**NNLC542 Understanding physical properties of settled Zirconium Molybdate (ZM) solid beds**

**Request for Information (RFI)**

This exercise is a request for information as part of a pre-tender market consultation, it does not constitute a formal procurement process. The purpose of this exercise is to assess the capabilities within current markets to delivery this service. National Nuclear Laboratory (NNL) may choose to contact suppliers because of completing this questionnaire, if you wish for us not to contact you please make this clear on your questionnaire response.

We are not requesting quotations at this stage, although some questions request examples of previous contracts. This is to understand the indicative cost of any future procurement, but respondents will not be held to these values.

**Background**

Zirconium Molybdate (ZM - ZrMo2O7(OH)2(H2O)2) is a solid which forms, from conversion of caesium phosphomolybdate in the presence of zirconium, during storage of highly active liquid waste generated from nuclear reprocessing operations [e.g. 1-4].

ZM has been observed to settle in nitric acid to form solid beds with variable yield stress (the force required to initiate the movement of the solid bed). The measured yield stress (bench scale rotational measurements at ambient temperature with a vane tool and applying incremental stress) have ranged from 10’s of Pa to 10’s of kPa. In some instances, we have observed that when a settled solid bed with a yield stress of >100 Pa is mobilised such that the ZM is re-suspended in nitric acid, the ZM subsequently settles to form a solid bed where the yield stress is reduced to 10’s of Pa. However, in other observations this is not the case with the solids re-settling to a solid bed with a yield stress of >100 Pa. The yield stress values measured of solid beds have been found to be variable under nominally identical conditions, leading to uncertainty in the understanding why such solid beds form and conditions which promote solid beds of varying yield stress.

Typical examples of ZM settled solid beds of varying yield stress which have been measured are:

* ZM was manufactured at 900 litre scale and subsequently washed with 0.1 M nitric acid or demineralised water. The yield stress of the resultant solid bed between nominally identical batches varied, forming solid beds of varying yield stress from several 10’s kPa or very mobile beds (<10 Pa).
* For ZM manufactured at 4 litre scale in 1.5–2 M nitric acid, the ZM always settled to form a solid bed of low yield stress (10’s of Pa).
* Washing of ZM solid beds of low yield stress (10’s of Pa) with water or nitric acid of low concentration (≤0.5 M) formed in some instances solid bed of >250 Pa, but in other instances the settled solid bed exhibited a low yield stress (10’s of Pa).
* After heating of ZM in boiling (50-60 °C) nitric acid solutions of up to 10 M under reduced pressure in some instances formed settled solid beds with estimated yield stress to be >100 Pa (in particular where the suspension of solids at the base of the vessel by the boiling action was hindered due to low heat flux or the presence of a hydrostatic head). Once re-suspended, the ZM settled to a solid bed of lower yield stress (10’s of Pa).

**Problem**

When ZM settles to a solid bed with high yield stress (>100’s of Pa), mobilisation of such solids for subsequent transportation through plant may be difficult potentially leading to solids being left behind in vessels. This is undesirable as it reduces the quantity of solids being vitrified for disposal, but also leaves a greater legacy for decommissioning activities. Understanding the mechanism to why ZM forms settled solid beds of varying yield stress, may allow conditions to be optimised for plant operations. Experiments have been carried out at NNL where the yield stress of the resulting settled ZM bed has been measured specifically under plant conditions. This approach has thus far been unsuccessful at elucidating an explanation of behaviour.

**Aim of task**

To investigate the formation of yield strength in settled ZM beds and determine parameters that impact the experimentally observed differences in yield stress. The task should aim to determine a reason for the behaviour and potential mechanism. Furthermore, it would be advantageous if the conditions which promote unwanted high yield stress is identified for nitric acid-based solutions.

**Proposed task**

The project should take around 1 year to complete. The work may be analytical based and may be mechanistic in principle. Suitable analysis and/or measurement technologies may be used to evaluate settled solid beds of ZM. Due to the yield stress potentially changing when settled solid beds are subsequently mobilised, in-situ measurements may be required. It is considered by NNL that the reason for yield stress variability may be due to the properties of ZM and its surface/ particle interactions. Therefore, an approach which could elucidate the mechanism of high yield stress formation in settled ZM solid beds is desired.

**References**

1. Bradley, D F; Quayle, M J; Ross, E; Ward, TR and Watson, N, “Promoting the conversion of caesium phosphomolybdate to zirconium molybdate”, P1-45, Atalante 2004, Nimes (France), 2004
2. Shiels, J; Harbottle, D; Hunter, T N, “Synthesis and Characterisation of Caesium Phosphomolybdate and Zirconium Molybdate found in the Highly Active Storage Tanks at Sellafield”, Paper 18154, WM2018 Conference, March 18-22, 2018, Phoenix, Arizona, USA, 2018
3. Paul, N; Biggs, S; Edmondson, M; Hunter, T; Hammond, R, “Characterising highly active nuclear waste simulants”, Chemical Engineering Research & Design 91, p742-751, 2013
4. Dunnett, B; Ward, T; Roberts, R and Cheesewright, J, “Physical properties of highly active liquor containing molybdate solids”, Procedia Chemistry 21, p24-31, 2016

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| **Contact Information** | | | |
| Company Name |  | | |
| Address |  | Postcode |  |
| Name of person completing this form |  | | |
| Contact Details |  | | |
| Email Address |  | | |
| Phone Number |  | | |

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|  | **Relevant Experience** | |
| Have you worked with the Nuclear Industry before? |  | |
| Have you worked with the National Nuclear Laboratory before? |  | |
| Please detail up to three contracts where you have previously provided .  You should highlight who the contracting body is/was, the work carried out and scope required. | Contract 1: |  |
| Contract 2: |  |
| Contract 3: |  |
| Please can you describe your area of expertise relevant to this RFI. |  | |
| Is your company prepared to provide a (Rough Order of Magnitude) ROM cost to provide the scope above to support with the project approval stage? |  | |
| If yes to the above question, please provide ROM cost here. |  | |

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| **Procurement** | | |
| Are you on any frameworks operated nationally by Framework providers, for example:  CCS – Crown Commercial Service,  NHS SBS - NHS Shared Business Services  LUPC – London Universities Purchasing Consortium  Yorkshire Purchasing Organisation - YPO,  Eastern Shires Purchasing Organisation – ESPO  Dynamic Purchasing System (DPS) - NNL |  | |
| If yes, please provide the following information in relation to the framework. | Framework Provider |  |
| Framework Reference |  |
| If Lots, list all applicable |  |
| Do you have any internal or external pressures facing your business which would prevent you from tendering for this opportunity, should it become available May 2024? |  | |

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| **Additional Comments** | |
| If you DO NOT wish to be contacted by NNL please write so here. Otherwise, you may provide any further information you think may be of use during this early market engagement exercise. |  |

**Please return this questionnaire to** [**david.brown@uknnl.com**](mailto:david.brown@uknnl.com) **before 17:00 on**

**Friday 8th March 2024.**