

EXPERIENCES WITH APPLICATION OF INHIBITORS TO REDUCE HEAVY METAL UPTAKE OF DRINKING WATER

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Summary

Drinking water as a basic food stuff is a valuable and limited good and thus underlies a high level of regulation to ensure its quality for the consumer. The European Drinking Water Directive (98/83/EC) provides microbiological and chemical parameters that define the minimum quality standards within the European Community, which have been transferred into national laws by the member states. In Germany the new Drinking Water Ordinance (Trinkwasserverordnung, TrinkwV) has come into force January 1st, 2003.

An important change in the new directive is the extension of the purview to the tap of the final consumer of drinking water. As a consequence the interaction of the drinking water with various kinds of materials of the household installations has to be taken into account, that can lead to a deterioration of the water quality e.g. due to heavy metal uptake from metallic tubes and installations. The metal uptake is strongly influenced by the individual regime of water usage, that varies considerably according to the individual habits of the consumer. Therefore, according to the German TrinkwV, the determination of the chemical parameters copper, nickel and lead has to be based on a representative sample reflecting the mean weekly water consumption of the end user. A standardised method to determine this parameter, that is widely accepted, is DIN 50931-1. According to the results a substantiated recommendation can be given about the compatibility of the installation materials with the water investigated. Furthermore, the efficiency of corrosion inhibitors, i.e. based on silicate and phosphate, can be measured.

This paper describes the fundamental approach of this method and the principle for the evaluation of the mean weekly metal concentration. It shows the results of studies on the influence of different corrosion inhibitors on the heavy metal uptake of different kinds of drinking waters. The focus is put on copper.

The data derived from selected field studies at German municipal water suppliers clearly show, that a continuous addition of inhibitors based on phosphate and silicate can significantly reduce the heavy metal uptake of critical waters. Therefore, the dosage of a corrosion inhibitor can be an effective measure to ensure, that the standards of the Drinking Water Directive can be met even under critical conditions.

Introduction

Due to the importance of the quality of drinking water (water intended for human consumption) for the human health, the European Community has defined quality standards, that this kind of water has to meet [1]. The so-called European Drinking Water Directive (98/83/EC) intends to prevent any water contamination that may have any impact on the consumers health and to protect its purity and usefulness for consumption.

Within the sense of the directive water intended for human consumption does include not only drinking water in the narrow sense of the word, but also water used for cooking, food preparing and other domestic purposes. Furthermore, water used in food plants is to a large extent subject of it. According to the principle of subsidiarity the Drinking Water Directive has been transferred into national, i.e. German law by the German Drinking Water Ordinance [2]. There, the term domestic purpose is specified more precisely to include water used for body care and for cleaning of objects duly having food contact.

The Drinking Water Directive defines the minimum quality standards for water intended for human consumption, whereas the member states may define more stringent or additional parameters, if necessary.

As the purview of the drinking water directive has been extended to the tap of the final consumer, the interaction of the drinking water with various installation materials used in the distribution network and, especially, households has to be considered. Therefore, the control of the water quality has to ensure, that the sampling represents the water quality during the whole year.

Amongst other parameters the uptake of heavy metals is of major concern for human health. Table 1 gives a summary of the parameters for metals most commonly used

in drinking water pipes and installations as defined by the European Drinking Water Directive and the German Drinking Water Ordinance. Additionally, the recommended limits from the World Health Organisation [3] are given for comparison. The uptake of heavy metals can lead to significant deterioration of water taste and color (“red water” formation) as well as toxic effects.

Table 1: Survey of limits and recommendations for heavy metals in drinking water

	Parameter value [mg/l]		Recommendations of WHO ⁴⁾ [mg/l]		
	89/83/EC ¹⁾	TrinkwV ²⁾	HBGV ³⁾	discoloration / staining	bad taste
Copper	2	2	2	-	>5
Lead	0.01	0.01	0.01	-	-
Zinc	-	-	-	3-5	>4
Iron	-	0.2	-	>0.3	~ 0.3
Nickel	0.02	0.02	0.02	-	-

¹⁾ Drinking Water Directive

²⁾ Drinking Water Ordinance (Trinkwasserverordnung TrinkwV)

³⁾ Health Based Guideline Value

⁴⁾ World Health Organization

Unfortunately, there is no unequivocal correlation between heavy metal uptake and water quality until now. Furthermore, the heavy metal uptake strongly depends on the specific installation design and the consumers habits, that may vary sharply with time and individual person. As a consequence, a sample taken randomly generally does not reflect the representative water quality, as demanded by legislation. Therefore, there is a need for a harmonised and standardised method in order to determine the parameter values of a water for different metals.

In Germany the control of the limits for lead, copper and nickel is based on a sample representing the mean weekly water consumption (weekly mean value). Single measured values higher than the parameter value of the drinking water directive, are not looked upon as an infringement of the parameter value.

The iron level in the drinking water is not only restricted by the limit itself, but also by the general requirements of drinking water having to be colourless and tasteful. Therefore, formation of ‘red water’ cannot be accepted.

Based on the available experiences the ranges of applicability for different standardised metallic materials have been specified in DIN 50930-6 [4]. E.g. copper is looked upon as compatible with a water without further testing, if the pH of the water is either above 7.4 or if the pH is between 7.0 and 7.4 and the TOC is below 1.5 g/m³. In case it is intended to use a material outside of these specified ranges of application an evaluation of the parameter value is required. DIN 50931-1 [5] describes the accepted procedure to evaluate these parameters with a test device simulating typical household conditions with stagnation periods of different lengths.

Corrosion Inhibitors

In cases where an installation material cannot be recommended for a water quality the dosage of corrosion inhibitors to the drinking water is an appropriate measure to enhance the range of applicability of this material. The use of inhibitors is not regulated in detail by the Drinking Water Directive but by the national regulations. According to § 11 of the German Drinking Water Ordinance the Federal Environmental Agency has set up a list [6] of accepted additives for drinking water preparation and disinfection. This list defines the scope of application, the maximum dosage for each component as well as the quality of the material itself, generally referring to an European Standard, e.g. EN 1209 for sodium silicate [7]. This well defined frame allows within its limits a safe application of corrosion inhibitors without any impact on the drinking water quality.

Different chemicals are allowed to use as corrosion inhibitors in drinking water: phosphate, i.e. ortho and poly phosphates, silicates or activated silicates [8] or combinations of these.

In Germany the continuous dosage of corrosion inhibitors to the central distribution network is successfully applied for many years [9-12]. Generally, the focus has been the prevention of red water formation. The mechanisms of red water formation have been extensively investigated [13-14] as well as the mode of action of the corrosion inhibitors [15-17].

Due to the new Drinking Water Directive the influence of corrosion inhibitors on the heavy metal uptake, especially on copper, has become more the attention of research [18-20]. It has been shown, that the addition of phosphate and silicate to drinking water can significantly reduce the copper uptake.

This paper provides data on heavy metal uptake in the field determined by the German Standard DIN 50931-1 and evaluated according to DIN 50930-6 for copper and lead. Additionally, the results of studies on iron corrosion are shown.

Copper and lead uptake

Test Method and Evaluation

Basically, the test equipment simulates the situation in the kitchen of a household installation at the final train of the pipes (figure 1, 2). The investigations to determine the copper uptake from copper pipes, respectively the zinc and lead uptake from galvanised steel pipes were carried out in accordance to DIN 50931-1. Sampling and evaluation followed the norms specifications. Operational conditions were characterised by 22 short phases with water running for 1 or 2 minutes and 22 stagnation periods of different lengths between a quarter of an hour and sixteen hours. The flow through each test tube was approx. 145 l per day at a flow-through of 300 l/h.

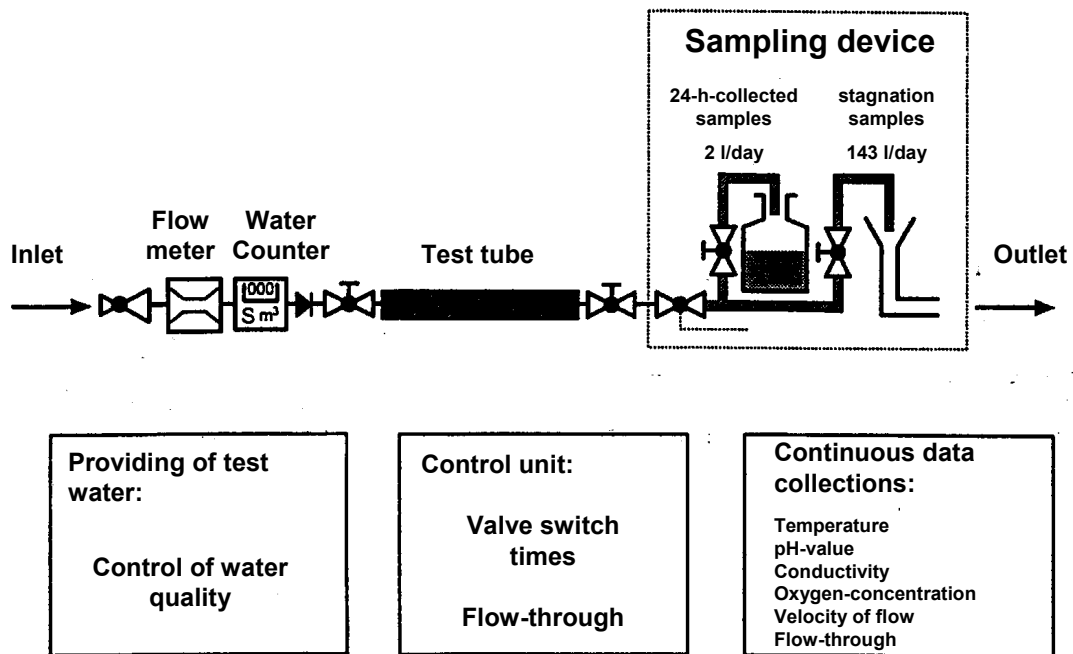


Fig. 1: Scheme of a corrosion test plant according to DIN 50931-1



Fig. 2: Picture of a corrosion test plant according to DIN 50931-1

To determine the heavy metal uptake properties of the water stagnation curves, i.e. concentration against time curves, were measured after defined weeks of operation, i.e. after 1, 2, 3, 6, 12, 18, 26, 52, 78 and 104 weeks. Water samples were taken from the stagnating water after stagnation periods of 0.5, 1, 2, 4, 8 and 16 hours and the heavy metal concentrations were measured. Sampling was done twice for the stagnation periods of 0.5 and 1 hour. Thus, in total eight samples finally were available.

The measured heavy metal concentrations were plotted versus the stagnation period to provide a concentration time curve (figure 3). From the analytical data the maximum concentration $c_{\max}(T)$ and the mean value $M(T)$ were determined. According to DIN 50930-6 the mean value $M(T)$ is equivalent to the mean weekly concentration of the Drinking Water Ordinance respectively the representative sample of the Drinking Water Directive and delivers the heavy metal load of the drinking water under the given conditions. $M(T)$ is the basis for the assessment of the suitability of a material or installation component for the investigated drinking water. This holds also for the dosage of corrosion inhibitors.

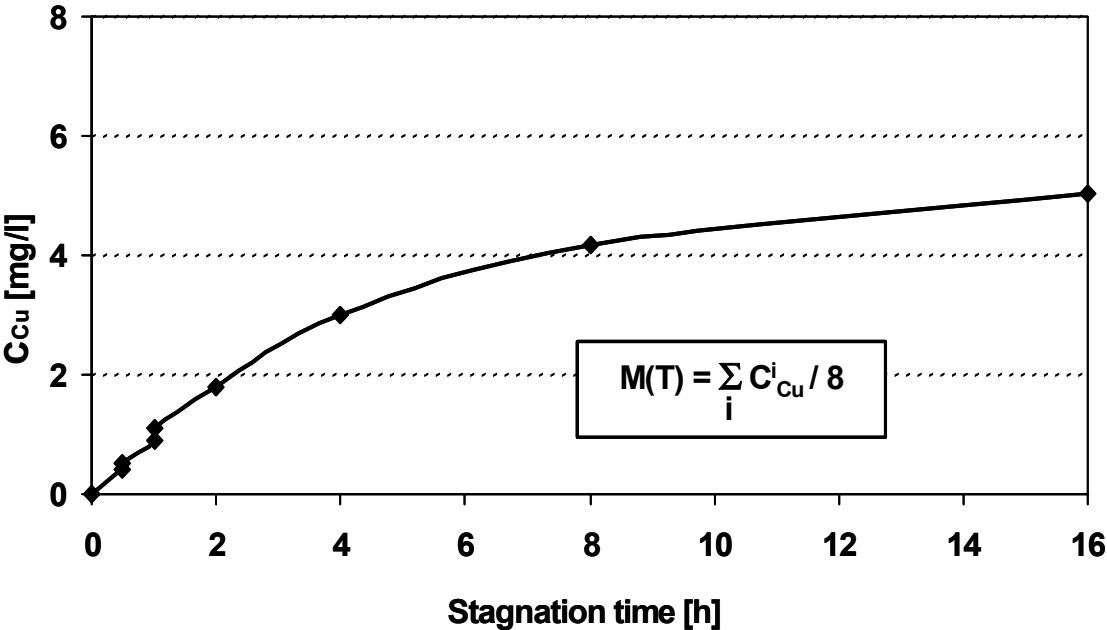


Fig. 3: Example of stagnation curve for copper, i.e. copper concentration in dependence of stagnation time

For further evaluation the mean value $M(T)$ and the maximum value $c_{\max}(T)$ are plotted versus operational time. Decisive for the assessment of the compatibility of a installation material with a water is total behaviour of the system. Thus, a single value is of no relevance. This kind of curves are discussed in detail later. In case that metal tubes of a certain diameter (DN 10 to DN 25) have been used for the test the tube material may be used, if the following conditions are obeyed:

I: for materials forming a protective layer

M (104 weeks) \leq Parameter value

M (6 weeks) $\leq 2 \bullet$ Parameter value

II: for stainless steel

M (12 weeks) \leq Parameter value

There are additional general requirements to the behaviour of $c_{\max}(T)$ and $M(T)$, for details see DIN 50930-6.

In addition to DIN 50931-1 during 24 hours water samples were collected weekly for the first 26 weeks of the trial respectively each fortnight afterwards. These samples show any distortion of the test plant especially in between the long periods of the test run. Furthermore they give an indication of the influence of any change in water quality.

Results and Discussion

During the last years various investigations about the heavy metal uptake of drinking water according to DIN 50931-1 were carried out at different German water-works. Generally, the target of these investigations was to prove the compliance with the new requirements of the Drinking Water Ordinance, especially with regard to the new copper parameter.

For this purpose a test plant was installed on-site at the water-works using the drinking water before its distribution in the net, but without addition of a corrosion inhibitor (reference). To study the influence of the effect of an inhibitor addition, several test strains were operated simultaneously. The dosage of the additives was done by automatic feeding systems in the test plant.

In the subsequent section selected results of some studies are presented.

• Study I:

The water supply of a major German city is done by bank filtered river water, that is prepared in several steps before distribution. For many years the application of a corrosion inhibitor based on silicate and phosphate successfully prevented red water formation.

Although the water quality (table 2) was not looked upon as critical for copper use according to DIN 50960-6 ($7.0 \leq \text{pH} \leq 7.4$; $\text{TOC} < 1.5 \text{ mg/l}$), the persons responsible for the water-works decided to carry out a study on the copper uptake. The test regime followed DIN 50931-1, but additional stagnation curves were determined after the 26th week of the trial.

Table 2: Typical analysis of the drinking water qualities used for the corrosion resp. metal uptake investigations

Parameter	Unit	Study I	Study II	Study III	Study IV	Study V
pH		7.3	7.3	7.4	7.8	7.5
$K_{S4.3}$ ¹⁾	mol/m ³	3.4	5.5	3.4	2.6	3.2
$K_{B8.2}$ ²⁾	mol/m ³	0.5	0.8	0.4	0.1	0.2
TH	mol/m ³	2.6	3.2	2.7	2.3	2.2
Ca	mol/m ³	2.1	3.0	2.5	1.7	0.5
Cl	mg/l	83.7	22.8	33.3	79.0	74.1
SO ₄	mg/l	57.6	13.8	94.1	72.0	63.0
TOC	mg/l	0.5	0.8	3.9	n.d.	n.d.

¹⁾ m value

²⁾ - p value

The trial was carried out with clean water, i.e. water fed into the net prior to addition of disinfectant and corrosion inhibitor, as blank value. For comparison in a second strain the corrosion inhibitor was added to the clean water. The corrosion inhibitor was a mixture of ortho, poly phosphate and silicate. From week 81 of the trial a different inhibitor was used with a reduced phosphate content, but increased silicate level.

Figure 4 shows the mean value $M(T)$ in dependence of the trial duration, Figure 5 the corresponding curve for the maximum concentration $c_{max}(T)$. Surprisingly, the water without corrosion inhibitor seems to be quite aggressive to copper, as the mean value hardly decreases below the parameter value throughout the whole test period. Thus a clear statement about the compatibility of copper with the untreated water is not possible. However, the values of $M(T)$ are always below the critical value of 4 mg/l (2 • parameter value of 2 mg/l).

The corresponding curve of $M(T)$ for the water with corrosion inhibitor is for the whole test period clearly below the parameter value as well as below the curve of the untreated water. Probably the slight increase of the data at the end of the test is due to the change of the corrosion inhibitor.

The curves of maximum concentrations $c_{max}(T)$ principally show a similar result, but on a much higher level. Furthermore, the scattering of data is higher.

The addition of a corrosion inhibitor leads to a significant reduction of the copper values. A level of 1 mg/l phosphate and 1 mg/l silicate is sufficient for this water quality to meet the parameter value of 2 mg/l copper of the Drinking Water Ordinance.

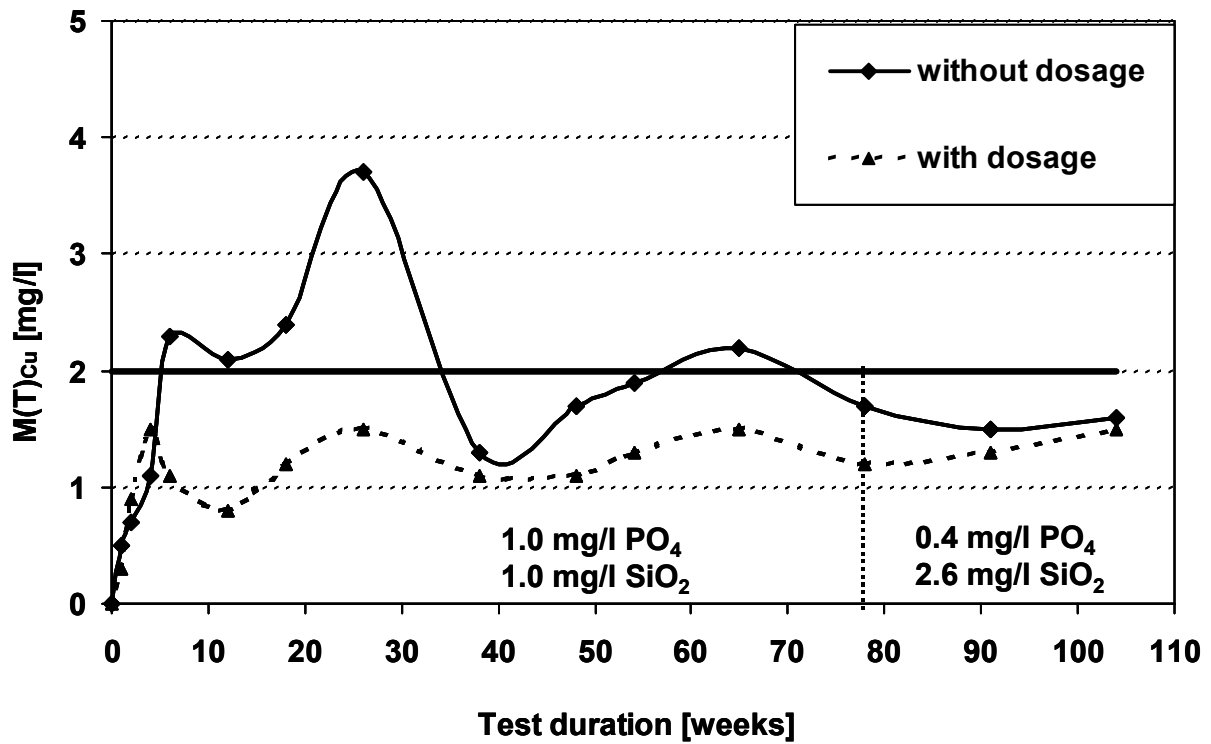


Fig. 4: Dependence of the mean weekly copper concentration – $M(T)_{Cu}$ – on time for a drinking water with and without addition of a corrosion inhibitor based on phosphate /silicate. The target concentrations of the inhibitor components and the application periods are indicated. The parameter value for copper is shown by the thick black line.

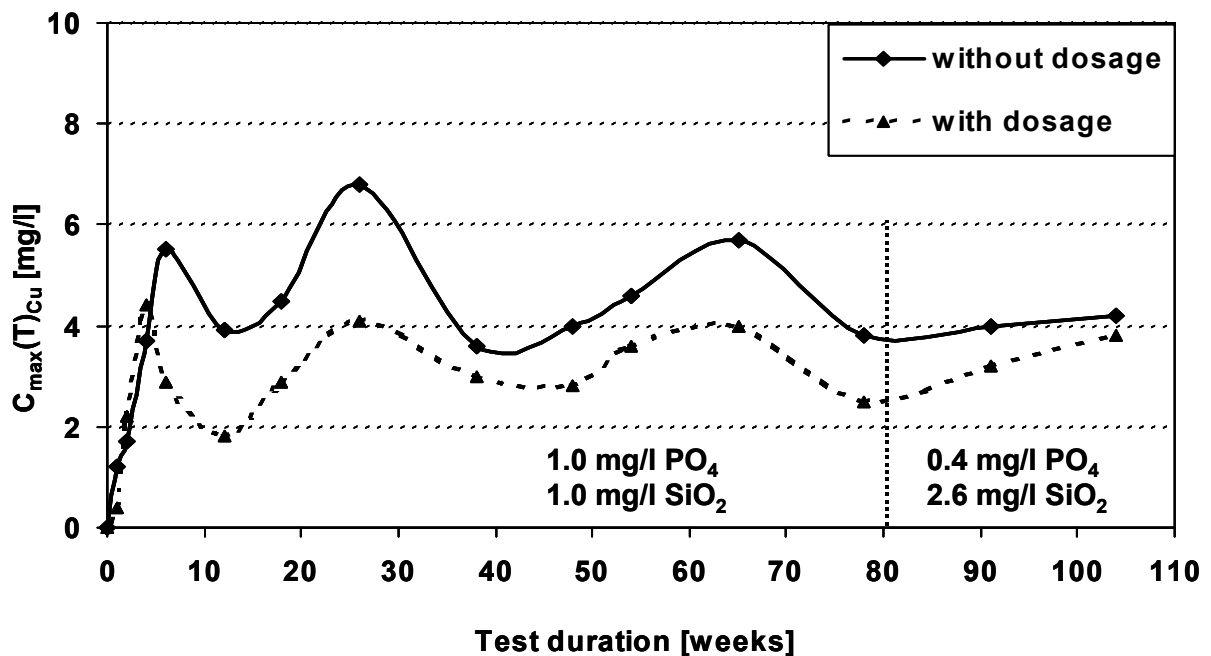


Fig. 5: Dependence of the maximum copper concentration – $c_{max}(T)_{Cu}$ – on time for a drinking water with and without addition of a corrosion inhibitor based on phosphate/silicate. The target concentrations of the inhibitor components and their application periods are indicated.

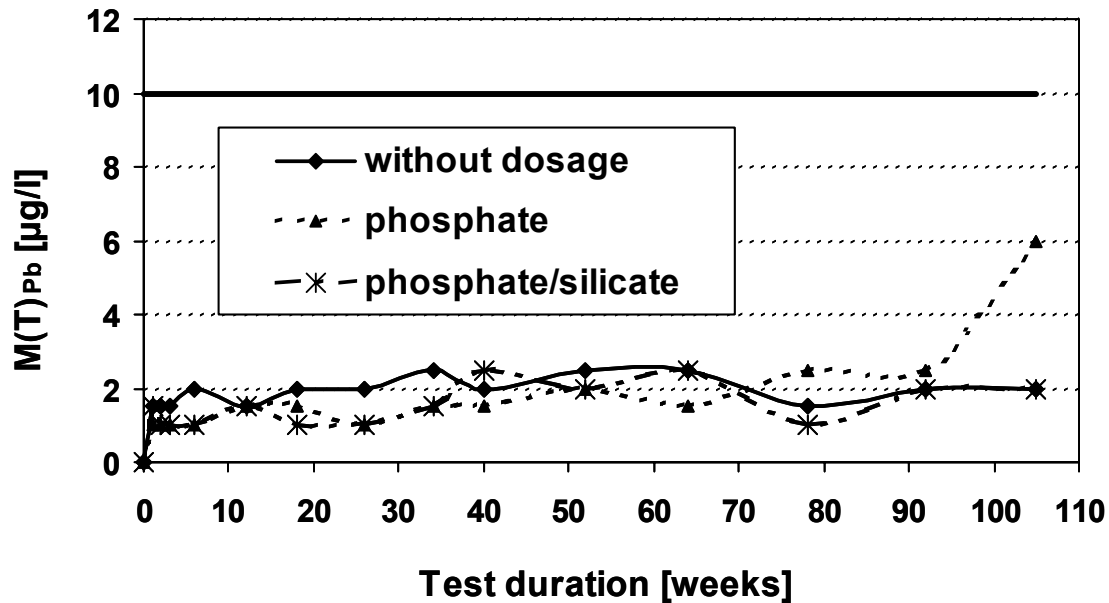
- **Study II:**

Within the supply area of a South German water-works consumers of the drinking water claimed red water formation as well as blocking of pipes due to scaling, mainly in the hot water sections of their installations. The quite hard water is taken from wells. Table 2 gives the typical water composition. The majority of the household piping was made from copper or galvanized steel.

The protection of galvanised steel against corrosion mainly is due to a slow and uniform dissolution of the zinc layer into the water. As a consequence there will be an entry of the other components of the zinc layer into the water, especially lead. Therefore, the range of application of galvanised steel tubes is limited by DIN 50960-6 to waters having a $K_{B8.2} \leq 0.5 \text{ mol/m}^3$ (-p value) and a $K_{S4.3} \geq 1.0 \text{ mol/m}^3$ (m value). Furthermore, the maximum lead content of the zinc layer must not exceed 0.25 %. The tubes used for the investigation fulfilled this demand.

The lead migration is shown in figure 6. All measured values for the mean weekly lead concentration are very low and clearly below the parameter value. A trend cannot be noted. Furthermore, there is no significant effect by the inhibitor dosage. The reason for the increase of M(T) in the water with ortho phosphate dosage at the end of the test duration is not clear. According to DIN 50930-6 the values of M(T) should continuously decrease during the last phase of the trial. If this is not obeyed, like in this study, all data of M(T) must be below the 0.8 fold of parameter value, i.e. 8 µg/l for lead. As a consequence, the data indicate that the parameter value for lead will be kept for this water, if the tested galvanised steel tubes are used.

Thus, the central dosage of a corrosion inhibitors prevents red water formation and scaling without an infringement of the parameter values for lead.



	Phosphate	
Dosage	3 mg/l o-PO₄	2 mg/l o-PO₄
	Phosphate/silicate	
	2.1 mg/l o-PO₄ 0.9 mg/l p-PO₄ 0.0 mg/l SiO₂	1.05 mg/l o-PO₄ 0.45 mg/l p-PO₄ 4.50 mg/l SiO₂

Fig. 6: Dependence of the mean weekly lead concentration – $M(T)_{Pb}$ – on time for a drinking water with and without addition of different corrosion inhibitor systems based on phosphate/silicate. The target concentrations of the inhibitor components and their application periods are indicated below the graph. The parameter value for lead is shown by the thick black line.

- **Study III:**

In the Northern part of Germany a water-works supplies drinking water from moorland, thus having a comparatively high level of organics. The DOC concentration is generally between 3.5 and 4.0 mg/l and the pH is around 7.4 (table 2). As a consequence the compatibility of copper with the water was uncertain. Therefore, investigations according DIN 50931-1 were done to prove this and, additionally, to measure the efficiency of corrosion inhibitors to reduce the copper uptake of the water. Thereby, emphasis was put on the minimisation of the amount of phosphate.

The test plant was operated with three strains each for the untreated water and for water with addition of 3 mg/l ortho phosphate. From the M(T) curve (figure 7) it can be clearly seen that copper installations are not suitable for this water, if no corrosion inhibitor is used, as the values scatter around 2.1 mg/l copper, which is above the parameter value of 2 mg/l. However, the addition of phosphate reduces the M(T) 4 to 5 fold in comparison to the untreated water, i.e. the parameter value can be safely kept.

As the target had been to use a corrosion inhibitor with low amount of or even without phosphate after 52 weeks the inhibitor in two of the treated strains was changed to a phosphate silicate mixture to provide a nominal dosage of 1.0 mg/l phosphate (the ratio of ortho to poly phosphate was 1 to 1) and 6.5 mg/l silicate. The M(T) curve versus test duration doesn't show any significant change caused by the inhibitor change (figure 8). Also with the silicate based corrosion inhibitor including a low amount of phosphate the same reduction of the mean copper value can be achieved and the parameter value kept.

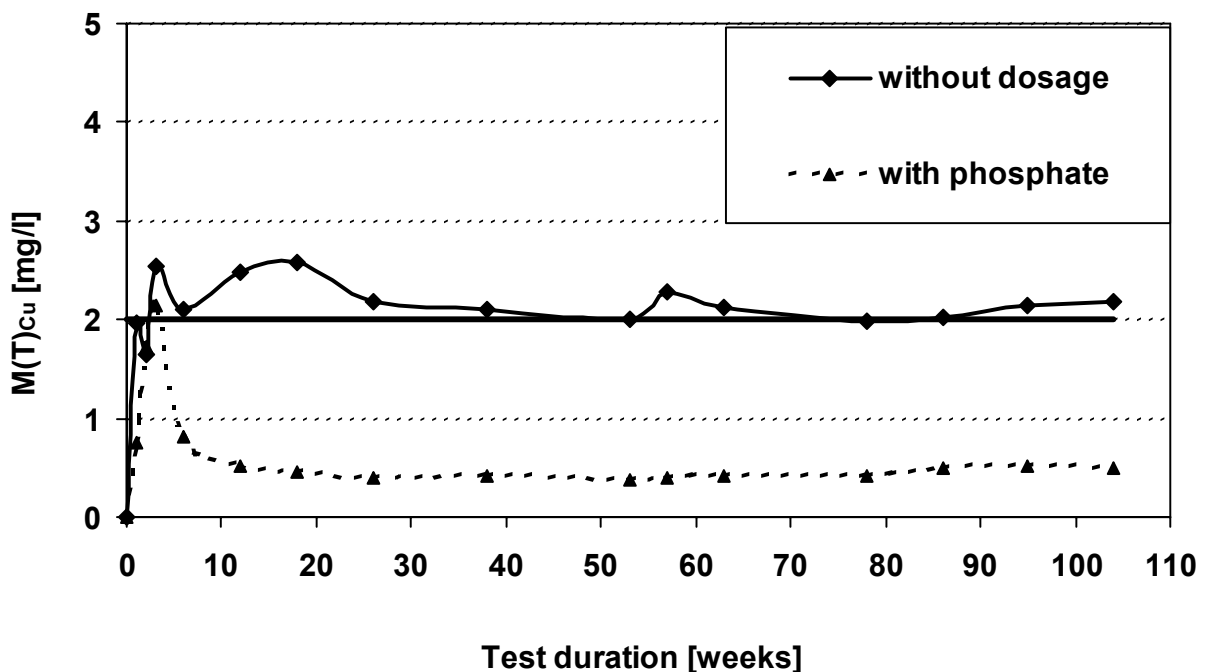


Fig. 7: Dependence on the maximum copper concentration – $M(T)_{Cu}$ – on time for a drinking with and without the addition of 3 mg/l ortho phosphate. The parameter value for copper is shown by the thick black line.

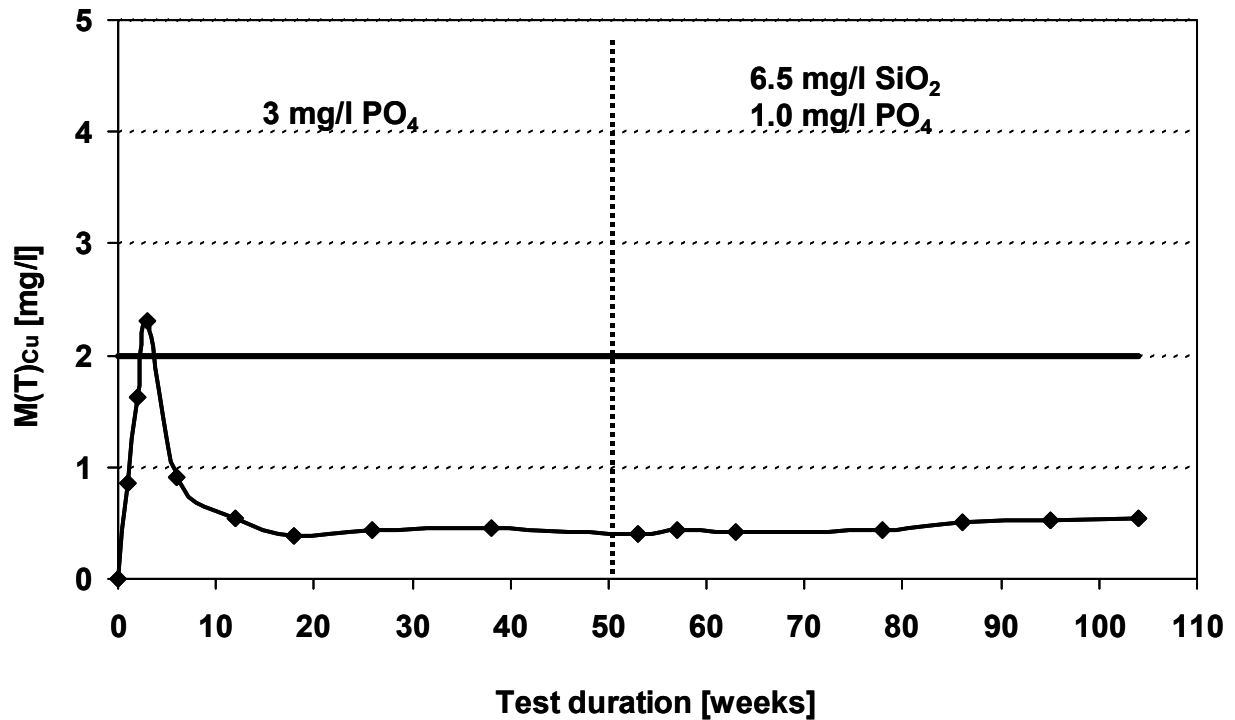


Fig. 8: Dependence of the mean weekly copper concentration – $M(T)_{Cu}$ – on time for a drinking water with and without addition of a corrosion inhibitor based on phosphate/silicate. The target concentrations of the inhibitor components and their application periods are indicated. The parameter value for copper is shown by the thick black line.

Therefore, one of these two strains was changed after 78 weeks to a total phosphate free corrosion inhibitor with a silicate dosage of 8 mg/l. However, as figure 9 indicates, the $M(T)$ value quickly increases to reach finally the same level as the untreated water. A completely phosphate free treatment seems not to be suitable for this water.

If a suitable corrosion inhibitor is used, i.e. either a silicate/low phosphate combination or only ortho phosphate, but at a higher level, these investigations according to DIN 50931-1 have shown, that the parameter value for copper can be met for this kind of drinking water. Without a corrosion inhibitor, however, the requirement of the Drinking Water Ordinance with regard to the copper parameter cannot be met under these test conditions.

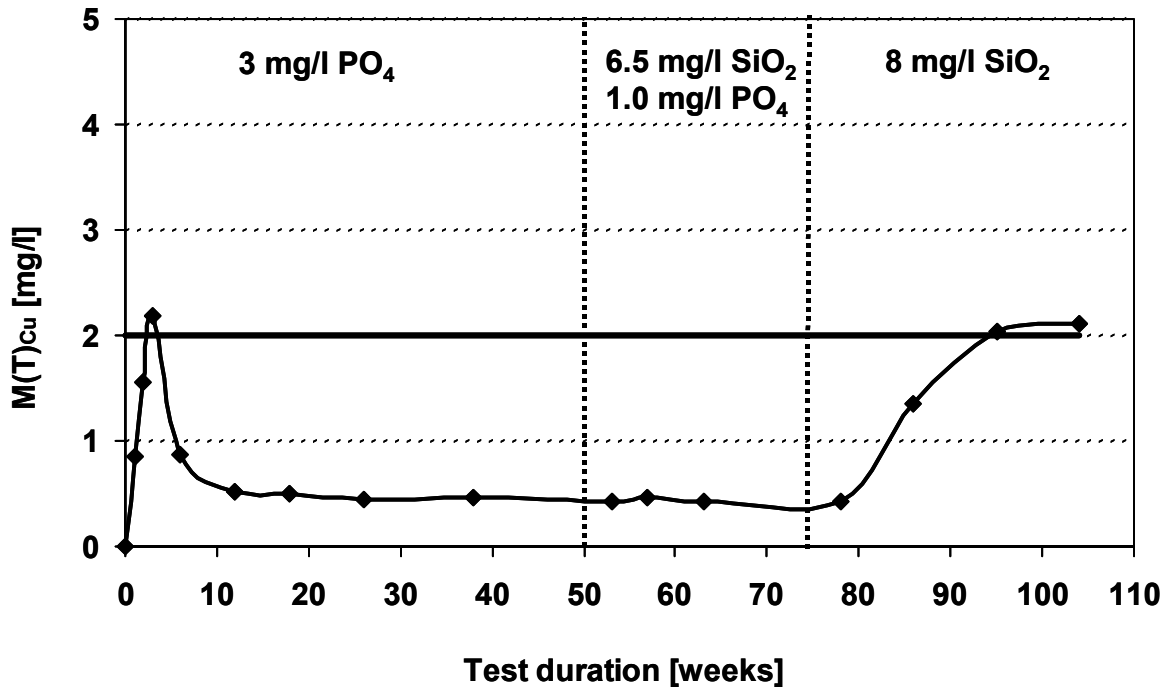


Figure 9: Dependence of the mean weekly copper concentration – $M(T)_{Cu}$ – on time for a drinking water with and without addition of a corrosion inhibitor based on phosphate/silicate. The target concentrations of the inhibitor components and their application periods are indicated. The parameter value for copper is shown by the thick black line.

Corrosion of steel

Drinking water that is transported through carbon steel pipes can be deteriorated by corrosion causing change of colour or taste due to small amounts of iron. To investigate corrosion phenomena in drinking water distribution systems a model system has been used, that has been applied successfully for many on-site studies at water works (Figure 10). This system is run under defined operation conditions and can be adapted to the specific plant conditions.

A single test unit uses pipes with a length of 1 m operated continuously during the whole trial period. For each measurement of the corrosion rate the tubes are removed and put into a closed circulating device completely made by non-corroding materials. In this device, that is operated with the same kind of water and the same water flow, the oxygen consumption is determined in the water and the corrosion rate calculated thereof.

The flow-through test units are equipped with tube pieces of app. 0.3 m length, that are mainly used for analysis of the protective layer formed during the trial period, i.e. by X-ray fluorescence and X-ray diffraction.

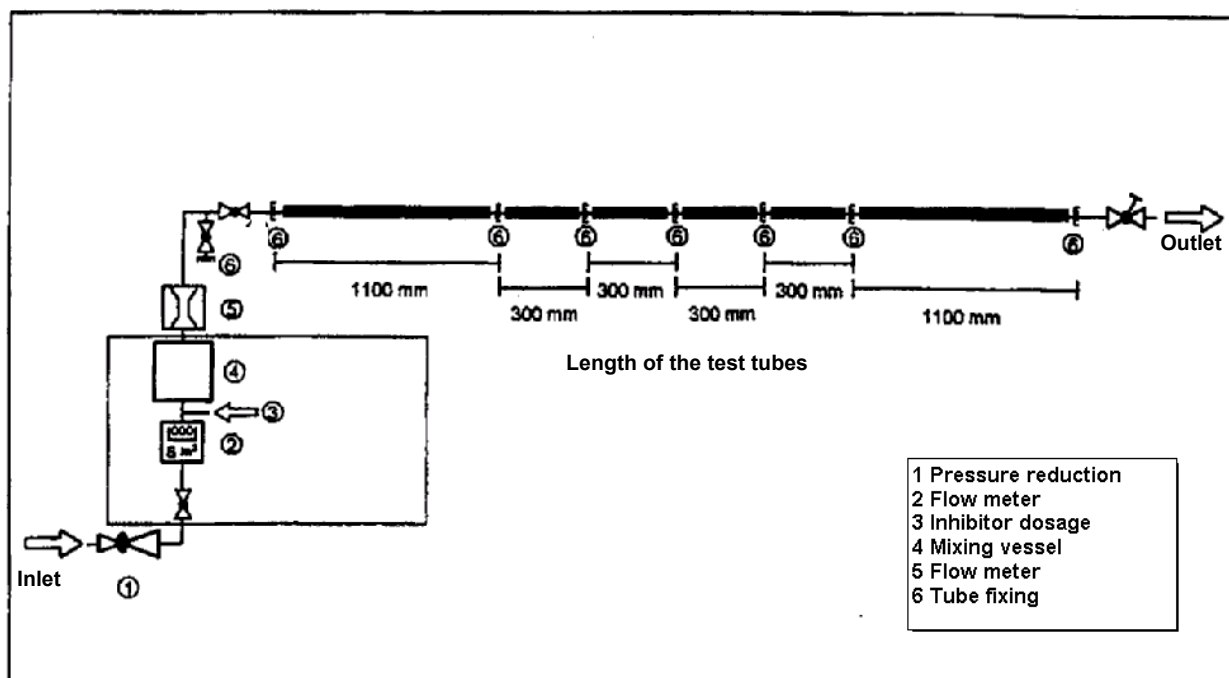


Fig. 10: Scheme of a test unit for the investigation of the corrosion of carbon steel in drinking water

- **Study IV:**

A water works in west Germany supplying considerable hard water decided to install a partial softening of its drinking water. Due to this the corrosiveness of the drinking water (table 2) increased finally leading to the installation of a central dosage of a corrosion inhibitor in order to avoid red water formation. Therefore, an investigation program was started to study the efficiency of different inhibitor systems.

Figure 11 shows the development of the corrosion rates for the untreated drinking water and for two corrosion inhibitors. To precondition the steel tubes all units were operated without addition of a corrosion inhibitor for 8 weeks, first. After this 3 mg/l ortho phosphate respectively 9 mg/l silicate and 3 mg/l of Phosphate were continuously added to the drinking water of two units, whereas one unit remained without inhibitor addition.

The data clearly show that the addition of a suitable corrosion inhibitor reduces the corrosion rate on carbon steel by more than 50 %. A combination of silicate and phosphate provides a higher efficiency compared to solely ortho phosphate. This result is supported by the analysis of the protective layer, that had been formed at the end of the test period: x-ray diffraction analysis could clearly determine γ -FeOOH in the layer formed by the untreated water. However, the layers formed by inhibited water did not show any evidence of this crystal form, playing an important role for red water formation.

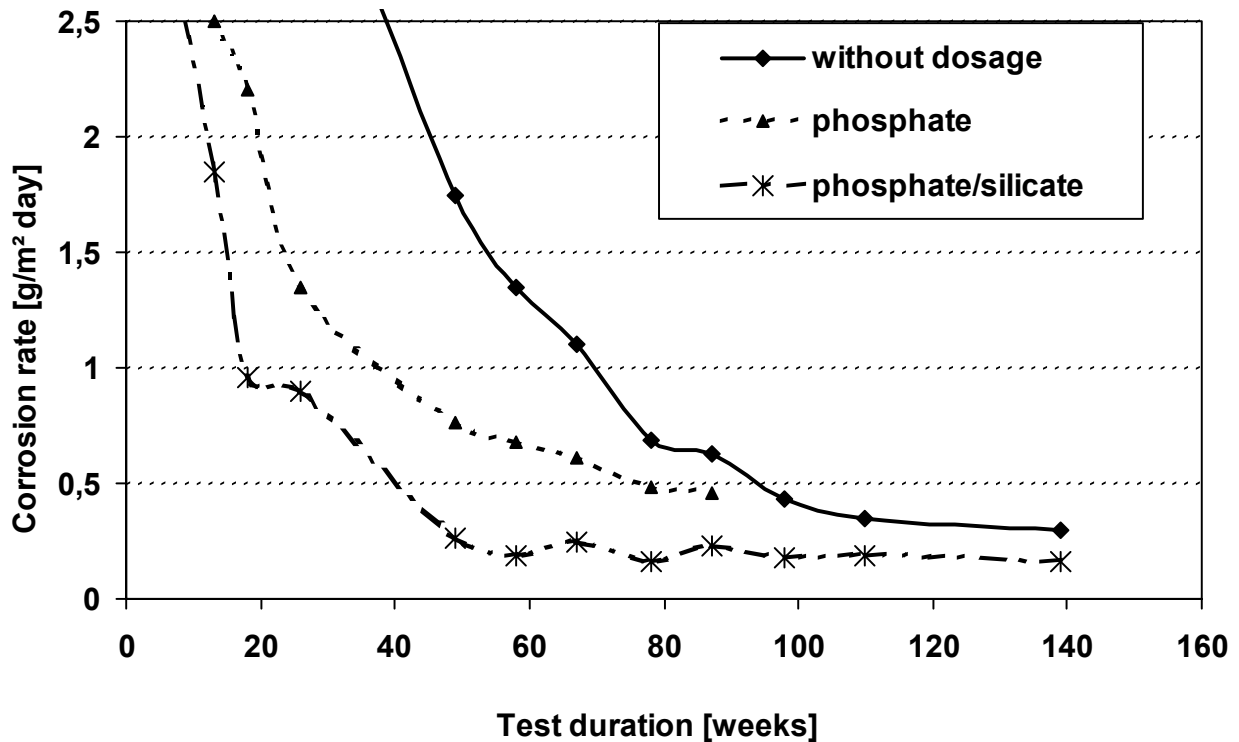


Fig. 11: Dependence of the iron corrosion rate on time for a drinking water with and without addition of corrosion inhibitors. (Phosphate: 3 mg/l ortho-phosphate; Phosphate/silicate: 3 mg/l phosphate + 9 mg/l silicate)

- **Study V:**

A water works in the South of Germany gains its drinking water from soft, acidic spring water. To meet the requirement of the drinking water ordinance it was necessary to pretreat the water, mainly by de-acidification. Table 2 shows a typical analysis of the water after physical de-acidification. After a change of the reagent used for the pH adjustment a test program was initiated to optimise the corrosion behaviour of the drinking water. The target was to achieve a suitable corrosion inhibition on carbon steel by a phosphate free treatment.

Figure 12 shows the corrosion rate for two selected strains of the test unit: untreated drinking water and drinking water with addition of corrosion inhibitor. The inhibition was started with a phosphate/ silicate mixture (3 mg/l phosphate/ 3 mg/l silicate) and was switched to only silicate after 32 weeks. Finally, the silicate level was reduced from 6 mg/l to 4.5 mg/l after 88 weeks.

The addition of an inhibitor reduced the corrosion rate by approx. 50 % compared to the untreated water. The corrosion rates measured with untreated water are increasing at the end of the test period, whereas the corrosion rates remained stable at a very low level in the strain operated with treated drinking water. Furthermore, the dosage of the silicate based inhibitor considerably improved the mechanical stability of the protective layer formed on the tube surface. The removal of iron from the tube

surface under increased water flow could be reduced by approx. 70 % by the addition of silicate to the drinking water. As a consequence, the continuous dosage of a suitable corrosion inhibitor sharply reduces the risk of red water formation.

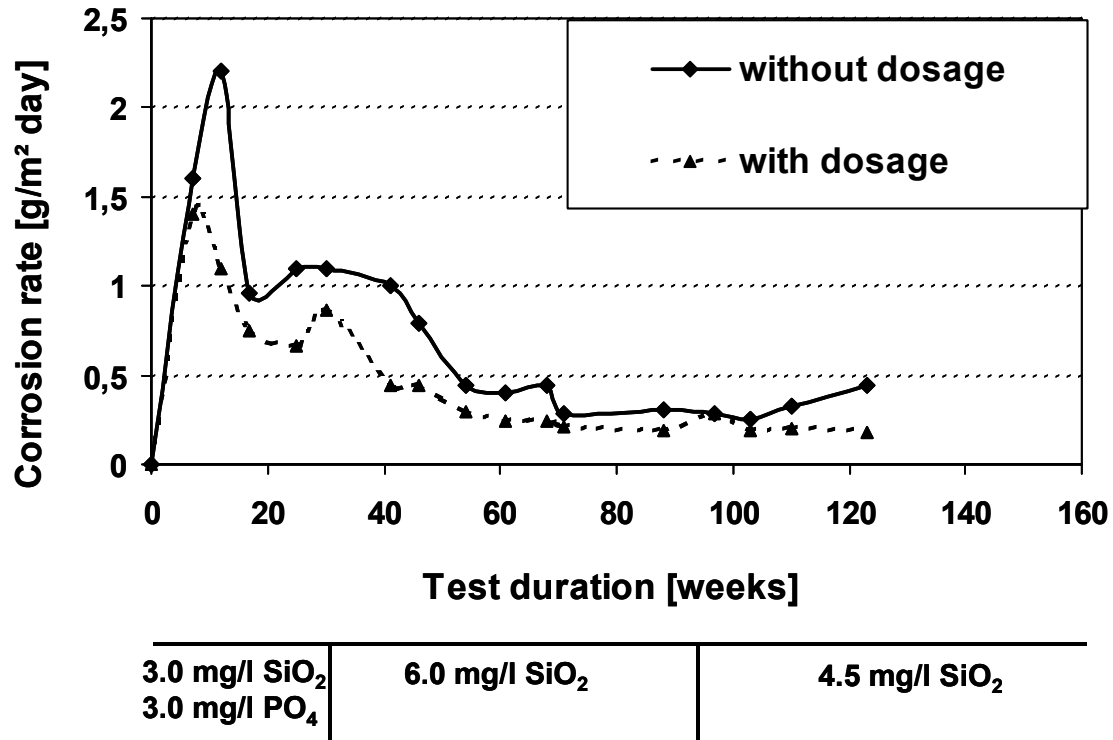


Fig. 12: Dependence of iron corrosion rate on time for a drinking water with and without addition of corrosion inhibitors. The target concentrations of the inhibitor components and their application period are indicated below the graph.

Conclusions:

The drinking water legislation in combination with the corresponding norms, i.e. DIN 50931-1 and DIN 50930-6, give a clear frame in order to judge the compatibility of a construction material with the drinking water supplied with respect to the parameter values. Furthermore, the efficiency of measures to reduce heavy metal migration, e.g. addition of corrosion inhibitors, can be assessed.

Various investigations according to DIN 50931-1 at water-works have been carried out mainly for the determination of the copper migration. Thereby, measurements on the efficiency of different corrosion inhibitor systems have been included. Additionally, studies on iron corrosion and the efficiency of corrosion inhibitors have been presented.

The presented results show:

- Galvanised steel tubes containing low lead amounts, i.e. that were in accordance to the requirements of DIN 50960-6, showed lead migration that was below the parameter value of the drinking water directive. For this water quality the dosage of a corrosion inhibitor showed no significant influence on the lead uptake from galvanised steel tubes.
- For critical drinking water qualities the parameter values of the drinking water directive cannot be kept for copper.
- The addition of corrosion inhibitors can significantly reduce the copper migration. Besides phosphate based inhibitors also silicate/phosphate combinations are suitable corrosion inhibitors.
- The addition of corrosion inhibitors is an efficient means to reduce iron corrosion and to avoid formation of red water in drinking water networks.

Literature:

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