



Geochemical monitoring at Soultz-sous-Forêts (France) between October 2006 and March 2007, after the chemical stimulations (RMA, NTA and OCA) carried out in the wells GPK-4 and GPK-3.

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GEOCHEMICAL MONITORING AT SOULTZ-SOUS-FORÊTS (FRANCE) BETWEEN OCTOBER 2006 AND MARCH 2007, AFTER THE CHEMICAL STIMULATIONS (RMA, NTA AND OCA) CARRIED OUT IN THE WELLS GPK-4 AND GPK-3

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ABSTRACT

This paper presents the main results obtained during the geochemical monitoring of the fluids and deposits collected from the wells GPK-4 and GPK-3 after the four operations of chemical stimulation carried out in May and October 2006, and February 2007, at Soultz-sous-Forêts. During these operations, solutions of Regular Mud Acid (RMA), Nitrilo-Triacetic Acid (NTA) and Organic Clay Acid HT (OCA) were successively injected with fresh water into GPK-4 whereas only the OCA treatment was used for GPK-3. This paper shows that these acids have actively reacted with minerals present in the fractured and porous areas of the granite. The injection of the NTA basic solution (pH 12) into GPK-4 has allowed cleaning this well and a part of the fractured areas, and recovering significant amounts of drilling wastes (grease, rests of cuttings), rock fragments and hydrothermal deposits. Dissolved and solid Ca-Fe-NTA compounds were also formed. The combination of the RMA, NTA and OCA treatments is possibly at the origin of the significant increase of GPK-4 productivity. No major improvement in injectivity was observed for the well GPK-3 for which only the OCA treatment was used.

INTRODUCTION

The main objective of the original European Hot Dry Rock (HDR) Project, renamed as an Enhanced Geothermal System (EGS) Project in 2001, is to develop a deep heat exchanger for electricity production at Soultz-sous-Forêts, France.

The construction of a scientific pilot plant was initiated in 2001. Three wells, drilled at a depth of about 5000 m, must make up the heat exchanger. GPK-3 will be the injection well and GPK-2 and GPK-4 will be the production wells. In order to enhance the GPK-3 injectivity and GPK-4 productivity, and after several tests of hydraulic stimulation which resulted in a moderate improvement of these parameters, three operations of chemical stimulation were selected and carried out in GPK-4 (against one in GPK-3) between May 2006 and February 2007. One of the positive points of these operations is their low induced microseismicity.

In this study, we report and comment the results obtained on the fluids and deposits collected from the wells GPK-4 and GPK-3 during the production tests carried out in 2006 and 2007 after the chemical stimulation operations. These works were carried out in the framework of the research activities accompanying the construction of the scientific pilot plant (European EGS Pilot Plant Project, 2004-2008).

GEOCHEMICAL MONITORING CARRIED OUT AFTER THE CHEMICAL NTA STIMULATION IN THE WELL GPK-4

Operation of chemical NTA stimulation conducted in the well GPK-4 (October 2006)

Previously to this operation, 200 m³ of RMA (12% HCl and 3% HF) had been injected into GPK-4 on the 17th May 2006. The objective of the RMA treatment was to attack the hydrothermal deposits (carbonates, clays, etc.) present in the main fracture and porosity zones of the granite to optimize their permeability. Before the injection of RMA, 2000 m³ of fresh water and 25 m³ of 12% diluted HCl had been introduced in this well. 2000 m³ of fresh water were still injected after the RMA injection. However, no production test was performed after these operations. If we assume that the injected acids only attacked calcite, the maximum volume of dissolved calcite can be estimated at about 19.2 m³.

If the RMA treatment was used to clean up the zones of hydrothermalized fractures and the rock porosity at the vicinity of GPK-4, the objective of the NTA stimulation was to forward the basic solution of NTA as far as possible in these zones to reach the hydraulic connections in the reservoir. NTA (C₆H₉NO₆) is a weak tri-acid with a strong chelating capacity with respect elements such Ca, Mg, Fe and other metals. Injection of NTA into GPK-4 was carried out by SAPS between October 16 and 21, 2006. In order to cool the well GPK-4, 4150 m³ of fresh water were injected at 20 to 30 l/s before the injection of 200 m³ of a basic solution (pH 12), constituted of caustic soda (NaOH) and 19% diluted Na₃NTA, at a flow rate of 35 l/s. The corresponding pressure curve showed no more straight increases indicating that the near-well fracture zones were become more permeable. After the NTA injection, 850 m³ of fresh water were injected at 20 to 40 l/s. As a radical increase of pressure was surprisingly observed at the well head after these injections, a production test was immediately carried out in GPK-4 between October 23 and 27.

During this test, a volume of about 2640 m³ of fluid was discharged from GPK-4. Mainly at the beginning of this test (the first 450 cubic meters), the discharged fluid was accompanied by numerous black solid and grease deposits which were collected by GEIE and analyzed by BRGM. These deposits will be later described. The mobilization and accumulation of these deposits in the fractured zones and rock porosity were due to the important cleaning of the well and a part of these zones by the basic NTA solution (especially the use of NaOH).

These factors were probably responsible of the plug which caused the total blocking of the thinnest hydraulic circuits in the vicinity of the well and the pressure increase at the well head. The effect of this plug was practically inexistent during the production test.

After and up to the end of the production test, the discharged fluid became clearer but significant amounts of very fine brown-yellowish particles in suspension were observed. These particles, which form flocculates at the bottom of the sampling flasks and are dissolved by a solution of concentrated HCl, were collected and analyzed by BRGM. Results will be later described and commented.

Another production test was carried out in GPK-4 between January 22 and 25, 2007. A volume of about 2400 m³ of fluid was discharged from this well. Brown-yellowish particles in suspension were observed at the beginning of this test (the first 500 cubic meters) but after disappeared. Some black solid deposits were still collected and analyzed by BRGM.

The computations performed by GEOWATT using the results obtained during these production tests showed that the GPK-4 productivity was clearly improved after the RMA and NTA treatments from about 3 l/s/MPa in July 2005 to a value close to 4 l/s/MPa (GEOWATT, 2007). The real effect of each chemical treatment could not be differentiated because no production test was carried out after the RMA stimulation. If the RMA treatment allowed to improve the injectivity of the well GPK-4 by about 30% (from 2.5 to 3.5 l/s/MPa; GEOWATT, 2006), an injectivity test (Step rate test) carried out in December 2006 suggested that the NTA stimulation had little positive effect on the GPK-4 injectivity. Only the connection between the well GPK-4 and the far field seemed to be slightly improved.

Two production tests were also carried out in GPK-3 between October 10 and 11, 2006, and between January 25 and 26, 2007. The volumes of discharged fluid were close to 1750 and 1300 m³, respectively. The computations done by GEOWATT indicated a value of productivity slightly higher than 4 l/s/MPa for GPK-3 in the last test (GEOWATT, 2007).

Geochemical monitoring of the discharged fluids from GPK-3 and GPK-4 in October 2006 and January 2007

The chemistry of the fluids discharged from GPK-3 and GPK-4 was monitored by BRGM during these production tests. Fluid sampling, on site measurements (temperature, conductivity, specific gravity, Redox potential, pH, dissolved oxygen and alkalinity) and some chemical analyses in laboratory (dissolved Cl, Ca, organic and inorganic carbon) were performed by BRGM for all the production tests. Classical techniques such as specific electrodes, titration, ICP-AES and carbon Infra Red detection after combustion were used. The relative uncertainties for these analyses ranged from 0.5 to 5%.

Free NTA was analyzed by EGI laboratory (P. Rose) using gas chromatography only for the GPK-4 production tests. The dissolved K, Li, Fe, Mn, Pb, Cu, Ni, Zn and Cr as well as the organic tracers previously injected such as fluorescein, 1,5-, 1,6-, 2,6- and 2,7-nds (naphthalene disulfonate) were only analyzed for the productions tests carried out in GPK-3 and GPK-4 in January 2007. The analyses of the inorganic elements were performed using inductively coupled plasma emission and mass spectroscopy techniques (ICP-AES and ICP-MS). Relative analytical uncertainty was 5% for potassium and 10-15% for the other inorganic elements. The organic tracers were analyzed using HPLC and fluorescence detection (Sanjuan *et al.*, 2006a). Relative analytical uncertainty was 10-15%.

Results obtained during the production tests carried in October 2006

All these results are reported in Table 1.

Well GPK-3

The conductivity and pH values measured in the fluid discharged from GPK-3 vary from 61 to 69 mS/cm and from 7.16 to 7.44, respectively. The conductivity and pH values are 66 mS/cm and 7.16 at the end of the test (October 11), after a fluid production of about 1750 m³. The concentrations of dissolved chloride and calcium are about 28 and 3.1 g/l (Table 1), which suggests this fluid is constituted of about 47% of native geothermal brine and 53% of fresh water (chemical compositions of the native geothermal brine are reported in Sanjuan *et al.*, 2006b). The last fluid injected into the well GPK-3 (December 19, 2005), at the end of the fluid circulation test between GPK-3 and GPK-2/GPK-4, had Cl and Ca concentrations of 42 and 5.2 g/l (Sanjuan *et al.*, 2006b). Consequently, a fluid dilution process has occurred in the meantime.

The concentrations of dissolved Total Organic Carbon (TOC) ranging from 32 to 76 mg/l (Table 1) are relatively high. A large amount of TOC probably comes from the dissolution of grease used for the drilling operations. Another part could have a natural origin (Ledésert *et al.*, 1996).

Well GPK-4

Except for the first samples, figures 1 and 2 show that the conductivity and pH values measured in the fluid discharged from GPK-4 are relatively stable around 55±2 mS/cm and 7.3±0.2, respectively. The Cl and Ca concentrations also indicate stable values around 20-21 and 1.8-1.9 g/l (Table 1, Fig. 1) after a fluid production of 2640 m³.

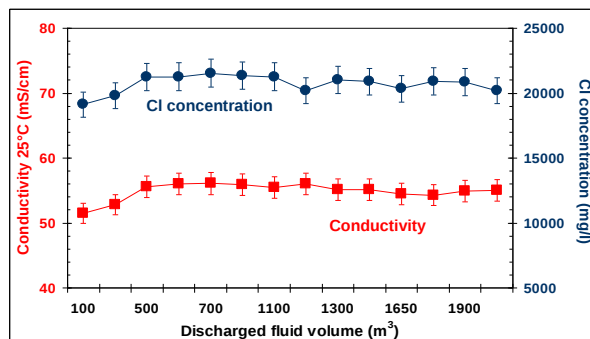


Figure 1 - Conductivity and concentrations of dissolved Cl measured in the fluid discharged from the well GPK-4 between October 23 and 27, 2006.

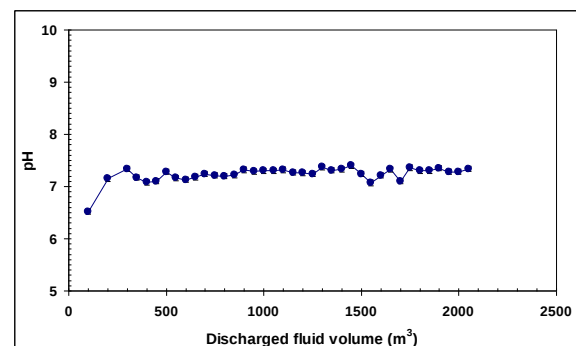
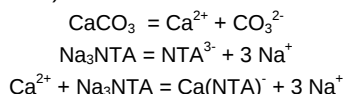


Figure 2 - pH values measured in the fluid discharged from GPK-4 between October 23 and 27, 2006.

At the end of the fluid circulation test carried out between GPK-3 and GPK-2/GPK-4, the last fluid samples collected from GPK-4 (November and December 2005) had Cl and Ca concentrations of 46 and 6.0 g/l, respectively (Sanjuan *et al.*, 2006b). The measured conductivity and pH values were close to 111 mS/cm and 5.7-5.8, respectively. The large volumes of fresh water injected into GPK-4 for the RMA and NTA treatments have diluted this fluid.

The Cl concentrations suggest that the fluid discharged from GPK-4 is constituted of about 36% of native geothermal brine whereas the Ca concentrations rather indicate a proportion of 28%. As chloride ions were introduced during the RMA treatment and calcium ions can have reacted, we will consider that this proportion is close to 32%.

The chemical attack of calcite (CaCO_3) by Na_3NTA in basic medium ($\text{pH} > 11$) can be schematized as follows:



38 tons of Na_3NTA correspond to 28.2 tons of NTA and can dissolve 14.8 tons (or 5.5 m^3) of calcite if this reaction is total. These data give an idea of the maximum amount (or volume) of calcite which can be attacked by this treatment. NTA can also attack other carbonate minerals such as dolomite ($\text{CaMg}(\text{CO}_3)_2$), ankerite ($\text{CaFe}(\text{CO}_3)_2$) or siderite (FeCO_3) by complexing cations such as Mg or Fe.

The concentration of NTA injected into GPK-4 was estimated at 3.5 to 5.4 g/l in the deep fluid before any chemical reaction and taking into account the fluid mixing processes. Most of the free NTA concentrations analyzed in the EGI laboratory ranging from 200 to 300 mg/l (Table 1 and Fig. 3) suggest that only 4 to 9% of the NTA injected into GPK-4 has not reacted. At the measured pH (7.1-7.4), free NTA is essentially in form of $\text{H}(\text{NTA})^{2-}$. The alkalinity values are higher than the concentrations of Total Inorganic Carbon (TIC) and suggest the presence of 1 to 2 meq/l of dissolved organic anion (Table 1). If we assume that this organic anion is mainly $\text{H}(\text{NTA})^{2-}$, the concentrations of free NTA are in relative good agreement with these alkalinity values.

The TOC values are much higher than those analyzed in the fluid discharged from GPK-3 (Table 1). Most of them vary from 283 to 333 mg/l (or from 23.6 to 27.7 mmol/l; Fig. 3). They probably traduce the presence of dissolved NTA chelates such as $\text{Ca}(\text{NTA})^-$, $\text{Fe}(\text{NTA})$ or other metal-NTA.

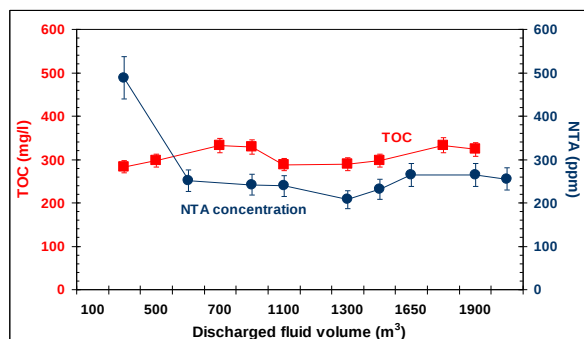


Figure 3 - Concentrations of Total Organic Carbon (TOC) and free NTA analyzed in the fluid discharged from GPK-4 between October 23 and 27, 2006.

A maximum of 0.38 g/l of dissolved $\text{Ca}(\text{NTA})^-$ is estimated if about 10 mmol/l of TOC are assumed to be associated to this compound. This implies a maximum concentration of chelated calcium of about 67 mg/l. 8 to 12 mmol/l of TOC are considered to correspond to free NTA.

As for GPK-3, the remaining TOC (estimated at about 6 mmol/l) could also come from the dissolution of grease used for the drilling operations and from a natural origin.

The Total Suspended Solids (TSS) were weighed on two fluid samples and gave the values of 229 and 314 mg/l (Table 1). These TSS, essentially constituted of calcite and flocculates of organic carbon and calcium (see next chapters), suggest the presence of Ca-NTA compounds.

After a production of 2640 m^3 of fluid and using the previous assumptions, it can be roughly estimated that about 2.2 tons of NTA have been recovered, which represent about 8% of the NTA injected into GPK-4.

Results obtained during the production tests carried in January 2007

All these results are reported in Table 2.

Well GPK-3

After a production of 253 m^3 of fluid, which essentially corresponds to the volume of the well (close to 200 m^3), fresh water is replaced by a mixing of native geothermal brine and fresh water. After, the conductivity and pH values tend to slightly increase from 76 to 79 mS/cm and 6.51 to 6.71, respectively (Table 2). The conductivity values are much higher than those determined during the October 2006 test whereas the pH values are lower. This suggests proportions of native geothermal brine much more large than in the previous test. Using the concentrations of dissolved Cl, Ca, K and Li, it can be estimated that the GPK-3 fluid is constituted of 65% of native geothermal brine and 35% of fresh water at the beginning of this production test. These proportions become 68 and 32% at the end of this test, after a production of 1263 m^3 . They were of 47 and 53% at the end of the October 2006 production test and 37% and 63% in the fluid injected into GPK-3 after this test (Table 1). This fast increase of proportion of native geothermal brine without any fluid production between the two production tests confirms the previous studies, which indicated a relatively high flux (1-1.2 m^3/h) for the native geothermal brine.

The analyzed concentrations of 1,5-, 2,7-, 1,6- and 2,6-nds (Table 2) allow to estimate that the fluid discharged from GPK-3, at the end of the production test, is constituted of about 1.5% of fresh water injected into GPK-2 in 2000, 2% of fresh water injected into GPK-2 in 2003, 9% of fresh water injected into GPK-3 in 2003 and 3% of fluid injected into GPK-4 in 2005. The initial concentrations of these tracers are reported in Sanjuan *et al.* (2006a). The analyzed concentrations of fluorescein tend to decrease (Table 2) confirming the increase of the proportion of native geothermal brine during the production test. These concentrations are higher than those analyzed in the fluids discharged from GPK-2 and GPK-4 and in the fluid injected into GPK-3 at the end of the 2005 circulation test (Sanjuan *et al.*, 2006b; Table 3). This result can be probably explained by the fact that fluorescein was injected into GPK-3 and residual fluorescein could have been recovered.

The concentrations of dissolved oxygen measured in the fluid discharged from GPK-3 are relatively high (Table 2) and consequently, the Eh values are rather positive. These values are probably due to the injection of an oxygenated fluid into this well in October 2006, after the production test (Table 1). Even if the Fe concentration is badly known for the native geothermal brine (50 to 100 mg/l; Sanjuan *et al.*, 2006b), the concentrations of dissolved Fe are excessively low if we consider that the discharged fluid is constituted of 65 to 68% of native geothermal brine. They are lower than the concentrations analyzed in 2005 in the fluids of all the wells (Table 3).

These results and the rust-colored deposits collected from this well suggest the precipitation of hematite (see later) probably due to the oxygenation of the deep fluid, its cooling and degassing. The concentrations of Zn and Pb are also rather low in comparison with the concentrations analyzed in 2005 (Table 3) and the concentrations of the native geothermal brine (Sanjuan *et al.*, 2006b), suggesting the precipitation of sulfide minerals (sphalerite and galena), which were also observed in small amounts in the collected deposits (see next chapters).

The values of TOC (Table 2) are much lower than those analyzed in the October 2006 production test (Table 1). This can be explained by the decrease of drilling grease in contact with the fluid as confirmed in the collected deposits (see next chapters). Alkalinity is constituted of both inorganic and organic anions but inorganic anions are predominant (Table 2). The TIC values are similar to those measured in the previous production test.

Well GPK-4

Fresh water has been replaced by a mixing of brine and fresh water, after a production of 460 m³ of fluid which exceeds the volume of the well (close to 200 m³). After, the conductivity values tend to slightly decrease from 75 to 67 mS/cm (Table 2; Fig. 4). pH values vary from 6.23 to 6.84. As observed for the well GPK-3, the conductivity values are much higher than those determined during the October 2006 test. This suggests proportions of native geothermal brine much more significant than in the previous test. Again, this fast increase of the proportion of native geothermal brine without any fluid production between the two production tests confirms the existence of a relatively high natural flux of the native geothermal brine.

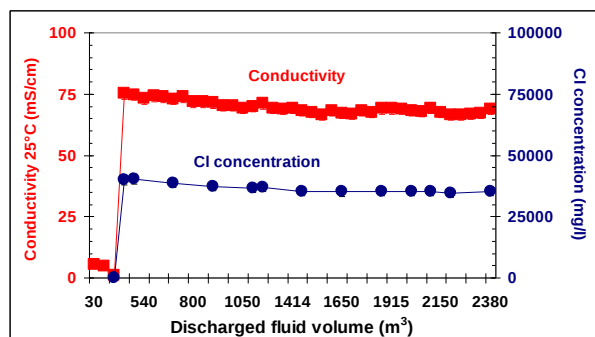


Figure 4 - Conductivity and concentrations of dissolved Cl measured in the fluid discharged from the well GPK-4 between January 22 and 25, 2007.

However, contrary to the behavior observed in GPK-3, the conductivity values mainly depending on the concentrations of dissolved Cl slightly decrease during this test (Fig. 4) whereas the concentrations of dissolved K and Ca increase (Fig. 5). These results suggest that the Cl ions don't only depend on the native geothermal brine but have also another origin (RMA treatment). Consequently, this element cannot be used to estimate the proportions of native geothermal brine in the fluid discharged from GPK-4 during this production test.

Contrary to the decreasing trend shown by the Cl concentrations, the contribution of native geothermal brine in the fluid discharged from GPK-4 slightly increase during this production test, as shown by the increasing concentrations of dissolved Ca and K. Using these concentrations, it can be estimated that this fluid is constituted of about 50% of native geothermal brine and 50% of fresh water at the beginning of the production test.

At the end of this test, the proportion of native geothermal brine is estimated to be close to 56%. The Cl concentrations give an estimation of 60% suggesting that the other source of chloride has a contribution much less significant than at the beginning of this test. The proportion of native geothermal brine was of about 32% at the end of the October 2006 production test.

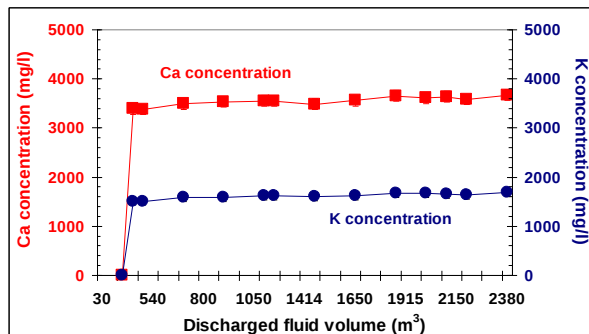


Figure 5 - Concentrations of dissolved K and Ca analyzed in the fluid discharged from the well GPK-4 between January 22 and 25, 2007.

The analyzed concentrations of 1,5-, 2,7-, 1,6- and 2,6-nds allow to estimate that the fluid discharged from GPK-4, at the end of the production test, is constituted of about 0.2% of fresh water injected into GPK-2 in 2000, 0.3% of fresh water injected into GPK-2 in 2003, 1.4% of fresh water injected into GPK-3 in 2003 and 9.4% of fluid injected into GPK-4 in 2005. Note that these proportions are slightly lower at the beginning of the production test (Table 2). The analyzed concentrations of fluorescein, lower than those determined in the fluid discharged from GPK-4 at the end of the 2005 fluid circulation test (Sanjuan *et al.*, 2006b; Table 3), also tend to increase during this production test, traducing a decreasing contribution of the fluids injected during the RMA and NTA treatments in the fluid discharged from GPK-4.

The concentrations of dissolved oxygen measured in the fluid discharged from GPK-4 are close to zero (Table 2). The concentrations of dissolved Fe are similar to those analyzed in the fluid discharged from GPK-4 during the 2005 fluid circulation test (Table 3) if we take into account the dilution factor. They are higher than those analyzed in the fluid discharged from GPK-3. The concentrations of Zn and Pb are much lower than those analyzed in 2005. As for GPK-3, low amounts of sulfides were observed in the deposits collected from this well (see next chapters).

The values of TOC are much lower than those analyzed in the October 2006 production test (Table 1) but remain much higher than those analyzed in the GPK-3 fluid (Fig. 6).

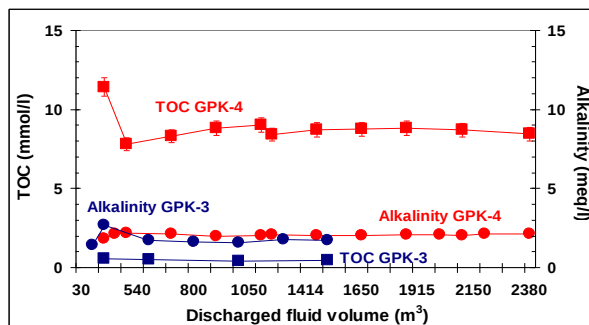


Figure 6 - Concentrations of TOC and alkalinity values analyzed in the fluids discharged from the wells GPK-3 and GPK-4 in January 2007.

As for GPK-3, the fall in TOC is mainly due to a large decrease of drilling grease in contact with the fluid as observed in the collected deposits, but it must also be caused by a decrease of the formation of dissolved NTA chelates. However, these latter most probably constitute the major part of remaining TOC (that we estimate at about 7.5 mmol/l). No free NTA was detected by the EGI laboratory (Table 2). After a production of 2380 m³ of fluid and using the concentration of NTA deduced from the value of TOC, it can be roughly evaluated that about 0.6 tons of NTA have been recovered. The sum of the two estimations of NTA recovery after each production test carried out in GPK-4 (2.8 tons) represents about 10% of the NTA injected into this well.

As shown in figure 6, the alkalinity values are similar for the fluids discharged from GPK-4 and GPK-3 in January 2007, confirming the absence of free NTA observed by EGI in the GPK-4 fluid. The values of TIC are also close (Table 2). The part of alkalinity which corresponds to the presence of organic anions is probably associated to the dissolution of drilling grease and the existence of natural organic acids.

According to the literature, the toxicity of NTA is rather low and its degradation is relatively fast. In this study, the fluid samples collected during the October 2006 production test which indicated the presence of free NTA (Table 1) were re-analyzed by EGI after four months and showed that free NTA had totally disappeared.

Chemical and mineralogical characterization of the solid deposits collected from GPK-3 and GPK-4

Chemical and X-ray diffraction analyses were done on samples of solid deposits collected from these wells by GEIE or BRGM during and after the October 2006 production test and after the January 2007 production test. Some samples were also observed using optical microscopes and a Scanning Electron Microscope (SEM). All these analyses were performed in the BRGM laboratories.

Most of the chemical analyses were performed on solid samples dried at 40 or 105°C and after chemical attack or combustion in a furnace. Classical methods such as inductively coupled plasma technique (ICP) for multi-elementary inorganic analyses (most of the major and trace elements), infra-red detection for carbon and sulfide detection, chemical technique for sulfate determination were used. Analytical precision is rather low for the multi-elementary ICP technique. For the other techniques, relative uncertainty is always lower than 5%. The X-ray diffraction analyses were performed using an automated SIEMENS D5000 diffractometer. The detection limit of a phase is generally considered to be 5%.

Solid samples collected in October and November 2006

All the results are reported in Tables 4, 5, 6 and 7.

Well GPK-3

Four solid samples were collected from this well by GEIE. Numerous mineralogical phases observed on a solid deposit collected on October 2, 2005, during the fluid circulation loop between GPK-3 and GPK-2/GPK-4, were detected on these samples but at different proportions (Tables 4 and 5).

All the samples are constituted of abundant grease and magnetite. Calcite, quartz and amorphous silica are also present. Barite and celestine are always observed but at proportions lower than 5%. Sulfide minerals such as sphalerite, galena and pyrite seem to be also present but at low amounts (< 1%). The excess of sulfur could not be associated to other elements (presence of more abundant pyrite, native sulfur ?).

These samples are relatively rich in arsenic and copper. The presence of manganese oxides is always observed. The detected halite and sylvite are formed during the sample drying, from the brine associated to these samples.

The most representative deposit sample of the fluid production, collected on October 11, 2006, is also constituted of alumino-silicate minerals such as feldspars, plagioclases, micas/illite and chlorite. Low amounts of auto-morph apatite are also identified. Some limpid rod-shaped hexagonal crystals of quartz seem to be neo-formed on a siliceous substratum.

Most of the detected phases suggest that all the deposit samples are mainly constituted of drilling wastes, remaining cuttings, natural rock fragments and hydrothermal deposits observed in the Soultz granite (Stussi *et al.*, 2002). Given the low concentrations of Zn and Pb observed in the discharged fluid (Table 2), it is probable that a part of the sulfide minerals is neo-formed (see previous chapters).

The rust color observed in the deposit samples collected on October 11 and November 13, 2006 (Tables 4 and 5), is due to the presence of hematite which has been probably formed after the injection of oxygenated fluid into GPK-3. The mixing between this fluid and the geothermal brine which is cooled and degassed represents a favorable factor to precipitate this type of mineral. By the way, the contents of dissolved oxygen are relatively high in the fluid discharged from GPK3 and the Fe concentrations are particularly low (Table 2). Apparently, even if chemical treatments were used to eliminate the dissolved oxygen in the fluids injected into GPK-3, these latter don't seem to have been sufficient faced with the volumes of surface fluids which were injected into this well. More cautionary measures during the fluid injection will have to be taken in the future.

Well GPK-4

Three types of solid deposits were collected from this well by GEIE and BRGM. Most of these deposits are probably at the origin of the plug and the pressure increase at the well head observed during the NTA injection.

The first type of solids, two samples of black deposits, are approximately constituted of the same phases as those observed for the more representative samples of the GPK-3 fluid production (Oct. 11 and Nov. 13, 2006), except the absence of hematite (Tables 4 and 5). The higher concentrations of organic carbon probably traduce the presence of additional NTA solid compounds and more significant amounts of grease recovered by means of the caustic soda injected during the NTA treatment. The proportions of magnetite are also relatively high. The rod-shaped crystals of quartz neo-formed in the siliceous substratum are again found. The origin of all these deposits seem to be similar to that of the deposits collected from GPK-3. However, the arsenic contents are much higher reaching up to 11.5% (Tables 4 and 5). Arsenic is omnipresent in these samples. The optical observations indicate that this element is not associated to a crystallized structure but rather to an abundant organic phase. This phase could be constituted of NTA molecules which have trapped this element. Identification of these compounds using FTIR-ATR (Fournier Transform Infrared - Attenuated Total Reflectance) has been attempted but for the moment, has not given conclusive results.

One of the other types of solids, three samples of brown-yellowish deposits, are mainly constituted of organic carbon, calcium and iron (Tables 6 and 7), which suggest the formation of solid Ca-Fe-NTA compounds after the injection of NTA into GPK-4. Calcite, barite and celestine are also observed.

The chemical analyses indicate the presence of silica (probably amorphous silica) and manganese oxides, the absence of aluminosilicate minerals and the probable presence of low amounts of sulfide minerals (sphalerite and galena). The detected halite, sylvite and gypsum are formed during the sample drying, from the brine associated to these samples. The presence of these brown-yellowish solids in GPK-4 occurred after the apparition of the black and grease deposits. Similar observations were done after the injection of a basic solution of NTA into a well in the framework of the Coso project (P. Rose, pers. comm.) but the NTA compounds were rather white-colored. This discrepancy of color can be explained by the significant difference of fluid chemical composition between the two sites.

The last type of solids, two samples of yellowish Total Suspended Solids (TSS) collected from the fluid samples, are mainly constituted of organic carbon and calcium (Tables 6 and 7), which suggest the formation of solid Ca-NTA compounds after the injection of NTA into GPK-4. Calcite, barite and celestine are also detected. As for the previous samples, the presence of halite and sylvite is due to the sample preparation.

Solid samples collected in January 2007

All the results are reported in Tables 4 and 5.

Two deposit samples were collected by GEIE after the fluid production tests carried out in January 2007. One of these samples (rust-colored deposit), collected after separator, is constituted of a mixing of deposits coming from GPK-3 and GPK-4. The other sample only concerns GPK-4 deposits (black deposits).

In comparison to the previously collected deposits, these two samples have much lower amounts of drilling grease. Consequently, the concentrations of organic carbon analyzed in these samples are lower than those previously determined in the GPK-4 deposits (Tables 4 and 5). However, the presence of NTA solid compounds is suspected in both samples because the concentrations of organic carbon remains similar to those previously analyzed in the GPK-3 deposits in spite of the significant decrease of grease. The chemical analyses of these two samples indicating high concentrations of barium and sulfur suggest the presence of abundant barite.

The other detected mineral phases are similar to those previously observed (Tables 4 and 5). Magnetite remains abundant. The amounts of calcite seem to be more significant in the GPK-3 deposits. Celestine is present. An excess of sulfur is also observed. The presence of apatite is suspected. Manganese oxides and high contents of arsenic are also analyzed. The presence of hematite is only detected in the rust-colored deposit (coming from GPK-3). Most of the analytical results suggest that these two deposit samples are mainly constituted of remaining cuttings, natural rock fragments and hydrothermal deposits observed in the Soultz fractured granite. Some minerals such as observed quartz and suspected poly-metallic sulfides (in low amounts) and hematite are most probably neo-formed phases.

GEOCHEMICAL MONITORING CARRIED OUT AFTER THE CHEMICAL OCA STIMULATION IN THE WELLS GPK-3 AND GPK-4

Operation of chemical OCA stimulation conducted in the wells GPK-4 and GPK-3 (February 2007)

The third and fourth chemical stimulation operations (injection of OCA) were carried out into the wells GPK-3 and GPK-4 by Schlumberger between February 7 and 22, 2007.

The main objectives of this operation were to dissolve the remaining solid deposits observed during the previous production tests and enhance the permeability as far as possible from the wells in the hydrothermalized fracture and rock porosity zones.

The OCA solution is constituted of 5-10% citric acid ($C_6H_8O_7$), 0,1-1% HF, 0,5-1,5% HBF_4 and 1-5% NH_4Cl (Schlumberger catalog). Its use is especially recommended at temperatures higher than 180°C for the sandstone formations or formations with contents of zeolite or chlorite superior to 5%. This solution was preliminary tested in laboratory by Schlumberger on samples of GPK-3 cutting. After 5 hours of reaction at 80°C, the OCA solution dissolved 24% of the cutting samples and 41% at 180°C. Other laboratory tests were carried out at BRGM showing that the OCA solution could also dissolve the NTA solid compounds.

In GPK-3, 1250 m³ of fresh water were injected at 35 to 55 l/s before the injection of 200 m³ of OCA at 55 l/s. The amount of fresh water was reduced to 685 m³ into GPK-4 whereas the amount of injected OCA was identical (200 m³ at 50 l/s). After the OCA injection, about 1400 and 750 m³ of fresh water were injected into the wells GPK-3 and GPK-4, respectively (at 30 to 45 l/s). If no significant modification was observed in the GPK-3 behavior during the OCA treatment, some high pressure increases were observed during the preliminary injection of fresh water into GPK-4 at 35 l/s. After the OCA treatment and only 7 hours after the GPK-4 closing, the pressure at well head drastically decreased from 126 to 40 bars. For the moment, no definitive interpretation is given for this fast and significant pressure decrease.

The computations performed by GEOWATT using the results obtained during these production tests showed that the GPK-4 productivity still increased from 4 to 5 l/s/MPa after the OCA treatment. For the well GPK-3, the OCA treatment did not significantly improve its previous injectivity.

Geochemical monitoring of the discharged fluids from GPK-3 and GPK-4 in February and March 2007

The chemistry of the fluids discharged from GPK-3 and GPK-4 was monitored by BRGM and CFG Services during these production tests. Fluid sampling, on site measurements and chemical analyses similar to those carried out in the previous production tests were performed. Taking into account the chemical composition of OCA, additional analyses of dissolved F (on site and in laboratory), B and citrate ions were done by BRGM using specific electrode, ICP-AES and chromatography ion. Dissolved Na, Mg and Br were also analyzed using ICP-AES for the two cations and ion chromatography for Br. Relative analytical uncertainty ranged from 5 to 15% following the used method. Free NTA was analyzed by EGI laboratory in some fluid samples collected from GPK-4. All the obtained results are reported in Table 8.

Well GPK-3

After a production of 300 m³ of fluid, which slightly exceeds the volume of the well, fresh water is replaced by a mixing of native geothermal brine and fresh water. After, the conductivity values tend to increase from 34 to 43 mS/cm (Table 8; Fig. 7). As fresh water has been injected into GPK-3 during the OCA treatment, the conductivity values are lower than those measured at the end of the production test carried out in January 2007. Using the analyzed concentrations of dissolved Cl, Na, K, Ca, Mg and Br, it can be estimated that the fluid discharged from GPK-3 is constituted of about 24% of native geothermal brine and 76% of fresh water at the beginning of this production test.

These proportions become 33 and 67% at the end of this test, after a production of about 1900 m³ of fluid. They were of 68 and 32% at the end of the January 2007 test.

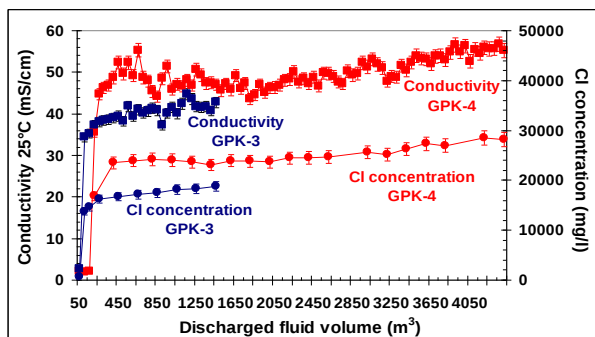


Figure 7 - Conductivity and concentrations of dissolved Cl measured in the fluid discharged from GPK-3 and GPK-4 in February and March 2007.

As observed for the conductivity values and the concentrations of dissolved Cl, K and Ca, the concentrations of 1,5-, 2,7-, 1,6-, 2,6-nds and fluorescein (Table 8) are lower than those analyzed in the previous test. Moreover, they tend to increase during this production test confirming the decreasing proportions of fresh water injected during the OCA treatment in the fluid discharged from GPK-3. The concentrations of 1,6- and 2,6-nds allow to estimate that this fluid, at the end of the production test, is constituted of about 4% of fresh water injected into GPK-3 in 2003 and 1% of fluid injected into GPK-4 in 2005. In comparison to those analyzed at the end of the January 2007 production test (Table 2), the concentrations of 1,6- and 2,6-nds suggest that the fluid discharged from GPK-3 at the beginning of this test has been diluted from 3 to 4 times by the fresh water injected into GPK-3 during the OCA treatment. These results are in good agreement with the concentrations of dissolved Cl, K and Ca, which indicate a dilution factor of about 3. Consequently, it can be estimated that the volume of fresh water injected into GPK-3 during the OCA treatment (2860 m³) has been mixed with about 1500 m³ of deep fluid.

Given its chemical composition, the OCA solution has a very low pH (< 1 at 20°C). In spite of its mixing with the fresh water injected into GPK-3 and the deep fluid, the pH values would have remained low if no chemical reaction with rocks and minerals had occurred. The pH values measured in the fluid discharged from GPK-3 ranging from 6.99 to 7.37 (Table 8; Fig. 8) indicate that the OCA solution has actively reacted with these latter (most probably silicate, aluminosilicate, carbonate and iron oxy-hydroxide minerals).

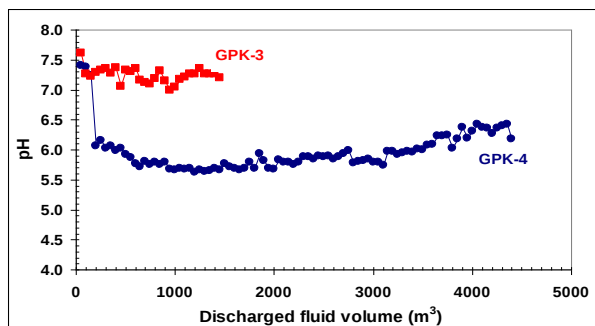


Figure 8 - pH values measured in the fluids discharged from GPK-3 and GPK-4 in February and March 2007.

At these pH values, citric acid is mainly present in form of C₆H₅O₇³⁻. The concentration of citric acid injected into GPK-3 during the OCA treatment was estimated at 2-5 g/l in the deep fluid taking into account the fluid mixing processes.

The concentrations of free citrate ions determined by ion chromatography ranging from 95 to 162 mg/l (Table 8) are in relative good agreement with the alkalinity values (Fig. 9), which are much higher than those analyzed in January 2007 (Table 2) and are mainly attributed to the presence of bicarbonate and citrate ions. Note that the TIC values (essentially bicarbonate ions) are similar to those analyzed in the previous production test. The low concentrations of free citrate relative to the concentration of injected citric acid suggest that at least 92% of this acid has reacted with minerals. The TOC concentrations (Table 8; Fig. 9) are much higher than those analyzed in January 2007 (Table 2) and decrease during the production test. If they are assumed to be mainly derived from the presence of free citrate and the formation of citrate complexes, it can be estimated that the concentrations of free and complexed citrate decrease from 1.9 to 1.2 g/l during the production test. These concentrations are concordant with those estimated for the citric acid injected in the deep fluid.

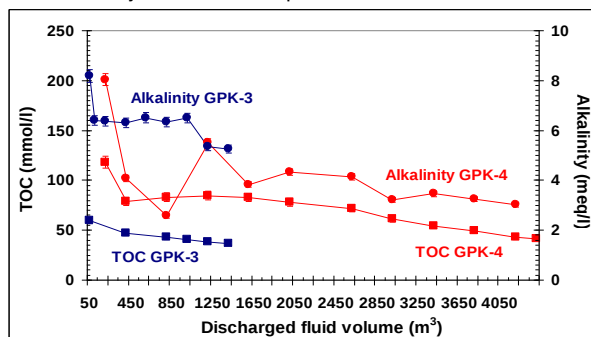


Figure 9 - Concentrations of TOC and alkalinity values determined in the fluids discharged from GPK-3 and GPK-4 in February and March 2007.

The analyzed concentrations of dissolved B (Table 8; Fig. 10) are in good agreement with the estimations done (35-92 mg/l) after the injection of 1-3 tons of HBF₄ in the deep fluid by taking into account the fluid mixing processes. As for the TOC concentrations, a decreasing trend is observed for this element during the production test (Fig. 10). The estimations done (245-1030 mg/l) for the concentration of dissolved F in the deep fluid after the injection of 0.2-2 tons of HF and 1-3 tons of HBF₄ are much higher than those analyzed in the fluid discharged from GPK-3 which also show a decreasing trend during the production test (Table 8; Fig. 10). These differences can be explained by the precipitation of fluorite (CaF₂) from the chemical attack of calcite, plagioclase, or clays by HF or HBF₄, releasing Ca ions in solution which are associated to F ions following the reaction:

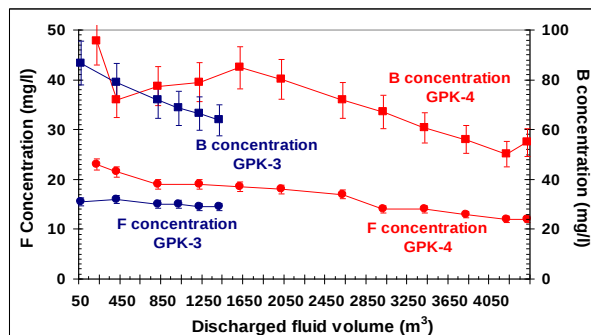
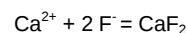


Figure 10 - Concentrations of dissolved F and B analyzed in the fluids discharged from GPK-3 and GPK-4 in February and March 2007.

Fluorite was observed in the deposit sample collected from GPK-3 using the optical microscope. Its amount was estimated at about 0.15% from the chemical fluoride analysis (Table 5). Calculations of mineral saturation using the EQ3NR geochemical code (Wolery, 1992) showed that the Soultz native geothermal brine was saturated with respect to fluorite (CaF_2) at 230-240°C (Sanjuan *et al.*, 2001). Other calculations performed at 200°C indicated that, if the fluids discharged from GPK-3 in January 2007 were under-saturated with respect to fluorite, they were over-saturated with respect to this mineral in February 2007. At lower temperatures, over-saturation was still more significant. In spite of the fluorite precipitation, the chemical attacks by HF and HBF_4 allow to increase the porosity volume because these acids dissolve numerous minerals such as quartz, feldspars, plagioclases, clays, carbonates, iron oxy-hydroxides, etc. Moreover, the molar volume of fluorite (24.542 cm^3) is lower than those of calcite (36.934 cm^3), plagioclase (100.79 cm^3) or clays. So, assuming that only calcite is attacked by 2 tons of HF and 3 tons of HBF_4 , it is estimated that these acids can dissolve a volume of about 3.1 m^3 of this mineral and form 2.1 m^3 of fluorite. The concentrations of dissolved F (Table 8; Fig. 10) compared to those estimated in the deep fluid after HF and HBF_4 injection suggest that at least 93% of fluoride have precipitated. The injection of 20 tons of citric acid can also dissolve a maximum volume of 3.9 m^3 of calcite.

The concentrations of dissolved oxygen measured in the fluid discharged from GPK-3 are close to zero (Table 8). The concentrations of dissolved Fe are much higher than those analyzed in the previous production test (Table 2), and even than those estimated for samples of diluted native geothermal brine with equivalent factors of dilution by fresh water. These results suggest a chemical attack of some iron oxy-hydroxide minerals and clays by the OCA treatment.

Well GPK-4

After a production of 200 m^3 of fluid, which is close to the well volume, fresh water is replaced by a mixing of native geothermal brine and fresh water. After, the conductivity values tend to increase from 35 to 55 mS/cm (Table 8; Fig. 7). As fresh water has been injected into GPK-4 during the OCA treatment, the conductivity values are lower than those measured at the end of the production test carried out in January 2007. Using the analyzed concentrations of dissolved Cl, Na, K, Ca and Br, it can be estimated that the fluid discharged from GPK-4 is constituted of about 29% of native geothermal brine and 71% of fresh water at the beginning of this production test. These proportions become 48% and 52% at the end of this test, after a production of about 4400 m^3 of fluid. They were of 56 and 44 % at the end of the January 2007 production test.

As observed for the conductivity values and the concentrations of dissolved Cl, K and Ca, the concentrations of 1,5-, 2,7-, 1,6-, 2,6-nds and fluorescein (Table 8) are lower than those analyzed in the previous test. Moreover, they tend to increase during this production test confirming the decreasing proportions of fresh water injected during the OCA treatment in the fluid discharged from GPK-4. The concentrations of 1,5-, 2,7-, 1,6- and 2,6-nds allow to estimate that this fluid, at the end of the production test, is constituted of about 0.1% of fresh water injected into GPK-2 in 2000, 0.07% of fresh water injected into GPK-2 in 2003, 0.9% of fresh water injected into GPK-3 in 2003 and 6% of fluid injected into GPK-4 in 2005. Compared to those analyzed at the end of the January 2007 test (Table 2), the concentrations of 2,7-, 1,6- and 2,6-nds suggest that the fluid discharged from GPK-4 at the beginning of this test has been diluted about 2 times by the fresh water injected into GPK-4 during the OCA treatment. These results are in good agreement with the Cl, K and Ca concentrations.

Consequently, it can be estimated that the volume of fresh water injected into GPK-4 during the OCA treatment (1635 m^3) has been mixed with about 1500 m^3 of deep fluid.

The pH values measured in the fluid discharged from GPK-4 ranging from 5.62 to 6.43 are lower than those determined in the GPK-3 fluid (Fig. 8). Even if these pH values still show that the OCA solution has actively reacted with minerals into GPK-4, they indicate that less H^+ ions have been consumed into this well where the RMA and NTA treatments had been previously carried out, contrary to GPK-3.

At these pH values, citric acid is mainly present in form of $\text{C}_6\text{H}_5\text{O}_7^{2-}$. The concentration of citric acid injected into GPK-4 during the OCA treatment was estimated at 3-6.5 g/l in the deep fluid taking into account the fluid mixing processes. The concentrations of free citrate ions analyzed by ion chromatography ranging from 34 to 77 mg/l (Table 8) are in relative good agreement with the alkalinity values, which are higher than those determined in January 2007 (Table 2) and are mainly attributed to the presence of bicarbonate and citrate ions. The TIC values (essentially bicarbonate ions and dissolved CO_2) are similar to those analyzed in the previous production test. The low concentrations of free citrate relative to the concentration of injected citric acid suggest that at least 97% of this acid has reacted with minerals. The TOC values (Table 8; Fig. 9) are lower than those analyzed in January 2007 (Table 2) and decrease during the production test. If they are assumed to be mainly derived from the presence of free citrate and the formation of citrate complexes, it can be estimated that the concentrations of free and complexed citrate decrease from 3.7 to 1.3 g/l during the production test. These values are concordant with those estimated for the citric acid injected in the deep fluid. No free NTA was detected (Table 8).

The analyzed concentrations of dissolved B (Table 8) are in good agreement with the estimations done (50-130 mg/l) after the injection of 1-3 tons of HBF_4 in the deep fluid by taking into account the fluid mixing processes. As for the TOC concentrations, a decreasing trend is observed for this element during the production test (Fig. 10). The estimations done (340-1435 mg/l) for the concentration of dissolved F in the deep fluid after the injection of 0.2-2 tons of HF and 1-3 tons of HBF_4 are much higher than those analyzed in the fluid discharged from GPK-4 which also show a decreasing trend during the production test (Table 8; Fig. 10). As for GPK-3, these differences can be explained by the fluorite precipitation from the chemical attack of calcite, plagioclase, or clays by HF or HBF_4 . Fluorite was detected in the two deposits collected from GPK-4 by X-ray diffraction and optical microscope. Its amounts were estimated at about 4.9 and 2.3% from the chemical fluoride analyses (Table 5).

Calculations of mineral saturation performed at 200°C indicated that, if the fluids discharged from GPK-4 in January 2007 were under-saturated with respect to fluorite, they were over-saturated with respect to this mineral in March 2007. At lower temperatures, over-saturation was still more significant. As for GPK-3, the chemical attacks by HF and HBF_4 allow to increase the porosity volume. The analyzed concentrations of dissolved F (Table 8; Fig. 10) compared to those estimated in the deep fluid after HF and HBF_4 injection suggest that at least 93% of fluoride have reacted. The concentrations of dissolved oxygen measured in the fluid discharged from GPK-4 are close to zero (Table 8). The concentrations of dissolved Fe are much higher than those analyzed in the previous production test (Table 2), and even than those estimated for samples of diluted native geothermal brine with equivalent factors of dilution by fresh water. They are much higher than those analyzed in the GPK-3 fluid (Table 8). These results suggest a chemical attack of some iron oxy-hydroxide minerals, clays and possible Ca-Fe-NTA compounds by the OCA treatment.

Chemical and mineralogical characterization of the solid deposits collected from GPK-3 and GPK-4

Three samples of deposit were collected by GEIE. One of the samples (rust-colored deposit) was sampled after the GPK-3 production on February 27, 2007. The two other samples (black and black and white deposits) were collected after the GPK-4 production on March 9, 2007. Similar chemical and mineralogical analyses to those done on the previous deposit samples were performed on these samples. As the two samples collected in January 2007, these samples are cleaner than those sampled in 2006. Drilling grease is much less abundant but organic phases constituted of crumbly and fragile grains are still observed in all the samples. These grains are rust, red and dark brown-colored in the GPK-3 deposit. They are rather black, olive-greenish gray, ocher-colored in the GPK-4 deposits.

Except the presence of neo-formed crumbly fluorite, the analyses give similar results to those obtained on the deposit samples collected in January 2007 (Tables 4 and 5). However, the amounts of barite and celestine, disseminated in small grains, are much lower. The proportions of silicate and alumino-silicate minerals (quartz, amorphous silica, feldspars, plagioclases, clays such as chlorite, illite, muscovite and biotite) are higher. Neo-formed automorph rod-shaped crystals of quartz present in a siliceous substratum and colorless vitreous grains of amorphous silica are also observed. Magnetite is still abundant and calcite is present. The presence of sulfide minerals (galena, sphalerite, pyrite) is detected or suspected and an excess of sulfur still exists (Table 5). Arsenic is present in all the samples but is much more abundant in the GPK-4 deposits (Table 4). It seems to be associated to present organic phases rather than a crystallized mineral. In the deposit sample collected after the GPK-3 production, traces of amphibole are detected and hematite is still present. Given the chemical composition of the fluid discharged from GPK-3 during the production test, this mineral probably comes from residual deposits of previously precipitated hematite. The white-colored deposit observed in the sample collected in the GPK-4 setting tank is constituted of 35% of silica (quartz and amorphous silica), barite and halite (formed during the sample drying).

All these samples contain rests of cuttings and granite fragments constituted of common minerals such as quartz, feldspars, plagioclases, micas, magnetite and hydrothermal deposits (calcite, chlorite, illite, muscovite). Organic phases rich in arsenic and neo-formed minerals such as quartz and crumbly fluorite are observed. Barite, celestine and traces of poly-metallic sulfides (galena, sphalerite, pyrite) are also probably neo-formed minerals. Residual hematite is only present in the GPK-3 deposit sample.

CONCLUSION

To conclude, the geochemical monitoring carried out by BRGM during this study shows that the RMA, NTA and OCA treatments have actively reacted with the encountered rocks.

In the well GPK-4, the successive injection of RMA and of a basic solution of NTA has allowed to improve the GPK-4 productivity from 3 to 4 l/s/MPa. After the injection of the basic solution, a plug and a corresponding increase of pressure were observed. The production test carried out just after this injection showed that this plug had been caused by the formation of abundant deposits extracted from GPK-4. These deposits were mainly made up of drilling wastes (grease, rests of cuttings) and rock fragments resulting from the cleaning of the well and some fractured areas by the basic NTA solution. These deposits were afterwards accompanied by brown-yellowish flocculates constituted of Ca-Fe-NTA compounds. The maximum volumes of calcite

dissolved by the RMA and NTA treatments were estimated at about 19.2 and 5.5 m³, respectively.

The OCA treatment has allowed improving the GPK-4 productivity from 4 to 5 l/s/MPa but seems to have been not very efficient on the GPK-3 injectivity. The maximum volume of calcite dissolved by the OCA treatment in both wells was estimated at about 7 m³. However, the OCA solution also attacks silicate, alumino-silicate and iron oxy-hydroxide minerals and Ca-Fe-NTA compounds. Precipitation of crumbly fluorite was observed in deposits mainly constituted of rests of cutting, rock fragments, organic phases rich in As and hydrothermal minerals. Even if the distribution and the size of the fractured areas are probably different in GPK-3 and GPK-4, the absence of an efficient cleaning in GPK-3 and neighboring fractured areas (which seems to be necessary) as well as the reduced use of chemical attacks could partially explain the less good results obtained on this well. The extraction of some rust-colored hematite deposits from GPK-3 where large volumes of surface fluids were injected suggest that more caution must be taken in the chemical treatments accompanying these injections.

The geochemical monitoring of the fluids discharged from GPK-3 and GPK-4 also indicates that the proportions of traced fresh water injected in 2000, 2003 and 2005 into GPK-2, GPK-3 and GPK-4 are always low (< 10%) compared to the proportions of native geothermal brine.

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Table 1 - Geochemical monitoring of the fluids discharged from GPK-3 and GPK-4 during the production tests carried out between October 10 and 27, 2006 (all the chemical analyses were performed by BRGM, except free NTA which was analyzed in the EGI laboratory, Utah University).

Operation	Date	Discharged volume (m ³)	T _{meas.} (°C)	TSS (mg/l)	Cond. 25°C (mS/cm)	pH	Cl (mg/l)	Ca ⁽¹⁾ (mg/l)	Ca ⁽²⁾ (mg/l)	% Ca diff.	TIC (mg/l)	TIC (mmol/l)	TOC (mg/l)	TOC (mmol/l)	TC (mg/l)	TC (mmol/l)	Alk. (meq/l)	NTA (ppm)
GPK-3 production	10/10/06 10:00				61.4	7.35	24139	2592	2509	2%	22	1.9	32	2.7	54	4.5		
	10/10/06 14:00				63.2	7.36												
	10/10/06 20:00				65.5	7.44												
	11/10/06 02:00				67.3	7.39												
	11/10/06 08:00				69.2	7.25												
	11/10/06 09:10	1750			65.8	7.16	28243	3076	2925	4%	20	1.7	76	6.3	96	8.0		
Injection of about 1300 m ³ of fluid into GPK-3	23/10/06 11:00				32.2	8.17	11724	1307	1260	3%	17	1.4	90	7.5	107	8.9		
	23/10/06 19:00				53.5	8.16												
	24/10/06 03:00				54.9	8.10												
	24/10/06 08:00				56.8	7.84	22098	2508	2481	1%	10	0.9	104	8.7	114	9.5		
GPK-4 production	23/10/06 14:30	219			50.3	6.61	18994	1706	1656	2%	58	4.8	261	21.7	318	26.5		
	23/10/06 14:50	250			49.3	6.82												
	23/10/06 15:26	320			49.3	7.16												
	23/10/06 16:00	370			49.9	7.15												
	23/10/06 16:21	420			50.5	7.15	18975	1554	1528	1%	58	4.8	377	31.4	434	36.2		
	25/10/06 18:10	100	17.1		51.5	6.52	19123	1770										
		200	17.4		41.8	7.15												
	25/10/06	300	16.8		52.8	7.34	19822	1704	1606	4%	41	3.5	283	23.6	325	27.1	4.54	488
		350	17.6		54.2	7.17												
		400	17.7		55.2	7.08												
		450	17.8		55.0	7.10												
	25/10/06	500	17.5		55.6	7.28	21258	1859	1761	4%	38	3.2	298	24.8	336	28.0		
		550	17.9		56.0	7.16												
		600	18.4		55.8	7.13												
	25/10/06	650	18.4		56.0	7.18	21266	1854										
	25/10/06	700	18.4		56.1	7.24	21541	1949	1823	5%	37	3.1	332	27.6	369	30.8		251
		750	19.4		55.9	7.21												
		800	20.4		56.0	7.20												
		850	20.4		56.2	7.22												
	25/10/06	900	19.1	314	55.9	7.32	21345	1899	1852	2%	36	3.0	329	27.4	365	30.4		242
		950	20.4		55.8	7.29												
		1000	20.4		55.8	7.30												
		1050	21.6		55.9	7.30												
	26/10/06	1100	21.7		55.5	7.32	21260	1871	1807	2%	35	2.9	288	24.0	323	26.9		239
	26/10/06	1150	22.0		56.0	7.27	20185	2019										
		1200	21.9		55.3	7.26												
		1250	22.1		55.5	7.24												
	26/10/06	1300	24.5		55.1	7.37	21023	1868	1775	4%	35	2.9	290	24.1	324	27.0		208
		1350	22.9		55.8	7.31												
		1400	23.8		55.0	7.33												
		1450	26.4		55.1	7.40												
	26/10/06	1500	20.6		55.1	7.24	20889	1855	1763	4%	35	2.9	297	24.8	332	27.7		232
		1550	20.4		54.6	7.07												
		1600	20.8		54.7	7.21												
	26/10/06	1650	21.1		54.5	7.33	20350	1799										
	26/10/06	1700	21.1		54.3	7.10	20898	1794	1714	3%	35	3.0	333	27.7	368	30.7		264
		1750	21.2		54.0	7.36												
		1800	21.4		54.4	7.30												
		1850	21.7		54.4	7.30												
	27/10/06	1900	22.5	229	54.9	7.35	20872	1834	1692	6%	34	2.8	323	27.0	357	29.7	4.47	264
		1950	22.0		54.9	7.28												
			23.4		55.0	7.28												
	27/10/06	2050	23.4		55.0	7.34	20197	1880										255
	27/10/06	2220																284

TSS: Total Suspended Solids

⁽¹⁾ Analyses done on 0.45 µm filtered and HNO₃ acidified samples

⁽²⁾ Analyses done on non filtered samples acidified using 30% diluted HCl

Table 2 - Geochemical monitoring of the fluids discharged from GPK-3 and GPK-4 during the production tests carried out between January 22 and 26, 2007 (all the chemical analyses were performed by BRGM, except free NTA which was analyzed in the EGI laboratory, Utah University).

Well	Date	Discharged volume (m ³)	T _{test} (°C)	Weight of Specific gravity	pH	Cond. 25°C (mS/cm)	Eh (mV)	Diss. O ₂ (mg/l)	Ca (mg/l)	K (mg/l)	Cl (mg/l)	Alk. (meq/l)	TC (mmol/l)	TC (mg/l)	TIC (mmol/l)	TIC (mg/l)	TOC (mmol/l)	TOC (mg/l)	N/A (ppm)	Li (mg/l)	Fe (mg/l)	Mn (μg/l)	Zn (μg/l)	Cr (μg/l)	Cu (μg/l)	Ni (μg/l)	Pb (μg/l)	Fluo. (μg/l)	1.5 mls 2.7 mls (μg/l)	1.5 mls 2.6 mls (μg/l)	Fluid colour			
GPK 4	22/01/2007 09:30	30	160	50.04	1.008	7.47	5.5	86	1.50	7.3	63.2	1.81	62.3	13.53	25.3	2.11	137.0	11.42	nd	0.128	20.1	13.9	406	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown		
	22/01/2007 10:20	100	162	50.12	1.0024	6.87	4.2	95	1.72	6.0	7.3	1.81	62.3	13.53	25.3	2.11	137.0	11.42	nd	0.128	20.1	13.9	406	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Translucent		
	22/01/2007 11:10	250	162	50.13	1.0026	6.27	3.7	106	1.63	3807	1506	4000	2.13	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 12:00	400	162	52.17	1.0434	6.23	7.52	106	1.63	3370	1509	4000	2.16	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 13:30	600	162	52.52	1.0204	6.53	74.7	4.0	0.43	3370	1509	4000	2.16	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 15:15	540	162	52.52	1.0434	6.53	74.7	4.0	0.43	3370	1509	4000	2.16	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 17:00	600	162	52.41	1.0466	6.49	74.1	4.0	0.43	3370	1509	4000	2.16	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 18:40	600	162	52.45	1.0466	6.49	74.1	4.0	0.43	3370	1509	4000	2.16	107.5	8.96	13.7	1.14	93.6	7.80	82.4	25.2	13.6	369	<25	<20	<25	<5	6.5	4.0	7.1	29.9	Brown yellow		
	22/01/2007 20:30	650	163	52.45	1.0490	6.58	73.8	4.8	0.27	3490	1581	3680	2.12	111.6	9.30	12.0	1.00	99.6	8.30	86.9											Pale yellow			
	22/01/2007 22:30	700	164	52.77	1.0458	6.62	73.0	3.5	0.55	3490	1581	3680	2.12	111.6	9.30	12.0	1.00	99.6	8.30	86.9												Pale yellow		
	23/01/2007 00:55	750	19.5	52.77	1.0564	6.51	73.9	-19	0.41																								Very pale yellow	
	23/01/2007 02:40	800	19.5	52.18	1.0436	6.76	71.8	-81	0.17																								Cloudy white	
	23/01/2007 03:30	850	19.5	52.18	1.0436	6.74	71.8	-71	0.35																								Cloudy white	
	23/01/2007 04:25	900	19.8	52.48	1.0448	6.65	71.4	-25	0.49	3530	1591	37300	1.99	117.0	9.75	11.3	0.94	105.7	8.81	88.4	24.4	13.3	363	<25	<20	<25	<5	8.2	1.7	7.5	37.4	211	Cloudy white	
	23/01/2007 05:00	950	19.7	52.45	1.0490	6.84	70.3	-96	0.14																								Cloudy white	
	23/01/2007 06:30	1000	19.9	52.31	1.0462	6.81	70.1	-92	0.11																								Very pale yellow	
	23/01/2007 08:00	1050	20.2	52.48	1.0456	6.82	69.1	-88	0.21																								Pale yellow	
	23/01/2007 09:30	1100	21.1	52.30	1.0460	6.62	69.9	-33	0.34	3553	1615	36800	2.04	119.4	9.95	11.3	0.94	106.1	9.01	88.6	24.2	13.1	332	<25	<20	<25	<5	9.1	2.1	7.5	30.0	213	Pale yellow	
	23/01/2007 11:00	1150	22.0	52.33	1.0426	6.66	69.1	-31	0.35	3549	1625	36900	2.07	110.7	9.23	9.7	0.81	101.0	8.42	86.3	27.3	12.5	354	<25	<20	<25	<5	9.2	2.1	7.8	33.9	218	Pale yellow	
	23/01/2007 12:30	1200	21.7	52.32	1.0424	6.68	69.1	-16	0.36																								Very pale yellow	
	23/01/2007 14:00	1250	21.7	52.32	1.0424	6.68	69.1	-16	0.36																								Very pale yellow	
	23/01/2007 15:30	1300	24.5	51.94	1.0388	6.88	69.3	-78	0.19																								Cloudy white	
	23/01/2007 17:00	1350	24.5	51.94	1.0388	6.88	69.3	-78	0.19																								Cloudy white	
	23/01/2007 19:30	1414	20.9	52.56	1.0512	6.38	69.1	-16	0.55	3487	1609	35400	2.03	112.3	9.36	7.7	0.64	104.6	8.72	nd	85.2												Pale yellow	
	24/01/2007 00:55	1514	20.9	52.34	1.0468	6.41	67.6	-57	0.09																									Pale yellow
	24/01/2007 02:40	1560	21.9	52.29	1.0458	6.35	66.4	-51	0.09																									Pale yellow
	24/01/2007 04:25	1600	21.6	52.17	1.0434	6.45	68.2	-7	0.06																									Pale yellow
	24/01/2007 06:15	1650	20.2	52.32	1.0464	6.41	67.2	-10	0.31	3556	1621	35100	2.04	114.3	9.53	9.4	0.78	104.9	8.74	87.0													Pale yellow	
24/01/2007 07:50	1700	15.4	52.29	1.0458	6.54	66.8	-33	0																									Very pale yellow	
24/01/2007 09:30	1750	20.5	52.09	1.0418	6.53	68.2	-19	0.04																									Very pale yellow	
24/01/2007 11:40	1800	21.1	52.37	1.0474	6.54	67.4	-17	0.31																									Very pale yellow	
24/01/2007 13:30	1850	22.2	52.26	1.0462	6.61	69.0	-25	0.04	3649	1689	35300	2.06	114.9	9.58	9.1	0.76	105.8	8.82	86.9	25.5	12.4	262	<25	<20	<25	<5	12.1	2.4	8.5	39.0	217	Very pale yellow		
24/01/2007 15:30	1915	15.3	52.96	1.0512	6.60	68.9	-41	0																									Pale yellow	
24/01/2007 17:00	1945	19.4	52.29	1.0458	6.72	68.9	-56	0																									Very pale yellow	
24/01/2007 18:30	1980	19.4	52.30	1.0458	6.72	68.9	-56	0	3611	1654	35200	2.07	119.7	9.98	0.94	0.84			nd	87.3												Very pale yellow		
24/01/2007 20:00	2060	19.4	52.40	1.0468	6.58	69.3	-32	0.54	3611	1654	35200	2.07	119.7	9.98	0.94	0.84			nd	87.3												Very pale yellow		
25/01/2007 01:05	2100	19.4	52.08	1.0468	6.58	69.3	-32	0.54	3639	1654	35300	2.01	111.0	9.25	6.5	0.54	104.5	8.71	85.3	19.0	12.3	252	<25	<20	<25	<5	13.0	2.5	8.0	41.7	218	Pale yellow		
25/01/2007 03:05	2160	18.3	52.31	1.0462	6.53	67.5	-9	0.61																									Pale yellow	
25/01/2007 05:00	2200	19.8	52.41	1.0482	6.63	66.4	-9	0.24	3680	1642	34700	2.12	115.4	9.62					nd	90.4												Pale yellow		
25/01/2007 06:55	2250	19.2	52.17	1.0434	6.72	66.4	-65	0																									Pale yellow	
25/01/2007 08:45	2300	19.1	52.22	1.0444	6.73	66.8	-51	0.43																									Pale yellow	
25/01/2007 10:40	2350	18.1	52.41	1.0482	6.81	67.0	-71	0																									Pale yellow	
25/01/2007 11:00	2380	19.9	52.07	1.0418	6.72	68.7	-1	0.74	3670	1692	35200	2.12	108.6	9.05	7.3	0.61	101.3	8.44	nd	91.5	25.7	13.0	246	<25	<20	<25	<5	14.5	3.4	8.1	39.5	217	Pale yellow	
25/01/2007 12:10	100	19.5	50.37	1.0474	7.23	4.3	103	2.25																									Pale red	
25/01/2007 12:40	177	20.5	50.10	1.0020	7.30	5.3	238	2.73	238	2.73	1960	1.43	2																					

Table 3 - Partial geochemical monitoring of the fluids discharged from GPK-3 and GPK-4 and injected into GPK-3 during the fluid circulation test carried out between July and December 2005 (all the chemical analyses were performed by BRGM).

Sample n°	Date	Cond. (25°C) mS/cm	pH	Cl mg/l	SO ₄ mg/l	Br mg/l	Alk. meq/l	Na mg/l	K mg/l	Ca mg/l	Mg mg/l	Li mg/l	SiO ₂ mg/l	Fe mg/l	Mn mg/l	Zn µg/l	Pb µg/l	Ni µg/l	Cu µg/l	Cr µg/l	Ag µg/l	Fluo. µg/l	1,5-nds µg/l	2,7-nds µg/l	1,6-nds µg/l	2,6-nds µg/l	TDS g/l	Cl/Br
GPK2-05-P1	06/07/05 13:00	96.7	5.90	41363	151	155	2.29	19774	1768	3880	88.4	86.7		16.5	15.0	1778	144	<25	<20	<25	<5	413	66.3	147	198	<10	67.3	267
GPK2-05-P69	15/08/05 08:45	108.2	5.25	46164	145	199	n.a.	20759	2313	5432	107	119		13.1	15.1	1740	28	<25	<20	<25	<5	266	70.5	138	247	75.5	75.2	233
GPK2-05-P79	24/08/05 08:45	108.5	5.25	46629	146	203	n.a.	21657	2410	5664	111	120	267	13.1	15.1	1740	28	<25	<20	<25	<5	266	67.9	131	249	72.9	77.4	231
GPK2-05-P99	13/09/05 09:00	109.0	5.30	47564	148	211	2.11	21894	2445	5781	113	123	240	16.4	15.2	1787	75	<25	<20	<25	<5	203	57.2	117	227	72.4	78.5	225
GPK2-05-P121	05/10/05 09:00	113.0	5.38	48571	151	212	2.11	23100	2510	5935	114	137	246	16.4	15.5	1851	33	<25	<20	<25	<5	165	53.7	113	212	72.8	81.0	229
GPK2-05-P130	19/10/05 09:00	111.1	5.83	49624	164	214	n.a.	21600	2440	5879	101	125	190	17.7	15.4	1831	22	<25	<20	<25	<5	132	50.7	104	204	77.7	80.5	233
GPK2-05-P149	22/11/05 09:00	110.3	5.39	49321	157	219	2.26	22891	2592	6100	106	130	255	14.7	14.9	1680	93	<25	<20	<25	<5	83.1	49.5	98.3	192	81.5	81.8	225
GPK4-05-P1	04/07/05 17:00	86.3	6.43	35826	214	92	1.61	18690	1077	2503	44.0	52.8		40.4	14.3	1072	34	<25	<20	<25	<5	<0.6	<0.2	4.2	10.8	694	58.5	389
GPK4-05-P83	15/08/05 08:45	97.9	5.35	40754	169	170	n.a.	18843	2062	4939	92.5	100		36.3	14.5	1118	37	<25	<20	<25	<5	1.1	1.4	9.0	57.9	646	67.1	240
GPK4-05-P101	24/08/05 08:45	100.0	5.52	42223	168	178	n.a.	19303	2120	5051	95.3	103	216	35.0	15.3	1253	117	<25	<20	<25	<5	8.3	2.7	11.6	73.7	511	69.5	237
GPK4-05-P141	13/09/05 09:00	101.5	5.00	43899	168	184	2.93	19999	2229	5289	99.3	109	221	35.0	15.3	1253	117	<25	<20	<25	<5	7	3.7	12.2	96.7	446	72.2	238
GPK4-05-P184	05/10/05 17:00	105.0	5.07	44748	177	190	2.62	20000	2330	5570	102	119	222	32.4	16.3	1504	111	<25	<20	<25	<5	15.5	4.0	13.6	91.8	413	72.8	222
GPK4-05-P183	19/10/05 09:00	105.2	4.87	44500	176	201	n.a.	20000	2209	5321	87.9	116	208	32.4	16.3	1504	111	<25	<20	<25	<5	22.5	8.0	20.8	130	334	75.1	229
GPK4-05-P212	22/11/05 09:00	106.4	5.16	44646	166	196	3.70	21020	2376	6030	102	96.6	231	34.6	16.1	1538	14	<25	<20	<25	<5	24.7	8.0	20.8	130	334	75.1	229
GPK3-05-BOUCL37	10/08/05 09:00	105.4	5.33	44900	153.2	188.4	n.a.	20540	2277	5363	105	113	189	19.6	14.7	1588	45	<25	<20	<25	<5		55.7	86.3	204	178	73.8	238
GPK3-05-BOUCL52	24/08/05 08:45	107.8	5.59	46018	153.4	197.1	n.a.	20986	2341	5486	108	117	241	18.4	15.0	1636	49	<25	<20	<25	<5	215	50.9	127	225	175	75.6	233
GPK3-05-BOUCL72	13/09/05 09:00	108.3	5.13	46877	150.1	200.7	2.08	21480	2402	5658	110	119	237	19.9	15.5	1721	92	<25	<20	<25	<5	158	46.1	93.5	203	155	77.2	234
GPK3-05-BOUCL94	05/10/05 09:00	111.4	5.26	47776	149.9	205.5	2.24	22700	2460	5820	112	129	242	20.2	15.6	1748	112	<25	<20	<25	<5	147	43.8	83.6	188	146	79.6	232
GPK3-05-BOUCL103	19/10/05 09:00	107.5	6.27	46500	169	203.1	n.a.	21200	2362	5633	99.1	125	160	<0.4	13.3	964	13	<25	<20	<25	<5	50.1	37.9	88.2	186	157	76.5	229
GPK3-05-BOUCL117	22/11/05 09:00	87.5	6.77	39264	119	174	1.33	17400	1970	5135	100	115	178	<0.4	11.7	793	<5	<25	<20	<25	<5	62.4	31.3	70.1	149	96.6	64.5	226

n.a. : not analyzed

GPK-2 : Opening on July 6, 2005 at 12h10
 GPK-4 : Opening on July 4, 2005 at 10h45
 GPK-3 : Opening on July 6, 2005 at 12h10

Table 4 - Chemical composition of deposits collected from the wells GPK-3 and GPK-4 in 2005, 2006 and 2007.

Parameter	Unit	Deposit ⁽¹⁾ (circulation test)	Sediment ⁽¹⁾ (circulation test)	GPK-3 ⁽¹⁾ (after production)	GPK-3 ⁽¹⁾ (Well Head Annulus)	GPK-3 ⁽²⁾ (Upper part of the tubing 9'5/8)	GPK-3 ⁽²⁾ (Upper part of the tubing 9'5/8)	GPK-3 ⁽²⁾ (Upper part of the tubing 9'5/8)	Separator ⁽²⁾ (GPK-3) Rust-colored deposits (27/02/2007)	Separator ⁽²⁾ (GPK-3/GPK-4 production)	GPK-4 ⁽¹⁾ (GPK-4-13) Black deposits (25/10/2006)	GPK-4 ⁽¹⁾ (Well Head Annulus) Black deposits (6/11/2006)	GPK-4 ⁽²⁾ (sediment trap after production) Black deposits (31/01/07)	Separator ⁽²⁾ (GPK-4 production) Black deposits (9/03/2007)	GPK-4 ⁽²⁾ settling tank Black/white deposits (9/03/2007)
Mass loss at 450°C	%	2.55	9.90	4.60	13.6	44.4	n.a.	n.a.	1.86	3.25	< 0.05	16.8	2.89	8.70	8.90
Fe ₂ O ₃	%	84.5	41.3	20.9	15.0	12.4	n.a.	n.a.	14.7	17.2	14.7	9.70	16.8	11.1	9.60
CaO	%	5.80	5.70	4.40	1.30	1.20	n.a.	n.a.	2.70	10.8	2.80	1.10	4.00	10.0	9.80
C _{organic}	%	0.87	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.80	3.66	n.a.	n.a.	3.35	4.88	2.62
C _{mineral}	%	2.50	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.48	1.84	n.a.	n.a.	0.45	0.82	1.45
C _{total}	%	3.17	n.a.	4.20	10.8	39.5	28.8	n.a.	1.28	5.30	7.75	18.1	3.80	5.70	4.07
S	%	1.10	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
SO ₄	%	0.76	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	4.70	5.20	5.80	7.80	11.3	3.20	3.72
S _{total}	%	1.35	n.a.	4.83	11.5	7.00	1.95	n.a.	0.07	n.a.	n.a.	n.a.	n.a.	2.40	1.13
F	%	1.80	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	42.7	18.4	13.6	4.60	10.8	36.9	38.0
SiO ₂	%	< 1	1.50	5.90	< 1	< 1	n.a.	n.a.	8.70	3.60	1.80	< 1	1.60	7.30	7.80
Al ₂ O ₃	%	0.08	n.a.	2.35	4.12	1.00	n.a.	n.a.	2.40	0.87	0.44	2.98	0.19	n.a.	n.a.
Na ₂ O	%	< 1	1.00	1.80	< 0.5	< 0.5	n.a.	n.a.	< 1	1.20	< 0.5	< 0.5	< 0.5	2.00	2.00
K ₂ O	%	< 1	< 1	< 1	< 1	< 1	n.a.	n.a.	< 1	< 1	< 1	< 1	< 1	< 1	< 1
MgO	%	0.73	0.29	0.12	0.07	0.07	n.a.	n.a.	0.07	0.32	0.04	0.07	0.16	0.07	0.22
MnO	%	< 0.01	0.09	0.35	0.01	0.04	n.a.	n.a.	0.36	0.20	0.24	0.04	0.20	1.09	0.36
P ₂ O ₅	mg/kg	3395	1410	4399	728	660	n.a.	n.a.	1260	2990	989	330	1460	891	749
Sr	mg/kg	5240	36200	12200	13600	20100	n.a.	n.a.	7840	19700	16300	26400	31300	8940	17100
Ba	mg/kg	27600	27600	8700	1020	1800	n.a.	n.a.	1493	103000	8630	2430	232000	5830	4090
As	mg/kg	2040	2910	8300	5300	5060	n.a.	n.a.	10700	38300	115000	24000	44400	60100	46800
Pb	mg/kg	29700	617	1886	650	5500	n.a.	n.a.	705	1540	1144	800	1330	1445	4130
Zn	mg/kg	5940	5660	5910	2454	2780	n.a.	n.a.	1493	4520	8120	2804	8110	5830	4090
Cu	mg/kg	1070	800	570	281	696	n.a.	n.a.	360	932	2331	820	2980	1824	1071
Sb	mg/kg	424	137	296	328	1126	n.a.	n.a.	367	716	1354	421	1250	912	813
Cr	mg/kg	145	625	886	133	98	n.a.	n.a.	662	544	1155	256	912	991	553
Cd	mg/kg	181	19	32	22	41	n.a.	n.a.	22	88	106	38	188	104	78
Ni	mg/kg	97	180	116	32	17	n.a.	n.a.	127	340	1416	283	784	762	480
Co	mg/kg	27	25	30	13	7	n.a.	n.a.	22	88	213	54	160	160	93
Sn	mg/kg	21	88	30	22	15	n.a.	n.a.	< 10	16	51	33	60	33	20
Ag	mg/kg	92	< 1	< 0.2	2	26	n.a.	n.a.	5	21	52	8	28	40	26
Li	mg/kg	58	141	74	12	21	n.a.	n.a.	38	48	36	13	32	151	167
B	mg/kg	17	< 10	23	26	54	n.a.	n.a.	58	40	25	39	40	151	156
W	mg/kg	48	87	146	119	57	n.a.	n.a.	93	108	39	23	56	56	36
Ce	mg/kg	43	28	43	< 10	< 10	n.a.	n.a.	80	92	75	< 10	84	160	98
Zr	mg/kg	29	54	181	< 20	44	n.a.	n.a.	102	72	192	< 20	60	382	211
V	mg/kg	28	15	82	64	46	n.a.	n.a.	51	< 10	< 10	< 10	< 10	11	< 10
Mo	mg/kg	22	64	58	48	39	n.a.	n.a.	13	28	360	109	188	158	111
Be	mg/kg	12	10	14	6	6	n.a.	n.a.	20	20	4	< 2	8	13	33
Nb	mg/kg	41	< 20	< 20	< 20	< 20	n.a.	n.a.	< 20	92	24	< 20	< 20	44	22
Y	mg/kg	< 20	< 20	21	< 20	< 20	n.a.	n.a.	< 20	92	< 20	< 20	40	100	91
La	mg/kg	< 20	< 20	25	< 20	< 20	n.a.	n.a.	42	44	33	< 20	48	78	51
Bi	mg/kg	< 10	< 10	< 10	< 10	< 10	n.a.	n.a.	24	44	< 10	11	32	16	18

n.a.: not analyzed

⁽¹⁾ Sample dried at 40°C before analyses⁽²⁾ Sample dried at 105°C before analyses⁽³⁾ Sample not dried before analyses

Table 5 - Reconstructed mineralogical composition of deposits collected from the wells GPK-3 and GPK-4 in 2005, 2006 and 2007.

Phase	Unit	Deposit ⁽¹⁾ (circulation test)	GPK-3 ⁽¹⁾ (after production)	GPK-3 ⁽¹⁾ (Well Head Annulus)	GPK-3 ⁽³⁾ (Upper part of the tubing Internal (Nov. 2006)	GPK-3 ⁽³⁾ (Upper part of the tubing External (Nov. 2006)	Separator ⁽²⁾ (GPK-3 production) Rust-colored deposits (27/02/2007)	Separator ⁽²⁾ (GPK-3/GPK-4 production) Rust-colored deposits (31/01/07)	GPK-4 ⁽¹⁾ (GPK4-13) Black deposits (25/10/2006)	GPK-4 ⁽¹⁾ (Well Head Annulus) Black deposits (6/11/2006)	GPK-4 ⁽²⁾ (sediment trap after production) Black deposits (31/01/07)	Separator ⁽²⁾ (GPK-4 production) Black deposits (9/03/2007)	GPK-4 ⁽²⁾ settling tank Black/white deposits (9/03/2007)
Water (H ₂ O)	%	2.6	4.6	14	44	n.a.	1.9	3.3	< 0.05	17	2.9	8.7	8.9
Organic carbon (C _{org})	%	0.67	3.3	11	38	29	0.80	3.7	7.2	18	3.4	4.9	2.8
Amorphous phases		traces	abundant grease	abundant grease	abundant grease	abundant grease	absent	low amounts	abundant grease	abundant grease	present	low amounts	present
Magnetite (Fe ₃ O ₄)	%	67	20	15	12	low amounts	14	traces	14	9.4	16	11	9.3
Hématite (Fe ₂ O ₃)	%		traces	traces			traces	traces					
Siderite (FeCO ₃)	%	12	7.9	2.3	2.1	n.a.	4.0	14	5.0	2.0	3.8	6.8	12
Calcite (CaCO ₃)	%	10				n.a.	0.15					4.9	2.3
Fluorite (CaF ₂)	%		30	4.0	9.0	n.a.	43	18	14	4.6	11	37	38
Silica (SiO ₂)	%	1.8	present	traces		traces	low amounts	traces	present	traces	low amounts	low amounts	traces
Quartz (SiO ₂)	%						0.46	3.2			1.9	6.2	3.0
Calcium oxide (CaO)	%		2.4	4.1	1.0	n.a.	n.a.	0.87	0.44	3.0	0.18	n.a.	n.a.
Sodium oxide (Na ₂ O)	%		1.6			n.a.	2.4	1.2				2.0	2.0
Potassium oxide (K ₂ O)	%		5.9			n.a.	8.7	3.6	1.8		1.6	7.3	7.8
Aluminium oxide (Al ₂ O ₃)	%		traces			traces	present	low amounts	traces			traces	traces
Albite (NaAlSi ₃ O ₈)	%		traces			traces	low amounts	low amounts	traces			traces	traces
Microcline (KAlSi ₃ O ₈)	%		traces			traces	low amounts	present	traces		present	low amounts	low amounts
Plagioclase			traces			traces	traces	low amounts	traces			traces	traces
Micasillite			traces			traces	traces	traces	traces			traces	traces
Chlorite			traces			traces	traces	traces	traces			traces	traces
Amphiboles			traces			traces	traces	traces	traces			traces	traces
Halite (NaCl)	%	1.0	1.5	0.17	0.27	traces	2.7	17.5	1.5	0.41	39	2.0	1.9
Sylvite (KCl)	%	1.0	2.6	2.9	4.2	traces	1.6	4.1	3.4	5.5	6.6	1.9	3.6
Barite (BaSO ₄)	%	present	3.9	10.9	6.0	2.0	4.0	1.8	4.6	6.6	4.3	2.3	2.6
Celestine (SrSO ₄)	%	3.5	0.20	0.08	0.63	traces	0.08	0.18	0.13	0.09	0.15	0.17	0.48
Sulphide (S)	%	0.80	0.88	0.37	0.41		0.22	0.67	1.2	0.42	1.2	0.87	0.61
Galena (PbS)	%		?	traces	?		?	?	?	?	?	traces	traces
Sphalerite (ZnS)	%	0.70											
Pyrite (FeS ₂)	%	0.73	0.12	0.07	0.07	n.a.	0.07	0.32	0.04	0.07	0.16	0.07	0.22
Manganese oxide (MnO)	%	0.34	0.44	0.07	0.07	n.a.	0.13	0.30	0.10	0.03	0.15	0.09	0.07
Phosphore oxide (P ₂ O ₅)	%		traces										
Apatite (Ca ₅ (PO ₄) ₃ (OH, Cl, F))	%	< 0.01	0.35	0.01	0.04	n.a.	0.36	0.20	0.24	0.04	0.20	1.1	0.36
Titane oxide (TiO ₂)	%	0.20	0.83	0.53	0.51	n.a.	1.1	3.8	11.5	2.4	4.4	6.0	4.7
Arsenic (As)	%	0.11	0.06	0.03	0.07	n.a.	0.04	0.09	0.23	0.08	0.30	0.18	0.11
Copper (Cu)	%												

n.a.: not analyzed

⁽¹⁾ Sample dried at 40°C before analyses⁽²⁾ Sample dried at 105°C before analyses⁽³⁾ Sample not dried before analyses

Table 6 - Chemical composition of deposits collected from the well GPK-4 in October 2006.

Parameter	Unit	GPk-4 Brown-Yellowish deposits ⁽¹⁾ (26/10/2006)	GPk-4 Brown-Yellowish deposits ⁽¹⁾ Wet	GPk-4 Brown-Yellowish deposits ⁽¹⁾ Dry	GPk-4 TSS (314 mg/l) ⁽²⁾ (collected from the fluid sample 900 m ³) (25/10/2006)	GPk-4 TSS (229 mg/l) ⁽²⁾ (collected from the fluid sample 1900 m ³) (27/10/2006)
Mass loss at 450°C	%	n.a.	22.9	17.2	n.a.	n.a.
Fe ₂ O ₃	%	14.0		11.2	< 1	< 1
C _{total}	%	13.9	3.7	6.8	2.8	3.6
S _{total}	%	0.76	0.23	0.30	0.11	0.16
SiO ₂	%	9.3	9.8	8.0	< 1	< 1
CaO	%	10.2	8.6	10.5	6.3	6.4
Na ₂ O	%	15.2	20.7	21.5	n.a.	n.a.
Al ₂ O ₃	%	< 1	< 1	< 1	< 1	< 1
K ₂ O	%	1.0	0.9	1.5	3.4	3.5
MgO	%	< 1	< 1	< 1	< 1	< 1
MnO	%	0.36	0.32	0.28	0.02	0.02
TiO ₂	%	0.03	< 0.01	0.01	< 0.01	< 0.01
P ₂ O ₅	mg/kg	1265	439	622	< 100	< 100
Ba	mg/kg	8090	1384	2010	91	120
Sr	mg/kg	4570	2890	4420	3291	3340
As	mg/kg	2250	728	1260	20	42
Pb	mg/kg	1986	352	386	< 10	< 10
Zn	mg/kg	1110	213	275	6	16
Li	mg/kg	468	367	801	1321	1384
B	mg/kg	196	247	253	331	356
Cr	mg/kg	226	28	63	< 10	< 10
Cu	mg/kg	219	37	48	< 5	< 5
Y	mg/kg	152	118	150	< 20	< 20
Sb	mg/kg	104	16	23	< 10	< 10
Be	mg/kg	48	32	45	< 2	< 2
Ni	mg/kg	75	< 10	28	< 10	< 10
Cd	mg/kg	4	< 2	< 2	< 2	< 2
Ag	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
W	mg/kg	< 10	< 10	< 10	< 10	< 10
Ce	mg/kg	10	< 10	< 10	< 10	< 10
Nb	mg/kg	< 20	< 20	< 20	< 20	< 20
Zr	mg/kg	35	< 20	< 20	< 20	< 20
V	mg/kg	< 10	< 10	< 10	< 10	< 10
Co	mg/kg	< 5	< 5	< 5	< 5	< 5
Mo	mg/kg	8	< 5	< 5	< 5	< 5
Sn	mg/kg	< 10	< 10	< 10	< 10	< 10
La	mg/kg	< 20	< 20	< 20	< 20	< 20
Bi	mg/kg	< 10	< 10	< 10	< 10	< 10

n.a.: not analyzed, TSS: Total Suspended Solids, ⁽¹⁾ Sample dried at 40°C before analyses, ⁽²⁾ Sample dried at 105°C before analyses

Table 7 - Reconstructed mineralogical composition of deposits collected from the well GPK-4 in October 2006.

Phase	Unit	GPk-4 Brown-Yellowish deposits ⁽¹⁾ (26/10/2006)	GPk-4 Brown-Yellowish deposits ⁽¹⁾ Wet	GPk-4 Brown-Yellowish deposits ⁽¹⁾ Dry	GPk-4 TSS (314 mg/l) ⁽²⁾ (collected from the fluid sample 900 m ³) (25/10/2006)	GPk-4 TSS (229 mg/l) ⁽²⁾ (collected from the fluid sample 1900 m ³) (27/10/2006)
Water (H ₂ O)	%	n.a.	22.9	17.2	n.a.	n.a.
Amorphous organic phase (NTA?)		abundant	abundant	abundant	abundant	abundant
Organic carbon (C _{org})	%	> 12	> 1.9	> 3.6	> 1.5	> 2.2
Total Carbon (C _T)	%	14	3.7	6.8	2.8	3.6
Iron (Fe)	%	9.8	6.5	7.8	< 0.7	< 0.7
Calcium (Ca)	%	< 7.3	< 6.1	< 7.5	< 4.5	< 4.6
Calcite (CaCO ₃)	%	present (< 18%)	present (< 15%)	present (< 19%)	present (< 11%)	present (< 12%)
Silica (SiO ₂)	%	9.3	9.8	8.0	< 1	< 1
Aluminium oxide (Al ₂ O ₃)	%	< 1	< 1	< 1	< 1	< 1
Neoformed Halite (NaCl)	%	29	39	41	present	present
Neoformed Sylvite (KCl)	%	1.6	1.4	2.4	5.4	5.6
Gypsum (CaSO ₄ · 2H ₂ O)			traces	traces		
Celestine (SrSO ₄)	%	1.0	0.61	0.93	0.69	0.70
Barite (BaSO ₄)	%	1.4	0.24	0.34	0.02	0.02
Sulphide (S)	%	0.32	0.08	0.07	0	0.03
Sphalerite (ZnS)	%	0.17	0.03	0.04		
Galena (PbS)	%	0.23	0.04	0.04		
Manganese oxide (MnO)	%	0.36	0.32	0.28	0.02	0.02
Titanium oxide (TiO ₂)	%	0.03	< 0.01	0.01	< 0.01	< 0.01
Phosphore oxide (P ₂ O ₅)	%	0.13	0.04	0.06	< 0.01	< 0.01
Arsenic (As)	%	0.23	0.07	0.13		

n.a.: not analyzed, TSS: Total Suspended Solids, ⁽¹⁾ Sample dried at 40°C before analyses, ⁽²⁾ Sample dried at 105°C before analyses

Table 8 - Geochemical monitoring of the fluids discharged from GPK-3 and GPK-4 during the production tests carried out between February 26 and 27, 2007, and between March 5 and 9, 2007 (all the chemical analyses were performed by BRGM, except free NTA which was analyzed in the EGI laboratory, Utah University).

Well	Date	Discharged volume (m ³)	Specific gravity (GCE)	Weight of 20 ml (g)	Specific gravity (BRGM)	T _{amb} (°C)	pH	Cond. (mS/cm)	Cond. 25°C (mS/cm)	En (mV)	E _h (mV)	Diss. O ₂ (%)	Na (mg/l)	K (mg/l)	Ca (mg/l)	Mg (mg/l)	Cl (mg/l)	Alk. (meq/l)	TC (mmol/l)	TC (mg/l)	TOC (mmol/l)	TOC (mg/l)	NTA (µmol/l)	chlato (mg/l)	chlato (CaH ₂ O ₂) (mg/l)	Fe _{tot} (mg/l)	F (mg/l)	Br (mg/l)	B (mg/l)	Fe (mg/l)	Fluo. 1,5 nds (µg/l)	Fluo. 2,7 nds (µg/l)	1,6 nds (µg/l)	2,6 nds (µg/l)	Field colour	Comments			
GPK-3	26/02/2007 09:15	100	1.01	49.81	0.996	20.0	7.61	2.7	165	281	165	43.5	7046	759	1653	121	683	1.73	739	61.50	25.7	2.14	712	59.33	144	0.87	15.5	65.6	96.7	117	23.2	<5	<11	96.5	19.1	brown green			
	26/02/2007 11:30	300	1.019	50.69	1.014	21.4	7.23	32.9	374	188	19	0	7046	759	1653	121	683	1.73	739	61.50	25.7	2.14	712	59.33	144	0.88	16.1	65.6	96.7	117	23.2	<5	<11	96.5	18.1	yellow			
	26/02/2007 13:25	500	1.021	50.7	1.016	22.3	7.29	35.5	374	188	19	0	7046	759	1653	121	683	1.73	739	61.50	25.7	2.14	712	59.33	144	0.88	16.0	65.6	96.7	117	23.2	<5	<11	96.5	18.1	yellow			
	26/02/2007 15:00	700	1.021	50.78	1.016	24.2	7.26	37.8	387	211	8	0	7046	759	1653	121	683	1.73	739	61.50	25.7	2.14	712	59.33	144	0.88	17.9	65.6	96.7	117	23.2	<5	<11	96.5	18.1	yellow			
	26/02/2007 16:40	880	1.010	50.68	1.013	24.2	7.26	38.1	387	211	8	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow			
	26/02/2007 17:12	880	1.010	50.68	1.013	24.2	7.26	38.1	387	211	8	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow			
	26/02/2007 18:35	900	1.010	50.76	1.015	22.0	7.23	36.8	383	173	31	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow			
	26/02/2007 19:10	980	1.016	50.78	1.016	22.0	7.23	36.8	383	173	31	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow			
	26/02/2007 20:00	1000	1.017	50.80	1.017	20.3	7.3	36.2	41	18	151	65	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow		
	26/02/2007 20:45	1060	1.017	50.80	1.017	20.3	7.3	36.2	41	18	151	65	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow		
	26/02/2007 21:30	1100	1.021	50.81	1.017	21.4	7.36	38.1	410	184	112	0	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow		
	26/02/2007 22:14	1150	1.021	50.85	1.017	21.8	7.12	37.8	402	126	79	0	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow		
	26/02/2007 23:00	1200	1.021	50.89	1.014	22.1	7.10	38.5	407	183	42	0	0	8616	917	2041	90.7	16742	6.31	599	49.08	22.4	1.97	566	47.17	105	1.67	17.5	16.0	79.8	86.5	24.8	<5	<11	96.5	25.5	yellow		
	26/02/2007 00:30	1300	1.023	50.88	1.018	22.9	7.43	38.1	408	179	56	0	0	8616	948	2137	85.9	17500	6.34	534	44.50	21.6	1.86	512	42.97	112	1.78	15.0	62.7	71.9	76.4	26.3	<5	<11	118	19.6	yellow	white deposits at the bottom of the flask	
	26/02/2007 01:46	1360	1.023	50.84	1.017	22.5	7.45	38.5	412	184	56	0	0	8616	948	2137	85.9	17500	6.34	534	44.50	21.6	1.86	512	42.97	112	1.78	15.0	62.7	71.9	76.4	26.3	<5	<11	118	19.6	yellow		
GPK-4	26/02/2007 02:30	1400	1.023	50.77	1.015	22.8	6.96	39.5	401	106	98	0	0	8616	921	1983	147	16940	6.04	1436	119.59	16.0	1.50	1417	118.08	nd	0.88	23.0	89.4	95.5	366	<0.6	<0.1	3.5	9.5	136			
	26/02/2007 03:30	1500	1.023	50.77	1.015	22.8	6.96	39.5	401	106	98	0	0	8616	921	1983	147	16940	6.04	1436	119.59	16.0	1.50	1417	118.08	nd	0.88	23.0	89.4	95.5	366	<0.6	<0.1	3.5	9.5	136			
	26/02/2007 04:15	1600	1.023	50.83	1.017	24.0	7.17	39.6	400	132	71	0	0	8616	981	2198	184.7	18177	6.49	505	42.08	17.5	1.46	467	40.58	125	1.99	16.6	15.0	85.2	88.6	66.3	25.5	<5	<11	115	25.5	yellow	
	26/02/2007 05:05	1650	1.023	50.94	1.019	20.5	7.21	38.9	424	153	53	0	0	8616	981	2198	184.7	18177	6.49	505	42.08	17.5	1.46	467	40.58	125	1.99	16.6	15.0	85.2	88.6	66.3	25.5	<5	<11	115	25.5	yellow	
	26/02/2007 05:50	1700	1.023	50.95	1.019	20.5	7.21	38.9	424	153	53	0	0	8616	981	2198	184.7	18177	6.49	505	42.08	17.5	1.46	467	40.58	125	1.99	16.6	15.0	85.2	88.6	66.3	25.5	<5	<11	115	25.5	yellow	
	26/02/2007 06:50	1800	1.025	50.97	1.019	20.7	7.26	40.1	435	161	45	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 07:45	1700	1.025	50.96	1.017	23.4	7.36	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 08:44	1750	1.025	50.96	1.019	23.4	7.27	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 09:30	1800	1.025	50.96	1.019	23.4	7.27	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 10:30	1800	1.025	50.97	1.019	23.4	7.26	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 11:20	1800	1.025	50.97	1.019	23.4	7.26	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 12:00	1800	1.025	50.97	1.019	23.4	7.26	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 13:00	1800	1.025	50.97	1.019	23.4	7.26	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	26.6	<5	<11	122	23.7	yellow	
	26/02/2007 14:00	1800	1.025	50.97	1.019	23.4	7.26	40.3	415	147	57	0	0	8616	987	2208	82.6	18291	5.36	477	39.75	17.8	1.48	460	38.33	162	2.57	17.0	14.5	85.9	65.5	61.3	2						