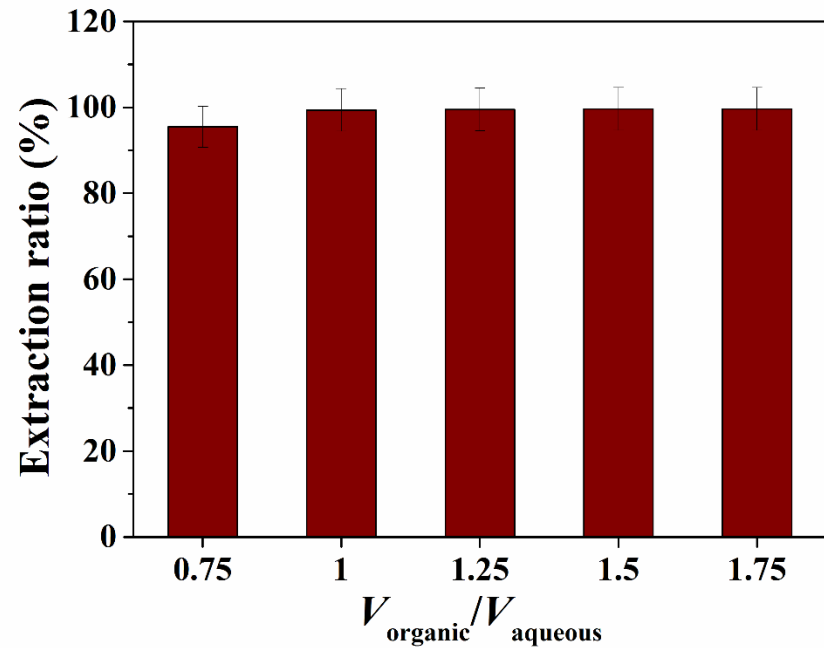
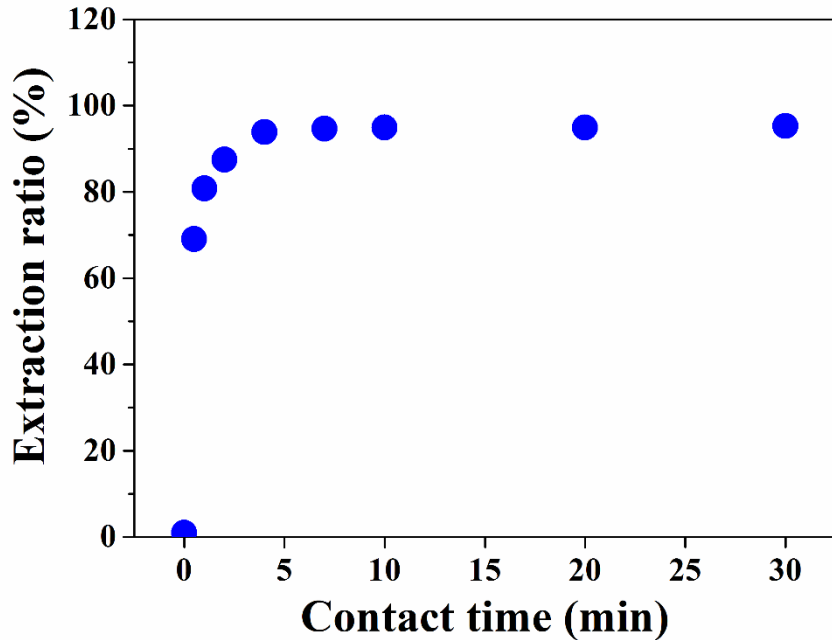


- 2,6-dimethylpyridine is miscible in acid medium;
- Two phase can be formed when the aqueous phase is strong alkaline medium;
- The volume expansion can be avoided when the extractant was pretreated with NaOH solution more than 3 time





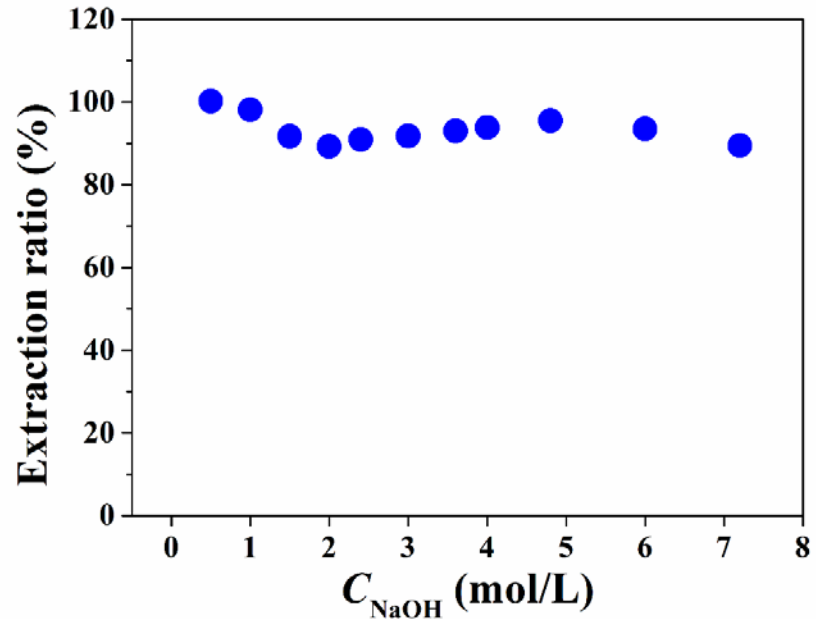
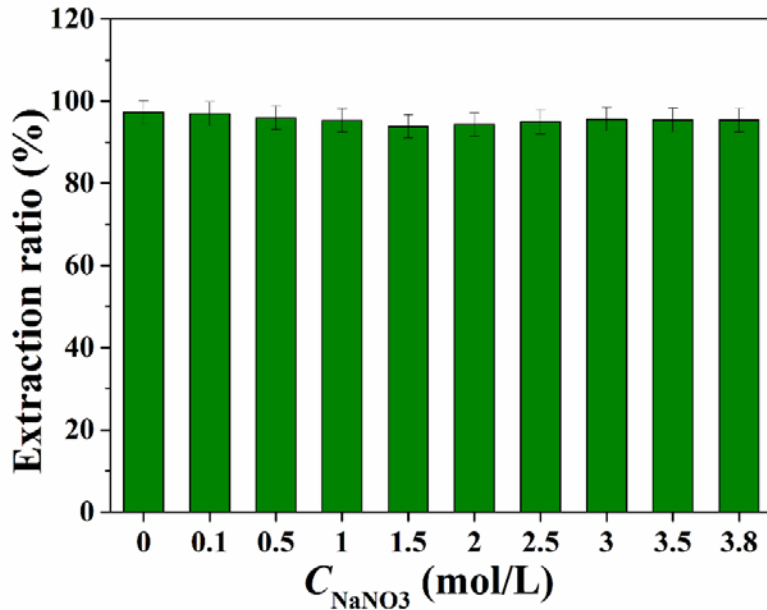
## Effect of contact time and volume ratio of organic to aqueous



- 1) The extraction rate of  $^{99}\text{Tc}$  from aqueous phase to organic phase is so fast, the equilibrium can be reached within 5 min;
- 2) The extraction ratio of  $^{99}\text{Tc}$  is close to 100% and keeps stable when the volume ratio of organic phase to aqueous phase is above 1:1.



## Effect of $\text{NO}_3^-$ and NaOH concentration on the extraction of $^{99}\text{Tc}$



- 1) The effect of  $\text{NO}_3^-$  concentration (from 0 to 3.8 mol/L) on  $^{99}\text{Tc}$  extraction is insignificant;
- 2) The lower concentration of NaOH, the higher extraction ratio of  $^{99}\text{Tc}$ ;
- 3) It is difficult to separate the organic phase and aqueous phase when the concentration of NaOH is less than 0.5 mol/L.

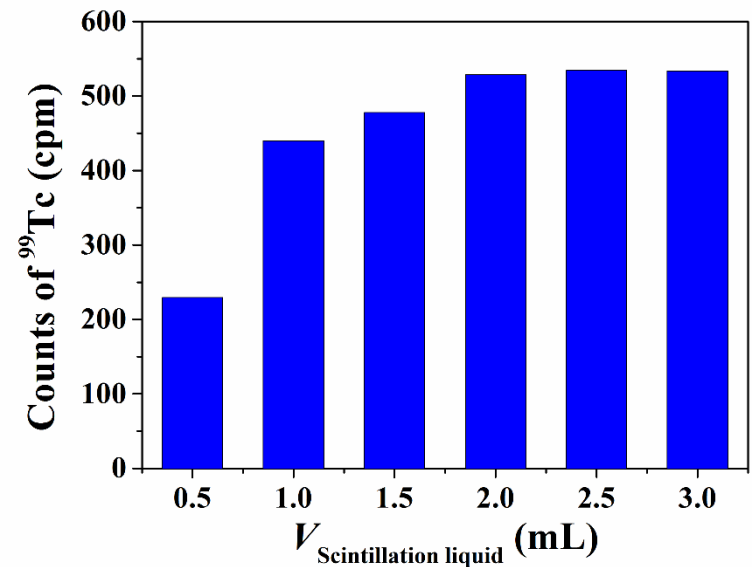


## Effect of U(VI) concentration on the extraction of $^{99}\text{Tc}$

Sample number	1	2	3	4	5	6	7
Counts in aqueous (cpm) (before extraction)	857	67297	99628	128990	157160	183974	232437
Counts in organic phase (after extraction) (cpm)	860	857	865	851	870	866	859
Quality of U(VI) added (mg)	0	28.24	42.36	56.48	70.6	84.7	112.96

## Effect of liquid scintillator volume

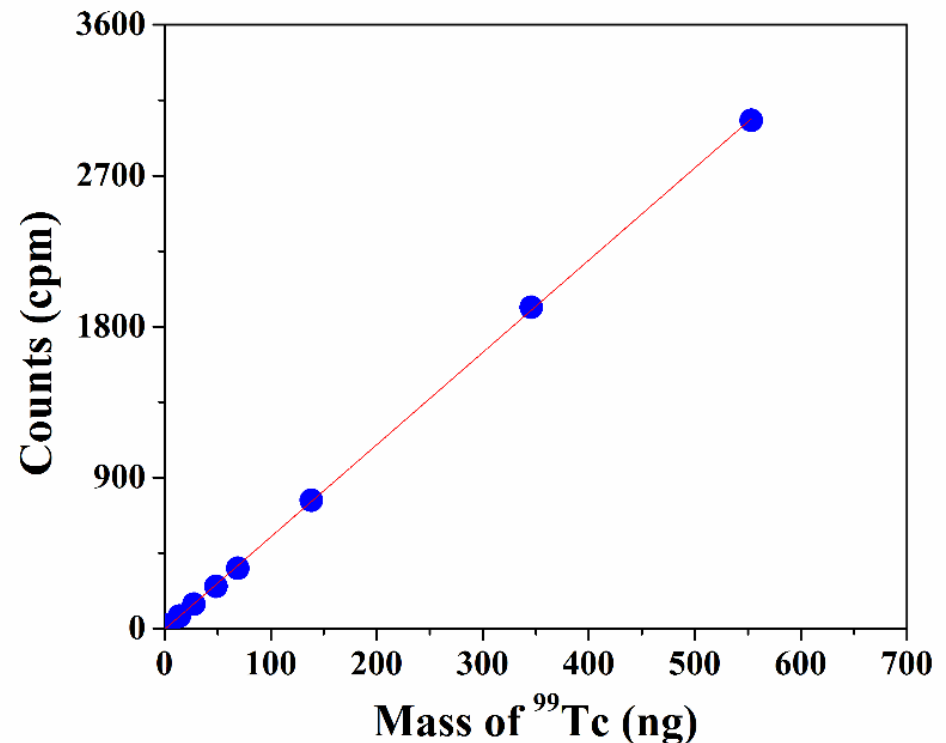
- ✓ Measurement time: 10 min;
- ✓ Volume of organic sample: 1 mL;
- ✓ The liquid scintillation cocktail is kept in dark room more than 6 h.





## □ Working curve

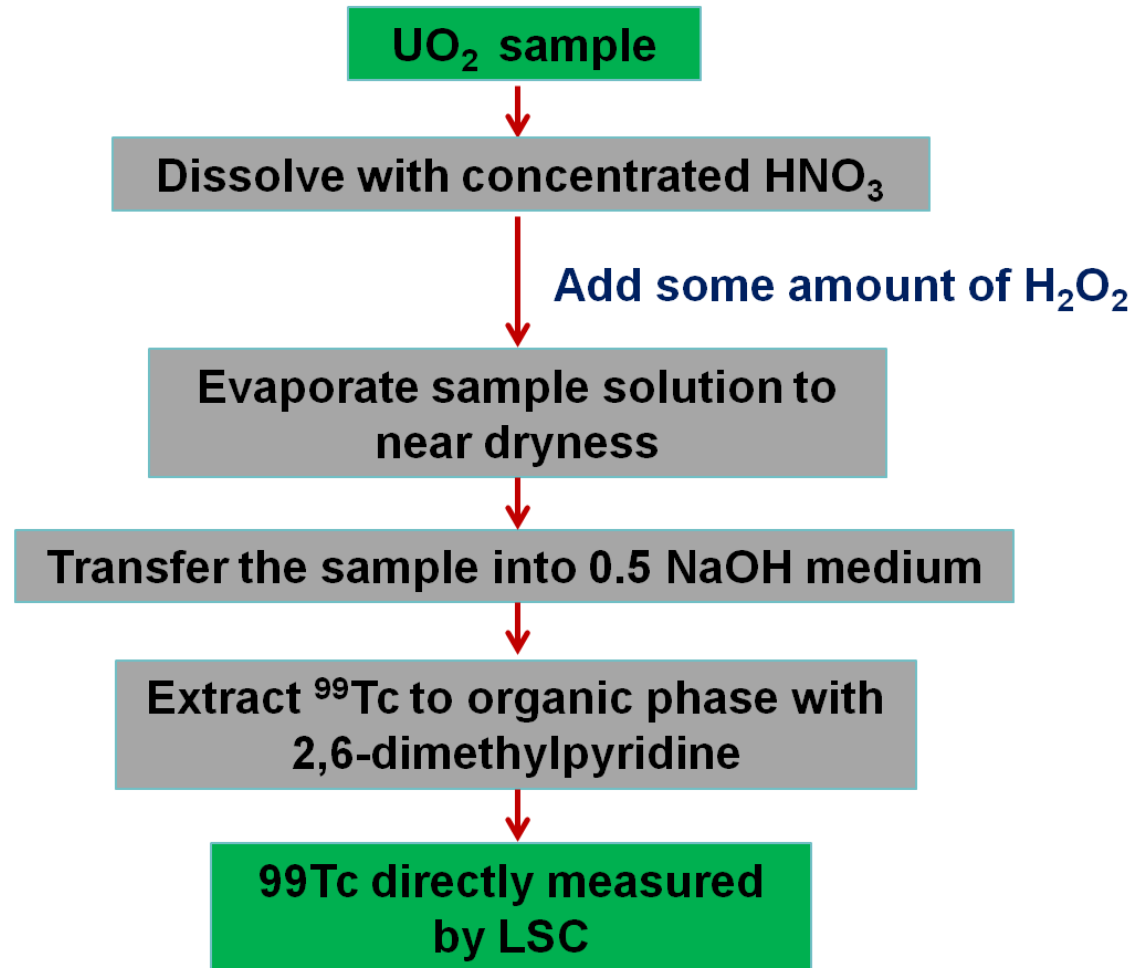
- ✓ Contact time for extraction: 10 min;
- ✓ Aqueous phase: 0.5 mol/L NaOH medium with  $^{99}\text{Tc}$  and uranium;
- ✓  $V_{\text{organic}}/V_{\text{aqueous}}$ : 2 mL/2mL;
- ✓ Content of U(VI): 0.16 g;
- ✓ For measurement:  
 $V_{\text{organic sample}} = 1 \text{ mL}$   
 $V_{\text{liquid scintillation}} = 2 \text{ mL}$







## □ Analytical procedure for $^{99}\text{Tc}$ determination in $\text{UO}_2$ samples





## □ Procedure evaluation using standard addition method

- Amount of  $\text{UO}_2$  sample: 0.2 g;
- Quality of  $^{99}\text{Tc}$  added: 0.25  $\mu\text{g}$ ;
- Volume of organic phase/aqueous phase: 2 mL/2mL



The analytical results for  $^{99}\text{Tc}$  spiked in  $\text{UO}_2$  samples

Sample number	1	2	3	4	5	6
$^{99}\text{Tc}$ measurement ( $\mu\text{g}$ )	0.235	0.239	0.246	0.235	0.238	0.238
Recovery (%)	94.4	95.9	98.8	94.3	95.6	95.5
RSD	1.76 %					



- Because of the interference of uranium, the direct determination of  $^{99}\text{Tc}$  in  $\text{UO}_2$  sample by LSC is difficult;
- $^{99}\text{Tc}$  can be effectively extracted from 0.5 mol/L NaOH solution by 2,6-dimethylpyridine, but keep uranium in aqueous;
- The parameters which affect the purification of  $^{99}\text{Tc}$  during extraction step were optimized;
- A procedure for rapid determination of  $^{99}\text{Tc}$  in  $\text{UO}_2$  sample was set up and applied for sample analysis;
- The analytical method was evaluated using standard addition method, the recovery of  $^{99}\text{Tc}$  is higher than 94%, and the RSD for 6 samples is lower than 2%.

**Thanks for your  
attention**

