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Mineralogy and mineral chemistry of snow filter residues in the vicinity of the nickel-copper processing industry, Kola Peninsula, NW Russia

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With 8 Figures

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Summary

Fifteen snow samples were collected at the end of the 1995/96 winter in the vicinity $(1.5-8 \text{ km})$ of the copper-nickel processing plants at Zapoljarnij, Nikel and Monchegorsk on the Kola Peninsula, NW Russia. Polished sections were made from the snow filter residues for microscopic and qualitative and quantitative electron microprobe investigations. Particles in snow are derived from either geogenic¹ or technogenic sources². Ore particles from two different Ni-Cu deposits can be identified in the snow: i) local Pechenga ore and ii) imported ore from Norilsk-Talnakh. Technogenic particles consist of a wide spectrum of sulphides (Ni-Cu-Fe-Co), oxides (Fe-Ni-Cu), metallic phases and alloys (Ni-Cu-Fe-Co), as well as slag particles and coke. Differences in the processing technologies used in the three plants (e.g. roasting, smelting to Cu-Ni matte, converting, refining) can be deduced from the mineralogy of the particulate emissions. The mineralogy of particulate matter in snow is characteristic for each emitter and can thus be used to fingerprint different sources. PGE-bearing sulphides (pentlandite with 0.2-0.3 wt% Pd) have been observed in the particulate emissions from Zapoljarnij and discrete PGE-phases at Monchegorsk.

Zusammenfassung

Mineralogie und Mineralchemie yon Schnee-Filtraten aus dem Bereich der Nickel-Kupfer Kombinate der Kola-Halbinsel, NW RuJ31and

Fünfzehn Schneeproben wurden in der Nähe (1.5–8 km) von Kupfer-Nickel-Minen und Htitten in Zapoljamij, Nikel und Monchegorsk auf der Halbinsel Kola, NW Rugland, am Ende des Winters 1995/96 genommen. Anschliffe von Schneefilterrückständen

wurden für die mikroskopischen Untersuchungen sowie für qualitative und quantitative Mikrosondenanalytik angefertigt. Die in den Anschliffen beobachteten Phasen sind teils geogener¹, teils technogener² Herkunft. Die geogenen Partikel sind Teil der lokalen Staubemissionen (Pechenga Ni-Cu-Tagebaue, Transport und Aufbereitung yon Erzen). Die technogenen Partikel repräsentieren ein breites Spektrum unterschiedlich zusammengesetzter Sulphide (Ni-Cu-Fe-Co), Oxyde (Fe-Ni-Cu), metallischer Phasen und Legierungen (Ni-Cu-Fe-Co), sowie Schlacke-Partikel und Koks. Variationen der Zusammensetzung und im Auftreten dieser Phasen ermöglichen Rückschlüsse auf die im Prozess eingesetzten Erze und auf verschiedene Stadien der Prozesstechnik (z.B. R6sten, Schmelzen auf Cu-Ni Stein, Konvertieren auf Cu-Ni Feinstein, Raffinieren) in den lokalen Hauptemittenten. Ausserdem konnten PGE-ftihrende Sulphide (Pentlandit mit 0.2-0.3 Gew% Pd) und separate PGE-Phasen in den partikulären Emissionen von Zapoljarnij und Monchegorsk identifiziert werden.

Introduction

The Kola Peninsula is second only to Norilsk, Siberia, as an area heavily polluted by nickel mining, roasting and smelting and includes some of the world's largest point source emitters of SO₂ and heavy metals *(Gunn et al., 1995)*. In 1994, official figures for annual emissions from the three plants in the area included 296,000 t SO_2 , 1900 t Ni, 1100 t Cu, 94 t V_2O_5 , and 92 t Co (Report of the Murmansk Region Committee of Ecology and Nature Resources, 1995 in *Reimann* et al., 1997a).

Over the past sixty years, the activities of the Russian non-ferrous metal processing industry have caused hundreds of square kilometers around the plants to become industrial deserts, where virtually all vegetation and humus are destroyed *(Tommervik* et al., 1995). The damaged areas can be easily detected and outlined on satellite images *(Hogda* et al., 1995). To assess the environmental impact of the nickel processing and associated industry in the Kola Peninsula, the Geological Surveys of Finland (GTK) and Norway (NGU), in cooperation with the Central Kola Expedition in Russia (CKE), in 1992 started an environmental geochemical mapping project-"Kola Ecogeochemistry" - (see http://www.ngu.no/Kola) in the western half of the Kola Peninsula and the adjacent areas of Norway and Finland. The Kola ecogeochemistry project consisted of three phases: a pilot project in 1992-1993, followed by a catchment study in 1994-1995, and a regional geochemical mapping phase in 1995-1996. A detailed description of the three Kola project stages as well as the results from different sampling media are given in *]{yriis* and *Reimann* (1995), *Niskavaara* et al. (1996), *Reimann* et al. (1996), *Caritat* et al. (1996a,b), *Caritat* et al. (1998), *Reimann* et al. (1997a,b,c), *Reimann* et al. (1998). The Geochemical Atlas of the Central Barents Region *(Reimann* et al., 1998) provides a comprehensive overview.

¹ Geogenic refers to particles originating from natural soil and/or bedrock sources (mainly silicates); in this paper, we include under this heading also anthropogenically generated geogenic dust derived from open cast mining, and handling of concentrates and tailings.

 $2^{\frac{1}{2}}$ Technogenic refers to products of technological processes (such as globular particles, slags, etc.)

In this work we report the results of mineralogical studies on snowpack samples. Geochemical data on trace elements and on platinum-group elements (PGE) in snow samples are given in *Gregurek* et al. (1998a) and *Gregurek* et al. (in press). The technique described here was originally developed by *H. Neinavaie* in the' late 1980's (personal communication; *Neinavaie* and *Pirkl,* 1996). An investigation using comparable techniques - but not quantitative microprobe analysis - on soil samples was carried out around Sudbury, the nickel mining and processing center in Canada, by *Adamo* et al. (1996). The main aim of this contribution is to correlate mineralogical observations and electron microprobe data with stages of processing technology and ore types. The results of mineralogical and chemical investigations of emitted particles deposited on snow permits distinction and comparison between the emission sources.

Sampling and analytical procedures

Snowpack sampling was carried out in April 1996 to collect the total atmospheric contribution of heavy and precious metals from the 1995/96 winter season. Fifteen snowpack samples were taken; seven near Monchegorsk, four near Nikel and four near Zapoljarnij, at distances of between 1.5 and 8 km from the plants. During sampling, two factors had to be considered; the distances from the plants and the prevailing wind direction (north-south). The total depth of snow cover was between 70 and 110 cm. A specially developed colourless Plexiglass tube of 1 m length and 92.5 mm internal diameter was used for sampling *(Soveri,* 1995). Snow samples were stored in polyethylene bags and kept frozen. After arrival at the GTK laboratory, snow samples were melted and filtered. The snow filter residues obtained after melting of 3 kg snow were embedded in Araldite resin, ground and then polished on a Rehwald polishing machine at the Institute of Geological Sciences, Leoben. Mineral compositions were determined using an ARL-SEMQ microprobe in the energy- and wavelength-dispersive mode $(20 \text{ kV}; 20 \text{ nA})$. Standards used included arsenopyrite, chalocopyrite, metallic Co, Ni, Pd and Pt, (Au, Ag)Te₂, chromite, ilmenite, magnetite, kaersutite, jadeite, anorthite, biotite and rhodonite. In addition, a JEOL scanning electron microscope (SEM) was used at the Institute for Ceramics at the University of Leoben for imaging fine-grained textures.

Geology and ore types

The Pechenga Zone is a part of an extended Early Proterozoic rift zone within the Baltic Shield *(Melezhik* et al., 1994a). It is composed of thick sequences of volcanic rocks (picritic to tholeiitic), minor intrusive bodies, and intervening sedimentary units, deposited upon the Archaean basement. The Pechenga Zone has been divided into two structurally different parts; the Pechenga series (sensu stricto) in the north and the South Pechenga series *(Abzalov* et al., 1997). The Pechenga series (sensu stricto) has been divided lithologically into the Ahmalahti, Kuetsjärvi, Kolasjoki and Pilgujärvi formations *(Abzalov* et al., 1997). Each formation comprises sediments which are capped by a thick volcanic sequence. The South Pechenga series is composed of mafic and intermediate volcanics and clastic sedimentary rocks; all the lithologies of the Pechenga Zone have been

affected by deformation and metamorphic overprinting, with metamorphic grades ranging from prehnite-pumpellyite to amphibolite facies *(Abzalov* and *Both,* 1997). The sedimentary part of the Pilgujärvi formation hosts Ni-Cu-bearing intrusions (the "Productive Pile"). All economic deposits occur in this zone *(Zak* et al., 1982). The disseminated, massive and breccia Ni-Cu-ores are hosted in gabbrowehrlite intrusions *(Melezhik* et al., 1994b). Several open-pit and underground mines (Tsentralny, Zapadny, Severny, Kaula Kotselvaara) are in operation with annual production of about 6.5 million tonnes of ore. The Pechenga ores contain 0.5-3 wt% Ni, 0.5-1.8 wt% Cu and 3-5 wt% S; the ores have Ni/(Ni+Cu) ratios of 0.5-0.7 *(Melezhik* et al., 1994a,b).

The Norilsk-Talnakh province includes several groups of ore bodies in picritic to doleritic sills associated with the Triassic Siberian flood basalt province *(Naldrett* et al., 1992, 1996). Contact metamorphism associated with the intrusive rocks has resulted in both prograde and retrograde metamorphic assemblages *(Likhachev,* 1994). The Norilsk-Talnakh ore province contains a wide spectrum of ore types; disseminated, massive and "copper" ore, hosted in different types of gabbro-dolerite intrusions. The nickel and copper contents in the disseminated ores are 0.4–0.6 wt% and 0.6–0.8 wt%, respectively. The massive ores contain $2-4$ wt% Ni and 3-25 wt% Cu, and are also rich in platinum metals with grades from 2 to 200 mg/kg PGE's *(Likhachev,* 1994). The Ni/(Ni+Cu) ratios of these ores range from 0.2 to 0.8.

Emission sources

Industrial activity in the western part of the Kola Peninsula, Russia started in the late 1930s with the discovery of the Petsamo (now Pechenga) nickel deposits, then part of Finland. The Pechenga area was ceded to the Soviet Union after World War II. Major sources of industrial emissions on the Kola Peninsula in NW Russia include: 1) the ore roasting and dressing (flotation) plants at Zapoljarnij, 2) the open pit mine near Zapoljarnij, 3) the nickel smelter at Nikel, and 4) the nickel, copper and cobalt smelter complex (refinery) at Monchegorsk (Fig. 1).

The ore roasting and dressing (flotation) plant at Zapoljarnij, built in 1965, processes local Pechenga ores only. Disseminated ores dominate in production (Pechenga Nikel, personal communication). The final products of this plant are Cu-Ni pellets i.e. a concentrate with 6wt% Ni. The smelter at Nikel started production in 1932 and, presently, processes Cu-Ni concentrates from local Pechenga ore, pellets from the Zapoljarnij roasting plant, rich Pechenga ores $(>3 \text{ wt\% Ni})$ and ores from Norilsk-Talnakh which are mixed in various proportions. The final product of the Nikel smelter is Cu-Ni converter matte. Annual production is estimated at 30,000-35,000 tons of Ni metal *(Melezhik* et al., 1994b). A flowchart of processing technology at Zapoljarnij and Nikel is given in Fig. 2. Roasting temperatures are in the 400° to 500° C range; smelting occurs at 1400° -1500 °C. Due to the high SO₂-emissions and losses of metals (compare also *Reimann* et al., 1996, 1997a) plans for modernisation of the Nikel smelter have been prepared, but their implementation has been delayed because of financial problems.

The much larger smelter complex and refinery at Monchegorsk, which is more advanced in comparison to Nikel, was built in 1938 and presently processes matte

Fig. 1. Location map of the study area, and the position of major industrial centers $(Zapoljarnij = ore$ roasting and dressing plant, Nikel = smelter, Monchegorsk = smelter complex and refinery)

and ore from Norilsk-Talnakh, pellets from Zapoljarnij and Cu-Ni converter matte from Nikel *(Boyd* et al., 1997; Pechenga Nikel, personal communication). Norilsk-Talnakh ore, processed at Monchegorsk since 1968, contains on average 2.35 wt% Ni and 2.7 wt% Cu *(Boyd* et al., 1997). The final products of the Monchegorsk smelter complex are refined nickel, copper and cobalt metal, and noble-metal sludge which is sent from Monchegorsk to Krasnoyarsk in western Siberia for further processing (Pechenga Nikel, personal communication). Sulphuric acid is also produced.

Mineralogy and mineral chemistry

Primary Pechenga and NoriIsk-Talnakh ores

The major and minor ore-forming minerals in the Pechenga and Norilsk-Talnakh ores are listed in Table 1. In the massive (breccia) and disseminated ores of the Pechenga deposits, pyrrhotite, pentlandite, chalcopyrite and magnetite are the

Ore roasting and dressing plant (Zapoljarnij)

Fig. 2. Flowchart of processing technology for the ore roasting and dressing plant at Zapoljarnij and for the smelter at Nikel (Pechenga Nikel, personal communication)

major ore minerals (Plate 1a). These are often integrown within a network of **metamorphic ino- and phyllo-silicates (Plate lb). Troilite, pyrite, cubanite, bornite, cobaltite-gersdorffite, nickeline, maucherite, breithauptite, galena, mackinawite, chromite and ilmenite are accessory constituents (see Table 1). A detailed**

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description of the mineralogy of these Ni-Cu ores is given by *Abzalov* and *Both* (1997) and *Abzalov* et al. (1997). Gangue minerals include olivine, pyroxene, amphibole (tremolite/actinolite, magnesio-hornblende), biotite, talc, chlorite and serpentine.

The mineral composition of the Norilsk-Talnakh ore is diverse *(Likhachev,* 1994) and includes the pyrrhotite group (troilite, monoclinic and hexagonal pyrrhotites), pentlandite, chalcopyrite-group (chalcopyrite, talnakhite, mooihoekite, putoranite), cubanite and magnetite. See Table 1 for mineral formulae. Accessory minerals include pyrite, valleriite, mackinawite, argentian penttandite, bornite, chalcocite, covellite, heazlewoodite, millerite, godlevskite, galena, sphalerite, chromite, chromian-magnetite, titanomagnetite, ilmenite and different platinumgroup minerals (see Table 1). There is variety of primary and secondary rockforming minerals: olivine, pyroxene (diopside - $En_{37-40}Fs_{13-17}Wo_{45-47}$), amphibole (tremolite/actinolite, hornblende), serpentine (antigorite), muscovite (sericite), biotite, talc, chlorite, garnet, epidote/clinozoisite, sphene, prehnite, pumpellyite, alkali-feldspars, plagioclase (An₀₋₇₀) and quartz *(Likhachev, 1994; Zolotukhin* and *Malyuk,* 1983).

Snow filter residues

All samples consist of a mixture of geogenic and technogenic particles in varying proportions *(Gregurek* et al., 1998b). Geogenic particles are primary minerals as can be found in the local bedrock and Pechenga ore. They are not affected by the industrial processes of roasting or smelting. The technogenic phases are particles which are products of different kinds of metallurgical processes. The size of opaque phases varies from a few up to $170 \mu m$. Larger rock-fragments exceeding several hundreds μ m in size and carrying different silicates, oxides and sulphides are also encountered.

Geogenic particles

The most widespread geogenic particles in snow fiom Zapoljarnij are sulphides (pyrrhotite, pentlandite, chalcopyrite, bornite, pyrite) which occur as independent grains, aggregates or as intergrowths within a network of metamorphic silicates such as serpentine (antigorite), amphiboles and chlorite (Plate lc). These metamorphic intergrowths are common in the local Pechenga ore processed in Zapoljarnij (compare Plate lc and Plate lb). Sulphides are often associated with oxides, mostly magnetite (Plate ld). Oxides (magnetite, chromite, Cr-spinel, ilmenite) associated with sulphides and silicates, or as independent grains, are common. Olivine, pyroxene, amphibole, serpentine, talc, chlorite, sphene, and feldspars are frequent.

In snow from Nikel and Monchegorsk a variety of geogenic phases (sulphides, oxides and silicates) is present. However, Monchegorsk samples carry more silicates than those from Nikel. There is quartz, alkali feldspars, plagioclase, serpentine, chlorite, talc, biotite, muscovite, amphibole, pyroxene, epidote/ clinozoisite, sphene and garnet. Magnetite, titanomagnetite, ilmenite, chromite, chromian-spinel, pentlandite, pyrrhotite, chalcopyrite and cubanite are mostly

Plate 1. a-f Photomicrographs of geogenic phases from primary Pechenga ores (a,b) and of filter residues (e-f) embedded in Araldite resin from Zapoljarnij, Nikel and Monchegorsk: a polished section of Pechenga ores with pyrrhotite, pentlandite, chalcopyrite and euhedral to subhedral magnetite, reflected light, oil immersion: b polished section of Pechenga ores with intergrowths of sulphides, oxides and silicates. Note flame-like crystals and aggregates of pentlandite in pyrrhotite, reflected light, oil immersion; e Zapoljarnij: sulphide (pyrrhotite) intergrowths within a network of metamorphic silicates (serpentine-antigorite), reflected light, oil immersion; d Zapoljarnij: chalcopyrite and pyrrhotite associated with magnetite, reflected light, oil immersion; e Monchegorsk: pentlandite associated with magnetite and isolated pyrrhotite grain, reflected light, oil immersion; f Nikel: rock fragment containing primary sulphides, reflected light. Width of photomicrographs a,e,f is 0.3 mm; b,c,d is 0.1 mm

present as monomineralic grains or as mineral aggregates (some of these are shown in Plate le). Rock-fragments containing silicates (chlorite, mica, amphibole, plagioclase, pyroxene/amphibole, prehnite) as well as oxides and sulphides have also been identified in polished sections (see Plate lf).

Mineral chemistry of geogenic phases

Cr-rich spinels from all three localities show a wide range of compositions with Crnumbers $[100 \text{Cr/(Cr+Al)}]$ varying from 30 to 78 (Fig. 3a), and Mg-numbers $[100\,\text{Mg/(Mg+Fe^{2+}})]$ from 1 to 30.

Clinopyroxene from Zapoljarnij is diopside $(En_{38-42}Fs_{11-18}Wo_{45-48});$ Mgnumber (Mg/(Mg+Fe²⁺+Fe³⁺+Mn) varies between 68 and 79), (see also Fig. 3b). In samples from Nikel and Monchegorsk, both clinopyroxene (diopside) and orthopyroxene (enstatite) are present. In Monchegorsk, diopside (En_{33-36} - $F_{s_{18-19}}W_{047-48}$ shows somewhat lower Mg-numbers (64-67) in comparison to Nikel (diopside - $En_{39}Fs_{14}Wo_{47}$; Mg-number 74), whereas orthopyroxene $(\text{En}_{68-78}F_{522-30}W_{00-2})$ at Nikel shows lower Mg-numbers (69–78) than at Monchegorsk (orthopyroxene - $En_{70-85}Fs_{13-25}Wo_{2-4}$; Mg-number 72-87) (see also Fig. 3b). Orthopyroxene contains 0.2 wt\% NiO and clinopyroxene 0.1 wt\% NiO, particularly in samples from Monchegorsk.

Different amphiboles can be distinguished from all three localities. Amphiboles (Mg-number 35 to 85) are enriched in Na₂O (up to 3 wt%), TiO₂ (up to 3.2 wt%) and K₂O (up to 2.3 wt%) and are low in Cr₂O₃ (<0.9 wt%). Cl⁻ and F⁻ contents in amphiboles are below the detection limit of the electron microprobe. The chemical compositions of these amphiboles are plotted in Fig. 3c and 3d. Two different trends can be observed - from tremolite (actinolite) towards tschermakite compositions, and towards pargasite (hastingsite) compositions (alkali- and A1 rich amphiboles) (see Fig. 3c). Mg-hornblende and tremolite (actinolite) are the most common amphiboles (Fig. 3d).

Technogenic particles

In spite of the dominance of geogenic particles in snow from Zapoljarnij, technogenic particles (oxides and sulphides) are also present. Magnetite is partly or completely transformed into hematite (Plate 2a) and probably maghemite. Sulphides are often surrounded by hematite or by magnetite and hematite/ maghemite (Plate 2b).

The morphological types in samples from Nikel and Monchegorsk include:

(1) globular particles approaching a spherical shape and representing melt relics emitted through smokestacks;

(2) irregular particles without spherical shapes and frequently showing oxidized rims, derived from material not heated to melting temperatures;

(3) euhedral to subhedral phases (N_3S_2 , Cu_2S) crystallised during Cu-Ni converter matte production *(Pawlek,* 1983).

In samples from Nikel, technogenic phases differ significantly in shape (globules and spherules) from the technogenic particles encountered at Zapoljarnij,

Fig. 3. a-d Compositional variation of geogenic phases (oxides and silicates) in filter residue samples from Zapoljarnij, Nikel and Monchegorsk; a 100 Mg/(Mg+Fe) vs 100Cr/ (Cr+Al) for Cr-rich spinel; **b** quadrilateral diagram for pxroxenes; **c** $(Na+K)$ _A vs Al(IV) **diagram for amphiboles; d 100Mg/(Mg+Fe) vs Si per formula unit (p.f.u.) diagram for amphiboles**

High reflectance sulphide globules (Plate 2c), (R_{air} > 40%) and lower reflectance oxide globules (R_{air} ca. 20%) predominate. Globules shown in Plate 3a consist of **Ni- and Cu-rich iron sulphide surrounded by a glassy matrix with crystallized dendritic iron oxides [wtistite/magnetite]; those in Plate 3b consist of Cu-(Ni)-Ferich sulphide, skeletal magnetite and replacement rims of iron oxide. The intergranular texture within a globule shown in Plate 3c is formed by thin Cu-rich iron sulphide occurring as exsolutions between individual Ni-(Cu)-Fe-S aggregates constituting the globule matrix. Subhedral to anhedral sulphide and oxide grains are also present. Sulphides are often surrounded by oxide rims similar to those** from Zapoljarnij (Plate 2d). Dark, very low reflectance (R_{air} < 10%) slag particles **are common. These may have different shapes and textures (see Plate 2e). Slag** particles usually consist of a glass phase with enclosed crystalline phases, e.g. skeletal or elongated, euhedral to subhedral olivine, as well as globular and euhedral to subhedral Fe-(Ni-Cu)-sulphides. Cr-Al-spinel, chromite and magnetite also occur in the glass matrix (Plate 2f). Particles of unprocessed ore and/or concentrate with small amounts of fluxes and coke are additional technogenic phases. Coke and graphite are also present (Plate 2g).

In samples from the vicinity of the Monchegorsk smelter complex (refinery), the technogenic phases resemble those from Nikel i.e. they are: a) globules, b) subhedrat to anhedral sulphides and oxides, c) slag particles, d) widespread particles of ore mixed with fluxes and coke, e) coke and graphite. However, phase associations in samples from Monchegorsk differ significantly from the Nikel samples. Differences include: 1) the occurrence of metallic phases and alloys (Plate 2h); 2) the presence of different Cu- and Ni-rich technogenic silicates (micas); 3) widespread euhedral to subhedral sulphides such as Cu -rich ($Cu₂S$) and Ni-rich $(Ni₃S₂)$ phases; 4) occurrence of Cu-chloride (CuCl₂), mostly as globular phases (Plate 3d); 5) relative scarcity of slag particles; 6) presence of independent PGE minerals.

The mineralogical form, composition and abundance of emitted particles differs between the local point emitters.

Mineral chemistry of technogenic phases

The chemical compositions of Cu-rich and Ni-rich technogenic sulphides from Zapoljarnij, as well as euhedral to anhedral sulphides from Nikel and Monchegorsk, are shown in Fig. 4a and 4b. In the Fe-Ni-S system such phases plot mostly within the monosulphide solid solution field (mss, $(Fe, Ni)_{1-x}S$). Especially in samples from Zapoljarnij (see Fig. 4a) a wide range of compositions between the two endmembers – pyrrhotite ($Fe_{1-x}S$) and millerite ($Ni_{1-x}S$) can be observed. The mss also carries minor amounts of copper (up to 2wt% in Zapoljarnij and < 1 wt% in Nikel and Monchegorsk). In the Cu-Fe-S system, the Cu-rich technogenic sulphides from Zapoljarnij, Nikel and Monchegorsk plot mostly close to the positions of natural ore minerals (Fig. 4b).

The wide compositional ranges of globules (spherules) from Nikel and Monchegorsk are plotted in the S-Fe-(Ni+Cu+Co)- and $(Fe+S)$ -(Ni+Co)-Cu-

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Plate 2. a-h Photomicrographs of technogenic phases in polished sections of snow filter residues from Zapoljarnij, Nikel and Monchegorsk: a Zapoljarnij: magnetite almost completely transformed into hematite, reflected light, oil immersion: b Zapoljarnij: technogenic sulphide surrounded by two different oxides (magnetite and hematite/ maghemite), reflected light, oil immersion: c Nikel: different globular phases, reflected light, oil immersion; d Nikel: sub- to anhedral sulphide surrounded by oxides (magnetite and hematite/maghemite), reflected light, oil immersion; e Nikel: slag particles of different shapes, reflected light; f Nikel: crystalline idiomorphic to hypidiomorphic olivine (ol, darker) and euhedral Cr-Al-spinel (sp, lighter) within slag particle, reflected light; g Nikel: Coke particles, reflected light; h Monchegorsk: metallic phase, reflected light, oil immersion. Width of photomicrographs a,b,h is 0.1 mm; $c-g$ is 0.3 mm

Plate 3. a-d Backscattered electron images on SEM showing textures of globules from Nikel and Monchegorsk: a Nikel: globule consisting of Ni-(darker) and Cu-rich (lighter) iron sulphide surrounded by glassy matrix with crystallized dendritic iron oxide (wüstite (FeO)/magnetite); b Nikel: Cu-(Ni)-Fe-rich sulphide globule with skeletal magnetite and rim of iron oxide: e Nikel: intergranular texture formed by Cu-rich iron sulphide (light) occurring between individual Ni-(Cu)-Fe-S aggregates constituting a globule; d Monchegorsk: Cu-chloride (CuCl₂) globular phase with light and dark constituents. The extremely fine grained particles precluded microprobe analysis

ternary systems (Figs. 5a and 5b). The globules from Nikel tend to be richer in iron and sulphur compared to Monchegorsk globules which have higher nickel, cobalt, and especially copper contents. The chemical compositions of some representative technogenic sulphide phases and globules from Nikel and Monchegorsk are given in Table 2.

Compositional variations of technogenic oxides are shown in an FeO_{tot} vs NiO diagram (Fig. 6). Two groups of technogenic oxides can be identified: a) high

sulphides from Nikel and Monchegorsk presented in a) Fe-Ni-S- (wt%) and b) Cu-Fe-S (at%) ternary systems. an-anilite Ni₇S₆), hc-haycockite (Cu₄Fe₅S₈), hz-heazlewoodite (Ni₃S₂), ma-marcasite (FeS₂), ml-millerite (NiS), mh-mooihoekite $\left(\text{Cu}_7\text{S}_3\right)$, *bbcv* "blaubleibender" covellite (Cu_{1+x}S), *bn*-bornite ($\text{Cu}_3\text{F}e\text{S}_4$), *cb*-cubanite ($\text{Cu}\text{Fe}_2\text{S}_2$), *cc*-chalcocite (Cu_2S), *cp*- $\stackrel{\sim}{\sim} \tilde{\rm e}$ **%** Ξ တွိ တွိ Z °~ $\hat{\mathcal{L}}$ $\tilde{\pi}$ $^{\circ}$ … ਨੀ $\rm (Cu_9(Fe,Ni)_{8}S_{16})$

Fig. 5. Chemical composition of globular phases in samples from Nikel and Monchegorsk plotted in a) S-Fe-(Ni+Cu+Co)- and b) (Fe+S)-(Ni-Co)-Cu-triangles

Fig. 6. Compositional variations of technogenic oxides shown in a FeO_{tot} vs NiO diagram

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	N-15	N-15	$N-15$	$N-14$	N-14	M-7	M-7
SiO ₂	45.00	29.88	26.17	40.95	32.54	43.46	39.94
TiO ₂	0.93	0.54	0.16	0.62	0.52	0.30	0.20
Al_2O_3	7.94	6.77	3.88	7.30	6.34	6.04	6.09
Cr_2O_3	0.17	0.08	<0.03	0.54	0.17	< 0.03	0.08
FeO _(tot)	26.71	52.96	63.67	35.73	49.58	42.55	41.68
MnO	0.11	0.09	0.08	0.09	0.13	0.12	0.28
MgO	9.61	2.80	0.79	7.06	3.78	3.35	7.16
CaO	5.56	2.75	1.48	3.00	2.91	2.75	3.56
K_2O	1.09	0.63	0.51	0.71	0.73	0.61	0.45
Na ₂ O	1.49	1.30	1.06	1.57	1.38	0.43	0.21
N _i O	0.15	0.07	0.60	< 0.06	0.08	0.11	0.81
CuO	${}_{< 0.07}$	0.18	0.68	0.12	0.22	0.13	0.45
S.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
\sum	98.76	98.05	99.08	97.69	98.38	99.85	100.91
pl	0.1	0.1	0.1	0.1	0.1	0.1	0.1
p2	0.3	0.2	0.1	0.2	0.2	0.1	0.3

Table 3. Representative microprobe analyses of slag particles from Nikel and Monche*gorsk. Same abbreviations as in Table 3. (<)- values below detection limit,* n.d. *not determined*

pl CaO/SiO2; *p2* (CaO+MgO)/SiO2

Fig. 7. Chemical composition of stag particles in snow samples from Nikel and of slags from the Nikel smelter plotted on an $SiO₂$ vs (FeO+Al₂O₃) diagram. *Data from *Sevryukov* (1975) and *Koch* and *Janke* (1984)

FeO_{tot} (up to 95 wt%) and low NiO (up to 25 wt%) from all three localities and b) low FeO_{tot} (up to 20 wt%) and high NiO (up to 90 wt%) from Monchegorsk only. Technogenic oxides from Nikel and Monchegorsk contain up to 4wt% MgO whereas technogenic oxides fiom Zapoljarnij contain up to 9 wt% MgO.

The chemical composition of slag particles from Nikel and Monchegorsk can be expressed by their major components $-$ CaO, SiO₂, FeO, MgO and Al₂O₃. Minor components are Na₂O (1-2 wt%), K₂O (up to 1.1 wt%), Cr₂O₃ (up to 0.5 wt%), CuO (up to 0.7 wt%) and NiO (up to 0.8 wt%) (Table 3). Slag acidity/ basicity has been calculated as $CaO/SiO₂$ (p1) and as $(CaO+MgO)/SiO₂(p2)$, *(Koch* and *Janke,* 1984). The values for pl are lower than 0.1 and for p2 range from 0.1 to 0.5. According to *Koch* and *Janke* (1984) values below 1 are characteristic of acid slags. The chemical composition of slag particles in snow samples from Nikel was compared with slags from the Nikel smelter and plotted in the $SiO₂$ $vs(FeO+Al₂O₃)$ diagram (Fig. 7). The Nikel slag data for melted snow samples and for the smelter plot close to the compositions of slags from the blast furnace and converter (data after *Sevryukov,* 1975; *Koch* and *Janke,* 1984). The slags from the blast furnace are Si-rich and have lower $FeO + Al₂O₃$ than the converter slags (lower $SiO₂$ and higher FeO+Al₂O₃) (compare Fig. 7).

The metallic phases and alloys in the Monchegorsk samples contain Ni (85–90 wt%), Cu (9–12 wt%), Fe(1–3 wt%) and Co (0.3–0.9 wt%).

Mineralogy of PGE-bearing phases

In snow filter residues from Zapoljarnij and Nikel no distinct PGE minerals have been found. This is consistent with the fact that PGE minerals (arsenides, selenides and tellurides) are rare in Pechenga type deposits *(Abzalov* and *Both,* 1997). However, in snow from Zapoljarnij PGE were detected by electron microprobe in some primary sulphide grains (e.g., $0.2-0.3 \text{ wt\%}$ Pd in solid solution in pentlandite). Although this is close to the detection limits of microprobe analysis, the observation is backed by Pd-values of up to 196ng/1 in filter residues from Zapoljarnij.

Independent PGE minerals (Pt-Fe-alloys) and Pt-Pd-Au-rich, Cu-Cr-Fe-Nisulphides (Fig. 8) have been found in snow from Monchegorsk. Unfortunately

Fig. 8. Back-scattered image of (1) Pd-Pt-Au rich technogenic Cr-Cu-Ni-Fe-sulphide. Cr-Cu-Ni-Fe alloy (2) are also present. Snow filter residue, Monchegorsk (M-7)

these phases are too small $\left(\langle 1 \mu m \rangle \right)$ for representative microprobe analyses, Isoferroplatinum (Pt_3Fe) and Tetraferroplatinum ($PtFe$) in Norilsk-Talnakh ore were reported by *Distler* (1994) and their occurrence in Monchegorsk snow points to their possible derivation. The importance of Norilsk ores for the Monchegorsk industrial complex has been stressed before.

Discussion and conclusions

The excellent preservation of a wide variety of geogenic and technogenic particles from the emissions of one winter season in snow samples and their suitability for reflected light and microprobe studies is the most remarkable result of this study. Distribution of geogenic and technogenic particles in snow samples is shown in Table 4. However, some important differences in mode of occurrence of geogenic and technogenic particles can be observed. These are summarized below:

a) In the surroundings of the Zapoljarnij open pit and ore roasting and dressing plant, primary ore particles (sulphides and oxides), silicates and rock fragments unaffected by industrial processes dominate the snow samples, thus reflecting the mineralogy of local Pechenga ores processed at Zapoljarnij. The "primary" sulphides contain PGE in solid solution; pentlandite with 0.2-0.3 wt% Pd has been observed. The °'primary" particles can be attributed to windblown dust from 1) the mining operations, 2) ore transport, 3) crushing and processing (flotation). Ore minerals may also be lost during flotation (70% recovery for Ni, and 80% recovery for Cu; Pechenga Nikel, personal communication); thus, windblown dust from tailings (grain size $\langle 74 \mu m \rangle$ can be considered as a further source of particles. Some of these may also escape during the initial stages of roasting (drying after flotation) in the Zapoljarnij roasting plant.

b) Geogenic particles in snow from Nikel can probably be attributed to dust emissions from ore transport and ore processing during production of the Cu-Ni concentrate at Nikel.

c) Geogenic particles are also encountered in snow samples collected in the vicinity of the Monchegorsk smelter complex. Silicates are more frequent than at Nikel. Mining activities have ceased in the Monchegorsk area (Pechenga Nikel, personal communication) so that dust emissions must be ascribed to dressing of Norilsk-Talnakh ores.

The chemical composition of geogenic phases reported above has not been affected by metallurgical processing. Thus, smokestack emissions can be excluded as potential sources.

Technogenic particles represent material which has escaped at different stages of metallurgical processing, thus showing different mineralogical forms and chemical compositions:

a) In Zapoljarnij, technogenic particles consist of oxides, sulphides, coke and graphite; globular particles are absent (Table 4). Primary pentlandite was changed to monosulphide solid solution (mss) plus heazlewoodite ($Ni_{3\pm x}S_2$) according to the reaction:

pentlandite = Fe-rich mss + $Ni_{3\pm x}S_2$

	Zapoljarnij	Nikel	Monchegorsk			
geogenic						
sulphides	$++$	$++$	$++$			
oxides	$++$	$++$	$^+$			
silicates	$++$	$^{+}$	$++$			
rock fragments	$++$	$^{+}$	$+$			
technogenic						
sulphide (globules)		$++$	$++$			
sulphide (phases [*])	$++$	$+$	$++$			
oxide (globules)		$++$	$++$			
oxide (phases*)	$++$	$++$	$++$			
alloys			$++$			
slags		$+ +$	$+$			
coke						

Table 4. *Distribution of geogenic and technogenic particles in snow samples from Zapoljarnij, Nikel and Monchegorsk (qualitative estimate, only)*

 $-$ absent; $+$ rare; $++$ frequent; $*$ technogenic phases other globules (lack of spherical shape)

during roasting at temperatures of approximately 600°C *(Kullerud,* 1963). Heazlewoodite should also be present if the reaction had gone to completion. However, its absence points to incomplete reactions. The mss is stable within a wide range of temperatures (300° to 1000°C, *Kullerud*, 1963). Sulphides surrounded by magnetite, and hematite/maghemite can probably be attributed to sequential oxidation during the ore roasting process.

b) In samples from Nikel, similar technogenic phases to those reported in Zapoljarnij are observed (Table 4). They frequently show oxidized rims, suggesting that oxidation of sulphides is caused by air blown into the furnace at an early stage of the smelting process. Temperatures (several hundred degrees C) at this stage were too low to melt sulphides; the "primary" shapes tend to be preserved, but not the chemical compositions. Compared to Zapoljarnij, the majority of particles consist of globules and slags. Globules, representing molten particles, show textures which can be attributed to different stages of smelting (e.g. smelting of ore/concentrate, melting of fluxes, and rapid crystallization of iron-bearing glassy material; they escape due to introduction of air into the furnace; compare with *Sevryukov,* 1975; *Pawlek,* 1983). On the other hand, slag particles can be attributed to transport of material from an adjacent slag dump (grainsize generally $< 100 \mu m$). Additionally, mixtures of ore/concentrate, fluxes and coke encountered at Nikel represent "primary" charge fed into the smelter. Coke particles may derive from fuel for combustion used in the pyrometallurgical processing.

c) In samples from Monchegorsk, technogenic particles seem at first glance to be similar to those from Nikel. However, some important differences are observed:

(1) Alloys $\ll 90$ wt% Ni and $\lt 12$ wt% Cu) occur exclusively at Monchegorsk. Ni-Cu-rich phases such as euhedral to subhedral $Cu₂S$ and $Ni₃S₂$ also occur at Nikel, but in smaller amounts. These sulphides and alloys are typical of Cu-Ni converter matte *(Pawlek, 1983)*. Globular Cu-chloride *(CuCl₂)* is restricted to Monchegorsk samples. The formation of $CuCl₂$ is ascribed to addition of NaCl as a flux and of gaseous chlorine $(Cl₂)$ during nickel refinement at the late stage of the metallurgical process *(Pawlek,* 1983). Thus, further sources of particulate emissions are associated with an advanced stage of processing.

- (2) The differences in chemical compositions between globules from Nikel and Monchegorsk are probably due to different technologies used.
- (3) The presence of technogenic PGE minerals (Fe-Pt-alloys) and Pt-Pd-Au-rich Cu-Cr-F-Ni-sulphides can be correlated with the PGE-rich Norilsk-Talnakh ore presently processed at Monchegorsk. Pt-Pd-Au-rich Cu-Cr-Fe-Ni-sulphides have probably been derived from technogenic material at an advanced stage of processing technology.

The mineralogical phases identified in snow and derived from emissions are thus a characteristic "fingerprint" of the industrial processes used. This approach can also be applied to deduce the technological processes utilised in different plants. Furthermore, it is possible to apportion the phases found at individual localities to their original pollutant source, thus allowing a direct evaluation of how much of an observed contaminant is derived from a particular source, even in complex situations with many likely polluters. Thus, semiquantitative estimation of the proportions of contaminants derived from specific emitters becomes possible. These results are the more remarkable if we consider that the weight of filter residue material available from each sample was in the 0.1–0.5 g range.

At present, soil samples taken at the same locations as snow samples are being investigated by identical techniques. It is intended to follow the particulate emissions from the melting snow to the soil, where particulate emissions have accumulated over many years and where their response to weathering can be studied. Data obtained from snow and soil underline the importance of mineralogical studies in solving environmental problems. These should always be performed in conjunction with chemical analyses; they provide unique indicators both for the type of residence of potentially toxic elements (sulphides, oxides, silicates, alloys, etc.) and for their derivation from distinct polluting sources.

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