



## Iron oxidation state in impact glass from the K/T boundary at Beloc, Haiti, by high-resolution XANES spectroscopy

Gabriele GIULI,<sup>1\*</sup> Sigrid Griet EECKHOUT,<sup>2</sup> Eleonora PARIS,<sup>1</sup>  
Christian KOEBERL,<sup>3</sup> and Giovanni PRATESI<sup>4, 5</sup>

<sup>1</sup>Dipartimento di Scienze della Terra and INFN, Università di Camerino, Via Gentile III da Varano, 62032 Camerino, Italy

<sup>2</sup>European Synchrotron Radiation Facility (ESRF), 6 rue Jules Horowitz, BP 220, 38043 Grenoble, France

<sup>3</sup>Department of Geological Sciences, University of Vienna, Althanstrasse 14, A-1090 Vienna, Austria

<sup>4</sup>Museo di Storia Naturale, Sezione di Mineralogia e Litologia, Università di Firenze, Via G. la Pira 4, 50121 Firenze, Italy

<sup>5</sup>Museo di Scienze Planetarie della Provincia di Prato, Prato, Italy

\*Corresponding author. E-mail: [gabriele.giuli@unicam.it](mailto:gabriele.giuli@unicam.it)

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**Abstract**—We examined the local iron environment in nine impact glasses from the Cretaceous-Tertiary (K/T) boundary section at Beloc, Haiti, which formed as the result of impact melting during the Chicxulub impact event. The samples have been analyzed by Fe K-edge high-resolution X-ray absorption near edge structure (XANES) spectroscopy to obtain data on both the Fe oxidation state and the coordination number. The pre-edge peak of our high-resolution XANES spectra display noticeable variations indicative of significant changes in the Fe oxidation state spanning a wide range from about 20 to 75 mol% trivalent Fe. All data plot along the same trend, falling between two mixing lines joining a point calculated as the mean of a group of tektites studied so far (consisting of four- and five-coordinated Fe<sup>2+</sup>) to [<sup>4</sup>]Fe<sup>3+</sup> and [<sup>5</sup>]Fe<sup>3+</sup>, respectively. Thus, the XANES spectra can be interpreted as a mixture of [<sup>4</sup>]Fe<sup>2+</sup>, [<sup>5</sup>]Fe<sup>2+</sup>, [<sup>4</sup>]Fe<sup>3+</sup>, and [<sup>5</sup>]Fe<sup>3+</sup>. There is no evidence for six-fold coordinated Fe; however, its presence in small amounts cannot be excluded from XANES data alone. Our observations can be explained by two possible scenarios: either these impact glasses formed under very reducing conditions and, because of their small size, were easily oxidized in air while still molten, or they formed under a variety of different oxygen fugacities resulting in different Fe oxidation states. In the first case, the oxidation state and coordination number would imply similar formation conditions as splash-form tektites, followed by progressive oxidation.

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### INTRODUCTION

Sigurdsson et al. (1991a, b), Izett (1991), and Kring and Boynton (1991) have described the presence of relict glass within alteration spherules from the K/T boundary at Beloc, Haiti, and interpreted the material as impact glass. This was confirmed by Koeberl and Sigurdsson (1992) and Koeberl (1992). Sigurdsson et al. (1992) demonstrated that the Haitian glasses have been quenched from temperatures much higher than is common for volcanic processes. Koeberl and Sigurdsson (1992) provided not only detailed geochemical data for the impact origin of these glasses but also demonstrated the existence of rare inhomogeneous glasses with lechatelierite and other mineral inclusions, which are typical for an origin by impact. Oxygen isotope studies by Blum and Chamberlain (1992) specifically rule out a volcanic origin of these glasses, and Blum et al. (1993), from Rb-Sr

and Sm-Nd isotopic data, showed that the Haitian glasses are mixtures of silicate rocks of upper crustal composition with a high CaO-endmember (e.g., limestone) (cf. also Premo and Izett [1992] and Bohor and Glass [1995]).

Chaussidon et al. (1994) demonstrated that the sulfur in the yellow glasses occurs in the form of sulfate, again incompatible with a volcanic source. Koeberl (1992) measured the water content in glasses from Haiti and found low, impact-characteristic abundances (0.013 to 0.021 wt% H<sub>2</sub>O), and Koeberl et al. (1994) have used Re-Os isotope systematics to detect a small meteoritic component in the Haitian glasses. The Haitian glasses are different from tektites in some respects; for example, they are more oxidized than tektites (Oskarsson et al. 1996).

Glasses with similar properties have later been recovered from some K/T boundary locations in Mexico (e.g., Mimbrol) as well (e.g., Smit et al. 1992a, b). It is interesting to note that,

despite all the data favoring an impact origin of the Haiti glasses, a few authors have suggested that the glasses are of volcanic origin (e.g., Stinnesbeck et al. 1993) or that there is no evidence at all for impact deposits at Beloc (e.g., Jéhanno et al. 1992). However, there is abundant evidence on the impact origin of these glass spherules based on water content (Koeberl 1992) and geochemistry (Koeberl and Sigurdsson 1992; Koeberl 1996).

X-ray absorption spectroscopy allows structural and chemical determinations and has the advantage over other spectroscopic techniques of also being used to study trace elements or complex chemical systems. The information obtained by XANES on the glasses is interpreted by comparison with data available for well-characterized model compounds. High-resolution XANES spectra give structural data on the local geometry around the selected element and a quantitative evaluation of the different oxidation states (Wilke et al. 2001).

### SAMPLES AND EXPERIMENTAL METHODS

The samples studied have been collected from the K/T boundary layer outcrops along the Jacmel highway in the Beloc region (Haiti) (Sigurdsson et al. 1991a, b). The glass is usually altered to smectite spherules; however, in some cases, relict glass is preserved in the interior of the spherules. To remove any adhering soil or other contamination, the glassy parts of the spherules were immersed for a few minutes in dilute HCl and subsequently cleaned in deionized water in an ultrasonic bath. The glass spherules examined are of the black glass type, with diameters in the 1 to 3 mm range. Complete spherules were analyzed (not fragments of larger samples). The glasses were embedded in epoxy and sectioned; polishing was done with standard diamond paste. More details about sample preparation can be found in Koeberl and Sigurdsson (1992). No micro-crystals or residual smectite were found after optical microscopic examination. The samples analyzed here were the same as those analyzed by Koeberl and Sigurdsson (1992).

Chemical compositions were obtained by electron microprobe (see Koeberl and Sigurdsson 1992) and are reported in Table 1.

The standards used for XANES measurements are a staurolite from Canton Ticino (Switzerland) and a synthetic Fe akermanite for Fe<sup>2+</sup> in tetrahedral coordination, a grandidierite from Madagascar for Fe<sup>2+</sup> in trigonal bipyramidal coordination, a synthetic kirschsteinite and a siderite from Erzberg (Austria) for Fe<sup>2+</sup> in octahedral coordination, a natural andradite for Fe<sup>3+</sup> in octahedral coordination, and a natural yoderite from Mautia Hills (Tanzania) for Fe<sup>3+</sup> in five-fold coordination. The natural standards were separated by hand picking from thumb-sized crystals choosing the clearest portions to avoid impurities. All the standards were checked for purity by both optical microscopy and X-ray diffraction.

Model compounds for XAS measurement were prepared by smearing finely ground powder on a kapton tape, while the K/T glass spherule samples were prepared as microprobe mounts. The flat surface was placed at 45° from the X-ray beam directed toward the fluorescence detector. Fe K-edge XANES spectra were recorded at room temperature at the ESRF on the undulator beamline ID26 (Gauthier et al. 1999; Solé et al. 1999) operating at 6 GeV in four bunch mode for samples one to five, and in 2 × 1/3 filling for samples six to ten. A fixed-exit Si(220) double-crystal monochromator was used, providing an energy resolution of ~0.4 eV at the Fe K-edge. However, the main limitation for energy resolution is the finite core-hole width of the absorbing element (~1.15 eV at the Fe K-edge; Krause and Oliver 1979), resulting in a convoluted energy resolution (FWHM) of ~1.4 eV. The energy was calibrated by defining the first derivative peak of a metallic Fe reference foil to be at 7112.0 eV. Two Si mirrors were used for the harmonics rejection of the incident X-ray beam. The beam dimension at the sample was 400 × 100 μm. Before each measurement, care was taken in sample centering so as to have the beam pointing at the sample's geometric center. XANES data were recorded in quick-scan mode by

Table 1. Major element compositions of black impact glasses from Beloc (Haiti).<sup>a</sup>

	H-1	H-2	H-3	H-4	H-5	H-6	H-7	H-8	H-10
SiO <sub>2</sub>	62.25	65.07	65.45	62.82	63.53	61.42	63.30	86.00	62.86
TiO <sub>2</sub>	0.71	0.62	0.63	0.69	0.63	0.71	0.68	0.47	0.67
Al <sub>2</sub> O <sub>3</sub>	15.06	15.31	15.29	15.05	15.61	15.40	15.31	6.93	15.16
FeO	5.40	5.14	5.03	5.34	4.87	5.32	5.44	2.45	5.33
MnO	0.18	0.17	0.18	0.17	0.17	0.17	0.18	0.05	0.19
MgO	2.77	2.28	2.35	2.63	2.33	2.75	2.58	1.15	2.64
CaO	8.12	5.77	5.65	7.56	6.72	8.39	7.23	0.38	7.54
Na <sub>2</sub> O	3.78	3.73	3.72	3.92	4.08	4.09	3.65	0.38	3.86
K <sub>2</sub> O	1.59	1.71	1.55	1.65	1.78	1.52	1.50	2.21	1.59
P <sub>2</sub> O <sub>5</sub>	0.07	0.09	0.02	0.09	0.10	0.06	0.06	0.02	0.08
S	0.004	0.003	0.002	0.002	0.002	0.002	0.001	0.002	0.004
Total	99.91	99.90	99.87	99.92	99.84	99.84	99.93	100.04	99.93

<sup>a</sup>Data in wt%, after Koeberl and Sigurdsson (1992).

simultaneously scanning the monochromator angle and the undulator gap with a typical energy step of 0.25 eV and counting 20 ms per point. Each scan took 30 sec and the average of 15 spectra was taken. The spectra were acquired in fluorescence mode, using a Si photo-diode, and  $I_0$  was monitored by measuring the fluorescence signal of a titanium foil using a Si photo-diode.

## DATA REDUCTION

Experimental XANES spectra were reduced by background subtraction with a linear function and then normalized for atomic absorption on the average absorption coefficient of the spectral region from 7150 to 7300 eV. The threshold energy was taken as the first maximum of the first derivative of the spectra. Pre-edge peak analysis was carried out following the same procedure reported in Wilke et al. (2001) and Giuli et al. (2002, 2003). The pre-edge peak was fitted by a sum of pseudo-Voigt functions and the integrated intensities along with centroid energies were compared with those of the model compounds analyzed here and others in the literature (Wilke et al. 2001; Farges 2001) to extract

information on Fe oxidation state and coordination number in the glasses studied.

## RESULTS

XANES spectra of the Fe model compounds are shown in Fig. 1a, while those of the K/T impact glass spherule samples are shown in Fig. 1b. The XANES spectra of all the K/T impact glass samples are less structured than those of the standards because of the amorphous nature of the former producing an absence of a long-range order. Apart from the different overall shape, due to the different structure of the model compounds, clear differences can be noted in the energy region before 7120 eV, called the pre-edge region. The peak located in the pre-edge region (the pre-edge peak) is the most useful feature to discriminate the oxidation state and coordination number of Fe (Fig. 2). This peak represents an  $1s-3d$  like transition and is, thus, dipole-forbidden but becomes partially allowed by mixing of the  $d$ -states of the transition metal with the  $p$ -states of the surrounding oxygen atoms. Its energy position depends mainly on the mean Fe oxidation state, gradually increasing from  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$ , while

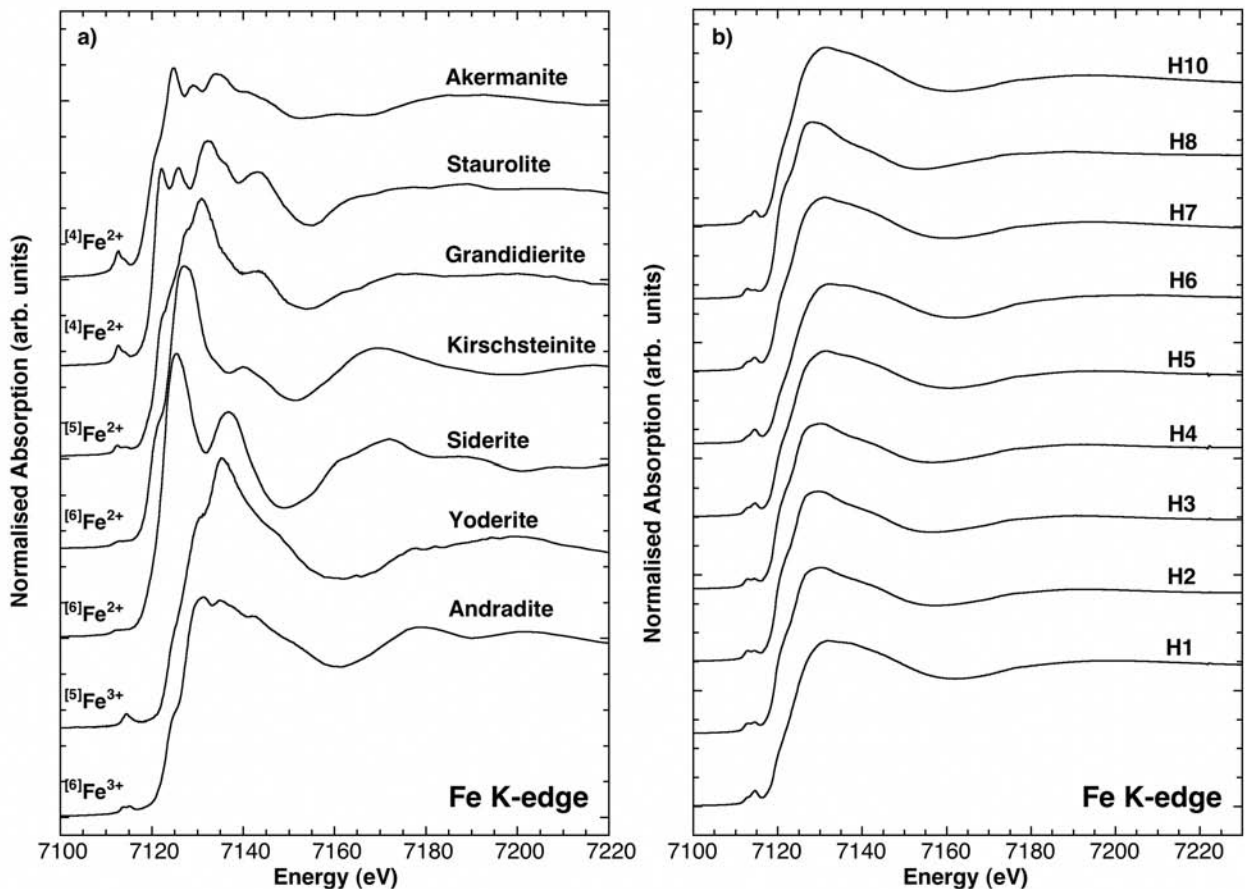


Fig. 1. Experimental Fe K-edge XANES spectra of: a) model compounds with Fe in different oxidation states and coordination numbers; b) K/T glass spherules. The spectra have been normalized setting the step height to one.

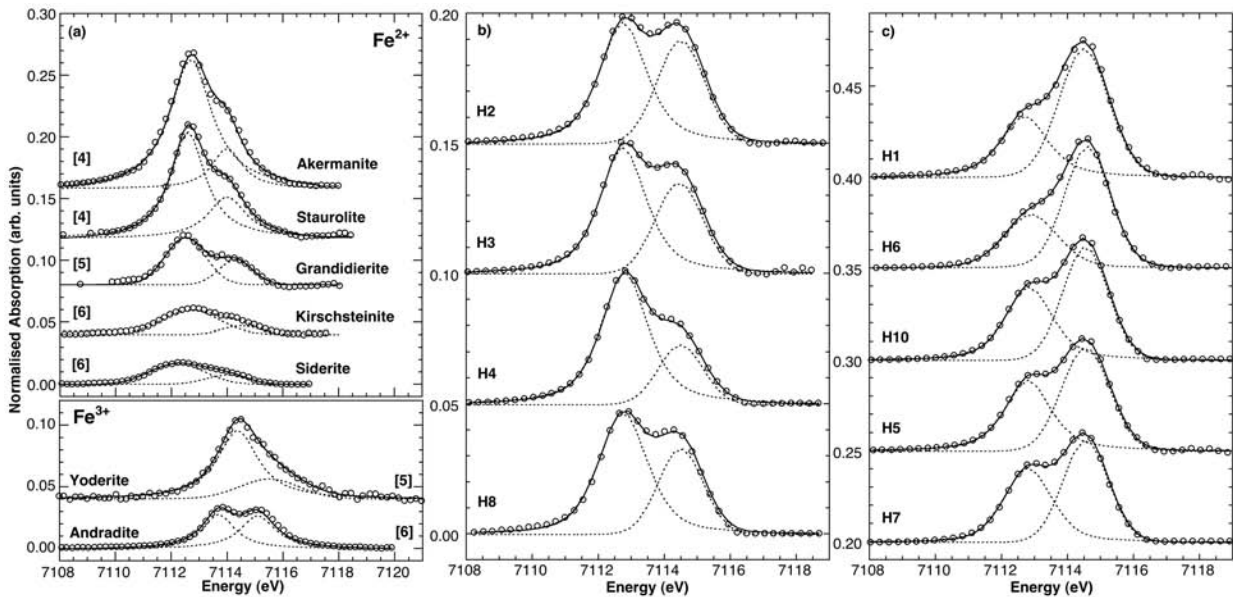


Fig. 2. Fit of the pre-edge peak of: a) model compounds with Fe in different oxidation states and coordination numbers; b) and c) K/T glass spherules.

its intensity depends on the local geometry around Fe (Calas and Petiau 1983; Brown et al. 1995; Wilke et al. 2001), so that it will be virtually zero in the case of regular octahedral symmetry around the absorber, while it will reach its maximum in the case of tetrahedral coordination. For these characteristics of the XANES spectra, we analyzed model compounds chosen to represent a variety of oxidation states (from  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$ ) and coordination numbers (from 4 to 6). Figure 2 (b and c) displays how the pre-edge peak varies as a function of these parameters in the K/T impact glass samples and also shows the results of the pre-edge peak deconvolution into single pseudo-Voigt components.

The pre-edge peaks of almost all the samples display two components. By calculating the pre-edge peak centroid and the integrated intensities (reported in Table 2), these data can be compared with those of the model compounds with known Fe oxidation states and coordination numbers (Wilke et al. 2001; Farges 2001; Giuli et al. 2002, 2003) to evaluate the contribution of the different Fe oxidation states and coordination numbers in our samples.

All the pre-edge peak data (integrated intensity versus centroid energy) are plotted in Fig. 3, where the positions of model compounds are also shown. One may notice that all the samples plot along a well-defined trend, and can be bracketed between the two mixing lines shown in the figures: the mixing lines are calculated from a point representative of divalent Fe in a mixture of five-fold and four-fold coordinated sites (which is taken as the mean of more than 20 tektites from the four known Cenozoic tektite strewn fields) and extending up to purely trivalent Fe in five-fold and four-fold coordination respectively. All the data shown can thus be interpreted in

Table 2. Pre-edge peak features of the Fe K-edge XANES spectra.

Sample name	Centroid (eV) <sup>a</sup>	Integrated intensity	Fit agreement index (%)
Siderite	7112.8	0.054	99.84
Kirschsteinite	7113.0	0.062	99.83
Grandidierite	7113.0	0.101	99.32
Staurolite	7113.0	0.218	99.88
Fe akermanite	7112.9	0.283	99.84
Andradite	7114.4	0.107	99.93
Yoderite	7114.3	0.169	99.91
H1	7113.9	0.202	99.97
H2	7113.4	0.169	99.98
H3	7113.3	0.153	99.95
H4	7113.4	0.138	99.96
H5	7113.8	0.179	99.97
H6	7114.0	0.182	99.97
H7	7113.7	0.171	99.98
H8	7113.2	0.142	99.97
H10	7113.8	0.182	99.98

<sup>a</sup>Precision and accuracy of the pre-edge peak centroid energy are  $\pm 0.05$  and  $0.1$  eV, respectively.

terms of different amounts of divalent and trivalent iron in five-fold and four-fold coordination. In particular, the Fe oxidation state spans a wide range from 20–70 mol%  $\text{Fe}^{3+}$  ( $\text{Fe}^{2+} + \text{Fe}^{3+}$ ).

## DISCUSSION AND CONCLUSIONS

XANES spectroscopy has been used to characterize the Fe local structure and oxidation state in a set of nine impact

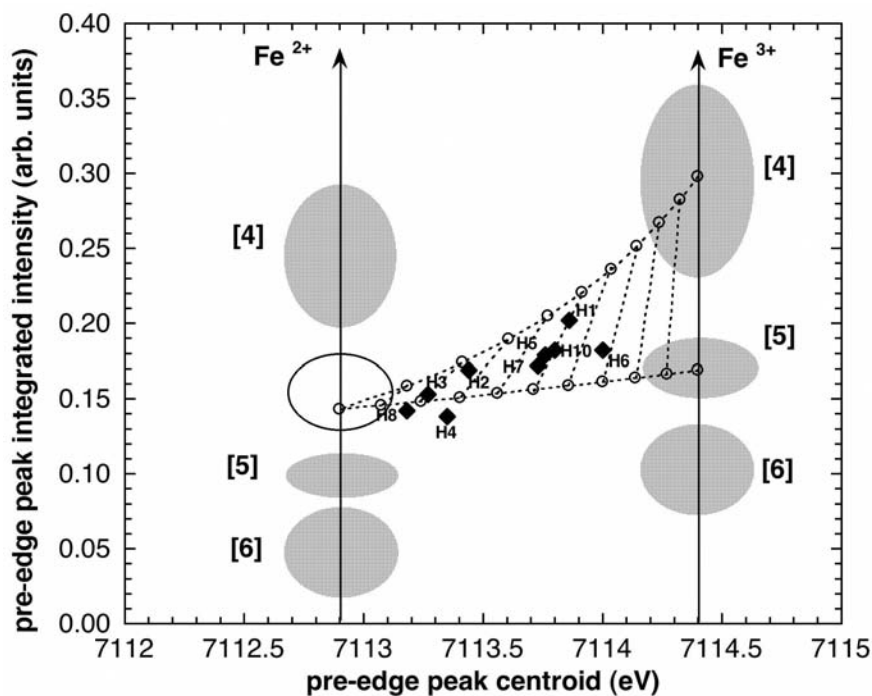


Fig. 3. Plot of the pre-edge peak integrated intensity versus centroid energy. The solid symbols refer to the samples studied here (diamonds = K/T glass spherules). The unshaded open ellipse on the left side indicates the field of data occupied by all splash-form tektites analyzed so far (Giuli et al. 2002; and Giuli et al., unpublished data). Also shown are the mixing lines (dashed lines + circles) between  $^{4}\text{Fe}^{3+}$  or  $^{5}\text{Fe}^{3+}$  and the mean of about 20 tektites analyzed (see text).

glass samples from the K/T boundary of Beloc (Haiti). Eight of the nine impact-glasses studied (which are all of the “black” type) display a relatively narrow range of compositions, while one sample (H8) has an unusually high Si content, and belongs to a new type of K/T impact glass referred to as High Si-K glass by Koeberl and Sigurdsson (1992).

The pre-edge peaks of the samples studied display a wide range of variations both in the energy position and in the integrated intensity. Comparison with spectral data from model compounds with known structure allows us to estimate the relative percentages of divalent and trivalent iron according to the procedure reported in the literature (Willke et al. 1991; Giuli et al. 2002, 2003). The oxidation state of the samples has been found to vary between 20 and 70 mol% trivalent iron. Such a wide range of Fe oxidation states in K/T impact glasses has never been reported in the literature, and thus, the conclusions reported in Oskarsson et al. (1996) should be revisited in the view of these new data.

It is interesting that nearly all pre-edge peak data form a trend pointing toward a point calculated by averaging data on tektites from the four Cenozoic strewn fields (Giuli et al. 2002 and unpublished data). In particular, the pre-edge peak data can be bracketed by two mixing lines: one between the tektite average and tetracoordinated  $\text{Fe}^{3+}$ , the other between the tektite average and five-fold coordinated  $\text{Fe}^{3+}$ . These data can be interpreted as resulting from the mixing of  $\text{Fe}^{2+}$  (in five- and

four-coordination) and  $\text{Fe}^{3+}$  (in five- and four-coordination) in various amounts. However, the presence of minor amounts of six-fold coordinated Fe cannot be excluded from these data.

The fact that this trend points to the average of tektites (prevalent  $\text{Fe}^{2+}$  in four- and five-fold coordination) could be explained by two possible scenarios:

1. these impact glasses were formed under very reducing conditions (as for splash-form tektites) and then, due to their small size, were oxidized in air while still in a molten state; or
2. these impact glasses were formed under a variety of different oxygen fugacities resulting in different Fe oxidation states.

Which of these scenarios is applicable cannot be decided from the present data alone. However, the target stratigraphy at Chicxulub indicates that the source rocks from which the Beloc black impact glasses were derived (and with which they show geochemical similarities; e.g., Blum et al. 1993) were not close to the surface at the time of the impact, while tektites in the Cenozoic strewn field were derived from near-surface material (e.g., Ma et al. 2004). Explosive decomposition of the overlying carbonate and evaporite rocks may have created zones of widely varying oxygen fugacities.

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