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SAMPLING PROCEDURES APPLICABLE FOR THE QUALITY ASSURANCE SYSTEMS IN THE STARCH INDUSTRY

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Abstract

Sampling is one of the most important factors influencing the analysis results. When the results of several analyses are used to develop and implement a monitoring system which must assure the product quality and food-safety, this becomes a primary element. As an analysis result can be used in the development of such system, the sampling should be as accurate as possible. The sampling methods are various and depend in principal by the sampled material, the material state of aggregation, physical and chemical properties and last but not least, by the presentation mode. At this moment, the sampling procedures applicable in the starch industry are presented in several standards and have a general description based on product categories (e.g.: ISO 24333:2009 Cereals and cereal products − Sampling). For the industry, however, these general guidelines are sometimes difficult to apply as such. This paper aims to review the sampling procedures applicable in the starch industry and to create specific sampling method guidelines developed for a starch factory in order to create an optimal monitoring system capable to assure the quality and food-safety for products and by-products.

Key words: *food-safety, quality, sampling, starch.*

INTRODUCTION

Sampling is one of the most important factors influencing the analysis results (Alexander *et al.* 2007; Thomas *et al*., 2012). The sample has to be big enough and representative for whole batch. The sample size is proportional to the batch size and depends, in principal on the material type and analysis type (Paakkunainen *et al*., 2009; Tokman N., 2007).

For the development of a monitoring system capable to assure the product quality and food safety the most important step is fixing the optimal and specific sampling procedure.

For the starch industry, excepting intermediary products, there are three types of materials: grains (corn and corn germs), milled products (starch, corn gluten feed and corn gluten meal) and viscous liquids (glucose syrups).

Taking into account all these aspects, this paper presents and compares all the international sampling instructions applicable for the cereals and cereals products in order to establish the specific optimal sampling procedure for the final products in a starch factory.

MATERIALS AND METHODS

In order to establish a specific optimal sampling procedure for the starch industry, we identified the main international sampling instructions included in various standards and regulations as follows: SR EN ISO 24333:2009 Cereals and cereal products − Sampling, Commission regulation (EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs, Commission Regulation (EU) No 836/2011 of 19 August 2011 amending Regulation (EC) No 333/2007 laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo (a) pyrene in foodstuffs, Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed, Commission Directive 2002/63/EC of 11 July 2002 establishing Community methods of sampling for the official control of pesticide residues in and on products of plant and animal origin and repealing Directive 79/700/EEC, Decree ANSVSA No. 27 of 06.06.2011 regarding the approval of hygiene and

microbiological criteria for foodstuffs other than those mentioned in Regulation (EC) no. 2.073/2005 of 15 November 2005 on microbiological criteria for foodstuffs, Commission Regulation (EU) No 619/2011 of 24 June 2011 laying down the methods of sampling and analysis for the official control of feed as regards the presence of genetically modified material for which an authorisation procedure is pending or the authorisation of which has expired.

The international standard SR EN ISO 24333:2009 Cereals and cereal products − Sampling was the starting point and it was adapted by other relevant sampling documents to be found in EU-regulation or specific literature. SR EN ISO 24333:2009 is applicable "for the dynamic or static sampling, by manual or mechanical means, of cereals and cereal products, for assessment of their quality and condition" (SR EN ISO 24333:2009). It is not appropriate for the determination of the presence of adventitious genetically modified material in non-GM products and for microbiological criteria. These omissions would be dealt with only the EU-regulation and specific literature.

The information contained in these documents were interpreted, adapted for the intended use and transposed into a centralised, specific and optimal sampling procedure.

RESULTS AND DISCUSSIONS

For cereals and cereal products we identified seven referential documents which are applicable for the starch industry.

Taking into account the instructions mentioned in these documents and the specific of the starch factory took for example, we centralized the information and issued a single sampling procedure for raw material and final products.

There were identified four categories of analyses: specific for product quality determination, for contaminants identification and determination, microbiological analyses and analyses for non-GMO products.

For contaminants the sampling procedures depend on the type of the contaminant, being different for mycotoxins, heavy metals and pesticides.

In Table 1, we present the documents where sampling instruction can be found. It can be seen that for specific quality analysis the only harmonized and internationally recognized sampling procedure is SR EN ISO 24333:2009. This standard indicates a single sample size (1 to 3 kg) depending on various criteria like the batch size, the minimum number of elementary samples and its size, the sampling method (mechanical or manual) or the packaged unit weight.

The minimum sample size is indicated also for contaminants like ochratoxin A (10 kg), pesticides, heavy metals, dioxin (1 kg), other contaminants (3 kg). To complete these values, the EU regulations establish the sample size depending on the batch size and packaging mode (bulk or packed).

Taking into account the product categories presented in these documents, we split starch products as follows:

- Grains:
  - in bulk:
    - corn
  - corn germs
  - packed:
    - corn germs:
      - in big-bags
- Milled products:
  - in bulk:
    - corn starch
    - corn gluten feed
    - corn gluten meal
  - packed:
    - corn starch:
      - in paper bags of 25 kilos
      - in big-bags
    - corn gluten feed:
      - in big-bags
    - corn gluten meal:
      - in big-bags
- Liquid products:
  - in bulk:
    - glucose syrup
  - packed:
    - glucose syrup:
      - in recipients of 45 and 150 kilos
      - in recipients of 1100 kilos

Table 1. Sampling procedures applicable for starch industry

Product category	**Analysis category**	**Sampling procedures applicable**	
		International standards	**Legislation (EU / RO) in force**
Food products	Specific for quality	SR EN ISO 24333:2009	-
	Contaminants		
	mycotoxins	SR EN ISO 24333:2009	Commission Regulation (EC) No 401/2006
	heavy metals	SR EN ISO 24333:2009	Regulation (EC) No 333/2007
	pesticides	SR EN ISO 24333:2009	Commission Directive 2002/63/EC
	Microbiological	-	Decree ANSVSA 27/2011
	non-GMO	-	2004/787/EC: Commission Recommendation
Feed products	Specific for quality	SR EN ISO 24333:2009	-
	Contaminants		
	mycotoxins	SR EN ISO 24333:2009	Commission Regulation (EC) No 152/2009
	heavy metals	SR EN ISO 24333:2009	Commission Regulation (EC) No 152/2009
	pesticides	SR EN ISO 24333:2009	Commission Directive 2002/63/EC
	dioxine	SR EN ISO 24333:2009	Commission Directive 2002/70/EC
	other contaminants	SR EN ISO 24333:2009	Commission Regulation (EC) No 152/2009; Commission Directive 2002/63/EC
	Microbiological	-	-
	non-GMO	-	2004/787/EC: Commission Recommendation

The analysis categories were split in six categories:
- specific for quality
- mycotoxins
- heavy metals
- pesticides
- microbiological
- non-GMO

For each product and analysis category we established the specific sampling procedure, taking into account the batch size, the minimum number of elementary samples and quantity of the total sample.

For an easier application of this procedure we used an excel file which calculated the sampling frequency depending on the number of packaging units and pallets.

An example for a batch of an approx. 25000 kilos is presented in Tables 2 and 3 for food products and in Tables 4 and 5 for feed products.

The types of analyses performed were grouped in accordance to the sampling procedure similarities, so that the analyses specific for quality and those for mycotoxins and heavy metals were presented together (Tables 2 and 4), while the analyses for the detection of pesticides, microbial contamination and the presence of genetically modified organisms were grouped and included in Tables 3 and 5.

In all these tables the abbreviations mean:

-parameters measured for all types of sampling procedures except those involving microbiological analyses:
- CP: product code
- DA: packed unit weight (kg)
- DP: number of packed units in a pallet
- DL: batch size (kg)
- NP: number of pallets in a batch
- NA: number of packed units in a batch
- FA: sampling frequency / packed unit
- FP: sampling frequency / pallet unit
- NPE: number of incremental samples
- MPE: size of incremental sample (kg)
- PG: size of global sample (kg)
- PL: size of laboratory sample (kg)

-parameters measured for microbiological analyses:
- FP: sampling frequency / pallet unit
- NPT: number of tested samples
- MPT: tested samples weight

-type of food product:
- PB: corn
- AV: bulk starch
- AS: starch packed in 25 kg bags
- AB: starch packed in big-bags
- GV: bulk glucose syrup

- G45: glucose syrup packed in 45 kg recipients
- G15: glucose syrup packed in 150 kg recipients
- G11: glucose packed in 1100 kg recipients
- GrV: bulk germs
- GB: germs packed in big-bags

-type of feed product:

- TV: bulk corn gluten feed
- TB: corn gluten feed packed in big-bags
- FV – bulk corn gluten meal
- FB – corn gluten meal packed in big-bags

The values in the table that are marked with yellow colour and font italic are fixed values established according to EU regulation and taking into account the factory possibilities. These values should not be changed.

The values coloured in green should be introduced by the operator in order to identify the correct values from the white cells. For example, to identify the sampling procedure for a batch of starch packed to bags of 25 kilos it was established according to SR EN ISO 24333:2009 that the size of incremental sample (MPE) should be 0.1 kg, the size of global sample (PG) and the size of laboratory sample should be 1 kg for each one.

As it is known that a bag has 25 kg and a pallet has 30 bags, it was easy to identify that for a batch of 24750 kg, the number of pallets in a batch (NP) is 33 and the number of packed units in a batch (NA) is 990, using the following formulas:

$$NP = \frac{\bar{D}L}{DA \times DP}; NA = DP \times NP$$

Then it was identified that the sampling frequency / packed unit (FA) should be 99, the sampling frequency / pallet unit (FP) should be 3.3 and the number of incremental samples (NPE) should be 10, using the following formulas:

$$FA = \frac{DL \times MPE}{PG \times DA}; FP = \frac{FA}{DP}; NPE = \frac{NP}{FP}$$

For all packed products, food and feed, the sampling frequency for all analyses types, except non-GMO and microbiological analyses, was calculated following the recommendations given by SR EN ISO 24333:2009, taking into account the possibilities of the starch factory (e.g.: only manual static sampling) and considering that there are no significant differences between the sampling methods used for its (Knight and Wilkin, 2010).

For bulk products, food and feed the sampling procedure is applied as shown in Table 2 and using the rules shown in figure 1.

These rules follows the examples presented in SR EN ISO 24333:2009 for products located in trucks: 3, 5, 8 and 10 sampling points.

For quality analyses, in order to determine the sampling frequency / packed unit (FA), the parameters DL (batch size), MPE (size of incremental sample), PG (size of total sample) and DA (packed unit weight) should be known; for the calculation of FA the formula was used:

$$FA = \frac{DL \times MPE}{PG \times DA}$$

The sampling frequency / pallet unit (FP) was after that calculated by using the following formula:

$$FP = \frac{FA}{DP}$$

For the packed products, the number of incremental samples, NPE value, can be also calculated, based on the number of pallets in a batch (NP) and the above calculated FP value, as follows:

$$NPE = \frac{NP}{FP}$$

Table 2. Example of sampling procedure for a 25000 kilos batch of food products for which analyses of quality, mycotoxins and heavy metals are performed

			FOOD PRODUCTS																			
			DL	NP	NA	QUALITY						MYCOTOXINS						HEAVY METALS					
			CP	DA	DP	FA	FP	NPE	MPE	PG	PL	FA	FP	NPE	MPE	PG	PL	FA	FP	NPE	MPE	PG	PL
PB	-	-	25000	-	-	-	-	*8*	*0.4*	*1*	*1*	-	-	*8*	*1.3*	*10*	*10*	-	-	*8*	*0.4*	*1*	*1*
AV	-	-	25000	-	-	-	-	*3*	*0.4*	*1*	*1*	-	-	*3*	*0.4*	*1*	*1*	-	-	*3*	0.33	*1*	*1*
AS	*25*	*30*	24750	33	990	99.0	3.3	10	*0.1*	*1*	*1*	9.9	0.3	*100*	*0.1*	*10*	*10*	330.0	11.0	*3*	0.33	*1*	*1*
AB	*1000*	*1*	25000	25	25	2.5	2.5	10	*0.1*	*1*	*1*	0.3	0.3	*100*	*0.1*	*10*	*10*	8.3	8.3	*3*	0.33	*1*	*1*
GV	-	-	25000	-	-	-	-	*3*	*0.4*	*1*	*1*	-	-	*3*	*0.4*	*1*	*1*	-	-	*3*	0.33	*1*	*1*
G45	*45*	*12*	24840	46	552	110.4	9.2	5	*0.2*	*1*	*1*	5.5	0.5	*100*	*0.1*	*10*	*10*	184.0	15.3	*3*	0.33	*1*	*1*
G15	*150*	*4*	24600	41	164	32.8	8.2	5	*0.2*	*1*	*1*	1.6	0.4	*100*	*0.1*	*10*	*10*	54.7	13.7	*3*	0.33	*1*	*1*
G11	*1100*	*1*	24200	22	22	2.2	2.2	10	*0.1*	*1*	*1*	0.2	0.2	*100*	*0.1*	*10*	*10*	7.3	7.3	*3*	0.33	*1*	*1*
GrV	-	-	25000	-	-	-	-	*8*	*0.4*	*1*	*1*	-	-	*8*	*0.4*	*3*	*3*	-	-	*3*	0.33	*1*	*1*
GB	*800*	*1*	12000	15	15	1.5	1.5	10	*0.1*	*1*	*1*	0.2	0.2	*100*	*0.1*	*10*	*10*	5.0	5.0	*3*	0.33	*1*	*1*

Table 3. Example of sampling procedure for a 25000 kilos batch of food products for which analyses of pesticides, microbiological contamination and non-GMO content are performed

			FOOD PRODUCTS																		
			DL	NP	NA	PESTICIDES						MICROB.			non-GMO						
			CP	DA	DP	FA	FP	NPE	MPE	PG	PL	FP	NPT	MPT	FA	FP	NPE	MPE	PG	PL
PB	-	-	25000	-	-	-	-	*8*	*0.4*	*1*	*1*	-	-	-	-	-	10	*0,5*	5	2,5
AV	-	-	25000	-	-	-	-	*3*	0.33	*1*	*1*	-	*3*	*0.5*	-	-	10	*0,5*	5	2,5
AS	*25*	*30*	24750	33	990	330.0	11.0	*3*	0.33	*1*	*1*	6.6	*5*	*0.5*	31	1	31	*0,2*	5	2,5
AB	*1000*	*1*	25000	25	25	8.3	8.3	*3*	0.33	*1*	*1*	5.0	*5*	*0.5*	5	5	5	*1,0*	5	2,5
GV	-	-	25000	-	-	-	-	*3*	0.33	*1*	*1*	-	*3*	*0.5*	-	-	10	*0,5*	5	2,5
G45	*45*	*12*	24840	46	552	184.0	15.3	*3*	0.33	*1*	*1*	9.2	*5*	*0.5*	23	2	23	*0,2*	5	2,5
G15	*150*	*4*	24600	41	164	54.7	13.7	*3*	0.33	*1*	*1*	8.2	*5*	*0.5*	13	3	13	*0,4*	5	2,5
G11	*1100*	*1*	24200	22	22	7.3	7.3	*3*	0.33	*1*	*1*	4.4	*5*	*0.5*	5	5	5	*1,1*	5	2,5
GrV	-	-	25000	-	-	-	-	*3*	0.33	*1*	*1*	-	*5*	*0.5*	-	-	10	*0,5*	5	2,5
GB	*800*	*1*	12000	15	15	5.0	5.0	*3*	0.33	*1*	*1*	3.0	*5*	*0.5*	4	4	4	*1,3*	5	2,5

For the determination of mycotoxins level the rules imposed by the EU regulations are stricter, therefore the number of fixed values, marked in the table with font italic and yellow colour is bigger than for the other analyses. In corn, as it is known that there is very heterogeneous distribution of the grains, the Commission Regulation (EC) No 401/2006 recommends a Guidance document for the sampling of cereals for mycotoxins, which specifies the impossibility of static sampling for very large batches stored in closed cylindrical silos (Malone *et al*., 2008). The corn used for the starch production in the factory, taken as reference, is stored in closed cylindrical silos of 5000 tonnes capacity. As a solution to apply sampling rules to specific possibilities, we adapted the instruction presented in the same Guidance for batches with reasonable size, as follows: if for a 25 tonnes batch of grains the sampling procedure requires to release into a recipient a quantity of 50 to 100 kilos and to take the sample in a representative way from this 50-100 kilos (meaning five incremental samples of 2 kilos to obtain a global sample of 10 kilos), then for a 5000 tonnes batch the sampling procedure may require to release into a recipient a quantity of 25 tonnes and to take the sample according to SR EN ISO 24333:2009 (meaning 8 incremental samples of approx. 1.3 kilos to obtain a total sample of 10 kilos) from this quantity located in a truck (Figure 1). The calculation was made by using the rule of three and the real result was between 10 and 20 tones. For this adaptation we consider also the literature. Hallier *et al*. showed in 2011 that the principal source of variability in the mycotoxin analysis result is the grain sampling, due to the heterogeneous repartition in the grain lots. It can decrease with the sample size increasing and can increase proportional with mycotoxin

concentration (Whitaker, 2003; 2006). For the cereal products, food and feed, although the heterogeneity is lower than in the case of grains, it is also very important to collect a representative sample (Duarte *et al*., 2010; Stroka *et al*., 2004). For bulk products (starch, glucose syrup, germs, corn gluten feed and corn gluten meal) we consider only SR EN ISO 24333:2009, but for packed products we take into consideration both Commission Regulation (EC) No 401/2006 (for food products) and Commission Regulation (EC) No 152/2009 (for feed products) regarding the number of elementary samples and the sample size and, according to that, we calculate the sampling frequency given by SR EN ISO 24333:2009 and the number of incremental samples by using the formula:

$$NPE = \sqrt{NA}$$

For heavy metals in food the sampling frequency was established according to SR EN ISO 24333:2009, considering the minimum sample size specified by Regulation (EC) No 333/2007 (1 kilo comprising 3 incremental samples). The formula used is:

$$MPE = \frac{PG}{NPE}$$

For heavy metals in feed it was applied the same sampling procedure as for mycotoxins in feed and in addition MPE was calculated as for heavy metals in food.

The sampling procedure for pesticides was issued considering the sample size and the number of elementary samples specified by Commission Directive 2002/63/EC for bulk and packed batches lower than 50 tonnes and calculating the sampling frequency according to SR EN ISO 24333:200 and MPE according to the formula used for heavy metals in food.

Table 4. Example of sampling procedure for a 25000 kilos batch of feed products for which analyses of quality, mycotoxins and heavy metals are performed

			FEED PRODUCTS																			
			DL	**NP**	**NA**	**QUALITY**						**MYCOTOXINS**						**HEAVY METALS**					
			CP	**DA**	**DP**	**FA**	**FP**	**NPE**	**MPE**	**PG**	**PL**	**FA**	**FP**	**NPE**	**MPE**	**PG**	**PL**	**FA**	**FP**	**NPE**	**MPE**	**PG**	**PL**
TV	-	-	25000	-	-	-	-	*3*	*0.4*	*1*	*1*	-	-	*8*	*0.5*	*4*	*1*	-	-	*8*	0.5	*4*	*1*
TB	*900*	*1*	24300	27	27	2.7	2.7	10	*0.1*	*1*	*1*	0.7	0.7	*5.2*	*0.1*	*4*	*1*	5.2	5.2	5	0.8	*4*	*1*
FV	-	-	25000	-	-	-	-	*3*	*0.2*	*1*	*1*	-	-	*8*	*0.5*	*4*	*1*	-	-	*8*	0.5	*4*	*1*
FB	*1000*	*1*	4000	4	4	0.8	0.8	5	*0.2*	*1*	*1*	0.1	0.1	*4*	*0.1*	*4*	*1*	1.0	1.0	4	1	*4*	*1*

Table 5. Example of sampling procedure for a 25000 kilos batch of feed products for which analyses of pesticides, microbiological contamination and non-GMO content are performed

			FEED PRODUCTS																		
			DL	**NP**	**NA**	**PESTICIDES**						**MICROB.**			**non-GMO**						
			CP	**DA**	**DP**	**FA**	**FP**	**NPE**	**MPE**	**PG**	**PL**	**FP**	**NPT**	**MPT**	**FA**	**FP**	**NPE**	**MPE**	**PG**	**PL**
TV	-	-	25000	-	-	-	-	*3*	0.33	*1*	*1*	-	-	-	-	-	10	*0,5*	5	2,5
TB	*900*	*1*	24300	27	27	9.0	9.0	*3*	0.33	*1*	*1*	27.0	*1*	*0.5*	5	5	5	*1,0*	5	2,5
FV	-	-	25000	-	-	-	-	*3*	0.33	*1*	*1*	-	-	-	-	-	10	*0,5*	5	2,5
FB	*1000*	*1*	4000	4	4	4.0	4.0	*1*	1.00	*1*	*1*	4.0	*1*	*0.5*	2	2	2	*2,5*	5	2,5

As for microbiology criteria of starch the only reference document is Decree ANSVSA 27/2011 we applied it as sampling instruction for all the products (food and feed) to be applied when required. For the packed products the sampling frequency / pallet unit was calculated with the following equation:

$$FP = \frac{NP}{NPT}$$

For non-GMO analysis we identified the specific sampling procedure taking into account the Commission Recommendation 2004/787/EC which specifies that "In case of lots smaller than 50 tonnes, the size of the bulk sample

should be 5 kg." According to this instruction, we calculate the number of the incremental samples which should be taken from bulk products, as follows:

$$NPE = \frac{PG}{MPE}$$

For packed products CEN/TS 15568:2006 contains relevant information about sampling strategies and it was easy to use, because of the number of incremental samples is given by the square root of total number of packages (Sisea, 2009). According to this indication and considering the global sample to be 5 kg as it is indicated in the Commission Recommendation 2004/787/EC, the values of NPE, MPE, FA, FP were calculated as follows:

$$NPE = \sqrt{NA} \quad ; \quad FA = \frac{NA}{NPE}$$

$$MPE = \frac{PG}{NPE} \quad ; \quad FP = \frac{FA}{DP}$$

For both, bulk and packed products, the laboratory sample (PL) is half of PG.

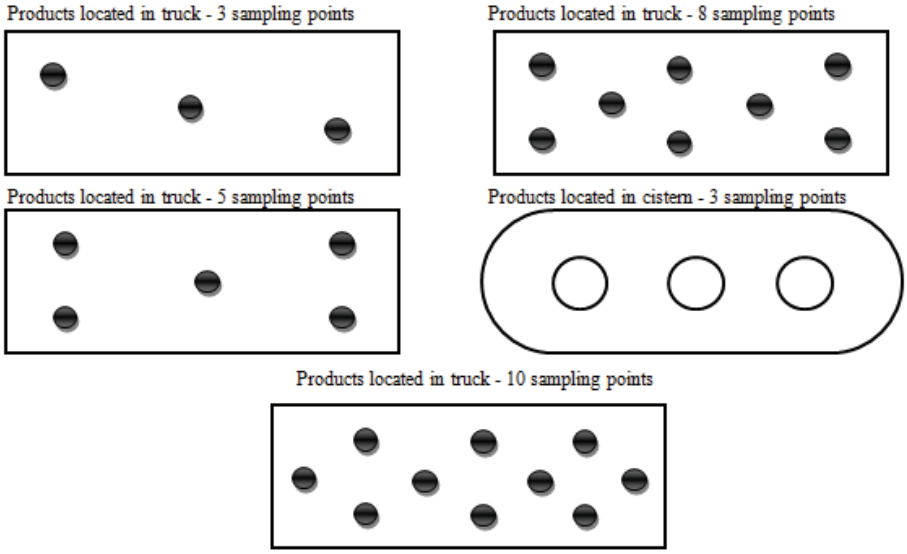

Figure 1. Examples of sampling distribution points

CONCLUSIONS

The development of a specific sampling procedure in the starch industry depends on the factory's specificity, including infrastructure possibilities, activities and analysis type.

Although the standards and legislation offer a general description of the sampling instructions, they can be applied in a harmonized way, on condition that the production process and products characteristics are known very well.

The sampling procedures to be applied for the analyses of pesticides, microbiological contamination and GMO-presence detection for packed products of food and feed are similar. However, differences appear when the analyses of mycotoxin presence should be performed, the rules for sampling for these types of analyses being more strict.

For food and feed bulk products the rules from regulations and standards are more specific and, that for the sampling procedure is simpler than for packed products.

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