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*Technical Article*

# Reverse Oxidation Zoning in Mine Tailings Generating Arsenic-rich Acidic Waters (Carnoulès, France)

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**Abstract.** The Carnoulès Pb mine closed 40 years ago, leaving tailings (pyrite-rich silts) exposed. In 1982, the tailings were gathered and placed behind a concrete dam, above a drainage pipe, and then covered with a clay layer. The As-rich pyrite in the tailings has oxidized; acidic water with high As concentrations (100-350 mg/L As) now emerges from the base of the dam. Strikingly, there is no oxidation zone at the surface of the tailings. The clay cover and the low hydraulic conductivity of most of the tailings ($10^{-7}$ m/s) strongly limit rainwater infiltration. Water table variations, water balance calculations, and flow modelling indicate subsurface water input and water flow along the bedrock within a more permeable sandy horizon. This lower horizon is strongly oxidized due to this flow pattern. The As-rich water is mainly produced in the northern part of the tailings area, where seasonal variations in the water table (a succession of aerobic/anaerobic periods) are important. This water flows through a drainage pipe to an acidic spring.

The disposal of tailings can release acidic and toxic waters into the environment. Monitoring and modelling of such tailings allow one to define the potential impact and propose remedies.

**Key words:** Arsenic, modelling, oxidation, tailings, water, zoning

## Introduction

Sulphide-rich ores are commonly exploited for their high metal contents. Metal-bearing sulphides are recovered by grinding and floatation processes; the resulting residual material (tailings) is very fine grained and can contain high levels of sulphides. Tailings are generally discharged as slurries and stocked behind containment dams. At the air-water contact, the sulphide minerals in the tailings are quickly oxidised, often generating acidic drainage and high concentrations of potentially toxic metals. Oxidation of sulphide minerals generally occurs in the unsaturated zone, where oxygen diffuses downwards through the tailings (Blowes and Jambor 1990). The presence of very fine-grained material reduces tailings permeability and oxidation; conversely, coarser horizons allow the circulation of sub-surface waters and promote oxidation of the tailings along specific layers (Robertson 1994). Generally there is no sulphide oxidation in the underlying saturated zone because of the very low diffusion of oxygen in water. In the framework of this pattern, a "dry" or "wet" cover is often used as an oxygen diffusion barrier at the surface of the tailings to avoid the formation of acid mine drainage.

This paper describes and discusses a case where oxidation does not occur in the unsaturated zone but along the bottom of the impoundment, producing acidic waters with extremely high arsenic concentrations. The objective is to understand the hydrogeological and geochemical processes that control the water flow and composition in order to propose a remediation strategy.

## Site Description

Carnoulès is a relatively important stratabound Pb-(Zn) orebody in the Pb-Zn belt of the southern border of the Massif Central, France (2.5 Mt containing 3.5% Pb and 0.8% Zn). Lower Triassic conglomerates, unconformably covering the Palaeozoic basement, host the Carnoulès mineralization: a pyrite-galena (sphalerite)-barite mineral association (Alkaaby et al. 1985). The Carnoulès orebody was mainly worked as an opencast operation and was abandoned in 1962, leaving 1 $km^2$ of quarries and 3 different tailings areas along the 1.5 km long glen of Reigous Creek (Figure 1). Fourteen years after mine closure, much of the tailings spilled down gradient for kilometres along the Amous Valley. In 1982, the remaining tailings and the recovered part of the spilled material were gathered together and stored behind a concrete dam built upon the uppermost course of Reigous Creek. The basement rocks are moderately dipping quartzite layers that are only crosscut by a few faults. The tailings (1.2 Mt, 6-20 m thick, and covering 54,375 $m^2$) have a sub-horizontal upper surface, and slope downwards at 20%. The total height gradient between the upper horizontal surface and the foot of the dam is

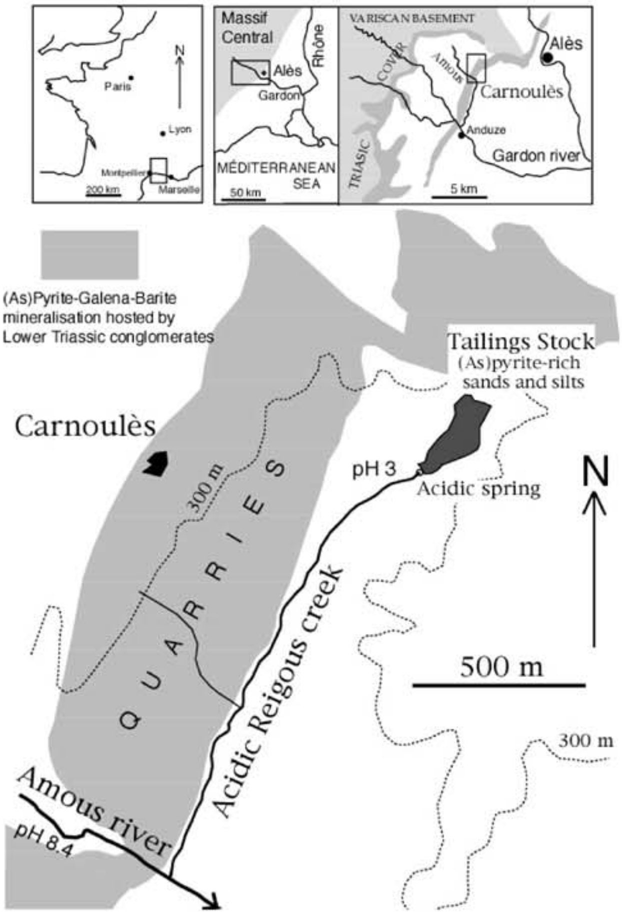


**Figure 1.** Location and sketch map of the Carnoulès mining site

35 m. A thin clay cover (30 cm thick) mitigates rainwater penetration and has allowed the spontaneous establishment of a vegetal association comprising *Pinus maritinus*, heathers, and mosses.

The tailings are mainly composed of very fine-grained material (30 µm on average) and generally display a sub-horizontal layering. They correspond to sulphide-bearing grey silts. Quartz is the main mineral component, with K-feldspar and biotite as minor phases. The tailings also contain 5-10 wt % of arsenic-rich pyrite (2-4 % As) with accessory galena and barite. The grain size and composition is generally homogeneous throughout the silt layer. Along the basal surface, there is a 1-3 m thick white to orange layer of sand (200 µm on average).

The surrounding runoff waters are collected by concrete ditches running along the borders of the tailings upper surface. They flow together into a small shaft, and then down-gradient to Reigous Creek through a concrete pipe crosscutting the tailings. Rainwater is collected by the same system along the upper surface of the tailings; sub-horizontal ditches drain rainwater laterally across the dipping face of the tailings deposit (Figure 2).

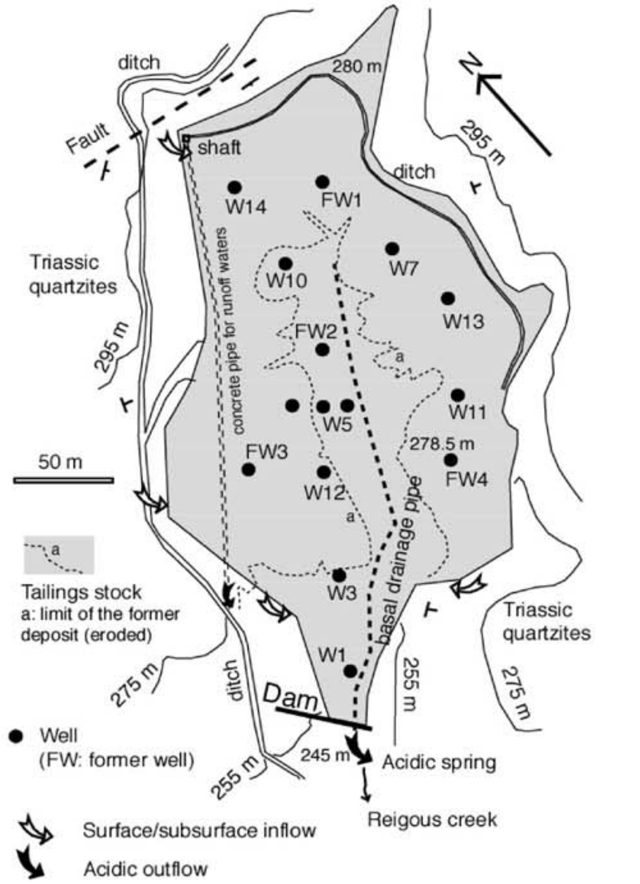


**Figure 2.** Map of the Carnoulès tailings deposit, showing the two stage history of the tailings, the location of the wells, the drainage system (ditches and pipes), and the inflow and outflow waters

The tailings were placed on the uppermost part of Reigous Creek and probably buried the former natural springs. Mining records give information on the presence of old concrete pipes built along the axis of the basal surface of the tailings to drain the creek and spring waters. Acidic waters (pH 3.3 on average) now emerge from the base of the tailings and constitute the initial source of Reigous Creek. The water has high concentrations of sulphate, iron, and arsenic (2000-7500, 750-2700 and 80-350 mg/L respectively). The temperature of the water is relatively stable all year (14-16°C), confirming an underground source. The annual discharge at this spring is 0.65L/sec, on average, with the variations (0.2-1.3) being seasonal and dependent on rainfall. The average annual rainfall is 1100 mm with a seasonal distribution that is typical of the Mediterranean climate, including long drought periods and spring and autumn rainstorms.

A few meters downstream of the spring, the redox potential and the $O_2$ concentration strongly increase. Simultaneously, As concentrations decrease from 200 to 8 mg/L (average values), as an Fe-As-rich material (up to 22% As) precipitates, covering the streambed. These sediments have been described as bacterial mats and result from the bio-oxidation and precipitation of soluble iron and arsenic (Leblanc et al. 1996, 2001).

## Methods

### Monitoring

Fifteen observation wells (5-18 m in depth) in the tailings allows the piezometric level to be monitored. They also permit injection/pumping tests, geochemical and microbiological analysis, and variations in the main physicochemical parameters in the water (pH, Eh, temperature, conductivity) to be tracked. Topography was checked by GPS levelling. This close network of wells was designed to optimise the recording of the water level and water sampling. Most of the wells have been drilled down to the bedrock of the tailings; some of them stopped in the grey silts (Figure 3). The boreholes are cased with slotted PVC screens, 5 or 10 cm in diameter, allowing water to enter throughout the saturated thickness of the aquifer; a geotextile screen and a gravel rim prevent the intrusion of the fine-grained material. The deepest well (W5), located in the central part of the tailings deposit, has been equipped to automatically monitor both rainfall (overturning rain recording gauge) and water level (every 30 minutes); atmospheric temperature and barometry are also recorded. Other wells are equipped with autonomous pressure gauges to record water level and temperature. Finally, a mobile self-contained data logger has been used to record variations in pH or redox potential at different locations relative to rain events. All these parameters were downloaded onto a portable computer, which allows visualisation and processing of the data in the field. In addition, piezometric data were regularly checked by hand measurements (electric piezometer).

At the beginning of the study, permeability was estimated from the grain size of the different tailings samples (measured from sieve separations and SEM observations). Then in-situ hydraulic tests, such as slug and pumping tests, were carried out in order to calculate the hydraulic conductivity and the storage coefficient values more accurately. In addition, infiltration tests were conducted both on the clay cover and on tailings outcrops, using a double-ring system conducted with a constant head (Sharma et al. 1980). Tracing the water paths within the tailings was unsuccessful due to the instability of the fluorescent dyes in acidic waters; other tracers will be tested.

The Reigous Creek spring is the main outlet of the tailings. Its discharge was monitored hourly using an electromagnetic flow meter to minimise the effect of

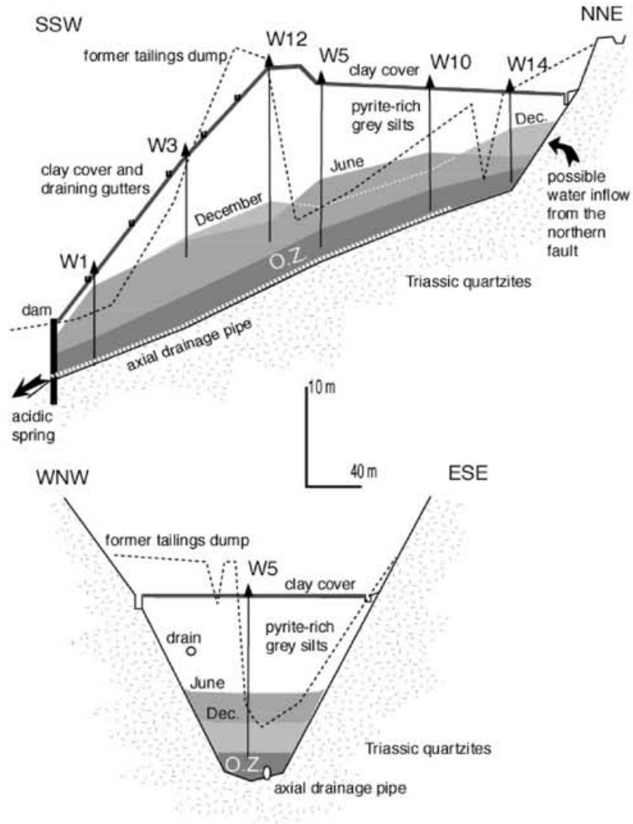


**Figure 3**. Cross-sections of the Carnoulès tailings deposit; starting from the surface, there are 3 horizons: a thin clay cover, hydraulic conductivity (cond.) = $10^{-7}$-$10^{-8}$ m/s, a pyrite-rich grey silt horizon, cond.=$10^{-7}$-$10^{-8}$ m/s, and a more permeable, strongly oxidized sand horizon, cond.=$10^{-5}$-$10^{-6}$ m/s (O.Z. = oxidized zone). The Triassic quartzites of the bedrock are assumed to have low permeability, cond.=$10^{-12}$ m/s. Water level variations are given for December 2001 and June 2002.

sediment precipitation and mobile bit coating. For the same reason and because of the aggressive nature of the fluid, delrin-coated toroidal-sensing-technology was used to monitor electrical conductivity. Temperature, pH, and Eh were also recorded at the outlet, together with pH 30 m downstream. All the probes were controlled by a programmable acquisition-system powered by 12 v sealed batteries, which gives up to 6 months of autonomy.

### Hydrodynamic modelling

A general model of the system in a permanent or transitional state has been developed to assess the hydraulic gradients, the flow lines of the tailings aquifer, and the water-balance. We used the modular finite-difference groundwater flow model, MODFLOW (McDonald and Harbaugh 1988; Harbaugh and McDonald 1996a, b), which is a program for simulating confined or unconfined, saturated flow in one, two, or three dimensions. It allows both steady state and transient simulations. Modflow is probably the most frequently used groundwater modelling program in the public domain (Winston 1999).

The main characteristics of our application were:

(1) The presence of two layers (a main layer of grey silt and a lower sand layer), as shown by the drilling logs.

(2) Both layers were considered to be anisotropic and homogeneous. Their hydraulic conductivities (average values from in-situ tests) are $K_{v1}=10^{-8}$m/s, $K_{v2}=10^{-6}$m/s, $K_{h1}=10^{-7}$m/s, and $K_{h2}=10^{-5}$m/s.

(3) The basement (unfractured Triassic quartzites) of the tailings was considered to be impermeable. The basement topography has been reconstructed from mining records, topographic and drillings observations, and geophysical exploration.

(4) The presence of a pipe draining the tailings stock from its upper part (Figures 2 and 3) to the spring. The conductance of the drain was defined during the calibration ($C = 3.5 \cdot 10^{-3}$ $m^2/s$). The discharge of the system corresponds to the acidic spring.

(5) The recharge is derived from both rainfall infiltration on the tailings and lateral subsurface inflow from the surrounding aquifer. Infiltration was estimated using the Thornwhaite formula (de Marsily 1981). The flow rate of the aquifer input, which remains unknown, is a parameter of calibration.

(6) April 2001 was chosen as a reference month for low rainfall (33 mm).

(7) The size of the grid cells was 20m x 10m.

The model was calibrated to match the recorded water levels at every borehole. Initially, the hydraulic conductivity of the drain was calibrated from piezometric data obtained during the low water level period. The storage coefficient was then calibrated with data corresponding to high levels. The boundaries of the system and its working cells were carefully defined before the simulations.

Water sampling and analysis

Most of the wells were sampled once a month, the spring and the main well (W5) at least twice a month. The main physicochemical parameters (conductivity, Eh, pH, temperature, dissolved oxygen (DO)) were measured using an Ultrameter™ Model 6P (Myron L Co., Camlab, Cambridge). DO was also determined with CHEMets tests (CHEMetrics, Calverton, USA) based on colorimetric detection. Water samples were collected in polyethylene bottles and filtered immediately through 0.45µm Millipore membranes. Fe(II) was determined immediately after filtration. Samples for total Fe and As determination were acidified with $HNO_3$ and stored at 4°C.

Samples for total Fe and As determination were acidified to pH=1 with $HNO_3$ (14.5M) and stored at 4°C in polyethylene bottles until analysis. The samples for Fe and As speciation and $SO_4$ determination were done within 24 hours. The total dissolved As was performed by inductively coupled plasma-mass spectrometry (ICP-MS). The elevated As concentrations necessitated a dilution factor of 1000, and no interferences due to ArCl was detected. Calibration of the ICP-MS was done by calibrating peak intensity, acquired in peak jump mode, with standard solutions. $^{115}$In was used as an internal standard to correct for instrumental drift.

For dissolved As(III) determination, the pH was adjusted to 4.8 using an acetic acid-sodium acetate buffer. Arsenic was determined using a hydride generation system coupled to the ICP-MS; this involves reaction of As(III) with sodium borohydride. The detection limit is 75 ng/L, with an accuracy better than ±5%. Dissolved As(V) was calculated as the difference between total dissolved As and As(III).

For Fe(II) determinations, filtered samples were buffered to pH 4.5 with an ammonium acetate/acetic acid buffer in the field, and Fe(II) was complexed by adding 1 ml of a 0.5% (w/w) phenantroline solution to 10 ml of sample. Analyses were undertaken by colorimetry at 510 nm. The detection limit is $2.10^{-4}$ mol.$L^{-1}$ and the precision is better than ±5%. Total dissolved Fe was determined by Flame Atomic Absorption Spectrometry.

Sulfate was determined by precipitation of $BaSO_4$ with $BaCl_2$ and spectrophotometric measurement at 650 nm.

Samples of tailings were air-dried, then mineralised using 20 ml of concentrated $HNO_3$ before analysis for total As, Fe, and $SO_4$. Tailings and bacterial sediments were examined by scanning electronic microscopy coupled with energy dispersive X-ray spectrometry.

## Results

Tailings aquifer

Every piezometric record shows that the water table comprises a northern upper flat part, with a slight central hollow and a downstream slope (Figure 4A). Considering a longitudinal section of the tailings stock (Figure 3), the water table roughly follows the basement topography, which corresponds to the upper part of Reigous Glen. In the upper, flat part of the tailings deposit, the thickness of the aquifer varies from 2 to 8 m, depending on seasonal variations and on well location. In the downstream, sloping part, the aquifer thickness remains more constant, and increases slightly against the barrier of the dam (W1). The hydraulic gradients, estimated from the hydrostatic level curves, vary from 1% upstream to 15% along the steep downstream slope of the tailings stock. Depending on hydraulic gradients (Figure 4B), the flow lines first turn towards a hollow, corresponding to the beginning of the drainage pipe, then diverge downstream in the wider part of the tailings stock before converging near the dam.

The values of hydraulic conductivity, obtained from slug tests, vary between $10^{-7}$ and $10^{-8}$ m/s for the main layer of grey silt. Infiltration tests on the fine-grained tailings also indicated very low conductivities, as low as $3.2 \times 10^{-7}$ m/s. Pumping tests at the bottom of the W5 well gave similar values for the storage coefficient, but higher values for conductivity ($1.2 \times 10^{-5}$ m/s) due to the presence of coarser material (sand) at the base of the tailings. These results are in agreement with the permeability ascribed to sand and silt materials, from $10^{-5}$ to $10^{-9}$ m/s (Domenico and Schwartz 1990). The clay cover of the tailings also displayed low hydraulic conductivity ($10^{-7}$-$10^{-8}$ m/s). The water level variations simulated by the hydrodynamic model using these values of hydraulic conductivity and storage coefficient can be compared with the observed variations (Figure 3).

Seasonal variations

A few wells (W1, W3, and W14) show a clear relationship between water level and rainfall: after rainy periods, the water table rises quickly to its maximum; after drought periods, it goes down progressively to very low levels. Actually, most of the wells show a delay of several weeks between rainfall and water level. However, the water variations of the central well (W5) apparently have no relationship with rainfall; in fact, it seems that there is a gap of up to 4 months between a rainy period and a water table rise. In this respect, Figure 5 clearly shows how water slowly migrates southwards and downwards (Figure 3), from W14 to W10 to W5; there is a 50 m gap between the first two and a 75 m gap between W10 and W5. After a rainy period, the maximum water level was observed in 1, 2, and 4 months for W14, W10, and W5 respectively; this corresponds roughly to a hydraulic conductivity of $1.5\ 10^{-5}$ m/s. Modelling correctly reproduces this transitory flow (Figure 4).

Water characteristics

Temperature is very stable (14-15°C) whereas the other physicochemical parameters display strong spatial and seasonal variations. Nevertheless, considering their average values, three different hydrologic sections, or blocks, are evident (Figure 6):

Block A, located just above the dam, represents a small volume of slightly acidic waters (pH 5.4). Total As concentrations are < 1 mg/L with up to 90% As

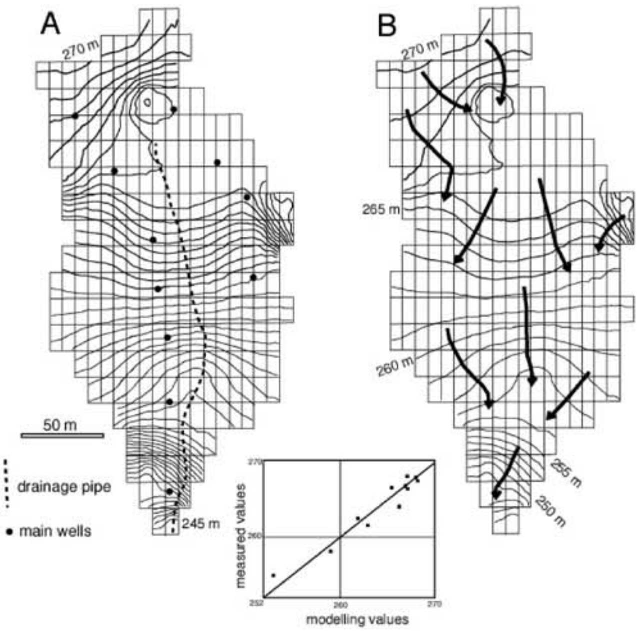


**Figure 4.** Modelling of the water table (**A**) and the water flow (**B**) for a dry period (April 2001)

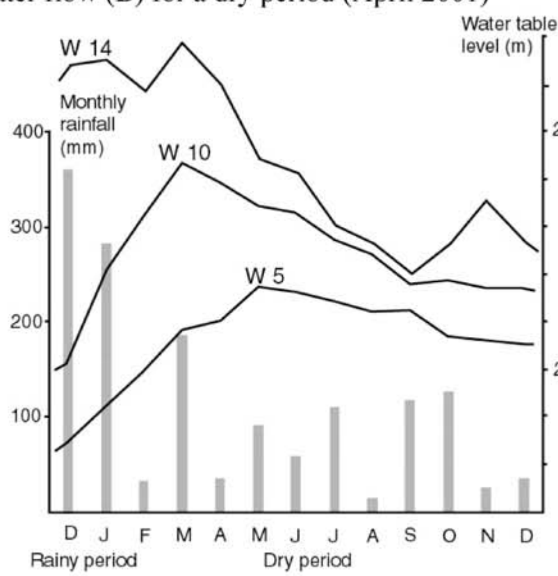


**Figure 5.** Seasonal variations of the water table level for wells W14, W10, and W5, showing the slow southwards migration of the water after a rainy period

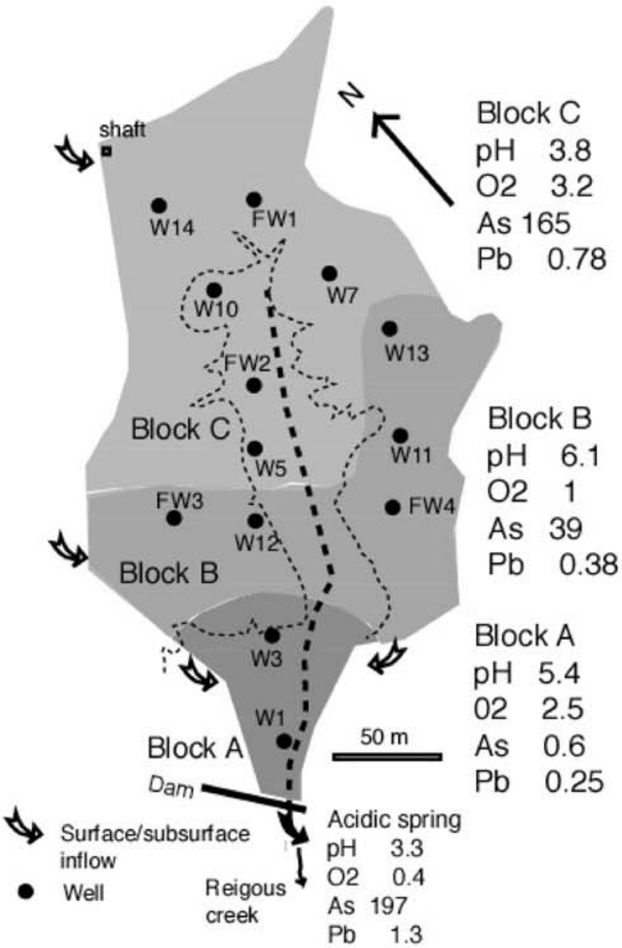


**Figure 6.** Spatial variation of water composition within the tailings stock; block C waters are also characterised by strong seasonal variations corresponding to hydrogeochemical reactions. Resulting acidic and As-rich waters probably flow to the acidic spring through the drainage pipe.

(V); DO is relatively high (2.5 mg/L) and conductivity relatively low (<2,000 µS/cm).

Block B is characterised by less acidic waters (pH 6.1) and negative Eh (-45 to –61 mV) with low DO (0.4-1 mg/L) and high conductivity (4,500 µS/cm). Total As concentrations are 39 mg/L with less than 15% As (V).

Block C, the more important central and northern section, is characterised by very strong seasonal variations. Referring to the W5 well: pH varies from 1.2-5.8; Eh from –80-+400 mV; conductivity from 6000-53,100 µS/cm; DO from 0.01-9 mg/L; total As from 250-12,150 mg/L with 1.5 to 100% As (III); total Fe from 2100-20,400 mg/L with 32-100% Fe (II), and $SO_4$ concentrations from 4200-42160 mg/L. The highest As, Fe, and $SO_4$ concentrations correspond to the lowest water levels, with high DO and low pH. Obviously, water block C is an active system. The characteristics of the spring water are clearly different from the A and B waters; conversely, they are quite similar to those of block C, and more precisely to wells W5-W7, suggesting a direct relationship between the acidic spring and this area of the tailings.

## Discussion

### Water balance

In a steady state model, the chosen parameters are constrained to obtain a good water balance on a yearly basis. Most of the parameters are well known (rainfall, tailings and cover hydraulic conductivities, water output); drainage pipe conductance and, evapotranspiration were calculated during calibration. Possible water input from fault zones or from seasonal surface runoffs cannot be precluded (Figure 2). Rainfall is the more obvious water input. Referring to the tailings deposit surface (54,375 $m^2$), about 60,000 $m^3$ of rainwater fall on the tailings annually. Nevertheless, rainwater can hardly enter the tailings. Along the dipping face, the strong slope (20%) and the drainage ditches don't allow much infiltration; on the flat surface (2/3 of the total tailings surface), the low permeability of the clay cover ($10^{-7}$-$10^{-8}$ m/s) strongly limits infiltration. Most of the rainwater stays on the surface to be quickly evaporated; furthermore, water that penetrates will be used by the plants growing on the cover. Evapotranspiration is obviously more important than previously calculated (30%) from the Thornwaite formula, which only considers temperature, and not wind (de Marsily 1981).

Calibrating this sensitive parameter, we obtain an evapotranspiration rate greater than 50%, with perfect concordance of the model with the observed evolution of the water table. Nevertheless, the water balance implies a water input of 6.2 $10^{-2}$ L/sec in the tailings. Considering the hydrogeological background (Figure 2), water table variations (Figure 5), and the mining records, this input probably corresponds to a buried spring in the northern part of the tailings area.

### Water circulation

Taking into consideration the low hydraulic conductivity ($10^{-7}$ m/s) of most of the tailings material (silts) and its clay cover ($10^{-8}$ m/s), the downwards penetration of rainwater should be very slow, taking 1 to 3 months to reach the water table.

Referring to hydraulic gradients in the tailings aquifer, the main flow is roughly horizontal, running from north to south and parallel to the quartzite bedrock. The presence of a lowermost horizon of coarser sand allows a circulation velocity of about 1.3

m/d. In the main part of the aquifer (block C), rainfall events first triggers increased water levels in the uppermost part (well 14), suggesting a possible input of surface and/or subsurface waters in this area (probably related to the fault zone, Figure 2). Blocks A and B seem to evolve in a different way. Block A is directly linked to rainfall events, suggesting a local and relatively quick input of surface water (Figure 2); in this small reservoir with high hydraulic gradients (Figure 4A) and high water levels, no efficient oxidizing reactions will occur. Block B waters are also clearly distinct from the block C waters, suggesting the presence of impermeable barriers. These barriers may correspond to ferric hydroxide-coated surfaces developed during the multistage history of the tailings stock (Figures 2, 3).

Block C waters are similar in composition to those of the spring, and are likely quickly transported there through the axial drainage pipe. Although located above the pipe that collects the block C waters, blocks A and B waters may also be drained and mixed with block C waters during their transfer to the acidic spring.

Role of oxygen

The water composition and physico-chemical characteristics generally change in the main (up-gradient) part of the tailings. For example, the seasonal variations of well W5 show a contrasting evolution (Figures 3 and 5). Stage 1 corresponds to low levels of the water table, and is characterised by high DO (8-9 mg/L) and low pH (<2). Stage 2 starts as the water table quickly rises (up to 2 m), has low DO (<1 mg/L) and higher pH (5-6). Interestingly, during the first stage, total arsenic levels are extremely high ($10^3$ mg/L As), mostly in the As (V) form, whereas during the second stage, total arsenic concentrations are lower (300-700 mg/L) and most of the arsenic is in the reduced form (As (III)). In the same way, the abundance of Fe (II) relative to total dissolved Fe varies from 10%, during the first stage to 100% during the second stage. This behaviour corresponds to that of a two-stage biohydrogeochemical reactor with a succession of aerobic and anaerobic stages. During stage 1, the input of oxygen-rich waters (6 mg/L) and the increase of the water table promote the oxidation of pyrite and the formation of strongly acidic waters with high As-Fe concentrations. In water leaching experiments, slightly oxidised pyrite-bearing tailings containing small amounts of Fe-sulphates and a low water/sand ratio (<1) generate a strongly acidic leachate (pH 1.2) with high arsenic content (3.3 $10^3$ mg/L As), mainly in the As (V) form. During stage 2, high water levels and low oxygen contents promote a decrease in pH and the formation and precipitation of secondary Fe-As oxidized phases.

Based on drill cores and remnant outcrops of former tailings deposits, there is an oxidized and more permeable zone along the bottom of the tailings. This oxidized zone, a few meters thick, consists of yellow and ferruginous sands that are directly overlain by unweathered pyrite-bearing grey silts. The quartz and K-feldspar detrital grains of the yellow sand are strongly coated, and even cemented, with poorly crystallised As-rich iron hydroxides (2-10% As). The oxidized zone, which contains 0.5-0.7% As, twice the average value of the overlying unweathered tailings, is obviously a strongly reactive zone.

**Conclusions**

The Carnoulès tailings stock shows an uncommon vertical zoning. The low permeability ($10^{-7}$ m/s) of the Carnoulès tailings and the presence of a clay cover prevent rainwater penetration and weathering of the pyrite-rich tailings. However, an oxidized horizon is present at the base of the tailings where a sandy layer allows water to flow parallel to the bedrock.

Depending on water level variations, an efficient oxic-anoxic reaction system produces acidic waters with high As contents.

Tailings deposits must be designed to account for possible subsurface water-input; moreover, strong hydraulic gradients will generate a draining system with horizontal water flows. In the case of Carnoulès, a remedial solution would be to divert or intercept the subsurface water input.

**Acknowledgements**

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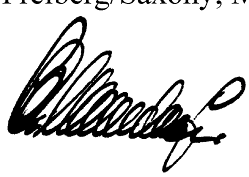
## International Mine Water Association – Executive Council Meeting

On October 22nd 2003 IMWA's Executive Council will hold its annual meeting in Johannesburg/South Africa. All members of the EC are encouraged to take part. If an EC member has any contribution he wishes to make and cannot attend the meeting, then please send your comments to President Peet NEL or the Secretary General Christian WOLKERSDORFER so that it can be read out at the meeting. Comments must arrive by September 22nd 2003.

***Agenda***

1. Present
2. Apologies
3. Previous Minutes
4. President's report (Peet Nel)
5. Secretary's report (Christian Wolkersdorfer)
6. Treasurer's report (Adrian Brown)
7. Editor-in-Chief's report (Bob Kleinmann)
8. 2003 Congress Report (Peet Nel)
9. Symposium 2004, Symposium 2005
10. IMWA's corporate design
11. IMWA statutes and by-laws
12. Any other Competent Business

Freiberg/Saxony, March 16th 2003



Christian Wolkersdorfer
Secretary General