Monitoring residue concentrations in milk from farm and throughout a milk powder manufacturing process

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Short title: **Milk quality from farm to milk powder**

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SUPPLEMENTARY FILE

Materials & Methods

Comparison between the residue levels in farm bulk tanks in mid- and late-lactation

In late-lactation (November 2016), the average (± SD) milk volume that was stored in each BT of the 67 farms during sampling was 1,683 ± 1,031 L (range: 125 to 4,519 L), which were stored for an average (± SD) of 34 ± 15 h, at 3.3 ± 1.2 ˚C.

Quantification of trichoromethane

In each sample vial, 2 mL of milk or reconstituted SMP were added with 5 µL of internal standard and 5 µL of of ethanol. The internal standard consisted of a solution prepared using 2-bromo-1-chloropropane and ethanol (0.2 mg/ mL). Samples were placed on an autosampler tray (CTC analytics Combi-pal; CTC Analytics AG Industriestrasse 20 CH-4222, Zwingen, Switzerland) and were incubated for 15 min at 80 ˚C and agitated at 750 rpm. Samples were injected (500 µL) into an Agilent 19095J-121LTM column (10 m x 0.53 mm x 2.65 µm; Agilent Technologies, Santa Clara, California, USA) with a heated gas-tight syringe (90 ˚C). Helium was used as the carrier gas, and the column temperature was kept at a constant temperature of 200 ˚C, which decreased to 70 ˚C in the end of analysis.

Quantification of total iodine

In each vial, 1:1 (w/v) solutions of sample and 5% tetramethyl-ammonium hydroxide (TMAH) extraction solution were added and gently swirled. Vials were placed in an oven at 90 ˚C for 3 h and afterwards they were allowed to cool. The standards used for the calibration consisted of solutions of iodine with 0.5 mL of a Tellurium solution (1,000 µg/ mL), which contained 1% TMAH.

Table S1. Comparison of mean trichloromethane (TCM) concentrations measured in each collection tanker (CT: 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11) during mid-lactation and those predicted (± standard error; S.E.) from the combined farm samples in each CT.

CT number	Number of farms	Total volume per tanker (L)	Mean (± SD) volume measured per farm (L)	Mean TCM concentration of each CT (mg/ kg)	Predicted TCM concentration (weighted means ± S.E.)† (mg/ kg)	95% CI‡		Mean TCM concentration of each CT covered by predicted C.I.
						LCL	UCL	
1	4	23771	5,943 ± 1,271	0.0015	0.0014 ± 0.0009	0.0000	0.0043	Yes
2	5	26503	5,301 ± 2,385	0.0008	0.0005 ± 0.0002	0.0000	0.0011	Yes
3	6	29122	4,854 ± 1,763	0.0012	0.0009 ± 0.0003	0.0001	0.0016	Yes
4	6	23780	3,963 ± 2,683	0.0012	0.0009 ± 0.0003	0.0002	0.0016	Yes
5	8	27585	3,448 ± 2,214	0.0008	0.0004 ± 0.0001	0.0002	0.0005	Yes
6	7	28628	4,090 ± 1,208	0.0011	0.0008 ± 0.0004	0.0000	0.0018	Yes
7	7	27188	3,884 ± 2,064	0.0006	0.0004 ± 0.0001	0.0002	0.0006	Yes
8	7	28470	4,067 ± 2,437	0.0007	0.0004 ± 0.0002	0.0001	0.0008	Yes
9	2	27147	13,574 ± 11,312	0.0010	0.0007 ± 0.00004	0.0002	0.0012	Yes
10	5	25248	5,050 ± 3,877	0.0007	0.0003 ± 0.0001	0.0000	0.0006	Yes
11	10	28561	2,856 ± 1,764	0.0008	0.0005 ± 0.0001	0.0003	0.0008	Yes

† Weighted means were calculated considering the volume of milk supplied by each farm or by each CT.

‡ Confidence interval (CI), lower (LCL) and upper (UCL) confidence limits.

Table S2. Comparison of mean trichloromethane (TCM) concentrations measured in the whole milk silo (WMS) during mid- and late-lactation and those predicted (± standard error; S.E.) from the combined collection tankers (CTs) samples.

	Mean TCM concentration of the WMS (mg/ kg)	Mean (± SD) volume measured per CT (L)	Predicted TCM concentration (weighted means ± S.E.)† (mg/ kg)	95% CI‡		Mean TCM concentration of WMS covered by predicted C.I.
				LCL	UCL	
Mid-lactation	0.0009	26,909 ± 1,902	0.0007 ± 0.00009	0.0005	0.0009	Yes
Late-lactation	0.0018	24,357 ± 3,768	0.0019 ± 0.0002	0.0014	0.0024	Yes

† Weighted means were calculated considering the volume of milk supplied by each CT.

‡ Confidence interval (CI), lower (LCL) and upper (UCL) confidence limits.

Table S3. Comparison of mean iodine concentrations measured in each collection tanker (CT: 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11) during mid-lactation and those predicted (± standard error; S.E.) from the combined farm samples in each CT.

CT	Number of farms	Mean (± SD) volume measured per farm (L)	Total volume per CT (L)	Iodine concentration measured in each CT sample (µg/ L)	Predicted iodine concentrations (weighted means ± SE) (µg/ L) †	95% CI ‡		Mean iodine concentration of each CT covered by predicted CI
						LCL	UCL	
1	4	5,943 ± 1,271	23,771	83.9	89.2 ± 21.8	19.8	158.6	Yes
2	5	5,301 ± 2,385	26,503	81.8	90.0 ± 23.8	23.9	156.2	Yes
3	6	4,854 ± 1,763	29,122	120.0	117.9 ± 45.6	0.6	235.3	Yes
4	6	3,963 ± 2,683	23,780	58.3	61.2 ± 8.5	39.3	83.7	Yes
5	8	3,448 ± 2,214	27,585	125.9	141.0 ± 27.8	75.4	206.7	Yes
6	7	4,090 ± 1,208	28628	138.4	144.1 ± 55.7	7.9	280.3	Yes
7	7	3,884 ± 2,064	27188	112.0	116.7 ± 15.7	78.4	155.1	Yes
8	7	4,067 ± 2,437	28470	76.3	82.9 ± 20.9	31.6	134.1	Yes
9	2	13,574 ± 11,312	27147	390.8	335.7 ± 91.6	0	1,500	Yes
10	5	5,050 ± 3,877	25248	202.9	282.7 ± 121.2	0	619.7	Yes
11	10	2,856 ± 1,764	28561	80.0	101.7 ± 12.1	74.3	129.1	Yes

† Weighted means were calculated considering the volume of milk supplied by each farm.

‡ Confidence interval (CI), lower (LCL) and upper (UCL) confidence limits.

Table S4. Comparison of mean iodine concentrations measured in the whole milk silo (WMS) during the mid- and late-lactation periods and those predicted (± standard error; S.E.) from the combined collection tankers (CTs) samples.

	Mean (± SD) iodine concentration of the WMS (µg/ L)	Mean (± SD) volume measured per CT (L)	Predicted iodine concentration (weighted means ± SE) (µg/ L) †	95% CI ‡		Mean iodine concentration of the WMS covered by predicted CI
				LCL	UCL	
Mid-lactation	135.5 ± 7.6	26,909 ± 1,902	134.2 ± 28.3	71.0	197.3	Yes
Late-lactation	419.0 ± 2.8	24,357 ± 3,768	421.4 ± 50.5	308.8	534.0	Yes

† Weighted means were calculated considering the volume of milk supplied by each CT.

‡ Confidence interval (CI), lower (LCL) and upper (UCL) confidence limits.

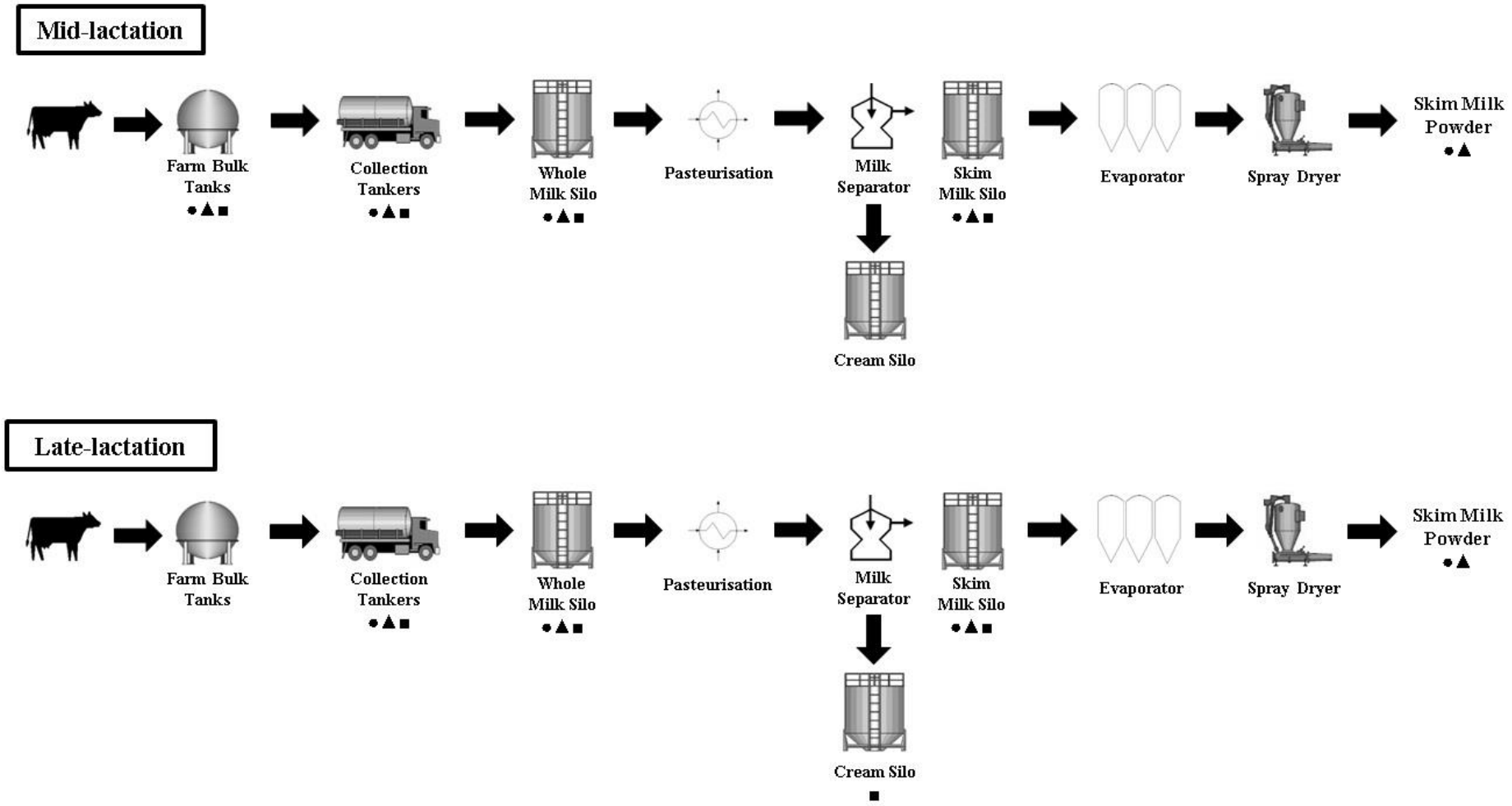

Figure S1. Milk supply chain and manufacturing process for conversion to low-heat skim milk powder, conducted in the mid- and late-lactation periods. The sampling points for chlorate (CHLO) and perchlorate (PCHLO), iodine and trichloromethane (TCM) are indicated with a ●, ▲and ■, respectively.