

Value Assignment of Vitamin D Metabolites in DEQAS Samples at NIST

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Brief History

- Assignment of values by the NIST Reference Measurement Procedure would allow for DEQAS to evaluate individual laboratory performance based on accuracy as opposed to consensus
- NIST began receiving DEQAS samples in October 2012 to value assign 25(OH)D₂, 25(OH)D₃, and 3-epi-25(OH)D₃

Process

- NIST receives DEQAS samples (5 per quarter) via overnight shipping
- DEQAS staff provide NIST with total 25(OH)D target values, as well as any additional information that may assist in individual value assignment (i.e., known high 25(OH)D₂ content)
- NIST performs a screening of the DEQAS materials for all 3 vitamin metabolites with NIST RMP to focus in on target values
- Based on NIST screening target values, a final set of samples is processed and analyzed for all 3 vitamin D metabolites
- All final values are detailed in an official NIST Report of Analysis and forwarded to the DEQAS staff

NIST RMP Screening

Sample Set 1

Fresh 25(OH)D2 and
25(OH)D3 calibrants
prepared



Single 1.5 mL samples
spiked with 25(OH)D2 and
25(OH)D3 stable isotope
internal standards



Samples processed
according to RMP



Duplicate analysis
of 25(OH)D3 by LC-
MS/MS on Column
1



Duplicate analysis
of 25(OH)D2 by LC-
MS/MS on Column
2

Sample Set 2

Fresh 3-epi-25(OH)D3
calibrants prepared



Single 1.5 mL samples
spiked with 3-epi-
25(OH)D3 stable isotope
internal standard



Samples processed
according to RMP



Duplicate analysis
of 3-epi-25(OH)D3
by LC-MS/MS on
Column 1

NIST RMP Final Analysis

Sample Set 1

Fresh 25(OH)D2 and
25(OH)D3 calibrants
prepared



Duplicate 2 mL samples
spiked with 25(OH)D2 and
25(OH)D3 stable isotope
internal standards



Samples processed
according to RMP



Duplicate analysis
of 25(OH)D3 by LC-
MS/MS on Column
1



Duplicate analysis
of 25(OH)D2 by LC-
MS/MS on Column
2

Sample Set 2

Fresh 3-epi-25(OH)D3
calibrants prepared



Duplicate 2 mL samples
spiked with 3-epi-
25(OH)D3 stable isotope
internal standard



Samples processed
according to RMP



Duplicate analysis
of 3-epi-25(OH)D3
by LC-MS/MS on
Column 1

Final Value Assignments

- For each of the 5 DEQAS samples, NIST delivers 4 values (duplicate LC-MS/MS analyses of duplicate preparations) for each of the 3 vitamin D metabolites in ng/g units
- Description of analyses and final data are detailed in a NIST Report of Analysis, which is delivered to DEQAS staff for use in their evaluation
- Total screening, value assignment, and reporting at NIST takes approximately 1 month

What have we delivered so far?

- NIST has successfully delivered Reports of Analysis for quarterly DEQAS samples from October 2012, January 2013, April 2013, and August 2013
 - October 2013 sample set in progress
- New staff has been trained to perform the NIST RMP to keep up with the demand for vitamin D measurements

Thanks!

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