Analytical Conditions for the Secondary Ion Mass Spectrometry (SIMS)

SIMS analyses were undertaken to determine the concentration of the minor rare-earth elements in the individual doped rare earth element (REE) glasses. With the purity of the rare earth starting material being typically 99.9%, it is proposed that this could result in glasses having a high concentrations of many of the rare earth elements, in addition to the dominant REE, and make them unsuitable as standard materials.

Samples were mounted in epoxy resin (Buehler, Epo-thin) ground and polished (6µm and 1µm diamond) to a flat surface. The sample was then cleaned and coated with 30nm layer of Au.

The concentrations of the REEs were determined using a CAMECA IMS-4f ion microprobe with a PXT interface and control system at the University of Edinburgh. A 5nA, focussed primary beam of O- was accelerated onto the polished sample surface with a net impact energy of ~14.5keV. High energy (120ev +/- 25eV) positive secondary ions were detected using an electron multiplier.

The analysed isotopes of the REE were selected to avoid the REE hydride (LaH), REE oxide (LaO and LaO2) Si+REE (SiLa), Al+REE (AlLa) combinations formed with the dominant REE of the glass. The isotopes selected are given in the tables below, together with an estimate of the error in the measurement based on counting statistics.

In some cases no interference-free isotope was available for analysis. Under these conditions the corrections were considered too large for the concentration of the underlying minor REE to be accurately determined. In all cases, the interference of the minor REE-Oxides on the heavier REE were corrected using the REE-Oxide/REE ratio of the glasses measured at 120eV high energy offset (Fig 1).

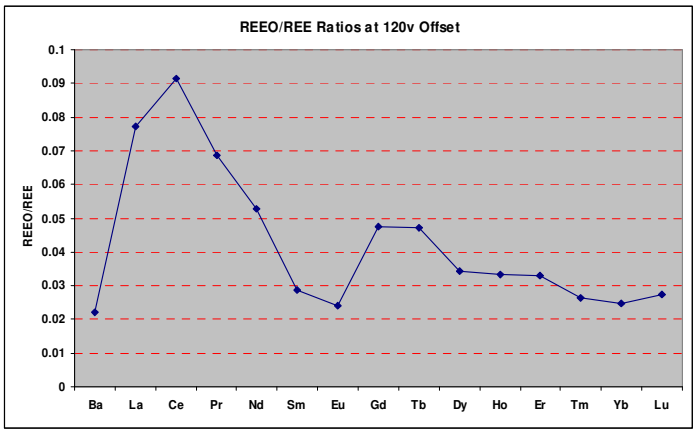

Fig1: Plot showing the ratio of the REEO/REE for the individual REE glasses. These ratios were used to correct for the interfering light REE oxide on a heavy REE.

The concentrations of the REE were calculated relative to SRM610 glass analysed under the same instrumental conditions.

Y Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.01	
Y	89	0.02	
Ba	138	7	
La	139	19	
Ce	140	7	
Pr	141	24	
Nd	143	19	
Sm	149	19	
Eu	151	70	BaO
Gd	157	16	PrO
Tb	159	25	NdO
Dy	161	5	NdO
Ho	165	18	SmO
Er	167	32	EuO
Tm	169	50	EuO
Yb	172	21	GdO, DyO
Lu	175	32	TbO

La Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	4	
Ba	138	0.6	^{138}La
La	139	0.02	
Ce	142	9	Nd
Pr	141	8	
Nd	143	15	
Sm	149	50	
Eu	151	24	BaO
Gd	157	4	PrO
Tb	159	50	NdO
Dy	161	57	NdO
Ho	165	15	SmO. Ignored minor $^{138}La^{27}Al$
Er	167		EuO + $^{139}La^{28}Si$
Tm	169		EuO + $^{139}La^{30}Si$
Yb	172	40	GdO, DyO
Lu	175	70	TbO
Pb	208	30	

Ce Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	4	
Ba	137	18	CeH
La	139	15	
Ce	140	0.03	
Pr	141	2	CeH
Nd	145	6	
Sm	149	10	
Eu	151	70	BaO
Gd	157	4	PrO
Tb	159	13	NdO
Dy	161	35	NdO
Ho	165		SmO + $^{138}Ce^{27}Al$ + $^{136}Ce^{29}Si$
Er	167		EuO + $^{140}Ce^{27}Al$ + $^{138}Ce^{29}Si$
Tm	169		EuO + $^{142}Ce^{27}Al$ + $^{140}Ce^{29}Si$
Yb	173	37	GdO
Lu	175	60	TbO
Pb	208	30	

Pr Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	9	
Ba	137	20	
La	139	6	
Ce	142	7	
Pr	141	0.02	
Nd	143	25	
Sm	149	44	
Eu	151	100	BaO
Gd	155	23	LaO
Tb	159	5	NdO
Dy	161	57	NdO
Ho	165	70	SmO
Er	167	80	EuO
Tm	169	18	EuO + $^{141}Pr^{28}Si$
Yb	172	40	GdO, DyO
Lu	175	57	TbO
Pb	208	33	

Nd Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	2	
Ba	137	20	
La	139	4	
Ce	140	10	
Pr	141	2	
Nd	143	0.05	
Sm	152	8	
Eu	153	25	BaO
Gd	155	10	LaO
Tb	159		Major NdO
Dy	163	9	SmO
Ho	165	12	SmO
Er	167	15	EuO
Tm	169	9	EuO + $^{142}Nd^{27}Al$
Yb	172		GdO + DyO + $^{145}Nd^{27}Al$ + $^{144}Nd^{29}Si$ + $^{143}Nd^{29}Si$ + $^{142}Nd^{30}Si$
Lu	175		TbO + $^{148}Nd^{27}Al$ + $^{146}Nd^{29}Si$ + $^{145}Nd^{30}Si$ + $^{143}Nd^{16}O_2$
Pb	208	40	

Sm Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	2	
Ba	137	16	
La	139	10	
Ce	140	14	
Pr	141	18	
Nd	143	7	
Sm	149	0.06	
Eu	151	14	BaO + $^{150}Sm^{1}H$
Gd	156	4	CeO + Dy
Tb	159	15	NdO
Dy	161	20	NdO
Ho	165		Major SmO
Er	167	14	EuO
Tm	169	11	EuO
Yb	172		GdO + DyO + $^{144}Sm^{28}Si$
Lu	175		TbO + $^{148}Sm^{27}Al$ + $^{147}Sm^{28}Si$
Pb	208	19	

Eu Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	13	
Ba	137	18	
La	139	1	
Ce	140	13	
Pr	141	9	
Nd	143	17	
Sm	149	21	
Eu	151	0.03	BaO
Gd	155	4	LaO
Tb	159	2	NdO
Dy	162	12	NdO
Ho	165	6	SmO
Er	166	16	SmO + NdO
Tm	169		Major EuO
Yb	172	13	GdO + DyO
Lu	175	0.8	TbO
Pb	208	35	

Gd Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	2	
Ba	137	20	
La	139	3	
Ce	140	4	
Pr	141	4	
Nd	143	11	
Sm	149	24	
Eu	151	7	BaO
Gd	155	0.06	LaO
Tb	159	4	NdO
Dy	162	21	Er + NdO
Ho	165	11	SmO
Er	167	26	EuO
Tm	169	18	EuO
Yb	172		Major GdO + DyO
Lu	175	4	TbO
Pb	208	28	

Tb Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	2	
Ba	137	15	
La	139	17	
Ce	140	18	
Pr	141	18	
Nd	143	33	
Sm	149	57	
Eu	151	18	BaO
Gd	155	7	LaO
Tb	159	0.02	NdO
Dy	162	3	Er + NdO
Ho	165	4	SmO
Er	167	6	EuO
Tm	169	25	EuO
Yb	172	6	GdO + DyO
Lu	175		Major TbO
Pb	208	57	

Dy Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	6	
Ba	137	15	
La	139	5	
Ce	140	5	
Pr	141	5	
Nd	143	20	
Sm	149	70	
Eu	151	8	BaO
Gd	155	15	LaO
Tb	159	4	$^{158}Dy^{1}H$ + NdO
Dy	162	0.04	Er + NdO
Ho	165	2	$^{164}Dy^{1}H$ + SmO
Er	167	4	SmO + NdO
Tm	169	1	EuO
Yb	171	3	GdO
Lu	175	2	TbO
Pb	208	13	$^{164}Dy^{44}Ca$ + $^{162}Dy^{46}Ca$ + $^{160}Dy^{48}Ca$

Ho Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	3	
Ba	137	14	
La	139	14	
Ce	140	15	
Pr	141	70	
Nd	143	44	
Sm	149	30	
Eu	151	100	BaO
Gd	155	3	LaO
Tb	159	5	NdO
Dy	162	3	Er + NdO
Ho	165	0.02	SmO
Er	167	4	SmO + NdO
Tm	169	16	EuO
Yb	171	7	GdO
Lu	175	23	TbO
Pb	208	4	$^{165}Ho^{43}Ca$

Er Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	6	
Ba	137	20	
La	139	8	
Ce	140	4	
Pr	141	25	
Nd	143	100	
Sm	149	45	
Eu	151	14	BaO
Gd	155	26	LaO
Tb	159	13	NdO
Dy	162	0.7	Er + NdO
Ho	165	9	SmO + $^{164}Er^{1}H$
Er	167	0.05	SmO + NdO
Tm	169	5	EuO + $^{157}Er^{1}H$
Yb	171	2	GdO
Lu	175	6	TbO
Pb	208	3	$^{168}Er^{40}Ca$ + $^{166}Er^{42}Ca$ + $^{164}Er^{44}Ca$ + $^{162}Er^{46}Ca$

Tm Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	14	
Ba	137	14	
La	139	9	
Ce	140	28	
Pr	141	22	
Nd	143	35	
Sm	149	35	
Eu	151	2	BaO
Gd	155	25	LaO
Tb	159	5	NdO
Dy	162	18	Er + NdO
Ho	165	16	SmO
Er	167	6	SmO + NdO
Tm	169	0.02	EuO
Yb	171	4	GdO
Lu	175	6	TbO
Pb	208	40	

Yb Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	5	
Ba	137	21	
La	139	12	
Ce	140	26	
Pr	141	18	
Nd	143	25	
Sm	149	30	
Eu	151	3	BaO
Gd	155	18	LaO
Tb	159	15	NdO
Dy	162	14	Er + NdO
Ho	165	13	SmO
Er	167	11	SmO + NdO
Tm	169	9	EuO + ^{168}YbH
Yb	171	0.06	GdO
Lu	175	2	TbO + ^{174}YbH
Pb	208	35	$^{168}Yb^{40}Ca$ + $^{176}Yb^{16}O_2$

Lu Glass: Analytical Conditions

Element	Mass Used	Estimated % Error	Major Interferences Corrections
Si	30	0.1	
Y	89	4	
Ba	137	16	
La	139	6	
Ce	140	10	
Pr	141	6	
Nd	143	21	
Sm	149	20	
Eu	151	16	BaO
Gd	155	5	LaO
Tb	159	5	NdO
Dy	162	50	Er + NdO
Ho	165	25	SmO
Er	167	23	SmO + NdO
Tm	169	1	EuO
Yb	171	6	GdO
Lu	175	0.03	TbO
Pb	208	20	$^{176}Lu^{16}O_2$