Characterisation of coated sand from iron removal plants

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Abstract Water treatment plants removing iron from groundwater are commonly expected to operate in oxidation-floc formation mode. However, there are indications that adsorptive iron removal plays a role as well. Adsorptive iron removal has several potential advantages over oxidation-floc formation iron removal. Therefore, the physical and surface chemical characteristics of coated sand from twelve different groundwater treatment plants in the Netherlands were analysed to assess their potential use in adsorptive iron removal. Specific surface areas, amounts of iron, manganese, calcium and TOC in the coatings and iron(II) adsorption capacities of the coated media were measured. Additionally, grain size distribution and density of the media were determined and X-ray diffractograms, scanning electron micrographs and EDAX spectra were prepared. Compared to new sand, coated sand had a very high porosity and a very large specific surface area. The surface extractable iron content of the coated sand increased with iron loading.

The iron content of the coatings ranged from 27% to 45%. At pH 6.5, the iron(II) adsorption capacities of different coated sands were 10 to 55 times that of new sand. In general, iron(II) adsorption capacities of the coated sand from different plants increased with the increase in the time in use and the iron content of the coating. However, the average annual increase of iron content of the coatings and the iron adsorption capacity were different for the coated sands from different plants, probably due to the difference in water quality, process conditions applied and time in use. The grain size of the filter sand increased and their density decreased with the development of the coating. The decrease in density was a function of the increase in the effective grain size. EDAX analysis showed that iron and oxygen were the main elements in the coating followed by manganese, calcium, silicon and carbon. All the coatings analysed were X-ray amorphous or poorly crystalline. The measured high adsorption capacities of coated sand from wet filters and dry filters of full-scale groundwater treatment plants indicate that, in wet filters, adsorptive iron removal also plays a role. In dry filters, this mechanism should be dominant due to a very short pre-oxidation time.

Process efficiencies of wet filters with iron oxide coated sand can be further improved by operating the filters predominantly in the adsorptive filtration mode.

Keywords Adsorption; groundwater; iron oxide coated sand; iron removal; surface characteristics

Introduction

Iron is commonly present in groundwater worldwide. The WHO recommended guideline value of iron in drinking water is 0.3 mg/l (WHO, 1996) and the EC directive has set a parametric value of 0.2 mg/l (EC, 1998). Conventionally iron is removed from groundwater by the process of aeration/oxidation followed by rapid sand filtration. It is commonly assumed in practice that this process is based on oxidation-floc formation mechanism for iron removal. Alternatively in adsorption-oxidation mechanism, iron in anoxic groundwater can be removed by the adsorption of iron(II) onto filter media. This adsorbed iron(II) subsequently undergoes oxidation in presence of oxygen thus creating a new surface for adsorption. Adsorptive iron removal has several potential advantages over the oxidation-floc formation iron removal namely longer filter run, better filtrate quality, less backwash water use and low volume of sludge produced.

Iron oxide coatings develop on the surface of the filter media used in iron removal plants with time. This indicates that in groundwater treatment plants adsorptive iron removal plays a role as well. Iron removal efficiencies are expected to increase with the
development such coatings (Stevenson, 1997) since these iron oxide coated media have high iron(II) adsorption capacity (Sharma et al., 1999). This high adsorption capacity can be utilised to improve the efficiency of the existing iron removal filters with iron oxide coated sand by changing the mode of operation from conventional oxidation-floc formation to adsorption-oxidation. However, iron oxide coating developed on the filter media under different water quality (e.g. pH, iron concentration, HCO₃⁻) and process conditions (e.g. time in use, pre-oxidation time, backwash conditions) may have different physical and chemical characteristics and hence different iron(II) adsorption capacities. No information is, however, available on the elemental composition of the coating, specific surface area and the adsorption properties of the iron oxide coated sand from iron removal plants. Furthermore, the role of these coatings in iron removal is not yet fully understood. Hence, the objective of this study was to analyse the physical and surface chemical characteristics of the iron oxide coated sand developed under different conditions in twelve full-scale groundwater treatment plants in the Netherlands to assess their potential use in adsorptive iron removal.

Theoretical background
Iron oxide coating development and its characteristics
Iron oxide coating development on filter media in iron removal plants is a natural process in which the coatings develop in situ within weeks to months on new (virgin) filter media during normal filter operation. The coatings thus developed consequently may reduce the filter ripening time after backwashing and improve the iron removal efficiency of a filter (O’Connor, 1971; Stevenson, 1997). Additionally, a broad range of commonly non-monitored (possibly unknown) improvements in the filtered water quality may result from the presence of such coatings as iron (hydr)oxide can adsorb various uncomplexed and complexed heavy metals, other inorganic anions and organic compounds (Bailey et al., 1992; Benjamin et al., 1996; Joshi and Chaudhuri, 1996; Chang et al., 1997; Korshin et al., 1997).

The physicochemical properties of the filter media, especially the specific surface area and pore size distribution, and the surface chemistry (type of functional groups present, nature of adsorption sites, point of zero charge, zeta potential and surface charge) have profound effects on both rate and capacity of adsorption (Weber, 1985). Furthermore, the physical and surface chemical characteristics of the filter media change with the development of the iron oxide coating. Specific surface area of the filter media increases with the development of coating (Chang et al., 1997; Lo et al., 1997). This is one of reasons why the adsorption capacity of iron oxide coated media is much higher than that of new media.

The iron (hydr)oxides formed under different water quality conditions may have different chemical composition and crystal structure with different specific surface areas, surface site densities and adsorption characteristics (Fasiska, 1967; Crosby et al., 1983). Secondly, iron (hydr)oxides age over time to form stable end products and different iron oxides can have different iron(II) adsorption capacities. Sung and Morgan (1980) reported that the initial products of iron(II) oxidation are lepidocrocite (γ-FeOOH) and amorphous FeOOH. These forms are unstable and age over time to form goethite (α-FeOOH).

Furthermore, water quality and the treatment schemes used in the plant may also have a significant effect on the nature of the coating developed on the sand grains. Iron oxide can adsorb a host of cations, anions (Dzombak and Morel, 1990; Benjamin et al., 1996) and water-borne humic substances (Tipping, 1981; Gu et al., 1995). The formation, morphology, transformation and adsorption capacity of iron (hydr)oxide is readily influenced or modified by the presence or adsorption of these “foreign” species (Cornell and Schwertmann, 1996). Stenkamp and Benjamin (1994) found that the surface characteristics...
of underlying sand could also have some effect on those of coated sand, specifically if the coating is thin or porous. Therefore, the surface characteristics of iron oxide coated filter media are physically and chemically heterogeneous and are expected to change with time.

**Change in grain size and density of the media**
The development of oxide coating increases the grain size (Galvin, 1982) and decreases the density (Prasad and Belsare, 1984) of the filter media. From practice, it is known that in several groundwater treatment plants there are substantial “losses” of filter media during backwashing. The extent of these losses varies considerably among different plants. These losses can be attributed to the growth of the filter media, which reduces the depth of supernatant and the free board in the filter. Differences in filter media losses among different plants indicate differences in the growth rate of the coating. Additionally, the media growth and consequent change in density of the grain may affect the head loss developments and the efficiency of filter backwashing. Ultimately, the filter media need to be cleaned or replaced. An understanding of the factors affecting the growth of filter media is useful to assess the frequency of cleaning or replacement of the filter media and to improve filter design by incorporating the expected changes in freeboard, depth of supernatant and backwashing.

**Materials and methods**
In order to study the physical and surface chemical characteristics of iron oxide coated sand developed under different water quality and process conditions, iron oxide coated sand from twelve groundwater treatment plants in the Netherlands were collected and the filter media coatings were analysed. The major quality parameters of the raw water treated at these water treatment plants (WTP) are presented in Appendix 1. Of the twelve plants, WTPs Helden, Lichtvoorde and Sellingen apply dry filtration, whereas all others apply wet filtration.

*Specific surface area and pore volume.* Specific surface areas of the filter media were measured using the Brunauer, Emmett and Teller (BET) nitrogen gas adsorption method (relative pressure range 0.05–0.25) with a Quantachrome Autosorb-6B gas adsorption analyser at 77 K. Before surface area measurement, the samples were vacuum treated for 40 hours at 60°C. Additionally, pore volumes of some selected samples were also determined from nitrogen adsorption isotherms.

*Chemical extraction.* Surface extractable iron, manganese, calcium and TOC contents of the media coatings were determined by chemical extraction. Replicate samples of about 2 to 4 grams of the coated sand were extracted in acidified hydroxylamine chloride (NH₂OH·HCl) at pH <2. Subsequently, the extracts were filtered and analysed by flame atomic absorption spectrometry (Perkin Elmer AAS 3110) and the amounts of iron, manganese and calcium in the coating per gram of the filter media were quantified (Standard Methods, 1995). The TOC content of the extract was measured using Model 700 TOC Analyser of O-I Corporation Texas, USA. The loss in weight of the media after extraction was determined to quantify the total amount of coating and the fraction of different elements in the coating.

*Measurement of iron(II) adsorption capacity.* Iron(II) adsorption capacities of the coated sand were measured by conducting a series of batch adsorption equilibrium experiments. The experimental set-up and the procedure were similar to that used in the earlier study (Sharma et al., 1999). Experiments were performed at a pH of 6.5 at room temperature (19
± 1°C) using HCO₃⁻–CO₂ buffer. Different amounts of 400 mg/l deoxygenated iron(II) stock solution were introduced into the closed reactor containing a known weight of coated sand in deoxygenated water. On equilibrium, the iron concentration in the reactor was measured (Standard Methods, 1995) and the iron(II) adsorbed onto the media was calculated from mass balance. The Freundlich isotherm was fitted to the adsorption equilibrium data and isotherm constants $K$ and $n$ were obtained. For comparison of the adsorption capacities of coated sand of different sizes, isotherm constant $K$ was recalculated in terms of amount of iron(II) adsorbed per unit geometric surface area of the filter sand.

Grain size and density. The grain size distribution and the dry density of the coated media were measured before and after the extraction of the coating to quantify the change in grain size distribution and density with the development of the coating (Degremont, 1991).

Media surface analysis. Powder X-ray diffractions (XRD) of the iron oxide coatings were conducted using Siemens D500 goniometer (anode CuKαkV-mA 45–30) to analyse chemical composition, mineral form and crystal structure of media coatings from different groundwater treatment plants. Scanning Electron Micrographs (SEMs) of the new and coated sand were prepared using JOEL Scanning Microscope (JSM-6400F) for the analysis of surface features and composition of the coatings. Filter media samples were air dried, glue mounted and gold coated by vacuum electric arc before scanning. Quantitative elemental analyses were performed with an Energy Dispersive Analysis of X-ray (EDAX) system attached to the SEM to determine the elemental compositions of the surface mineral deposits on the media. The operating conditions were as follows: live time 100 seconds, dead time 35 seconds, operating voltage 10–25 kV, area analysed 25 × 25 micrometre.

Results and discussion
Specific surface area and pore volume
The BET specific surface areas of the coated filter sand from different plants are presented in Table 1. These specific surface areas are 3,400–221,000 times larger than their nominal geometric surface area calculated from the sieve analysis data for the equivalent spherical grains. Measurements showed that the pore volume and specific surface area of the media increase considerably with the development of iron oxide coating. The coated sand from WTP Gilze had an average pore volume of $1.16 \times 10^{-2}$ cm³/g and BET surface area of 11.4 m²/g, whereas the new sand of the same size had an average pore volume of $0.13 \times 10^{-2}$ cm³/g and BET surface area of 1.0 m²/g. Among the filter media tested, coated sand

<table>
<thead>
<tr>
<th>Coated sand from water treatment plant</th>
<th>Time in use years</th>
<th>Grain size mm</th>
<th>Specific surface area m²/g</th>
<th>Isotherm constants</th>
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<tr>
<td>WTP1 Roosendaal, WNWB</td>
<td>0.6</td>
<td>1.4–2.0</td>
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<td>3.0</td>
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<td>36.8</td>
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<td>69.3</td>
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<tr>
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<tr>
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</tr>
<tr>
<td>WTP12 Seevingen, WGr</td>
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<td>2.0–4.0</td>
<td>108.0</td>
<td>136</td>
</tr>
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</table>
from WTP Noord Bargares had the highest specific surface area of 201 m²/g with an average pore volume of $14 \times 10^{-2}$ cm³/g. These results reveal that compared to the new media, the iron oxide coated media have a much higher porosity and larger specific surface area and, hence, are likely to have a higher iron(II) adsorption capacity.

**Chemical extraction**

The surface extractable iron content (SEIC) can be used as a relative measure of the amount of surface iron oxide coating present on the media. SEIC of the media coatings were in the range of 27% to 45%. The fractions of manganese, calcium and TOC in the coating ranged from 0.04% to 5.8%, 0.05% to 1.95% and 0.11% to 7.22%, respectively. This indicated a wide variation in the chemical composition of the coatings. The loss of weight of different coated sand tested on extraction of the coating ranged from 6% to 91%. This shows that the extent of media growth and, consequently, the thickness of the iron oxide coatings also varied widely.

Figure 1 shows the general trend that the SEIC of the coated sand was higher when the iron loading (time in use $\times$ raw water iron concentration) was higher. Higher SEIC in turn resulted in higher BET specific surface area of the coated sand. It was also found that the higher the manganese, calcium and TOC content of the raw water, the higher were their fractions in the coating.

**Iron(II) adsorption capacity**

The Freundlich isotherm constants $K$ and $n$ for iron(II) adsorption onto different coated sand at pH 6.5 are presented in Table 1. The iron(II) adsorption capacity of different iron oxide coated sand varied widely. The iron(II) adsorption isotherm constant $K$ for new sand...
at a pH of 6.5 is 2.5 mg/m² (Sharma et al., 1999). Depending on the type of the coating, the iron(II) adsorption capacity per unit geometric surface area of the different types of iron oxide coated sands were 10 to 55 times that of new sand. The measured high adsorption capacities of coated sand from wet filters and dry filters of full-scale groundwater treatment plants indicate that, in wet filters, adsorptive iron removal also plays a role. In dry filters, this mechanism should be dominant due to a very short pre-oxidation time.

Figure 2 presents the iron(II) adsorption capacity of coated sand from different plants as a function of time in use. In general, the adsorption capacity increased with the time in use and an increase in the iron content of the coating. However, the annual average increase of the iron content and the adsorption capacity was different for the coated sand from different plants. This can be attributed to the difference in water quality, process conditions applied and time in use.

There was no linear correlation between the iron(II) adsorption capacity and the SEIC (Figure 3). This is likely because the density of iron(II) adsorption sites depends not only on the iron content and the specific surface area, but also on the chemical composition, crystal structure and surface properties of the coating. Secondly, there can be a significant difference between the total BET specific surface area of an adsorbent and the actual surface area available for a particular adsorbate. This is related to the pore sizes and their distribution within the adsorbent particle. It is very likely that a portion of the total available surface area is inaccessible to the iron(II) ions (Weber, 1985) due to blocking and/or filling of the pores with newly deposited layers of iron hydroxides.

Grain size and density

The grain sizes of filter sand from iron removal plants had increased with time in use. The average annual growth rate of the effective grain size ($d_{10}$) varied from 0.7% to 20.7%/year. Sieve analysis showed that regardless of the extent of increase in the grain size of the media from different WTPs with coating development, there was no significant change in the uniformity coefficient ($d_{60}/d_{10}$) of the filter grains. This indicates that there was a uniform growth of the filter grains in a given treatment plant suggesting that the filter sand was properly mixed during backwashing. This is likely the case because backwashing with air and/or air and water is commonly applied.

The coating development was much faster in case of dry filters (WTPs Sellingen, and Lichtvoorde). This is probably because, in the case of dry filters, pre-oxidation time is minimal and more iron(II) is available for adsorption. However, in the case of WTP Helden, the growth rate of the filter media was low even when they were from the dry filter likely due to low raw water pH. Additionally, it was observed that the coating development
was also faster in the case of groundwater with high concentration of iron and organic matter (WTPs Boxmeer, Noord Bargares and Brucht). The effect of raw water quality on the rate of growth of filter media in iron removal plants needs further detailed investigation.

It was observed that when the iron oxide coating was relatively thin (coating constituting <20% of total volume or weight of the coated sand), there was no significant change in the density of the sand with the coating development. However, with an increase in coating thickness, the density of the grain decreased considerably, probably due to the development of a porous coating. Prasad and Belsare (1984) also reported the decrease in density of filter sand with the development of oxide coating. Figure 4 shows that the decrease in density of the coated sand was a function of the increase in the effective grain size. The increase in grain size and consequent decrease in density of the coated sand implies that the tendency of the “grown” grains to move towards the bottom of the filter bed during backwashing is reduced and filter beds would probably be fully mixed even after many years of operation.

Theoretically, the average density of the coated sand is given by

$$\rho_{acs} = (\rho_s - \rho_c) \left(\frac{d_s}{d}\right)^3 + \rho_c$$  \hspace{1cm} (1)

where \(\rho_{acs}\) = average density of the coated sand (kg/m\(^3\)); \(\rho_s\) = average density of the sand (kg/m\(^3\)); \(\rho_c\) = average density of the iron oxide coating (kg/m\(^3\)); \(d_s\) = average diameter of the sand grain, without coating (m) and \(d\) = average diameter of the coated sand (m).

This shows that the average density of coated sand is a function of the increase in grain size, density of sand and density of the iron oxide coating. As the filter media grows bigger with the development of the coating, the density of the coated sand approaches the density of the coating.

**Media surface analysis**

**X-ray diffraction.** Powder X-ray diffraction (XRD) of the media coatings of sand from different plants showed that there were no distinct iron oxide crystals present on the coatings. The coatings were X-ray amorphous or very poorly crystalline. It is likely that the filter media coatings consist of a mixture of poorly ordered iron oxides together with some built-in impurities. X-ray diffractograms of all the coated sand were very similar and overlapping except that of the coated sand from WTP Helden, which had some small peaks indicating poor crystallinity due to the presence of maghemite (\(\gamma-\text{Fe}_2\text{O}_3\)).

Iron, oxygen, manganese, calcium, silicon and carbon were the main elements found in the coatings. The presence of silica favours the formation of amorphous iron oxides (Robinson *et al.*., 1981; Carlson and Schwertmann, 1996). Huang and Wang (1997) reported that adsorption of silica, phosphate or organic matter on iron (hydr)oxide inhibits or
retards its natural transformation to more crystalline or stable forms. Manganese oxides also modify the crystallisation process of iron oxides, resulting in the formation of X-ray amorphous and/or different crystalline iron oxides. This can be an explanation for the iron oxide coating on filter sand being very poorly crystalline or X-ray amorphous.

**Scanning Electron Micrographs and EDAX analysis.** SEMs of the filter media coatings were prepared to get an insight of the nature of the coating. SEMs of the surfaces of new sand and coated sand from WTP Gilze are presented in Figures 5 and 6 respectively. Figure 6 shows some cracks on the surface of the coating, which may have already existed on the coating or might have developed during the preparation of the samples for SEM analysis. SEMs of the coated sand revealed nodules and globular forms on the coating and there was a high degree of surface roughness.

EDAX analysis revealed that iron and oxygen were the two major elements of all the coatings tested. Other elements present in the coating were manganese, calcium, silicon and carbon. This shows that, in addition to iron, different ions present in the raw groundwater like manganese, calcium, silicate, and organic matter are also adsorbed on the iron oxide coating. Adsorption of these elements influences the mineralogy and, hence, probably alters the adsorption capacity of the coating and influences the regeneration of iron(II) adsorption sites.

Two different types of surface features, namely “smooth region” (a) and “rough region” (b), were observed on the SEM of the coated sand from WTP Gilze (Figure 6). EDAX analysis showed that there was a significant difference in the elemental composition of the coating in these two regions. The region with the smooth surface was mainly iron oxide with some silicon and calcium but no manganese (Figure 7), whereas the region with the rough surface was composed of iron and manganese oxides together with silicon and calcium (Figure 8). This shows that iron oxide coating developed on the filter media was not uniform throughout the filter grain and different regions of the coated sand could have different elemental composition and different surface characteristics and, hence, different adsorption properties. Gold peaks observed in the spectrum were from the gold coating applied to the filter media before SEM analysis.

**Conclusions**

Based on the analysis of the physical and chemical characteristics of iron oxide coated sand from twelve iron removal plants in the Netherlands, the following conclusions can be drawn.

![Figure 5 SEM of new sand surface](image)

**Figure 5** SEM of new sand surface

![Figure 6 SEM of the surface of the coated sand from WTP Gilze showing two different surface features (a) smooth region and (b) rough region](image)

**Figure 6** SEM of the surface of the coated sand from WTP Gilze showing two different surface features (a) smooth region and (b) rough region
• Compared to new sand, iron oxide coated sand had a very high porosity (up to 110 times) and a very large specific surface area (5–200 times).

• The higher the iron loading, the higher was the surface extractable iron content of the coatings. The iron content of the coatings ranged from 27% to 45%, indicating a substantial difference in chemical composition of the coatings.

• At pH 6.5, the adsorption capacities of different coated sands were 10 to 55 times that of new sand. In general, iron(II) adsorption capacity was higher for the coated sand with a high surface extractable iron content and large specific surface area. However, the average annual increase of the iron content and the adsorption capacity varied for the coated sands from different plants, probably due to the difference in water quality, process conditions applied and time in use.

• The grain size of the filter sand increased and the density decreased with the development of iron oxide coating. The decrease in density of coated sand with
the iron oxide coating development was a function of the increase in the effective grain size.

- Iron oxides on the coatings were X-ray amorphous or poorly crystalline. SEM/EDAX analysis showed that iron and oxygen were the main components of the coating and elements like manganese, calcium, silicon, and carbon were also present in the coatings. Analysis of coated sand from WTP Gilze showed that the coating developed on a filter grain was not uniform and different regions of the coating on a sand grain could have a different elemental composition and surface characteristics.

- The measured high adsorption capacities of coated sand from wet filters and dry filters of full-scale groundwater treatment plants indicate that, in wet filters, adsorptive iron removal also plays a role. In dry filters, this mechanism should be dominant due to a very short pre-oxidation time.

- As all the iron oxide coated sand tested demonstrated high iron(II) adsorption capacity, process efficiencies of wet filters with coated sand can be further improved by operating the filters predominantly in the adsorptive filtration mode.

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References


### Appendix 1  Major raw water quality parameters of groundwater treatment plants

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Roosendaal</th>
<th>Gilze</th>
<th>Helden</th>
<th>Ossendrecht</th>
<th>Oosterhout</th>
<th>Boeimeer</th>
<th>Vlijmen</th>
<th>Macharen</th>
<th>N.Bargares</th>
<th>Lichtvoorde</th>
<th>Brucht</th>
<th>Sellingen</th>
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<td>pH</td>
<td>–</td>
<td>7.2</td>
<td>7.65</td>
<td>5.75</td>
<td>6.9</td>
<td>7.6</td>
<td>7.05</td>
<td>6.65</td>
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<td>7.07</td>
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<tr>
<td>Fe</td>
<td>mg/l</td>
<td>7.2–10</td>
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<td>9</td>
<td>12</td>
<td>1.2–2</td>
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<td>0.39</td>
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<td>Ca²⁺</td>
<td>mg/l</td>
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<td>0.7</td>
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<td>4</td>
<td>1.5</td>
<td>7.1</td>
<td>16.5</td>
<td>13</td>
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<td>(DOC)</td>
<td>6.1</td>
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