Appendix 6: Quality control data for IRMS analyses

An in-house reference material 'Fera casein' (with assigned δ-values generated during the European TRACE-006942 project of FP6), was analysed twice in triplicate within each analytical batch. The performance of the mean values is shown in the Quality control charts, Figures 5.1A to D, for each of the stable isotopes.

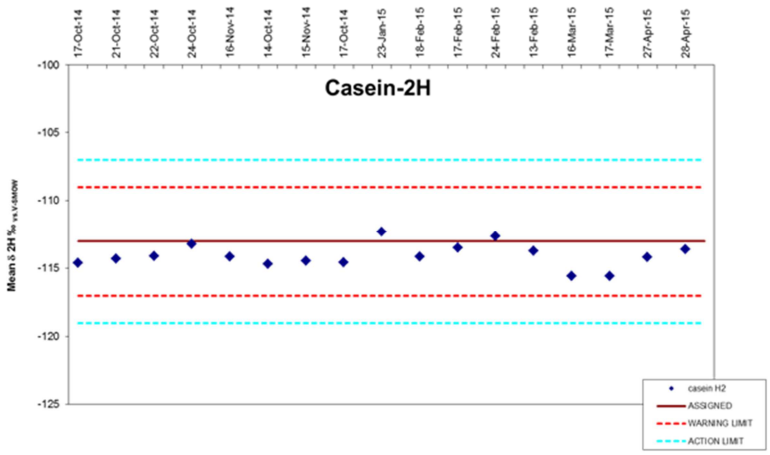

Figure 5.1A: Illustration of QC for 'Fera Casein' with assigned value of -113‰ for hydrogen

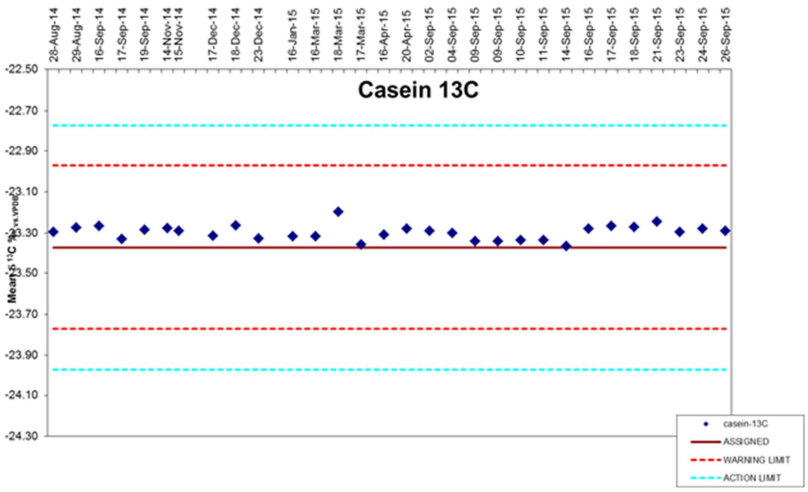

Figure 5.1B: Illustration of QC for 'Fera Casein' with assigned value of -23.37‰ for carbon

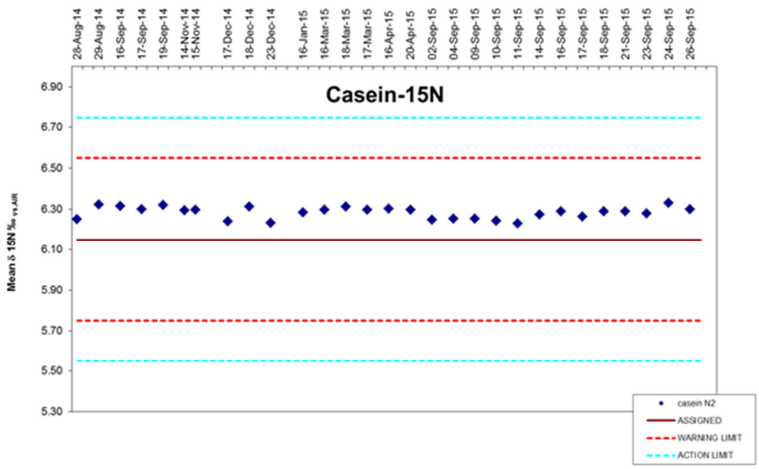

***Figure 5.1C*:** Illustration of QC for 'Fera Casein' with assigned value of 6.15‰ for nitrogen

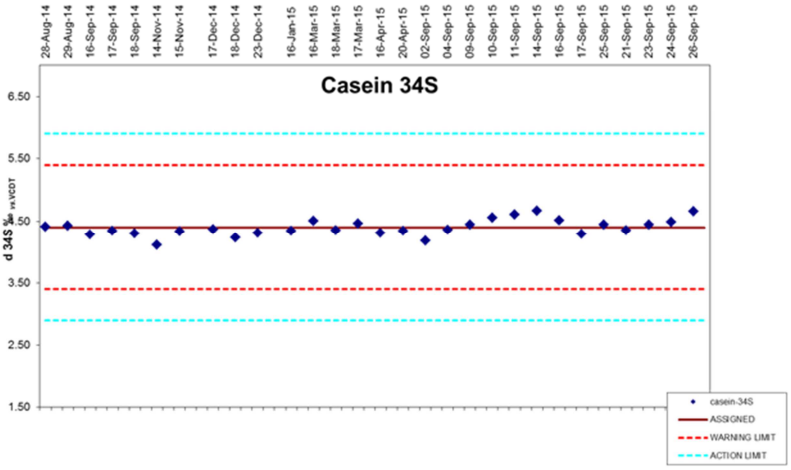

Figure 5.1D: Illustration of QC for 'Fera Casein' with assigned value of 4.4‰ for sulfur

The following acceptance criteria were applied:

- Hydrogen, SD: 2.0‰
- Carbon, SD: 0.2 ‰
- Nitrogen, SD: 0.2 ‰
- Sulfur, SD: 0.5 ‰

These resulted in a warning (2*SD, red line) and action limit (3*SD, turquoise line), non-compliance of the latter would have triggered a repeat analysis of a batch; but all batches conformed.

A number of certified reference materials and additional in-house standards were measured at two occasions throughout the project, to ensure consistency of the data.

Fera and QUB used different defatting procedures, based on the equipment available, to gain the beef protein for the IRMS analyses. To verify this approach, five beef samples were chosen, defatted and analysed by both institutes. The deviations deemed acceptable as shown in Figures 5.2 A and B.

	Defatted by Fera			Defatted by QUB			
Fera sample ID	**15N**	**SD or ABS**	**N**	**15N**	**SD or ABS**	**N**	**Diff.**
S14-030064	8.05	0.05	3	8.53	0.07	3	**0.48**
S14-030070	8.95	0.03	3	9.28	0.06	3	**0.33**
S14-030076	8.06	0.03	3	8.53	0.03	3	**0.47**
S14-030081	6.65	0.01	3	7.30	0.06	3	**0.65**
S14-030086	6.57	0.04	3	7.07	0.02	3	**0.50**
				mean of difference			**0.49**
				SD of difference			**0.11**

	Defatted by Fera			Defatted by QUB			
Fera sample ID	**13C**	**SD or ABS**	**N**	**13C**	**SD or ABS**	**N**	**Diff.**
S14-030064	-26.91	0.01	3	-26.64	0.03	3	**0.27**
S14-030070	-24.96	0.02	3	-24.73	0.02	3	**0.23**
S14-030076	-24.91	0.01	3	-24.64	0.02	3	**0.27**
S14-030081	-25.61	0.03	3	-25.35	0.02	3	**0.26**
S14-030086	-25.90	0.03	3	-25.64	0.04	3	**0.26**
				mean of difference			**0.26**
				SD of difference			**0.02**

	Defatted by Fera			Defatted by QUB			
Fera sample ID	**34S**	**ABS**	**N**	**34S**	**ABS**	**N**	**Diff.**
S14-030064	12.92	0.01	2	13.18	0.21	2	**0.26**
S14-030070	6.57	0.05	2	6.57	0.01	2	**0.00**
S14-030076	4.68	0.12	2	4.54	0.03	2	**0.14**
S14-030081	9.01	0.02	2	8.65	0.18	2	**0.36**
S14-030086	6.46	0.11	2	6.47	0.05	2	**0.01**
				mean of difference			**0.15**
				SD of difference			**0.16**

Figure 5.2A: Comparison of CNS data, Fera only.

	samples measured at Fera							samples measured at QUB						
	Defatted by Fera			Defatted by QUB				Defatted by Fera			Defatted by QUB			
Fera sample ID	2H	SD or ABS	N	2H	SD or ABS	N	Diff.	2H	SD or ABS	N	2H	SD or ABS	N	Diff.
S14-030064	-102.1	0.3	3	-102.0	1.8	3	**0.2**	-99.0	4.2	3	-96.0	4.1	3	**2.9**
S14-030070	-100.8	0.8	3	-101.0	0.9	3	**0.1**	-100.2	3.5	3	-96.4	3.0	3	**3.8**
S14-030076	-97.9	1.2	3	-101.1	0.9	3	**3.2**	-97.7	5.1	3	-99.6	3.3	3	**1.9**
S14-030081	-100.0	0.2	3	-101.5	1.7	3	**1.5**	-97.8	3.0	3	-100.8	2.2	3	**3.0**
S14-030086	-108.4	1.1	3	-105.7	0.6	3	**2.7**	-103.4	2.2	3	-106.4	0.7	3	**3.0**
				mean of difference			1.5				mean of difference			2.9
				SD of difference			1.4				SD of difference			0.7

Figure 5.2B: Comparison of hydrogen data.