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The geochemical and fractionation study on toxic elements in road dust collected from the arterial roads in Kraków

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Keywords: air pollution, heavy metals, traffic, fractionation, road dust

Abstract: Road dust should be considered as a secondary source of contamination in the environment, especially when re-suspended. In our study road dust samples were collected from 8 high-capacity urban roads in two districts of Kraków (Krowdrza and Nowa Huta). Total concentration of toxic elements, such as Cd, Cr, Cu, Mn, Zn, Co, Pb, Ni, Ba and Se were determined using ICP–MS ELAN 6100 Perkin Elmer. A fractionation study were performed using VI step sequential extraction, according to the modified method provided by Salomons and Förstner. Appropriate quality control was ensured by using reagent blanks and analysing certified reference material BCR 723 and SRM 1848a. Concentration of metals in the road dust varied as follows [mg/kg]: Cd 1.02–1.78, Cr 34.4–90.3, Cu 65–224, Mn 232–760, Zn 261–365, Co 4.32–6.46, Pb 85.6–132, Ni 32.2–43.9, Ba 98.9–104 and Se 78.3–132. Degree of contamination of road dust from Nowa Huta was very high (Cdeg 54) and considerable for road dust from Krowdrza (Cdeg 25). Results revealed that road dust samples were heavily contaminated with Cd, Cu, Zn, Mn, Co, Pb, Ni, Ba and Se, in amounts exceeding multiple times geochemical background values. The chemical speciation study using VI step sequential extraction, followed by assessing risk assessment code (RAC) revealed that elements in road dust are mostly bound with mobile and easy bioavailable fractions such as carbonates and exchangeable cations, with the exception for Cr and Cu being mostly associated and fixed with residual and organic matter fraction.

Introduction

Emission from transport is a well-known key source of particulate matter in urban environments (Holnicki et al. 2021, Michalski and Pecyna-Utylska 2022). For instance, according to a study conducted by Air Quality Expert Group (AQEG 2012), road traffic emissions are estimated to contribute 30–50% of the increase in PM_{2.5} levels in urban environments in the UK. The U.S. Environmental Protection Agency (EPA), on the other hand, has estimated that transportation is responsible for less than 10% of PM_{2.5} and PM₁₀ emissions (EPA 2020). Road dust consists of secondary re-suspended particles in the atmosphere (Vlasov 2022), deposited as fine solids on road surfaces. The chemical composition of road dust in urban environments is a very complex issue, due to the fact that it is a heterogenous mixture of particles of both anthropogenic and geogenic origin. Gunawardana et al. 2012 report that road dust consists primarily of geogenic soil-derived minerals such as quartz and clay-forming minerals (above 60%), small percentage (about 2%) of plant organic matter, including pollen, and the remaining portion of dust (approximately 30%) containing potentially toxic elements

that are mostly sourced from vehicular traffic emission. These toxic elements are mostly heavy metals such as Zn, Cu, Pb, Cr, Cd, Al, Se, Fe, Bi, Ti (e.g. Adamiec et al. 2016 Ali Taleshi et al. 2020, 2021, Sabouhi et al. 2020) polycyclic aromatic hydrocarbons (PAHs) or black carbon (Gunawardana et al. 2012, Vlasov 2022) derived from multiple sources. Ali-Taleshi et al. (2022) in their recent study have employed positive matrix factorization model and identified five main sources of elements in road dust collected from Tehran, i.e., elements derived from local soils and geogenic sources (28.2%), from demolition and construction activities (20.2%), from vehicle exhaust emissions (VEE) (16.2%), from vehicle non-exhaust emissions (VNEE) (18%) and finally elements derived from industrial and metallic substances emissions (17.5%). Tracing source profiles of traffic-associated elements in road dust is not an easy task, especially owing to the fact that heavy metals in road dust can be sourced from both non-exhausted and exhaust emission. When incorporated in road dust, these contaminants may become a significant threat to both human health and environment due to possibility of resuspension of dust, thus, for instance, contributing to the secondary PM emission. Moreover, as road dust can be considered as a sink

for heavy metals, it can also become a secondary source of contamination for other environmental media such as surface water and soil. Ayrault et al. 2013 in their research have proven by using isotopic lead signature $^{206}\text{Pb}/^{207}\text{Pb}$ ratio that street dust collected from an urban area in France may contain up to 50% of lead originating from leaded gasoline additives, twelve years after their prohibition. That only proves that contaminants such as heavy metals remain trapped in road dust for decades and should be considered as an ecological ticking bomb. However, it is important to emphasize that the information on the total concentration of contaminants in a sample is insufficient to conclude about the real threat related to the bioavailability and mobility of the contaminants in the environment. Sequential extractions provide detailed information about the origin, occurrence, biological and physicochemical availability and mobilization potential of trace metals under changing environmental conditions. The bioavailability of heavy metals depends on the characteristics of the particle surface, metal's bonding strength or simply the aggressiveness of the leaching solution being in contact with the solid sample.

To the best of our knowledge, there are only scarce research and advanced studies focused on the transformation, mobility and speciation of traffic-related metals bound in the solid phase of road dust, such as those conducted by Fuigueiras et al 2002, Świetlik et al. 2015, Adamiec 2017a,b, Sutherland et al 2012 Xu et al. 2011, and Zhang & Wang. Most of the abovementioned researchers report that metals binding potential is strongly dependent on dust fraction size. Our prior fractionation study (Adamiec 2017a) was conducted on the finest fraction of road dust (below 20 μm) and the results revealed that the mobility of metals in dust decreases in the following order $\text{Zn} > \text{Cd} > \text{Ni} > \text{Cu} > \text{Pb}$. To fill in the gap this new fractionation research was conducted on the bulk road dust samples instead. The aim of our research was:

- 1) to study total concentration of the selected metals, priorly considered as key tracers of traffic-related emission, in the bulk sample of road dust collected

from the surface of heavily trafficked roads in Kraków, Poland;

- 2) to assess metal contamination of road dust by determining the Contamination Factor index (Cf) and the degree of contamination (C_{deg})
- 3) to conduct VI step fractionation study on road dust, to deliver information on real mobility and bioavailability of heavy metals in dust.
- 4) To calculate Risk Assessment Code to forecast the envirototoxicity of the road dust.
- 5) The source identification of certain metals in road dust was not part of this research.

Materials and methods

Study area and sample collection

The material in this research consisted of road dust collected from the surface of major, heavy trafficked roads in Kraków, Poland. The results of air quality monitoring in Krakow presented by Godłowska et al. (2022) confirm the significant impact of traffic-related pollutants. Two representative city districts were selected for the study. One was the city center, a residential area with a very high congestion, the vicinity of the old town, offices and areas with a high density of pedestrian, bicycle and car traffic. The other study area was Nowa Huta District, also residential area but with multiple industrial facilities in the vicinity, such as the Łęg heat and power plant, the Philips Morris tobacco plant and the Accelormittal Steelwork. Eight arterial, high-capacity urban roads (4 roads within the first sampling area and 4 roads in the second sampling area) were sampled (dust was swept from the road surface using brush). Two samples of dust were collected from each road, which accounts for a total of 16 road dust samples of approximately 0.5 kg each. Detailed sampling areas and sites location are presented in Table 1.

For further analysis all individual samples collected from each road but within the same area location were combined,

Table 1. Sampling campaign details

City district	Sampling site location (street names)	GPS coordinates and number of collected samples (n)
Kraków Krowdrza District	Adama Mickiewicza Ave	50°06,452'N 19,923728'E n=2
	Czarnowiejska St.	50°06,607'N 19°92,375'E n=2
	Dolnych Młynów St.	50°06,564'N 19°92,551'E n=2
	Reymonta St.	50°063,916'N 19°92,336'E n=2
Kraków Nowa Huta District	Nowohucka St.	50°06,568'N 19°99,671'E n=2
	Pokoju Ave.	50°06,664'N 20°00,151'E n=2
	Centralna St.	50°06,364'N 20°00,426'E n=2
	Lema St.	50°06,491'N 19°98,907'E n=2

averaged and homogenized. The samples were compared to the geochemical background values, which were provided as the average element content for the specific city district. The background values used for further analysis were provided separately for the two districts in Krakow by Lis and Pasieczna in 1995, taking into account their different dominant sources of pollution and contamination.

Analytical Methods

Total metal concentration

The total concentrations of heavy metals such as Cd, Cr, Cu, Mn, Zn, Co, Pb, Ni, Ba and Se were determined in bulk road dust samples, representing two different city areas locations, i.e., Nowa Huta District and Krowodrza District. Prior analysis, the samples were dried in 105°C and digested with *aqua regia*, according to 3050A protocol and then analyzed using ICP-MS ELAN 6100 Perkin Elmer.

Appropriate quality control of the performed analysis (both total content as well as fractionation study) was ensured by using reagent blanks and analyzing certified reference material BCR 723 (road dust) and SRM 1848a (urban particulate matter).

Metal contamination assessment of road dust

The contamination factor index (C_f), provided by Hakanson (1980), Suryawanshi et al. (2016), was then calculated according to the equation 1 as a ratio of the total concentration of a certain metal (C_i) in roadside dust to the geochemical background concentration of that metal for the city of Krakow C_n , provided by Lis and Pasieczna, (1995).

$$C_f = C_i / C_n \quad (1)$$

Contamination of the road dust with each individual metal was then classified according to the scale provided by (Hakanson 1980), where:

- $C_f < 1$: low degree of contamination
- $1 \leq C_f < 3$: moderate degree of contamination
- $3 \leq C_f < 6$: considerable degree of contamination
- $C_f \geq 6$: very high degree of contamination

Furthermore, the sum of contamination factors for all elements was used to quantify the degree of contamination C_{deg} of road dust.

$$C_{deg} = \sum C_f \quad (2)$$

- Where: $C_{deg} < 8$: low degree of contamination
- $8 \leq C_{deg} < 16$: moderate degree of contamination
- $16 \leq C_{deg} < 32$: considerable degree of contamination
- $C_{deg} \geq 6$: very high degree of contamination

VI step sequential extraction protocol

A fractionation study of 2 representative road dust samples was performed using VI-step sequential extraction, according to the modified method provided by Salomons and Förstner (1985). In order to fractionate metals in road dust and to obtain information on main phases in which metals are actually bound in the dust, different sets of reagents were employed as depicted in the protocol provided in Table 2.

Risk Assessment Code

Furthermore, to forecast the envirototoxicity of road dust metal pollution, the risk assessment code (RAC) was calculated according to the following equation provided by Perin et al. (1985).

$$RAC = (C_{F1,F2} / C_{total}) \times 100 \quad (3)$$

Where $C_{F1,F2}$ - is the concentration of elements in exchangeable, soluble fractions (first step of sequential extraction) and carbonates (second step of sequential extraction)

C_{total} - is the concentration of elements in the sample

Risk assessment code has been commonly used by multiple authors (e.g. Matabane et. al 2021, Marin et. al. 2022, Kowalik et al. 2022) to assess the availability of contaminants bound in various environmental samples and thus to determine the ecological risk posed by those toxic elements based on the quantified risk scale.

RAC risk is then classified according to I-V scale, when RAC value <1% is considered as no risk, RAC between 1-10%

Table 2. Sequential extraction procedure (modified Salomons and Förstner,1985)

Step of extraction	Extracted fractions	Detail extraction protocol
I	Exchangeable cations	1 M ammonium acetate, pH 7, solid/solution ratio 1:20, 2 hrs shaking time
II	Carbonate fraction	1 M sodium acetate buffer, pH 5; 1:20, 5 hrs shaking at 20°C
III	Easy reducible phases e.g. Mn oxide, partly amorphous Fe-oxyhydrates	0.1 M $NH_2OH \cdot HCl$ + 0.01 M HNO_3 , pH 2, dilution 1:100, 12 hrs shaking
IV	Moderately reducible phases (e.g., amorphous and poorly crystallized Fe oxyhydroxides)	0.2 M ammonium oxalate + 0.2 M oxalic acid, pH 3, dilution 1:100, 24 hrs shaking time
V	Organic fraction, inc. sulfides	30% H_2O_2 + HNO_3 , pH 2, 85°C, extracted with 1 M ammonium acetate, dilution 1:100, 24 hrs shaking
VI	Residual fraction (e.g. detrital silicates, crystalline Fe-oxides)	<i>Aqua regia</i> digestion*

* modification of the digestion protocol in 6th step, allowing comparison with the digestion method used in determining total concentration of metals in dust sample

is low risk, RAC between 11–30% is medium risk, RAC 31–50% is considered as high risk and finally RAC value >50% indicate a very high risk.

Results and discussion

Concentration of metals in road dust

The concentrations of metals in road dust samples, priorly averaged for each district location, are summarized in Table 3. Road dust (in bulk sample) from both locations was heavily enriched with heavy metals such as Cd, Cr, Cu, Mn, Zn, Co, Pb, Ni and Ba when compared to the geochemical values of these elements considered as natural content in the surrounding soils. The content of cadmium in road dust exceeded background values about 1.4–2.5 times, Cr 4.3–11 times, Cu 5.9 up to even 20 times, Mn about 2 times, Zn 3.6 up to 5 times, Pb 4–6 times, Ni 3.5–5 times and, finally, Ba concentration in road dust was a two-fold increase compared to the background value for this element in the surrounding soils. Cumulatively the road dust collected from 4 streets in Nowa Huta district was two times more contaminated with all of the examined metals than the dust collected from City Center Krowodrza District, that is 1989 mg/kg to 892 mg/kg respectively. This may be due to the fact that Nowa Huta district is a more industrialized part of the city.

The results on total metal content in urban road dust obtained in this study are mostly consistent with our previous research conducted in 2016 but also with the results reported by Miazgowicz et al. (2020) for road dust from Kraków.

The contamination factor index determined as a ratio of metal content in the road dust to the geochemical background value confirmed that road dust in Krowodrza is moderately contaminated with Cd, Mn and Co, considerably contaminated with Cr, Zn, Pb and Ni and highly contaminated with Cu. Road dust collected from Nowa Huta district was moderately contaminated with Mn and Co, considerably contaminated with Cd, Zn and Ni and highly contaminated with Cr, Cu and Pb. When considering contamination degree of road dust as a sum of individual C_i determined for each metal it was found that the road dust from Krowodrza represents a considerable degree of contamination according to the classification first introduced by Hakanson (1980), whereas the road dust from Nowa Huta should be classified as of very high degree of contamination.

However, since as previously stated, the information on the total concentration of metals in road dust is only semi conclusive when considering real ecotoxicity of dust, more informative on that matter, will be the results of the fractionation study, depicted in Fig. 1.

The results of chemical speciation revealed that despite the fact that Cr is present in the road dust in elevated concentrations,

Table 3. Heavy metals concentration in road dust

Element (mg/kg)	Road dust from the Krowodrza District	Road dust from the Nowa Huta District	Geochemical background value*
	mg/kg d.m.		
Cd	1.02±0.13	1.78±0.24	0.7
Cr	34.4±5.05	90.3±14	8.0
Cu	64.9±12	224±41	11.0
Mn	232±24	760±81	319
Zn	261±29	365±41	73
Co	4.32±0.46	6.46±0.69	4.0
Pb	85.6±11	132±17	22.0
Ni	32.2±6.8	43.9±9.3	9.0
Ba	98.9±18	104±19	52
Se	78.3±17	132±28	–

* background value for Kraków, according to Lis and Pasieczna, 1995

Table 4. Contamination Factor Index and Contamination degree determined for road dust

Location	Contamination Factor index C_f								C_{deg} sum of C_f
	Cd	Cr	Cu	Mn	Zn	Co	Pb	Ni	
Krowodrza	1	4	6	1	4	1	4	4	25
Nowa Huta	3	11	20	2	5	2	6	5	54

Cf classification	
<1	Low contamination
1–3	Moderate contamination
3–6	Considerable contamination
≥6	Very high contamination

C_{deg} classification	
<8	Low degree of contamination
8–16	Moderate degree of contamination
16–32	Considerable degree of contamination
≥32	Very high degree of contamination

which exceed 4 to even 10 times the concentration of this element in the background, still about 63% up to even 96% of this element is rather permanently fixed in residual and organic fractions, considered as immobile, thus not causing real ecotoxicological threat to the surrounding environment. Also Cu was found to be predominantly fixed with residual and organic matter fraction (77–81%), which should be considered as rather safe for the environment. However, as reported by Li et al. 2009, Cu^{2+} ions have a high affinity to soluble organic ligands, which in fact may attribute to the increase of this metal mobility in road dust.

Details regarding the speciation profile of each individual metals in road dust from both sampling locations are also presented in Table 5.

From 45% up to even 58% of cadmium in road dust was associated with the mobile and bioavailable fractions, such

as carbonates and exchangeable cations. The remaining part of Cd (31%) was bound with residual fraction, 14% with immobile organic fraction and only 7% with Mn oxides, 1% with amorphous and poorly crystallized Fe oxyhydroxides. Similarly, 43–50% of zinc was also bound with easily labile fractions (exchangeable and carbonates), 19–21% of zinc was associated with Mn-oxides and amorphous and poorly crystallized Fe-oxyhydroxides. 61–52% of Mn in road dust should also be considered as bioavailable, since it was bound with carbonates and exchangeable fractions.

Risk Assessment Code (RAC) was then used to assess and classify the ecological risk pose by toxic elements incorporated in the dust. It was found that there is a very high risk for Mn and Cd to be released from the road dust collected from Nowa Huta to the environment (RAC >50%) and a high risk (31–50%) for Zn, Mn and Cd to be released from the road dust collected from

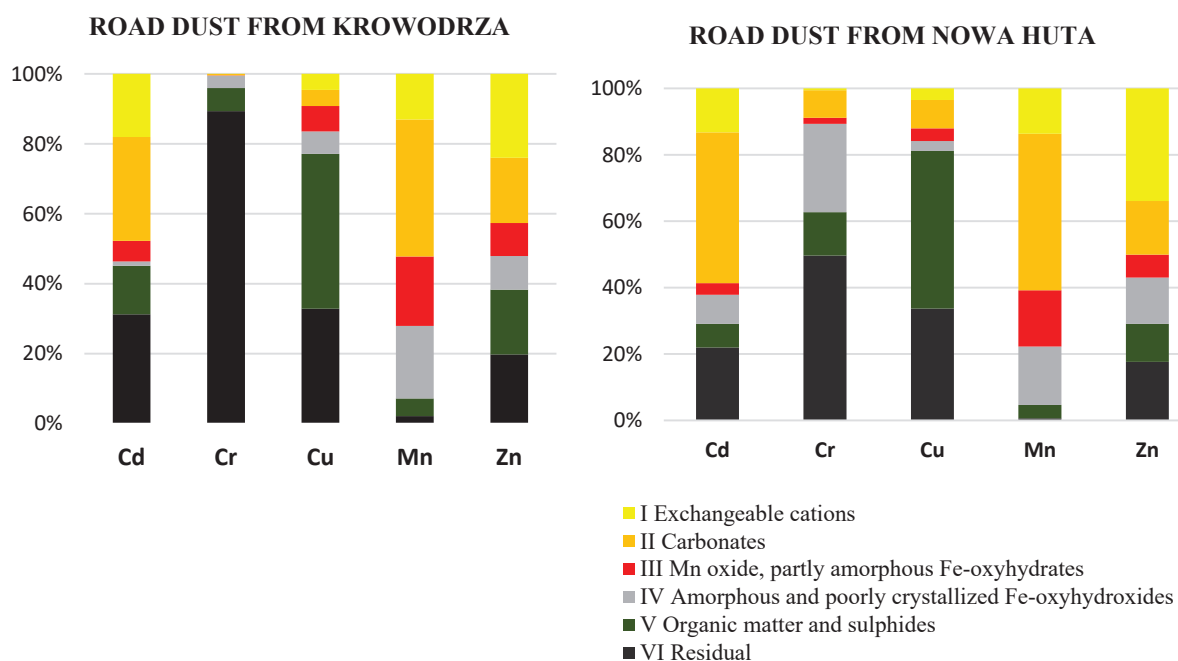


Fig. 1. Road dust fractionation

Table 5. Details on chemical speciation of metals in road dust

Chemical speciation of metals in road dust from Krowodrza District							
Metal	Residual fraction [%]	Organic matter and sulphides [%]	Amorphous and poorly crystallized Fe-oxyhydroxide [%]	Mn oxide, partly amorphous Fe-oxyhydrates [%]	Carbonates [%]	Exchangeable cations [%]	RAC [%]
Cd	31	14	1	6	29	18	43
Cr	89	7	4	0	0	0	0
Cu	33	44	6	7	5	5	10
Mn	2	5	21	20	39	13	42
Zn	20	18	10	9	19	24	43
Chemical speciation of metals in road dust from Nowa Huta District							
Cd	22	7	9	4	45	13	58
Cr	50	13	27	2	8	1	9
Cu	34	47	3	4	9	3	12
Mn	1	4	18	17	47	14	61
Zn	18	11	14	7	16	34	50

Krowodrza. Other metals such as Cu and Cr were classified as low to medium risk to the environment.

Conclusions

Our study revealed that the bulk fraction of road dust from two different districts in Kraków was heavily contaminated with Cd, Cr, Mn, Cu and Zn in amount multiple times exceeding geochemical background values, considered as pre-industrial content of metal in the surrounding soils. The content of cadmium in road dust exceeded background values by about 1.4–2.5 times, Cr by 4.3–11 times, Cu by 5.9 up to even 20 times, Mn by about 2 times, Zn by 3.6 up to 5 times, Pb by 4–6 times, Ni by 3.5–5 times and Ba by 2 times.

Cumulatively, road dust collected from Nowa Huta district was twice as contaminated with all the examined metals (classified as very high degree of contamination according to C_{deg} index) compared to dust collected from City Center Krowodrza District (classified as a considerable degree of contamination), probably due to the more industrial character of this part of the City. The metal's chemical speciation study followed by assessing the risk assessment code revealed, that except for Cr and Cu, all remaining metals such as Cd, Mn and Zn are mostly bound in dust with relatively labile and easy bioavailable fractions such as carbonates and exchangeable cations, indicating high and very high envirotoxicity (according to RAC classification).

Acknowledgement

This work was supported by the “Excellence initiative – research university” program for AGH University of Krakow and by the subsidy of the Ministry of Education and Science, grant No. 16.16.140.315.8

The article was partially presented during the XIIth Scientific Conference Air Protection in Theory and Practice, Zakopane, Poland, October 18–21, 2022.

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Badania geochemiczne i frakcjonowanie pierwiastków toksycznych w pyłach drogowych pobranych z głównych arterii komunikacyjnych Krakowa

Streszczenie: Zanieczyszczenia pochodzące z transportu drogowego uważane są za główny czynnik ryzyka środowiskowego odpowiedzialny za przedwczesne zgony na całym świecie. Rosnący udział emisji zanieczyszczeń związanych z komunikacją potwierdza konieczność oceny jakości środowiska drogowego poprzez określenia stopnia zanieczyszczenia pyłu drogowego oraz ocenę zagrożenia związanego z potencjalnym uwalnianiem się pierwiastków toksycznych z pyłów drogowych do środowiska wodno-glebowego. Do badań pobrano pył drogowy z 8 odcinków dróg miejskich o dużej kongestii w dwóch dzielnicach Krakowa (Krowodrza i Nowa Huta).

Stężenie metali ciężkich, takich jak Cd, Cr, Cu, Mn, Zn, Co, Pb, Ni, Ba i Se oznaczano metodą ICP–MS (ELAN 6100 Perkin Elmer). Frakcjonowanie przeprowadzono z zastosowaniem VI stopniowej ekstrakcji sekwencyjnej. Badania wykazały silnie zanieczyszczony Cd, Cu, Zn, Mn, Co, Pb, Ni, Ba i Se w ilościach przekraczających wielokrotnie wartości poziomów tła geochemicznego. Stężenia metali w pyłach drogowych kształtowały się następująco [mg/kg]: Cd 1,02–1,78, Cr 34,4–90,3, Cu 65–224, Mn 232–760, Zn 261–365, Co 4,32–6,46, Pb 85,6–132, Ni 32,2–43,9, Ba 98,9–104 i Se 78,3–132. Stwierdzono bardzo wysoki stopień zanieczyszczenia pyłu drogowego pobranego w Nowej Hucie (Cdeg 54) i wysoki w Krowodrzy (Cdeg 25). Badania form związków metali z zastosowaniem VI stopniowej ekstrakcji sekwencyjnej, a następnie ocena kodu oceny ryzyka (RAC) wykazały, że metale w pyłach drogowych są w znacznym stopniu mobilne i potencjalnie łatwo mogące się uwalniać do środowiska (metalami występującymi na pozycjach jonowymiennych i/lub węglany), z wyjątkiem Cr i Cu, które w większości związane są z siarczkami, materią organiczną, ewentualnie pozostają wbudowane w sieć krystaliczna minerałów.