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^{90}Sr Liquid Scintillation Urine Analysis Utilizing Different Approaches for Tracer Recovery

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NOMENCLATURE

cpm	counts per minute
dpm	disintegrations per minute
LEB	low-energy beta
LEBAB	low-energy beta/alpha/beta
LSC	liquid scintillation counting
MDA	minimum detectable activity
QC	quality control
RPSD	Radiation Protection Sample Diagnostics
SNL	Sandia National Laboratories
TSIE	transformed spectral index of an external standard
UGXR™	Ultima Gold (Extended Range) ™
UGLLT™	ULTIMA Gold (Low Level Tritium) ™
Sr carrier	Stable elemental strontium
Sr85 tracer	radioactive isotope of strontium
Sr85 LEBAB	LSC protocol with specific window settings and calibration curve
Sr90 DPM	LSC protocol with specific window settings and calibration curve

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ABSTRACT

^{90}Sr is one of the isotopes most commonly produced by nuclear fission. This medium lived isotope presents serious challenges to radiation workers, the environment, and following a nuclear event, the general public. Methods of identifying this nuclide have been in existence for a number of years (e.g. Horwitz, E.P. [1], Maxwell, S.L.[2], EPA 905.0 [3]) which are time consuming, requiring a month or more for full analysis. This time frame is unacceptable in the present security environment. It is therefore important to have a dependable and rapid method for the determination of Sr. The purposes of this study are to reduce analysis time to less than half a day by utilizing a single method of radiation measurement while continuing to yield precise results. This paper presents findings on three methods that can meet this criteria; (1) stable Sr carrier, (2) ^{85}Sr by gamma spectroscopy, and (3) ^{85}Sr by LSC.

Two methods of analyzing and calculating the ^{85}Sr tracer recovery were investigated (gamma spectroscopy and a low energy window-Sr85LEBAB by LSC) as well as the use of two different types of Sr tracer (^{85}Sr and stable Sr carrier). Three separate stock blank urine samples were spiked with various activity levels of ^{239}Pu, ^{137}Cs, ^{90}Sr /^{90}Y to determine the effectiveness of the Eichrome Sr-spec™ resin 2mL extractive columns. The objective was to compare the recoveries of ^{85}Sr versus a stable strontium carrier, attempt to compare the rate at which samples can be processed by evaluating evaporation, neutralization, and removing the use of another instrument (gamma spectrometer) by using the LSC spectrometer to obtain ^{85}Sr recovery.

It was found that when using a calibration curve comprised of a different cocktail and a non-optimum discriminator setting reasonable results (bias of ± 25%) were achieved. The results from spiked samples containing ^{85}Sr demonstrated that a higher recovery is obtained when using gamma spectroscopy (89-95%) than when using the LEB window from LSC (120-470%). The high recovery for ^{85}Sr by LSC analysis may be due to the interference/cross talk from the alpha region since alpha counts were observed in all sample sets. After further investigation it was determined that the alpha counts were due to ^{239}Pu breakthrough on the Sr-spec™ column. This requires further development to purify the Sr before an accurate tracer recovery determination can be made. Sample preparation times varied and ranged from 4-6 hours depending on the specific sample preparation process.

The results from the spiked samples containing stable strontium nitrate $Sr(NO_3)_2$ carrier demonstrate that gravimetric analysis yields the most consistent high recoveries (97-101%) when evaporation is carefully performed. Since this method did not have a variation on the tracer recovery method, the samples were counted in 1) LEB/Alpha/Beta mode optimized for Sr-90, 2) DPM for Sr-90, and 3) general LEB/Alpha/Beta mode. The results (from the known) ranged from 79-104%, 107-177%, and 85-89% for 1, 2, and 3 respectively. Counting the prepared samples in a generic low energy beta/alpha/beta protocol yielded more accurate and consistent results and also yielded the shortest sample preparation turn-around-time of 3.5 hours.

INTRODUCTION

Background

Strontium-ninety (^{90}Sr) is the most hazardous of the radioactive isotopes of the chemical element strontium. ^{90}Sr is formed in nuclear reactors or during the explosion of nuclear weapons. It gives off beta particles and decays into yttrium-ninety (^{90}Y); ^{90}Y is also radioactive and gives off radiation to form zirconium-ninety (^{90}Zr), which is a stable isotope. ^{90}Sr has limited use and is considered a waste product. Once strontium enters the bloodstream, it is distributed throughout the body and behaves like calcium. A large portion of the strontium will accumulate in bone. In adults, strontium commonly attaches to the surfaces of bones. In children, whose bones are still growing, strontium may be used by the body to create the hard bone mineral itself. As a result the strontium will be stored in the bone for extended periods of time (years). Strontium is eliminated from the body through urine, feces, and sweat. Elimination through urine may occur over long periods when small amounts of strontium are released from bones [3].

As a pure beta emitter all of the energy from the decay process will be deposited within a few millimeters of the source. The long biological half-life of more than 10,000 days will result in a chronic exposure to the surrounding tissue. Possible health effects include a reduction of the number of red blood cells, which may lead to anemia. A reduction in platelets which could cause increased bleeding, especially in the intestine and a lower white blood cell count reducing the body's ability to resist infection and disease. These serious health risks necessitate a reliable, rapid method of urine analysis to monitor anyone who may be exposed to this isotope. It is vital that a method be devised whereby a large population could be rapidly screened for Sr intake following a nuclear event.

The traditional methods for the analysis of Sr utilize Sr-Spec™ resin columns or cartridges to isolate elemental Sr. Losses can occur during the purification process so it is important to know how much strontium was lost during the process. This loss is described as the Sr recovery and is taken into account during the quantification process for the sample. The Sr recovery during the purification process can be determined by several different methods: ^{85}Sr spike (quantified by gamma spectroscopy), ^{85}Sr spike (quantified by Liquid Scintillation), or stable strontium nitrate carrier (quantified by drying and gravitation method).

Objective

The objective of the study was to determine if variations of traditional Sr analysis methods could be used to decrease the overall turnaround time for sample results, while maximizing Sr recovery. The following recovery methods were investigated (1) stable Sr carrier, (2) ^{85}Sr by gamma spectroscopy, and (3) ^{85}Sr by LSC.

EXPERIMENT

For all the following analysis the ^{90}Y is removed from the ^{90}Sr sample. If a value for ^{90}Y or ^{89}Sr is required, the activity can be determined by reprocessing the Sr-spec columns at a later date. The initial activity reported is for ^{90}Sr only, which enhances the speed of analysis.
A stock urine solution was divided into three parts and was spiked with differing activity levels (low, medium, and high) of ^{239}Pu, ^{137}Cs, and $^{90}Sr/^{90}Y$ and are presented in the Table 1.

Table 1: Stock Urine Spike Levels

Nuclides	Level	Source Aliquot (mL)	Stock Urine (mL)	Total (mL)	Specific Activity (Bq/mL)	Sample Activity (Bq/5mL)
^{239}Pu	Low	0.13	20.28	20.56	1.09	5.45
^{239}Pu	Medium	0.11	19.33	20.73	4.10	20.49
^{239}Pu	High	0.52	17.33	20.40	19.51	97.55
^{137}Cs	Low	0.04	20.28	20.56	0.35	1.77
^{137}Cs	Medium	0.22	19.33	20.73	1.96	9.80
^{137}Cs	High	0.44	17.33	20.40	3.91	19.53
$^{90}Sr/^{90}Y$	Low	0.11	20.28	20.56	1.01	5.03
$^{90}Sr/^{90}Y$	Medium	1.06	19.33	20.73	9.76	48.78
$^{90}Sr/^{90}Y$	High	2.10	17.33	20.40	19.26	96.32

From the three spiked urine stock solutions four sets of samples were created. Each set contained a sample from each of the three spiked urine stock solutions as well as a blank sample. For each created sample set, an internal radioactive tracer or stable carrier was added to the 5ml aliquot of stock urine solution with the exception of the blank sample. The four sets of samples created are described in further detail in the Table 2.

Table 2: Urine Set Description

Set #	Process Description	Internal Tracer or Stable Carrier	^{90}Sr Analysis Method	Internal Tracer or Stable Carrier Analysis Method
1	5ml Solution + Sr-85	Sr-85	LSC	Gamma Spec
2	5ml Solution + Sr carrier	Strontium Nitrate Carrier	LSC	Gravimetric Weighing
3	5ml solution + Sr-85	Sr-85	LSC	LSC
4	5ml stock solution (no tracer)	N/A	LSC	N/A

Sets #1, 2, and 3, were passed through Sr-Spec™ columns to isolate the total Sr from the sample as described by S. Maxwell [2]. The final stripping of Sr from the columns was performed with 10 mL of 0.05M HNO_3.

For Set #1: After the samples were passed through the Sr-Spec™ column, the eluent containing the ^{90}Sr were evaporated on a hotplate and volumed up with DI H_2O. Ultima Gold LLT™ was then added to the sample for analysis on the liquid scintillation counter as described by reference [4]. The samples were counted in a low energy beta (LEB)/beta/alpha mode with the beta window optimized for ^{90}Sr . The ^{85}Sr recovery was determined by two methods: 1) analyzing the samples by gamma spectroscopy and 2) counting the sample by LSC. The preparation process took approximately 4 hours to complete.

For Set #2: After the samples were passed through the Sr-Spec™ column, the eluent containing the ^{90}Sr were evaporated on a hotplate and gravimetrically weighed to determine the Sr recovery from the addition of the $Sr(NO_3)_2$ carrier. The samples were then volumed up with DI H_2O. Ultima Gold LLT™ was then added to the DI H_2O solution and was analyzed on the liquid scintillation counter as described by reference [5]. The preparation process took approximately 3.5 hours to complete.

For Set #3: After the samples were passed through the Sr-Spec™ column the eluent containing the ^{90}Sr were neutralized with NH_4OH and divided in half (to reduce the quenching effects for LSC). Ultima Gold LLT™ was then added to the samples for analysis by LSC. The ^{85}Sr recovery was then calculated by two methods: 1) using the results from the low energy beta window on the liquid scintillation analysis, and 2) counting the sample by gamma spectroscopy. The preparation process took approximately 6 hours to complete.

Set #4 was the control set. There was no internal tracer added to the samples. Ultima Gold LLT™ was then added to the samples for analysis by LSC using a generic LEB/beta/alpha protocol for Ultima Gold XR™.

Table 3 describes the counting conditions used for the liquid scintillation analyses.

Table 3: LSC count conditions

Set #	Cocktail	Analysis Time (min)	Aliquot Size (mL)
1	Ultima Gold LLT	10	10 + 10 cocktail
2	Ultima Gold LLT	10	10 + 10 cocktail
3	Ultima Gold LLT	10	5 + 15 cocktail

*Discriminator setting=148, LEB/beta/alpha window ranges 0-55, 55-2000, 20-600, respectively.

RESULTS AND DISCUSSION

Set #4 samples were prepared by taking the 5mL of the urine stock solution and adding 15mL of Ultima Gold LLT™. The samples were then counted on a generic LEB/gross alpha/gross beta protocol that was calibrated for the cocktail Ultima Gold XR to determine if there were any significant differences between the calibrations for differing cocktails. The results are presented in Table 4 below.

Table 4: Set#4 Direct Count Results for Baseline Values

Sample ID	Aliquot (mL)	tSIE	Gross Beta Observed (Bq/mL)	Gross alpha Observed (Bq/mL)	Beta Known Spike Value (Bq/ml)	Alpha Known Spike Value (Bq/mL)
Low	5	190	2.98	0.64	2.37	1.09
Medium	5	188	24.6	3.14	21.47	4.10
High	5	206	49.80	17.72	41.46	19.51
Blank	5	201	0.022	-0.0048	N/A	N/A

*Discriminator setting=119, low level beta/beta/alpha window ranges 0-12, 12-2000, 20-600, respectively.

Analysis of prepared urine spikes without any carrier for gross alpha/beta by LSC showed a reasonable correlation between the observed and known values. The gross beta, and gross alpha biases could be a result of the discriminator setting being optimized for Ultima Gold XR while the samples contained Ultima Gold LLT™.

The dried samples from Set #1 were placed in solution with 10 mL of DI H_2O and homogenized with 10 mL of Ultima Gold LLT™ cocktail. The samples were then analyzed on a Packard LSC unit using the ^{85}Sr LEB/beta/ alpha protocol with the beta window optimized for Sr. The samples were then analyzed on an HPGe gamma detector to calculate the ^{85}Sr recovery. The results are presented below in Table 5.

Table 5: Results Set#1 (^{85}Sr tracer recovery by gamma spectroscopy)

Sample ID	^{85}Sr Activity (Bq)	^{85}Sr recovery by gamma	Sr85LEB Activity (Bq)	Alpha Activity (Bq)	Beta Activity (Bq)	^{90}Sr Target Activity (Bq)	^{90}Sr Recovery (%)	Bias
Low	18	0.9	22.6	2.84	5.35	5.04	106.1	-0.061
Medium	15	0.89	62.7	14.0	40.14	48.78	82.29	0.177
High	15	0.95	111.00	70.2	87.55	96.32	90.89	0.091
Blank	15	0.93	15.0	0.048	0.63	N/A	N/A	N/A

The SNL method [5] was successful for the analysis of Sr. However, there was alpha activity present in the gross alpha window. It was also decided to compare the activity found in the LEB window to the quantities of known ^{85}Sr internal tracer added for the determination of tracer recovery. The results are shown in Table 6.

Table 6: Results Set#1 (^{85}Sr tracer recovery by LSC)

Sample ID	^{85}Sr Activity Bq	^{85}Sr recovery by LSC	Sr85LEB Activity (Bq)	Alpha Activity (Bq)	Beta Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low	18	1.26	22.6	2.84	3.4	5.04	67.46	0.325
Medium	15	4.18	62.7	14.0	8.64	48.78	17.71	0.823
High	15	7.40	111.00	70.2	11.36	96.32	11.79	0.882
Blank	15	1.00	15.0	0.048	0.59	N/A	N/A	N/A

*Sr85LEB/Alpha/Beta protocol - Gross Beta window optimized for ^{90}Sr

This experiment demonstrated that the gamma spectrometer analysis for ^{85}Sr recovery provides reliable results (recovery 89-95%) as compared to the LSC Sr85LEB analysis for ^{85}Sr (recovery 120-470%). The ^{90}Sr results for Set #1 samples are close to the target results when the gamma spectrometer analysis was used for ^{85}Sr recovery. The bias ranged from -6% to 18%. The high recovery for ^{85}Sr by LSC may be due to the interference/cross talk from the alpha region since alpha counts were observed. Also, the sample set was able to be prepared in a relatively short period of time (4 hours).

Set #2 was carefully evaporated on the hot plate for about 2 hours. The recovery of stable strontium nitrate $Sr(NO_3)_2$ was calculated using gravimetric analysis. Ten (10) ml of DI H_2O was then added to the dry samples and homogenized with 10 ml of Ultima Gold LLT™ cocktail, and counted on the LSC for Sr using the DPM mode specifically calibrated for ^{90}Sr . The results are presented in the Table 7.

Table 7: Results Set#2 (Stable Sr carrier recovery by gravimetric analysis)

Sample ID	$Sr(NO_3)_2$ recovery	^{90}Sr Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low	0.992	5.98	5.04	118.65	-0.187
Medium	0.967	50.85	48.78	107.24	-0.042
High	1.008	170.02	96.32	176.52	-0.765
Blank	0.992	-0.10	N/A	N/A	N/A

*counted in Sr90DPM mode

The stable strontium nitrate $Sr(NO_3)_2$ carrier samples demonstrate that gravimetric analysis yields consistent high recoveries (97-101%) when evaporation is carefully performed. The high recovery for ^{90}Sr by LSC using the Sr90DPM mode were not valid due to the interference/cross talk from the ^{239}Pu signal with ^{90}Sr signal. Since there is no discrimination performed in the DPM mode between alpha and beta signals the DPM mode is not optimal for samples with the presence of alpha interferences.

The samples were then counted in the LEB/Alpha/Mode optimized for ^{90}Sr . The results are presented in Table 8.

Table 8: Results Set#2 (Stable Sr carrier recovery)

Sample ID	$Sr(NO_3)_2$ Recovery	LEB Activity (Bq)	Alpha Activity (Bq)	Beta Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low	0.992	5.51	1.34	4.28	5.04	84.9	0.15
Medium	0.967	51.10	10.2	38.72	48.78	79.4	0.21
High	1.008	106.00	62.4	100.03	96.32	104	-0.04
Blank	0.992	0.0392	-0.009	0.01	N/A	N/A	N/A

*gravimetric analysis – counted in Sr85LEB/Alpha/Beta mode

The results demonstrate that the analysis was successful for ^{90}Sr with recoveries ranging from 79-104%. As with Set #1 results there appears to be a small amount of alpha activity present.

Set#2 samples were then recounted using the generic LEB/Alpha/Beta protocol which was not optimized for Sr and was calibrated with Ultima Gold XR™ cocktail. The results are shown in Table 9.

Table 9: Results Set#2 (Stable Sr carrier recovery by gravimetric analysis)

Sample ID	$Sr(NO_3)_2$ recovery	LEB Activity (Bq)	Alpha Activity (Bq)	^{90}Sr Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low	0.992	2.43	2.33	4.35	5.04	86.3	0.14
Medium	0.967	30.80	15.5	41.92	48.78	85.9	0.14
High	1.008	47.4	92.8	86.09	96.32	89.4	-0.12
Blank	0.992	-0.0859	-0.0269	0.05	N/A	N/A	N/A

*counted in LEB/Alpha/Beta mode XR Calibration

The results from Table 9 counted by the generic LSC LEB/Alpha/ Beta protocol demonstrates more consistency in the ^{90}Sr recovery (86% to 89%) This counting protocol was more effective in discriminating the alpha signals from the beta signals producing a more consistent set of results. Samples prepared by this method resulted in the shortest amount of preparation time (3.5 hours).

Set #3 was neutralized using NH_4OH. The sample was split in half to avoid quenching with the LLT cocktail which produced twice as many samples. Each sample was homogenized with 15mL of Ultima Gold LLT™ cocktail and counted by LSC using the LEB/Alpha/ Beta protocol optimized for Sr. Simultaneously the samples were counted by gamma spectrometry to obtain the ^{85}Sr recovery. Results are presented in Table 10.

Table 10: Results Set#3 LEB/Alpha/Beta protocol

Sample ID	^{85}Sr Activity (Bq)	^{85}Sr Recovery	LEB Activity (Bq)	Alpha Activity (Bq)	^{90}Sr Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low A	8	0.92	7.71	0.45	4.30	5.04	85.3	0.15
Low B	8	0.68	7.14	0.41	5.34	5.04	106	0.19
Med A	8	0.78	24.3	1.56	30.65	48.78	62.8	0.37
Med B	8	0.87	25.6	1.47	28.35	48.78	58.1	0.42
High A	8	0.83	44.1	7.84	93.18	96.32	96.7	0.03
High B	8	0.67	32.9	7.50	90.67	96.32	94.1	0.06
Blank A	8	0.82	7.51	-0.02	0.55	0	N/A	N/A
Blank B	8	0.75	6.71	0.02	0.52	0	N/A	N/A

*With ^{85}Sr tracer recovery by gamma spectroscopy

The data was then analyzed using the ^{85}Sr tracer recovery using the LEB window from the LSC count. The results are presented below in Table 11. As with the previous sample sets there appeared to be a small amountof alpha activity present in the samples.

Table 11: Results Set#3 LEB/Alpha/Beta Protocol

Sample ID	^{85}Sr activity, Bq	^{85}Sr Recovery	LEB Activity (Bq)	Alpha Activity (Bq)	^{90}Sr Activity (Bq)	^{90}Sr Target (Bq)	^{90}Sr Recovery (%)	Bias
Low A	8	1.02	7.71	0.45	3.90	5.04	77.4	0.23
Low B	8	0.94	7.14	0.41	3.83	5.04	76.0	0.24
Med A	8	3.20	24.3	1.56	7.49	48.78	15.4	0.85
Med B	8	3.37	25.6	1.47	7.35	48.78	15.1	0.85
High A	8	5.90	44.1	7.84	13.07	96.32	13.6	0.86
High B	8	4.37	32.9	7.50	13.92	96.32	14.5	0.86
Blank A	8	1.00	7.51	-0.02	0.45	N/A	N/A	N/A
Blank B	8	0.89	6.71	0.02	0.44	N/A	N/A	N/A

*with Tracer Recovery by LSC

The results for Set #3 samples demonstrated reasonable results (3-42%) when the ^{85}Sr tracer recovery was calculated by using the gamma spectroscopy results. The results when calculating the tracer recovery by the LEB window in LSC was not adequate. The biases ranged from 23-86%. This was due to the alpha cross talk contribution into the LEB region which caused the LEB activity values to be incorrectly reported. Additionally using a 5mL aliquot for analysis improves the quench contribution from the sample but increased the minimum detectable activity. The MDA increase could be reduced by counting the sample for a longer period of time. Sample preparation by this method resulted in the largest sample preparation time of 6 hours.

Since there appeared to be some amount of alpha activity present in the samples it was suspected that the ^{239}Pu was not being retained on the column or rinsed out with oxalic acid, but being rinsed out with the Sr sample.

Conclusions

Two methods of calculating the ^{85}Sr tracer recovery was investigated (gamma spectroscopy and a low energy window in LSC) as well as the use of two different types of Sr tracer (^{85}Sr and Sr carrier).

Three sets of spiked urine samples were prepared; 1) Set #1 – ^{85}Sr tracer, 2) Set #2 – stable Sr carrier, and 3) Set #3 –^{85}Sr tracer. Since Sets #1 and #3 were spiked with a ^{85}Sr carrier they were analyzed by both gamma spectroscopy as well as the low energy beta (LEB) window in LSC for ^{85}Sr recovery. Set #2 Sr carrier tracer recovery was determined gravimetrically but counted using the DPM mode in LSC and also using a generic LEB/alpha/beta protocol. Set #4 was the control set and was counted in the generic LEB/alpha/beta window calibrated in Ultima Gold XR and optimized for ^{36}Cl rather than the prepared sample cocktail of Ultima Gold LLT.

Analysis of prepared urine spikes without any carrier (Set #4) for the general LEB/alpha/beta by LSC showed a very reasonable correlation between the observed and known values. The average beta bias was (-20%) while the average alpha bias was 24%. This bias could be a result of the discriminator setting being optimized for Ultima Gold XR™ cocktail and ^{36}Cl / ^{244}Cm while the samples were contained in Ultima Gold LLT™ and had ^{239}Pu /^{137}Cs. This demonstrated that when using a calibration curve comprised of a different cocktail and a non-optimum discriminator setting will still yield reasonable results.

The results from spiked samples containing ^{85}Sr demonstrated that a higher recovery is obtained when using gamma spectroscopy (89-95%) than when using the LEB window from LSC (120-470%). The high recovery for ^{85}Sr by LSC analysis may be due to the interference/cross talk from the alpha region since alpha counts were observed in all sample sets. After further investigation it was determined that the alpha counts were due to ^{239}Pu breakthrough on the Sr-spec™ column. This requires further development to purify the Sr before an accurate tracer recovery determination can be made. Sample preparation times ranged between 4-6 hours depending on the sample preparation process.

The results from the spiked samples containing stable strontium nitrate $Sr(NO_3)_2$ carrier demonstrate that gravimetric analysis yields consistently high recoveries (97-101%) when evaporation is carefully performed. Since this method did not have a variation on the tracer recovery method, the samples were counted in 1) Sr85LEB/Alpha/Beta mode optimized for ^{90}Sr, 2) DPM for ^{90}Sr, and 3) general LEB/Alpha/Beta mode. The results (from the known) ranged from 79-104%, 107-177%, and 85-89% for 1, 2, and 3 respectively. Counting the prepared samples in a generic low energy beta/alpha/beta mode yielded more accurate and consistent results and also yielded the shortest sample preparation turn-around-time of 3.5 hours.

This study finds that superior methods of Sr identification and quantification exist. Using the methodologies presented in this paper highly accurate results from difficult matrix samples can be prepared in less than four hours, allowing for a much more rapid assessment of exposure to strontium.

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