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Control charts in multi-element analysis of human urine

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Within a study to evaluate the level of depleted uranium in urine from Italian soldiers involved in a peacekeeping mission, the concentration of eight other elements was determined by means of Inductively Coupled Plasma Mass Spectrometry. An internal quality control scheme was planned together with the method validation and carried out along with the analysis of samples. Two certified reference materials (CRMs) containing Arsenic (As), Cadmium (Cd), Lead (Pb), Molybdenum (Mo), Nickel (Ni), Tungsten (W), Vanadium (V), Zirconium (Zr) and Uranium (U) were used: Seronorm™ Trace Elements Urine Blank and Seronorm™ Trace Elements Urine. Due to the large number of specimens to be analysed (~1350) an in-house control material was also prepared and its suitability for IQC evaluated.

PREPARATION OF IN-HOUSE SPIKED MATERIAL FOR IQC (RM-IH): a pooled human urine sample was acidified with HNO_3 and centrifuged to remove any precipitate. The clear supernatant was analyzed to verify the concentration of each element. Since the levels of U, V and Zr were very low, the urine pool was spiked with 0.045, 0.40, 0.05 µg/L of U, V and Zr, respectively. The final material was homogenized by gentle mixing and stored in polypropilene tubes at +4 ℃.

BUILDING OF SHEWART CONTROL CHARTS (CCs): target values for RM-IH were assigned by analyzing it several times together with the CRMs. The analytical results were accepted only when the values obtained for CRMs in the same run fell within the range certified value ± 2u (Fig.1). The observed mean values and SDs for each analyte in the RM-IH, given in the table, were used to set the central line, the upper/lower warning limits (± 2SD) and the upper/lower action limits (± 3SD). For the CRMs, the certified value was chosen as the central line and warning and action limits were set at ± 2u and ± 3u, respectively. Control charts for the determination of Uranium in RM-IH and CRM are shown as examples (Fig. 2 and Fig. 3).

Building of CCs: basis for central line and limits

Analyte	Pooled urine (n>30)		CRM	
	Mean value (µg/L)	SD (µg/L)	Certified (µg/L)	u (µg/L)
As	62.6	2.7	85	2.5
Cd	0.41	0.028	0.31	0.025
Mo	67.1	1.3	49.3*	1.3*
Ni	2.4	0.26	2.4	0.3
Pb	1.7	0.08	0.75	0.025
U	0.045	0.003	0.037	0.003
V	0.43	0.04	0.53	0.04
W	0.18	0.01	0.14	0.025
Zr	0.05	0.02	0.081	0.0022

* Reference value and SD reported for Seronorm Urine

CONCLUSIONS: the SDs observed during the assessment of the RM-IH, were confirmed for each analyte at the end of a long-term evaluation. This values were comparable with the uncertainties reported for CRM values, confirming that RM-IH was suitable for IQC purpose. Furthermore, the use of such material has some analytical advantages: it is more representative of the real sample; it is easy to dilute; the occurrence of sample contamination is reduced and the overall analysis is less expensive.

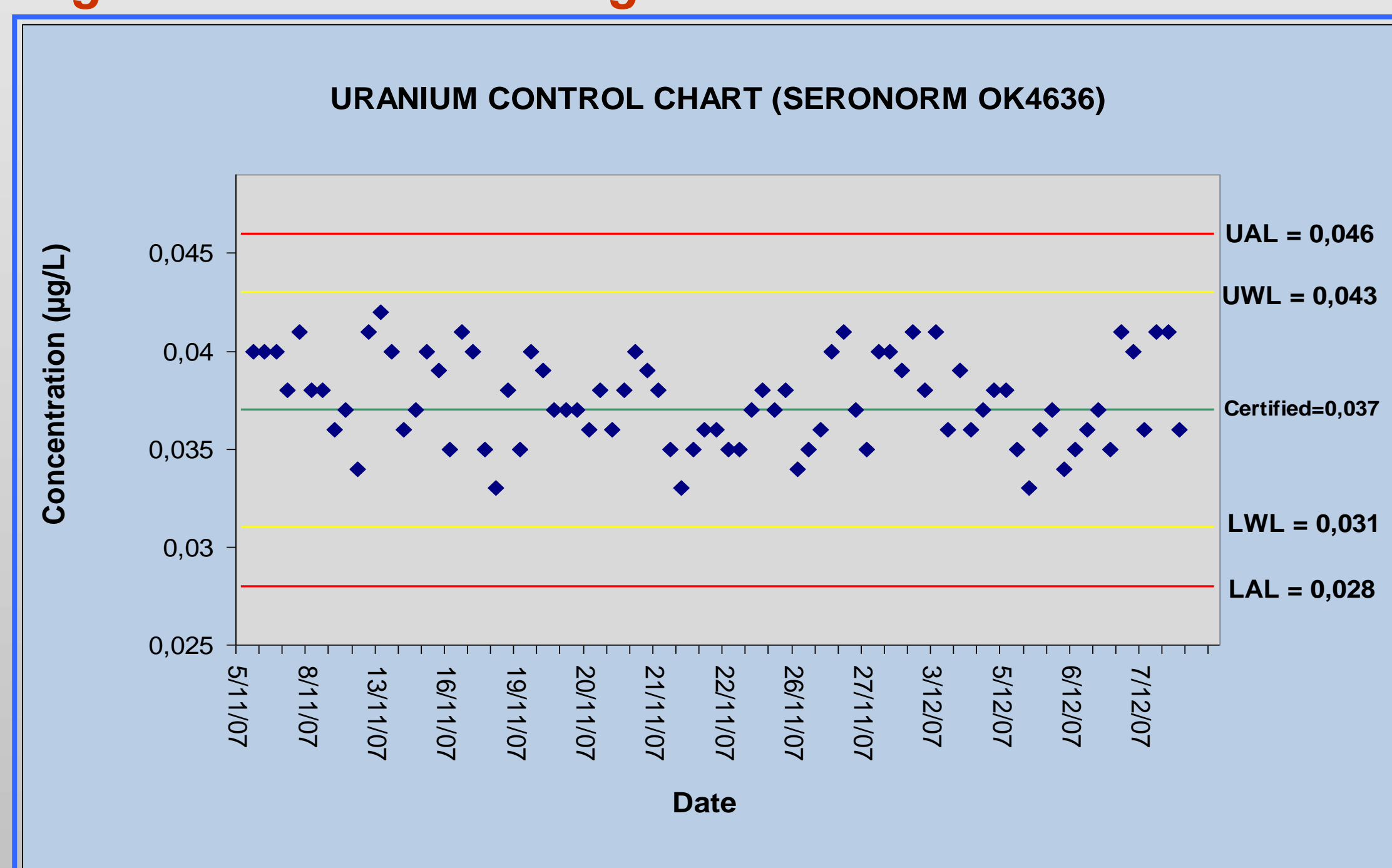**Fig. 1: CC for CRM during the assessment of the RM-IH**

URANIUM CONTROL CHART (SERONORM OK4636)

Fig. 2: CC for RM-IH during the analysis of the samples
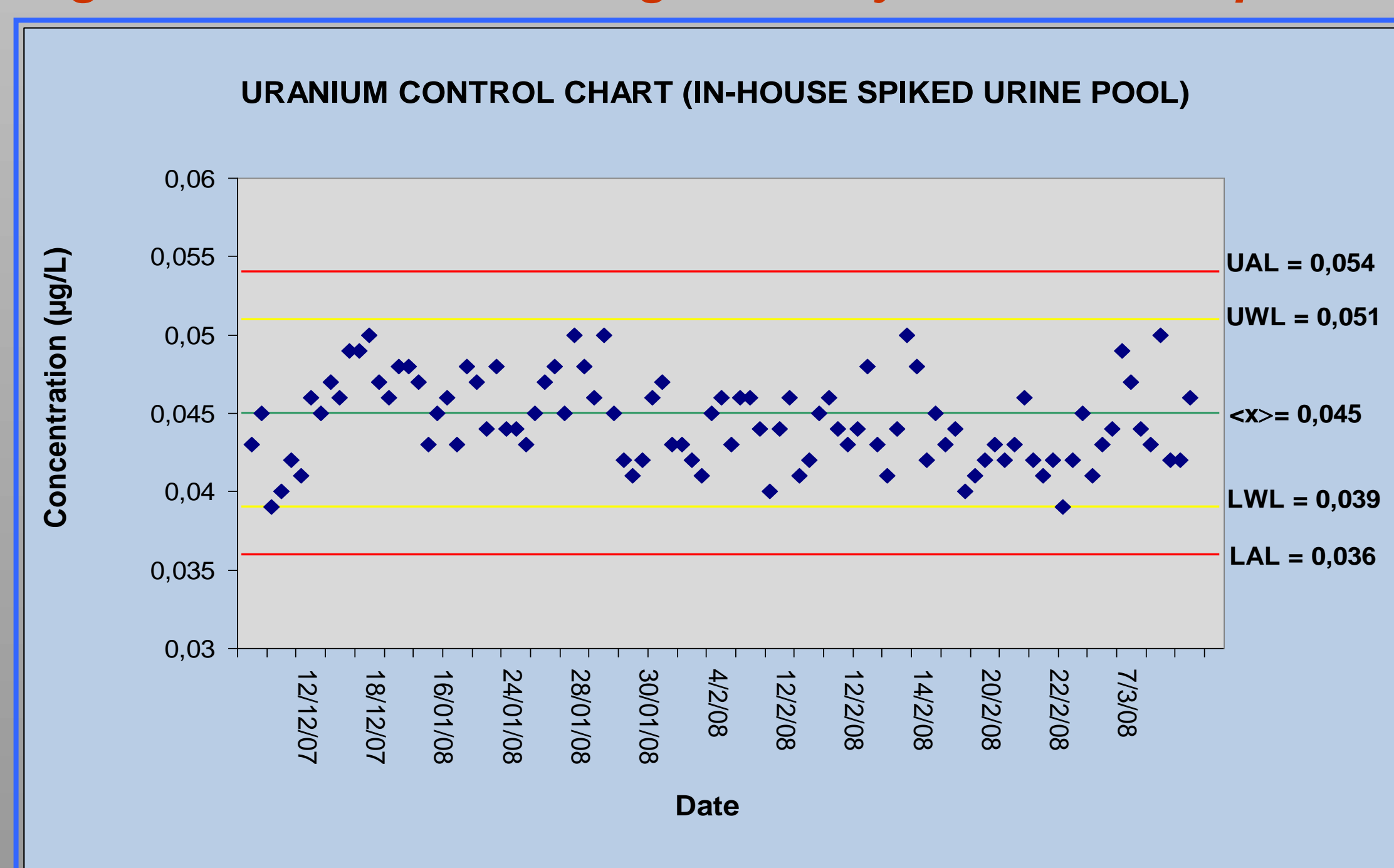

URANIUM CONTROL CHART (IN-HOUSE SPIKED URINE POOL)

Fig. 3: CC for CRM during the analysis of the samples

URANIUM CONTROL CHART (SERONORM OK4636)