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# MINERAL NOVELTIES IN SOILS, FORMED ALONG THE GAS PIPELINES IN SLOVAKIA

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**Abstract.** Presented are selected results, found by the assessment revision of X-ray analyses records. Analyses were made from the bottom soils layers and thin coatings along the older gas pipelines in different regions in Slovakia. In some places the tendencies to form solid crustal were found. Apart of common soil minerals with some amorphous phases, the crystalline coatings, precipitated along the pipelines were analyzed. According to specific local conditions, rare mineral novelties were revealed there. Using the XRD method, the carbonates nahcolite, thermonatrite and also Ca-nesquehonite were determined; beyond its acurate identity also phase sX and amorphous admixtures we found.

**Key words:** *gas-pipeline soil, X-ray diffraction, phase analysis, mineral novelties*

## 1. INTRODUCTION

Nearly all gas-pipeline branches – similarly the other antropogene underground transportation or communication devices – represent the breakdown, or highly porous zones, ideal for the water solutions migration. By the investigation, focused onto gas pipeline defects (held by SPP a.s., Slovakia), also some mineralogical research works were engaged. These should solve the task on composition of local solid incrustations, formed along the pipelines construction, same as identification of found newly formed phases, present as dusty thin film, crystaline coatings to restite layers. More of it have formed directly at the pipeline isolation cover, but also in soil pores and cavities. Among the common suite of soil mineral, several naturally very rare phases were identified. Here´s getting up the questions, related to its presence, to interprete its origin, the influence of local soil mineral composition, eventually possible using of additive bed materials were searched too.

The article we suppose as the contribution of applied mineralogy, based on utilizing the X-ray analysis, allowing to point out onto some rare phases occurence. It also follow the ambition to interprete formation of found novelties in soil by the precipitation, accelerated along the pipes. Because of specific interest of company SPP a.s. we ommit any technical specifications connected to exact position, construction or diameter of pipeline branches, or threating technical informations.

## 2. USED ANALYTICAL METHOD

Mineralogical interpretation of soil material phase composition was based on X-ray analyses of around 30 various samples, which offer exact information about very fine soil minerals identity, same as the crustal novelties. As most suitable, the powder X-ray diffraction method (XRD) was chosen. By this method the pulverized sample, set in flat holder was exposed to monochromatic X-rays under various angles to record diffracted reflexes from crystalline structural planes. Positions of detected diffraction lines is typical for each mineral / group of minerals, so the calculated $d_m$-data file is fully sufficient for phase objective identification. Most samples from three localities were mineral mixtures. Because of X-ray diffraction apparatus susceptibility, the records contain the information about main and minor minerals only, which contents exceed 1-3 % and its mutual ratio too.

Nearly all samples were analyzed in Dept. Metallography and Failure Analysis, GM Research and Development, U. S. Steel Košice, using the high quality diffractometer Seifert XRD 3003 PTS. The technical conditions were: extinction CuKα, ($\lambda$ = *0,1541nm*), monochromatic Ni-filter, goniometer´ angle positions of diffracted lines: 10° 2θ to 110° 2θ . Each made XRD record was recalculated to dm (nm) and determined by apparatus software ZDS-Search Match. All analyses were included to several USS Technical Reports (Černík 2007, 2008a and 2008b).

Realized mineralogical revision of wide diffraction patterns had included import of measured dm/Im data from the format .nja to .xls, extraction of relevant angles extent (~up to 65° 2 theta) and amplifying of repressed suite of peaks to contrast level. All peaks (diffraction lines) in graph were described by $d_m$ $[nm]$ in accordance to Bragg equation for $CuK_\alpha$; with missing data completion using the Cheman software. Most of former identifications, using the automatic system ZDS-Search Match was correct, but some proclaimed minerals was necessary get to correspond with proper, low temperature minerals, real in soils. Appraisal of all diffraction patterns use to make by hand, comparing it to JCPDS tabs, matching the $d_m$ / $I_m$ to $d_t$ / $I_t$ standard files, but here we had used more exact American Mineralogist Crystal Structure Database. Comparing tabular data summarize tabs 1 and 2.

Absence of low angle diffraction span (below 10° 2θ) in some samples did not allow revision of some phylosilicates presence, which was primarily beyond the study scope. On the other hand, composition of several samples (in relation to pipeline position and treating operation) was checked also using the chemical analyses. Documentation contains also series of photographs.

## 3. STUDIED MATERIAL

Soil samples came from three distant localities, where the investigation of gas-pipeline defects and its protection was pursued. Nearly all samples came not from the surface soil in searched line, but from the soil surroundings laid close to isolation cover – especially bottom side along the pipes. First locality was gas distribution branch by Chynorany, close to Nitra in the western Slovakia. Second locality was pipeline branches by Košice city – in eastern Slovakia – same as the third locality Kluknava.

Soil samples of all three localities were from the long outcrops, without seen application, or any remarks of imported bed dusting material, other than original local loam. Because of older age of each pipeline branch (up to 40 years), the relevant information, whether sandy or any other bed dusting were applied was missing. In opposite to crumble soil layers from Chynorany and Košice, the third locality samples were from compact incrustation of rigid consistency (Fig. 1b).

Outcrops samples were taken off by employees of SPP-Distribution a.s. and delivered to GM Unit X-ray laboratory, USS Košice, with next step selection, processing for the analyses, eventually separation. Each sample, analyzed by XRD and its documentation was suffixed by internal marking (for ex. V574, V675…).

Nearly all analyzed and reinterpreted (revised) samples were of polymineral / polyphase composition. Therefore fitting the individual peaks in XRD pattern to contained different mineral, they belong to, was marked by species abbreviation (Fig. 2 and 3), for ex. Qz, It, Pl…., as mentioned in text, but found out XRD-data show not only crystalline mineral contents. In almost all made X-ray records – as seen from the presented diffraction lines intensities – among found minerals quartz (*Qz*) dominate here (despite its low apsorption coefficient).

To be the focus of attention, frequent contents of carbonate minerals in soil samples were recorded. Beside calcite (*Cal*), dolomite (*Dol*) in two samples and perhaps also magnesite (*Mag*; only sample) were recorded too. We suppose mostly its natural origin, (clasts or binding constituent in sediment?), but along with next stage precipitation proceses, also secondary origin in soil samples we cannot exclude. Generally as another soil mineral components were found: plagioclase (*Pl*), illite (*It*), muscovite (-sericite?; *Ms*) and chlorite (*Chl*). In case of plagioclase, its closer chemistry by X-ray method it is not possible to determine; but with highest probability all are “oligoclase”, or albite. Samples´ X-ray records frequently contained main reflex of K-feldspar (*K-F*, microcline). Studied material contained only small amounts of typical clay´ mineral: illite. Having this analogue vs. muscovite show its lower ordered structure: illite manifest lesser of coherently diffracted domains by typical diffusive reflexes. Analogue phylosilicate structures and therefore equivalent angle positions of its basal diffraction lines have also chlorite group if compare to the other clay mineral: kaolinite. Prevailingly to chlorite addition show calculated lower dm-data value of the I=100 reflex (dm 0,705 to 0,710 nm), in some patterns present 1st chlorite diffraction line dm 1,412 nm, and also other decisive reflexes differences.

Except of common natural minerals, as much more interesting we suppose found admixtures of the phases, very rare or nearly absent in natural rocks. Such are the crystalline hydrated Na-carbonates: nahcolite (*Nah*), thermonatrite (*Tn,* Fig 1a), Ca-nesquehonite (*Nes*), event. sulphate phase sX. In found position we take them for the mineral novelties. Because in most samples they form minor components, for its correct analysis and identification the monomineral samples were separated from thin sheets or coatings from gas pipeline envelope.

With the exception of three samples, the latitudes of revised X-ray patterns allocate the amorphous phases indicia too. It expresses itself by oval uplift of the graph background intensity, nearly ever within the angle section 9° to 18-19° 2θ.

Fig.1a. Mineral novelty coating, Chynorany;

Fig.1 b. Solid incrustation, Kluknava

## 4. RESULTS AND DISCUSSION

### 4.1. Composition of soil bed samples from the locality Chynorany by Nitra

The common soil sample around the local gas distribution branch show to be descendant after the silt sediments. It is dominantly formed by quartz, with minor plagioclase, K-feldspar (microcline), and also some amorphous phase. The only local sample is unusual with high contents of dolomite; the X-ray analyses of another samples detected rather calcite, admixtures of muscovite, illite and chlorite. All these minerals we consider to be original sediment components.

Interesting from the mineralogical sight of view is proved nahcolite presence. Sample for the analysis was separated from the white crystalline coating in the pipeline isolation (*sample V574*). This naturally very rare sodium hydrocarbonate $Na(HCO_3)$ is known only from the salt lakes environment. Similarly another sample, taken from the bottom side with welded pipe join, the thin mineral layer of the crystaline phase (Fig.1a) was identified as thermonatrite, $Na_2(CO_3)\cdot H_2O$ (*V577*), mixed with more hydrated impurity of phase $Na_2(CO_3)\cdot 7H_2O$. Besides of mentioned natural occurences in lake evaporites, the thermonatrite was rarely found in contaminated salty soils from the Danube basin in Southern Slovakia. Here it should occure as the consequence of soil agrochemical pollution and seasonal irrigation. Especially the thermonatrite efflorescence (together with gypsum and nathron) are considered to be the product of groundwater dissoluted components and its crystallization around underground steel structures, with developed corrosive phenomena (Milička et al, 2005).

Table 1: Interplanar structural distances d and intensities I of diffracted lines of 5 mineral standards, frequent in revised X-ray records (by AMS-DATA)

| main tabular diffraction lines | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Quartz (Qz) | | Calcite (Cal) | | Dolomite (Dol) | | Plagioclase (Pl) | | K-feldspar | |
| $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ |
| | | | | | | 0,6387 | 10 | | |
| 0,4257 | 20 | | | | | | | 0,4213 | 83 |
| | | | | | | 0,4032 | 92 | | |
| | | 0,3855 | 4 | | | | | | |
| | | | | | | 0,3776 | 25 | 0,3784 | 47 |
| | | | | 0,3693 | 5 | 0,3683 | 22 | 0,3756 | 47 |
| | | | | | | 0,3664 | 41 | | |
| | | | | | | 0,3505 | 10 | | |
| 0,3345 | 100 | | | | | 0,3373 | 10 | 0,3472 | 33 |
| | | | | | | | | 0,3321 | 63 |
| | | | | | | | | 0,3296 | 62 |
| | | | | | | | | 0,3284 | 75 |
| | | | | | | 0,3194 | 100 | 0,3240 | 100 |
| | | | | | | 0,3155 | 30 | | |
| | | 0,3036 | 100 | | | | | 0,3001 | 35 |
| | | | | | | 0,2955 | 16 | 0,2982 | 37 |
| | | | | | | 0,2930 | 15 | | |
| | | | | 0,2885 | 100 | 0,2862 | 12 | | |
| | | | | | | | | 0,2580 | 23 |
| | | | | 0,2537 | 6 | 0,2561 | 18 | 0,2560 | 24 |
| | | 0,2495 | 70 | | | | | | |
| 0,2458 | 7 | | | | | 0,2447 | 12 | | |
| | | | | 0,2404 | 11 | | | | |
| | | | | | | 0,2319 | 9 | | |
| 0,2282 | 7 | 0,2285 | 14 | | | | | | |
| 0,2237 | 3 | | | | | | | | |
| | | | | 0,2191 | 28 | | | 0,2162 | 29 |
| 0,2129 | 5 | | | | | 0,2127 | 9 | | |
| | | 0,2095 | 28 | | | | | | |
| | | | | 0,2014 | 14 | | | | |
| 0,1981 | 3 | | | | | | | | |
| | | 0,1927 | 6 | | | | | | |
| | | 0,1912 | 20 | | | | | | |
| | | 0,1875 | 20 | 0,1846 | 5 | 0,1851 | 10 | | |
| 0,1814 | 13 | | | 0,1803 | 17 | | | 0,1801 | 27 |
| | | | | 0,1785 | 22 | 0,1785 | 9 | | |
| 0,1672 | 3 | | | | | | | | |
| | | 0,1626 | 4 | | | | | | |
| | | 0,1604 | 9 | | | | | | |
| 0,1542 | 9 | | | 0,1544 | 9 | | | | |
| | | 0,1525 | 5 | | | | | | |
| 0,1453 | 2 | 0,1440 | 7 | 0,1464 | 7 | 0,1868 | 36 | | |
| 0,1383 | 5 | | | 0,1388 | 8 | | | | |
| 0,1375 | 7 | | | | | | | | |

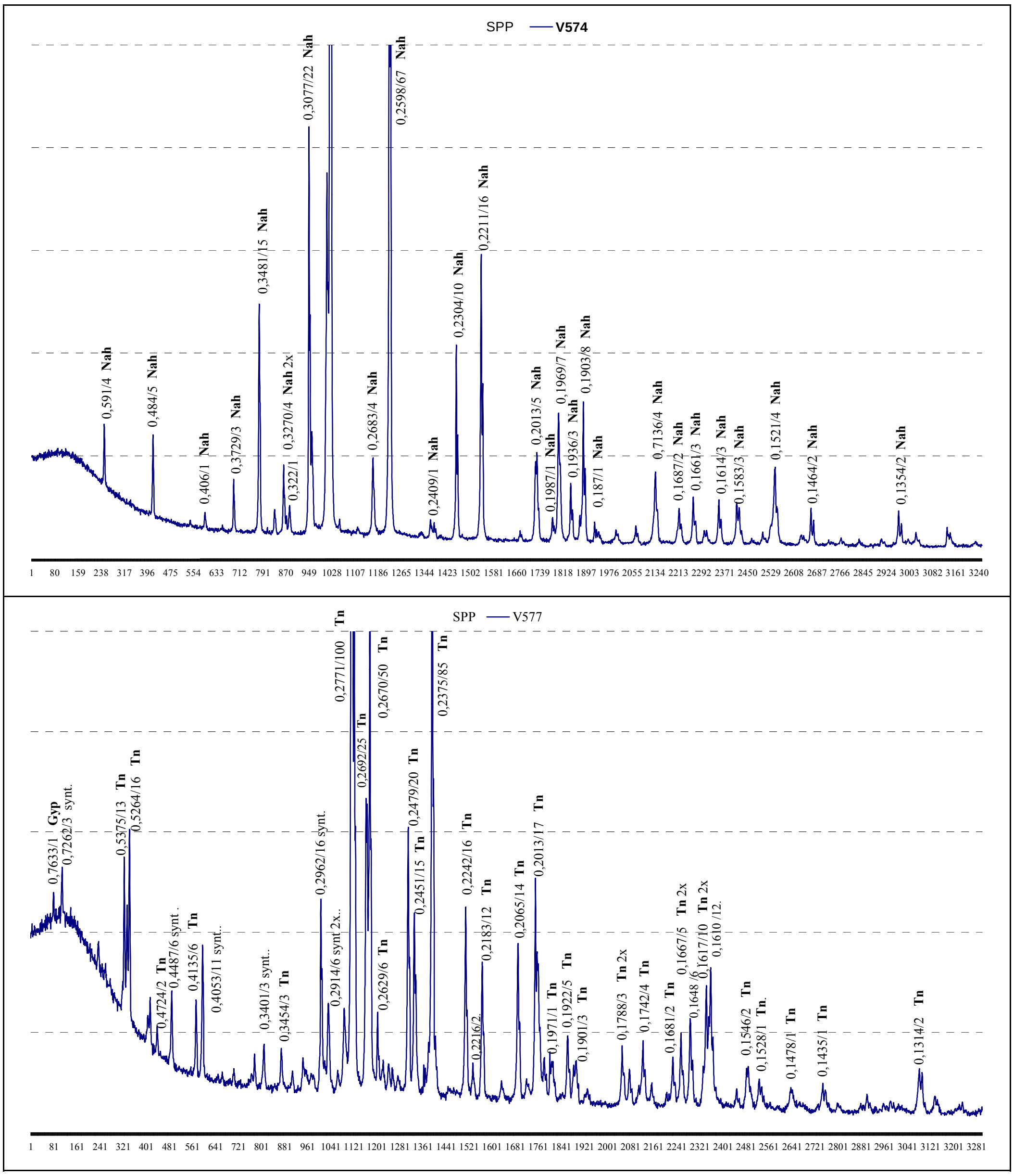


Figure 2. X-ray diffraction patterns of nahcolite (upper) and thermonatrite (below), both carbonates were found as mineral novelties along the pipeline from locality Chynorany.

Both relatively easy soluble hydrated carbonates we regard for secondary mineral phases, originated by precipitation from shallow underground, or rain water. Soils around the gas distribution pipelines represent permeable network for water migration. Sick to defective surfaces here offer the suitable places with changed, reduction environ, supporting precipitation processes.

### 4.2. Composition of soil bed samples from the locality Košice

All this locality samples came from the gas pipeline soil surroundings too. By the results of XRD analyses and consequent revision are formed mostly by dominant quartz and other minor minerals, taken for natural one. Among them usually distinct is plagioclase with less K-feldspar, chlorite, illite and muscovite additives. Diffraction patterns of some samples also comprise obvious "peaks" of calcite. The amorphous phase admixtures – assumed from the background uplift – were commonly registered too. Local mineralogical exception is found admixture of magnesite; questionable is gypsum (slightly above the detection limit).

Among the series of analyzed samples from the Košice, the extraordinary were a few soil samples with identified Ca-nesquehonite $(Ca,Mg)(CO_3)(OH)\cdot 2H_2O$ (Fig. 3, *V667*). We suppose it for synthetic novelty; its different contents in samples are accompanied by previous mentioned minerals of natural origin, mostly quartz. Partly of synthetic origin we assume here also calcite. Relevant Mg-calcite sample diffraction pattern is typical by systematically lower $d_m$ data of all its interplanar distances. Such a structural deformation in carbonates we can explain in accord with higher isomorphic $Mg^{2+}$ substitution instead of $Ca^{2+}$ in its cationic positions (Weiss & Chmielová, 1981). Mg-calcite can be the consequence not only of novelty formation, but also of long-therm metasomatic processes due to highly magnesian water solutions engagement, if permanently migrating in non-compact rocks along the gas pipeline.

The only sample – unusual by amorphous phase absence – contains another synthetic phase of partly ordered structure. Because of missing mineral equivalent it wasn't entirely identified; but span and position of diffraction lines show we could take it for hydrated sulphate close to basaluminite. In analysed sample it is accompanied by higher calcite content; with some plagioclase and illite too.

Formation of Ca-nesquehonite and Mg-calcite we interpret in connection with found magnesite admixture. Twenty to over forty years before, Košice was well-known by former intensive exploitation of the large magnesite deposit at Bankov, and its processing too. This mineral cannot represent the genuine ground component, but along the gas pipeline it is highly probable, its dusty scrap was utilized as technical bed dusting, poured into conduit crop bottom. Such a terrain use of available magnesite waste was regionally widespread and utilized for numerous "building" activities.

### 4.3. Composition of incrustation from the locality Kluknava

Investigated crustal sample came from the gas pipeline branch, close to vilage Kluknava. Rocks around the river valley are mostly schists, permian tuffs and sandstones. Outcrop sample form local, relatively rigid icrustation around the pipeline bottom side. By its brief microscopic study *(Technival, Zeiss Jena)*, we can characterize it as homogeneous, finegrained rock of ochree-grey colour (Fig. 1b). It contain dissaminated sandy detritus – isolated grains of quartz, mostly 0,3 ÷ 0,4 mm in diameter; larger irregular grains (up to 18 x 10 x 8 mm) are rare. Grain distribution is regular in all profile, without any concentration layers. Higher magnification reveal also tiny muskovite scales (below 0,1mm). Whole rock is not entirelly compact, some scarce 1÷2 mm cavities and fissures are filled by white calcite.

Table 2: Interplanar structural distances d and intensities I of diffracted lines of mica-clay minerals and found rare phases standards, (by AMS-DATA).

| main tabular diffraction lines | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Muscovite (Ms) | | Illite (It) | | Nahcolite (Nah) | | Thermonatrite | | Nesquehonite | |
| $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ | $d_t$ | $I_t$ |
| 1,0007 | 100 | 1,0022 | 57 | | | | | | |
| | | | | | | | | 0,6522 | 50 |
| | | | | | | | | 0,6480 | 80 |
| | | | | 0,5921 | 9 | 05362 | 20 | | |
| 0,5004 | 46 | 0,5011 | 35 | 05259 | 24 | 0,4906 | 9 | | |
| 0,4480 | 48 | 0,4476 | 100 | | | | | | |
| 0,4455 | 93 | 0,4399 | 23 | | | | | | |
| 0,3879 | 56 | 0,3891 | 31 | | | | | 0,3850 | 100 |
| 0,3732 | 48 | 0,3738 | 38 | | | | | | |
| 0,3493 | 70 | 0,3501 | 65 | 0,3487 | 31 | | | 0,3589 | 17 |
| 0,3336 | 67 | 0,3352 | 49 | | | | | | |
| 0,3202 | 73 | 0,3215 | 71 | | | | | 0,3228 | 27 |
| | | 0,3130 | 33 | 0,3089 | 38 | | | | |
| 0,2992 | 72 | 0,2996 | 54 | 0,2961 | 58 | | | 0,3031 | 28 |
| | | | | 0,2945 | 100 | | | | |
| 0,2863 | 50 | 0,2875 | 33 | | | | | | |
| 0,2795 | 34 | 0,2800 | 25 | | | 0,2771 | 100 | 0,2787 | 17 |
| | | | | | | 0,2756 | 57 | | |
| | | | | 0,2705 | 29 | 0,2689 | 30 | | |
| | | | | | | 0,2669 | 58 | | |
| 0,2590 | 32 | 0,2597 | 39 | 0,2607 | 92 | 0,2629 | 8 | 0,2612 | 56 |
| | | 0,2578 | 48 | | | | | | |
| 0,2557 | 77 | 0,2564 | 57 | | | | | 0,2508 | 21 |
| | | | | 0,2495 | 70 | 0,2477 | 34 | 0,2492 | 9 |
| 0,2461 | 30 | | | | | 0,2451 | 24 | | |
| | | 0,2406 | 24 | | | | | | |
| 0,2378 | 37 | 0,2385 | 31 | 0,2324 | 24 | 0,2376 | 82 | | |
| | | | | | | 0,2241 | 23 | | |
| 0,2129 | 38 | 0,2135 | 24 | 0,2160 | 14 | 0,2183 | 20 | | |
| | | | | 0,2049 | 25 | 0,2064 | 21 | | |
| 0,2001 | 28 | 0,2004 | 26 | 0,2035 | 11 | 0,2013 | 30 | 0,2021 | 9 |
| 0,1972 | 21 | | | 0,1967 | 16 | 0,2005 | 16 | | |
| | | | | 0,1946 | 13 | | | | |
| | | | | 0,1909 | 23 | 0,1912 | 10 | 0,1925 | 29 |
| | | | | 0,1751 | 12 | 0,1787 | 9 | 0,1799 | 21 |
| | | | | 0,1704 | 12 | | | 0,1715 | 10 |
| | | | | 0,1672 | 15 | 0,1666 | 11 | | |
| 0,1648 | 28 | 0,1652 | 23 | | | 0,1648 | 15 | 0,1648 | 8 |
| | | | | 0,1618 | 10 | 0,1610 | 23 | | |
| 0,1499 | 37 | 0,1506 | 25 | 0,1526 | 12 | | | | |

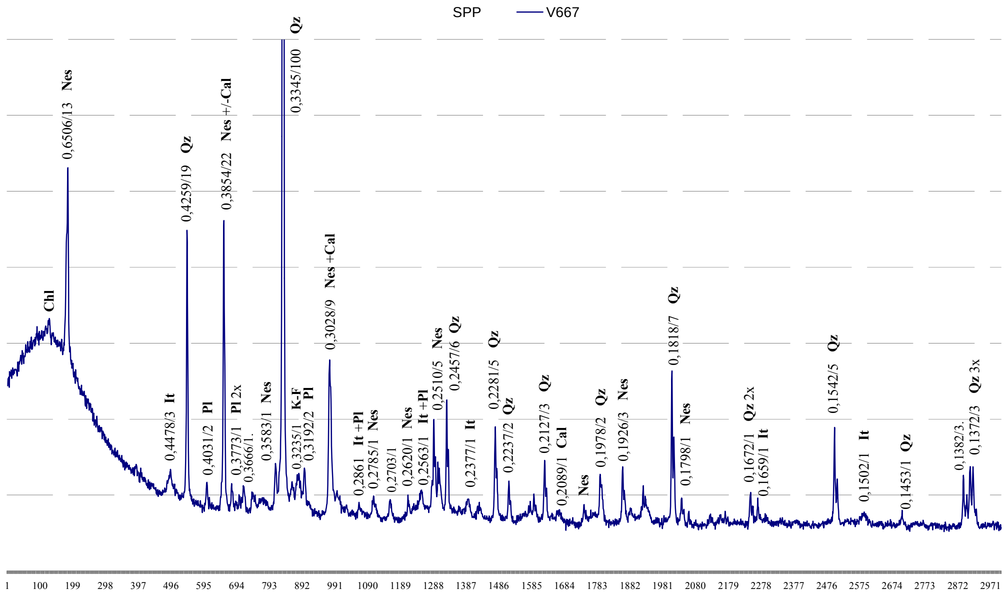


Figure 3. X-ray diffraction pattern of Ca-nesquehonite from the pipeline locality Košice

Edge parts of this indurated mudstone were polluted by asfalt particles from the pipeline isolation cover. Based on rock positive HCl reaction tests, studied claye incrustation is calcified accross all its profile.

More detail information, related to incrustation mineral composition was revealed using the XRD analyses. All sample´s diffraction pattern contain reflexes of several minerals. Four made X-ray analyses are typical by excessive quartz (75 - 90% Qz). Other minor to accessory ground minerals are plagioclase, K-feldspar, chlorite and also relatively resistant soil phases: muscovite, illite and kaolinite. All these minerals could be also the components of sandy bed dusting, that is at present day impossible to determine. One samples contain more (~9,5 %) calcite, obviously the possible cohesive ingredient. In the bottom, where the incrustation was found we can take it for real product, precipitating from calcareous undergroud water. Recorded RTG-diffraction patterns point out to possible gypsum? ev. siderite? additions too.

In all revised X-ray diffraction patterns from Kluknava, the presence of amorphous phase was confirmed too. Without its closer determination, in powder incrustation samples we can match it to asphalt admixture, seen by microscopic study; eventualy by the bacground uplift extent – there could be also nearly amorphous „disordered“ claye phase, close to kaolinite. All here found crystalline mineral species (calcite included) we can qualify both as natural components of local silty soils, same as could be constituents of imported bed dusting. Despite of some limstone rocks, situated SSW of Kluknava, in case of calcite we consider it for the secondary phase, precipitated step by step along the gas pipeline bottom from soaking water solutions. Time horizon about 40 years seem to be real for the formation of such indurated incrustation, over 10 cm thick.

## 5. CONCLUSIONS

This article refer about some posibilities of used X-ray diffraction method for the soil composition study. Treatment or processing of soil material needs not only chemical composition, but more detail knowledges about its phase / mineral composition. Revision of investigated samples X-ray records, made by use Seifert XRD 3003 PTS diffractometer, had required amplifying of minor diffraction lines against main quartz reflexes to determine maximum of present crystaline constituents. Analyzed soil samples were all related to relative shallow gas pipeline deposition from three different localities in Slovakia.

Series of X-ray diffraction patterns got evident the polymineral character of samples, all with quartz highly prevailed over other minerals. The suite of minor soil minerals, found in all three localities included plagioclase, K-feldspar (microcline), muscovite, illite and chlorite. Rarely dolomite and kaolinite were found, the second one not sure because of its pattern similar to chlorite; questionable was gypsum or siderite presence too. Many samples had contained calcite, from low admixture to quite common. This carbonate we consider here of various origin. Beside calcite grains as detritic component in natural soil profile, f.e. limestone particles, and common is its presence as fine sediment binder. In permeable rock medium along the pipelines we take it preferentially for the secondary calcareous adhesive agent. We suppose it is the principal constituent for such indurated incrustation formation, as the sample from the pipeline bottom from the third locality Kluknava.

XRD analyses were useful for semi-quantitative estimation of crystalline mineral contents, evidence of amorphous phases admixture and exact identification of found rare mineral novelties. In selected samples from the direct pipeline surroundings by the Chynorany and Košice the crystallization of different hydrated carbonates were documented. Along the pipeline bed we assume wide migration of shallow water. Precipitation and crystallization of novelties were positively influenced by deffective isolation / exposed steel surface, which offer changed pH and Eh conditions. In the locality Chynorany nahcolite and thermonatrite were determined, acompanied by phase $Na_2(CO_3){\cdot}7H_2O$. Local formation of Na-carbonates we assume as consequence of intensive agricultural production, related to fertilizing and irrigation. In Danube lowland with warm climate, the salty soils are quite frequent phenomena. Connected to climate changes, even earlier occurrences of the same mineral novelties were described here as risky corrosive or even environmental factor. In Košice locality the other hydrated carbonate – Ca-nesquehonite was found, and also sX sulphate? – without closer identification. According to same principles of its precipitation and crystalization as in Chynorany, we assume also influence of additives (magnesite bed dusting). This could serve as heritage example after former magnesite industrial boom in Košice region, with wide utilizing of cheap and available technical wastes.

Related to local soil vs. imported bed dusting material, there is no registration about its application around older pipeline branches. In case of massive bed application of imported material, sandy or loamy dusting use to be homogeneous. Found in more samples from the same locality, the variable ratio of individual minerals show onto utilizing of local soil material.

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