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Ages of several xenotime megacrysts by ID-TIMS: potential reference materials for ion microprobe U-Pb geochronology

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Abstract: Four museum specimens of xenotime were tested for homogeneity in their U-Pb ages using isotope dilution – thermal-ionization mass spectrometry (ID-TIMS). The U-Pb ages of the samples ranged from about 500 Ma to 1020 Ma. Sample z6413, from a granitic pegmatite in the Grenville Province, yielded homogeneous $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages of 994 Ma and 997 Ma, respectively. This sample was selected as a primary candidate reference material for ion microprobe U-Pb geochronology. A second sample, z6410, yielded $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages of 897 Ma and 901 Ma, respectively, and is a potential secondary standard. The other samples displayed significant age heterogeneities, including the effects of Pb loss, making them unsuitable as standards.

Résumé : Quatre spécimens de musée de xénotime ont été analysés par dilution isotopique et spectrométrie de masse à thermoionisation afin d'évaluer l'homogénéité de leurs âges U-Pb, qui s'échelonnent entre 500 Ma et 1020 Ma environ. L'échantillon z6413, qui provient d'une pegmatite granitique de la Province de Grenville, a livré des âges $^{206}Pb/^{238}U$ et $^{207}Pb/^{206}Pb$ homogènes de 994 Ma et 997 Ma, respectivement. Cet échantillon a été choisi comme candidat idéal pour devenir matériau de référence pour la datation U-Pb par microsonde ionique. Un deuxième échantillon, z6410, a livré des âges $^{206}Pb/^{238}U$ et $^{207}Pb/^{206}Pb$ de 897 Ma et 901 Ma, respectivement, et pourrait devenir un étalon secondaire. Les autres échantillons affichent des hétérogénéités d'âge substantielles, dues en partie à la perte de Pb, qui les rendent impropres à servir d'étalons.

INTRODUCTION

Xenotime [(Y, HREE)PO_4] is a common accessory mineral in granitic rocks, particularly peraluminous pegmatites and migmatites, and is also found in a range of metamorphic rocks, including some low-grade metasedimentary rocks where it occurs as a diagenetic mineral. Like zircon ($ZrSiO_4$), xenotime is tetragonal and possesses MO_8 polyhedra alternating with MO_4 tetrahedra (xenotime: $REEO_8$ + PO_4; zircon: ZrO_8 + SiO_4). The MO_8 polyhedra also accommodate the smaller lanthanide ions (Y and HREEs) in preference to the larger ones (LREEs), opposite to the case for monazite ((LREE)PO_4) with its larger MO_9 polyhedra (Ni et al., 1995). Actinide-element (U, Th) abundances of several hundred to a few thousand parts per million are typical for xenotime. These levels are generally higher than for zircon, but compared with monazite, xenotime generally has similar or lower U contents, and much lower Th. Incorporation of actinides in xenotime involves coupled substitutions with Si or Ca (Van Emden et al., 1997; Förster, 1998).

Ion microprobe U-Pb geochronology (e.g. SHRIMP; Stern, 1997) of xenotime requires the use of natural xenotime reference materials in order to correct for inherent discrimination of Pb and U secondary ions. A minimum requirement for such reference materials is their homogeneity in $^{206}Pb/^{238}U$ ratios as determined by isotope dilution – thermal-ionization mass spectrometry (ID-TIMS) on bulk samples. In this report, ID-TIMS U-Pb data are reported for several xenotime specimens, only one of which met this minimum requirement.

DESCRIPTION OF MINERAL SPECIMENS

Four xenotime megacrysts were obtained: three from the National Mineral Collection of Canada (NMCC) Systematic Reference Series, which is held at the GSC in Ottawa (samples z6414, z6410, z6409), courtesy of Dr. R. Herd, and one from the Canadian Museum of Nature (z6413), courtesy of Dr. S. Ercit.

z6413

Alternate names for this sample, used in preliminary examination, are 'XENO1' and 'PM3.' It is recommended that usage of these names be discontinued.

We obtained a hand sample of granite pegmatite (PM3-A, Fig. 1a) containing numerous large xenotime crystals. The pegmatite body is found within the Purdy #3 Mine, near Purdy Lake, Mattawan Township, Ontario, Canada (approximately UTM zone 17, 661400E, 5131700N), within the Mesoproterozoic Grenville Province. Major minerals present within the pegmatite are quartz, feldspar, apatite, xenotime, and fergusonite. A dark reddish-brown, subequant, semi-transparent, multifaceted xenotime crystal measuring ~7 mm across (total mass 0.81 g) was extracted from the pegmatite matrix. The crystal was subsequently fragmented using a tool-steel mortar and pestle. The crystal fragments typically appear transparent and pale yellow-brown under the binocular microscope (Fig. 2a).

Backscattered-electron (BSE) imaging reveals the xenotime to be broadly zoned (Fig. 2b), and ion microprobe analysis confirms that the brighter areas correspond with higher uranium contents. The BSE images of some fragments also show the presence of 1 to 5 µm uraninite inclusions (Fig. 2c and inset). Although heterogeneously distributed, the

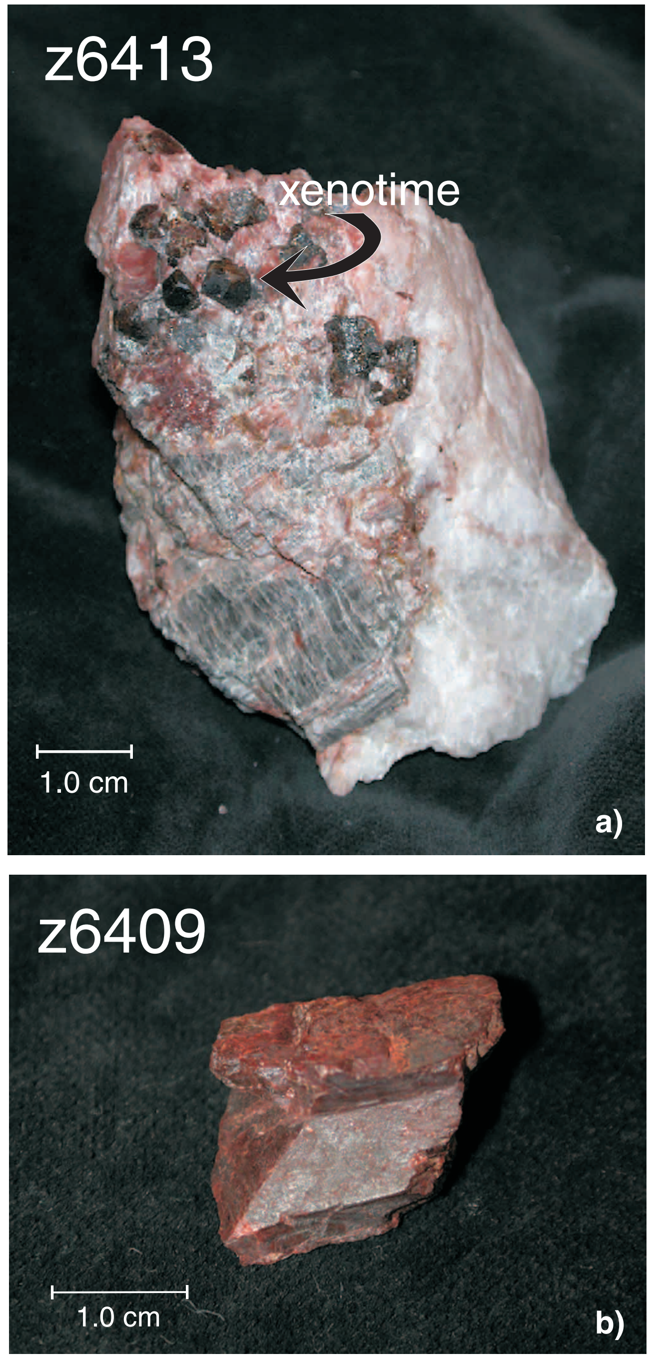

Figure 1. *Photographs of two of the xenotime megacryst samples tested as potential ion microprobe standards: (**a**) z6413 and (**b**) z6409. The typical reddish-brown lustre is apparent. Xenotime z6413 proved suitably homogeneous in U-Pb isotopes.*

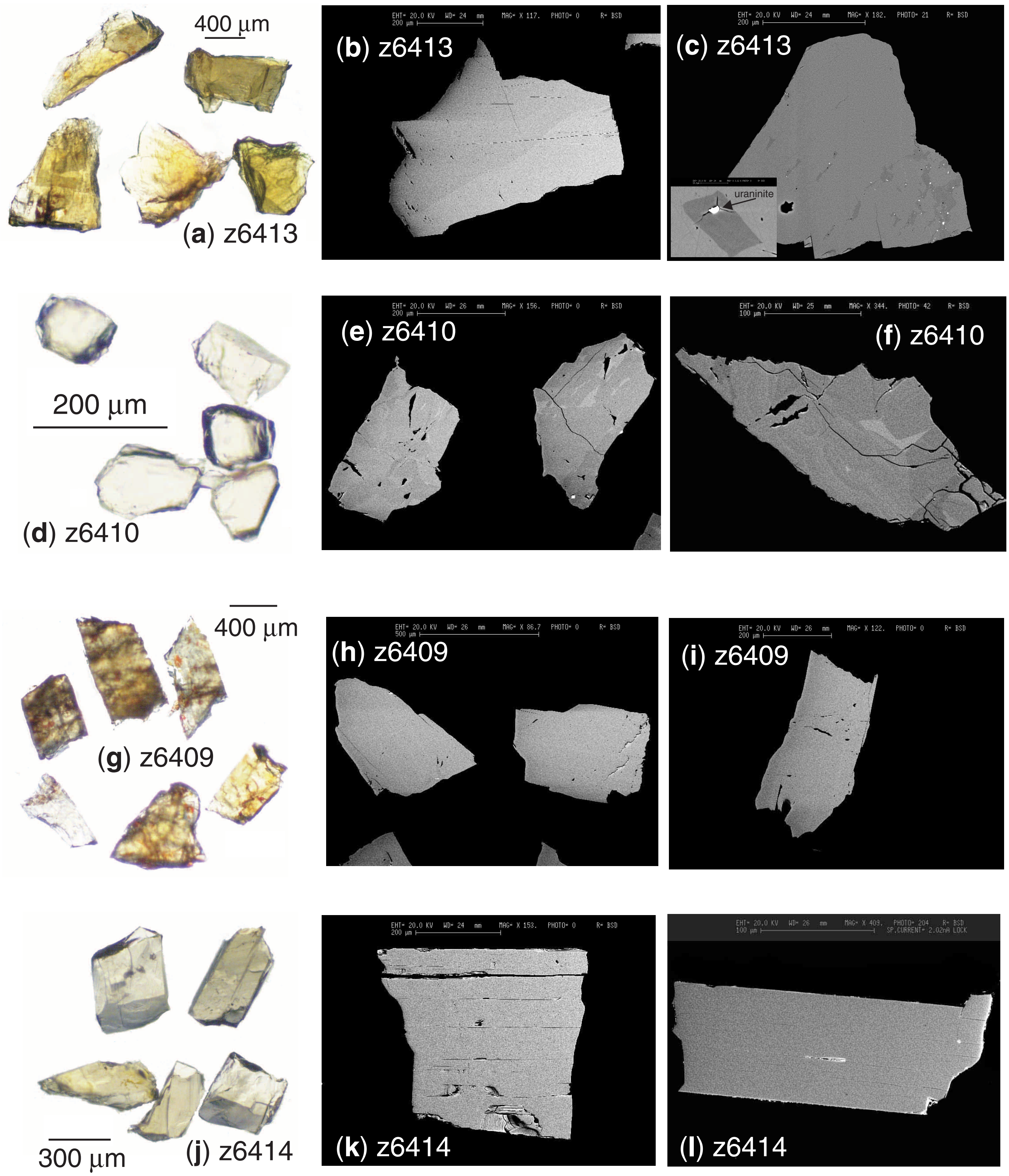

Figure 2. *Transmitted-light and backscattered-electron micrographs of xenotime fragments. (**a, b, c**) z6413; note broad zoning corresponding with variations in U abundance in (**b**) and numerous uraninite inclusions (bright specks) associated with darkened xenotime zones in (**c**). (**d, e, f**) z6410. (**g, h, i**) z6409. (**j, k, l**) z6414.*

uraninite makes up < 0.05‰ by volume of most fragments. The xenotime immediately surrounding the uraninite inclusions has anomalously dark BSE response (Fig. 2c and inset).

z6410

Alternate names for this sample are 'XENO2' and 'N3' (usage to be discontinued), and 18947 (NMCC number).

The geological origin of the sample is not known in detail, but it was collected from the Aust-Agder county, southern Norway, which is underlain by rocks of the Proterozoic Sveconorwegian Province. A fragment with mass 2.1 g contained several twins with well developed prismatic faces. The xenotime was pale brown, opaque, with a resinous lustre. The crystal fragments are colourless to pale yellow and transparent under the binocular microscope (Fig. 2d). BSE images show irregular, patchy zoning (Fig. 2e, 2f).

z6409

Alternate names for this sample are 'XENO4' and 'NP1' (usage to be discontinued), and 18950 (NMCC number).

The exact origin of this sample is unknown, but it was collected from Calvin Township, Nipissing District, Ontario, Canada (approximately NTS zone 17, 659500E, 5127500N). The geological locale is similar to that of z6414 (described below), and the sample is also likely of pegmatitic origin. The single crystal (Fig. 1b; total mass 6.7 g) was prismatic, with well developed facets on two sides; its colour was a dark, rusty brown, with a resinous, almost submetallic lustre. Crystal fragments under the binocular microscope range from colourless to pale yellow-orange, and opaque inclusions are common (Fig. 2g). BSE images lack obvious zoning (Fig. 2h, 2i).

z6414

Alternate names for this sample are 'XENO3' and 'NHX' (usage to be discontinued), and 67514 (NMCC number).

The origin of this sample is not known in detail, but it was collected from the Novo Horizonte area, Bahia, Brazil, and is likely from a granitic pegmatite. The specimen was a single, medium brown, translucent, elongate, twinned crystal measuring 3 x 5 x 11 mm. A coating of yellowish, powdery mineral was present in some of the cracks within the crystal. After fragmentation, the yellowish coating was removed using an ultrasonic bath. Crystal fragments observed under the binocular microscope are colourless to pale yellow-brown and have good cleavage (Fig. 2j). The BSE images appear featureless (Fig. 2k, 2l).

ANALYTICAL METHODS

Individual fragments with masses of 2 to 181 μg were selected for spiking, acid dissolution, and column chemistry. No particular selection criteria were applied other than avoiding fragments with obvious inclusions. Fragments were not air abraded. U-Pb chemistry, including dissolution in concentrated HCl, and mass spectrometry followed the methods used for monazite, as described by Parrish et al. (1987). Analytical blanks for Pb were 4 to 10 pg over the course of this study. Analytical errors are quoted at the 95% confidence level in the text and concordia plots, and at the 68% confidence level in Table 1.

RESULTS

z6413

U-Pb data for five fragments with masses of 2 to 4 μg are presented in Table 1 and shown in Figure 3a. All data are analytically indistinguishable, having a weighted mean $^{207}Pb/^{206}Pb$ age of 996.7 ± 0.8 Ma (MSWD = 0.67; probability, P, = 0.61) and mean $^{206}Pb/^{238}U$ age of 993.8 Ma ± 0.7 Ma (MSWD = 0.27, P = 0.90). The xenotime appears discordant by about 0.3% relative to the origin. Common-Pb content was negligible, i.e. mostly contributed by the analytical procedures. The U, Th, and total radiogenic-Pb contents ranged from 1300 to 6700 ppm, 230 to 1180 ppm, and 211 to 1080 ppm, respectively, whereas Th/U ratios were quite uniform at ~0.18.

Although only fragments appearing free of inclusions were selected, the presence of small uraninite inclusions cannot be ruled out. From independent evidence (i.e. electron- and ion-microprobe analysis), it is known that the total radiogenic-Pb content of uraninite of this age would be about 12 weight per cent. Assuming 0.05‰ uraninite by volume and 750 ppm Pb in xenotime, it is calculated that a maximum of 0.8% of the Pb in the TIMS analyses could be attributed to the uraninite inclusions. As there is no correspondence between the U contents of the fragments and their discordance, it is unlikely that the small amount of discordance can be attributed to uraninite inclusions.

z6410

Six fragments with masses ranging from 4 to 10 μg vary from concordant to 1.5% discordant (Table 1; Fig. 3b). Linear regression yields an upper intercept of 901 +3/-2 Ma (MSWD = 1.14, P = 0.33; lower intercept = 157 +215/-209 Ma), which is the best estimate of the crystallization age. Excluding the most discordant fraction (*see* Table 1: z6410, fraction 1), the mean $^{206}Pb/^{238}U$ age is 897 Ma. Although the $^{206}Pb/^{238}U$ data scatter well beyond analytical error, this value is nevertheless a potentially useful reference age for the purpose of ion microprobe standardization. Common-Pb contents were negligible to slightly above analytical blanks. The U, Th, and total-Pb contents range from 665 to 1150 ppm, 700 to 1700 ppm, and 119 to 226 ppm, respectively, and Th/U ranges from 1.1 to 1.6.

z6409

Nine fragments with masses ranging from 2 to 181 μg were analyzed; the data are presented in Table 1 and illustrated in Figure 4a. Eight of the analyses scatter slightly below and

Table 1. TIMS U-Pb data for potential xenotime reference materials.

Fraction	No. of grains	Wt. (µg)	U^2 (ppm)	Th^m (ppm)	Th^m/U	Pb^{*2} (ppm)	Pb_C^1 (pg)	$^{206}Pb^1/^{204}Pb$	$^{208}Pb^2/^{206}Pb$	$^{207}Pb^2/^{235}U$	$\pm ^{207}Pb/^{235}U$	$^{206}Pb^2/^{238}U$	$\pm ^{206}Pb/^{238}U$	Corr. coeff.	$^{207}Pb^2/^{206}Pb$	$\pm ^{207}Pb/^{206}Pb$	Apparent ages (Ma) $^{206}Pb/^{238}U$	$\pm ^{206}Pb/^{238}U$	$^{207}Pb/^{235}U$	$\pm ^{207}Pb/^{235}U$	$^{207}Pb/^{206}Pb$	$\pm ^{207}Pb/^{206}Pb$	Disc. (%)
z6413																							
1	1	2.2	4580	850	0.186	741	6.7	15590	0.0565	1.6619	0.117	0.16663	0.094	0.921	0.07234	0.048	993.5	1.7	994.1	1.5	995.4	1.9	0.2
2	1	3.2	6019	1062	0.177	971	5.5	36200	0.0537	1.6633	0.113	0.16662	0.088	0.943	0.07240	0.042	993.5	1.6	994.6	1.4	997.2	1.7	0.4
3	1	2.9	6689	1179	0.176	1079	4.5	44870	0.0536	1.6630	0.113	0.16665	0.088	0.941	0.07238	0.042	993.6	1.6	994.5	1.4	996.5	1.7	0.3
4	1	4.2	1537	278	0.181	248	6.6	10172	0.0550	1.6650	0.118	0.16678	0.092	0.917	0.07241	0.050	994.3	1.7	995.3	1.5	997.3	2.0	0.3
5	1	3.6	1303	234	0.180	211	6.4	7619	0.0547	1.6645	0.121	0.16677	0.095	0.908	0.07239	0.053	994.3	1.7	995.1	1.5	996.8	2.1	0.3
z6410																							
1	1	9.9	889	1308	1.471	172	4.5	17870	0.4502	1.4023	0.114	0.14740	0.089	0.930	0.06900	0.045	886.4	1.5	889.9	1.4	898.7	1.9	1.5
4	1	5.4	1046	1554	1.486	205	11.3	4658	0.4547	1.4245	0.127	0.14949	0.095	0.872	0.06911	0.064	898.1	1.6	899.3	1.5	902.1	2.6	0.5
5	1	4.2	683	734	1.075	122	7.0	3822	0.3292	1.4159	0.130	0.14871	0.094	0.887	0.06906	0.064	893.7	1.6	895.7	1.6	900.4	2.6	0.8
6	1	3.9	1127	1837	1.630	228	6.9	5927	0.4988	1.4238	0.123	0.14956	0.096	0.881	0.06904	0.060	898.5	1.6	898.9	1.5	900.0	2.5	0.2
7	1	4.3	665	706	1.062	119	24.9	1082	0.3250	1.4141	0.188	0.14884	0.103	0.713	0.06891	0.135	894.5	1.7	894.9	2.2	895.9	5.6	0.2
8	1	5.9	1149	1706	1.485	226	23.1	2747	0.4546	1.4250	0.136	0.14973	0.096	0.858	0.06903	0.073	899.4	1.6	899.5	1.6	899.6	3.0	0.0
z6409																							
1	1	27.6	2128	2511	1.180	449	6.3	98870	0.3587	1.7271	0.115	0.17090	0.092	0.946	0.07329	0.041	1017.1	1.7	1018.7	1.5	1022.1	1.7	0.5
2	1	80.4	1893	2351	1.242	402	9.4	172000	0.3778	1.7142	0.121	0.16988	0.099	0.952	0.07318	0.041	1011.5	1.8	1013.9	1.6	1019.0	1.6	0.8
3	1	106.2	2377	2703	1.137	513	22.5	123200	0.3458	1.7798	0.127	0.17638	0.106	0.955	0.07319	0.041	1047.2	2.1	1038.1	1.7	1019.1	1.7	-3.0
4	1	180.8	2012	2443	1.214	422	16.7	228800	0.3694	1.6972	0.144	0.16885	0.126	0.963	0.07290	0.041	1005.8	2.3	1007.0	1.8	1011.2	1.7	0.6
5	1	3.4	928	1172	1.263	199	22.3	1518	0.3842	1.7276	0.152	0.17091	0.097	0.797	0.07331	0.095	1017.1	1.8	1018.9	2.0	1022.6	3.9	0.6
6	1	4.3	729	1082	1.483	176	24.7	1464	0.4510	1.7034	0.161	0.16893	0.102	0.778	0.07314	0.104	1006.2	1.9	1009.8	2.1	1017.7	4.2	1.2
7	1	1.9	1113	1332	1.197	235	24.5	932	0.3640	1.7244	0.206	0.17040	0.100	0.700	0.07340	0.154	1014.3	1.9	1017.7	2.6	1024.9	6.2	1.1
8	1	5.3	394	486	1.232	82	9.9	2210	0.3750	1.6693	0.147	0.16686	0.099	0.770	0.07256	0.095	994.8	1.8	996.9	1.9	1001.6	3.9	0.7
9	1	2.4	395	642	1.627	86	9.9	973	0.4961	1.5835	0.268	0.16105	0.210	0.580	0.07131	0.225	962.6	3.8	963.7	3.3	966.2	9.2	0.4
z6414																							
1	1	32.1	268	1749	6.521	58	4.8	8934	2.0403	0.6324	0.120	0.08032	0.094	0.860	0.05710	0.062	498.0	0.9	497.6	0.9	495.4	2.7	-0.5
2	1	17.1	154	1017	6.588	34	3.6	3722	2.0613	0.6373	0.152	0.08099	0.124	0.757	0.05707	0.100	502.0	1.2	500.7	1.2	494.3	4.4	-1.6
3	1	14.7	100	638	6.402	21	6.0	1259	2.0047	0.6332	0.342	0.08097	0.144	0.489	0.05671	0.299	501.9	1.4	498.1	2.7	480.4	13.2	-4.7
4	1	9.1	147	1025	6.979	33	76.4	104	2.1830	0.6277	1.940	0.07952	0.540	0.699	0.05726	1.609	493.2	5.1	494.7	15.2	501.4	69.3	1.7
5	1	19.7	243	1586	6.526	53	23.2	1055	2.0417	0.6353	0.213	0.08069	0.099	0.684	0.05711	0.162	500.2	1.0	499.4	1.7	495.6	7.1	-1.0
6	1	23.1	161	1058	6.589	36	20.0	968	2.0600	0.6537	0.275	0.08251	0.127	0.647	0.05746	0.216	511.1	1.2	510.8	2.2	509.4	9.5	-0.3
7	1	16.9	154	1078	7.023	34	8.2	1588	2.1989	0.6200	0.229	0.07927	0.145	0.656	0.05673	0.173	491.7	1.4	489.8	1.8	481.0	7.6	-2.3
8	1	22.9	181	1230	6.786	41	21.5	1010	2.1214	0.6535	0.211	0.08246	0.097	0.701	0.05748	0.158	510.8	1.0	510.6	1.7	510.0	6.9	-0.2

Notes

Errors are 1 SE % on ratios, 2 SE on ages

^{1} = spike- and fractionation-corrected only

^{2} = spike- fractionation- and blank-corrected

^{m} = model value calculated from $^{208}Pb/^{206}Pb$ assuming concordance between Th-Pb and U-Pb ages.

Pb_C = common Pb

Corr. coeff. = correlation coefficient

Disc. = discordance

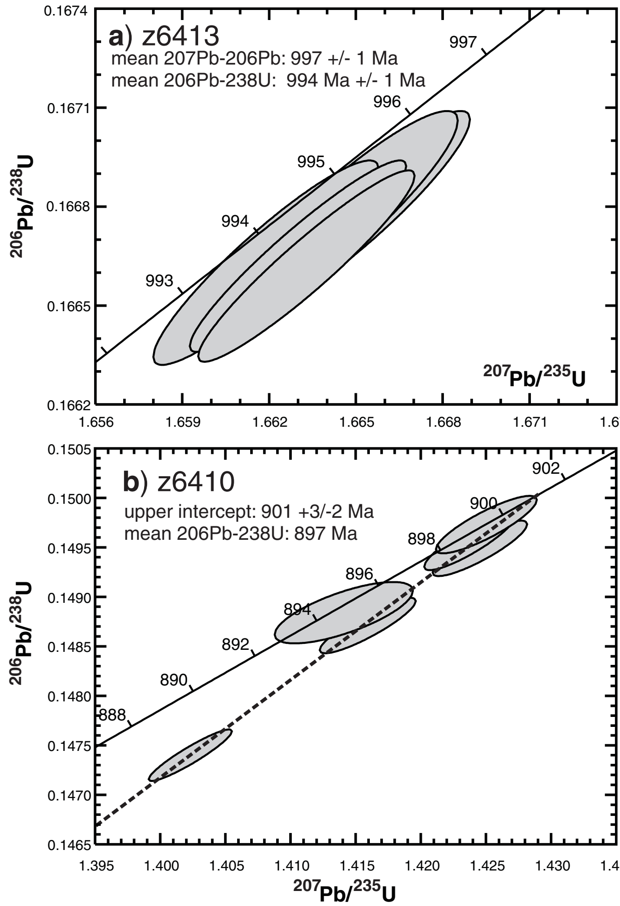

*Figure 3. TIMS U-Pb concordia plots for single fragments of xenotime samples (**a**) 6413 and (**b**) z6410. These two samples are potential ion-microprobe reference materials.*

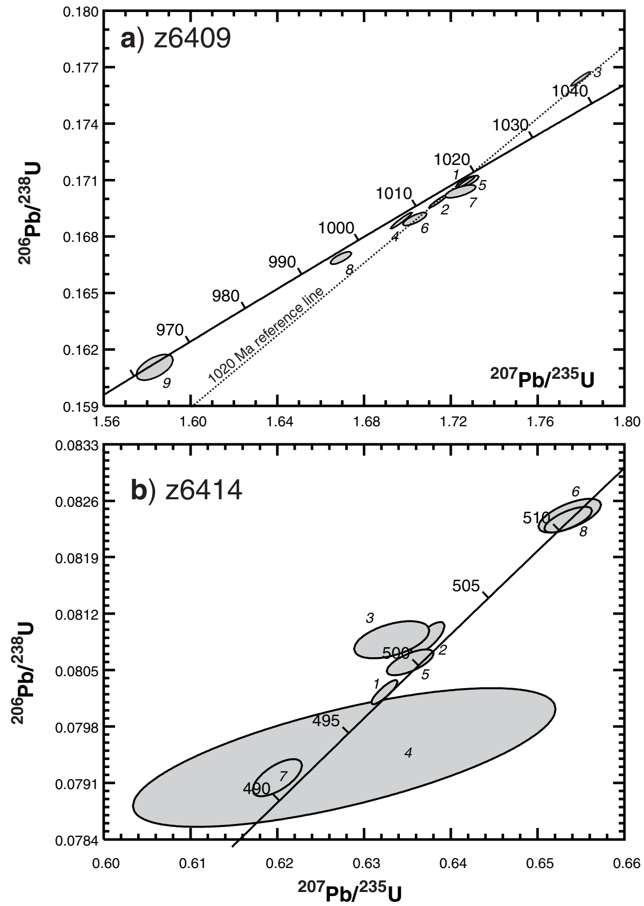

*Figure 4. TIMS U-Pb concordia plots for single fragments of xenotime samples (**a**) z6409 and (**b**) z6414. These samples are unsuitable as ion-microprobe reference materials.*

parallel to concordia between about 965 Ma and 1020 Ma. Fraction 3 is 3% reversely discordant, most likely an analytical artifact. Fractions 1, 2, 3, 5, 6, and 7 have a mean ^{207}Pb/^{206}Pb age of 1020 ± 2 Ma (MSWD = 2.9, P = 0.014), which may approximate the original age of the xenotime, assuming Pb loss was dominantly of zero age. However, ancient Pb loss may be indicated by fractions 4, 8, and 9, and thus the original age could be greater than this estimate. For example, a linear regression of all data except fraction 3 yields an upper intercept of 1068 +86/-31 Ma (lower intercept = 923 +55/-106 Ma; MSWD = 1.97, P = 0.065). Total common-Pb contents of 6 to 25 pg were at or slightly above expected blank levels. The U, Th, and total-Pb contents varied markedly, i.e. 395 to 2380 ppm, 485 to 2700 Th, and 80 to 510 ppm, respectively, whereas Th/U displayed a narrower range of 1.1 to 1.6.

z6414

U-Pb data for eight fragments with masses of 9 to 32 µg (Table 1) are shown in Figure 4b. The results show significant scatter beyond analytical errors, spreading out along or slightly above concordia. The ^{206}Pb/^{238}U ages range from 492 to 511 Ma, with a mean of 501 Ma. This sample is distinguished by its low U (100–268 ppm) but relatively high Th (640–1750), yielding high and uniform Th/U (6.4–7.0). Total common-Pb contents in the analyses ranged from negligible to significant (76 pg), possibly due to the variable presence of inclusions. There are no obvious correlations between the ages and other characteristics of the fractions. The slight reverse discordance in some fractions may indicate over-correction for common Pb, but despite this the overall spread in ^{206}Pb/^{238}U is real and either reflects some Pb loss from ca. 511 Ma or older xenotime, or mixing of different generations of xenotime.

CONCLUSIONS

The current practice in ion microprobe U-Pb geochronology of using separate fragments of reference material embedded within each sample mount requires, at a minimum, grain-to-grain homogeneity in the ^{206}Pb/^{238}U ratios. Evaluation of the ID-TIMS U-Pb ages of fragments from four different xenotime megacrysts reveals that only one, z6413, appears sufficiently homogeneous on a bulk scale to potentially serve as an ion microprobe U-Pb reference material. The reference

$^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages for z6413 are 994 Ma and 997 Ma, respectively. The small extent of isotopic discordance is inconsequential for the purposes of ion microprobe calibration of $^{206}Pb/^{238}U$ ratios, due to the inherently large analytical errors. The small uraninite inclusions can also be easily avoided if BSE images are available. All the other xenotime samples showed considerable age heterogeneity, for reasons that were not fully explored in this study. Of these, sample z6410 displayed a simple pattern of minor recent Pb loss, but is, nevertheless, a good secondary standard with reference $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages of 897 Ma and 901 Ma, respectively.

The U, Th, and Th/U characteristics of the samples varied quite markedly. Actinide abundances in xenotime samples z6413 and z6410 are suitably elevated for the purposes of ion microprobe analysis, and span the range likely to be encountered in unknown samples.

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The xenotime samples were generously supplied by Dr. Richard Herd (GSC) and Dr. Scott Ercit (Canadian Museum of Nature). The analytical expertise of personnel within the GSC TIMS facility is gratefully acknowledged. Critical review by Yuri Amelin helped to clarify the significance of the inclusions in the xenotime crystals. This is part of Geochronology Project P153.

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