The assessment of physicochemical properties and macronutrient content of reclaimed soil material and hard coal ash 15 years after the experiment setup

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Abstract. This paper presents the results obtained during reclamation of hard coal ash. The model was developed by covering the layer of ash with layers containing ash, organic by-products and mineral fertilizers in 10 different combinations. The experiment on reclamation of hard coal ash was carried in 2003 on the premises of Dolna Odra Power Station in Poland. Vegetation in the form of grass mixture was introduced to the plots of an area of 100 m² each. The layer of ash, i.e. underlay, located below each top laver was divided into two parts. One part was fertilized with mineral fertilizers NPK ratio of 60-70-70, the other part was left unfertilized. Particularly favourable was the introduction of fermented municipal sewage sludge to top layers. In this way, the appropriate quality of the analysed top layers was obtained, as manifested by good abundance of forms of magnesium, potassium and phosphorus available for vegetation. The results demonstrate the process of accumulation of the available forms of magnesium, potassium and phosphorus in underlays when the fertile surface layer is supplemented with fermented municipal sewage sludge. The results obtained in the experiment indicate good effectiveness of reclamation of coal ash waste and justify the continuation of the reclamation.

Keywords: physicochemical properties, macroelements, top layer, underlay, hard coal ash

INTRODUCTION

In Poland, despite the gradual reduction of hard coal mining, the consumption of coal for the production of electricity and heat in 2018 amounted to 42568 thousand tons (Statistics Poland, 2019). Consequently, enormous amounts of ash-slag mixture were generated. According to the Regulation of the Minister of Climate on the waste

Corresponding autor: Marzena Gibczyńska e-mail: marzena.gibczynska@zut.edu.pl phone: +48 608 511 430 catalog (2020), combustion by-products (CBP) coded 10 01 01, such as ash generated by the power industry and specified in the Regulation as slag, bottom ash and boiler ash, are not hazardous and their agricultural use is possible (Journal of Laws of 2020, item 10). Storage of ash-slag mixture results in formation of areas without vegetation cover, exposed to erosion and in absolute need of reclamation as a significant element of the functioning of the natural environment. Land reclamation is the process of establishing or restoring the utility value to the degraded or devastated land which, among others, includes restoration of soil. The processes are long-term and require significant financial resources. Numerous studies of the available literature on the subject present the realistic possibilities of reclamation of lands used for storing hard coal ash (Labidi et al., 2017; Theisen, 2015). The presence of macroelements and compounds showing strong adsorptive properties in ash indicates the potential environmental use (Wyszkowski et al., 2014).

This paper presents the results obtained during reclamation of hard coal ash. The model was developed by covering the layer of ash with layers containing ash, organic by-products and mineral fertilizers in 10 different combinations. The results of the studies conducted 15 years after the set-up of experiment, were aimed at determining the efficiency of reclamation of ash waste and were based on the assessment of the physicochemical properties and macroelement content in particular layers.

MATERIALS AND METHODS

Experiment characteristics

The experiment on reclamation of hard coal ash was conducted in 2003 on the premises of Dolna Odra Power Station in Nowe Czarnowo (53.20° N; 14.48° E), near

	Top layer and underlay number					
	Ι	II	III	IV	V	
Top laver	 low peat, ash. loose sand, compact produced 		 loose sand, compost produced with GWDA method, formanted municipal loose sand, ash, compost produced with 		 loose sand, coniferous wood bark, compost produced with GWDA method 	
Top layer		 compost produced with GWDA method[#], ash. 	sewage sludge (dry weight: 70% sludge, 15% urban green waste, 15% straw).	 fermented municipal sewage sludge (dry weight: 70% sludge, 30% urban green waste). 	 fermented municipal sewage sludge (dry weight: 70% sludge, 30% straw). 	
	Ratio 1:3	Ratio 1:1:2:4	Ratio 1:1:2	Ratio 0.5:0.5:1:2	Ratio 1:1:2:4	
Unfertilized underlay	Ash	Ash	Ash	Ash	Ash	
Fertilized underlay	Ash + NPK (60-70-70)	Ash + NPK (60-70-70)	Ash + NPK (60-70-70)	Ash + NPK (60-70-70)	Ash + NPK (60-70-70)	

Table 1. Fertilization scheme.

GWDA method - Composting sludge with structural material using the static method with forced aeration.

Gryfino in Poland. Surface layer (fertile) of a thickness of 40 cm consisting of a mixture of various materials, i.e. top layer, was applied to the ash underlay. The experiment was performed in four replications. Treatment number I is control. There were five treatments in the experiment in the form of top layer and ten as underlay. The top layer was used to enable the grass to grow properly. The composition of five types of top layer is presented in Table 1. Vegetation in the form of grass mixture (red fescue, perennial ryegrass, blue grass) was introduced to the plots of an area of 100 m² each. The layer of ash, i.e. underlay, located below each top layer was divided into two parts, when the experiment was set up. One part was fertilized with mineral fertilizers NPK ratio of 60-70-70, the other part was left unfertilized. The ratio is enough as a starting dose for most of plants. During the period 2008 grass was fertilized and cut off, later only cut once a year.

After 15 years, in the autumn of 2018, samples of top layer (0–40 cm in depth) and underlay (40–60 cm) were taken from the plots covered with grass. The samples taken from top layer are anthropogenic soils formed by applying fertile surface layer to ash. The anthropogenic soils formed in such a way are humic industrozems (Alpr) of a profile sequence Aan-2Can (Marcinek, Komisarek, 2011; Świtoniak et al., 2016).

Chemical analyses

Samples were taken in three replication with Egner's cane from appropriate depths. The samples were dried and ground according to the requirements set out in the Polish Standard (ISO 10381-2:2007). Soil pH was determined potentiometrically with the use of a pH-meter Orion Star A 211, according to ISO standard ISO 10390 2021. The

material loss at annealing at 550 °C was adopted as the content of organic matter. Electrical conductivity (EC) was measured with conductometric method (conductometer Orion 3Star) (suspension with a soil/water weight ratio 1:2.5) (ISO 11265:1994/Cor 1:1996). Total content of carbon (C), nitrogen (N) and sulphur (S) was determined with the use of elemental analyser CHNS (Costech Instruments Elemental Combustion System) by ELTRA Poland. To determine the total content of potassium, calcium and magnesium, the soil samples were wet digested in the mixture of nitric(V) acid and chloric(VII) acid in the ratio 1:1. The content of available magnesium was determined with Schachtschabel method using the extraction with a solution of calcium chloride (0.025n CaCl₂) (DIN-R-04020:1994+Azl:2004). In the obtained extracts, the content of metals was determined with the use of Atomic Absorption Spectrometer Apparatus (Thermo Fisher Scientific iCE 3000 Series). The available forms of phosphorus and potassium in the soil were determined using the Egner-Riehm method based on extraction of calcium lactate with buffer solution characterised by a pH value of 3.55 (Egner et al., 1960). The paper presents average values from three replication for each variant.

Statistical analysis

The results of the two-factor experiment were statistically processed using the analysis of variance in complete randomization design. The factors were as follows: 1st factor -5 top layer, 2nd factor - two underlay. Confidence sub-intervals were calculated using Tukey's multiple test, assuming a significance level of P<0.05 (Hill et al., 2006). The statistical analysis of the results was carried out using the Statistica 10.0 software.

Morka	Unit -	Top layer number				
IVIAIRS	Unit	Ι	II	III	IV	V
Electrical conductivity (EC)	μS cm ⁻¹	122b±9.7	92.1b±6.4	171a±20	155a±7.4	195a±29
pH _{KCl}	-	7.61a±0.05	7.44a±0.05	6.71b±0.02	6.99b±0.03	6.66b±0.06
pH _{H2O}	-	7.88a±0.05	7.64b±0.07	7.35c±0.04	7.28c±0.05	7.04 d±0.04
Organic matter	%	6.73a±1.06	4.66a±0.67	5.83a±0.74	5.68a±1.02	4.53a±0.88
Nitrogen	%	0.234b±0.02	0.165b±0.02	0.255b±0.02	0.319a±0.02	0.357a±0.02
Sulphur	%	$0.037b{\pm}0.02$	0.013b±0.02	0.022b±0.03	0.045a±0.02	0.052a±0.02
Carbon	%	3.34b±0.005	3.11b±0.001	3.18b±0.002	4.40a±0.005	4.77a±0.004
N/S	-	6.83b±0.87	12.50a±0.96	11.40a±0.40	7.48b±0.45	7.03b±0.21
C/N	-	15.2ab±2.21	19.1a±0.64	12.5b±0.33	13.9b±0.36	13.4b±0.20
Total magnesium		3.42a±0.67	3.79a±0.16	3.68a±0.20	4.16a ±0.17	3.27a±0.16
Total potassium	g kg-1	2.08a±0.03	2.64a±0.24	2.33a±0.10	2.51a±0.06	2.68a±0.10
Total calcium		17.9a±0.66	17.6a±0.77	16.2a±0.97	18.6a±0.71	17.9a±0.99
Available magnesium		249.8b±22.8	256.3b±11.9	322.5a±19.2	347.3a±29.9	273.5b±16.9
Available potassium	mg kg-1	215.0a±7.5	248.5a±12.0	141.8b±4.4	155.0b±11.2	177.5b±5.1
Available phosphorus		165.0a±8.8	176.5a±4.2	146.3b±7.2	140.5b±4.0	149.0b±4.2

Table 2. Physicochemical properties, total content of nitrogen, sulphur, carbon, calcium, magnesium and available forms of phosphorus, potassium and magnesium in top layers.

Values in rows followed by the same lower case letters are not statistically different

RESULTS AND DISCUSSION

Physicochemical properties and macroelements content in top layer

Average electrical conductivity of soil taken from top layers showed differentiation (from 92.1 to 195 μ S cm⁻¹) (Table 2). Higher electrical conductivity was found in top layer containing fermented municipal sewage sludge, i.e. number III, IV and V. The field experiment conducted by Tsadilas et al., (2014) produced comparable results in terms of increased electrical conductivity in soil due to fertilization with sewage sludge which the authors attributed to high content of salt in sludge. On the assumption that 1000 μ S cm⁻¹ represents 2.23 g NaCl in 1 kg of soil, the top layer is to be considered as non-saline (ISO 11265:1994/ Cor 1:1996).

Provided that underlay is to be treated as soil, the top layer types labelled I and II manifested alkaline pH, according to applicable standards (ISO 10390: 2021). The remaining three types of top layer showed neutral pH, most likely due to the presence of municipal sewage sludge (Table 2). The acidifying action of ash resulting from the addition of sewage sludge is widely known and well documented in the literature on the subject (Antonkiewicz et al., 2020; Masto et al., 2012; Tsadilas et al., 2014).

There was no differentiation in terms of organic matter content in top layer, which can be explained by the development of vegetation on the reclaimed area. The relationships concerning the amounts of nitrogen and sulphur in top layer were identical. The content of these elements in top layers I, II and III was found to be lower than in the remaining two types of the top layer (Table 2).

Sulphur is found in soil mainly in the form of organic compounds connected with the presence of humus and, it affects the growth and development of vegetation. According to Pietrzak (2015), in grassland mineral soils, the average content of total sulphur is 0.039%, with fluctuations from 0.034% to 0.044%. In top layer, the content of sulphur ranged from 0.013 to 0.052%, which does not prevent cultivation on the reclaimed area. The relationships concerning the content of sulphur and carbon in individual top layers were comparable - in top layers IV and V the content of both elements was found to be higher compared to the other top layers (Table 2). Janzen and Bettany (1984) demonstrated that the optimum ratio of available nitrogen to sulphur is 7:1. The ratio below 7 results in reduced yield. Carbon/nitrogen ratio (C:N) affects the decomposition of organic matter and represents the potential fertility of soils. Therefore, it can be applied for the purpose of the assessment of the reclaimed areas. The results obtained in the experiment show that in the discussed top layers the ratio is appropriate. The prevalence of carbon content over the content of nitrogen (C:N) in mineral soils generally amounts to 8-12 (on average 10) (Siuta, 2003). The results of the experiment manifest that owing to the addition of municipal sewage sludge to top layer type III, IV and V, appropriate values of C:N ratio were established. In top layer types I and II an excess of carbon was found, and in soil poor in nitrogen the decomposition of organic matter occurs at a slower rate.

The metal elements such as magnesium, potassium and calcium form cations in soil solution directly affecting the changes in soil pH. The elements may be taken up by the plants after introduction to soil solution. Interchangeably bound alkaline cations constitute a nutrient pool for vegetation. The presence of divalent cations of alkaline pH (calcium, magnesium) in the sorption complex positively affects the structure of soil. Mean content of total magnesium and potassium in top layers was 3.66 g Mg kg⁻¹ and 2.45 g K kg⁻¹. The experimental top layers showed total calcium content of 17 g Ca kg⁻¹.

The presence of hard coal ash did not result in a significant differentiation of the total content of the three macroelements, (Mg, K i Ca) in top layers, and the values did not exceed those characteristic for arable lands (Table 2). By way of comparison, soils contain from trace to a few percent of total magnesium (Nowosielski, 1959). The content of magnesium in soils in Poland is low which, to a considerable extent, is due to the soils being light and very light with low humus content. The total content of potassium in soils varies from 0.8 to 2.5% and depends on the proportion of soil silt elements and their mineral composition. Calcium content in soils is within the range of 0.7-36 g Ca kg⁻¹, (Medaj et al., 2017).

On the basis of 20 year long studies on the monitoring of the chemistry of arable lands, the Institute of Soil Science and Plant Cultivation (IUNG) states that in 2010, the content of available magnesium in Poland ranged from 5– 358 mg Mg kg⁻¹ of soil which shows high variability (Monitoring Chemizmu Gleb...). The whole surface layer of the reclamation experiment was characterised by a very high content of available magnesium (DIN-R-04020:1994+AzI:2004). The highest content of available magnesium (322.5 and 347.3 mg Mg kg⁻¹) was found in top layer type III and IV which contained urban green waste (Table 2).

Potassium is a nutrient indispensable for plant growth and one of the three main macronutrients – others being nitrogen and phosphorus. The natural potassium content in soils depends on the mineralogical composition and granulation, particularly on the content of clay minerals, composition of associated minerals, the capacity for cation exchange and soil pH (Dhaliwal et al., 2006). Due to high solubility of potassium salts, sewage sludge is not rich in potassium compounds. The addition of fermented municipal sewage sludge to top layers III, IV and V served as a way of diluting the present content of available potassium. In these top layers, the content of available potassium was high, whereas in the remaining top layer types, i.e. I and II, it was very high (Egner et al., 1960).

Phosphorus is one of the three macronutrients, the deficiency of which reduces the yield of crops to the greatest extent. The shortage of phosphorus in agricultural soils is generally common worldwide. The main reason of limited bioavailability of phosphorus is its high rate of retrogradation and slow release to soil solution (Shen et al., 2011; Zhu et al., 2018). On the basis of the adopted criteria, in all analysed top layers the content of available phosphorus was very high (Egner et al., 1960). Similarly to the determined content of available potassium, in top layers III, IV and V the identified content of available phosphorus was, on average, 145 mg P kg⁻¹ (Table 2).

Analysis of physicochemical properties and macroelements in underlays

The comparison of electrical conductivity values determined in the samples taken from underlays and top layers show comparable relationships. Higher electrical conductivity was found in underlays with fermented municipal sewage sludge – number III, IV and V, compared to the other two. All underlays are classified as non-saline (ISO 11265:1994/Cor 1:1996).

Mean pH w KCl value of underlays amounted to 8.39 and was higher than the values determined in the top layers. According to the standards for soils, the analysed underlays are classified as alkaline (ISO 10390: 2021).

The analysis of the content of organic matter shows the increase in underlay I. This can be explained by the presence of peat resulting in an increased development of vegetation. In the remaining four underlays, mean content of organic matter amounted to 4.92% (Table 3).

The mineral fertilisation (NPK) applied in the reclamation experiment did not result in pH differentiation and caused higher values of electrical conductivity and organic matter content in underlays (Table 3).

The total nitrogen content in underlays was from 3.6 to 8 times lower than that in top layers. Out of all underlays in the present experiment, only underlay I contained less total nitrogen. However, this is explained by the fact that the components of the surface layer (peat and ash) are not rich in this element.

In underlays I and II, the sulphur content was lower, whereas in the remaining three underlays. The average sulphur content in hard coal ash is 0.03% (Gluzińska et al., 2016). The reclaimed ash showed a lower content, i.e. 0.01%. There was no differentiation in terms of carbon content in underlays. The underlays showed a 40% lower carbon content in comparison with the surface layer (Table 4).

The value of N/S ratio, i.e. from 2.50 to 5.45, was below 7. This indicates the excessive content of sulphur in underlays containing hard coal ash.

The ratio of carbon to nitrogen (C:N) indicating the potential fertility of soil, in the case of the analysed underlays showed the value of 50, i.e. far from adequate with respect to arable soils, which manifested very large excess of carbon in comparison with nitrogen. The analysis of the values of N/S and C/N ratio shows a definite lack of nitrogen in underlays, which is explained by the chemical composition of hard coal ash (Table 4). The application of

Mortes	Unit -	Underlay number					
IVIAI KS		Ι	II	III	IV	V	
Electrical conductivity (EC)	μS cm ⁻¹	150.6b±10.4	142.6b±10.2	170.3a±11.9	174.4a±15.0	161.5a±15.9	
pH _{KCl}	-	8.46a±0.32	8.55a±0.32	8.16a±0.30	8.45a±0.31	8.37a±0.31	
pH _{H2O}	-	8.60a±0.34	8.86a±0.35	8.60a±0.33	8.71a±0.32	8.59a±0.32	
Organic matter	%	6.42a±0.75	5.08b±0.42	5.28b±0.72	5.17b±0.32	4.15b±0.88	
				Underlays			
	-	Unfe	rtilized underlay		Fertilized une	derlay	
Electrical conductivity (EC)	μS cm ⁻¹	138.5b±5.7			173.2a±8.4		
pH _{KCl}	-	8.51a±0.19			8.28a±0.18		
pH _{H2O}	-	8.84a±0.20			8.50a±0.1	19	
Organic matter	%	4.51b±0.43		5.92a±0.64			

Table 3. Physicochemical properties of underlays.

Values in rows followed by the same lower case letters are not statistically different

Table 4. Total content of nitrogen, sulphur and carbon in underlays.

Marks	Units -	Underlay number						
		Ι	II	III	IV	V		
Nitrogen	%	0.039b±0.004	0.046a±0.004	0.048a±0.004	0.046a±0.003	0.046a±0.004		
Sulphur	%	$0.009b{\pm}0.001$	$0.008b{\pm}0.002$	0.015a±0.003	0.017a±0.002	0.017a±0.003		
Carbon	%	2.08a±0.17	2.44a±0.09	2.30a±0.31	2.17a±0.08	2.36a±0.24		
N/S	-	4.37b±0.34	5.45a±0.20	3.75b±0.49	2.75c±0.22	2.50c±0.34		
C/N	-	53.89a±2.65	54.94a±4.37	46.91a±2.79	47.42a±1.84	50.50a±2.05		
		Unfe	rtilized underlay		Fertilized under	erlay		
Nitrogen	%	0.	.038b±0.001		0.052a±0.00)2		
Sulphur	%	0.	.015b±0.002		$0.020a{\pm}0.002$			
Carbon	%		1.96b±0.09		2.58a±0.10			
N/S	-		3.93a±0.41		3.59a±0.27			
C/N	-	5	51.60a±2.41		49.87a±1.24			

Values in rows followed by the same lower case letters are not statistically different

fertilizers (NPK) to the upper layer resulted in a significantly higher percentage of nitrogen, sulphur and carbon with lack of differentiation in terms of values of N/S and C/N ratio (Table 4).

The content of total magnesium, potassium and calcium amounted to, on average, 7.00 g Mg kg⁻¹, 4.60 g K kg⁻¹ and 18.5 g Ca kg⁻¹. The comparison of the content determined in underlays and top layers shows that the underlays were characterised by twice as large content of magnesium and potassium and comparable content of calcium. The study did not find a significant differentiation of the total content of the discussed macro elements in ash underlays with respect to the composition of the surface layer. As a result of the previously applied NPK fertilization, the content of magnesium in the fertilized underlays was determined to be 8% higher (Table 2 and 5). The identified content of the discussed cations was similar to the content in arable soils (Medaj et al., 2017; Nowosielski, 1959). In view of the standards of soil quality, the determined content of available magnesium in underlays containing hard coal ash is to be classified as very high (DIN-R-04020:1994+Azl:2004). The highest content of available magnesium, i.e. 290 mg Mg kg⁻¹, was found in underlay types III, IV and V containing fermented municipal sewage sludge (Table 5). It appears that the phenomenon of an increased content of magnesium is real and explained by the location of this element in grass roots. This is also in line with information that the content of magnesium in municipal sewage sludge is 5 g kg⁻¹ (Gondek, Filipek-Mazur, 2006). The recent studies by Głąb et al., (2020) show that organic fertilisation of soil results in an increased root length, size, volume and diameter.

With respect to the amount of available potassium, all underlays were characterised by very high content (Egner et al., 1960). Mean content of available potassium in underlays and top layers showed variability – in underlays I

Marler	Units –	Underlay number					
IVIATKS		Ι	II	III	IV	V	
Total magnesium		7.02a±0.30	6.95a±0.36	7.14a±0.27	6.89a±0.26	7.01a±0.81	
Total potassium	g kg ⁻¹	4.67a±0.17	4.81a±0.32	4.52a±0.39	4.47a±0.35	4.55a±0.14	
Total calcium		17.6a±0.74	19.2a±0.72	19.6a±1.41	18.2a±0.89	18.1a±2.21	
Available magnesium		215.0b±12.6	238.5b±9.2	284.5a±11.2	287.8a±13.1	298.3a±9.9	
Available potassium	mg kg ⁻¹	180.5b±12.8	186.0b±15.9	275.8a±15.4	249.2a±10.3	246.0a±9.0	
Available phosphorus		138.0b±13.5	138.5b±12.5	174.3a±5.6	191.5a±7.8	185.8a±5.1	
				Underlays			
		Unfer	tilized underlay		Fertilized und	erlay	
Total magnesium		6.54a±0.25			7.07b±0.21		
Total potassium	g kg ⁻¹	4.71a±0.27			4.50a±0.20		
Total calcium			18.8a±1.0		18.1a±0.5		
Available magnesium		2	64.7a±12.3		264.9a±9.4	4	
Available potassium	mg kg ⁻¹	213.7b±14.0		241.3a±9.9			
Available phosphorus		1	53.1b±9.6		178.2a±5.2		

Table 5. The content of total and available forms of magnesium, potassium, calcium and phosphorus in underlays.

Values in rows followed by the same superscript lower case letters are not statistically different

and II the average content was 183, with the average content in top layers of 230 mg K kg⁻¹. In the remaining underlays, the identified relationship was opposite – average in underlays was 257, whereas in top layers it was 158 mg K kg⁻¹.

The content of available phosphorus in underlays corresponds with the category of very high abundance (Egner et al., 1960). In underlay types I and II, located under the fertile layer, the amount of available phosphorus was 138 mg P kg⁻¹, whereas that determined under the layer containing fermented municipal sewage sludge was 33% higher. The obtained results indicate the process of accumulation of magnesium, potassium and phosphorus in underlays in a combination with the fertile layer being supplemented with municipal sewage sludge. Mineral fertilization applied in the beginning of the experiment had no effect on differentiation of available magnesium content in underlays and resulted in an increase of available potassium and phosphorus content.

CONCLUSIONS

1. It was particularly advantageous to introduce fermented municipal sewage sludge into the upper layers, which is manifested by a good abundance in forms of magnesium, potassium and phosphorus. According to the standards applicable to soils, the underlays analysed 15 years after the set-up of the experiment are classified as alkaline, without differentiation from the first application of NPK fertilisation to the top layers.

2. The carbon/nitrogen ratio (C:N) characterising the potential fertility of soils, in the analysed underlays amounted to 50 and was far from adequate for arable soils, indicating a very large excess of carbon in relation to nitrogen. The analysis of N/S and C/N ratio shows the definite lack of nitrogen in underlays, which is understandable given the chemical composition of hard coal ash.

3. The results demonstrate the process of accumulation of the available forms of magnesium, potassium and phosphorus in underlays when the fertile surface layer is supplemented with fermented municipal sewage sludge. Mineral fertilisation applied in the beginning of the reclamation experiment showed no effect on differentiation of available magnesium content and resulted in an increased amount of available potassium and phosphorus.

4. The results obtained in the experiment indicate good effectiveness of reclamation of coal ash waste and justify the continuation of the reclamation

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received – 26 July 2021 revised – 24 September 2021 accepted – 7 December 2021



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