Stability Assessment from Laboratory Control Samples

Advantages and Limitations

AAFCO Proficiency Testing Program

Laboratory Control Samples

AAFCO PT Samples repeatedly analyzed over a period of time.

Sample storage ranges across:
Controlled Ambient – Refrigeration – Freezer – -15ºC.
These are **not necessarily** Shipping or Transportation conditions.

Elapsed time between Sample delivery and 1st LCS measurements **sometimes lengthy**. Particularly true of "Quality Reference Materials" purchases.

I received LCS Time vs Value and Time vs % Recovery data for several Samples from several labs. **Thank you!**

Analytes Included: Vet Drugs, Vitamin A, Minerals, Protein, Fat and Moisture. **No alpha-tocopherol (Vit. E)!**

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What do we mean by STABILITY ??

From the IHP:

*"Materials distributed in proficiency tests must be **sufficiently stable** over the period in which the **Assigned Value is to be valid**."*

The Assigned Value is decided by Consensus over this period.

"**Sufficiently Stable**" means that Z scores are not significantly affected.

Do the Analyte values remain constant over a 12 week period?

Has the distribution unit "Sample" changed in any significant way?

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STABILITY can be a Tricky Problem!

In the usual approach a sample is split and one half, the "Control" is analyzed. Then a **period of time passes** and the second half, the "Test" is analyzed.

Our sources of variance now must include; "**Intermediate Variance**".

$$\sigma^2_{SampleResults} = \sigma^2_{Analytical} + \sigma^2_{Labs} + \sigma^2_{Sampling} + \sigma^2_{Intermediate}$$

We can control for:

- Analytical variance ($\sigma^2_{Analytical}$) by using a very precise method.
- Lab Bias (σ^2_{Labs}) by using one expert lab.
- Sampling ($\sigma^2_{Sampling}$) after a successful Homogeneity test.

Despite protestations, **significant $\sigma^2_{Intermediate}$** can be introduced over time. This may have **nothing to do with Stability** which is particularly true for "difficult" analytes.

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Using Laboratory Control Samples to Assess Stability

From ISO 13528:2015 "B.5 Assessment criteria for a stability check"

$$\left|\bar{y}_1 - \bar{y}_2\right| \leq 0.3\sigma_{pt}$$

Where:

- $\bar{y}_1$ is mean of analyte values at 0 weeks.
- $\bar{y}_2$ is mean of analyte values at 12 weeks (in our case!).
- A statistical test is recommended to detect **real differences**.
- σ_{pt} is SD chosen for proficiency testing (Horwitz).

If repeatability is suspect check to see if: $s_r > 0.5\ \sigma_{pt}$

Using 0.67 due to repeated sample handling!

Over time some intermediate variance **will interfere** with repeatability.
If s_r is greater than 2/3 σ_{pt} we have a repeatability problem.

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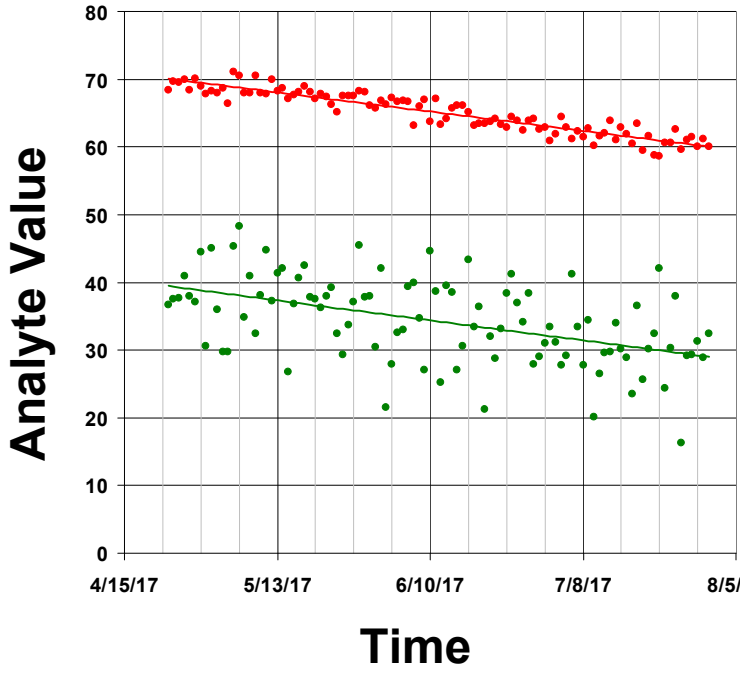
Using Laboratory Control Samples to Assess Stability

Set up a **Linear Regression** of Time vs Analyte Value

- Calculate the **Slope** of the line.
- Calculate the significance of the Slope (P).
- H_0: Slope = 0 (null hypothesis)
- If P **≤ 0.05**, there is evidence of instability.
- If P **> 0.05**, there is no evidence of instability.



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Using Laboratory Control Samples to Assess Stability

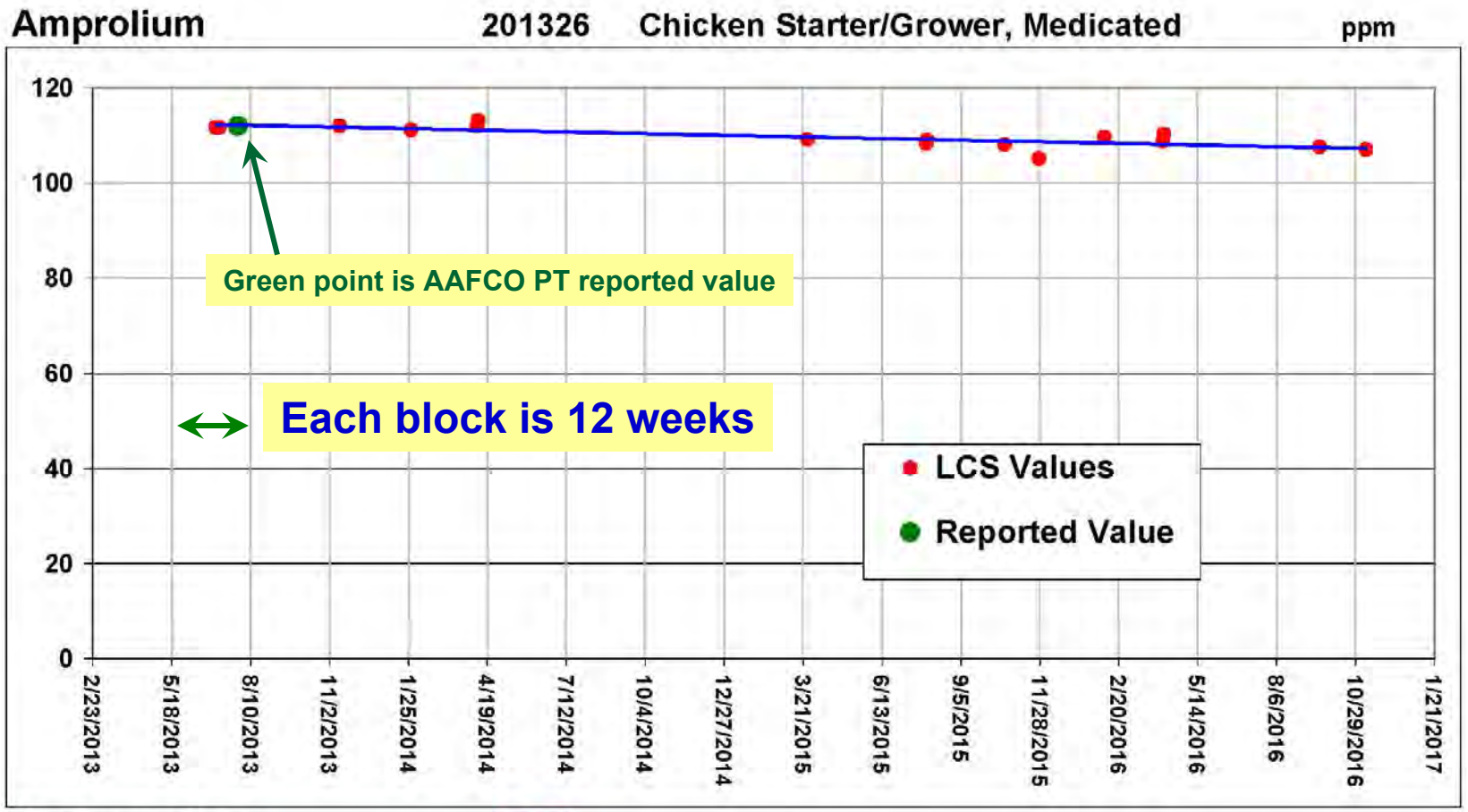
Remember!
We are only interested in the change over 12 weeks.

- If there is **NO** evidence of instability:
- Slope = 0; in our best estimate no instability predicted.
- If there is evidence of instability (Slope <> 0):
- Using the Slope calculate a **difference over 12 weeks**.
- **This is equivalent to the ISO recommendation**:

$$\left|\bar{y}_{0\,weeks} - \bar{y}_{12\,weeks}\right| \leq 0.3\sigma_{pt}$$

Does the analytical variance swamp out σ_{pt}?
Check by looking for $s_r > 0.67\ \sigma_{pt}$!

16 measurements of Amprolium (ppm) over 172 weeks!



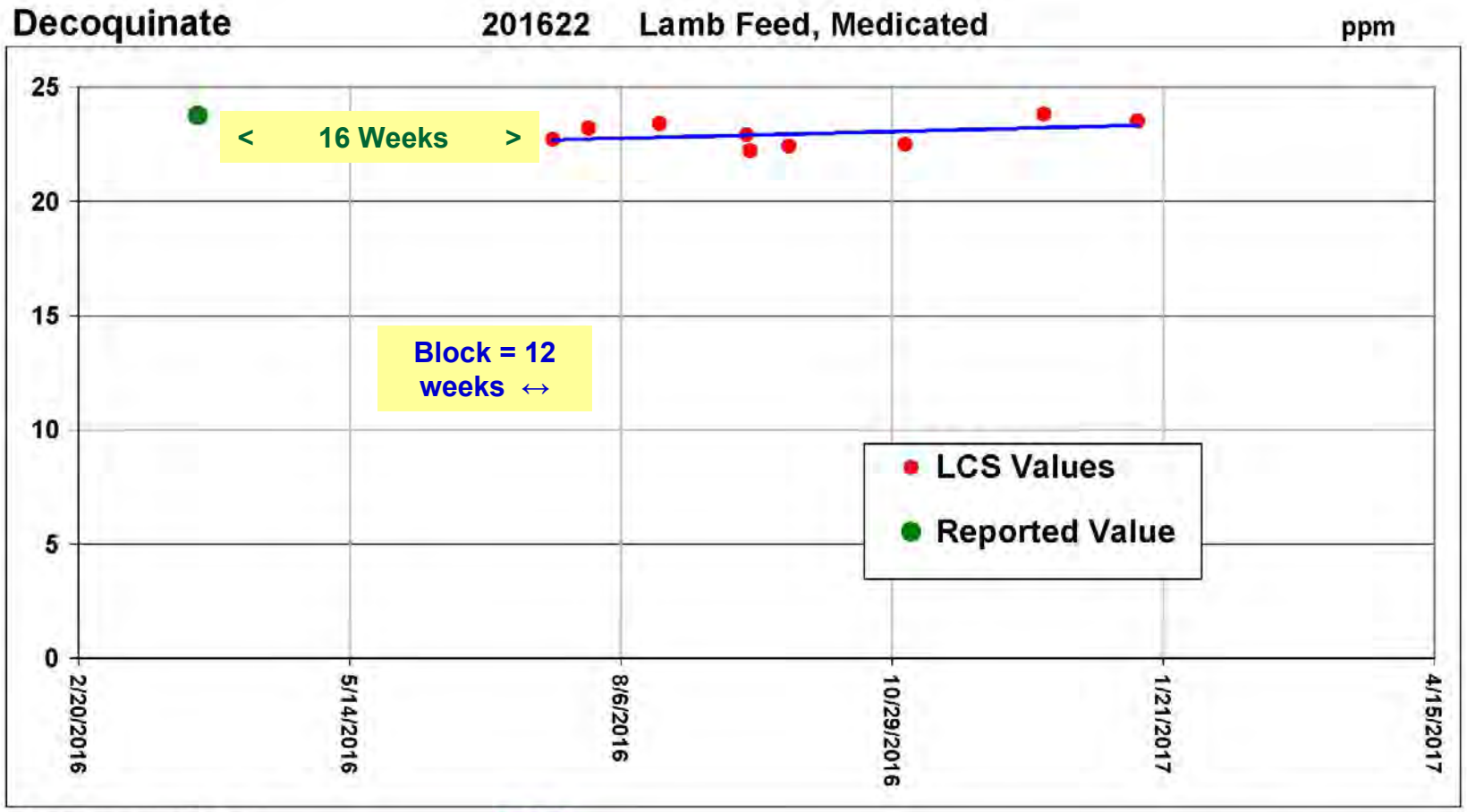
Significance of Slope	0.0002	
Evidence of Instability over 172 weeks.		
12 Week % Rel. Diff.	0.48%	
Sigma PT (%RSD)	7.89%	(Horwitz)
Allowed Var , 33%σ PT (A)	2.919	
12 Week Difference (B)	0.348	
Stability Decision, Is B < A ?	PASS Allowed Var.	

Analytical %rsd	1.26%
Analytical Variance Flag	sr OK
Should be > 1	4.21

Use SE of Y estimate

Weeks from End of Round -3

9 measurements of Decoquinate (ppm) over 25 weeks!



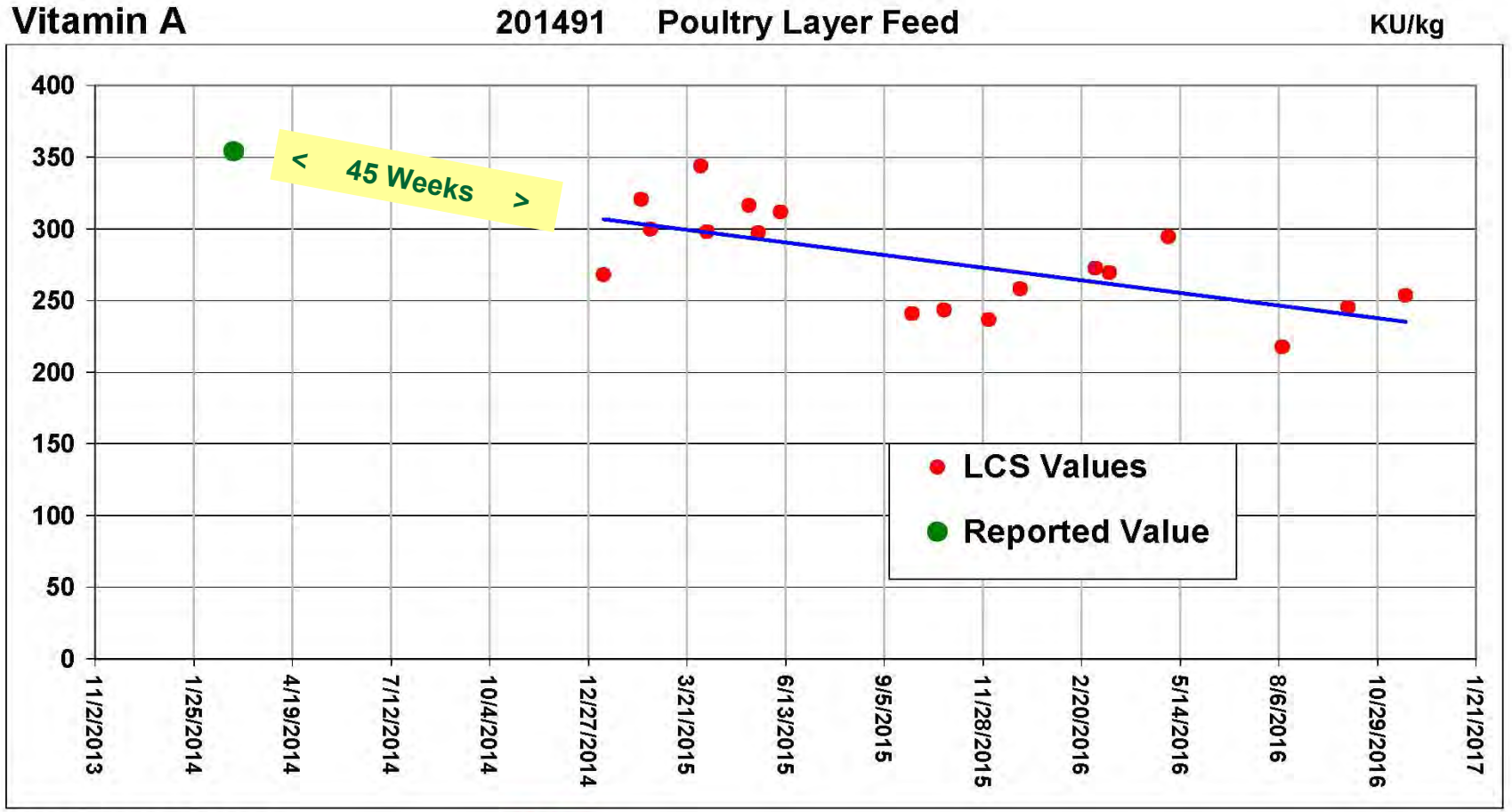
Significance of Slope	0.3040	
No Evidence of Instability over 25 weeks.		
12 Week % Rel. Diff.	NA	
Sigma PT (%RSD)	9.98%	(Horwitz)
Allowed Var , 33%σ PT (A)	0.700	
12 Week Difference (B)	0.000	
Is B < A ? YES, as NO difference seen.	PASS Allowed Var.	

Analytical %rsd	2.40%
Analytical Variance Flag	sr OK
Should be > 1	2.83

Use SD of 9 measurements

Weeks from End of Round 16

18 measurements of Vitamin A (KU/kg) over 96 weeks!

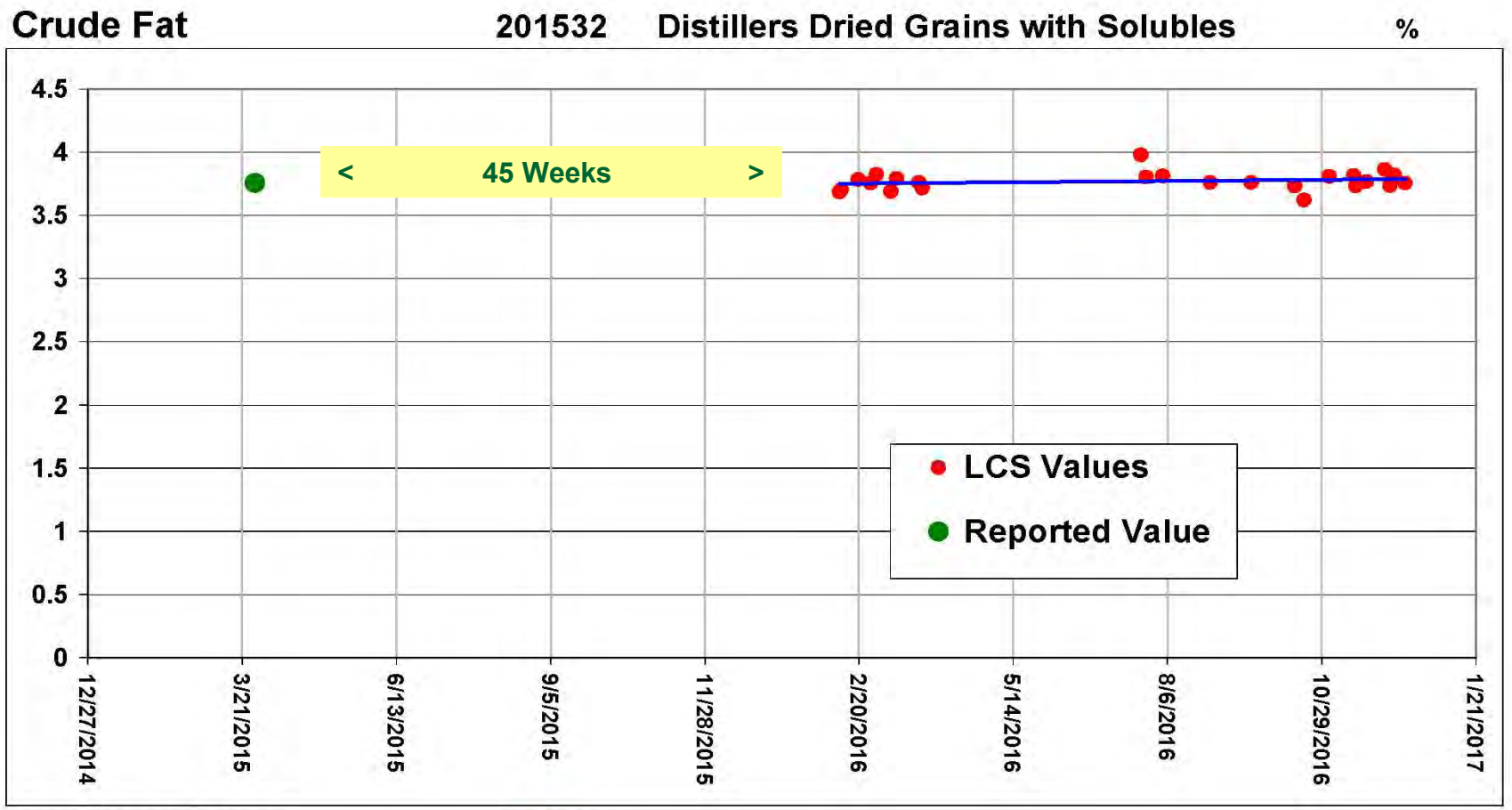


Significance of Slope	0.0030	
Evidence of Instability over 96 weeks.		
12 Week % Rel. Diff.	4.78%	
Sigma PT (%RSD)	18.56%	(Participants)
Allowed Var , 33%σ PT (A)	19.595	
12 Week Difference (B)	8.819	
Stability Decision, Is B < A?	PASS Allowed Var.	

Analytical %rsd	9.73%
Analytical Variance Flag	sr OK
Should be > 1	1.28

Important for QRM's but Not for 12 week window

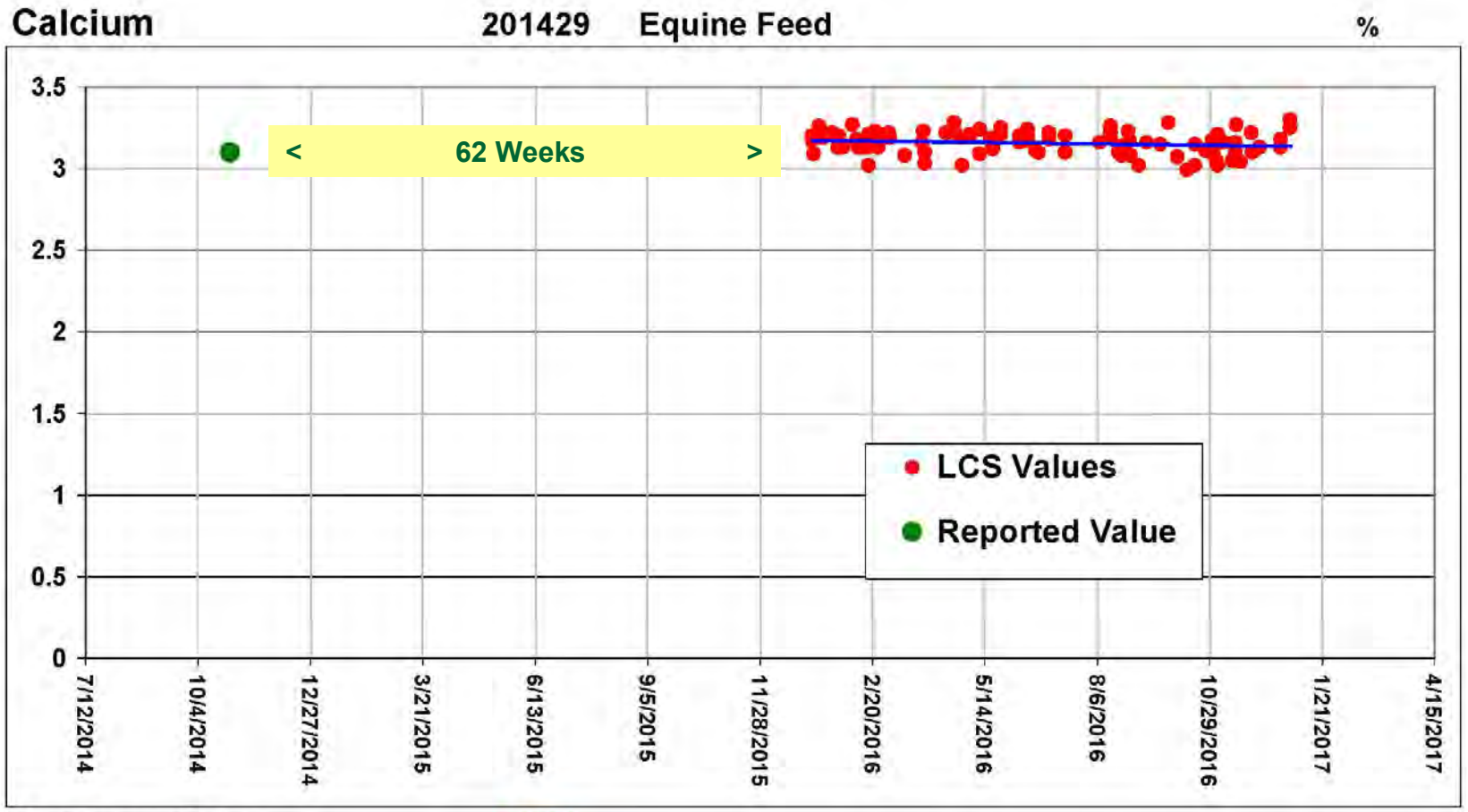
18 measurements of Crude Fat (%) over 43 weeks!



Significance of Slope	0.3503		
No Evidence of Instability over 43 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	3.28%	(Horwitz)	
Allowed Var , 33%σ PT (A)	0.038		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	1.83%
Analytical Variance Flag	sr OK
Should be > 1	2.35
Weeks from End of Round	45

18 measurements of Calcium (%) over 50 weeks!



Significance of Slope	0.0994		
No Evidence of Instability over 50 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	3.36%	(Horwitz)	
Allowed Var , 33%σ PT (A)	0.033		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	2.21%
Analytical Variance Flag	sr OK
Should be > 1	1.30
Weeks from End of Round	62

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Using LCS to Assess Stability Data Summary

Analyte	# Data Sets
Amprolium	4
Carbadox	1
Decoquinate	3
Lasalocid	2
Monensin	2
Vitamin A	10
Calcium	5
Copper	1
Magnesium	4
Potassium	1
Zinc	4
Crude Fat	2
Moj. Fat	1
Moisture	1
Protein	1

42 Data sets from 25 Samples Reviewed

- 4 Significant slopes **(P < 0.05)**
  - 1 had a positive slope??
  - 2 Vitamin A
  - 1 Amprolium
  - 1 Decoquinate
- 6 Marginally significant slopes **(0.05 <P< 0.1)**
- 38 show “No Evidence of Instability”
- 11 show “sr Suspect”
- 41 of 42 “PASSED” 12 week difference criteria.
- So 29 out of 41 “PASSED”, no decision on 13.

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Using LCS to Assess Stability Data Summary

Some Observations:

- Does not account for period between grinding and analysis (12 week window).
- Does not account for fluctuating environmental conditions (temperature, humidity, etc.).
- Advantage of many measurements.
- Exposes long term stability for QRM's.
- In many cases analytical variance not as precise as controlled study.
- Success in these studies requires excellent repeatability.

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Using LCS to Assess Stability Data Summary

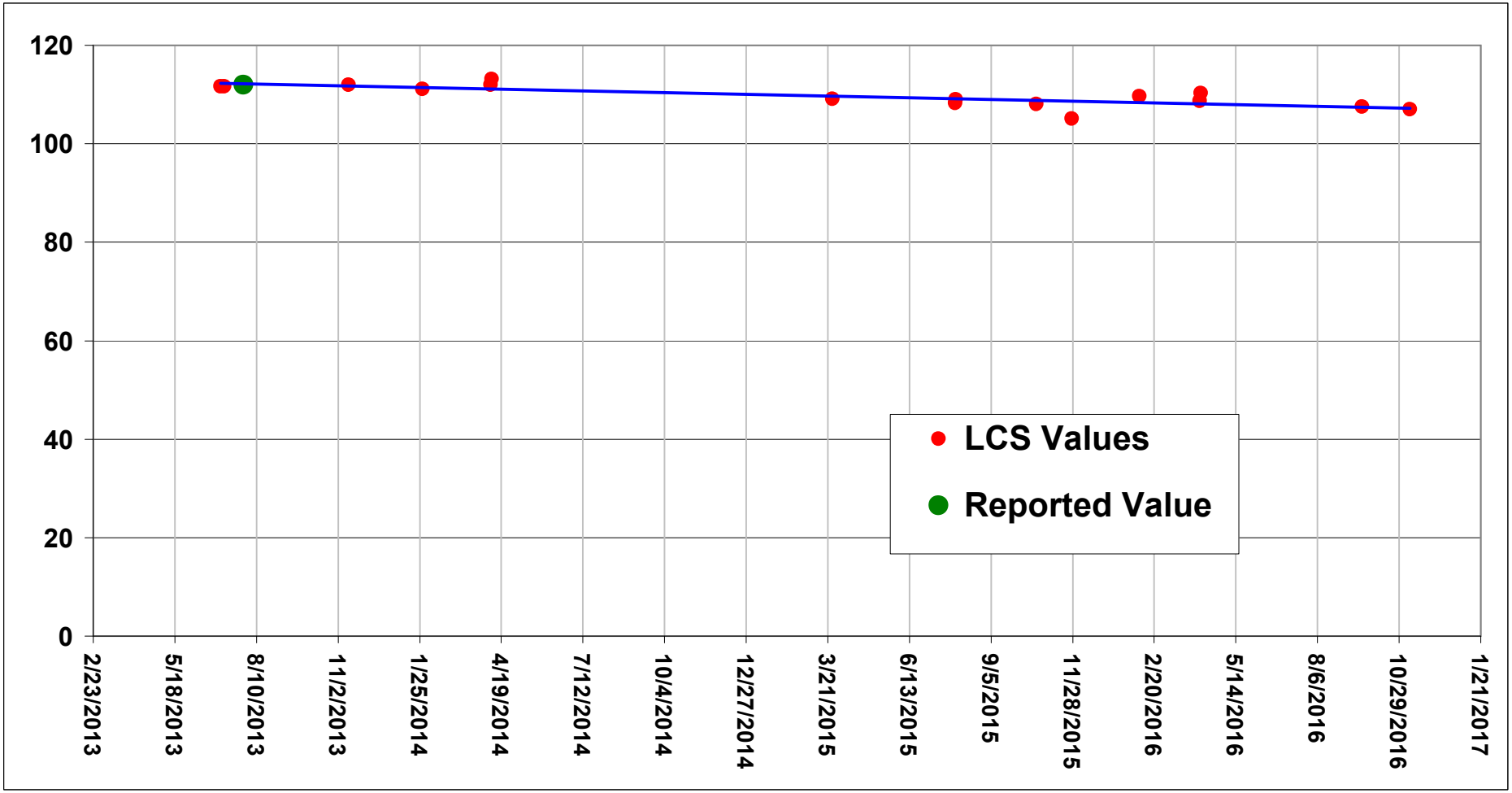
Some Thoughts:

- Unlike Homogeneity, Stability is **extremely Analyte specific**.
- The data suggests that this specificity may also be Sample specific.
- Identify Analytes of concern and direct studies at materials containing analytes of interest.

My take on this: From the reviewed data and many general observations of LCS, overall we are in good shape regarding PT sample sufficient stability.

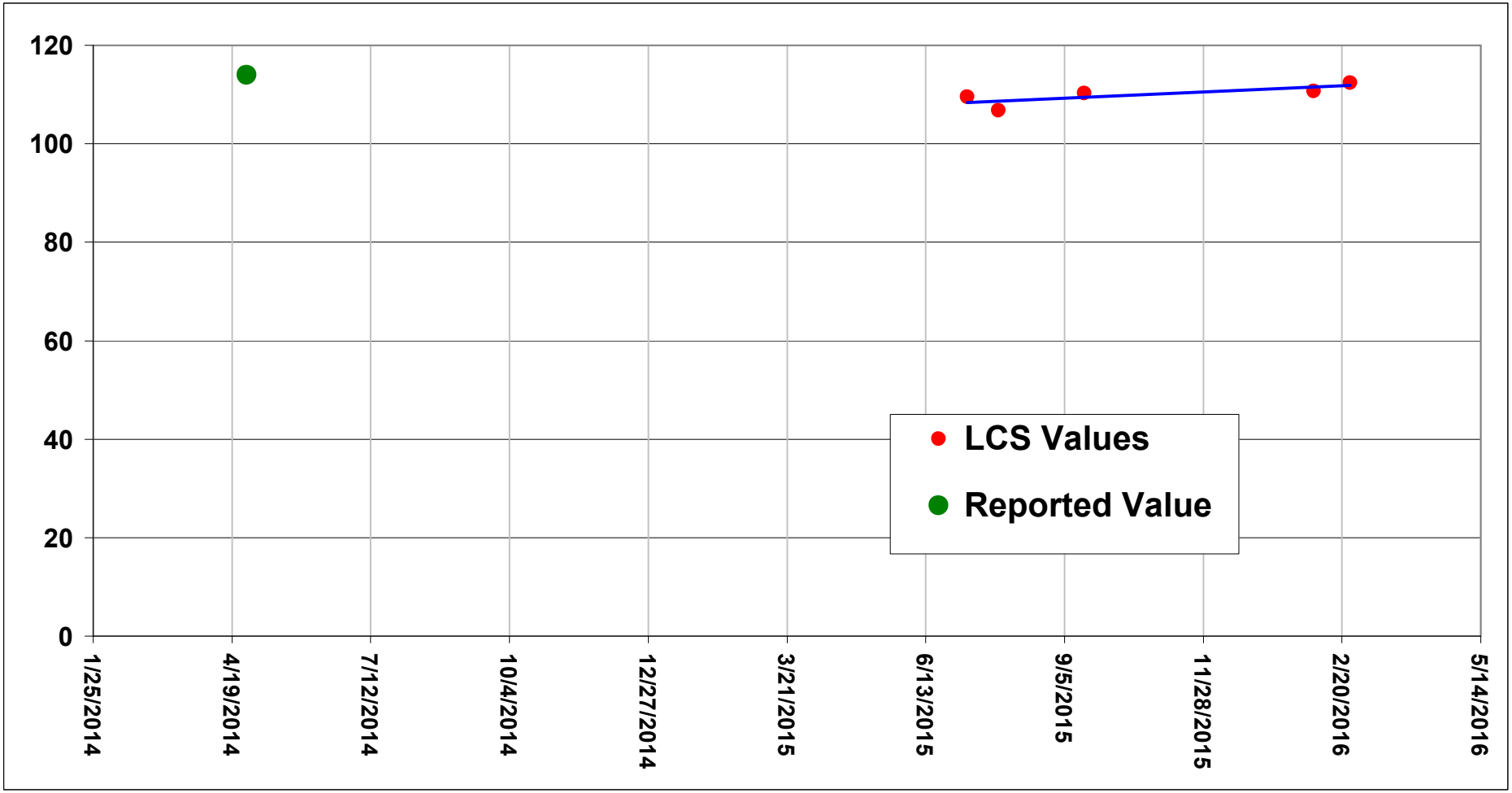

Thank you!
Can we review the additional data?

Amprolium 201326 Chicken Starter/Grower, Medicated ppm



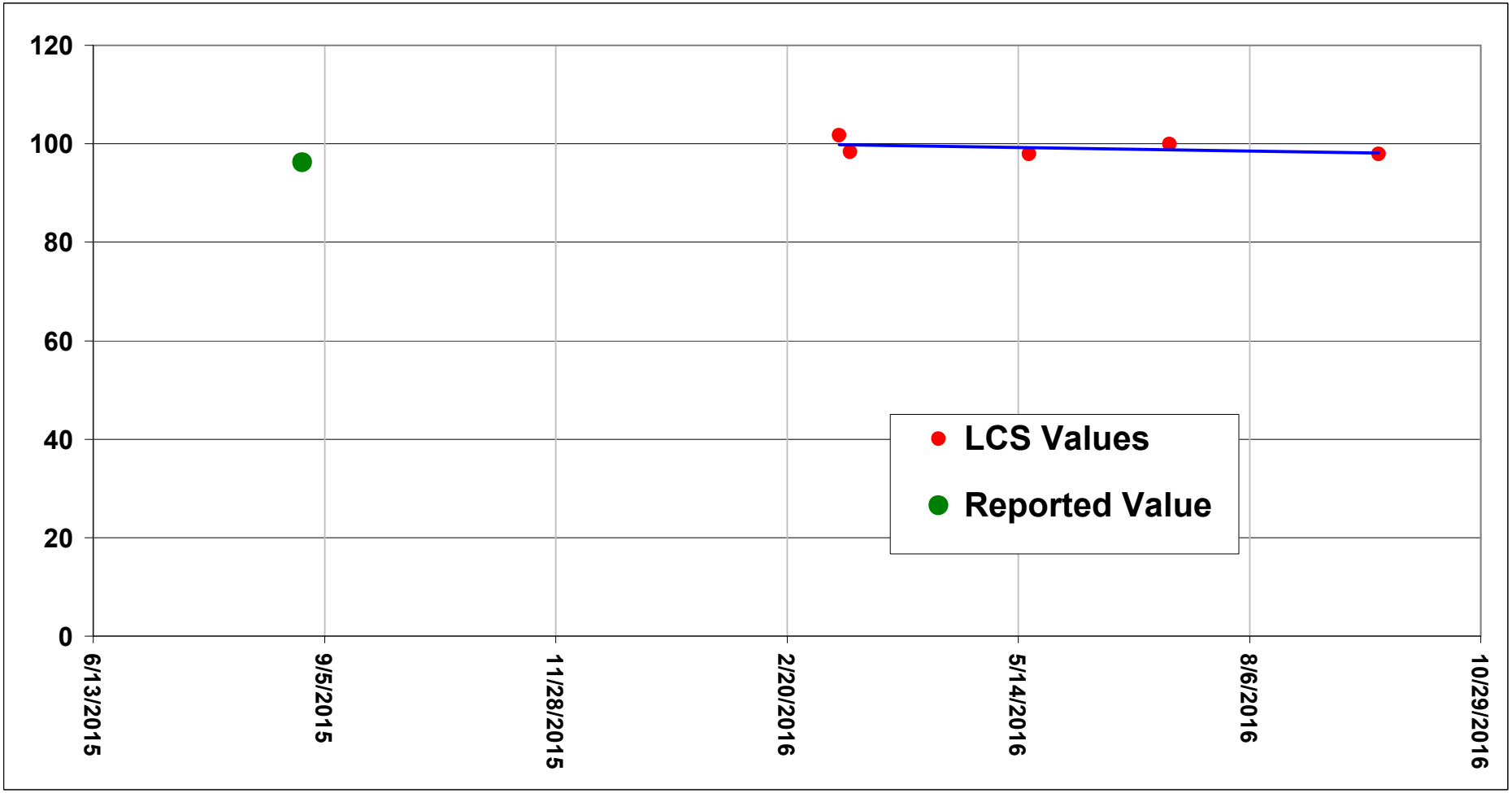
Significance of Slope	0.0002			
Evidence of Instability over 172 weeks.			Analytical %rsd	1.26%
12 Week % Rel. Diff.	0.48%		Analytical Variance Flag	sr OK
			Should be > 1	4.21
Sigma PT (%RSD)	7.89%	(Horwitz)		
Allowed Var , 33% σ PT (A)	2.919			
12 Week Difference (B)	0.348			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	-3

Amprolium 201423 Chicken Starter, Medicated ppm



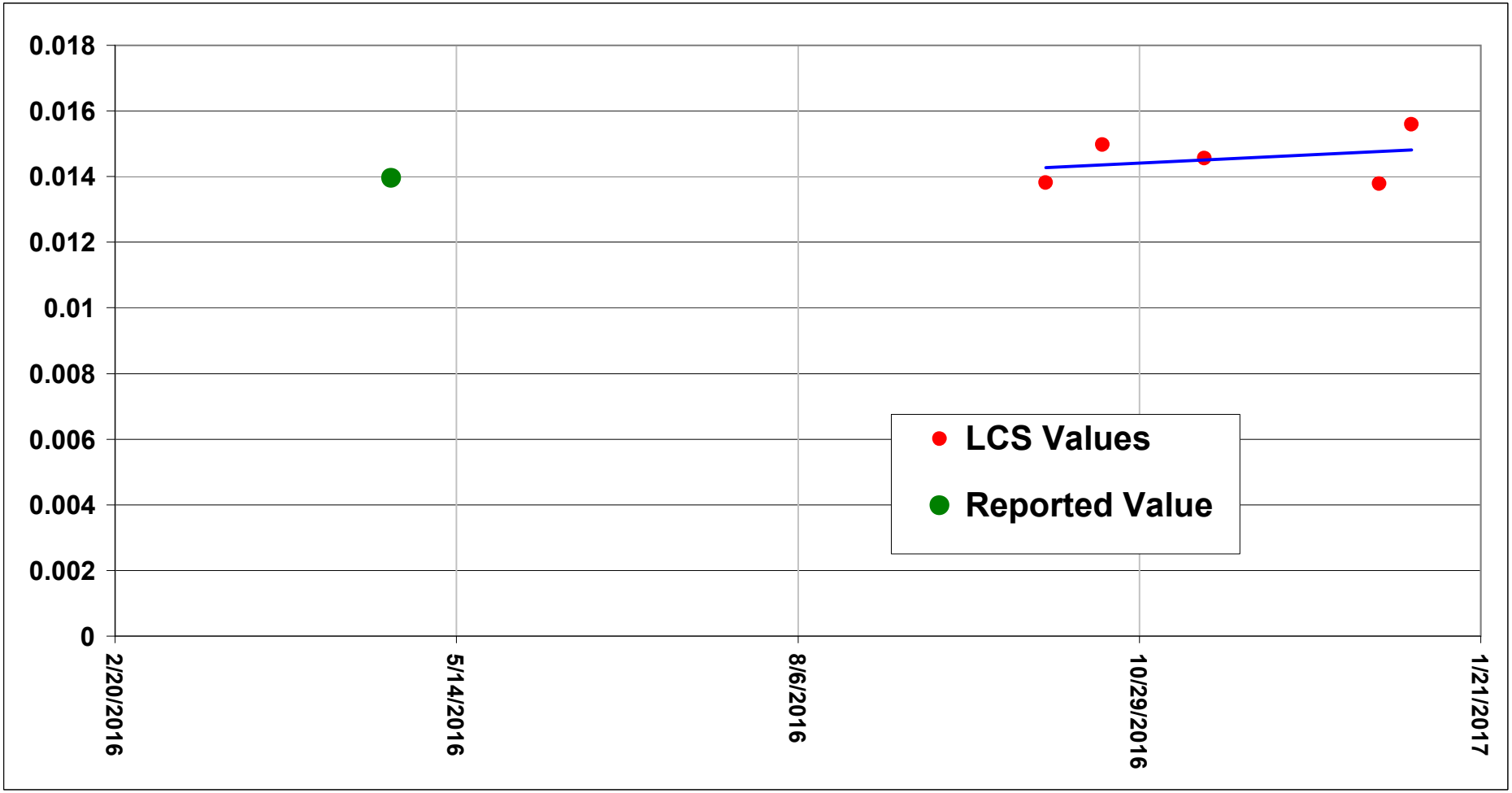
Significance of Slope	0.1155			
No Evidence of Instability over 32 weeks.				
12 Week % Rel. Diff.	NA		Analytical %rsd	1.88%
			Analytical Variance Flag	sr OK
			Should be > 1	4.04
Sigma PT (%RSD)	7.89%	(Horwitz)		
Allowed Var , 33% σ PT (A)	2.733			
12 Week Difference (B)	0.000			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	62

Amprolium 201527 Chick Starter, Medicated ppm



Significance of Slope	0.4492		
No Evidence of Instability over 27 weeks.		Analytical %rsd	1.69%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	3.07
Sigma PT (%RSD)	8.01%	(Horwitz)	
Allowed Var , 33% σ PT (A)	2.620		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	28

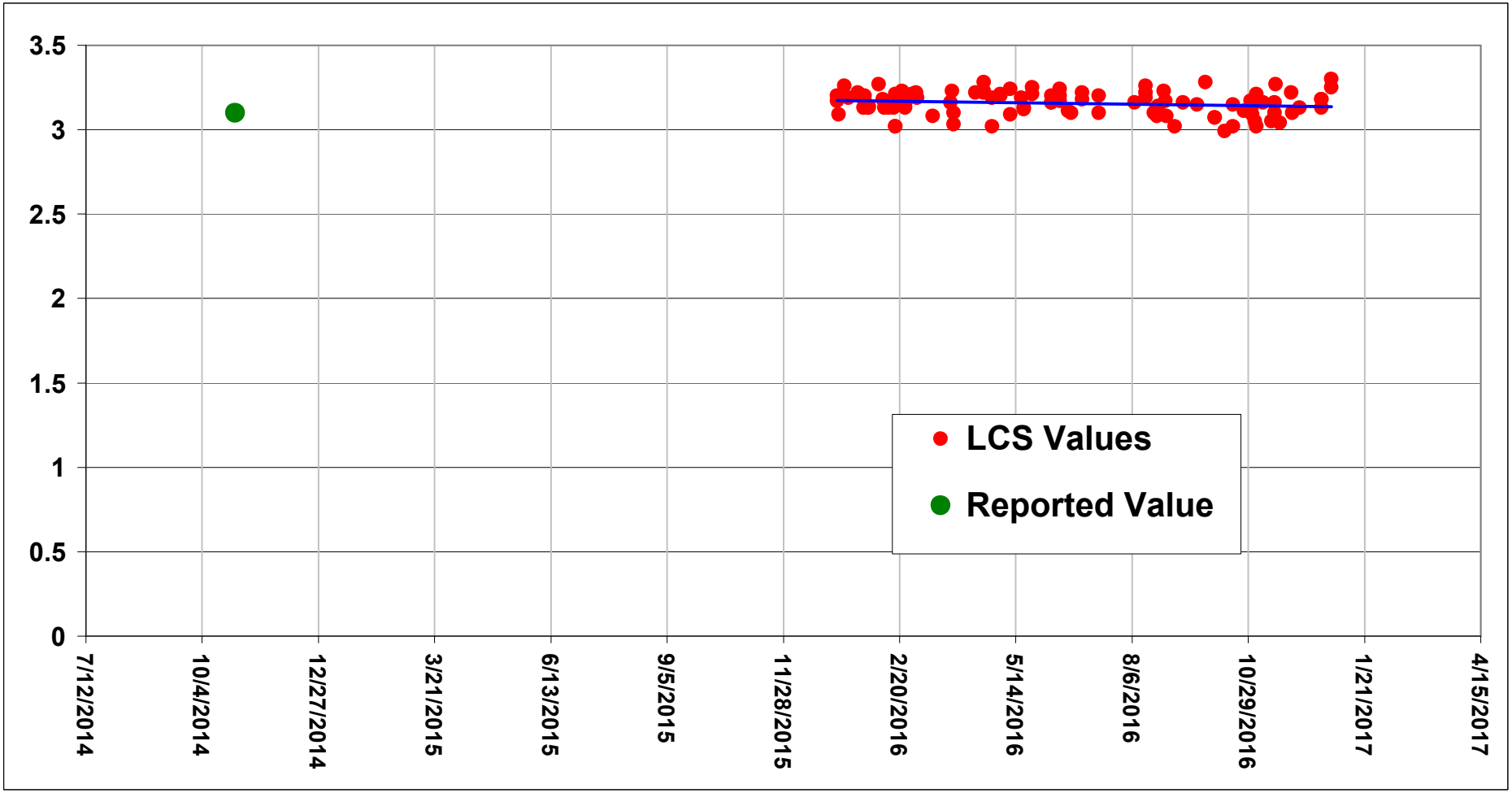
Amprolium 201623 Poultry Feed, Medicated %



Significance of Slope	0.6110		
No Evidence of Instability over 13 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	7.56%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.000		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	5.32%
Analytical Variance Flag	sr Suspect
Should be > 1	0.99
Weeks from End of Round	23

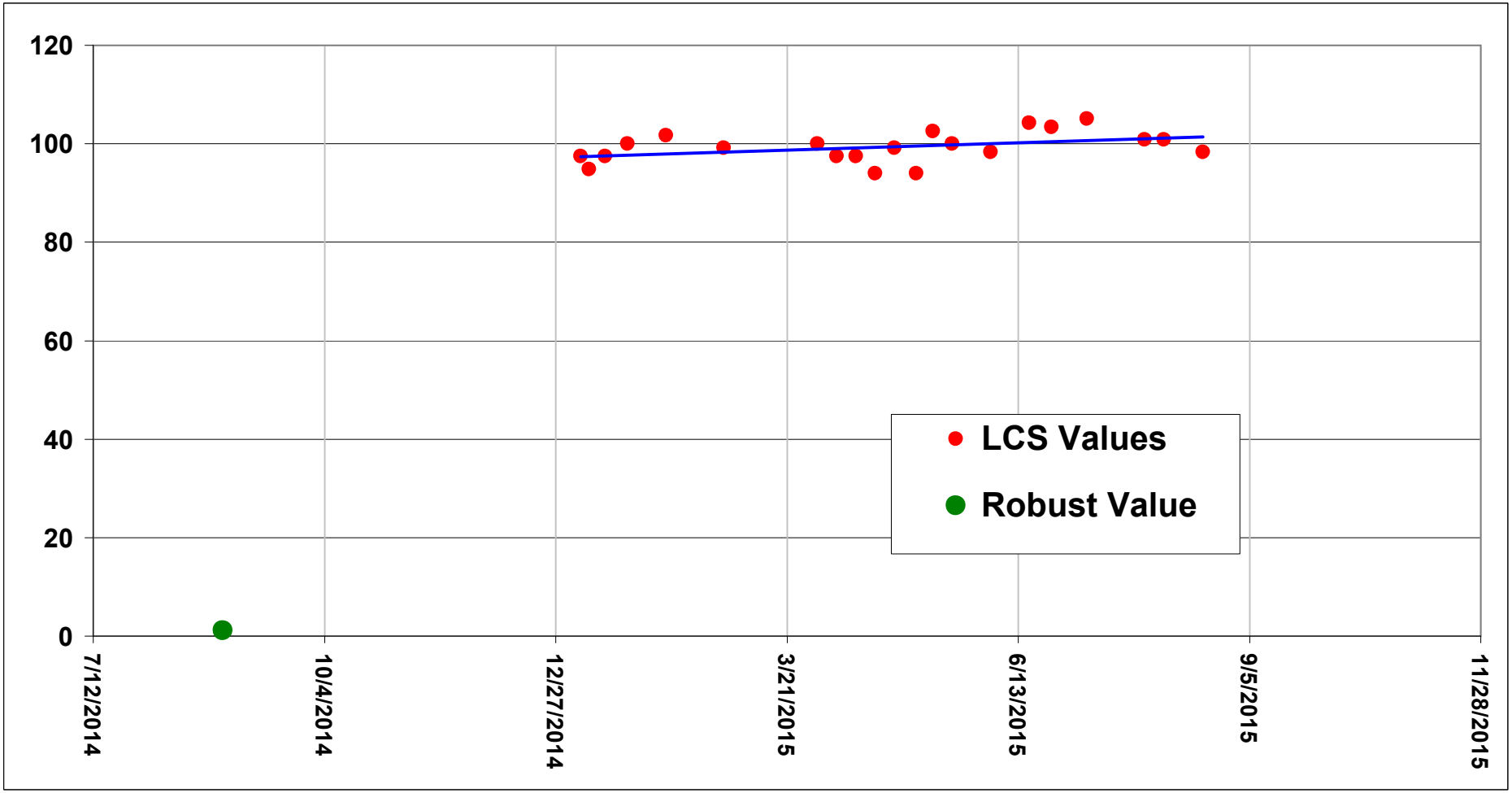
Calcium 201429 Equine Feed %



Significance of Slope	0.0994		
No Evidence of Instability over 50 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	3.36%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.033		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	2.21%
Analytical Variance Flag	sr OK
Should be > 1	1.30
Weeks from End of Round	62

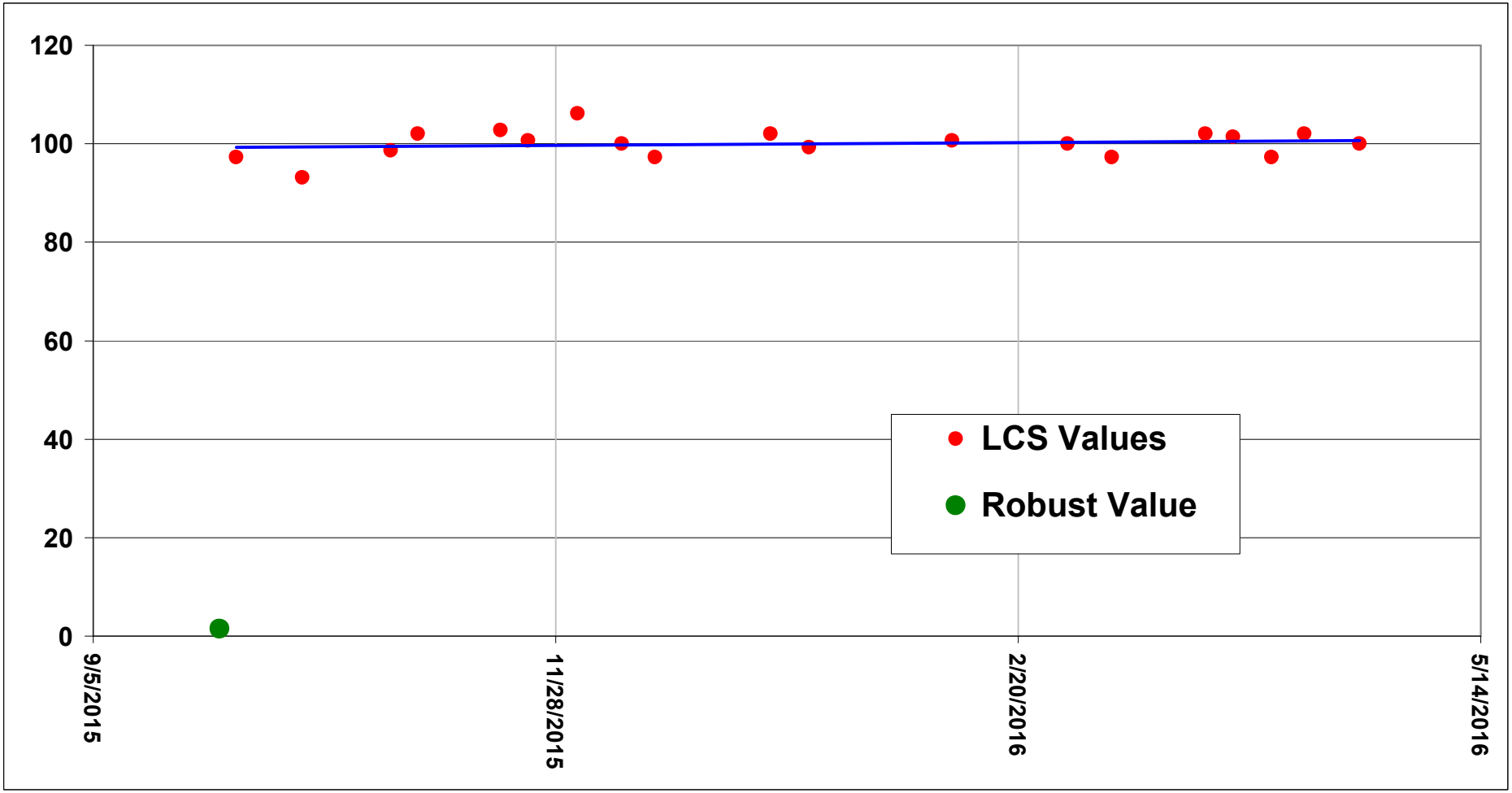
Calcium 201427 Calf Starter / Grower, Medicated % Recovery



Significance of Slope	0.0669		
No Evidence of Instability over 32 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	5.19%	(Participants)	
Allowed Var , 33% σ PT (A)	0.019		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

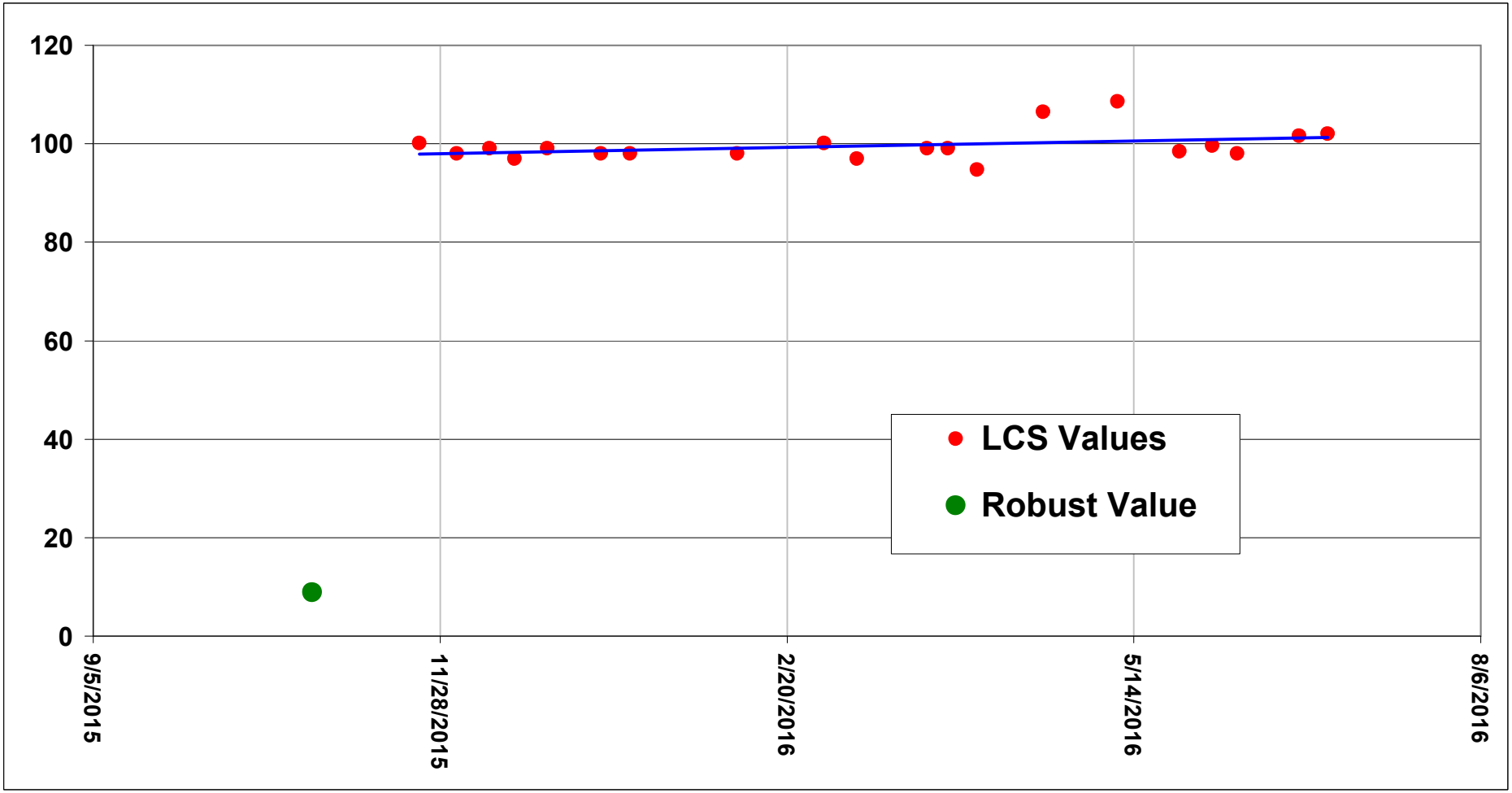
Analytical %rsd	3.10%
Analytical Variance Flag	sr OK
Should be > 1	1.20
Weeks from End of Round	19

Calcium 201528 Dog Food % Recovery



Significance of Slope	0.5023		
No Evidence of Instability over 29 weeks.		Analytical %rsd	2.81%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.15
Sigma PT (%RSD)	4.92%	(Participants)	
Allowed Var , 33% σ PT (A)	0.023		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	0

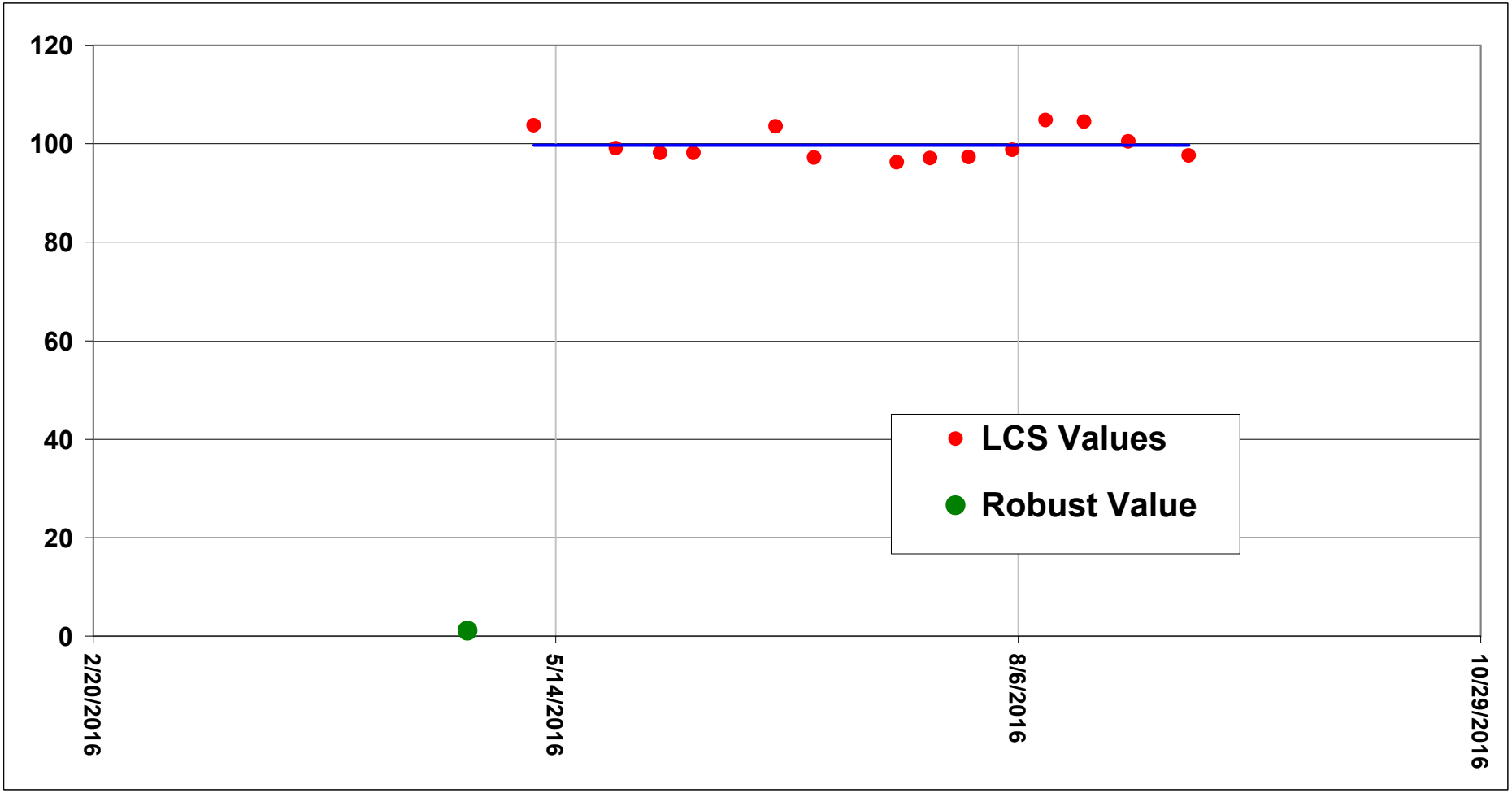
Calcium 201529 Calf Feed, Medicated % Recovery



Significance of Slope	0.1160		
No Evidence of Instability over 31 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	5.64%	(Participants)	
Allowed Var , 33% σ PT (A)	0.157		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

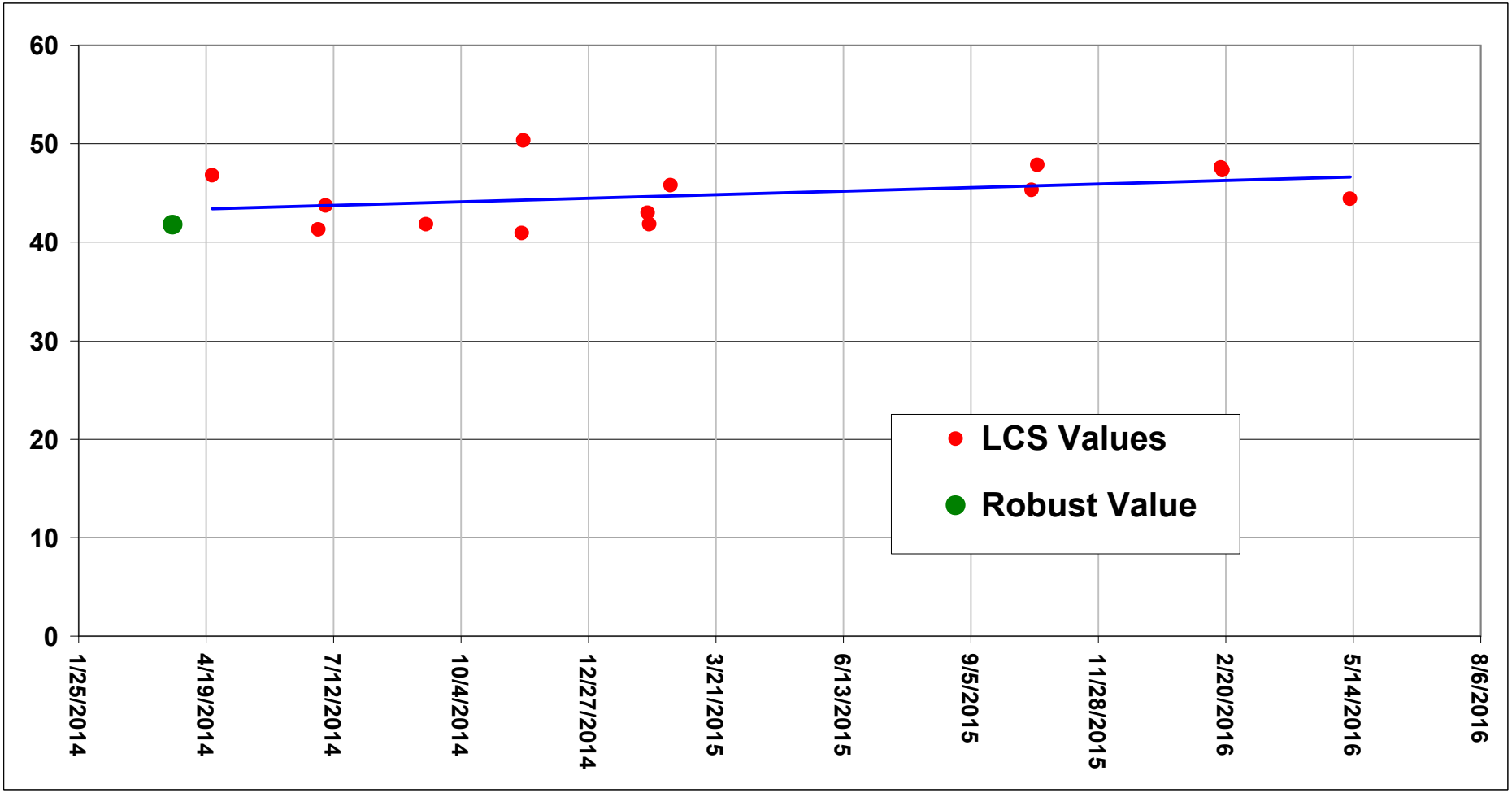
Analytical %rsd	3.20%
Analytical Variance Flag	sr OK
Should be > 1	1.23
Weeks from End of Round	4

Calcium 201623 Poultry Feed, Medicated % Recovery



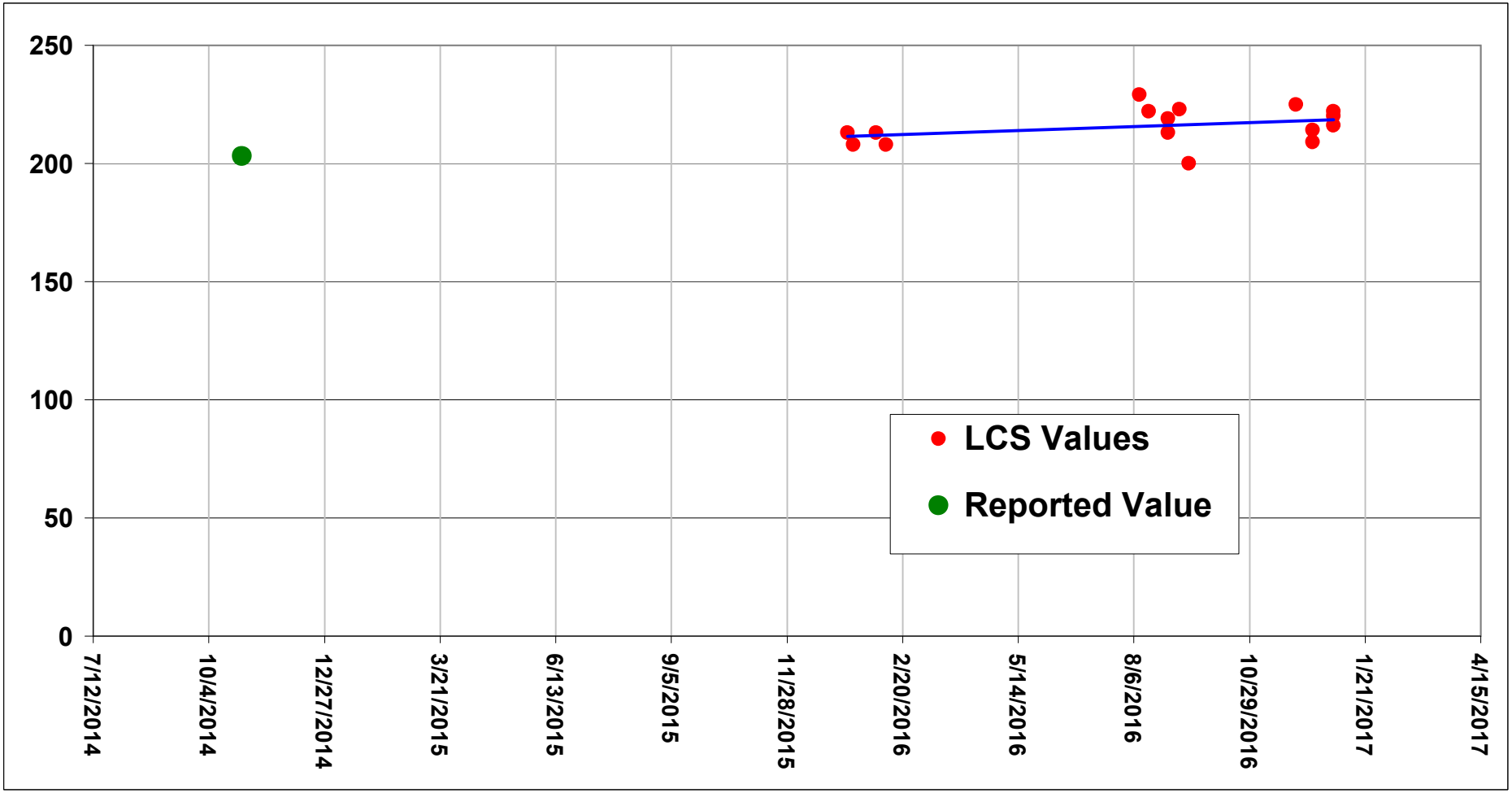
Significance of Slope	0.9966			
No Evidence of Instability over 17 weeks.			Analytical %rsd	3.07%
12 Week % Rel. Diff.	NA		Analytical Variance Flag	sr OK
			Should be > 1	1.16
Sigma PT (%RSD)	5.53%	(Participants)		
Allowed Var , 33% σ PT (A)	0.019			
12 Week Difference (B)	0.000			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	2

Carbadox 201422 Swine Grower, Medicated ppm



Significance of Slope	0.1982		
No Evidence of Instability over 106 weeks.		Analytical %rsd	6.45%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	6.03
Sigma PT (%RSD)	9.02%	(Horwitz)	
Allowed Var , 33% σ PT (A)	1.192		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	4

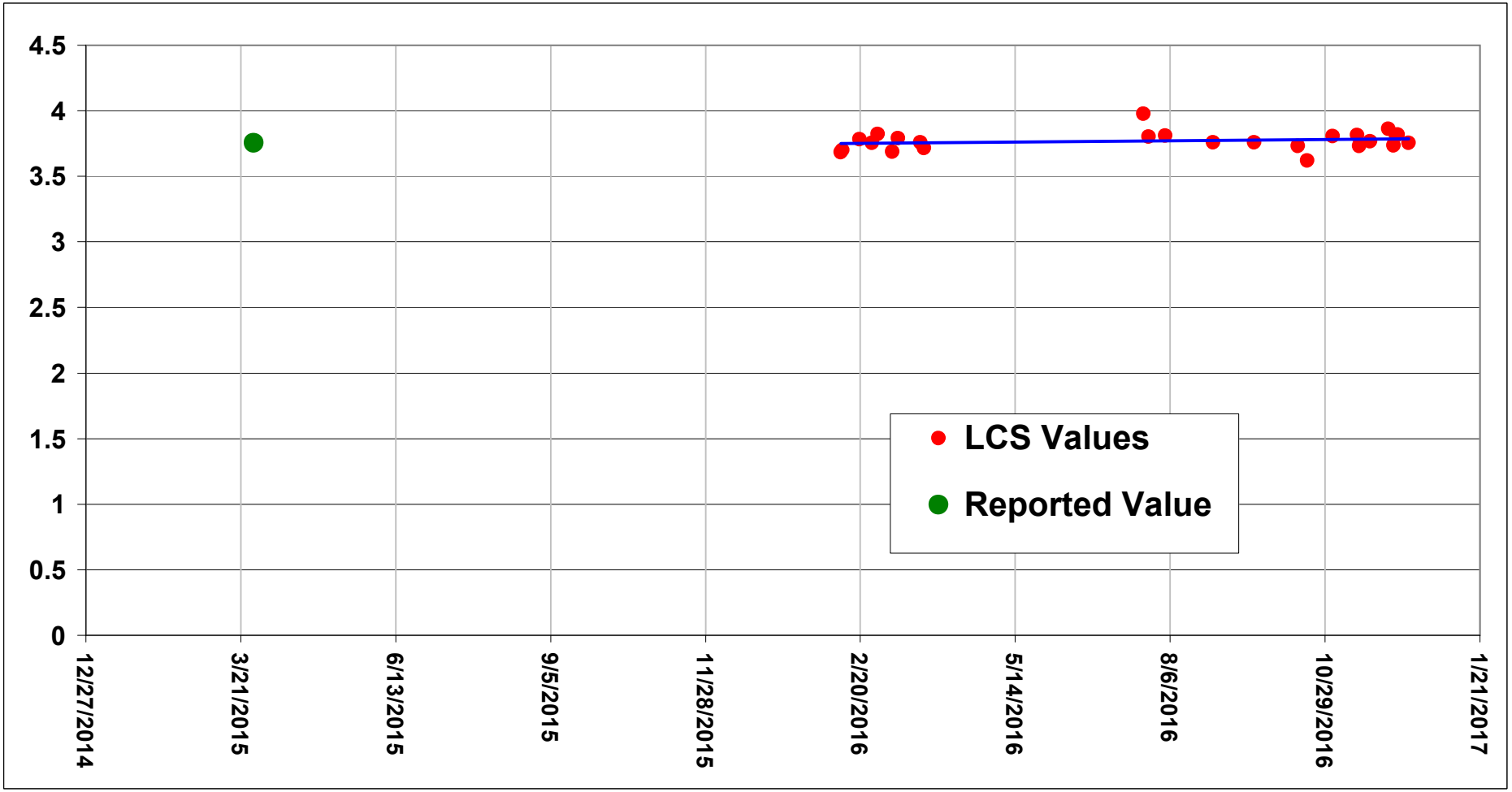
Copper 201429 Equine Feed %



Significance of Slope	0.1800		
No Evidence of Instability over 50 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	0.68%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.434		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

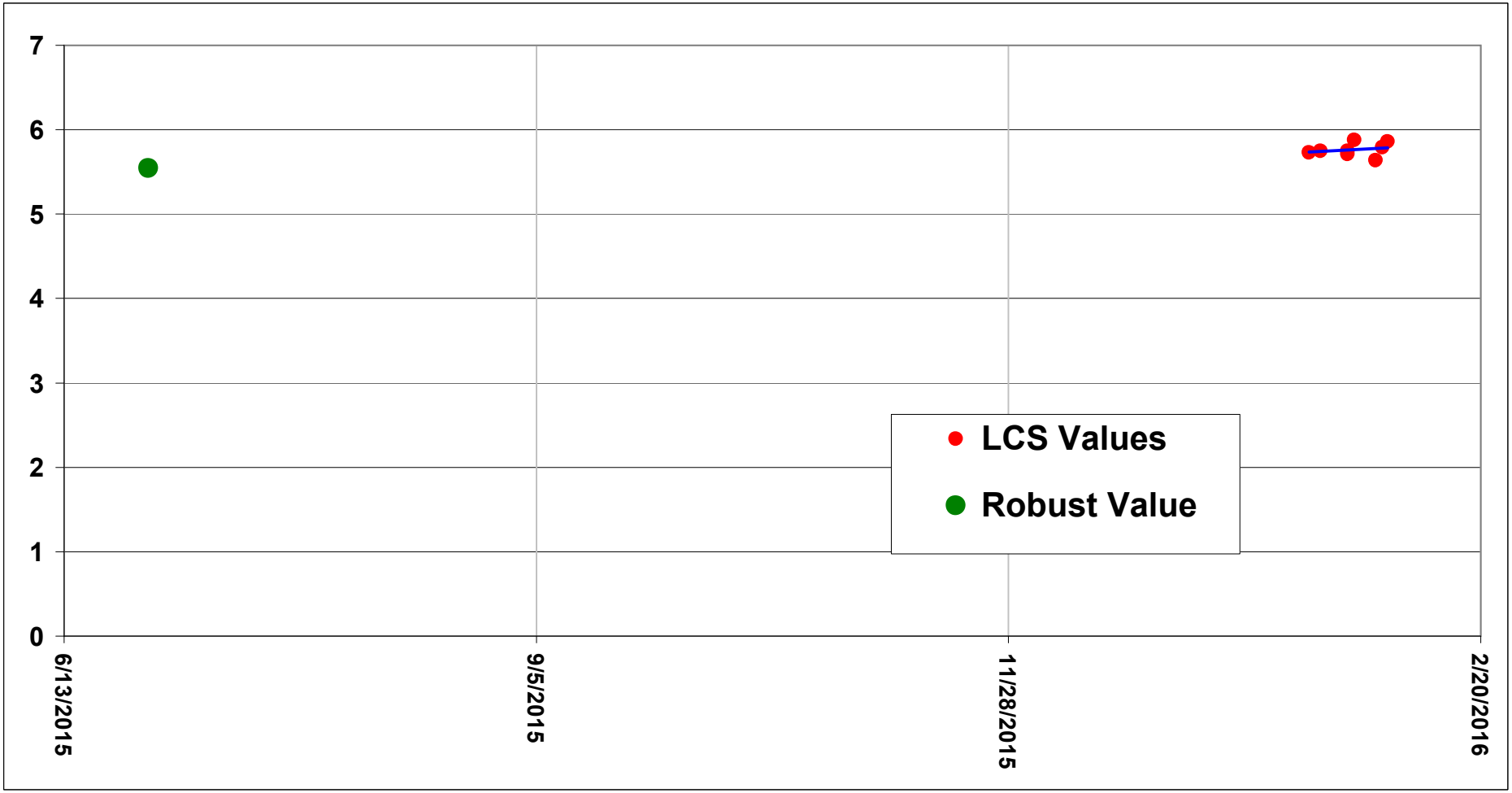
Analytical %rsd	3.51%
Analytical Variance Flag	sr OK
Should be > 1	1.30
Weeks from End of Round	63

Crude Fat 201532 Distillers Dried Grains with Solubles %



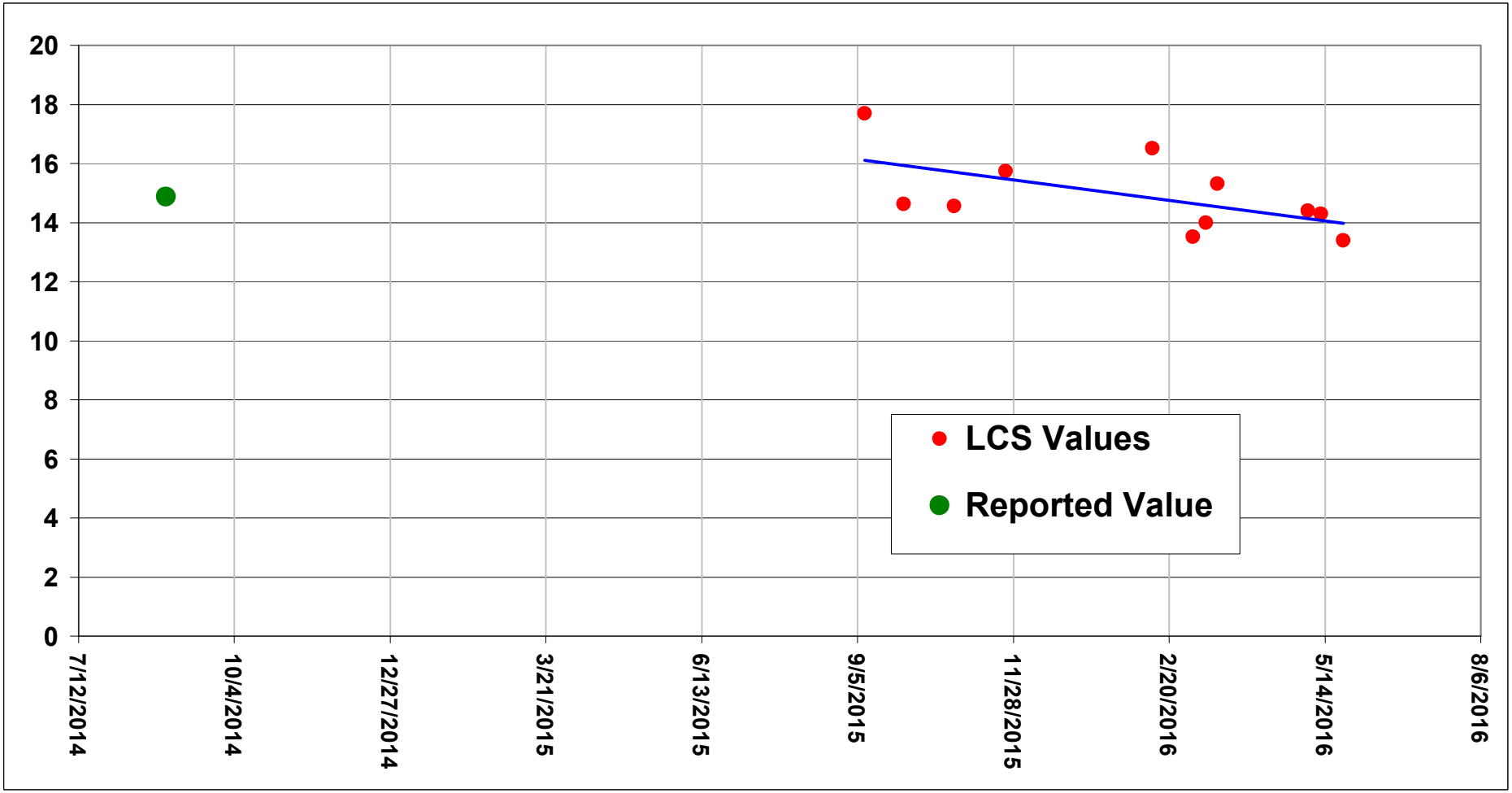
Significance of Slope	0.3503		
No Evidence of Instability over 43 weeks.		Analytical %rsd	1.83%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	2.35
Sigma PT (%RSD)	3.28%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.038		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	45

Crude Fat 201525 Equine Feed %



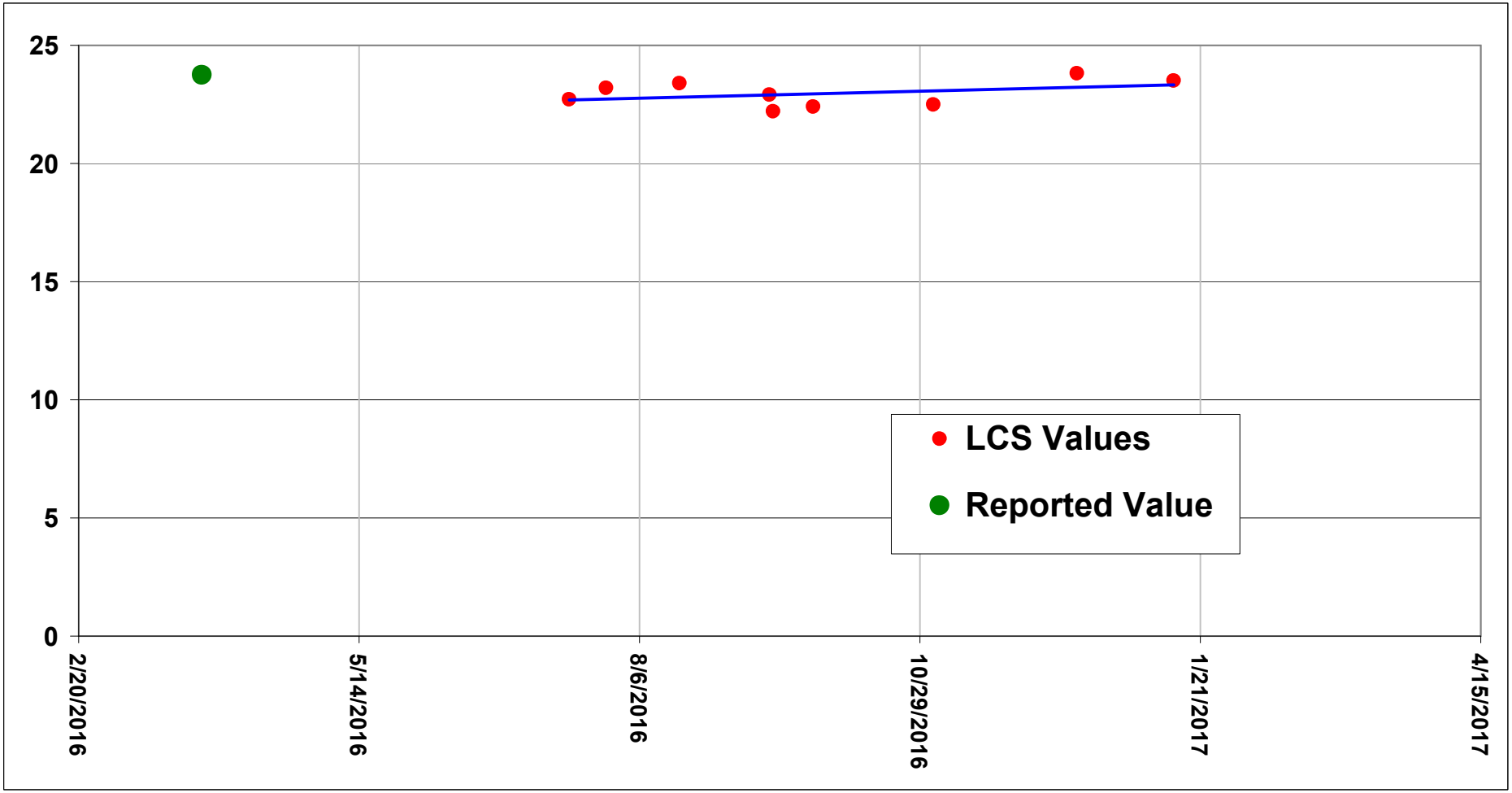
Significance of Slope	0.5685		
No Evidence of Instability over 2 weeks.		Analytical %rsd	1.36%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	2.48
Sigma PT (%RSD)	3.07%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.054		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	29

Decoquinate 201427 Calf Starter / Grower, Medicated mg/lb



Significance of Slope	0.0561		
No Evidence of Instability over 36 weeks.			
12 Week % Rel. Diff.	NA	Analytical %rsd	8.77%
		Analytical Variance Flag	sr Suspect
		Should be > 1	0.85
Sigma PT (%RSD)	9.46%	(Horwitz)	
Allowed Var , 33% σ PT (A)	1.050		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	54

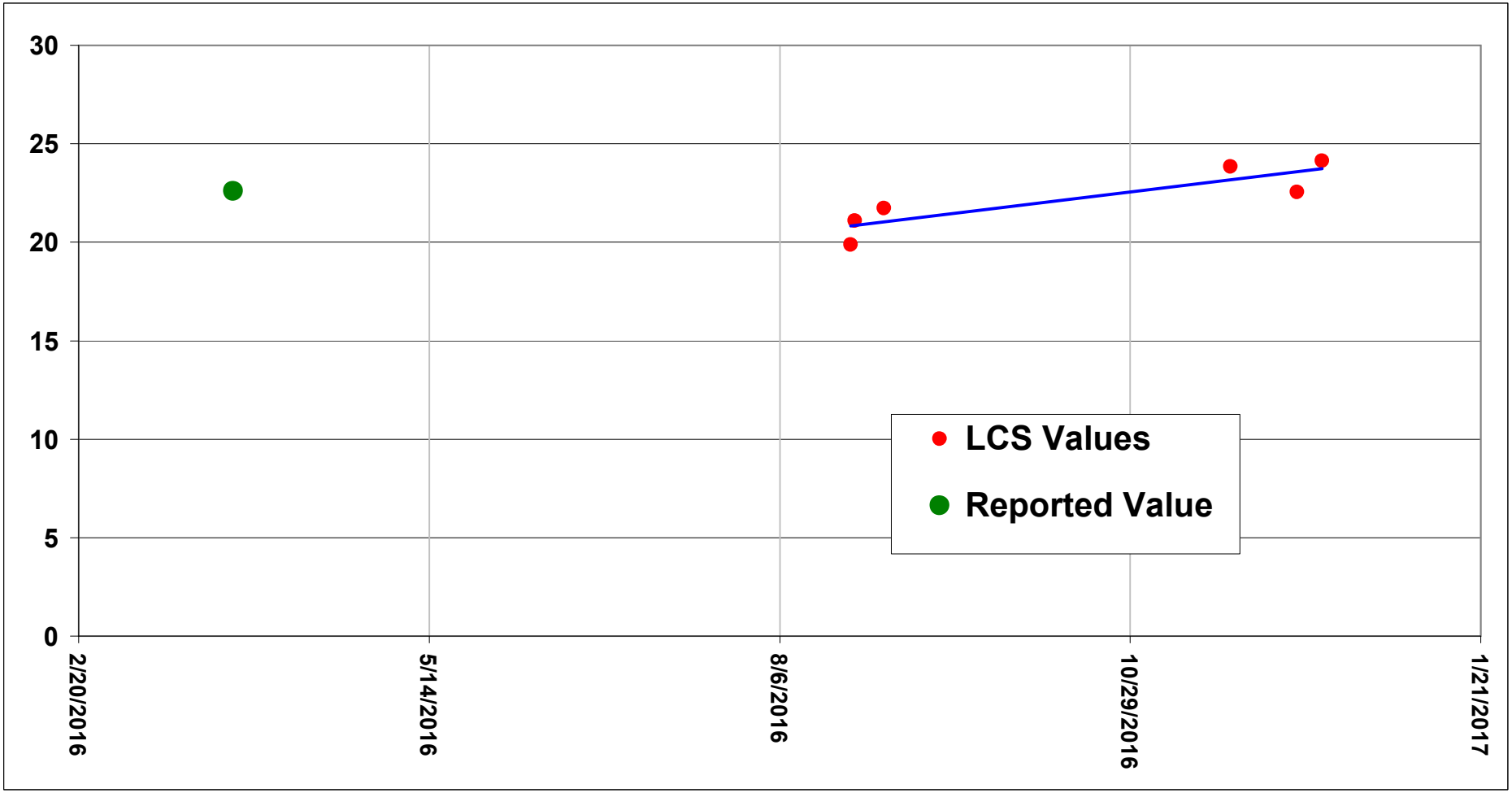
Decoquinate **201622** **Lamb Feed, Medicated** **ppm**



Significance of Slope	0.3040		
No Evidence of Instability over 25 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	9.98%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.700		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

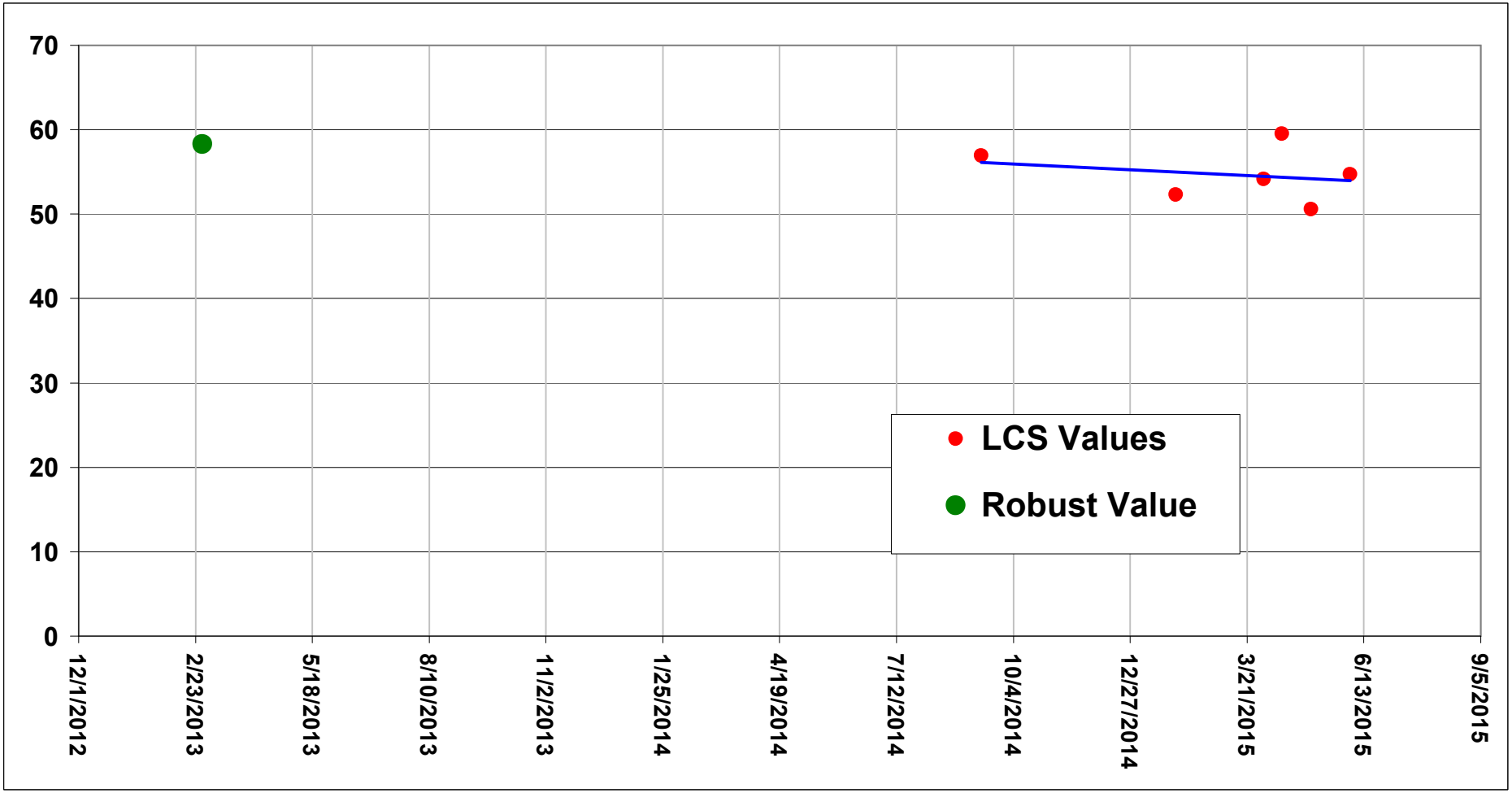
Analytical %rsd	2.40%
Analytical Variance Flag	sr OK
Should be > 1	2.83
Weeks from End of Round	16

Decoquinate 201622 Lamb Feed, Medicated ppm



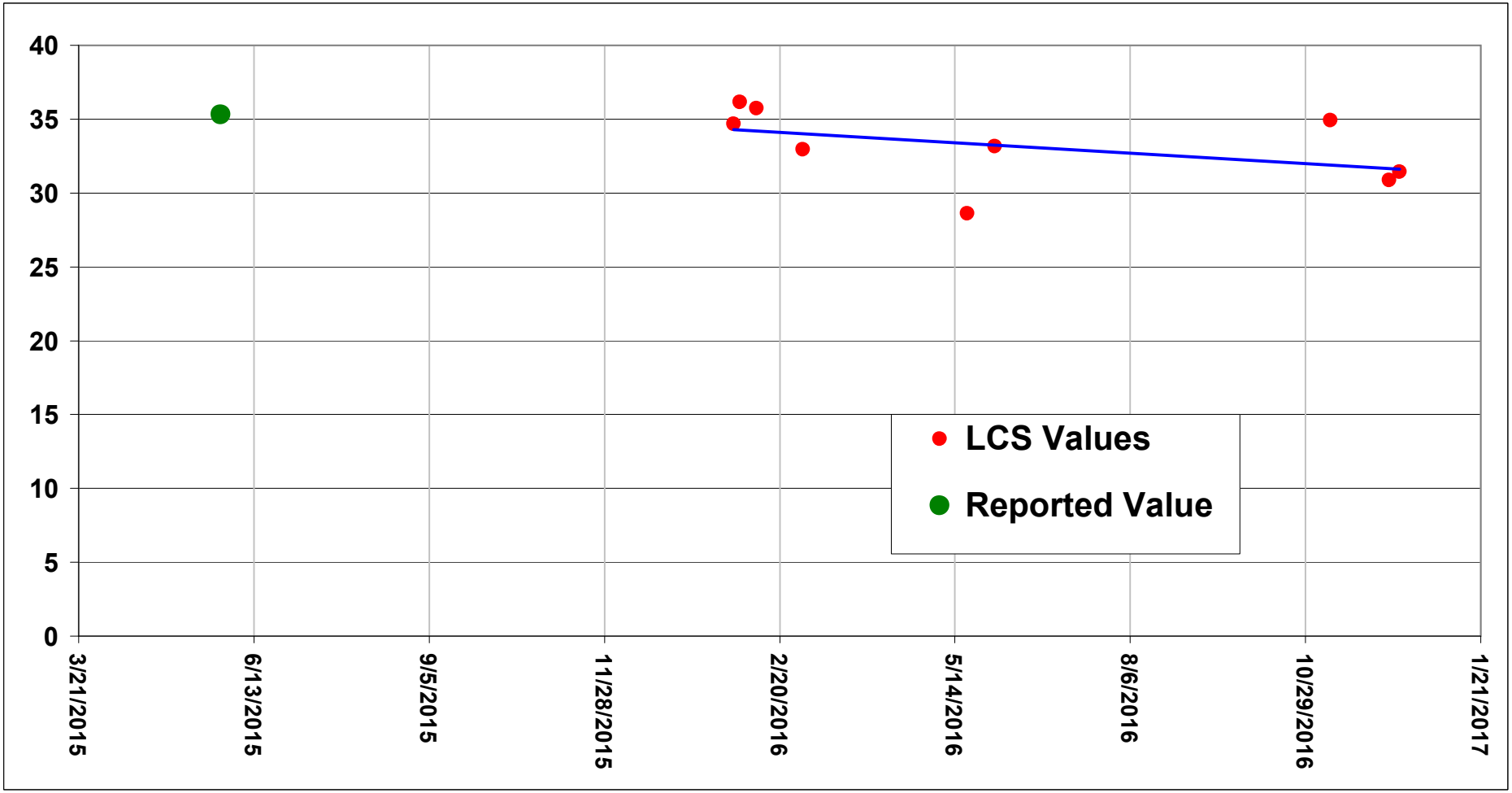
Significance of Slope	0.0220	+'ve Slope ??		
Evidence of Instability over 16 weeks.			Analytical %rsd	3.97%
12 Week % Rel. Diff.	14.62%		Analytical Variance Flag	sr OK
			Should be > 1	1.69
Sigma PT (%RSD)	10.03%	(Horwitz)		
Allowed Var , 33% σ PT (A)	0.704			
12 Week Difference (B)	2.164			
Stability Decision, Is B < A?	Low Allowed Var.		Weeks from End of Round	21

Lasalocid 201331 Cattle grower, Medicated ppm



Significance of Slope	0.6378			
No Evidence of Instability over 37 weeks.				
12 Week % Rel. Diff.	NA		Analytical %rsd	5.83%
			Analytical Variance Flag	sr Suspect
			Should be > 1	0.93
Sigma PT (%RSD)	8.76%	(Horwitz)		
Allowed Var , 33% σ PT (A)	1.614			
12 Week Difference (B)	0.000			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	80

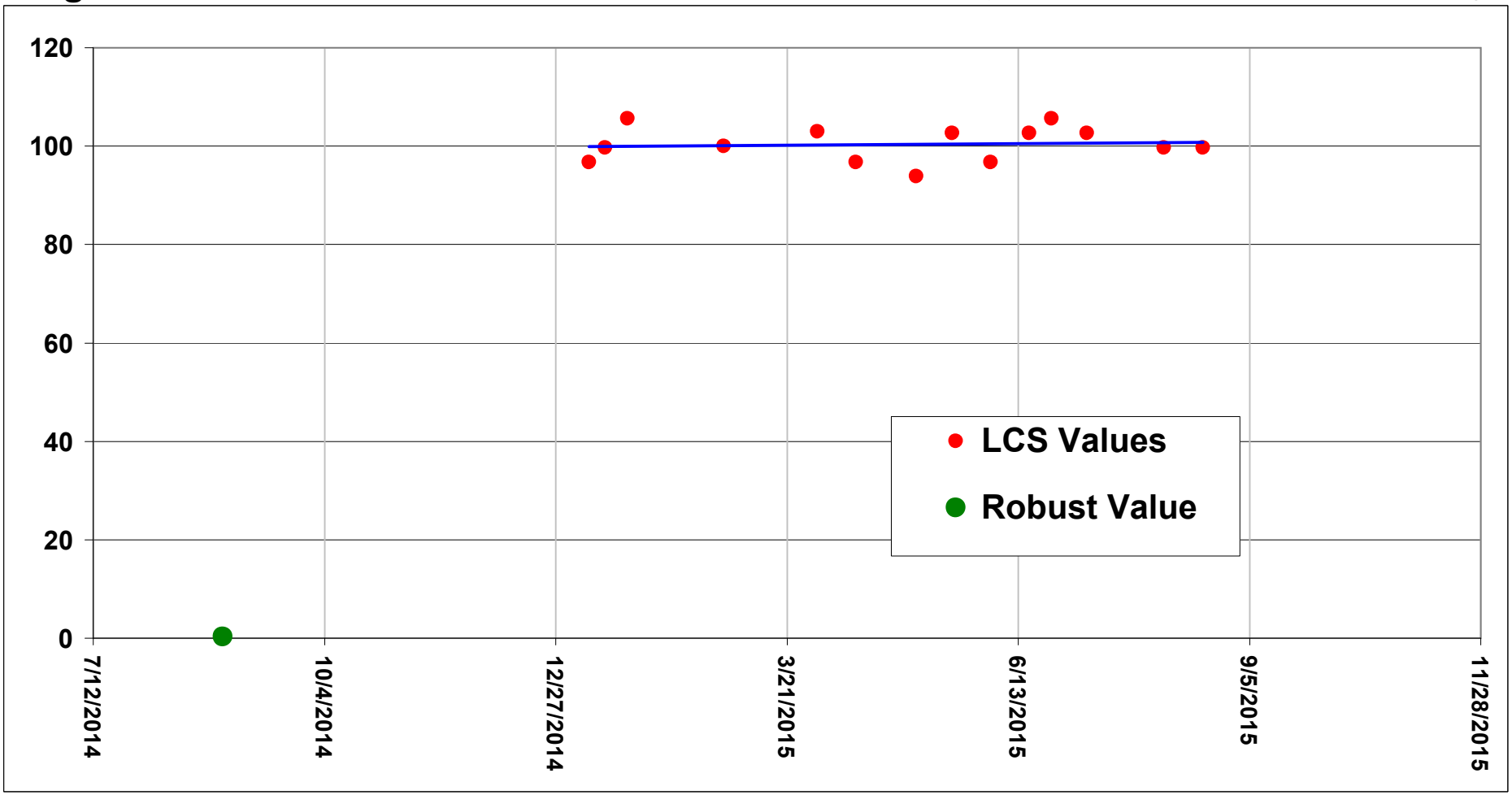
Lasalocid 201524 Goat Feed, Medicated ppm



Significance of Slope	0.2173		
No Evidence of Instability over 45 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	9.44%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.986		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	7.54%
Analytical Variance Flag	sr OK
Should be > 1	1.07
Weeks from End of Round	35

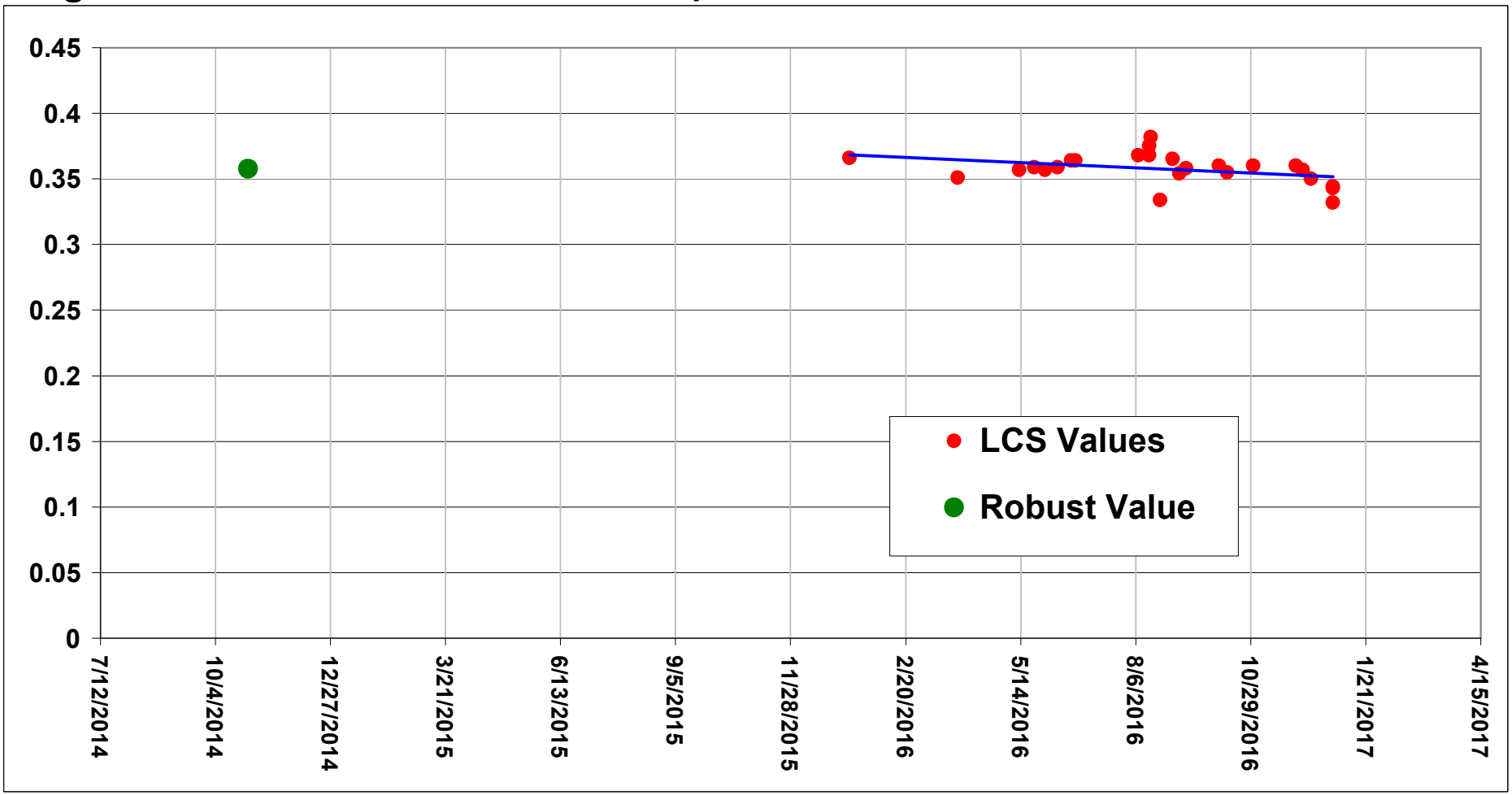
Magnesium 201427 Calf Starter / Grower, Medicated % Recovery



Significance of Slope	0.7923		
No Evidence of Instability over 32 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	4.12%	(Participants)	
Allowed Var , 33% σ PT (A)	0.004		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	3.48%
Analytical Variance Flag	sr Suspect
Should be > 1	0.76
Weeks from End of Round	19

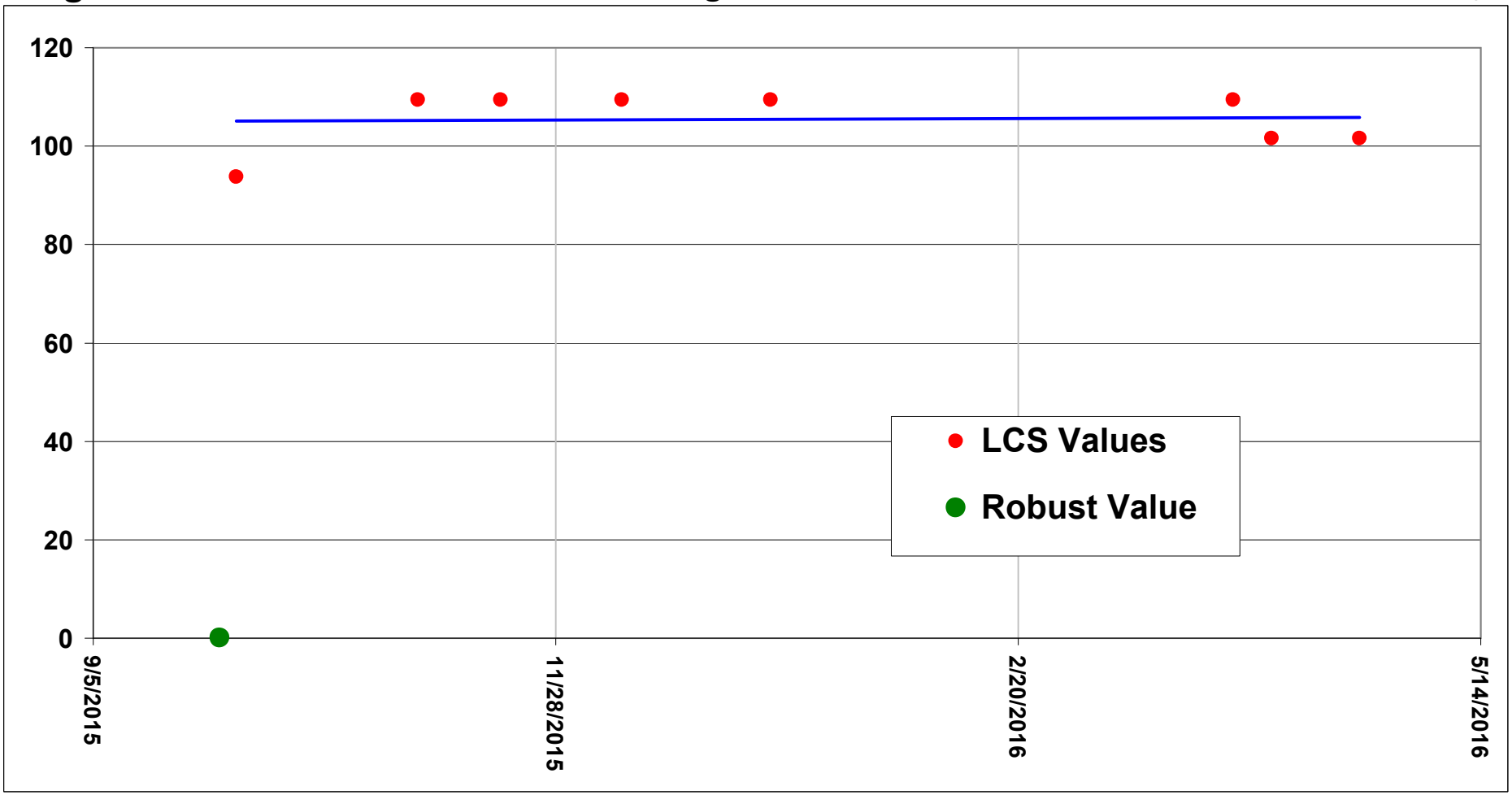
Magnesium 201429 Equine Feed %



Significance of Slope	0.0555		
No Evidence of Instability over 50 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	4.67%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.005		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

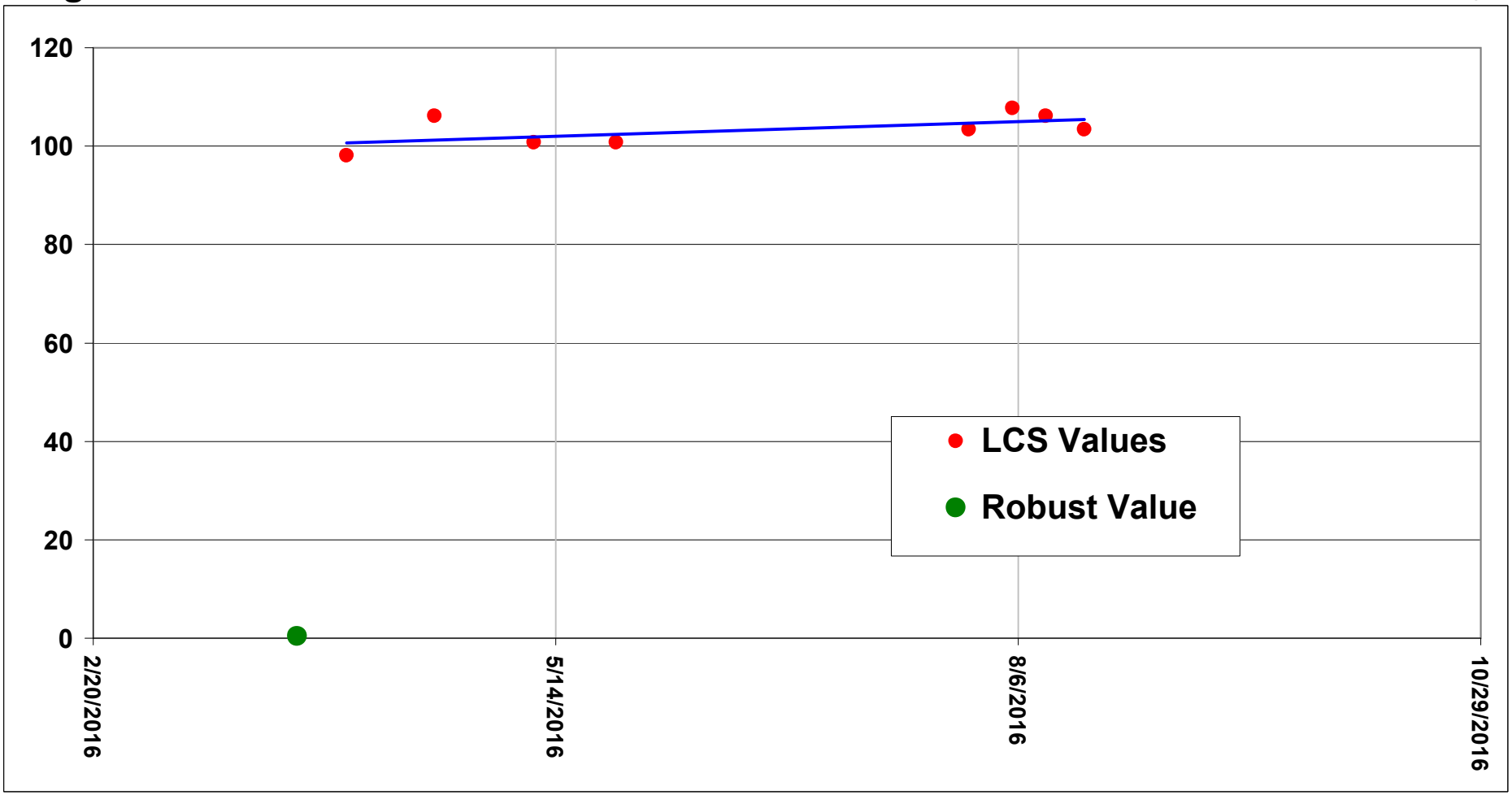
Analytical %rsd	3.16%
Analytical Variance Flag	sr OK
Should be > 1	1.05
Weeks from End of Round	63

Magnesium 201528 Dog Food % Recovery



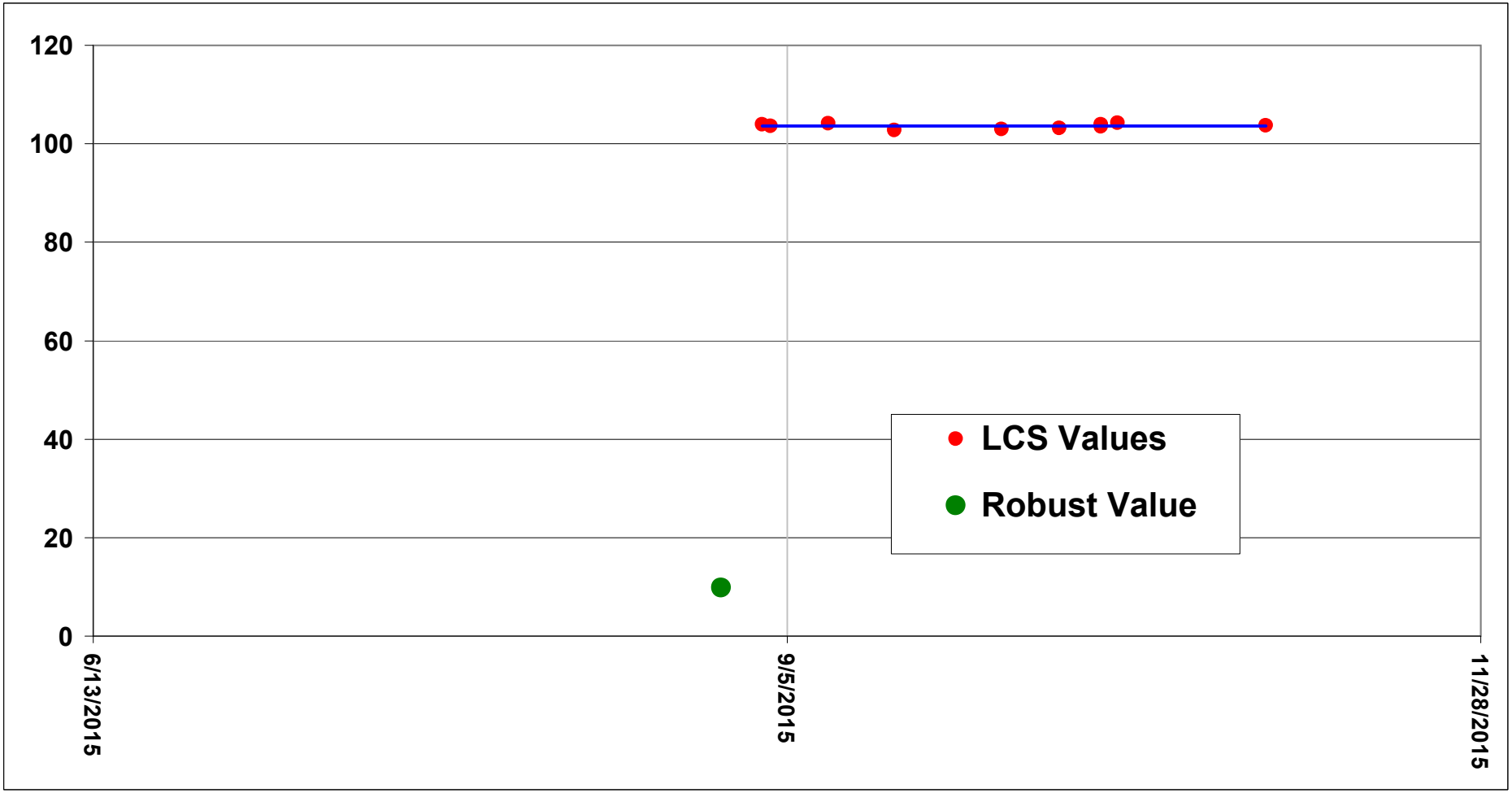
Significance of Slope	0.9166		
No Evidence of Instability over 29 weeks.		Analytical %rsd	5.60%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr Suspect
		Should be > 1	0.67
Sigma PT (%RSD)	6.08%	(Participants)	
Allowed Var , 33% σ PT (A)	0.003		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	0

Magnesium 201622 Lamb Feed, Medicated % Recovery



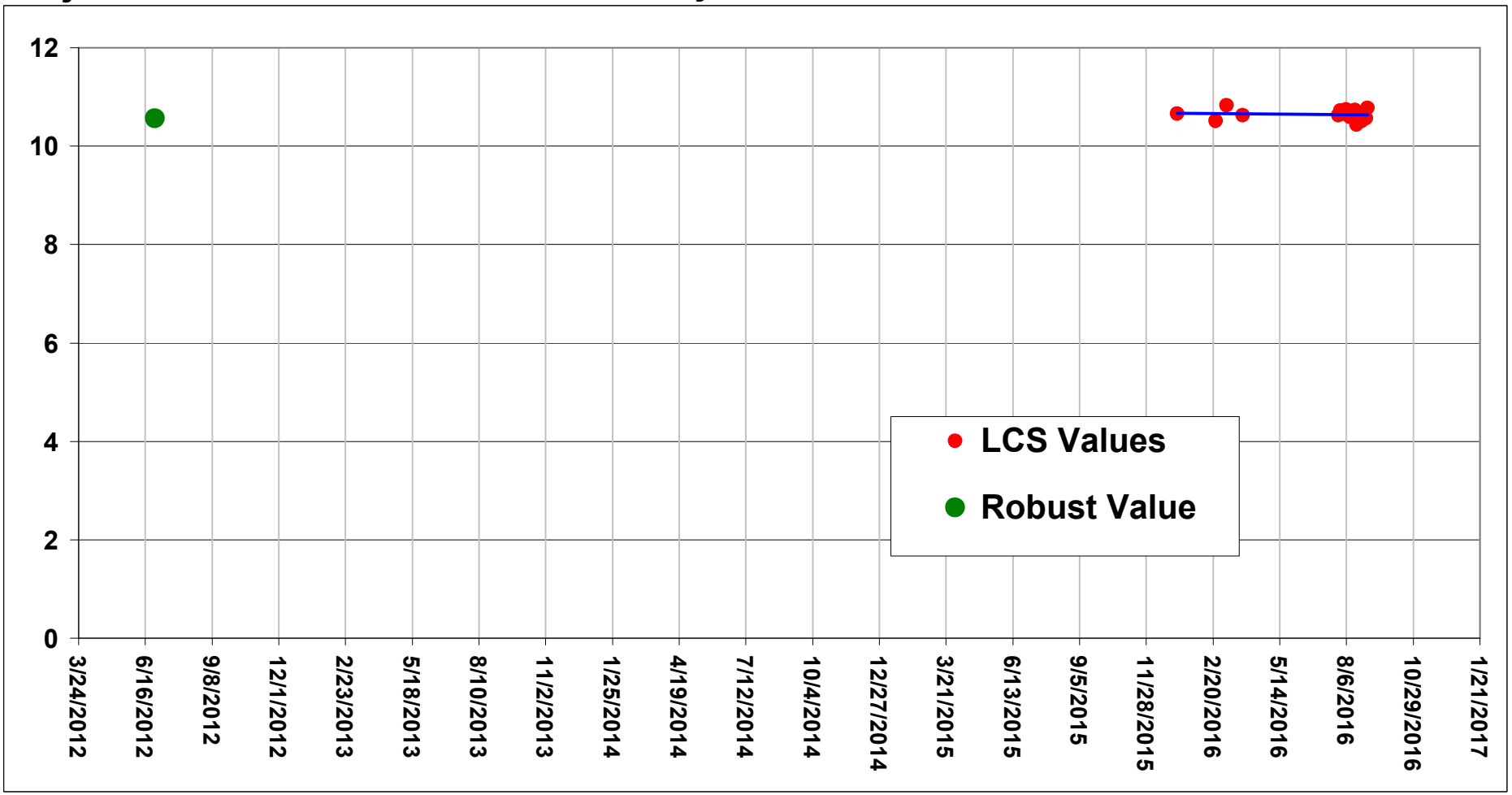
Significance of Slope	0.1164		
No Evidence of Instability over 19 weeks.		Analytical %rsd	3.15%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.26
Sigma PT (%RSD)	5.11%	(Participants)	
Allowed Var , 33% σ PT (A)	0.006		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	1

Moisture **201527** **Chick Starter, Medicated** **% Recovery**



Significance of Slope	0.9475		
No Evidence of Instability over 9 weeks.		Analytical %rsd	0.46%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	4.55
Sigma PT (%RSD)	3.31%	(Participants)	
Allowed Var , 33% σ PT (A)	0.104		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	1

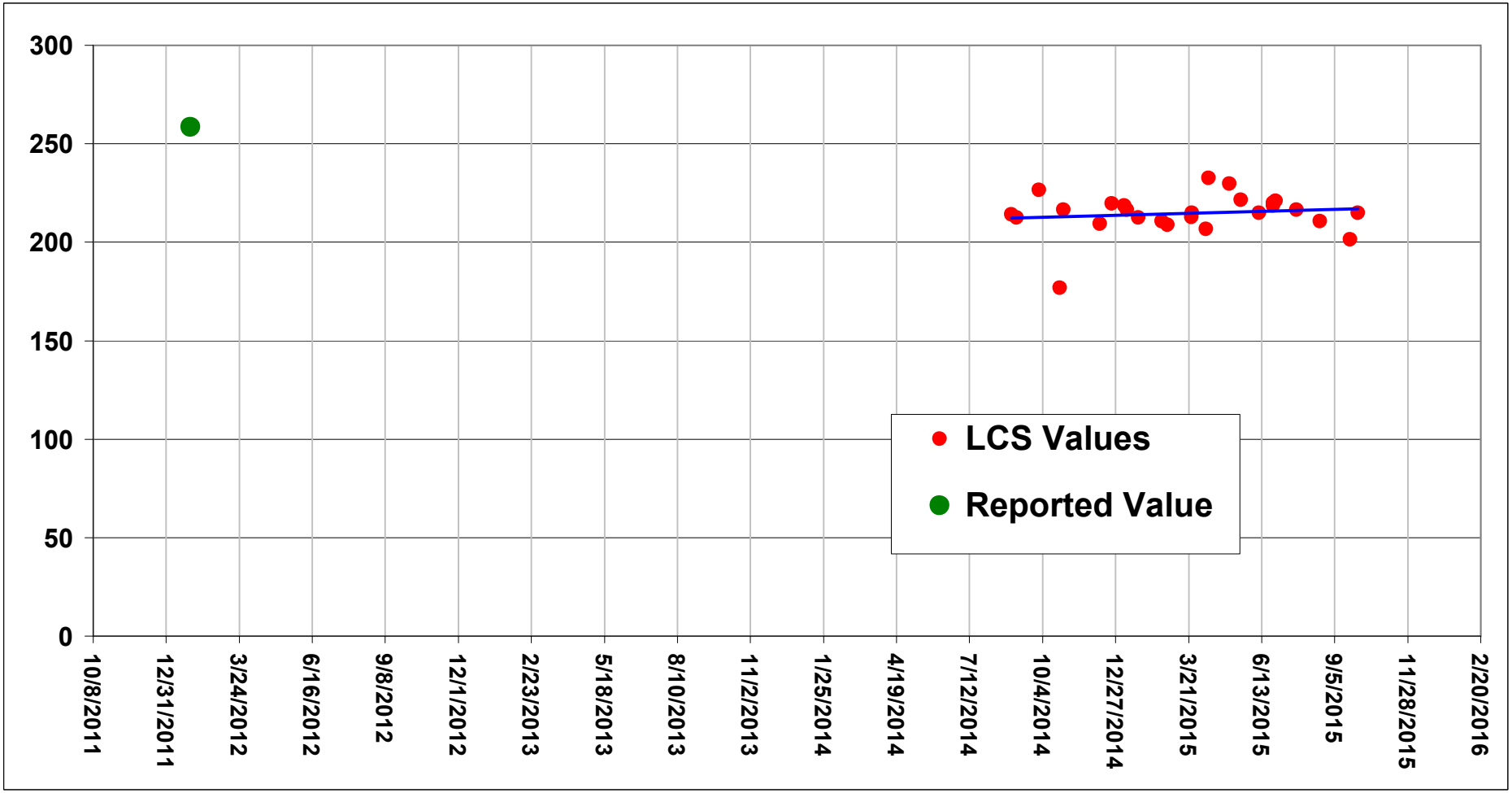
Moj. Fat 201225 Dry Cat Food %



Significance of Slope	0.7066		
No Evidence of Instability over 34 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	2.80%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.094		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	0.98%
Analytical Variance Flag	sr OK
Should be > 1	2.62
Weeks from End of Round	184

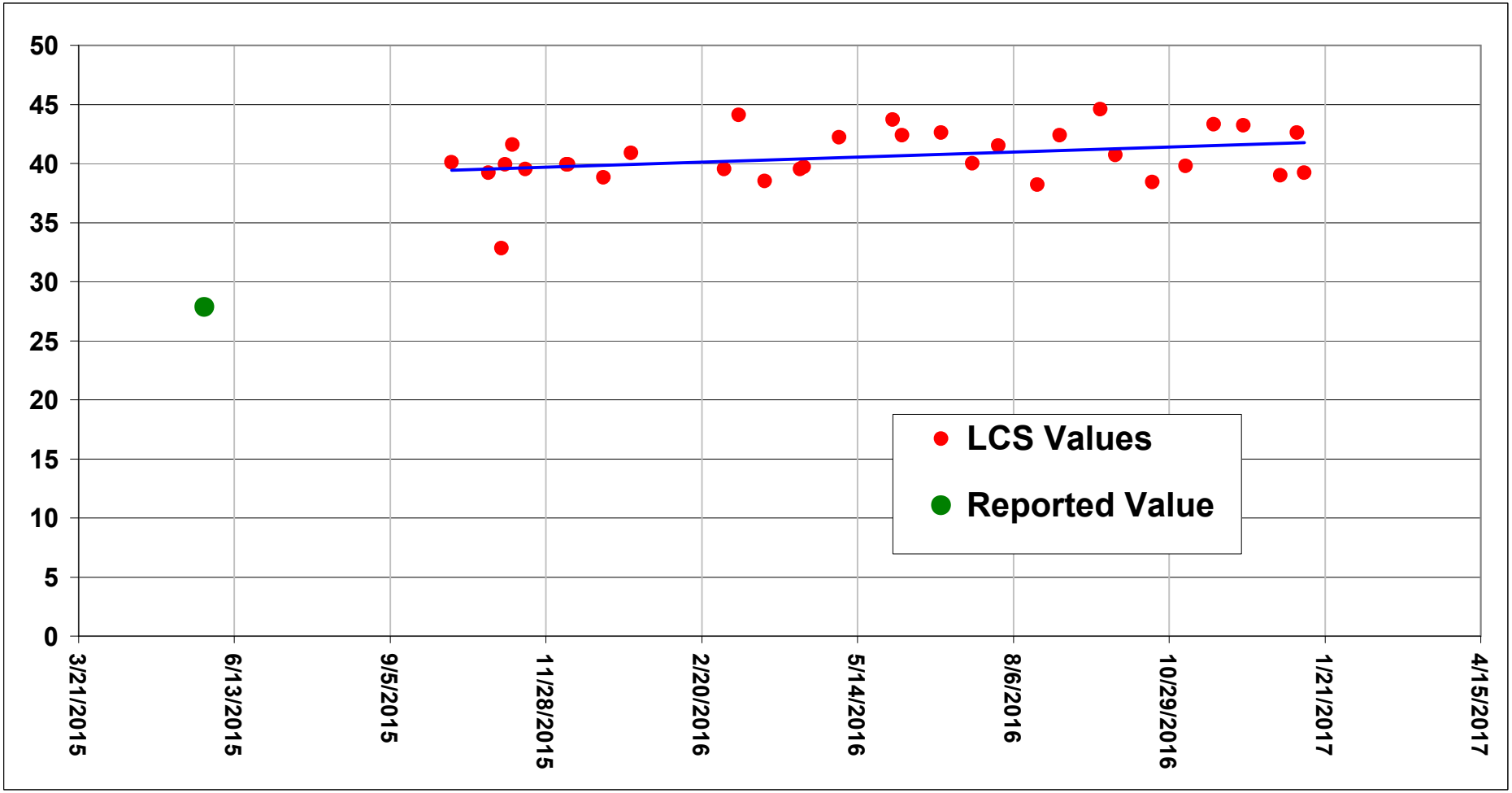
Monensin 201230 Beef Feedlot special, Medicated g/ton



Significance of Slope	0.5189		
No Evidence of Instability over 56 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	7.03%	(Horwitz)	
Allowed Var , 33% σ PT (A)	5.788		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

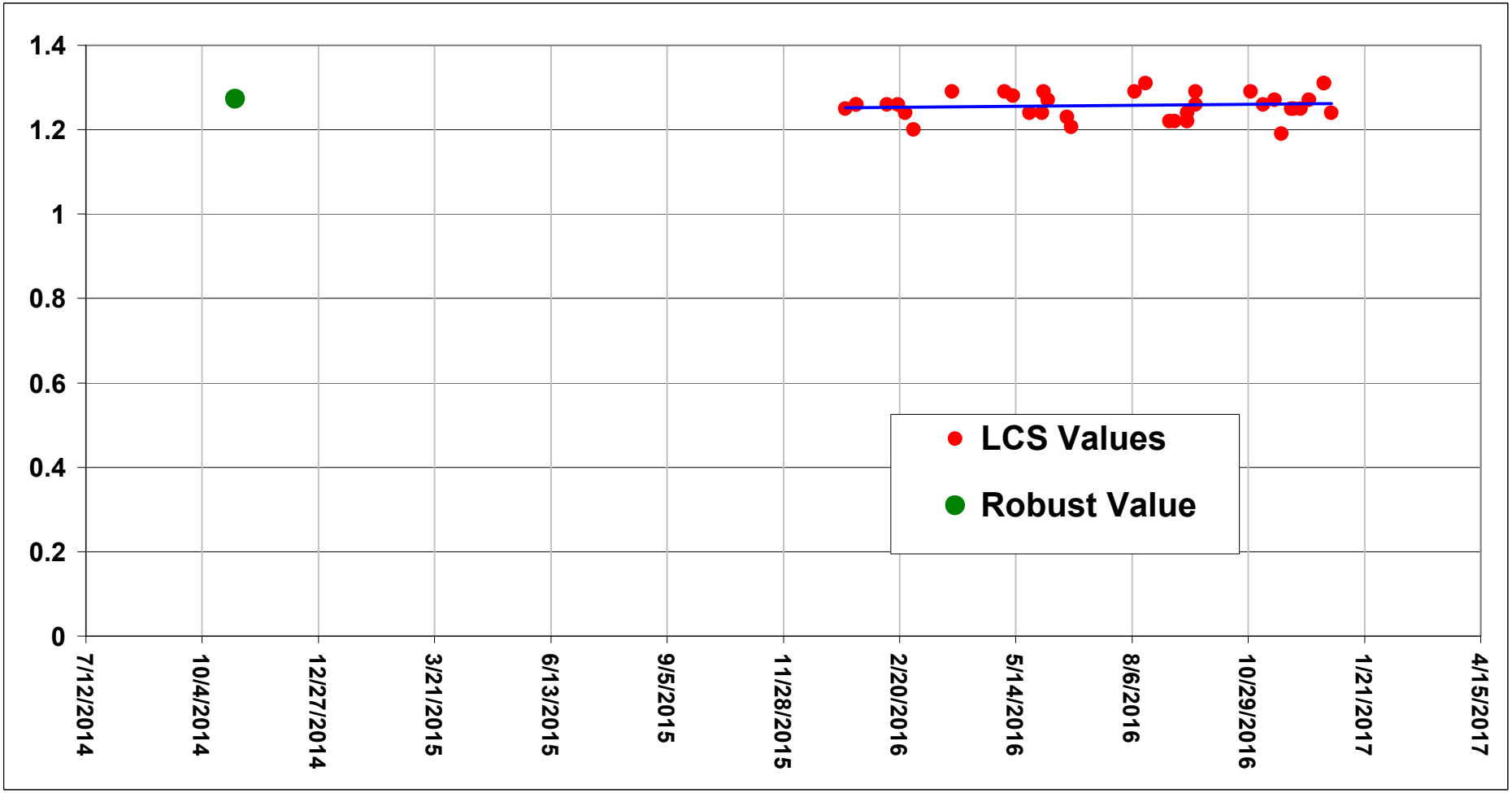
Analytical %rsd	4.80%
Analytical Variance Flag	sr Suspect
Should be > 1	0.97
Weeks from End of Round	135

Monensin 201524 Goat Feed, Medicated g/ton



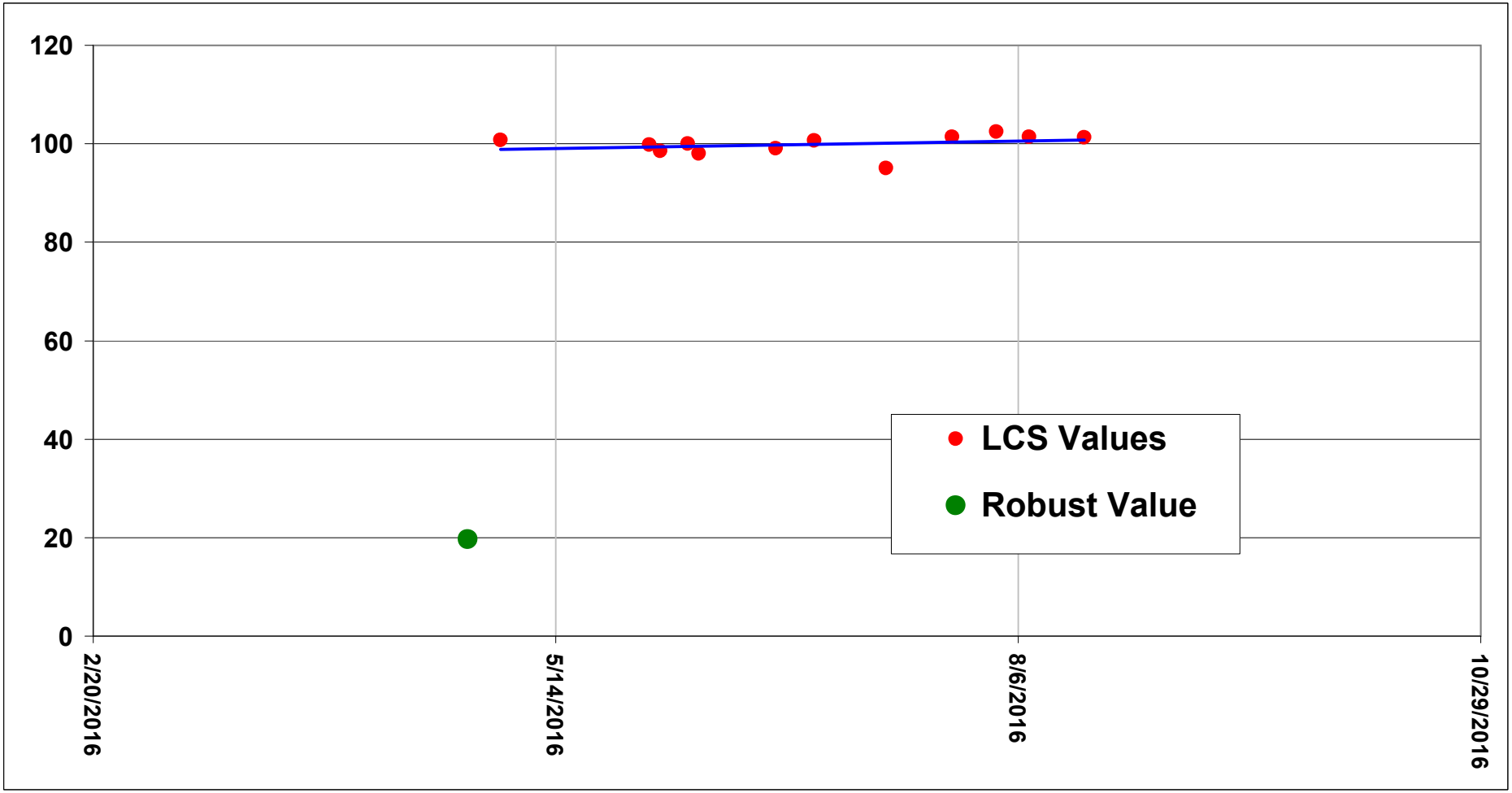
Significance of Slope	0.0640			
No Evidence of Instability over 65 weeks.				
12 Week % Rel. Diff.	NA		Analytical %rsd	5.66%
			Analytical Variance Flag	sr OK
			Should be > 1	1.11
Sigma PT (%RSD)	9.03%	(Horwitz)		
Allowed Var , 33% σ PT (A)	0.611			
12 Week Difference (B)	0.000			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	19

Potassium 201429 Equine Feed %



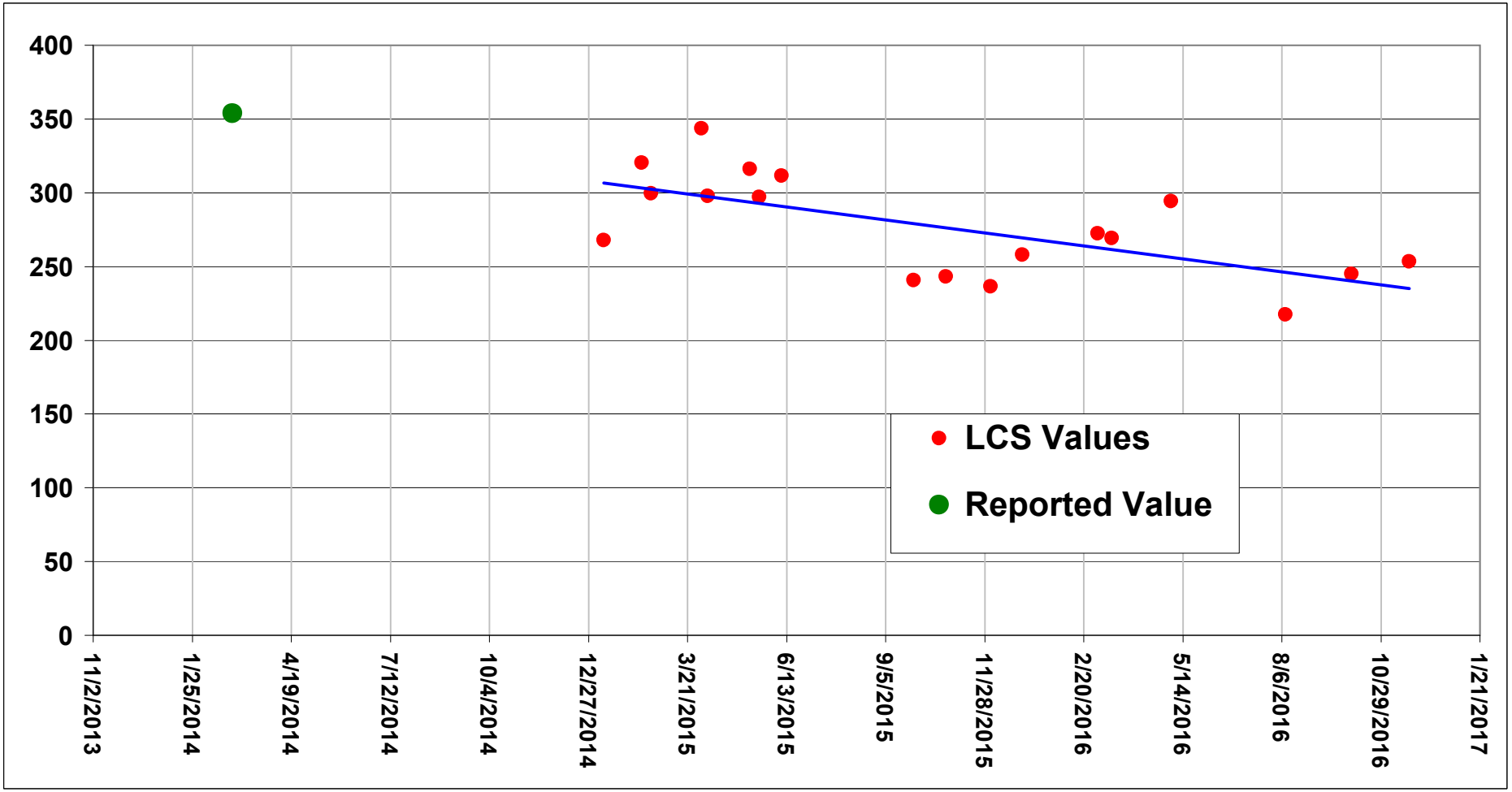
Significance of Slope	0.5903		
No Evidence of Instability over 49 weeks.		Analytical %rsd	2.47%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.48
Sigma PT (%RSD)	3.86%	(Horwitz)	
Allowed Var , 33% σ PT (A)	0.016		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	63

Protein **201623 Poultry Feed, Medicated** **% Recovery**



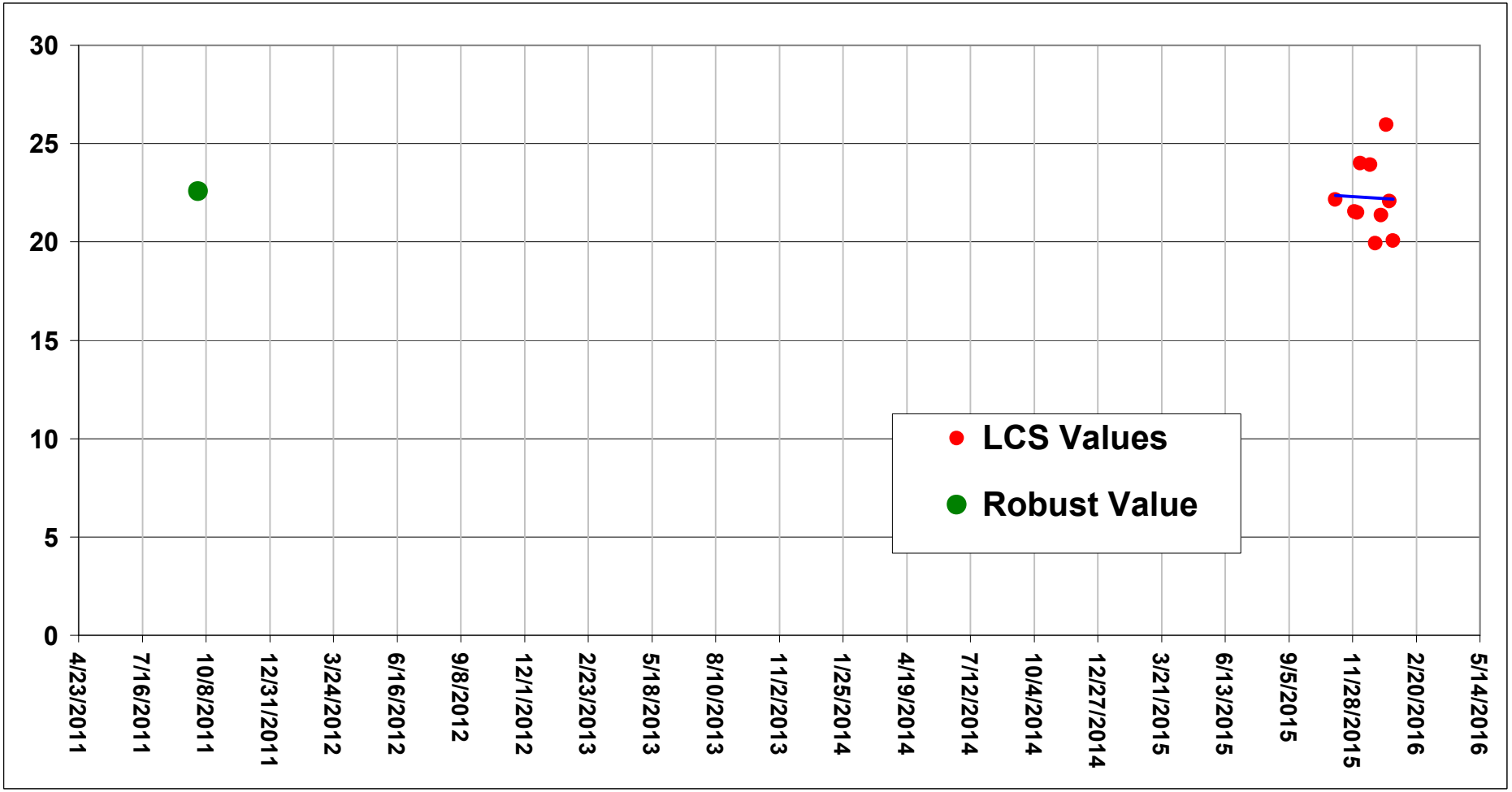
Significance of Slope	0.3302		
No Evidence of Instability over 15 weeks.		Analytical %rsd	2.01%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr Suspect
		Should be > 1	0.57
Sigma PT (%RSD)	1.71%	(Participants)	
Allowed Var , 33% σ PT (A)	0.106		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	1

Vitamin A 201491 Poultry Layer Feed KU/kg



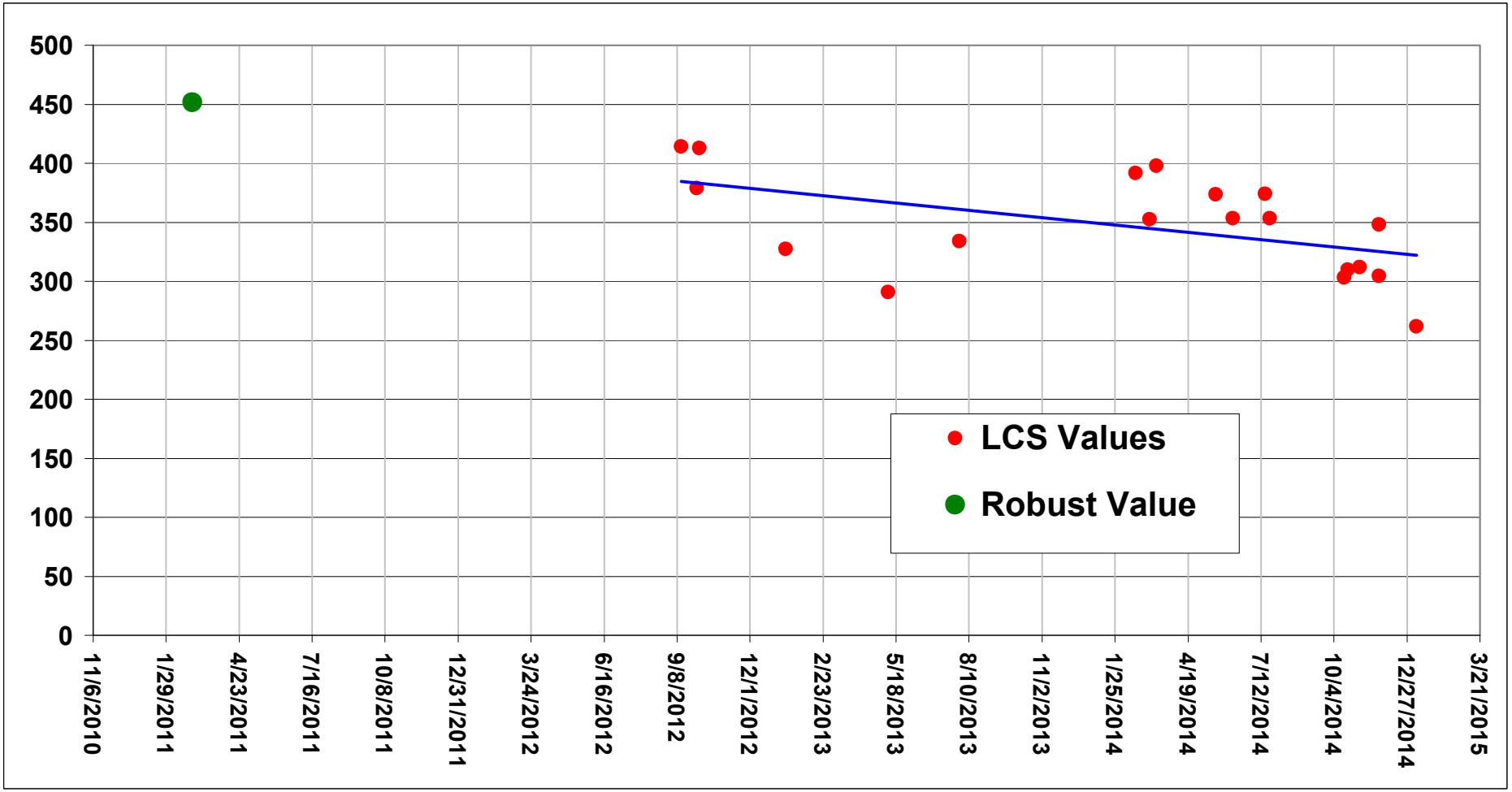
Significance of Slope	0.0030			
Evidence of Instability over 96 weeks.			Analytical %rsd	9.73%
12 Week % Rel. Diff.	4.78%		Analytical Variance Flag	sr OK
			Should be > 1	1.28
Sigma PT (%RSD)	18.56%	(Participants)		
Allowed Var , 33% σ PT (A)	19.595			
12 Week Difference (B)	8.819			
Stability Decision, Is B < A?	PASS Allowed Var.		Weeks from End of Round	45

Vitamin A 201128 Beef Feedlot Special, Medicated KU/kg



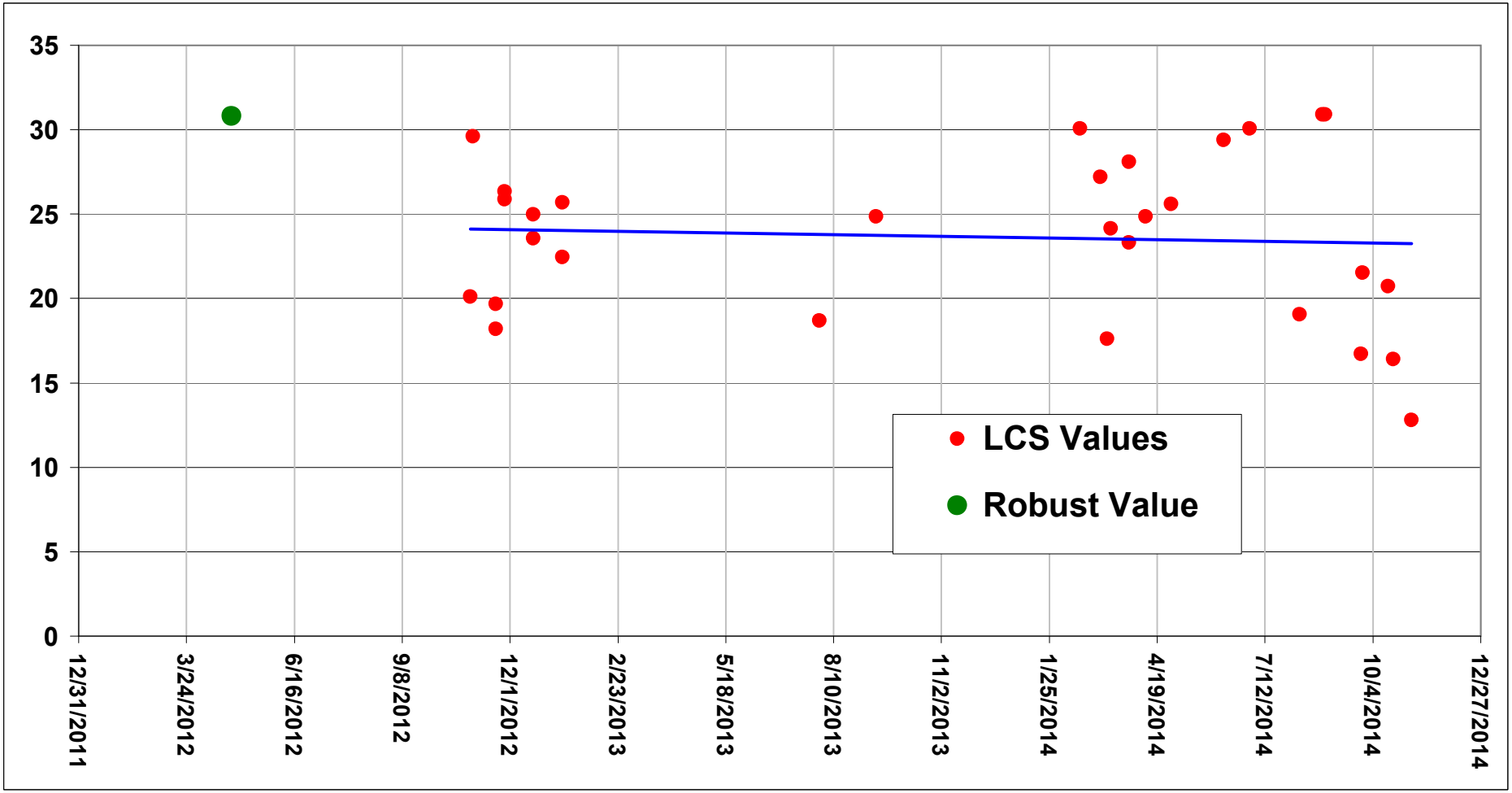
Significance of Slope	0.9359		
No Evidence of Instability over 11 weeks.		Analytical %rsd	8.43%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.02
Sigma PT (%RSD)	13.67%	(Participants)	
Allowed Var , 33% σ PT (A)	0.975		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	214

Vitamin A 201191 Dairy Cattle mineral supplement, Medica KU/kg



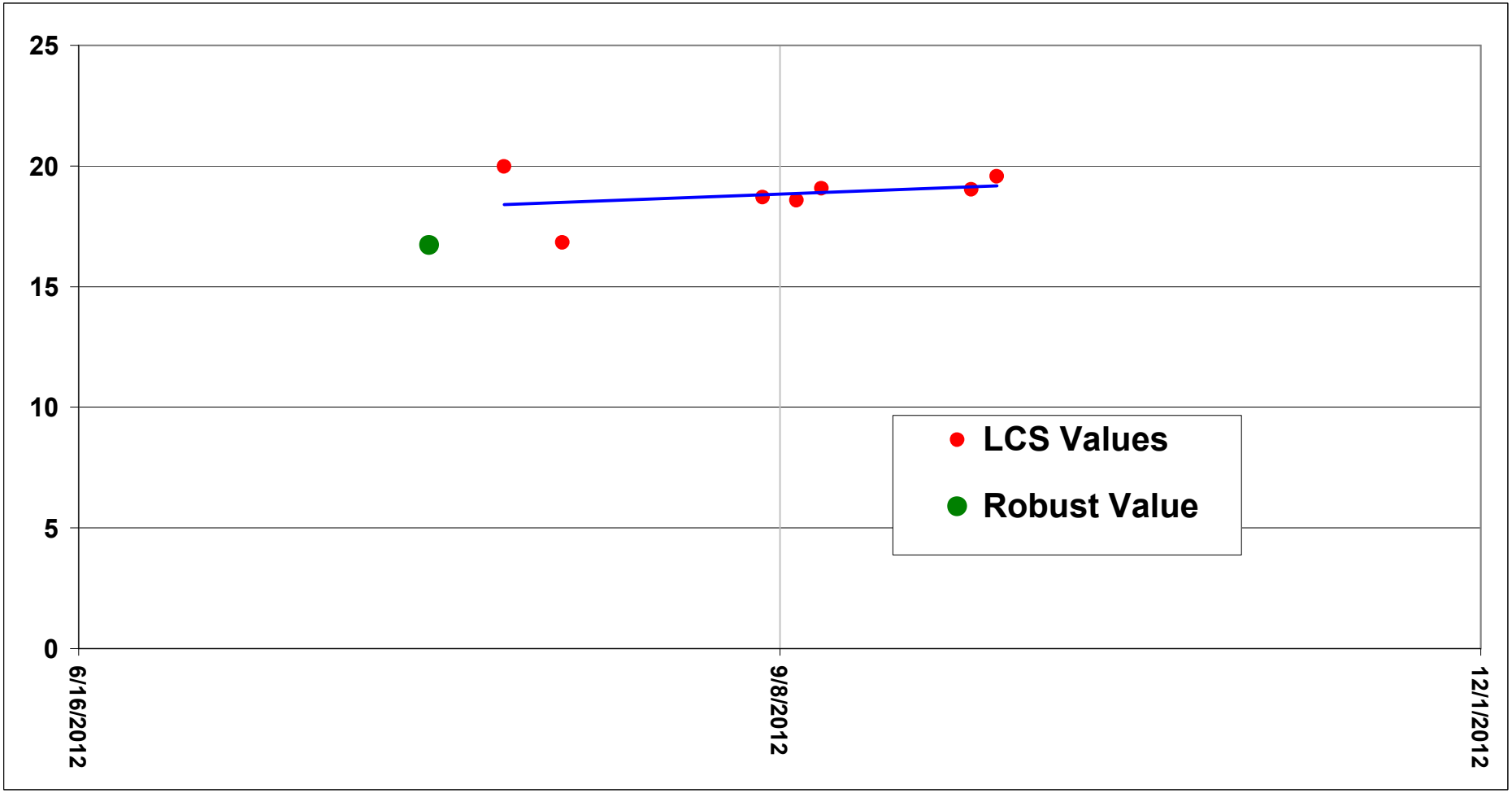
Significance of Slope	0.0271		
Evidence of Instability over 119 weeks.		Analytical %rsd	11.03%
12 Week % Rel. Diff.	2.68%	Analytical Variance Flag	sr OK
		Should be > 1	1.13
Sigma PT (%RSD)	18.61%	(Participants)	
Allowed Var , 33% σ PT (A)	26.569		
12 Week Difference (B)	6.199		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	80

Vitamin A 201223 Beef Feedlot special, Medicated KU/kg



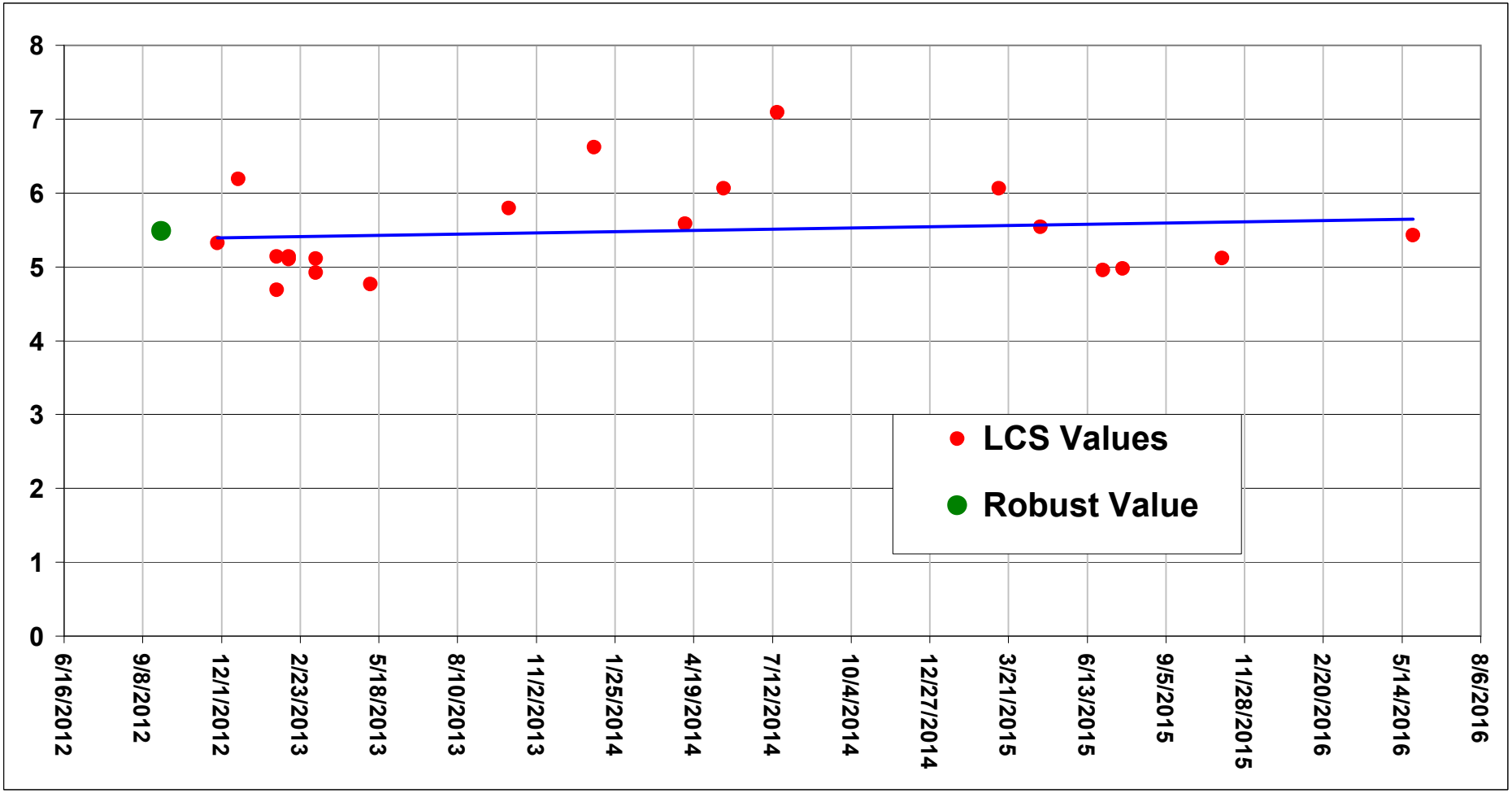
Significance of Slope	0.7254		
No Evidence of Instability over 103 weeks.			
12 Week % Rel. Diff.	NA	Analytical %rsd	20.58%
		Analytical Variance Flag	sr Suspect
		Should be > 1	0.48
Sigma PT (%RSD)	15.00%	(Participants)	
Allowed Var , 33% σ PT (A)	1.461		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	27

Vitamin A 201226 Dairy Herd & Beef Calf Milk Replacer, Me KU/kg



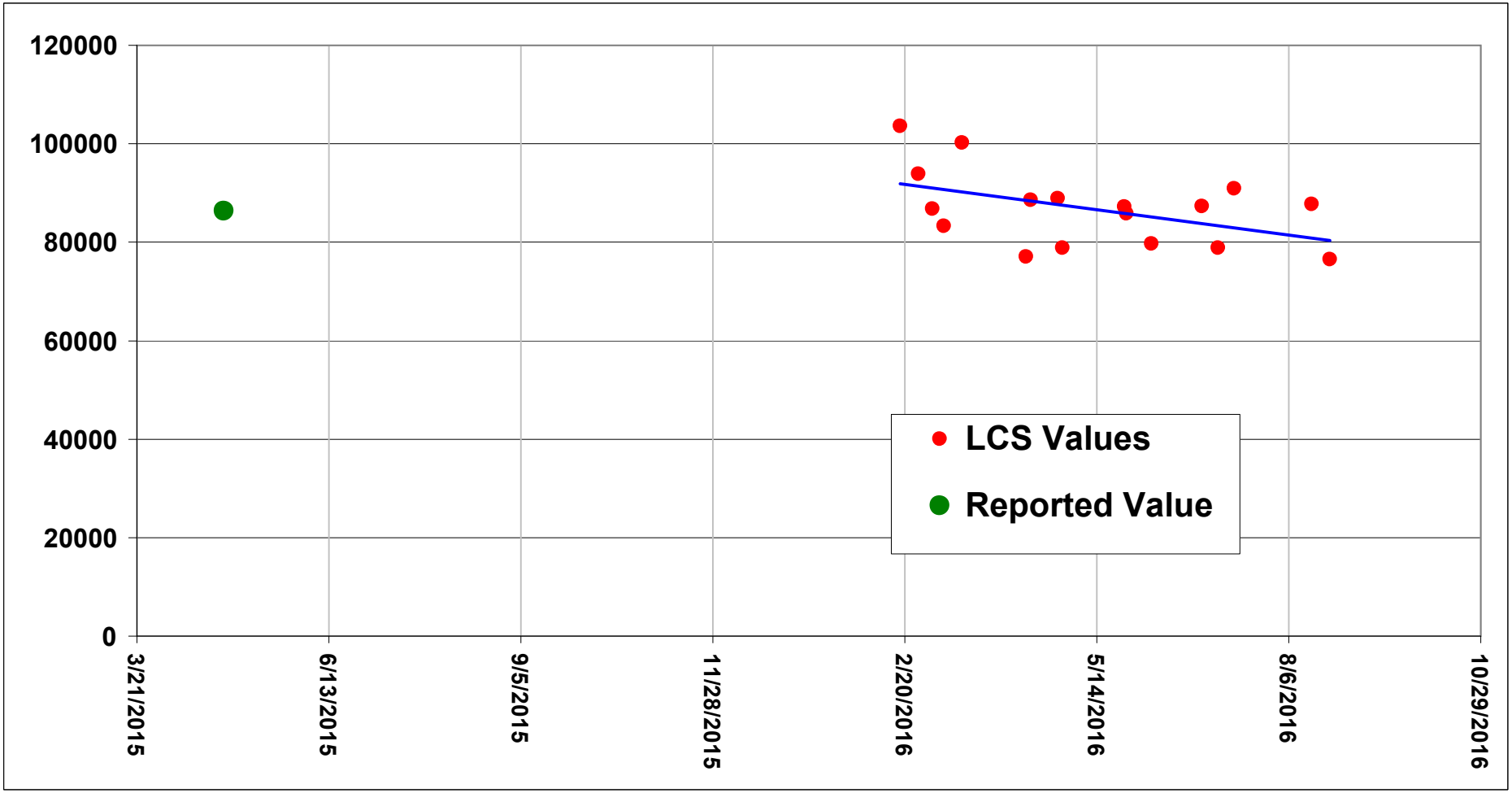
Significance of Slope	0.5265		
No Evidence of Instability over 8 weeks.		Analytical %rsd	5.34%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.88
Sigma PT (%RSD)	15.70%	(Participants)	
Allowed Var , 33% σ PT (A)	0.830		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	1

Vitamin A 201228 Sheep and Goat Grower/Finisher, Medica KU/kg



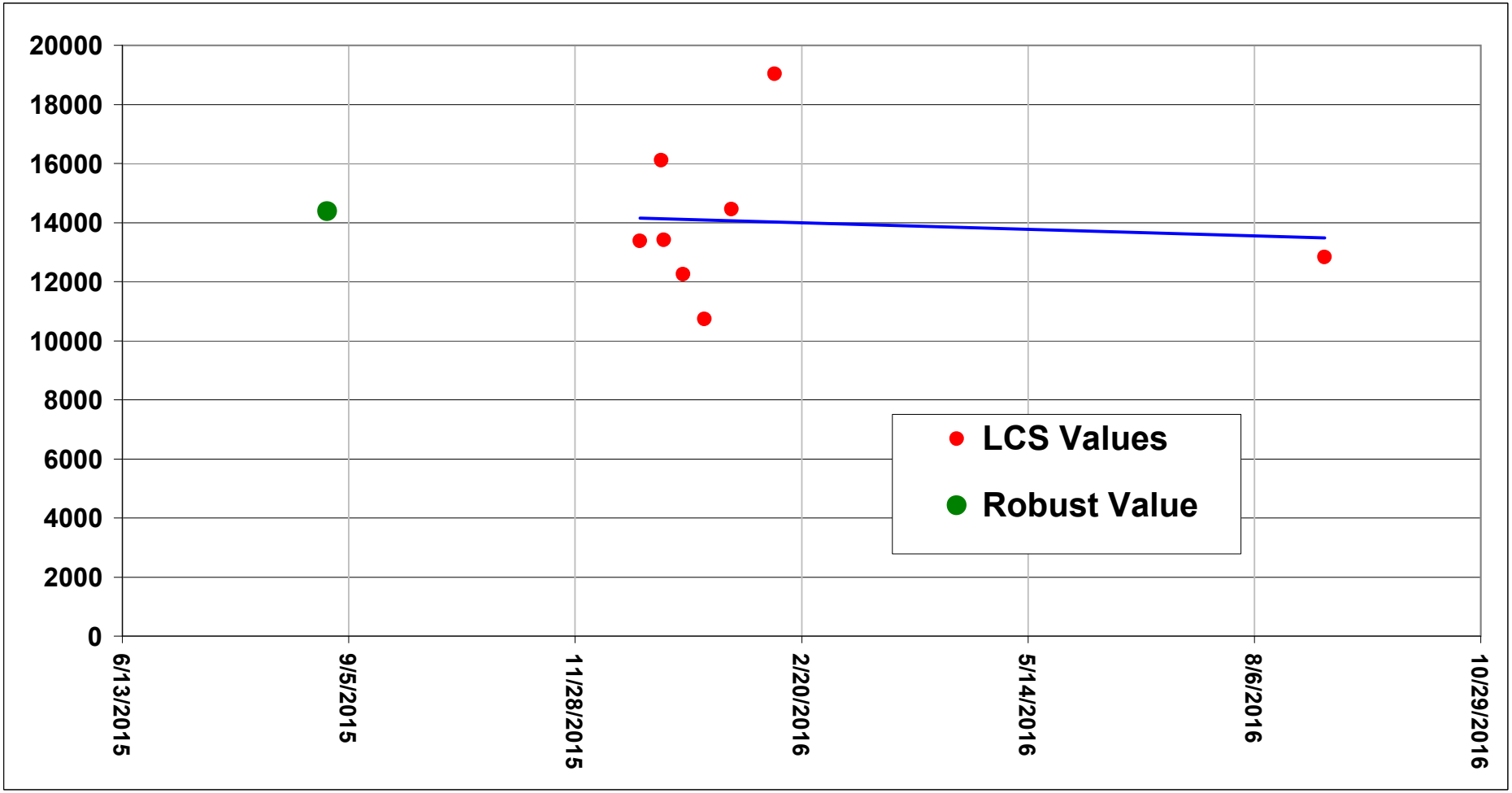
Significance of Slope	0.5819		
No Evidence of Instability over 180 weeks.		Analytical %rsd	11.73%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.12
Sigma PT (%RSD)	20.00%	(Participants)	
Allowed Var , 33% σ PT (A)	0.347		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	9

Vitamin A 201523 Milk Replacer, Medicated U/kg



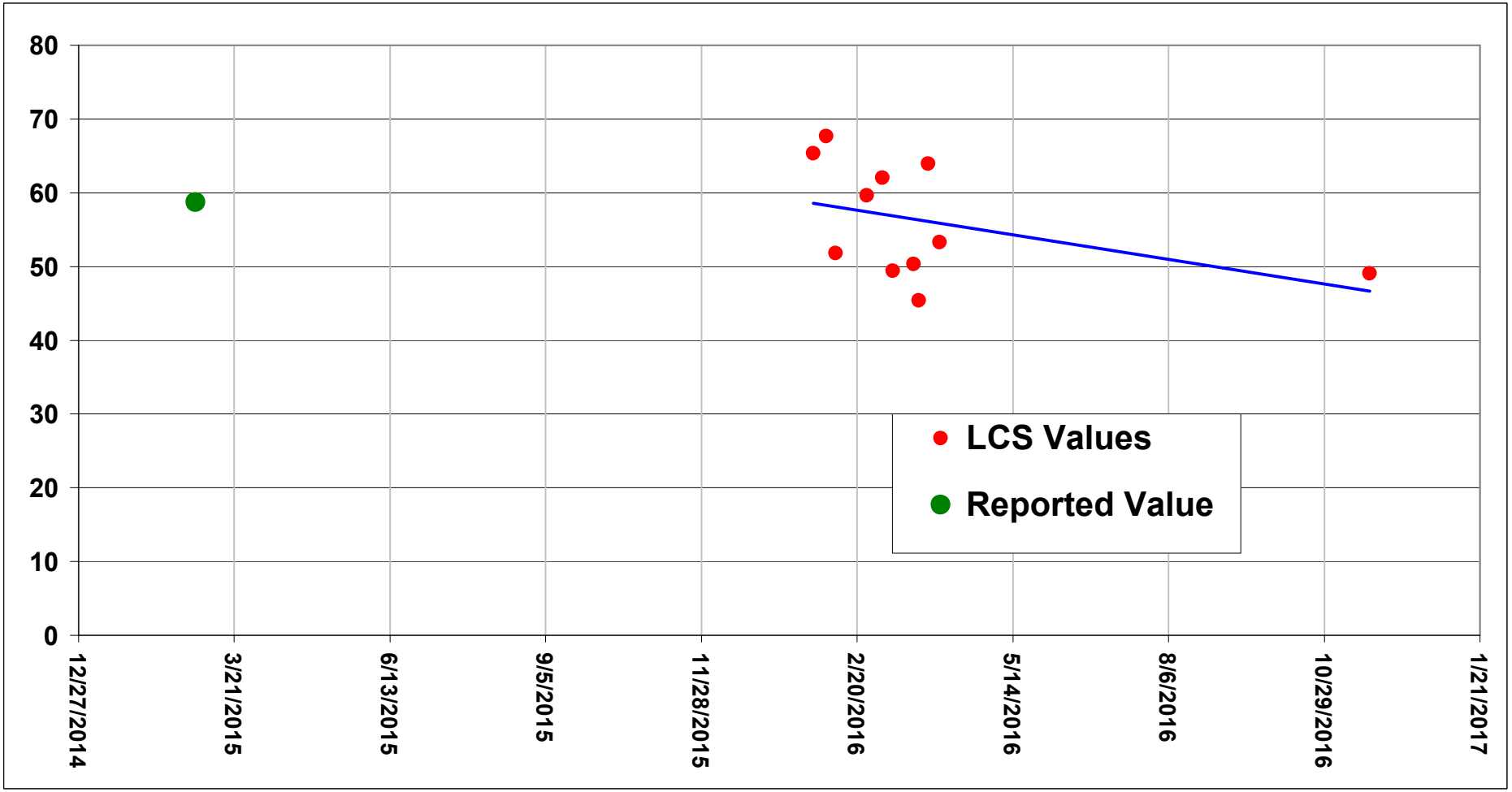
Significance of Slope	0.0516		
No Evidence of Instability over 27 weeks.			
12 Week % Rel. Diff.	NA	Analytical %rsd	8.78%
		Analytical Variance Flag	sr OK
		Should be > 1	1.65
Sigma PT (%RSD)	19.63%	(Participants)	
Allowed Var , 33% σ PT (A)	4506.246		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	42

Vitamin A 201527 Chick Starter, Medicated U/kg



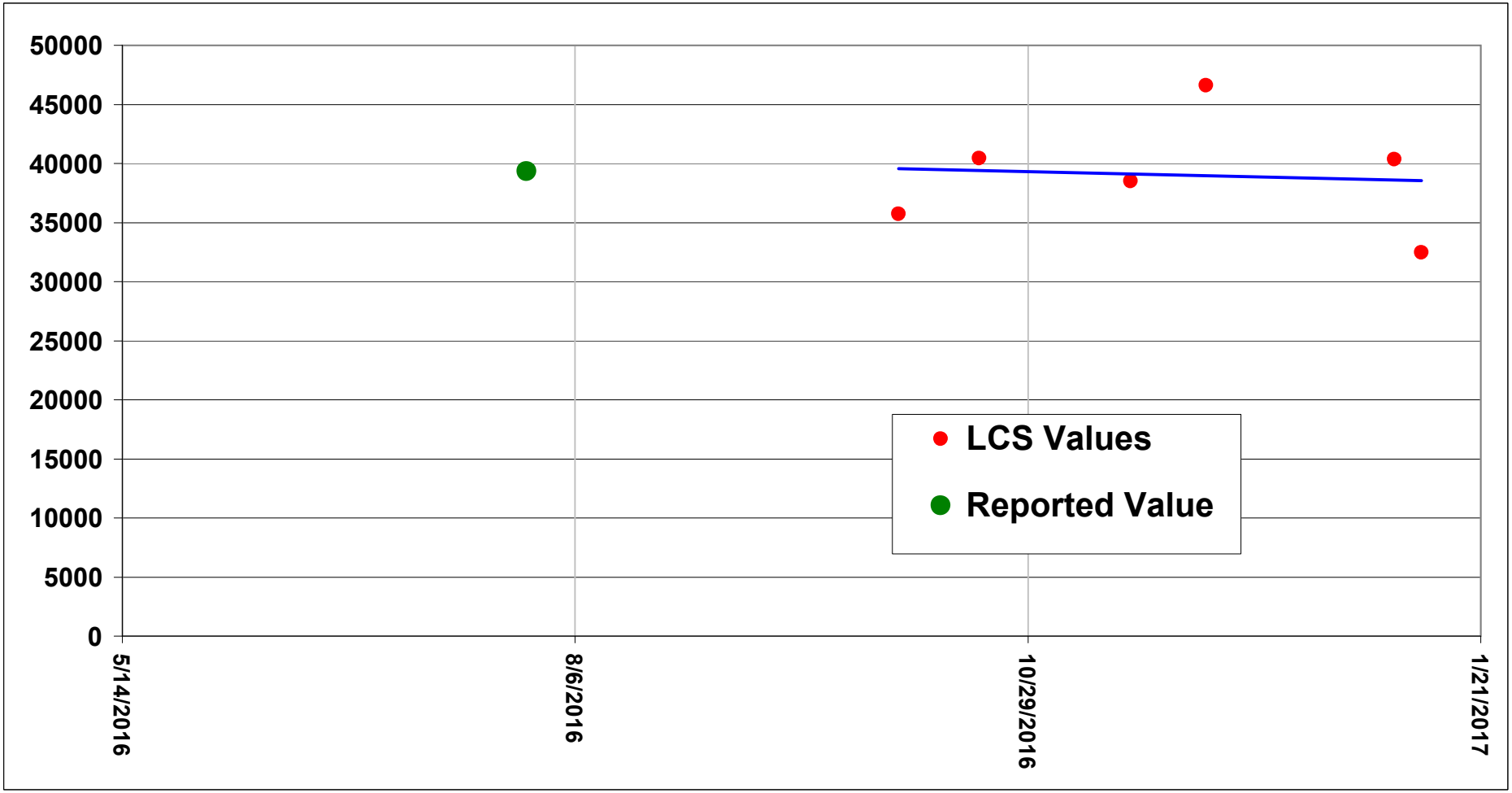
Significance of Slope	0.8357		
No Evidence of Instability over 36 weeks.		Analytical %rsd	18.27%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr Suspect
		Should be > 1	0.83
Sigma PT (%RSD)	24.23%	(Participants)	
Allowed Var , 33% σ PT (A)	1101.818		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	17

Vitamin A 201591 Barley KU/kg



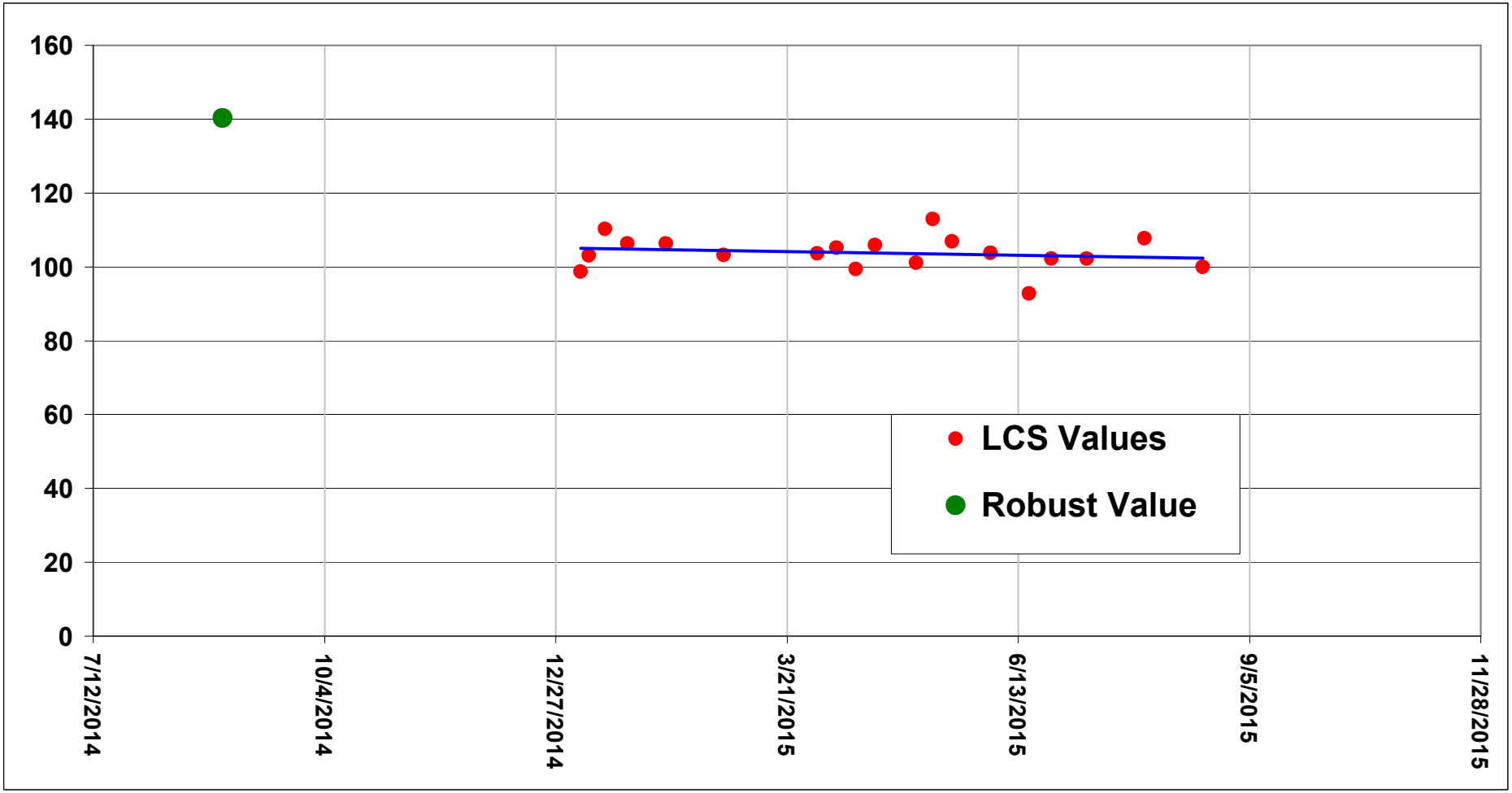
Significance of Slope	0.1916		
No Evidence of Instability over 42 weeks.		Analytical %rsd	13.76%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.51
Sigma PT (%RSD)	29.55%	(Participants)	
Allowed Var , 33% σ PT (A)	4.901		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	48

Vitamin A 201626 Llama Feed U/kg



Significance of Slope	0.8716		
No Evidence of Instability over 14 weeks.		Analytical %rsd	12.30%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr Suspect
		Should be > 1	0.77
Sigma PT (%RSD)	15.66%	(Participants)	
Allowed Var , 33% σ PT (A)	1813.001		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	10

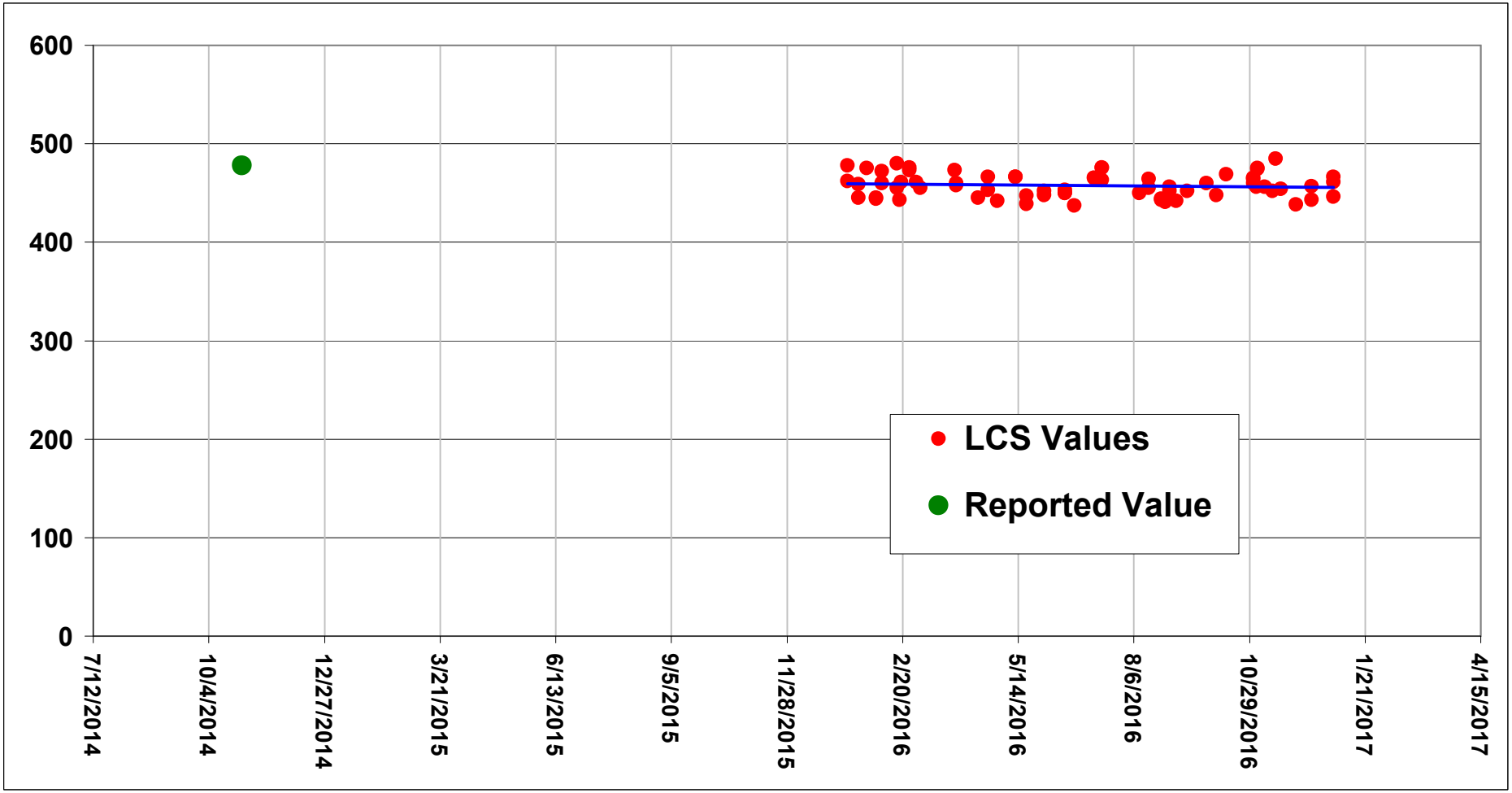
Zinc 201427 Calf Starter / Grower, Medicated % Recovery



Significance of Slope	0.4504		
No Evidence of Instability over 32 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	6.48%	(Participants)	
Allowed Var , 33% σ PT (A)	2.876		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

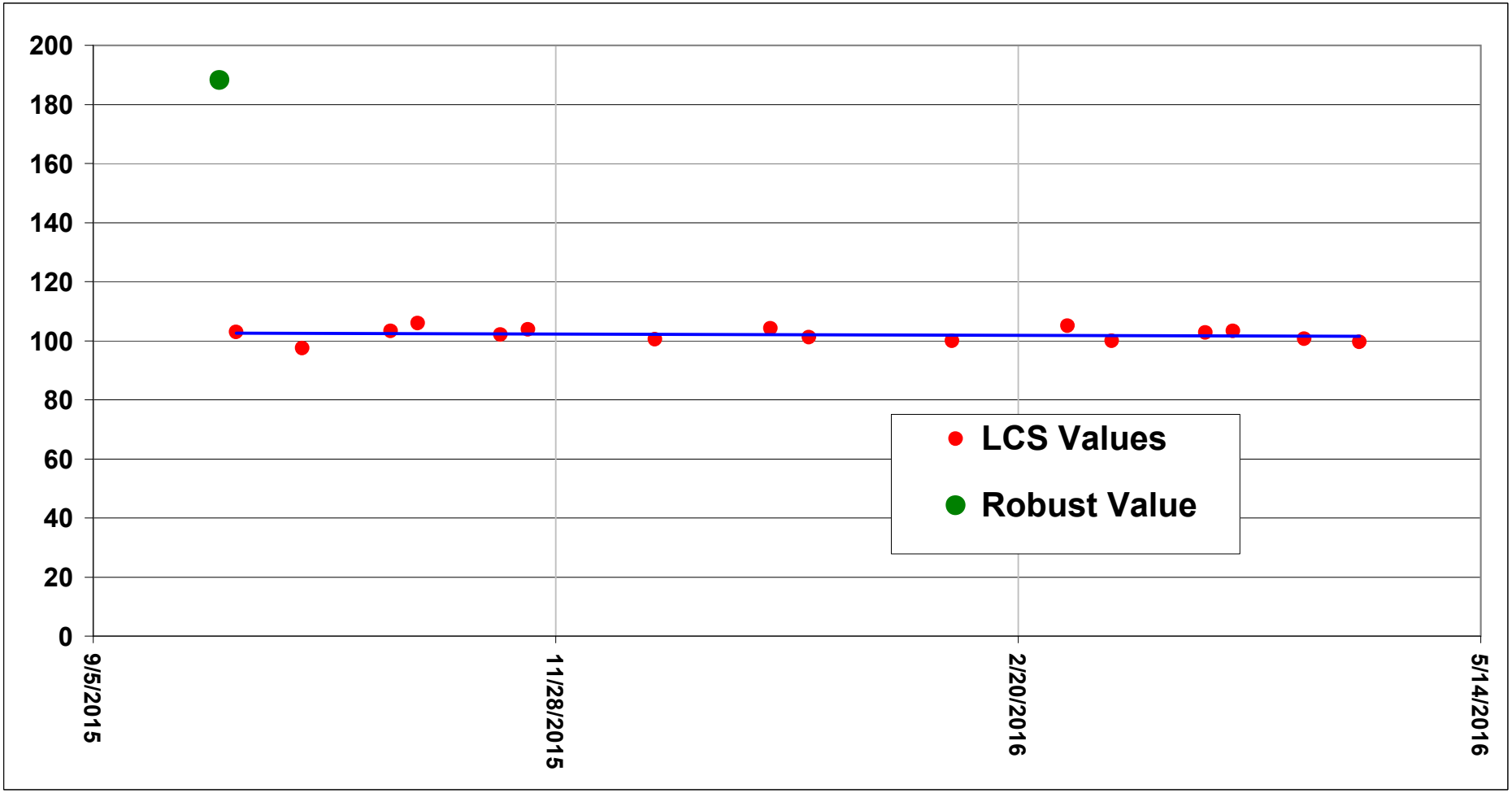
Analytical %rsd	4.33%
Analytical Variance Flag	sr Suspect
Should be > 1	0.99
Weeks from End of Round	19

Zinc 201429 Equine Feed ppm



Significance of Slope	0.4195		
No Evidence of Instability over 50 weeks.		Analytical %rsd	2.56%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.66
Sigma PT (%RSD)	6.36%	(Horwitz)	
Allowed Var , 33% σ PT (A)	9.227		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	63

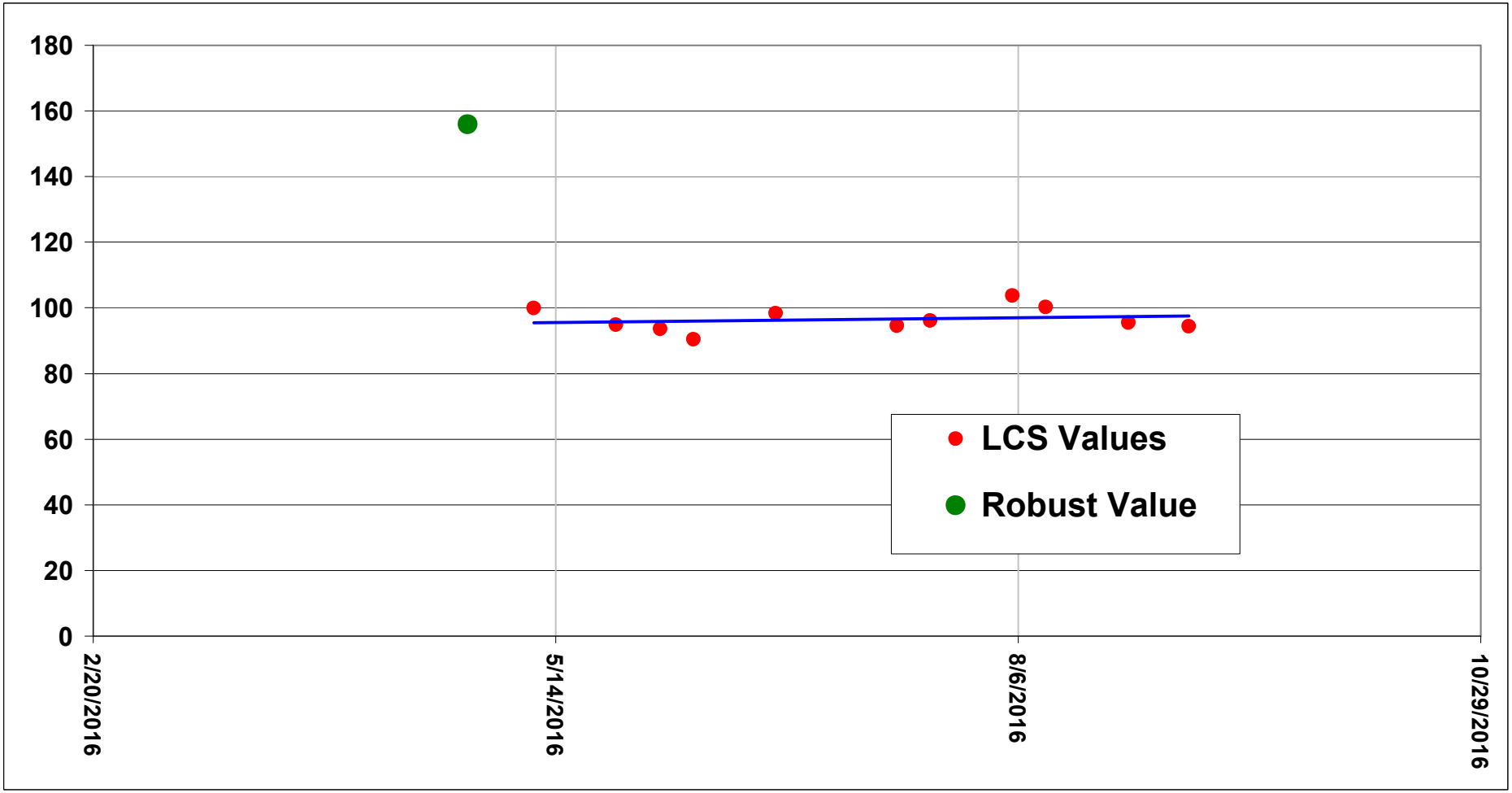
Zinc 201528 Dog Food % Recovery



Significance of Slope	0.5427		
No Evidence of Instability over 29 weeks.			
12 Week % Rel. Diff.	NA		
Sigma PT (%RSD)	7.05%	(Participants)	
Allowed Var , 33% σ PT (A)	4.194		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.		

Analytical %rsd	2.23%
Analytical Variance Flag	sr OK
Should be > 1	2.07
Weeks from End of Round	0

Zinc 201623 Poultry Feed, Medicated % Recovery



Significance of Slope	0.5960		
No Evidence of Instability over 17 weeks.		Analytical %rsd	3.86%
12 Week % Rel. Diff.	NA	Analytical Variance Flag	sr OK
		Should be > 1	1.27
Sigma PT (%RSD)	7.59%	(Participants)	
Allowed Var , 33% σ PT (A)	3.741		
12 Week Difference (B)	0.000		
Stability Decision, Is B < A?	PASS Allowed Var.	Weeks from End of Round	2