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MÖSSBAUER STUDY OF THE FERRO - GALLIC INK FROM A XV CENTURY MANUSCRIPT

by

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Mössbauer absorbers were made from fragments of the damaged part of the manuscript, by assembling together about 25 to 50 paper sheets. The percentage of absorption was found to be between 1 and 3%.

From 295K to 30K the absorption spectrum exhibits two quadrupole split doublets, arising from Fe^{2+} and Fe^{3+} ions, whose relative proportions are respectively 45 and 55%. At 1.4K the component arising from the Fe^{2+} ions is split, giving a single magnetic hyperfine pattern from which all hyperfine parameters were derived by computer fitting. At this temperature the Fe^{3+} fraction is present as a quadrupole split doublet and a Zeeman sextet. The Fe^{2+} ion magnetic hyperfine interaction vanishes at about 20K, which is thus the magnetic ordering temperature of the corresponding phase. The ratio (Fe^{3+} magnetic)/(Fe^{3+} total) goes from 41% at 1.4K to 20% at 10K and decreases slowly at higher temperatures. At 4.2K an external field of 55KG markedly increases the magnetic fraction of the Fe^{3+} spectrum and aligns the corresponding magnetic moments. These results show that the Fe^{3+} ions form a superparamagnetic phase, probably with very small particle size, or even amorphous, and this phase could be related to the black colour of the ink. The phase containing the Fe^{2+} ions has been identified as ferrous oxalate FeC_2O_4, $2H_2O$. The presence of oxalate is particularly interesting since it is probably connected to the degradation of parts of the manuscript.

We have also investigated by Mössbauer spectroscopy and X-ray diffraction inks which have been prepared according to ancient recipes and have compared the results with those from ancient inks.

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Fitted hyperfine parameters for the ink of the manuscript.

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Mössbauer absorbers were made from fragments of the damaged part of the manuscript, by assembling together about 25 to 50 paper sheets. The percentage of absorption was found to be between 1 and 3%.

From 295K to 30K the absorption spectrum exhibits two quadrupole split doublets, arising from Fe^{2+} and Fe^{3+} ions, whose relative proportions are respectively 45 and 55%. At 1.4K the component arising from the Fe^{2+} ions is split, giving a single magnetic hyperfine pattern from which all hyperfine parameters were derived by computer fitting. At this temperature the Fe^{3+} fraction is present as a quadrupole split doublet and a Zeeman sextet. The Fe^{2+} ion magnetic hyperfine interaction vanishes at about 20K, which is thus the magnetic ordering temperature of the corresponding phase. The ratio (Fe^{3+} magnetic)/(Fe^{3+} total) goes from 41% at 1.4K to 20% at 10K and decreases slowly at higher temperatures. At 4.2K an external field of 55KG markedly increases the magnetic fraction of the Fe^{3+} spectrum and aligns the corresponding magnetic moments. These results show that the Fe^{3+} ions form a superparamagnetic phase, probably with very small particle size, or even amorphous, and this phase could be related to the black colour of the ink. The phase containing the Fe^{2+} ions has been identified as ferrous oxalate FeC_2O_4, $2H_2O$. The presence of oxalate is particularly interesting since it is probably connected to the degradation of parts of the manuscript.

We have also investigated by Mössbauer spectroscopy and X-ray diffraction inks which have been prepared according to ancient recipes and have compared the results with those from ancient inks.

T(K)	contributions	IS(mm s^{-1})	QS(mm s^{-1})	H_{hf}(Koe)	P(%)
295	paramagnetic Fe^{3+}	0.27	0.59	—	55
	paramagnetic Fe^{2+}	1.08	-1.73	—	45
1.4	paramagnetic Fe^{3+}	0.41	0.68	—	32.2
	magnetic Fe^{3+}	0.42	——	535	22.7
	magnetic Fe^{2+}	1.23	-2.04	150	45.1

Fitted hyperfine parameters for the ink of the manuscript.