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For the determination of iron (II) and total iron with 2,2' - dipyridyl in mineral waters. Reduction of iron (III) with ascorbic acid.

Z. analyt.Chem. 209, 340-341 (1965)

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The determination of bi- and trivalent iron in proximity, in mineral waters has gained in significance, on biological and technical grounds. In order to bring about the fixation of the bivalent iron susceptible to oxidation, at the emergence of the water source, we added the mineral water directly to a solution of 2,2'- dipyridyl and measured the red-coloured complex, which remained stable over several hours photometrically in the laboratory. This method is simple in its execution, sensitive, and the results are easily reproduced.<sup>2,10,11</sup> In order to eliminate as much as possible, any errors in the method, we endeavoured to employ the same analytical procedure for the determination of the trivalent iron. The reducing agents given in the literature are: hydroxylammonium chloride, hydrogen sulphite, hydroquinone and others.<sup>2,9-11</sup> From our experiences, however, ascorbic acid tends to be the best reducing agent. The following have reported at various times, on the volumetric analysis of iron (III) ions, with ascorbic acid: ERDEY et al.<sup>4-7</sup>, FLASCHKA et al. also BARAKAT et al.<sup>3</sup>. The reduction with ascorbic acid was performed in acidic solution of pH-value under 3, in order to dissolve properly all the iron (III) which has already precipitated or is in colloidal solution. Photometric measurement is possible between pH 3 and 8, so that in the direct determination of bivalent iron in mineral waters, buffering is superfluous and is only necessary in the determination of total iron after acidification.

The absorption maximum of the iron - 2,2'-dipyridyl complex in aqueous solution lies at 522nm. It can be measured at this wavelength as well as from the mercury line at 546 nm. Measuring range: 0.02 - 1.0 mg Fe/100 ml. For checking the results, we determined simultaneously the total iron photometrically with theoglycolic acid<sup>12</sup> and by the sulfosalicylic acid procedure. The agreement of the results of these methods with the results after reduction with ascorbic acid and photometric determination with 2,2'-dipyridyl are good.

Directions for work. 1. Determination of bivalent iron.

10 - 20 ml of a 0.1% aqueous 2,2'- dipyridyl solution were placed in a 100 ml- measuring cylinder. Measuring cylinder and reagent solution were weighed and an arbitrary quantity of water added in the field. The red-coloured compound forms immediately. The exact weight of the water is established by weighing the water again in the laboratory. After making up the volume it is read in a 1cm - cuvette against water. The calibration

curve is linear between 0 and 1.0mg Fe<sup>2+</sup>/100 ml.

## 2. Determination of total iron

The weighed water sample (for the purpose, the contents of a whole flask) is clearly acidified with hydrochloric acid p.a. and transferred to a beaker. Possible insoluble residues in the flask are dissolved by the addition of hydrochloric acid p.a. and likewise transferred to the beaker. The water sample is then maintained at about 90 - 95°C for about 30 mins. This procedure is also necessary, when no visible ferric precipitation is present, since our experience shows that iron (II) ions can in spite of this be oxidated and present in a colloidal state. After cooling, the solution is transferred to a measuring cylinder of suitable size and the level made up. An aliquot part is taken and transferred to a 100ml measuring cylinder. The solution is neutralized with caustic soda p.a. to a pH-value of about 2 to max. 3, and 300 - 500 mg ascorbic acid are added to reduce the iron. After the addition of 20 ml reagent solution it is buffered with sodium acetate (about 1-3g) to a pH-value of about 5-6. It is now made up to the mark and measured in a 1cm cuvette.

Detailed work on the collective problems of bi- and trivalent iron in mineral springs will be reported elsewhere.

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