

ESR spectroscopy – an analytical tool for the glass industry

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In the past, wet chemical methods have been developed for the quantitative analysis of polyvalent elements in glasses. The major disadvantage of these chemical methods is that all structural information is lost during analysis. In addition, the analysis of species by wet chemical methods is unreliable due to possible redox reactions during the decomposition process. Therefore, the emphasis is on the physical methods which can directly detect species in bulk glasses. Especially optical and ESR spectroscopy are suitable methods due to their wide range of applications and sensitivity. Concerning quantitative measurements, no comparison between ESR results and chemical analysis of species has been published so far. This work discusses the possible application of ESR spectroscopy in the glass industry by focussing on routine interpretation, the correlation to chemical procedures and quantitative analysis. Results are presented and discussed for iron and chromium in packaging glasses. For example an excellent correlation was found between the Fe^{3+} ESR signal and the chemically determined Fe^{3+} concentration in glass.

ESR-Spektroskopie – ein Analyseverfahren für die Glasindustrie

In der Vergangenheit wurden hauptsächlich naßchemische Methoden zur quantitativen Bestimmung polyvalenter Elemente in Gläsern entwickelt. Der Hauptnachteil dieser chemischen Methoden ist, daß durch die Analyse die strukturelle Information verloren geht. Zusätzlich kann es während des Aufschlußverfahrens zu einer Redoxreaktion kommen. Daher sind physikalische Methoden vorzuziehen, die direkt die Elementspezies im Glas nachweisen. Insbesondere die optische und die ESR-Spektroskopie sind geeignete Verfahren wegen ihres weiten Anwendungsbereiches und ihrer hohen Empfindlichkeit. Was quantitative Messungen betrifft, so wurde bisher noch kein Vergleich zwischen den Ergebnissen der ESR-Spektroskopie und der chemischen Elementanalyse publiziert. Diese Arbeit untersucht die möglichen Anwendungen der ESR-Spektroskopie für industrielle Fragestellungen, vor allem als Routineverfahren für eine quantitative Alternative oder als Ergänzung zu den naßchemischen Methoden. Untersucht wurde speziell der Eisen- und Chromgehalt von Verpackungsgläsern. Im Falle des Eisens ergab sich eine sehr gute Korrelation zwischen dem Fe^{3+} -Signal und dem naßchemisch bestimmten Fe^{3+} -Gehalt der Gläser.

1. Introduction

Polyvalent transition metals in glasses have an effect on the macroscopic properties. Although the concentration of the transition metals is often only in the ppm range, they have, for example, a significant influence on the optical properties. Not only has the nature of the metal an important effect on glass color, but also the oxidation state and the microstructure influence these properties.

In the past, chemical methods have been developed for the quantitative analysis of polyvalent elements in glasses. At present, most of the research activities concentrate on the improvement of these methods [1 and 2]. The major disadvantage of these chemical methods is

that all structural information is lost during analysis. In addition, the analysis of species by wet chemical methods is unreliable due to possible redox reactions during the decomposition process. Therefore, the emphasis is on the physical methods which can directly detect species in bulk glasses. These physical methods are important for the verification of the wet chemical methods for analyzing elements in the ppm range. Table 1 shows the potential of several important methods that can be used to analyze transition metals in glasses. Especially optical and ESR spectroscopy are suitable methods due to their wide range of applications and sensitivity. In the case of ESR spectroscopy not only glass products but also ceramics, glazings and raw materials can be analyzed by this method alone if they have relevant paramagnetic centers such as transition metals or structural defects [3 and 6]. Table 2 summarizes the most important oxidation states that can be detected by ESR.

Concerning quantitative measurements, no comparison between ESR results and chemical analysis of spec-

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Table 1. Overview of several important characterization methods and their power for analysis of oxidation states; – = poor, ○ = fair, + = good, ++ = excellent.

method	species	defects	structure	sensitivity
NMR	–	–	+	–
Mössbauer	+	–	+	–
ESCA	+	–	○	–
Optical	++	+	+	++
ESR	++	++	+	++

Table 2. Important oxidation states that can be analyzed by ESR. The species marked by RT can usually be detected at room temperature. To analyze the LT-indexed elements, low temperature measurements have to be carried out.

Ti _{RT} ³⁺	V _{RT} ⁴⁺	Cr _{RT} ^{3+/5+}	Mn _{RT} ²⁺	Fe _{RT} ³⁺	Co _{LT} ²⁺	Ni _{LT} ²⁺	Cu _{RT} ²⁺
Zr _{RT} ³⁺	Nb	Mo _{RT} ^{3+/5+}	Tc	Ru	Rh	Pd	Ag
Hf	Ta	W _{RT} ^{3+/5+}	Re	Os	Ir	Pt	Au
Ce _{LT} ³⁺	Pr _{LT} ³⁺	Nd _{LT} ³⁺	Pm _{LT} ³⁺	Sm _{RT} ³⁺	Eu _{RT} ³⁺	Gd _{LT} ³⁺	Tb _{LT} ³⁺

ies has been published yet. Although in the past many ESR investigations have been carried out on laboratory melts, only a few industrial glasses have been analyzed in detail. This work discusses the possible application of ESR spectroscopy in the glass industry by focussing on routine interpretation, the correlation to chemical procedures and quantitative analysis.

The main object was to compare the wet chemical analysis with the results of ESR spectroscopy and to show the detection limits of the methods. For this purpose we have prepared laboratory melts with different concentration of transition metal ions. Further, we have restricted our work to the quantitative measurements of the iron and chromium content in glasses. Iron is the main impurity in glasses due to the raw materials, and the redox ratio of Fe²⁺/Fe³⁺ plays an important role in the coloration of green and amber glasses. Chromium has been critically discussed in view of its toxic effect [7 and 8]. Therefore, there is a great need on the part of the container glass industry to develop a reliable and sensitive detection method for Cr⁶⁺ ions in the ppm range.

2. Experimental procedures

2.1 Preparation of the glasses

Two series of glasses with different contents of iron and chromium were prepared based on a model glass, with the composition (in wt%): 76 SiO₂, 14 Na₂O, 10 CaO. Batches to give 300 g of glass were prepared from reagent-grade Na₂CO₃, CaCO₃ and SiO₂ powder. The batches were melted in platinum crucibles in an electric

furnace in normal atmosphere at 1500°C for 6 h. When the melting was completed, the glass was poured into a graphite form and annealed at 550°C for 2 h. Finally the glass blocks were pulverized in an agate ball mill. Iron and chromium as Fe₂O₃ and Cr₂O₃ were added to this basic frit to provide glasses with approximately 0.01; 0.02; 0.05; 0.1; 0.25; 0.5; 0.75 and 1.0 wt% total iron and chromium. 50 g of every composition were mechanically mixed and melted in platinum crucibles at 1400°C in normal atmosphere also for 6 h. The melt was poured again into a graphite form and annealed at 550°C for 2 h.

The Fe²⁺ and the Cr⁶⁺ concentrations were analyzed with the methods developed at our institute by Hahn [9 and 10]. These methods were tested in a round robin experiment performed by the subcommittee "Glass Analysis" of Technical Committee I: Physics and Chemistry of Glass of the German Society of Glass Technology (DGG). For the Fe²⁺ analysis o-phenantroline is added during the cold conditioning of the glass. The o-phenantroline complexates the Fe²⁺ ions and shifts the redox potential over the oxygen redox potential, so that the analysis need not be carried out under inert gas atmosphere. To determine the concentration of the Cr⁶⁺ ions, fine glass powder (100 µm) is dissolved in a mixture of HF/H₂SO₄. Diphenylcarbazide solution is added, mixed and the molecular absorption of Cr⁶⁺ is measured photometrically at 540 nm. The results of the measured Fe³⁺ and Cr⁶⁺ concentrations are collected in table 3.

For comparison we also investigated iron and chromium containing glasses prepared by the Institut für Silikattechnik, Technische Universität Freiberg (Germany) [8]. The basic glass composition was (in wt%): 70 SiO₂, 20 Na₂O, 10 CaO. The wet chemically determined Fe³⁺ and Cr⁶⁺ concentrations are listed also in table 3 (sample nos. F1 to F21).

2.2 ESR measurements

We used a Bruker ESP300E spectrometer with X-band microwave frequency (9 to 10 GHz). For accurate measurements a double rectangular resonator was chosen. It allows one to compare the spectrum of an unknown sample with a reference sample in the same cavity under identical experimental conditions. Glass powder samples with different grain size fractions were tested. Quartz glass tubes were used as sample holders. To examine the effect of different ESR parameters on the signal intensity *I* and the line width ΔH of the Fe³⁺ signal at *g* = 4.3 (figure 1), we varied the modulation amplitude, the modulation frequency and the microwave power. For this purpose one parameter was varied and all the other parameters were fixed. In that way optimum conditions for the ESR measurements were found (table 4). Also the influence of the sample preparation, particle size and the sample weight of the glass powder were investigated.

Table 3. Fe³⁺ and Cr⁶⁺ content of the glasses; sample nos. F1 to F12 were prepared by Technische Universität Freiberg.

sample no.	[Fe ³⁺] given as [Fe ₂ O ₃] in wt%	[Cr ⁶⁺] given as [Cr ₂ O ₃] in wt%
1	0.008	0
2	0.016	0
3	0.037	0
4	0.196	0
5	0.356	0
6	0.560	0
7	0	0.003
8	0	0.011
9	0	0.007
F1	0	0.006
F2	0	0.060
F3	0	0.070
F4	0.001	0.069
F5	0.09	0.0009
F6	0.23	0.038
F7	0.23	0.048
F8	0.23	0.06
F9	0.317	0.0013
F10	0.324	0.0012
F11	0.340	0.016
F12	0.340	0.053
F13	0.42	0.018
F14	0.47	0.046
F15	0.80	0.0026
F16	0.82	0.026
F17	0.88	0.0024
F18	0.90	0.038
F19	0.90	0.038
F20	0.95	0.0024
F21	0.001	0.069

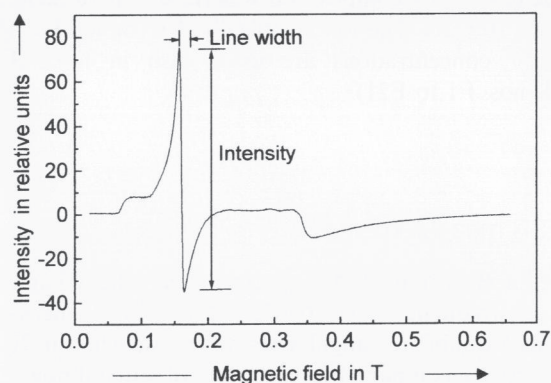
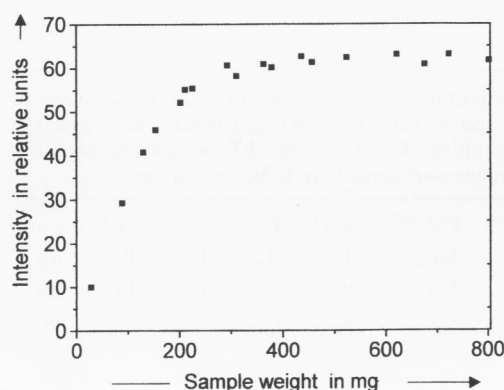


Figure 1. ESR spectrum of an iron-containing glass.

Best results were obtained when the glass was ground and a grain size fraction 75 to 150 μm was sieved. The intensity of the Fe³⁺ signal at $g = 4.3$ increases with increasing sample weight up to 300 mg. In this case the maximal filling height of the resonator is reached at about 300 mg (figure 2). For each concentration the measurement was repeated four times, so that the statistical error could be estimated. The comparison of the

Table 4. ESR instrument parameter.

microwave power	63.6 mW
modulation frequency	100 kHz
modulation amplitude	0.1 mT
delay time	10.24 ms
resolution	0.114 mT

Figure 2. Intensity of the Fe³⁺ signal in dependence on the sample weight.

measurements with 100 and 600 mg glass powders shows that the deviations are much smaller in the case of 600 mg. Therefore, all the following ESR measurements were carried out with a sample quantity of 600 mg.

3. Results

3.1 ESR spectra of Fe³⁺

Figure 3 shows the first derivative of ESR spectra of glasses with different Fe³⁺ concentrations. With increasing content also the signals at $g = 2$ and $g = 4.3$ increase. For lower concentrations (0.1 to 0.25 wt%) the signal at $g = 4.3$ shows a stronger increase compared with the signal at $g = 2$. At higher concentrations (>0.25 wt%) the $I_{4.3}/I_2$ ratio decreases, that means the signal at $g = 2$ becomes more and more distinctive. This observation is in accordance with the investigations of Camara and Rüssel [11 and 12].

To determine the signal intensity and the line width, only the signal at $g = 4.3$ was used and compared with the wet chemically determined Fe³⁺ concentrations. The line width ΔH and the peak-to-peak value ΔI were measured because these signal values can be detected very easily and exactly.

The area under the ESR signal is a measure for the number of the paramagnetic centers N and with that for the Fe³⁺ concentration. At lower concentrations the peak shows a Lorentzian line distribution, at higher con-

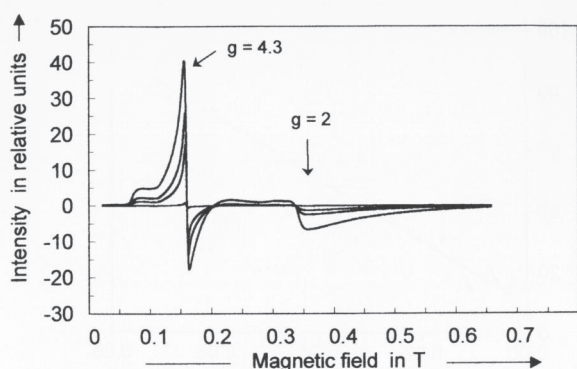


Figure 3. ESR spectra of glasses with different Fe^{3+} content (0.196, 0.356, 0.560 wt%).

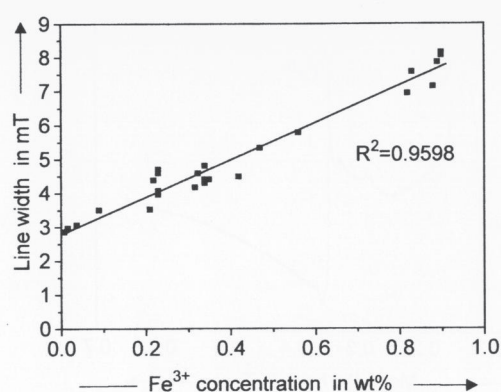


Figure 5. Line width ΔH of the Fe^{3+} signal in dependence on the Fe^{3+} concentration.

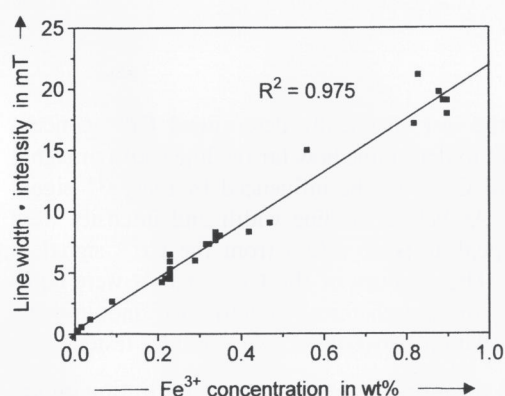


Figure 4. Product of line width ΔH and intensity ΔI of the Fe^{3+} signal in dependence on the Fe^{3+} concentration.

centration a Gaussian line form. The area is, in the former case, determined by the product line width times signal intensity, in the latter by the square of the line width times the signal intensity. Both possibilities were checked to find which of the two approximations agrees with the actual data.

In the second case the intensity of the ESR signal is proportional to

$$I_E \sim \Delta I \cdot (\Delta H)^2.$$

The comparison of the calculated signal intensities with the wet chemical results yielded no linear correlation. In contrast to this the relationship

$$I_E \sim \Delta I \cdot \Delta H$$

shows a linear dependence on the concentration of the Fe^{3+} ions with a regression coefficient of determination $R^2 = 0.975$ (figure 4).

The reason for this is the line width of the signal. The line width of the Fe^{3+} signal at $g = 4.3$ shows an obviously linear coherence (figure 5) in dependence on the Fe^{3+} concentration. The linear increase of the line

width is explained by the dipole-dipole interaction of the transition metal ions. The line width is determined by the interaction of the electron spin with the surrounding media by the spin-lattice and the spin-spin interaction. The strength of the interaction determines two characteristic lifetimes t_1 associated with the spin-lattice interaction and t_2 with the spin-spin interaction. The whole lifetime t is given by:

$$\frac{1}{t} \cong \frac{1}{t_1} + \frac{1}{t_2}.$$

The line width is proportional to $1/t$. $1/t_1$ determines the natural line width of the ESR signal and is independent of the concentration of the Fe^{3+} ions. The spin-spin interaction term $1/t_2$ depends on the amount of the dipole-dipole interaction. The linear dependence on the ion concentration shown in figure 5 means that the dipole-dipole interaction is also proportional to the Fe^{3+} content. This result is in accordance with theoretical investigations of Kittel et al. [13 and 14]. The linear relationship is only given for a purely statistical distribution of the Fe ions in the glass network. As our results show that the linear dependence exists up to a concentration of 0.95 wt%, the formation of iron clusters can be excluded. Normally iron clusters induce spin exchange interaction that lead to a nonlinear behavior. The line width shown in figure 5 can now be described by the following linear relation:

$$\Delta H = \Delta H_0 + a \cdot c(\text{Fe}^{3+})$$

where $c(\text{Fe}^{3+})$ is the Fe^{3+} concentration in wt% and a is the proportional factor. ΔH_0 means the natural line width which is the intersection of the correlation line with the abscissa at 2.75 mT. The important result of our investigation is the correlation between the ESR signal $I_E = \Delta I \cdot \Delta H$ and the Fe^{3+} concentration $c(\text{Fe}^{3+})$ in form of the following linear calibration curve (figure 4):

$$I_E = b \cdot c(\text{Fe}^{3+})$$

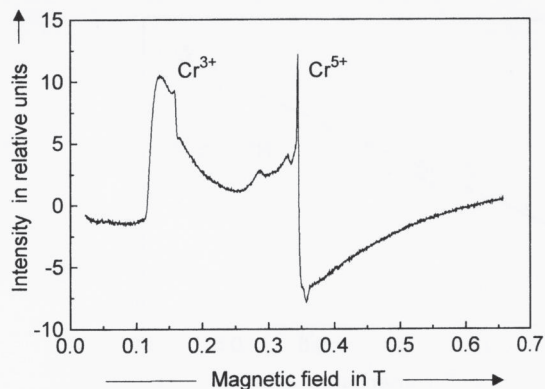


Figure 6. ESR spectrum of a chromium-containing glass.

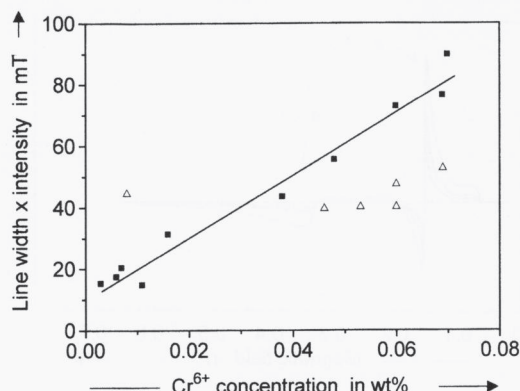
which allows the determination of the Fe^{3+} content in the glass matrix via ESR measurements. The linear correlation is valid between 0 to 0.95 wt%, which covers the whole range of Fe^{3+} concentration in practice. With the help of statistical analysis of linear regression methods the lower detection limit can be estimated for the ESR spectroscopic analysis of Fe^{3+} concentration. Assuming a 95 % confidence interval the detection limit is about 0.02 wt%.

The linear relationship between the ESR signal and the Fe^{3+} concentration holds for all measured samples although the glasses differ in their basic compositions of SiO_2 , Na_2O and CaO . This is an important result for practical applications because container glasses always differ slightly with respect to their compositions. Also the chromium ions have no influence on this result.

The ESR spectroscopy allows a quantitative determination of the Fe^{3+} concentration without and with chromium ions. But it is necessary to calibrate the system carefully. To minimize the error of wet chemical analysis, the concentrations of each calibration glass have to be measured several times to obtain a reliable mean value. Further, it is important to use exactly the same quantity of the glass sample both for determination of the calibration line and for the measurement of the unknown glass sample.

3.2 ESR spectra of Cr^{5+}

Unlike Fe^{3+} ions it is not possible to detect Cr^{6+} ions directly with the help of ESR spectroscopy. The ESR spectra of chromium ion containing glasses show two prominent ESR signals at $g = 5$ and $g = 1.98$ (figure 6). The broad peak at $g = 5$ belongs to Cr^{3+} and the sharp line at $g = 1.98$ to Cr^{5+} . As, however, in glasses the Cr^{6+} concentration is in equilibrium with the Cr^{5+} concentration, it should be possible to determine the Cr^{6+} content via the Cr^{5+} ESR signal. Therefore, the objective was to prove the correlation between the Cr^{5+} ESR sig-

Figure 7. Product of line width ΔH and intensity ΔI of the Cr^{5+} signal in dependence on the Cr^{6+} concentration; \blacksquare = low iron content, \triangle = high iron content.

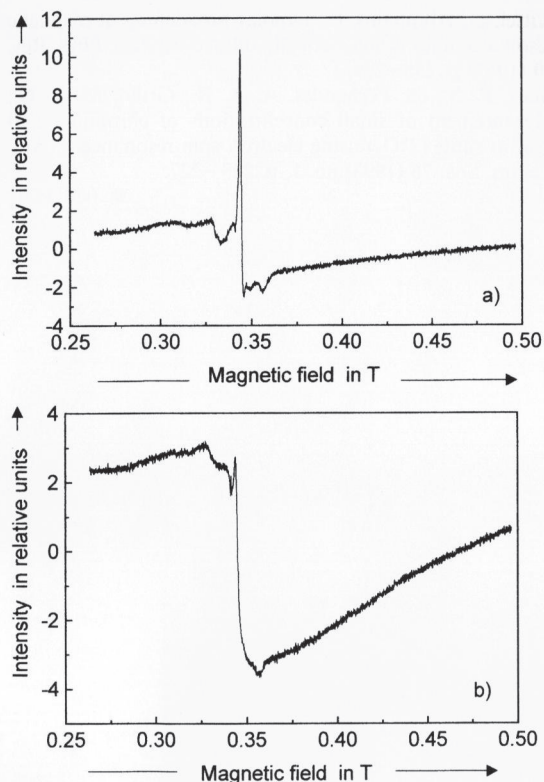
nal and the wet chemically determined Cr^{6+} concentration and to determine how far the line width or signal intensity of Cr^{5+} will be influenced by the Fe^{3+} signal at $g = 2$. As before the line width and intensity were taken as peak-to-peak values from the Cr^{5+} signal at $g = 1.98$. These values of the Cr^{5+} signals were compared with the wet chemically determined data and the linear correlation between the two variables tested.

To test the influence of the iron concentration on the line width, only the ESR spectra of the iron-free glasses (table 3: nos. 7 to 9 and F1 to F21) were recorded. The result was that in contradiction to iron the line width of the Cr^{5+} signal reveals no significant dependence on the iron concentrations. That means in the case of chromium ions the dipole-dipole interaction is too weak in order to influence the lifetime.

The comparison of the product $\Delta I \cdot \Delta H$ with the concentration of the wet chemically determined Cr^{6+} ions reveals a more complicated correlation as shown in figure 7. Compared to iron no universal straight line exists. To understand this behavior one has to distinguish between three types of glasses: glasses without iron, glasses with low iron content and glasses with high iron content relative to the chromium concentration. Glasses without iron or with low iron content show a linear dependence on the Cr^{6+} concentration. Deviations from this dependence appear in connection with high iron contents.

This behavior is mainly due to the superimposition of the Cr^{5+} signal with the Fe^{3+} signal at $g = 2$ (figures 8a and b). In the case of glasses without iron or with low iron content the ESR signal of Fe^{3+} is very low. Therefore, it is possible to separate the Cr^{5+} signal from the broad Fe^{3+} line and to measure the signal intensity very precisely.

Also the evaluation of the signal intensities is possible for glasses with medium Cr^{6+} and Fe^{3+} concentration, though the statistical uncertainty becomes



Figures 8a and b. ESR spectra of chromium-containing glass with low iron content; figure a) sample no. F4, figure b) sample no. F12.

stronger. As figure 8b clearly shows a determination of the Cr^{5+} content and therefore of the Cr^{6+} content is impossible for high iron contents. The linear dependence exists if the ratio of $\text{Fe}^{3+}/\text{Cr}^{6+}$ is < 6 . At higher values the statistical uncertainty is too high for a precise result. The lower detection limit depends on the iron content in a decisive way. For iron-free glasses this value is about 0.005 wt% Cr^{6+} .

4. Conclusions

The aim of this work was to show if ESR spectroscopy can be used as a quantitative analytical instrument for practical purposes in the glass industry. The necessary studies were carried out on glasses with iron and chromium content due to their industrial importance. For the special case of iron this could obviously be proved.

For the ESR signal at $g = 4.3$ the product of line width times signal intensity shows a linear dependence on the wet chemically determined Fe^{3+} content. This proportional relationship holds for the Fe^{3+} concentration region between 0 to 0.95 wt% and is independent of the $\text{Fe}^{3+}/\text{Cr}^{6+}$ ratio. No influence of the ESR signal of the chromium ions on the signal of iron at $g = 4.3$ was observed. Therefore ESR spectroscopy allows a

quantitative measurement of the Fe^{3+} concentration. The lowest detection limit for Fe^{3+} is 0.02 wt%. The line width itself also shows a linear dependence on the Fe^{3+} content. This phenomenon is explained by the dipole-dipole interaction of the iron ions.

Cr^{6+} ions, the detection of which is important for the container glass industry due to their toxicity, can not be proved by ESR spectroscopy. Therefore, an indirect proof was tested, namely the linear correlation of the wet chemically determined Cr^{6+} concentration with the intensity of the Cr^{5+} ESR signal. The comparison of the results of the ESR-spectroscopic measurements with the Cr^{6+} concentrations shows a more complicated relationship compared to the case of iron. For iron-free glasses the linear correlation is excellent. The detection limit is about 0.005 wt% for this case. For glasses with iron the ESR spectra show a superimposition of the Cr^{5+} signal at $g = 1.98$ with the broad Fe^{3+} signal at $g = 2$. This effect decisively influences the detection limits. A linear correlation holds only for a $\text{Fe}^{3+}/\text{Cr}^{5+}$ ratio < 6 .

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