

Table S1. Manufacture characteristics of the studied Spanish sheep cheese.

		I-CL	T-CL
Production location		<i>Castilla-y-León</i> , Spain	<i>Castilla-y-León</i> , Spain
Elaboration		Big industries under the regulations of the PGI	Small/familiar cheeseries under artisanal procedure
Commercial designation		Castellano sheep cheese	Artisanal/Traditional Castellano sheep cheese
Sheep breed		Castellana & Churra	Castellana & Churra
Milk pre-treatment		Raw	Raw
Starter culture		Homofermentative mesophilic cultures (LAB)	Homofermentative mesophilic cultures (LAB)
Rennet		Recombinant chymosin	Natural rennet from lambs
Coagulation	T (°C)	32	28 - 32
	Time (min)	35 - 40	30 - 45
Cutting curd		Automated process in continuous	Semi-continuous process
	Grain size (mm)	5 - 8	5 - 10
Stirring		Automated process in continuous	Manual discontinuous
Pre-drainage of whey		Automated process in continuous: stirring and bombing the whey milk	By soft draining
Cooking/Scalding	T (°C)	35 - 38	35 - 40
	Time (min)	40	30 - 40
Pre-pressing		Automated process in continuous: in blocks, according to the mold size	Manual: according to curdling vats
	T (°C)	30 - 32	30 - 32
	Time (min)	30	30
Molding		Manual	Manual
Pressing		Automated process	Automated process
	Time (h)	10 - 12	10
Unmolding		Automated process	Manual
Salting		Soaking in brine (0.2 g NaCl/mL)	Soaking in brine (0.2 g NaCl/mL)
	T (°C)	10	10 - 15
	Time (h)	24 - 30	24 - 32
Ripening		Automated process in rooms with automated control of temperature and relative humidity.	Rooms with natural conditions
	T (°C)	10	10 - 12
	Humidity (%)	75 - 80	75 - 80
	Time (d)	- Minimum of 60 d for cheeses heavier than 1.5 kg. - Semi-cured cheese: 60 - 90 d. - Cured cheese: 180 d.	

I-CL = pieces produced in a large industry in *Castilla y León*; T-CL = manufactured according to a traditional procedure in small cheesery in *Castilla y León*. LAB = lactic acid bacteria. T = temperature (°C). RH = relative humidity (%).

Table S2. Studied range and selected values for the optimization of the NMR parameters.

	Studied range	Selected value	1D experiment
Temperature (°C)	4-25	25	1D-NOESY, T_2 filter (CPMG), DIFF
Spinning frequency (kHz)	4-6	6	
Spectrum Width (ppm)	16.7-25.0	25	

Figure S1. Example of a ^1H , ^1H -COSY spectrum for the structural elucidation of metabolites.

