

THE EXTRACTABLE ZINC CONTENT OF SOILS IN THE NETHERLANDS  
DETERMINED BY VARIOUS METHODS

KEY WORDS: zinc in Avena sativa, zinc in Freesia hybr., zinc in soils,  
glasshouse soils, soils in the Netherlands.

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ABSTRACT

102 Soil samples collected from arable soils in the Netherlands were analysed for zinc by various extraction methods. Two crops, viz. oats (Avena sativa L.) and freesias (Freesia hybr.) were grown on the samples in pots and also analysed for zinc. Zinc in soil extracted with 1 N  $\text{NH}_4\text{Ac}$  of pH 7 gave the best correlation with the zinc content of the crops. Compared with data in literature more zinc was found in soils in the Netherlands than in some other countries. The average zinc value for glasshouse soils was higher than for outdoor soils.

INTRODUCTION

In a fluorine investigation samples were collected from arable soils all over the Netherlands. Two crops - oats and freesias - were grown on the soil samples in pots and the plant material was analysed.<sup>1</sup> The samples were also used in the study of other trace elements. In this publication the extractable zinc content of the soil and its relation with the zinc content of the plant is studied. Another publication (in press) deals with the manganese content. About the zinc content of arable soils

in the Netherlands little is known. The few publications in this field are mainly studying excess of this element.<sup>2</sup>

For the determination of plant available zinc in the soil many extractants are described in the literature.<sup>3,4</sup> In this study two extractants are compared, viz. solutions of ammonium acetate and of ethylenediamine tetraacetate (EDTA) in water. The pH of the solution being very important the latter was tested as solutions with low or high pH. Furthermore HCl was used as an extractant in order to obtain figures indicating "total" zinc contents.

#### MATERIALS AND METHODS

Of the 102 samples a relatively high percentage (39) consisted of glasshouse soils. The lowest percentage (7) came from grassland. There was a wide variation in the soil types. The pH ranged from 3.9 to 8.05, the calcium carbonate content from 0 to 10.2%, the clay content from 1 to 46% and the organic matter content from 1.4 to 76%.

The samples of about 50 litres were collected with a spade from the upper soil layer (0 to 20 cm deep) over an area of a few square metres. After mixing, two 10-litre plastic buckets with a surface area of 491 cm<sup>2</sup> were filled from each sample. Eight freesia corms of the variety Rijnveld's Golden Yellow were planted out in one bucket and oats of the variety Leande were sown in the other at a rate of 19½ g per bucket. The buckets were placed in a glasshouse in which the air was filtered through charcoal. The crops were grown without fertilisers and demineralised water was used.

The freesias were cut just before flowering and the aerial and subterranean parts were weighed fresh and again after drying. The soil in the pots was loosened, reagent-grade chemicals were added at a rate of 4.3 g NH<sub>4</sub>NO<sub>3</sub> and 6.6 g KH<sub>2</sub>PO<sub>4</sub> per pot and a new freesia crop was started by planting out 8 freesia corms of the variety Rijnveld's Golden Yellow. The second freesia crop was treated like the first one.

The first oat crop was harvested when it was about 20 cm high. The plants were cut off at soil level. The soil in the pots was loosened and root debris was removed. After this preparation analar chemicals were added as in the case of the freesias and oats of the variety Leende were sown again. The second crop was treated in the same way as the first one.

#### ANALYTICAL METHODS

##### Zinc

The soil samples were dried at 45°C for one night after which they were milled. Four extractants were used for the determination of zinc :

a. Ammonium acetate 1N, pH 7, according to Shaw & Dean.<sup>5</sup> Extraction ratio soil : extractant = 1 : 10 w/v, shaken at room temperature for 15 minutes. No dithizone was added.

b. EDTA-Na<sub>2</sub> 0.02 M, pH 4.6, according to Jensen & Lamm.<sup>6</sup> Extraction ratio 1 : 2½ w/v, shaken at room temperature for 60 minutes.

c. EDTA-Na<sub>2</sub> 0.01 M plus (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> 1 M, pH 8.6, according to Trierweiler & Lindsay.<sup>7</sup> Extraction ratio 1 : 2 w/v, shaken at room temperature for 30 minutes.

d. HCl 0.1 N, pH 1, according to Nelson et al.<sup>8</sup> Extraction ratio 1 : 10 w/v, shaken at room temperature for 15 minutes.

For filtering Whatman 540 paper was used.

The crop samples were dried at about 80°C for 24 or 48 hours after which they were milled with an ordinary household type electric coffee grinder.

The samples of the first oat crop (Oats I) were digested with a mixture of nitric, perchloric and sulphuric acid mixture, a modification of Schaumlöffel.<sup>9</sup> The samples of the other crops (Oats II, Freesias I and II) were extracted with trichloroacetic acid (2%) according to Legget & Westerman.<sup>10</sup> An extraction ratio 1 : 100 w/v and a shaking time of 16 hours were used. A few of these crop samples were also digested. The values obtained by the two methods were very similar which confirms the results reported by Legget & Westerman.<sup>10</sup> In the case of the freesias

only the aerial parts of the plants were used for analysis. The zinc content in all extracts was determined with the Varian Techtron model AAS atomic absorption spectrophotometer. The results are expressed in ppm Zn (mg per kg) of air dry soil or of the dry matter of the crop samples.

#### Other determinations in soil samples

pH - using a glass electrode in a soil-water suspension 1 : 5 w/v and measured after one night.

CaCO<sub>3</sub> - with the Scheibler method.

Organic matter - by loss of weight on ignition at 600°C.

Clay content - by the macro pipet method.

P water - extraction with water 1 : 5 w/v at room temperature, expressed as mg P<sub>2</sub>O<sub>5</sub> per 100 mg dry soil.

Pw value - extraction with water 1 : 60 w/v according to Sissingh.<sup>11</sup>  
Expressed as mg P<sub>2</sub>O<sub>5</sub> per dm<sup>3</sup> dry soil.

Resin extractable P - extraction with water 1 : 50 w/v with the addition of an anion resin, according to Hislop & Cooke,<sup>12</sup> shaken at room temperature for 16 hours. Resin percolated with 7% NaCl. Expressed as ppm P of dry soil.

Total P - digestion with Fleischmann acid in 1 : 10 ratio w/v.  
Expressed as % P<sub>2</sub>O<sub>5</sub> of the dry soil.

The P values of the extracts were determined with the method of Murphy and Riley.<sup>13</sup>

#### RESULTS OF THE ANALYSIS FIGURES

The extreme and the mean values of the four zinc analysis in the soils are given in Table 1. The figures have been tabulated separately for outdoor and glasshouse soils since the latter might contain more zinc as a result of heavy fertiliser dressings and the fact that part of the glasshouse structure consists of galvanised iron.

Table 2 - comprising all data (n = 102) - shows the correlation coefficients between the soil zinc contents determined with the aid of four

TABLE 1. Extreme and mean values of zinc in glasshouse and outdoor soils determined by four analytical methods (ppm Zn of dry soil)

	$\text{NH}_4\text{Ac}$ -Zn	EDTA -Zn	$\text{EDTA}+(\text{NH}_4)_2\text{CO}_3$ -Zn	HCl -Zn
Glasshouse soils (n = 30)				
Lowest value	5	40	32	180
Highest value	98	428	328	1250
Mean	29.1	135.9	94.7	585.0
Outdoor soils (n = 72)				
Lowest value	0.5	3	2	10
Highest value	134	340	192	1420
Mean	15.6	51.4	40.7	237.1
Total mean (n = 102)	19.6	76.3	56.6	339.4

analytical methods. The coefficients for the relationships between the zinc values and other soil analytical data are also shown.

As Table 1 shows, the extractable zinc contents of arable soils in the Netherlands ranged from 10 to 1420 ppm Zn of dry soil if the strongest extractant - HCl - was used. Glasshouse soils on average contained more zinc than outdoor soils. The reason for this is difficult to explain and will be discussed later.

Zinc in glasshouse soils was also analysed by Laake<sup>14</sup> using EDTA (1 : 10 w/v). The data reported by this author were lower with an average value in the upper soil layer of 67.5 ppm Zn and lowest and highest values of 22 and 84 ppm Zn respectively. In a recent paper this author<sup>15</sup> reported about the same values.

Jensen & Lamm<sup>6</sup> found considerably lower levels of zinc extracted with EDTA in Danish soils. Their highest value was 41 ppm Zn with an average of about 4 ppm Zn. Klemm & Bergmann<sup>16</sup> published results of zinc extracted

TABLE 2. Correlation coefficients, comprising all data ( $n = 102$ ), for the relationships between four zinc values in soil and other soil analytical figures.

	NH <sub>4</sub> Ac -Zn	EDTA -Zn	EDTA + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> -Zn	HCl -Zn
NH <sub>4</sub> Ac -Zn	-			
EDTA -Zn	0.68 **	-		
EDTA + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> -Zn	0.79 **	0.95 **	-	
HCl -Zn	0.59 **	0.81 **	0.77 **	-
pH	-0.44 **	-0.15	-0.22 **	-0.16
CaCO <sub>3</sub>	-0.26 **	-0.26 **	-0.23 **	-0.29 **
Organic matter	0.31 **	0.54 **	0.57 **	0.45
Clay	-0.36 **	-0.12	-0.17	-0.13
P water	0.23 **	0.45 **	0.36 **	0.57 **
Pw value	0.19	0.48 **	0.38 **	0.60 **
Resin extractable P	0.20 *	0.59 **	0.49 **	0.66 **
Total P	0.12	0.58 **	0.48 **	0.62 **

\*  $p = 0.05$

\*\*  $p = 0.01$

with EDTA + (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> from soils in Germany (DDR). Their lowest and highest values were 1.6 and 25.3 ppm Zn respectively, with 5.2 ppm Zn as the average.

Table 2 shows that soils with high organic matter content generally contain more zinc. This fact has been recorded in the literature by other research workers.<sup>4</sup> An interesting point was the relationship which was found between the phosphate contents in soil determined by various methods and the zinc contents extracted with HCl or with the EDTA extractants. The correlation between phosphate and zinc extracted with

$\text{NH}_4\text{Ac}$  was only slight. Laske<sup>15</sup> found using EDTA a correlation between soil zinc and lactate soluble phosphate.

In the search for an explanation for the positive relationship between the zinc and phosphate contents the length of time during which the glasshouse soil has been in use (the age of the glasshouse) was taken into account. It is known for instance that the phosphate content of the glasshouse soil has a strong correlation with the age of the glasshouse.<sup>17</sup> Table 3 shows the relationship between the age of the glasshouse (ranging from 0 to 50 years) or the logarithm of the age and a number of soil determinations amongst which those of phosphate and zinc.

The data in Table 3 confirm the experience that there are positive relationships between the age of the glasshouse and the organic matter and phosphate contents of the glasshouse soil. The data also seem to indicate that the zinc content of the glasshouse soil increase with the

TABLE 3. Correlation coefficients for the relationship between the age of the glasshouse or the logarithm of the age and some analytical figures obtained from 30 samples of glasshouse soil.

	<u>Age of the glasshouse</u>	<u>Logarithm of the age</u>
Organic matter	0.08	0.27
P water	0.20	0.21
Pw value	0.24	0.24
Resin extractable P	0.17	0.20
Total P	0.29	0.39 *
$\text{NH}_4\text{Ac}$ -Zn	0.12	0.17
EDTA -Zn	0.30 (*)	0.34 (*)
EDTA + $(\text{NH}_4)_2\text{CO}_3$ -Zn	0.16	0.25
HCl -Zn	0.22	0.22

(\*) P = 0.10

age of the glasshouse. In tracing the reason for the relationship between the phosphate and zinc contents in the soil and the possible connection with the age of the glasshouse, at least three factors should be taken into account : a. the organic matter content in the soil, b. the solubility of the zinc compounds present in the soil and c. the supply of zinc.

a. The organic matter content may affect the relationships between phosphate and zinc in different ways. An increase of the organic matter content will change the zinc and phosphate contents as it decreases the weight per volume but the effect should be similar for both elements. It is interesting that a clear relationship was found between the percentage organic matter and the extractable zinc content of the soil (Table 2) but with regard to phosphate, the existence of a significant relationship could only be established between the percentage organic matter and total P ( $r = 0,54^{**}$ ) as well as resin extractable P ( $r = 0.36^{**}$ ), but not between the percentage organic matter and the other two phosphate determinations (P water and Pw value).

In the case of glasshouse soils in particular, it is likely that the regular application of farmyard manure increases the organic matter content, the phosphate content and also the zinc content of the soil. This will be discussed in greater detail under g. Another aspect is the formation of less soluble organic zinc (phosphate) compounds which will be discussed under b.

b. The solubility of zinc phosphate compounds.

The literature is not very clear on this point. Lindsay<sup>4</sup> found  $Zn_3(PO_4)_2 \cdot 4 H_2O$  more soluble than soil zinc. Olsen<sup>3</sup> stated that phosphate induced zinc deficiency is not caused by the precipitation of zinc phosphate. However, it is possible that some zinc phosphate compounds have such a low solubility that there is little leaching of zinc which would lead to high zinc levels in soils rich in phosphates. The origin of the zinc is not important in this case. Lindsay<sup>4</sup> suggests that zinc may

become a component of immobile organic soil constituents which act as a fixation mechanism preventing the release or leaching of zinc.

c. The supply of zinc by fertilisers or other materials.

According to Swaine<sup>18</sup> most fertilisers contain only traces of zinc. Small amounts of zinc are carried sometimes by phosphates and by superphosphates and rock phosphates in particular. Dutch superphosphate has been found to contain between 3 and 1,340 ppm Zn with 300 ppm as an average.<sup>19</sup> This means that with a normal dressing of 10 kg superphosphate per 100 m<sup>2</sup>, 3 g zinc is applied. According to Jensen & Lamm<sup>6</sup> farmyard manure contains 50 to 500 ppm Zn on dry weight basis, 180 ppm Zn being an average. This means that with every ton of farmyard manure containing 180 ppm Zn on dry weight basis applied to 100 m<sup>2</sup> one also applies 45 g Zn if the manure contains 75% moisture.

Pesticides are another source of zinc. One zineb spray applies nearly 4 g Zn per 100 m<sup>2</sup>. About 1,000 mm water is used every year for glasshouse irrigation. Dhasee<sup>20</sup> found that surface water contained an average of 0.04 ppm Zn. Irrigation with such water applies another 4 g Zn per 100 m<sup>2</sup>. The surface water in the glasshouse area around Naaldwijk originates for the greater part from the river Rhine. Unfiltered Rhine water contains as an average 0.15 ppm Zn.<sup>21</sup> In this area 15 g Zn per 100 m<sup>2</sup> is applied with the irrigation annually. Corrosion of the galvanised parts of the glasshouse plays a part, particularly if the gutters are made of galvanised steel and CO<sub>2</sub> enrichment is applied by burning sulphur containing paraffin. Nevertheless, zinc from glasshouse gutters must be considered of minor importance as some of the older glasshouses examined in this investigation (40 to 50 years old) had no or only smaller galvanised parts.

#### Comparison of the contents of extractable zinc in the soils and the zinc contents of the crops.

The zinc contents of the aerial parts of the freesia crops ranged from 28 to 228 ppm. In the young oat plants the zinc contents ranged from

44 to 316 ppm Zn on dry weight basis. No symptoms of zinc deficiency or toxicity were observed. According to Lindsay<sup>4</sup> a range of 10 to 100 ppm zinc may be considered normal. Smilde et al.<sup>2</sup> studied zinc excess in a polluted acid sandy soil. They found 36 to 54 ppm  $\text{NH}_4\text{Ac}$  extractable zinc together with a low pH (pH-KCl 4.3), resulting in zinc contents of 1,000 to 2,000 ppm in various crops. In Table 4 the correlation coefficients are given for the relationship between the zinc contents of two crops (oats and freesias) and some soil analytical figures. Table 5 shows the relationship between extractable zinc in the soil and the zinc content of the crops.

It may be deduced from Table 4 that the pH and clay content have some effect on the zinc content of the plants. For this reason the relationship between extractable zinc in the soil and zinc in the crop was also computed as a multiple linear regression with the pH and the clay content as the third or the third plus fourth variable. The equations used were  $y = ax + bp$  and  $y = ax + bp + cq$  in which  $y$  = zinc in the crop,  $x$  = zinc in the soil,  $p$  = pH or clay and  $q$  = clay content. Apart from these factors the percentage organic matter was used as the third or the pH plus organic matter as the third and fourth

TABLE 4. Correlation coefficients for the relationship between the zinc content of the crop and some important soil analytical figures.

Zinc in crops :	freessias I	freessias II	Oats I	Oats II
pH	-0.51 **	-0.55 **	-0.56 **	-0.40 **
$\text{CaCO}_3$	-0.28 **	-0.43 **	-0.26 **	-0.24 *
Organic matter	0.03	0.06	0.09	0.02
Clay content	-0.49 **	-0.50 **	-0.31 **	-0.30 **

TABLE 5. Simple and multiple correlation coefficients for the relationship between the zinc content of some crops and zinc in the soil determined by various analytical methods, with and without the pH and the clay content or organic matter content of the soil as concomitant variables (all values highly significant)

X = y = zinc in crops	NH <sub>4</sub> Ac	EDTA	EDTA + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	HCl	Concomitant variables
	-Zn	-Zn	-Zn	-Zn	
Freesias I	0.65	0.39	0.46	0.39	
	0.70	0.60	0.62	0.60	pH
	0.68	0.53	0.57	0.53	Clay
	0.71	0.63	0.64	0.62	pH + clay
	0.68	0.45	0.54	0.42	Organic matter
	0.73	0.69	0.72	0.66	pH + organic matter
Freesias II	0.69	0.38	0.44	0.49	
	0.74	0.63	0.64	0.69	pH
	0.74	0.60	0.62	0.66	Clay
	0.76	0.68	0.69	0.73	pH + clay
	0.70	0.42	0.50	0.53	Organic matter
	0.77	0.70	0.72	0.75	pH + organic matter
Oats I	0.60	0.32	0.40	0.32	
	0.68	0.61	0.63	0.60	pH
	0.61	0.43	0.47	0.42	Clay
	0.68	0.61	0.63	0.61	pH + clay
	0.60	0.34	0.43	0.33	Organic matter
	0.70	0.65	0.68	0.64	pH + organic matter
Oats II	0.58	0.27	0.34	0.26	
	0.61	0.45	0.48	0.45	pH
	0.59	0.38	0.42	0.35	Clay
	0.61	0.47	0.49	0.46	pH + clay
	0.62	0.33	0.43	0.30	Organic matter
	0.65	0.54	0.59	0.51	pH + organic matter

variable. Figures 1 and 2 illustrate the relationship between the zinc content of the crop and zinc in the soil ( $\text{NH}_4\text{Ac}$  extracted) and the pH of the soil.

It is evident from the data in Table 5 that the zinc determination in soil using  $\text{NH}_4\text{Ac}$  as the extractant gives the best correlation with the zinc content of the crop. The other extractants yielded distinctly lower correlation coefficients. The correlation may be improved by including the pH, the organic matter or the clay content of the soil in the regression equation as concomitant variables. The pH in particular proved to be important in this respect. By introducing two variables simultaneously - the pH and another factor - the multiple correlation coefficients were further improved but the degree of improvement was rather small in the case of the clay content and slightly larger if the organic matter content was used. The same computations carried out for the relationship between zinc in the crop and zinc in the soil extracted

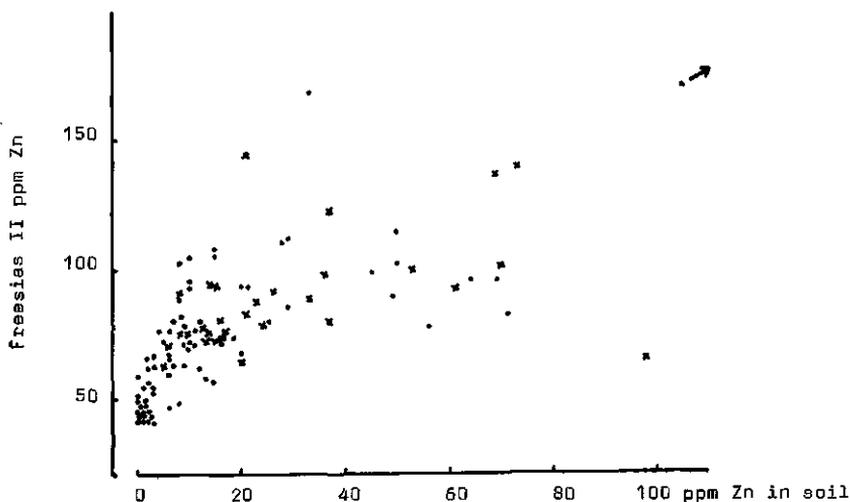


FIG. 1. The relationship between  $\text{NH}_4\text{Ac}$  extractable zinc in soil and the zinc content of the aerial parts of the second freesia crop (ppm Zn on dry weight basis)

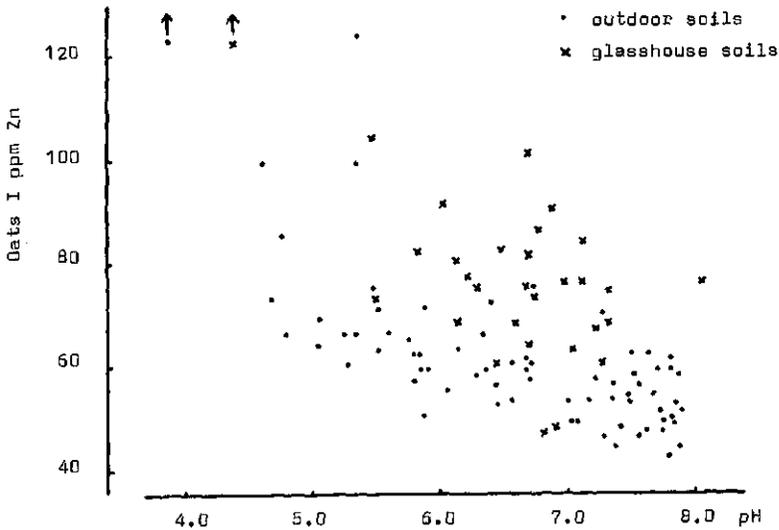


FIG. 2. The relationship between the pH of the soil and the zinc content of the aerial parts of the first oat crop (ppm Zn, on dry weight basis)

with  $\text{NH}_4\text{Ac}$ , also produced an improvement compared with the simple correlation coefficients, but the improvement was relatively small. The introduction of the percentage of organic matter as the fourth variable gave rather better results than the introduction of the clay content. This is the more noteworthy as the organic matter content showed no significant correlation with tissue zinc whilst clay did (see Table 4). Besides the fact that the organic matter content is related to soil weight and moisture content and in this way has some effect on the relationship between the zinc contents of the soil and the plant, it is believed that organic matter also has a special effect on zinc uptake.<sup>4</sup> The conclusion based on the data in this investigation must be that zinc determination with the aid of  $\text{NH}_4\text{Ac}$  provides the best way of predicting the zinc content in the crop. The other methods may also be useful provided the figures are corrected for the pH and the organic matter or clay contents. The same corrections carried out in the case of  $\text{NH}_4\text{Ac}$  extracted zinc enhance the

correlation coefficients, but the necessity for these corrections is questionable in this case.

#### Relationships with other trace elements

Significant correlations were found between the zinc and manganese contents of the crops ( $r = 0.34^{**}$  to  $0.62^{**}$ ), between the fluorine content in the second freesia crop and the zinc levels in the four crops ( $r = 0.36^{**}$  to  $0.41^{**}$ ). Water soluble manganese and water soluble fluoride in the soil were also correlated with the zinc content of the crop. This may be explained by the negative relationship which exists between all these values and the pH of the soil.

#### CONCLUSIONS

Soils in the Netherlands are generally rich in zinc compared with the published results from some other countries. The fact that zinc deficiency does not occur in the Netherlands confirms this conclusion. Glasshouse soils on average contain more extractable zinc than outdoor soils. Of the analytical methods tested soil extraction with ammonium acetate produced the best correlation with zinc content of the crop. If the other extractants are used it is necessary to adjust the values obtained for pH or pH plus organic matter or clay content.

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