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Original Research

Effect of Vegetable Freezing and Preparation of Frozen Products for Consumption on the Content of Lead and Cadmium

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Abstract

The aim of our study was to determine the levels of lead and cadmium in fresh vegetables, in vegetables after pre-freezing (blanching or boiling), and in frozen products after a 12-month period of storage at -30°C and prepared for consumption (by boiling samples blanched before freezing, and by defrosting and heating in a microwave oven samples boiled before freezing). Analyses were carried out in four groups of vegetables perfectly suitable for freezing, namely brassicas (broccoli, Brussels sprouts, and green and white cauliflower), leafy vegetables (kale, New Zealand spinach, and spinach), root vegetables (celeriac, carrot, parsnip, and red beet), and three species of leguminous vegetables (seeds of broad bean, and pea, and French bean pods). Vegetables of the studied species contained 1-86% and 3-26% of the European admissible levels of lead and cadmium, respectively. Technological processing before freezing, freezing and frozen storage, and culinary preparation of frozen products for consumption did not significantly affect the level of lead. However, boiling reduced the content of cadmium in brassicas, in leafy vegetables, apart from New Zealand spinach, and in root vegetables, apart from carrot and red beet.

Keywords: lead, cadmium, vegetables, blanching or boiling, frozen storage, culinary treatments

Introduction

Vegetables are necessary constituents of the human diet [1] that provide important nutritional constituents and also numerous biologically active compounds [2, 3]. On the other hand, vegetables contain compounds which are undesirable in human diet, among them chiefly nitrates and nitrites, and also such metals as lead and cadmium. The degree of accumulation of lead and cadmium in vegetables depends on various factors [4-7]. Hence their content varies according to the species, cultivar, usable part of the plant and the location and conditions of cultivation. A great number of studies deal with the level of lead and cadmium in vegetables depending on these factors [4, 8-11]. However, the effect of technological and culinary processing on the concentration of these metals in products prepared for consumption has not been sufficiently reported in literature.

Because of their seasonal nature, vegetables are processed to lengthen their availability on the market. Freezing is the best method of processing and, as the statistical data show, the supply of frozen vegetables is steadily increasing. Owing to the widespread interest of consumers in ready-to-eat food, easily prepared dishes (which only have to be heated before consumption) are becoming increasingly popular [12]. The products which meet these criteria are frozen vegetables; however, the production

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process should be modified so as to obtain the proper consistency of the vegetable before freezing.

The aim of this study was to measure the level of lead and cadmium in fresh vegetables, in vegetables after pre-freezing treatments or processing (blanching and boiling) and in products after a 12-month period of frozen storage prepared for consumption (by boiling samples blanched before freezing, and by defrosting and heating in a microwave oven samples boiled before freezing).

Material and Methods

Material

The studied material included of four species of brassicas: Brussels sprouts – *Brassica oleracea* var. *gemmifera* L. (Lunet F_1 cv.); broccoli – *Brassica oleracea* var. *italica* Plenck (Lord F_1 cv.); green cauliflower – *Brassica oleracea* var. *botrytis* L. (Trevi F_1 cv.) and white cauliflower – *Brassica oleracea* var. *botrytis* L. (Planita F_1 cv.); three leafy species: kale – *Brassica oleracea* L. var. *acephala* D.C. (Winterbor F_1 cv.); New Zealand spinach – *Tetragonia expansa* Murr., and spinach – *Spinacia oleracea* L. (Greta F_1); four root species: celeriac – *Apium graveolens* L. (Dukat cv.); carrot – *Daucus carota* L. (Koral cv.); parsnip – *Pastinaca sativa* L. (Fagot cv.); and red beet – *Beta vulgaris* L. (Czerwona Kula cv.); and three species of leguminous vegetables, namely broad bean – *Vicia faba* L. var. *major* (Windsor Biały cv.), French bean – *Phaseolus vulgaris* L. (Delfina cv.), and pea – *Pisum sativum* L. (Consul cv.). The seeds of broad bean and pea were at the stage of milk-wax maturity.

The content of Pb and Cd was determined in fresh vegetables (A), after blanching (B), after boiling in 2% (w/w.) brine of table salt to consumption consistency (C), and in frozen products after 12 months of storage at -30°C and then prepared for consumption. Frozen products from B were boiled in brine, thus yielding D, and frozen products from C were defrosted and heated in a microwave oven, yielding E.

Cultivation Measures

The vegetables were grown in an experimental field of the department carrying out the presented study. The field was in good horticultural condition, on the western outskirts of Kraków in southern Poland. The cultivation was conducted on brown soil with the mechanical composition of silt loam in the third year after manure fertilization. The pH of the soil in H_2O was 7.08, with humus content of 1.66%, nitrogen 24 mg NO_3 dm^{-3}, phosphorus 53 mg dm^{-3}, potassium 101 mg dm^{-3} and calcium 1020 mg dm^{-3}.

The fertility of the soil and the nutritional requirements of the crops have been taken into account, doses of mineral fertilizer were applied as given in Table 1. Cultivation measures included sprinkler watering, mechanical weed control and, where necessary, protective treatments against diseases and pests. The harvest time is also given in Table 1.

Table 1. Doses and kinds of mineral fertilizers used in vegetables growing and dates of harvest.

Vegetable species	Mineral fertilizer (kg ha^{-1})			Date of harvest
	N	P_2O_5	K_2O	
	ammonium nitrate, 34% N	superphosphate, 46% P_2O_5	potassium chloride, 60% K_2O	
Brassicas				
Broccoli	150	100	150	01.10
Brussels sprouts	120	80	150	13.10
Green cauliflower	150	100	150	28.09
White cauliflower	150	100	150	03.10
Leafy				
Kale	90	80	150	23.10
New Zealand spinach	60	60	100	12.07
Spinach	60	60	100	20.10
Root				
Celeriac	80	80	150	17.10
Carrot	60	80	150	15.09
Parsnip	60	80	150	19.10
Red beet	60	80	150	22.09
Leguminoses				
French bean	50	80	150	05.08
Broad bean	50	80	150	20.07
Pea	40	80	150	10.07

Preparation of the Raw Material

Directly after harvest mean samples representing the whole batch of the material were taken for analysis and preparation of the frozen products. All the vegetables were cleaned in running water. The study covered the following vegetables: Brussels sprout heads 25-30 mm in diameter were cleaned of stipules and the stems were cut; florets of broccoli and cauliflowers (white and green) were cleaned of leaves and divided into pieces 5 cm in diameter, the stems being cut 2 cm below the lowest ramification. Leafy vegetables were prepared as follows: the main rib was removed from healthy undamaged leaves of kale and the leaf blades were cut in crosswise strips 2-3 cm in width; whole healthy leaves with shoots 15-17 cm in length of New Zealand spinach were used; and, in the case of common spinach, whole healthy leaves without petioles were frozen. In root crops, carrot was 3-4 cm in diameter; parsnip 4-5 cm; celeriac 13-15 cm; and red beets about 6 cm in diameter. The carrot, parsnip and celeriac were peeled; the carrot and celeriac were cut into cubes 10x10x10 mm and parsnip into matchsticks 30x10x10 mm. Seeds of broad bean and pea were shelled and sorted, immature and overripe ones being discarded. In French bean, after discarding immature and overripe pods, the tips were cut off. After the processing described above (washing and suitable cutting), the vegetables were ready for blanching or boiling (A). Before freezing, the vegetables were blanched (B) or boiled (C). Red beet was blanched and also boiled whole in the skin to protect it from loosing juice and natural colour; then the beets were peeled and cut.

Preparation of Frozen Products

Two variants of processing the raw materials before freezing were used. In variant 1, the traditional technology of blanching the raw material was used (B); after freezing and frozen storage the product was boiled to consumption consistency (D). In variant 2, the raw material was boiled to consumption consistency to obtain a ready-to-eat product (C), which after freezing and storage only had to be defrosted and heated in a microwave oven (E).

In variant 1, the fresh material was blanched in a stainless steel vessel in water, the proportion of water to the raw material being 1:5 and the blanching temperature 95-98^{o}C. The time of blanching of the different species is given in Table 2. The applied parameters of blanching allowed a decrease in the activity of catalase and peroxidase to a level not exceeding 5% of the initial activity (unpublished data). After blanching the material was im-

Table 2. Pre-treatment times of vegetables before freezing (blanching – B, boiling – C) and preparing frozen vegetables for consumption (boiling – D, defrosting and heating in a microwave oven – E).

Vegetable species	Vegetables before freezing		Frozen vegetable	
	B	C	D	E
Brassicas				
Brussels sprouts	5 min	15 min	9 min	8 min 15 sec.
Broccoli	3 min	5 min	4 min 30 sec.	8 min 15 sec.
Green cauliflower	3 min	6 min	5 min	8 min 15 sec.
White cauliflower	3 min 15 sec.	6 min	5 min	8 min 15 sec.
Leafy				
Kale	3 min	15 min	12 min	8 min 15 sec.
New Zealand spinach	2 min 15 sec.	4 min	2 min	8 min 15 sec.
Spinach	2 min	6 min	2 min	8 min 15 sec.
Root				
Celeriac	2 min 30 sec.	8 min	5 min	8 min 15 sec.
Carrot	2 min 45 sec.	12 min	6 min	8 min 15 sec.
Parsnip	2 min 30 sec.	10 min	4 min 30 sec.	8 min 15 sec.
Red beet	15 min	35 min	*	8 min 15 sec.
Leguminoses				
French bean	3 min	9 min	6 min	8 min 15 sec.
Broad bean	3min 15 sec.	12 min	8 min	8 min 15 sec.
Pea	2 min 30 sec.	8 min	6 min	8 min 15 sec.

* The blanched frozen red beet root was not boiled since it was regarded as a semi-finished product to be used in preparing beetroot soup (borsch); in this case it would be boiled in a different proportion to water while both the vegetable and the liquid fractions would be utilized.

mediately cooled in cold water and left to drip on sieves for 30 min.

In variant 2, the vegetables were boiled in a stainless steel vessel in a brine containing 2% (w/w.) of table salt (NaCl), the proportion of the weight of the raw material to brine being 1:1. The vegetables were placed in boiling brine. The boiling time measured from the moment when the water came to a boil is given as well in Table 2. After boiling to consumption consistency, the material was left on sieves and cooled in a stream of cold air.

The materials from blanched and boiled samples were placed on trays and frozen at -40°C in a Feutron 3626-51 blast freezer. The time required for reaching the inside temperature of -30°C was 120 min. The frozen vegetables were then packed in 500 g polyethylene bags (0.08 mm thickness) and stored at -30°C for 12 months.

Preparation of Frozen Product for Evaluation

Samples of the vegetables blanched before freezing (B) were boiled in 2% brine, the proportion of the brine to the material being 1:1 (w/w.). As was the case when boiling fresh vegetables, the frozen product was placed in boiling brine. The time of boiling measured from the moment when the water came to the boil is given in Table 2. After boiling, the water was immediately drained; the product was cooled to 20°C (D) and analyzed. Samples of vegetables boiled before freezing (C) were defrosted and heated in a Panasonic NN-F621 microwave oven (E). In this case a 500 g portion was placed in a covered heatproof vessel. The time required to defrost and heat the material to consumption temperature [13] is given in Table 2. The samples were then cooled to 20°C and analyzed.

The content of lead and cadmium in the tap water used in technological and culinary processing was measured in each case. The used water samples did not significantly differ (P < 0.01) in the content of lead and cadmium, the range between the extreme concentrations of the metals did not exceed 15%. The maximum levels of lead and cadmium in the tap water were 60% and 70%, respectively, of the maximum level permitted in the Council Directive of 1998 [14]. Whenever table salt was added, it was from the same batch.

Table 3. Content of lead in raw, blanched, boiled and frozen, stored and then prepared for consumption vegetables (μg kg^{-1} fresh matter).

Vegetable species	A	B	C	D	E	LSD	
						P < 0.01	P < 0.05
Brassicas							
Broccoli	91 ±9	89 ±8	83 ±6	82 ±8	81 ±8	ns	ns
Brussels sprouts	82 ±5	79 ±8	79 ±7	80 ±7	79 ±7	ns	ns
Green cauliflower	78 ±6	78 ±10	75 ±7	74 ±9	75 ±9	ns	ns
White cauliflower	88 ±6	88 ±6	88 ±8	80 ±6	88 ±6	ns	ns
LSD P < 0.01, P < 0.05	ns, ns	ns, ns	ns, ns	ns, ns	ns, ns		
Leafy							
Kale	134 ±16	130 ±32	125 ±22	133 ±30	130 ±28	ns	ns
New Zealand spinach	3 ±1	2 ±1	2 ±1	2 ±2	3 ±1	ns	ns
Spinach	81 ±5	81 ±5	80 ±7	78 ±18	83 ±13	ns	ns
LSD P < 0.01, P < 0.05	21.8, 15.2	42.7, 29.7	31.0, 21.6	46.5, 32.3	40.7, 28.3		
Root							
Celeriac	77 ±9	76 ±14	77 ±10	75 ±14	77 ±10	ns	ns
Carrot	86 ±11	85 ±7	84 ±9	80 ±13	87 ±13	ns	ns
Parsnip	3 ±1	3 ±2	2 ±1	3 ±1	2 ±1	ns	ns
Red beet	84 ±13	82 ±12	83 ±14	81 ±11	84 ±15	ns	ns
LSD P < 0.01, P < 0.05	20.9, 14.9	21.5, 15.3	20.6, 14.7	23.1, 16.5	24.1, 17.2		
Leguminoses							
French bean	37 ±7	35 ±4	35 ±6	33 ±7	36 ±5	ns	ns
Broad bean	2 ±1	2 ±1	2 ±1	2 ±1	2 ±1	ns	ns
Pea	2 ±1	2 ±1	2 ±1	2 ±1	2 ±1	ns	ns
LSD P < 0.01, P < 0.05	9.1, 6.4	5.3, 3.7	8.0, 5.5	9.3, 6.4	7.6, 5.3		
for all species LSD P < 0.01, P < 0.05	14.9, 11.2	20.9, 15.6	17.3, 12.9	22.5, 16.8	21.0, 15.7		

A – raw material before blanching and boiling; B – blanched material before freezing; C – boiled material before freezing; D – product after frozen storage, prepared from B by boiling; E – product after frozen storage, prepared from C by defrosting and heating in microwave oven

Chemical Analyses

All chemical analyses were carried out in two parallel determinations, each in two replications. For the determination of lead and cadmium, the material was incinerated in a Nabertherm model L 9/S 27 furnace oven at 460°C for 8 h, and further mineralization of the material was carried out in a 3:1 mixture of nitric and perchloric acids. A 50 g portion of the material and 30 cm³ of the acid mixture were placed into 250 cm³ test tubes of the Tecator Kjeltec Auto Plus II mineralization set. The treated samples were left until the next day when complete mineralization was carried out. The mineralized samples were diluted with ultra-pure water to a volume of 100 cm³ and filtered to dry flasks. All glassware was soaked in 10% nitric acid for 24 h and then rinsed with ultra-pure water before use. The content of lead and cadmium in the solution was determined using an inductively coupled argon plasma emission spectrophotometer JY 238 Ultrace-Jobin Yvon (France). The most sensitive wavelengths for the determination of analyses were as follows: 220.353 nm for lead and 228.802 nm for cadmium. The accuracy of vegetable analysis method was verified on the basis of certified reference material (cabbage) (GBW 08504 – State Metrology Bureau, Beijing, China) with concentration of lead and cadmium 0.28 ±0.09 µg g^{-1} and 0.029 ±0.006 µg g^{-1}, respectively.

Statistical Analysis

The differences in the content of lead and cadmium between the compared species of vegetables at the same stage of processing (vertical comparing in Tables 3 and 4) and within one species between stages of processing (horizontal comparing in Tables 3 and 4) were established using single-factor analysis of variance (ANOVA) on the basis of the Snedecor F and Student's t tests and the least significant difference (LSD) was calculated at the probability level $P < 0.05$ and $P < 0.01$. The Statistica 6.1 program was applied.

Table 4. Content of cadmium in raw, blanched, boiled and frozen, stored and then prepared for consumption vegetables (µg kg^{-1} fresh matter).

Vegetable species	A	B	C	D	E	LSD	
						P < 0.01	P < 0.05
Brassicas							
Broccoli	5.1 ±0.8	4.0 ±0.4	4.7 ±0.5	2.0 ±0.2	4.9 ±0.7	1.15	0.83
Brussels sprouts	5.6 ±0.4	5.2 ±0.3	4.7 ±0.5	3.9 ±0.6	5.0 ±0.3	0.88	0.64
Green cauliflower	6.0 ±0.5	6.0 ±0.6	5.6 ±0.5	2.7 ±0.3	5.3 ±0.9	1.25	0.90
White cauliflower	4.5 ±0.6	4.5 ±0.2	4.5 ±0.5	2.6 ±0.5	4.5 ±0.8	1.16	0.84
LSD P < 0.01, P < 0.05	ns, 0.90	0.92, 0.66	ns, 0.75	0.92, 0.66	ns, ns		
Leafy							
Kale	7.4 ±1.4	6.0 ±0.6	4.5 ±1.2	3.0 ±0.7	4.6 ±0.7	2.05	1.48
New Zealand spinach	27.6 ±6.3	24.2 ±4.4	25.8 ±4.6	24.6 ±4.9	28.0 ±3.8	ns	ns
Spinach	42.2 ±3.0	40.0 ±3.8	40.1 ±4.2	30.4 ±3.2	41.0 ±6.0	8.65	6.26
LSD P < 0.01, P < 0.05	9.44, 6.57	7.77, 5.41	8.43, 5.87	7.85, 5.46	9.39, 6.54		
Root							
Celeriac	9.6 ±1.6	9.1 ±1.9	6.1 ±0.9	6.1 ±1.6	6.1 ±0.9	2.97	2.15
Carrot	12.9 ±2.3	7.4 ±1.1	6.6 ±1.6	5.7 ±1.1	6.8 ±1.3	ns	ns
Parsnip	6.7 ±1.8	4.7 ±1.2	3.6 ±0.8	3.5 ±0.4	3.8 ±0.5	2.26	1.64
Red beet	6.2 ±0.9	5.9 ±1.0	5.2 ±0.6	5.1 ±0.7	5.2 ±0.7	ns	ns
LSD P < 0.01, P < 0.05	ns, 2.45	2.87, 2.05	2.29, 1.63	ns, 1.59	1.95, 1.39		
Leguminoses							
French bean	1.9 ±0.3	1.9 ±0.3	1.8 ±0.3	1.8 ±0.4	1.8 ±0.2	ns	ns
Broad bean	1.3 ±0.3	1.2 ±0.3	0.8 ±0.4	0.8 ±0.2	0.8 ±0.4	ns	ns
Pea	1.9 ±0.4	1.4 ±0.3	1.3 ±0.3	1.1 ±0.3	1.4 ±0.3	ns	ns
LSD P < 0.01, P < 0.05	ns, ns	ns, 0.47	0.82, 0.57	0.70, 0.49	0.75, 0.52		
for all species LSD P < 0.01, P < 0.05	4.03, 3.01	3.32, 2.48	3.47, 2.59	3.24, 2.42	3.81, 2.85		

A – raw material before blanching and boiling; B – blanched material before freezing; C – boiled material before freezing; D – product after frozen storage, prepared from B by boiling; E – product after frozen storage, prepared from C by defrosting and heating in microwave oven

Results and Discussion

The content of lead and cadmium given in the literature varies within wide limits. According to Moćko and Wacławek [6], in 320 vegetable samples the lead content varied from trace amounts to 2380 μg kg^{-1} fresh matter, the admissible level [15] being exceeded in 6% of samples. The authors also determined the level of cadmium varying from trace level to 651 μg kg^{-1} fresh matter. The admissible level [15] was exceeded in as many as 47% of samples. According to Ni et al. [16] the highest content of cadmium reached the level of 1400 μg kg^{-1} fresh matter. This great variation depended on air pollution [4], type of soil [10], location of the cultivation sites [7, 8], concentration of metals in soil [17] and the competition for soil nutrients [5, 9], the vegetable species [18], and the usable part of a plant [16]. As reported Kawashima and Soares [19], the content of elements considerably varied in samples of the same species. According to Bhattacharjee et al. [20], there were large differences in micro-elements between samples. In the presented study, differences between samples-replications were not so high; however, they should be considered significant, as shown by standard deviations from the mean values (Tables 3 and 4).

In the studied vegetables, the content of lead depended on species and – compared with the admissible level [15] (0.3 mg kg^{-1} fresh matter for brassicas and leafy vegetables and 0.1 mg kg^{-1} for other vegetables) – in brassicas it was fairly constant, amounting to 26-30%. In leafy vegetables it varied considerably, since New Zealand spinach contained only 1% of the admissible amount, while spinach contained 27% and kale 45% (Table 3). Of the root vegetables, parsnip contained only 3% of the admissible amount, while the remaining species contained as much as 77-86%. Among leguminous vegetables, French beans showed the highest content of lead (37%), while the tested seeds contained only 2%.

The studied vegetable species contained 3-26% of the maximum admissible level of cadmium (0.2 mg kg^{-1} fresh matter for leafy vegetables and celeriac, 0.1 mg kg^{-1} for root vegetables, and 0.05 kg^{-1} for other vegetables). The lowest content of this metal (3-4%) was found in leguminous vegetables, while in brassicas it was 9-12%; in leafy vegetables 4-21%; and in the root vegetables 12-26% (Table 4). Ni et al. [16] and Järvan and Põldma [21] also found more cadmium in root than in leafy vegetables, with twice the content being recorded in some cases. However, the results obtained by Mikuła [22] showed that leaves accumulated more heavy metals than did roots. It should be stressed that in the presented work the vegetables were washed, peeled (if necessary in the case of a given species) and cut before analyses, i.e. prepared to pre-freezing procedures. Rosada and Nijak [23] showed that, depending on species, the content of lead in leafy vegetables after washing was 1-3 times lower than before washing. These authors also found 2-10 times lower lead content in root vegetables before peeling than in the removed peel. Zurera and Moreno [24] reported a 21% decrease in lead content in asparagus after washing and a further 12.5% decrease after peeling. These authors also found that the thicker the removed peel, the greater the decrease. Similar observations also concerned cadmium.

The process of freezing and preparing the studied vegetable for consumption did not significantly affect the level of lead whether the traditional method or the modified method was applied (Table 3). Compared with the material ready for blanching or boiling (A), changes in lead concentration were small, varying in the range of -11% to 2% in all the studied samples, with the exception of changes in the content of lead in New Zealand spinach and parsnip. This was no doubt associated with the very low content of lead in these species, hence even small changes in this level brought about a high percentage change. Since the content of lead was not affected by technological and culinary processing, virtually the same statistical relationship between the material before blanching or boiling (A) was established for all the stages of analysis (B, C, D, and E) (Table 3).

Haring and Delft [25] even showed an increase in the content of lead in boiled vegetables of four species when "hard" water was used, and a small decrease if the water was "soft"; however, these authors did not give the units of water hardness, which would have specified this trait in the water. In all the processes carried out in the presented experiment, water hardness varied in a relatively small range of 360-400 mg $CaCO_3$ dm^{-3}, this corresponding to the lower values of hard water.

As opposed to lead, the cadmium content in some cases depended on technological and culinary processes. After blanching (B), the content of cadmium was lower, but only in broccoli and parsnip and only at $p=0.05$; after boiling (C), the level of this metal was significantly lowered in Brussels sprouts, kale, celeriac and parsnip. In frozen products prepared for consumption when the traditional method was used (D), cadmium content was lower than in the fresh material (A) of most vegetables, except for New Zealand spinach, carrot, red beet and all the leguminous crops. In products obtained using the modified technology (E), a lower cadmium content was found only in kale, celeriac and parsnip when compared with the fresh material (A).

As in the present experiment, other research on pea and French bean did not indicate that processing with water had any effect on the levels of lead and cadmium [26, 27]. However, Zurera and Moreno [24] found a decrease in the content of both lead and cadmium after the sterilization of asparagus; these authors suggest that the decrease was due to water extraction. Svoboda et al. [28] found that the greater the grinding of mushroom tissue, the more lead and cadmium was leached during boiling, with significantly greater leaching of cadmium being recorded. Varo et al. [29] measured the content of lead and cadmium in seven species of fresh vegetables and in frozen products of the same seven species; however, the samples were taken from the retail market, thus it was not the same material and the frozen products had not undergone culinary processing. These authors found that in comparison with fresh vegetables, frozen products contained less, the same or higher amounts of lead, while the content of cadmium was lower or the same. The results of the presented study confirmed that processing resulted in greater decreases in cadmium than in lead content.

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