Chapter 16

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Zircon U–Pb and Fission–Track Dating Applied to Resolving Sediment Provenance in Modern Rivers Draining the Eastern and Central Cordilleras, Colombia

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Abstract Determining the crystallization and cooling ages of detrital zircons from ancient sedimentary rocks or modern river sediments is a powerful method for tracing the sediment provenance and exhumation of orogenic mountain belts. Here, we present a study of the U–Pb and fission–track dating of detrital zircons from: (1) the sedimentary cover units of the Eastern Cordillera between Bogotá and Villavicencio and (2) the modern river sediments of the Guatiquía and Guayuriba Rivers, which drain the eastern flank of the Eastern Cordillera, and those of the Magdalena River at Girardot, which drains the western flank of the Eastern Cordillera and the eastern part of the Central Cordillera. We use our data to highlight the advantages and limitations of using zircon U–Pb and fission–track dating in provenance studies, including the identification of original source areas, sediment recycling and the difficulty of detecting amagmatic orogens in the detrital zircon record. The data obtained in this study allow us to better understand the association between the exhumation of sources and their detrital zircon signatures in the modern rivers that drain part of the Eastern Cordillera.
Keywords: *Detrital zircon, Eastern Cordillera of Colombia, Exhumation, Provenance.*

Resumen La determinación de edades de cristalización y de enfriamiento de circones detríticos en rocas sedimentarias antiguas o sedimentos de ríos actuales es un poderoso método para trazar la proveniencia del sedimento y la exhumación de cinturones orogénicos. Aquí presentamos un estudio de dataciones U–Pb y trazas de fisión en circones de (1) las unidades sedimentarias de la cordillera Oriental entre Bogotá y Villavicencio y (2) sedimentos fluviales actuales de los ríos Guatiquía y Guayuriba, los cuales drenan el flanco oriental de la cordillera Oriental, y sedimentos del río Magdalena en Girardot, que drena el flanco occidental de la cordillera Oriental y la parte oriental de la cordillera Central. Usamos nuestros datos para resaltar las ventajas y limitaciones de usar dataciones U–Pb y trazas de fisión para estudios de proveniencia, incluyendo la identificación de áreas fuente originales, el reciclaje de sedimentos y la dificultad de detectar orógenos

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no magmáticos en el registro de circones detríticos. Los datos obtenidos en este estudio nos permitieron entender mejor la asociación entre la exhumación de fuentes y sus firmas detríticas en ríos actuales que drenan parte de la cordillera Oriental.

Palabras clave: *circón detrítico, cordillera Oriental de Colombia, exhumación, proveniencia.*

1. Introduction

Provenance studies that utilize the crystallization or cooling ages of detrital zircons have been proven to be useful in making paleogeographic reconstructions (e.g., Gehrels & Pecha, 2014), identifying tectonically induced changes in drainage patterns (e.g., Davis et al., 2010), placing time constraints on surface uplift (e.g., Horton et al., 2010), and fingerprinting pulses of magmatism (Caricchi et al., 2014). In recent decades, much progress has been made in the use of different geo– and thermochronological methods to gain information about sediment provenance and the exhumation of sediment source areas. Each individual dating technique offers unique information about provenance and exhumation. The strength of these analyses lies in combining different dating techniques on the same samples or even within the same grains to obtain crystallization and cooling ages that represent the geological history and processes that control a given source–to–sink system. Although apatite fission–track and U–Pb double–dating have been developed and used in provenance studies (Mark et al., 2016), the most suitable mineral for this type of analysis is zircon (e.g., Bernet et al., 2006; Rahl et al., 2003; Reiners et al., 2005), as zircons are present in many upper crustal magmatic, metamorphic, and sedimentary rocks and are resistant to weathering. In this work, we use new examples of modern river samples from the Colombian Andes to explain how zircon fission–track (ZFT) and U–Pb dating can be used in provenance studies to better understand the temporal association between sources and depositional sites (Carter & Moss, 1999) and how the evolution of orogenic mountain belts in settings where large amounts of sediment are recycled from sedimentary source rocks and volcanic input may complicate the exhumational signal in such data (e.g., Carter & Moss, 1999; Jourdan et al., 2013). ZFT data, which provide information about the most recent thermal history and exhumation of source rocks, can complement U–Pb data, which reflect the original zircon crystallization age and its ultimate provenance.

Zircon U–Pb geochronology is the most common technique applied to provenance studies of detrital materials, including the sedimentary basins of the Colombian Andes (Horton et al., 2010; Nie et al., 2010). The strength of this method lies in the ability of detrital zircons to retain the spectra of ages that characterize the timing of the igneous and/or metamorphic (re)–crystallization of their source terranes. This type of information is useful for establishing stratigraphic correlations and identifying sediment source areas and/or their transport and depositional histories (Kosler & Sylvester, 2003). In the same sense, ZFT data provide robust information about the most recent thermal history of a sediment source area after it cools below the ZFT closure temperature of approximately 250–200 °C, depending on the cooling rate, which is invaluable for elucidating the tectonic and exhumation processes in a range of geodynamic settings, especially during the evolution of convergent orogenic belts (Bernet & Garver, 2005; Brandon et al., 1998; Reiners & Brandon, 2006). Nonetheless, in cases of very slow cooling and/or reheating in the source area, detrital zircons may also reflect partial annealing in the source area, with apparent cooling ages that cannot be directly associated with a tectonic event or a particular orogenic phase (Bernet et al., 2001, 2006, 2009).

The purpose of this chapter is to present the U–Pb and fission–track data from the first source–to–sink study of the Servicio Geologico Colombiano (SGC) Geochronology Laboratory, using the tested analytical procedures and their application to resolve specific geological problems. The goal of this preliminary study is to determine the provenance signal in the modern river sediments of rivers draining the eastern and western foothills of the Eastern Cordillera in Colombia using the combined ZFT and zircon U–Pb dating of the same samples. The detrital sediments studied here were taken from the Magdalena River at Girardot on the western flank of the Eastern Cordillera and from the Guatiquía and Guayuriba Rivers in the eastern foothills of the Eastern Cordillera (Figure 1).

2. Geological Framework

The geology of Colombia is tectonically and morphologically characterized by the stable Precambrian basement of the Amazon Craton in the eastern part of the country (Ibañez–Mejia & Cordani, 2020), as well as by the highlands of the Andean Belt, in which three cordilleras are separated from each other by intermountain valleys (Figure 1). Rivers such as the Bogotá River drain the Eastern Cordillera to the west into the Magdalena River (Figure 2). The Magdalena River is one of the most important drainage systems of the Northern Andes, as it

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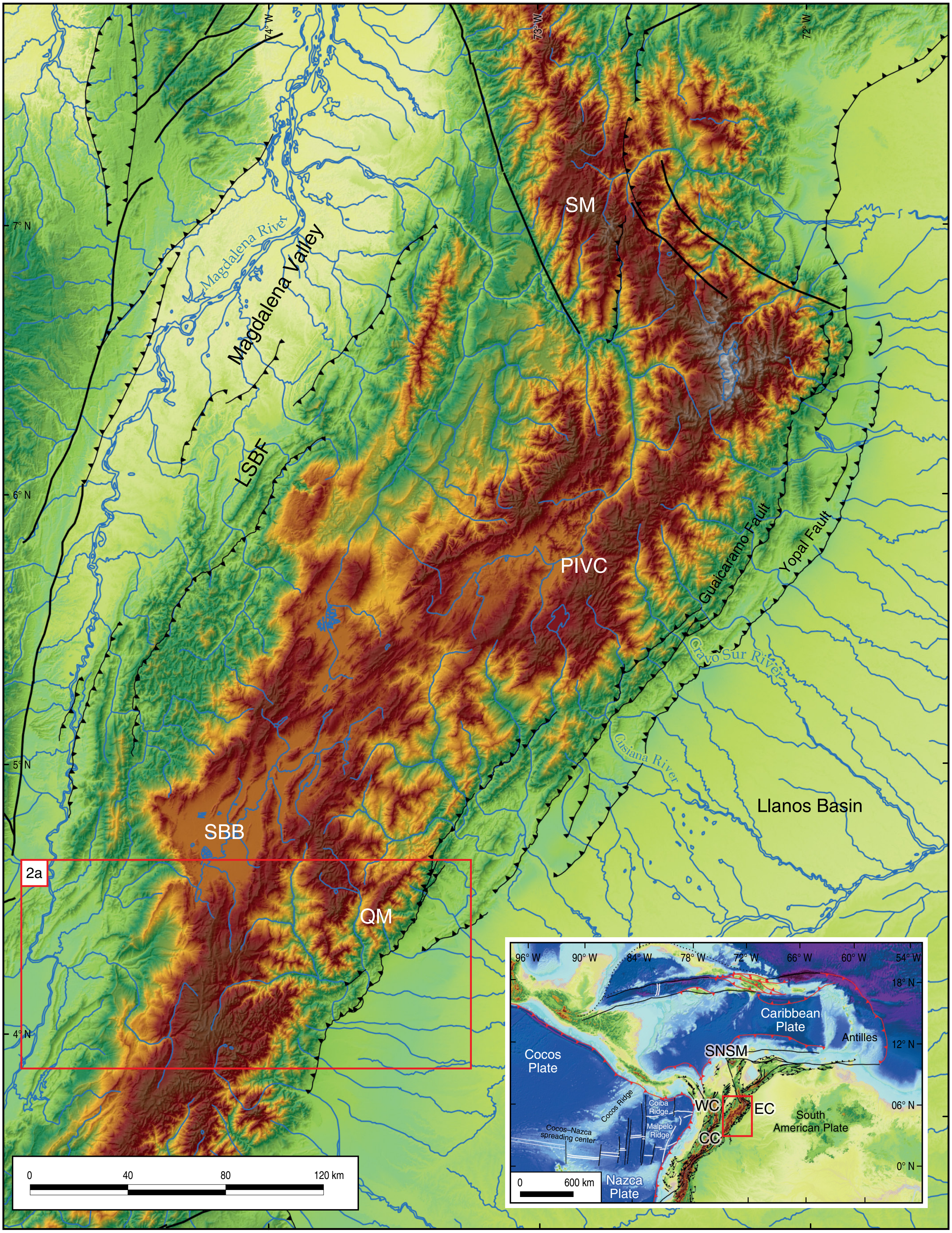
Figure 1. Shaded relief image of the Eastern Cordillera showing the main geomorphological and tectonic structures: Magdalena Valley; (SM) Santander Massif; (LSBF) La Salina–Bituima Fault System; (PIVC) Paipa–Iza Volcanic Complex; (QM) Quetame Massif; (SBB) Sabana de Bogotá Basin; Llanos Basin. Note the NE–SW trend of the cordillera, as well as the location of the study area (red box). The box in the smaller map shows the tectonic setting: (EC) Eastern Cordillera; (CC) Central Cordillera; (WC) Western Cordillera; (SNSM) Sierra Nevada de Santa Marta.


74° W
73° W
72° W
7° N
6° N
5° N
4° N
SM
Magdalena River
Magdalena Valley
LSBF
PIVC
Gualcaramo Fault
Yopal Fault
Cravo Sur River
Cusiana River
Llanos Basin
SBB
2a
QM
0
40
80
120 km
96° W
90° W
84° W
78° W
72° W
66° W
60° W
54° W
18° N
12° N
06° N
0° N
Caribbean Plate
Antilles
Cocos Plate
SNSM
Cocos Ridge
Coiba Ridge
Malpelo Ridge
Cocos–Nazca spreading center
WC
EC
CC
South American Plate
0
600 km
Nazca Plate

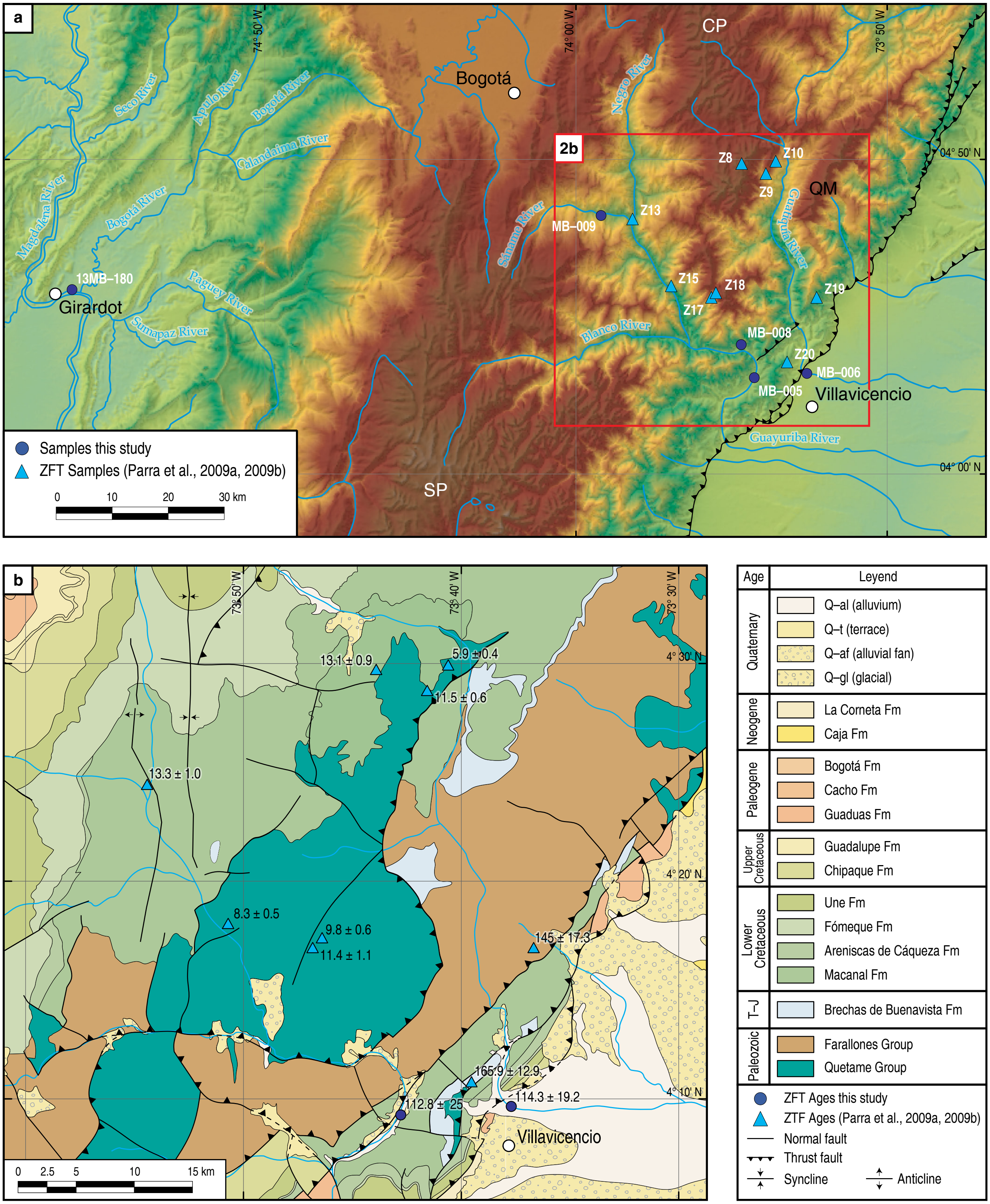


Figure 2. (a) Shaded relief image of the study area showing the main river discharge in the Eastern and Western Cordilleras (Bogotá River, Guayuriba River, Guatiquía River). Locations of samples collected in this study are shown as blue circles, and those of Parra et al. (2009a, 2009b) are shown as blue triangles. (QM) Quetame Massif; (SP) Sumapaz Paramo; (CP) Chingaza Paramo. **(b)** Simplified and modified geological map from Gómez et al. (2015) with the locations of samples analyzed for zircon fission–track ages in this study shown as blue circles and those of Parra et al. (2009a, 2009b) shown as blue triangles.

crosses Colombia from south to north over a distance of 1000 km, collecting all of the tributaries coming from the western flank of the Eastern Cordillera, the eastern flank of the Central Cordillera, and, further downstream, the western Cordillera at the confluence with the Cauca River near Magangué. On the east side of the Eastern Cordillera, the drainage configuration is different. The rivers run from the foothills across the Llanos Basin from west to east to ultimately join either the Orinoco or Amazon Rivers. The Guatiquía and Guayuriba Rivers are two of the most important tributaries of the Meta River, which is part of the Orinoco Basin. With their springs in the Chingaza and Sumapaz Paramo regions in the Eastern Cordillera (Figure 2a), they drain Precambrian (Garzón Massif), Paleozoic (Quetame Massif), and Cretaceous sedimentary cover rocks (Figure 2b).

Different authors have used thermochronology and U–Pb geochronology to describe how the surface uplift, exhumation and deformation history of the Eastern Cordillera is related to the drainage and sedimentary basin evolution of the Llanos Foreland, which is limited by the Guaicáramo Fault System (i.e., Bande et al., 2012; Horton et al., 2010; Mora et al., 2008; Nie et al., 2010; Parra et al., 2009a, 2009b; Saylor et al., 2013). The evolution of the Eastern Cordillera as a highland started during the middle Eocene to Oligocene, and it intensified during the Miocene – Pliocene, after the tectonic inversion of pre–existing Jurassic and Early Cretaceous graben structures and mid–crustal low–angle detachment faults (Colleta et al., 1990; Cooper et al., 1995; Dengo & Covey, 1993; Mora et al., 2006, 2009; Sarmiento–Rojas et al., 2006).

Since the Eocene, compressional deformation has migrated to the east (Mora et al., 2010; Parra et al., 2009a, 2009b, 2012), and a late Paleogene to Neogene foreland basin sequence began recording the erosional exhumation history of the adjacent basement highs in the Eastern Cordillera. According to Mora et al. (2008), the eastern flank has the highest mean elevations, and its topography exhibits deeply incised river canyons with dissected basement rocks. Based on thermochronological analyses, this topography has been interpreted as a cause and consequence of rapid exhumation rates, which have been attributed to climatic and tectonic forcing (Parra et al., 2009a, 2009b). The asymmetry in orogenic processes is believed to be caused by two main factors: (1) structural inheritance during inversion, and (2) initial topographic growth between 6 and 3 Ma, which built an orographic barrier that subsequently intercepted easterly moisture–bearing winds, thus leading to focused precipitation and enhanced erosion (Mora et al., 2008).

Although the initial surface uplift was probably moderate, after the Oligocene, the rise of the Eastern Cordillera blocked the arrival of zircons derived from the Guiana Shield to the hinterland interior. This dramatic change in local topography caused a shift in the sediment provenance of the sedimentary units deposited in the intermontane Magdalena River Basin (Horton et al., 2010; Nie et al., 2010), which has developed between the Central and Eastern Cordilleras since the Eocene.

3. Materials and Methods

3.1. Sampling

In this study, two samples were collected along a Bogotá–Villavicencio transect in a stratigraphic section of Paleozoic to Upper Cretaceous rocks (Figures 2, 3; Table 1); additionally, two river sediment samples of the main tributaries of the eastern flank of the Eastern Cordillera were collected close to Villavicencio, and one was collected from a sandbar in the Magdalena River at Girardot. The U–Pb dating of detrital zircons was conducted on river sediment samples, and ZFT dating was performed on both sedimentary rock and river samples. The oldest units in the area correspond to the metamorphic and metasedimentary rocks of the Quetame Group, which are stratigraphically overlain by the Devonian sedimentary rocks of the Farallones Group (Areniscas de Gutiérrez Formation), the Jurassic sedimentary rocks of the Brechas de Buenavista Formation, and a Cretaceous sedimentary section represented by the rocks of the Cáqueza and Une Formations, as well as the Upper Cretaceous Chipaque Formation. Active sediment samples from the Guatiquía and Guayuriba Rivers were collected in proximity to the drainage slope inflection that represents the transition from the orogenic hinterland to the foreland basin system. These two rivers extend from approximately the central axis of the mountain range and flow towards the east, mainly eroding Cretaceous and tertiary units. On the western flank of the Eastern Cordillera, one sample was taken from the Magdalena River on a sandbar close to the Girardot bridge, downstream of its confluence with the Bogotá River (Table 1). The Upper Magdalena River Basin drains not only the western flank of the Eastern Cordillera but also a large part of the Central Cordillera volcanic arc and crystalline basement.

3.2. U–Pb Dating by LA–ICP–MS

Sedimentary provenance studies commonly use the U–Pb dating of detrital zircon grains as a tool with which to characterize the provenance of sediments, provided that the age spectra of their potential source areas are known. This approach allows researchers to establish potential correlations between different sedimentary units and permits the quantification of the maximum depositional ages of strata in the absence of datable volcanic material (Dickinson & Gehrels, 2009; Gehrels, 2011). Laser ablation–inductively coupled plasma–mass spectrometry (LA–ICP–MS) is particularly well suited for provenance studies, which are usually based on a large number of measurements (typically approximately 100–120 grains per sample) in order to identify all major sedimentary source components (Vermeesch, 2004; Bernet & Garver,

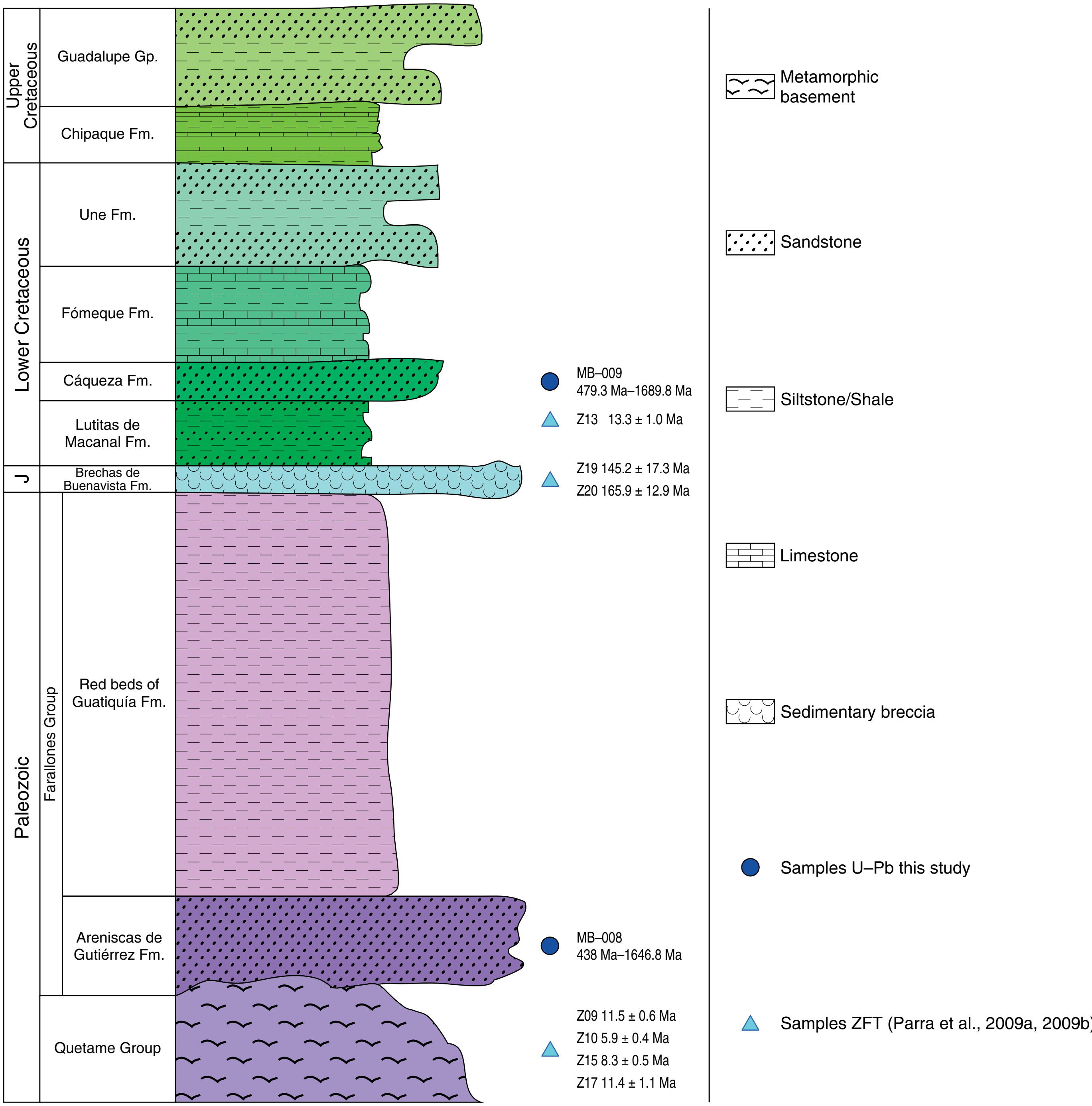


Figure 3. Schematic Paleozoic – Upper Cretaceous stratigraphic section with the locations of bedrock samples presented in this study (blue circles) and by Parra et al. (2009a, 2009b) (blue triangles).

Table 1. Sample information.

Sample	Latitude N	Longitude W	Altitude (masl)	Lithology	Stratigraphic unit	Depositional age
MB005	4° 9’ 12.312”	73° 42’ 49.284”	714	Sand	Recent	0 Ma
MB006	4° 9’ 36.468”	73° 37’ 44.76”	450	Sand	Recent	0 Ma
MB008	4° 12’ 21.204”	73° 44’ 4.776”	880	Metasandstone	Areniscas de Gutiérrez Fm.	Devonian
MB009	4° 24” 40.608”	73° 57’ 35.496”	1713	Quartz arenite	Cáqueza Fm.	ca. 127 Ma
13MB180	4° 17’ 35.16”	74° 48’ 36.036”	261	Sand	Recent	0 Ma

2005). Nevertheless, Pullen et al. (2014) recently demonstrated that large–n datasets (n= 300 to 1000) yield better precision in the distribution of U–Pb detrital zircon data and better approximate the true relative abundances of the major components in samples with multi–modal age spectra. In this study, we analyzed approximately 220 detrital zircon grains from each sample.

Detrital zircons from the Guayuriba, Guatiquía, and Magdalena River samples were mounted in epoxy resin together with age standards and polished to expose their internal grain surfaces. Cathodoluminescence (CL) images were taken using a Gatan MiniCL detector, attached to a JEOL JSM IT–300–LV scanning electron microscope (SEM) at the SGC in order to guide where analytical spots were placed on cores and/or rims; the operating conditions for SEM–CL images included an accelerating voltage of 15 kV and a probe current of 60 nA.

Following imaging, zircons were analyzed at the Geochronology laboratory of the SGC using LA–ICP–MS. The instruments used were a Thermo Scientific® Element2 magnetic–sector ICP–MS coupled to a Photon Machine® 'Excite' excimer laser system (193 nm). The mosaic images used for the selection of ablation spots were constructed using the Chromium2 software at 75% magnification. The following laser settings were used: a spot size of 20 or 35 µm; a laser fluence of 6.91 J/cm^2; and an output energy of 94%. The number of pulses per burst was 126; they were fired at a frequency of 8 Hz for a total ablation duration of 28 seconds per analysis. The mass spectrometer was tuned with Sri Lanka zircons to maximize the signals of the isotopes of interest, i.e., ^{238}U, ^{235}U, ^{207}Pb, ^{206}Pb, ^{208}Pb, ^{232}Th, ^{202}Hg, and ^{204}(Pb + Hg), and to minimize the oxide (^{254}(UO)$^+$/^{238}U$^+$) production at the sample interface.

Instrumental fractionation was corrected using a standard–sample bracketing approach, i.e., by analyzing reference zircon materials after every five unknowns and normalizing the data relative to the known (±CA)–ID–TIMS ages of the reference zircons. For further discussion of the data acquisition and reduction routines followed in this study, see Ibañez–Mejia et al. (2015) and Pullen et al. (2014). To validate the accuracy of our method, Plešovice crystals were analyzed alongside each sample and treated as unknowns during data reduction. These crystals have a known ID–TIMS age of 337.13 ± 0.37 Ma (Slama et al., 2008); thus, the results obtained here (see Supplementary Information) demonstrate that our analytical approach is accurate and precise within ±2%, which is typical for U–Pb zircon analyses using LA–SC–ICP–MS methods (e.g., Chang et al., 2006; Frei & Gerdes, 2009; Ibañez–Mejia et al., 2015; Schaltegger et al., 2015; Pullen et al., 2014).

Probability distribution diagrams were constructed using only ^{206}Pb/^{238}U ages that were concordant to determine the frequency and distribution of the values represented (i.e., the individual apparent ages) in each sample. The results were represented in concordia diagrams (Wetherill, 1956), where the obtained ^{206}Pb/^{238}U and ^{207}Pb/^{235}U isotopic ratios were plotted to assess age concordance. Diagrams were constructed by discarding data that were more than 10% discordant based on the difference between the calculated ^{206}Pb/^{238}U and ^{207}Pb/^{206}Pb apparent ages for each spot. These diagrams were constructed in ISOPLOT V3.75® (Ludwig, 2012). The individual spot uncertainties in the data presented here include internal analytical uncertainties only and are reported at the 2σ level. Determining the total uncertainties for each spot or mean age in this method requires the propagation of external reproducibility and other systematic sources of uncertainty, which, in our sessions, yielded average values of ca. 0.9% for the determination of ^{206}Pb/^{238}U and ca. 0.7% for that of ^{207}Pb/^{206}Pb. Consequently, the total uncertainties in the mean ages of the samples and reference zircons are reported in the form of ±A/B, where the first level of uncertainty A considers only the internal analytical uncertainties, and the second level B reflects the propagation of the external reproducibility of the standards and other sources of systematic error (see Ibañez–Mejia et al., 2015 for more details).

3.3. Zircon Fission–Track Dating

The sample preparation used for fission–track analysis at the Thermochronology Laboratory of the SGC consisted of mounting zircon aliquots (with grain sizes of approximately 75 to 250 μm) in Teflon® sheets, polishing the mounts to expose their internal grain surfaces, and etching the grains in a NaOH–KOH melt at 228 °C. Two to three grain mounts were prepared per sample and were etched for different lengths of time in order to obtain countable fission–tracks in the full grain age spectrum (e.g., Bernet et al., 2004). After etching, the grain mounts of all samples were cleaned and covered with mica sheets as external detectors. All samples were irradiated with thermal neutrons together with age standards and IRMM541 dosimeter glasses at well–thermalized reactor in Garching, Germany, using a nominal fluence of 0.5×10^{15} n.cm^{-2}. After irradiation, mica detectors were etched for 18 minutes at 21 °C with 48% HF to reveal induced tracks. All samples were analyzed using an Olympus BH2 optical microscope and the FTStage 4.04 system at the Thermochronology Laboratory of the Institut des Sciences de la Terre, University Grenoble Alpes. Tracks were counted dry at a magnification of 1250X; 100 grains were analyzed per sample, and fission–track grain ages were calculated using the Binomfit software of Brandon (see Ehlers et al., 2005). The Fish Canyon Tuff and Buluk Tuff age standards were used for zeta calibration.

4. Results

4.1. Zircon U–Pb Dating

Devonian sedimentary rocks (sample MB–008) show relevant age peaks at 468.3 ± 0.7 Ma, representing 24% of the data, as well as main peaks at 905, 991, 1158, 1304, and 1500.1 ± 1.7

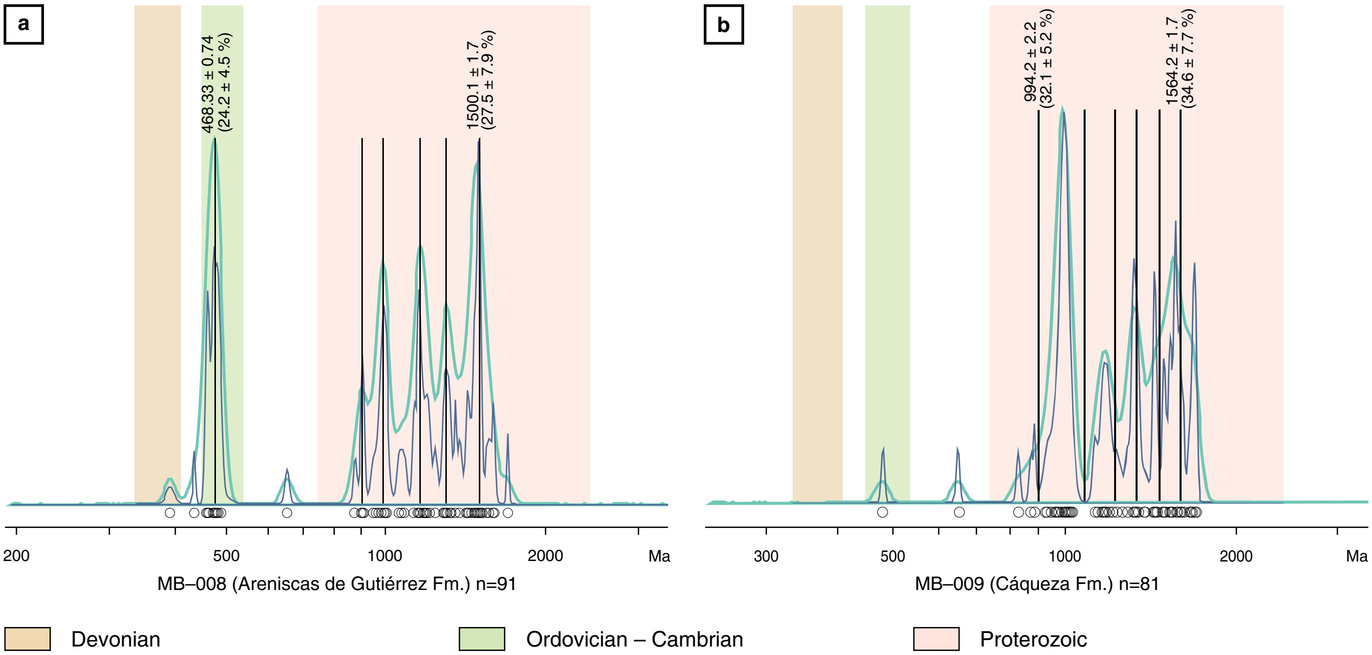


Figure 4. Zircon U–Pb ages with peak ages from probability density plots (dark blue line) and kernel density estimates (light blue line) (using RadialPlotter by Vermeesch, 2009). **(a)** Sample MB008, Devonian bedrock. **(b)** Sample MB009, Cretaceous bedrock.

Ma, representing 27.5% of the data; the oldest age is 1702.3 Ma (Figure 4a). In contrast, Cretaceous sedimentary rocks (sample MB–009) have only two grains with apparent ages of 479.3 ± 3.1 Ma and 650 ± 4.2 Ma; the remaining 94% of the data comprise peaks at 994.2 ± 2.2 Ma, 1177 Ma, 1320 Ma, 1438 Ma, 1564.2 ± 1.7 Ma and 1685 Ma (Figure 4b).

The detrital zircon U–Pb ages of the modern river sands from the eastern flank of the Eastern Cordillera are distributed between 830 and 1830 Ma, with a discrete peak at ca. 443 Ma (Figure 5). The Guayuriba River (MB–005) data define several (concordant) apparent age populations at 443.6 ± 3.6 Ma (which contains only a small proportion of the dated grains), 1027.8 ± 3.1 Ma, 1198.3 ± 2.9 Ma, 1336.4 Ma, 1407.2 Ma, 1527.7 Ma, and 1665.8 Ma (Figure 5a). Similarly, the zircons from the Guatiquía River (MB–006) define five populations at 451.4 ± 2.2 Ma, 1022.8 ± 3.3 Ma, 1189.5 Ma, 1322 Ma, 1518.3 Ma, and 1756.6 Ma (Figure 5b). On the other hand, the detrital zircon age spectra from our Magdalena River sample (13MB–180) are markedly different, as they are represented by four populations separated in two broad age ranges: the first range is represented by peaks falling between 156.76 ± 0.45 Ma and 268.56 ± 0.9 Ma, and the second range is represented by peaks distributed between 1068.9 ± 4.8 Ma and 1483.6 ± 6.5 Ma (Figure 5c). All of these data are included in the Supplementary Information.

The SEM–CL images of the detrital zircons from the river samples show the internal structures and zoning patterns of the different age populations found with U–Pb dating. The Guayuriba River sample (Figure 6, MB–005) shows at least seven age populations, and all of its imaged grains mainly exhibit oscillatory zoning. Some of these grains are often perturbed by convoluted local textures and characterized by complex crystal zoning patterns with local magmatic resorption (e.g., early Mesoproterozoic crystals), and the oldest grains show thin overgrowth rims. In the Guatiquía River sample (Figure 7, MB–006), oscillatory zoning patterns are also predominant, but some of the middle to late Mesoproterozoic grains display convoluted to chaotic zoning; as with the Guayuriba zircons, the oldest grains show thin overgrowth rims. The Magdalena River detrital zircons (Figure 8, 13MB–180) mainly show oscillatory zoning patterns, which often exhibit areas of local recrystallization and homogenization. Although it is not possible to determine the mechanisms and number of cycles that many of these re–worked zircons have experienced, their inheritance patterns suggest a magmatic origin for most of them. The grains that show overgrowth rims may suggest the presence of overprinting metamorphic events, but most of the observed rims are too thin to be dated.

4.2. Detrital Zircon Fission–Track Analysis

The fission–track data obtained from the three river samples are presented in Table 2 and Table 3. The fission–track ages obtained for the Guayuriba River sample (MB005) show a spectrum of ages ranging from 8.8 to 530.1 Ma (Figure 9). The dispersion is very high (99.4%); accordingly, the P(χ^2) value is zero. Using the peak–fitting algorithm (Galbraith & Green, 1990), three statistically representative peaks can be detected at 15.6 ± 2 Ma, 110 ± 10 Ma, and 229 ± 18 Ma (Figure 9a). The Guatiquía River sample (MB006) shows a similar grain

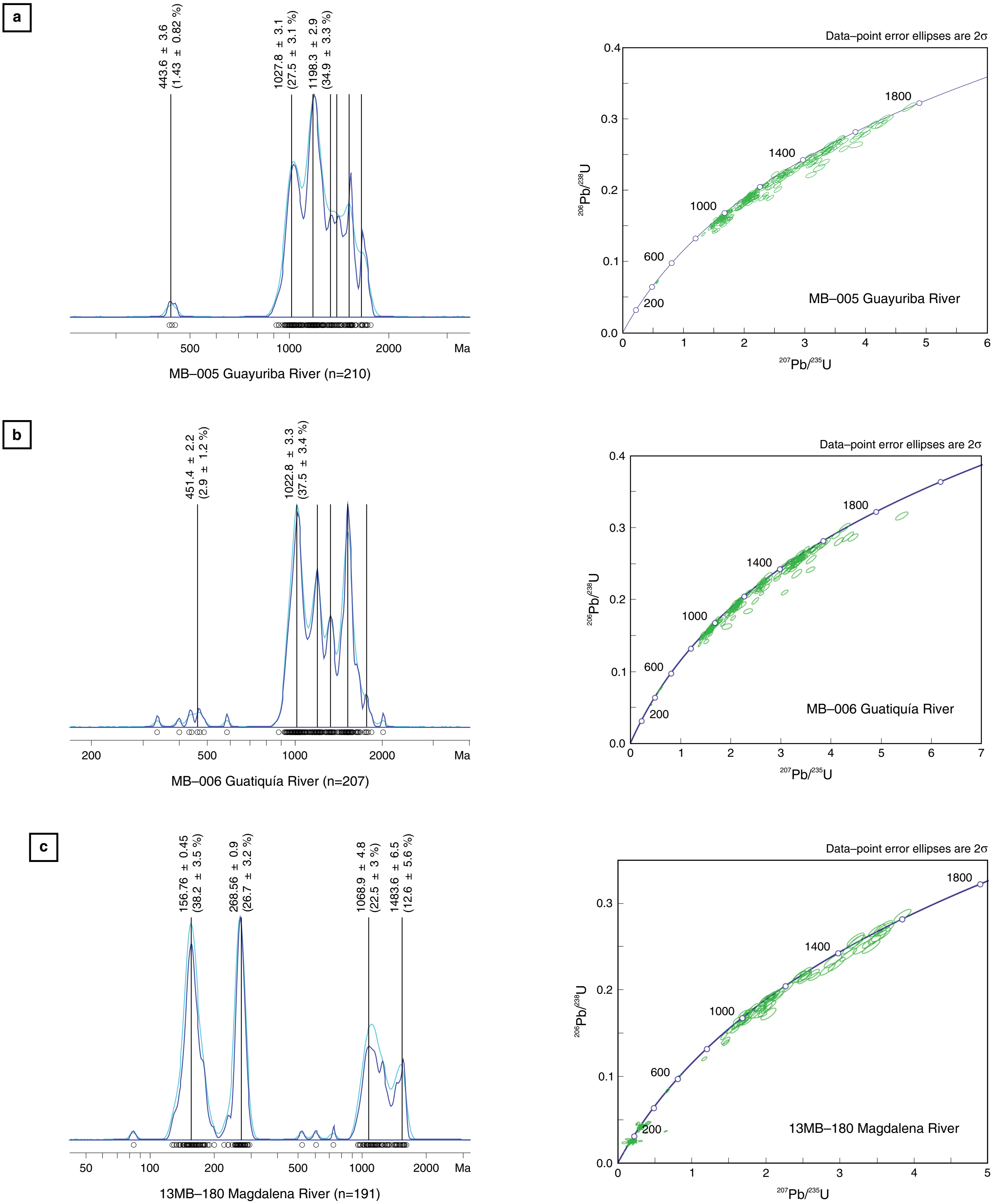


Figure 5. Zircon U–Pb age spectra with peak ages from probability density plots (dark blue line), kernel density estimates (light blue line) and concordia diagrams depicting $^{206}Pb/^{238}U$ and $^{207}Pb/^{235}U$ data for zircon grains from: **(a)** Guayuriba River sample MB005, **(b)** Guatiquía River sample MB006, and **(c)** Magdalena River sample 13MB180.

Neogene

Paleogene

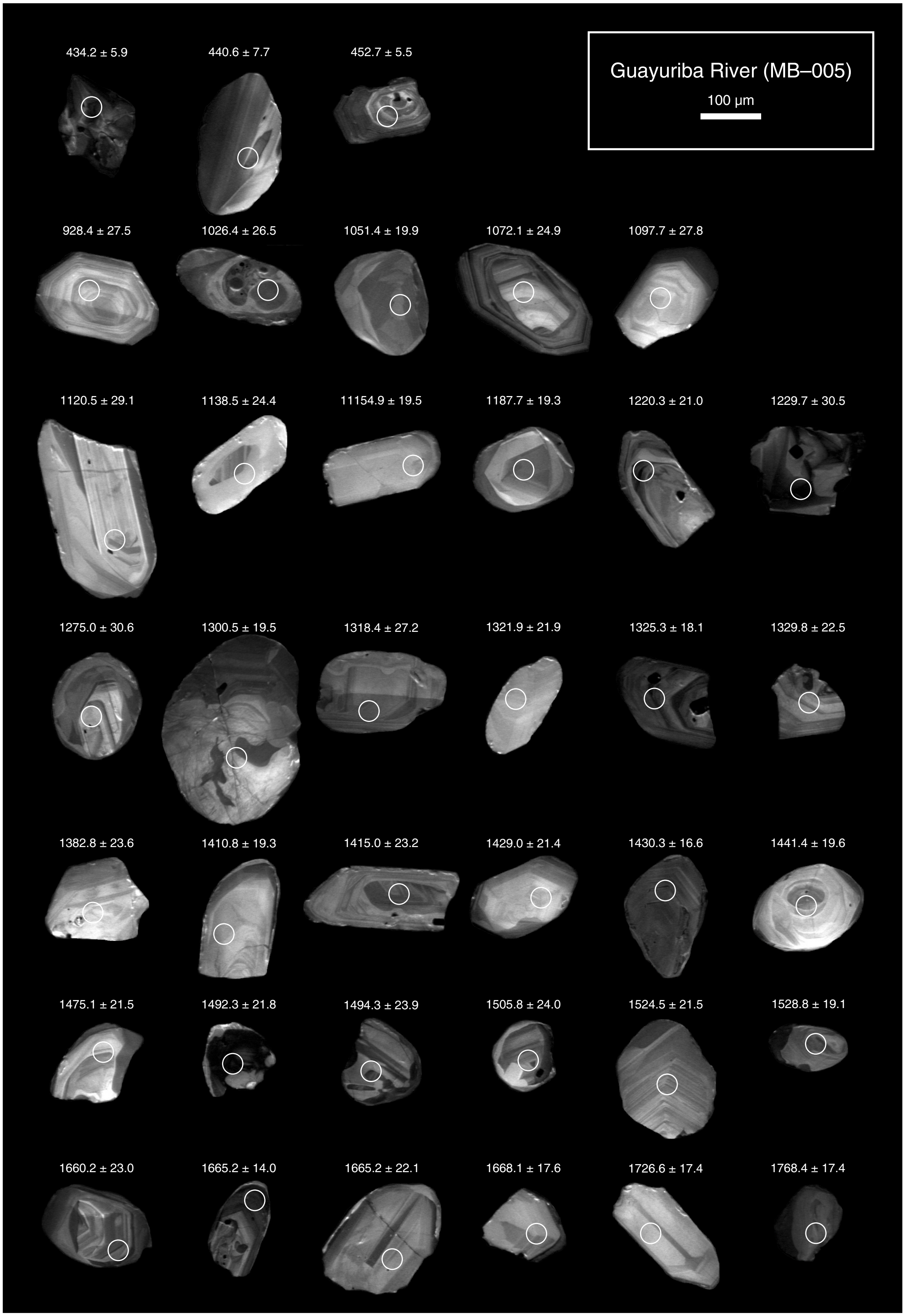


Figure 6. Cathodoluminescence images (SEM–CL) of representative detrital zircon populations of Guayuriba River sample (MB–005); U/Pb ages in Ma with 2–sigma uncertainty. Circles represent the areas used for analyses.

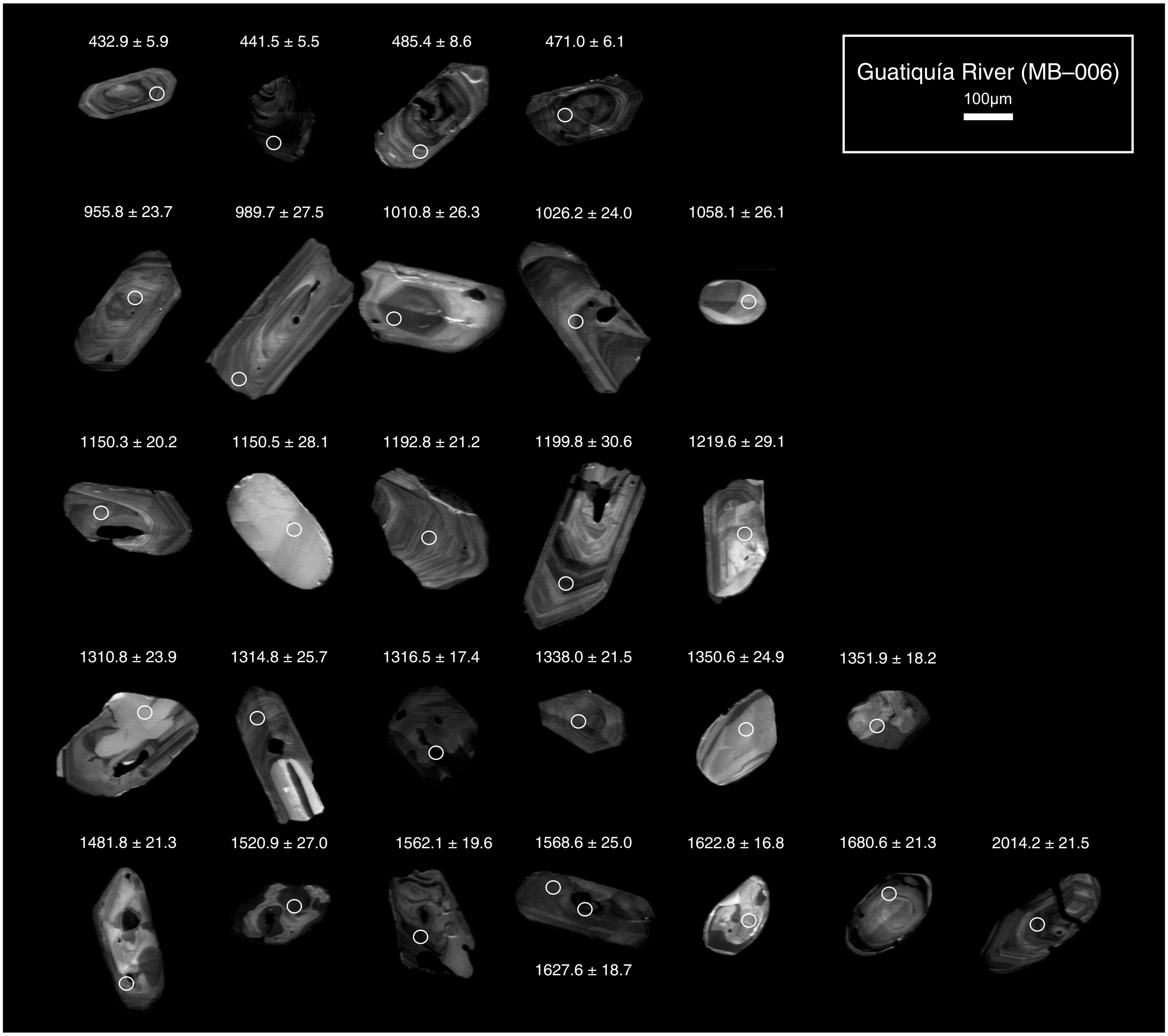


Figure 7. Cathodoluminescence images (SEM–CL) of representative detrital zircon populations of Guatiquía River sample (MB–006); U/Pb ages in Ma with 2–sigma uncertainty. Circles represent the areas used for analyses.

age distribution between 19.4 and 476.0 Ma, although it shows a smaller dispersion of 66.6%. Nevertheless, four statistically robust age peaks can be detected at 22.5 ± 3.5 Ma, 49.3 ± 4.6 Ma, 98.2 ± 9.2 Ma, and 194 ± 13 Ma, although the first one only corresponds to 3.8% of the data (Figure 9b).

The Magdalena River sample (13MB180) shows ages ranging from 28.1 to 327.9 Ma, with a dispersion of 36.3%. The main peak ages are 46.4 ± 4.6 Ma, 91.5 ± 5.6 Ma, and 233 ± 42 Ma (Figure 9c). Using Kolmogorov–Smirnov (KS) statistics, we compared the grain age distributions between rivers. The ZFT grain age distributions from the Guayuriba and Guatiquía Rivers are similar, whereas the ZFT grain age distribution of the Magdalena River sample is statistically significantly different (Figure 10a).

5. Discussion

5.1. Provenance Information from Detrital Zircon U–Pb Ages

The U–Pb dating of detrital zircons is a well–established provenance tool that has been widely used in many different tectonic settings. This method provides information about the distribution of crystallization ages from the original source region, which can be combined with careful analyses of igneous and/or metamorphic overgrowth rims (if present) to track intermediate source areas and sediment recycling. The data obtained here provide an opportunity to discuss the strengths and short-

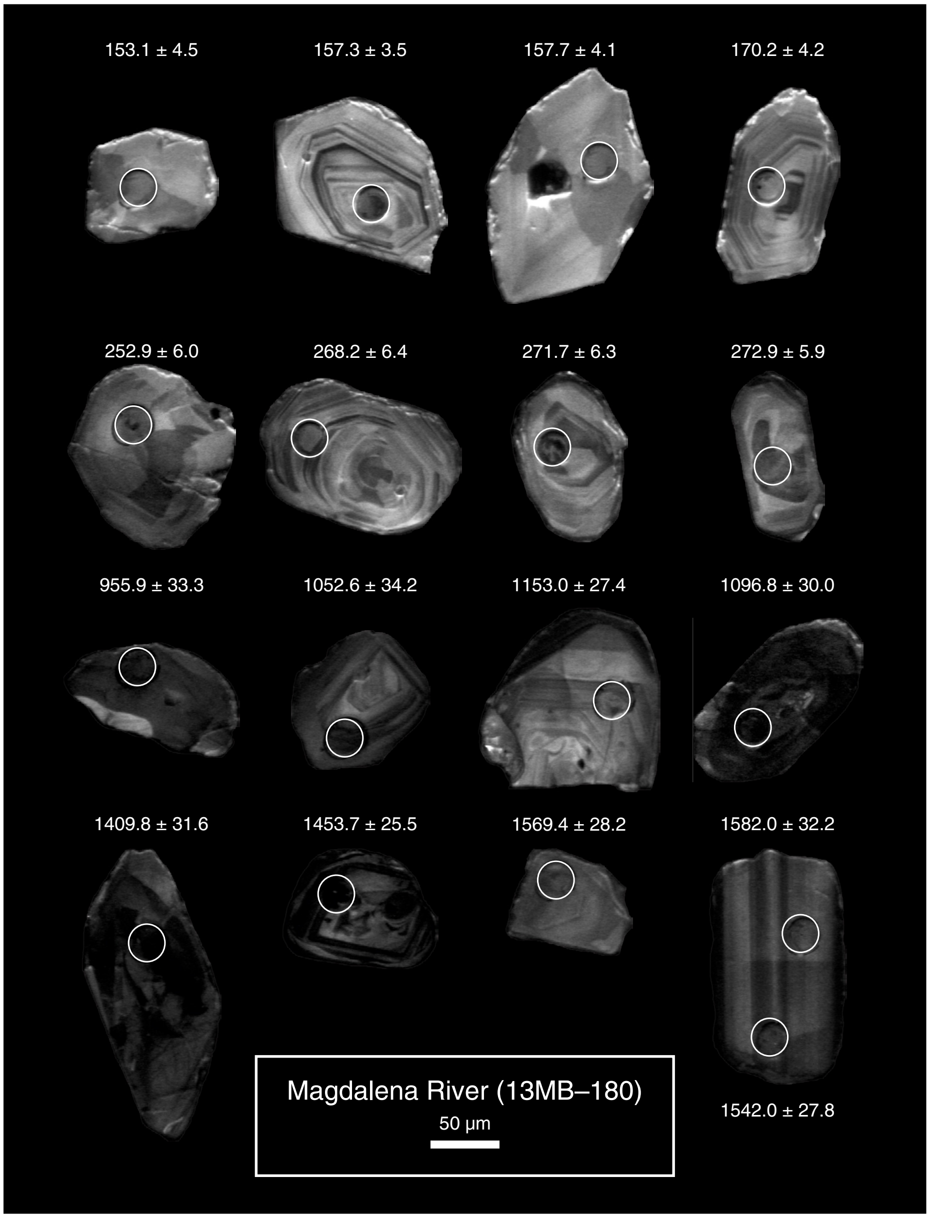


Figure 8. Cathodoluminescence images (SEM–CL) of representative detrital zircon populations of Magdalena River sample (13MB–180); U/Pb ages in Ma with 2–sigma uncertainty. Circles represent the areas used for analyses.

comings of these methods by combining our new bedrock and modern river data. There is currently no consensus on the best way to interpret detrital zircon U–Pb age spectra in terms of the significance of peak heights when plotted on probability density function plots, differences in the relative abundances of peaks between different samples in stratigraphic successions, or which statistics should be applied to decompose the observed grain age spectra (Gehrels, 2011). Nonetheless, the different age peaks determined here using the RadialPlotter program (Vermeesch, 2009, 2012) are consistent with the zircon U–Pb data obtained from other studies in the Eastern Cordillera (Horton et al., 2010; Nie et al., 2010). The comparison of bedrock detrital zircon U–Pb data from Devonian and Cretaceous sedimentary rocks in the Eastern Cordillera along the Bogotá–Villavicencio stratigraphic section hints at possible changes in the sediment provenance of these units. For the Devonian sedimentary rocks, a significant Ordovician zircon U–Pb age peak represents a source in the Cambrian – Ordovician crystalline basement of the Eastern Cordillera. The new zircon U–Pb data presented in this study are compatible with the zircon U–Pb data of Horton et al. (2010), who also proposed an igneous source for Ordovician zircons corresponding to the local lower Paleozoic Andean basement rocks, which are related to magmatic activity in the Floresta and Santander Massifs (e.g., García–Ramírez et al., 2017; Jiménez–Triana, 2016; Restrepo–Pace, 1995; van der Lelij et al., 2016). On the other hand, zircons with Meso-

Table 2. Zircon fission–track central age data.

Sample	n	ϱ_s (10^{+6} cm^{-2})	N_s	ϱ_i (10^{+6} cm^{-2})	N_i	ϱ_i (10^{+5} cm^{-2})	P(χ^2)	Dispersion (%)	Age (Ma)*	± 2 σ	U (ppm)	± 1 σ
MB005	100	6.72	9231	1.30	1792	3.84	0.0	99.4	112.8	25	170	9
MB006	100	7.40	8140	1.71	1880	3.83	0.0	66.6	114.3	19.2	223	12
13MB180	100	7.33	8131	2.32	2575	3.82	0.0	36.3	84.1	10.5	304	14

*Fission–track age is given as Central Age (Galbraith & Laslett, 1993).

Note: Samples were counted dry with a BX51 Olympus microscope at 1250X magnification. Central ages and age ranges were determined with the BINOMFIT program of Brandon (see in Ehlers et al., 2005), using a zeta factor of 142.39 ± 6.48.

Table 3. Zircon fission–track peak age data.

Sample	n	Age range (Ma)	P1	± 2 σ	%	P2	± 2 σ	%	P3	± 2 σ	%	P4	± 2 σ	%	P5	± 2 σ	%
MB005	100	8.8–530.1	15.6	4.0	11.4	110	20	33.9	229	36.0	54.7	–	–	–	–	–	–
MB006	100	19.4–476.0	22.5	7.0	3.8	49.3	9.2	12.4	98.2	18.4	26.0	194	26.0	57.8	–	–	–
13MB180	100	28.1–327.9	33.6	8.4	5.0	58.5	17	15	85.0	11.0	50.0	113	34.0	24.0	245	82	5.0

Note: Peak ages were determined with the RadialPlotter program of Vermeesch (2009).

proterozoic and Neoproterozoic U–Pb ages were most likely derived from basement sources such as the Garzón Massif or the Putumayo Basin, which are related to orogenic events associated with the assembly of Rodinia (Ibañez–Mejia et al., 2011). Therefore, we conclude that two principal sediment source areas existed for the Devonian sedimentary rocks. In the detrital zircon age spectra obtained from the modern river samples analyzed here, the Upper Ordovician signal is relatively minor but likely corresponds to the recycling of inherited zircons from the Farallones Group.

In contrast, the zircon ages of the Lower Cretaceous sedimentary rocks are restricted to Proterozoic ages. The disappearance of Phanerozoic zircons in the detrital age spectra is most likely related to the evolution of the basin from late–stage extension to thermal subsidence during the mid–Cretaceous and a lack of proximal basement erosion due to sedimentary infilling and burial (Horton et al., 2010). This suggests that the Ordovician arc terranes that were exposed in the Devonian were buried during the Cretaceous.

The modern river detrital zircon U–Pb spectra of the Guayuriba and Guatiquía Rivers samples indicate that the formation of the Eastern Cordillera, which has been forming since the Eocene, is not detectable in the zircon U–Pb record. Even the data reported by Saylor et al. (2013) for the Cusiana and Cravo Sur Rivers, which are located further to the north of our study area, show similar signals as those obtained here, in addition to the appearance of some younger grains with ages of approximately 100 Ma in Cravo Sur derived from Cenozoic units that crop out in the foothills of the Eastern Cordillera. These zircons are simply recycled from the sedimentary cover units or sourced from the Precambrian basement; however, no zircons with U–Pb ages reflecting the Eastern Cordilleran orogenesis exist. This interpretation is complemented by CL images showing zircons with internal oscillatory zoning related to primary magmatic sources and recrystallized edges due to ancient metamorphic events. Furthermore, the oldest grains are broken, with sub–rounded edges. All of these data are indicative of sedimentary recycling. This is not a surprising result, as the Eastern Cordillera is, for the most part, an amagmatic mountain belt, and the level of regional metamorphic overprinting that it experienced was insufficient to leave a trace in the detrital zircon U–Pb age record. The magmatic activity related to the evolution of the Eastern Cordillera and that close to the study area is restricted to only two known events, namely: (1) the intrusion of gabbros and doleritic dikes during peak extension in the Late Cretaceous (Fabre & Delaloye, 1983; Moreno–Murillo et al., 2007; Vásquez et al., 2010); and (2) acidic volcanism occurring in the Paipa–Iza Volcanic Complex (Figure 1), which was active throughout the Pliocene – Pleistocene surface uplift of the Eastern Cordillera (Bernet et al., 2016). Nevertheless, evidence of these two sources is difficult to detect in the detrital zircon record because: (1) gabbros have low fertility for zircon crystallization and thus will be poorly represented in the detrital record (Moecher & Samson, 2006), and (2) Paipa–Iza Volcanic Complex zircons have dominantly Proterozoic and Paleozoic core and rim ages, and only a few rims and overgrowths reflecting Pliocene – Pleistocene volcanic activity have been detected (Bernet et al., 2016).

The Magdalena River sample detrital zircon U–Pb data reflect the zircon U–Pb ages of the Upper Magdalena River Basin, which covers the western flank of the Eastern Cordillera and the eastern flank of the Central Cordillera. Figure 10b shows the differences between the detrital zircon age distributions found in the rivers on the east flank of the Eastern Cordillera and those the west flank, as represented by the Magdalena River, thus denoting the influence of the sedi-

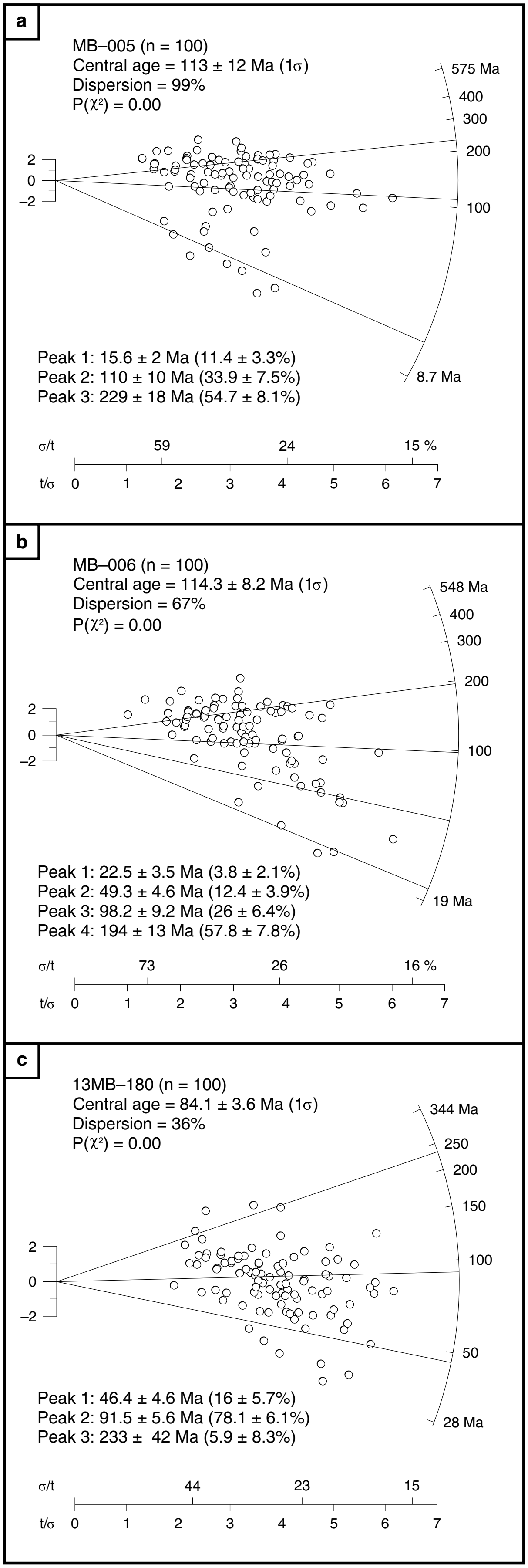


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Figure 9. Radial plots of zircon fission–track data with main peak age populations of three river samples: **(a)** Guayuriba River (MB005), **(b)** Guatiquía River (MB006), and **(c)** Magdalena River (13MB180). The plots were made and peak ages were determined with the RadialPlotter program of Vermeesch (2009).

ments of the Central Cordillera. Although this drainage basin includes the active Nevado del Huila Volcano of the Central Cordillera, the youngest zircon U–Pb age peak determined in the Magdalena River sample reflects Jurassic plutonism (Bustamante et al., 2010, 2016; Zapata et al., 2016) but not recent volcanic input. The volcanic signal is too weak to be detected, as the quantity of young volcanic zircons is negligible compared to the zircons derived from the erosion of the crystalline basement and sedimentary cover rocks. Considering that 191 detrital zircons from sample 13MB–180 were analyzed, we conclude that the abundance of modern volcanic crystals in this sample must be <0.5% (at least 1 data point) for them to have avoided detection. Because of this detection limit problem, it is useful in many provenance and exhumation studies to analyze a much larger number of zircons (e.g., Pullen et al., 2014) and/or to combine detrital zircon U–Pb dating with fission–track dating on the same samples or within the same individual grains (e.g., Bernet et al., 2006; Carter & Bristow, 2000, 2003; Carter & Moss, 1999; Jourdan et al., 2013).

Other peaks in this sample, such as those with Permian ages, could be related to magmatic activity in the Upper Magdalena Valley (Leal–Mejía, 2011; Rodríguez et al., 2017). The old peaks at 1068.9 Ma and 1483.6 Ma are related to either the erosion of basement units in the Eastern Cordillera and serranía de Las Minas (e.g., Ibañez–Mejia et al., 2011) or the reworking of Paleozoic and Mesozoic sedimentary rocks in the area containing material from Amazon Craton sources.

5.2. Provenance Information from Detrital Zircon Fission–Track Ages

It is very common for cooling ages derived from detrital samples to cluster in certain age groups instead of representing a continuum of ages across the drainage area (Bernet & Spiegel, 2004; Bernet et al., 2001, 2004; Cerveny et al., 1988; Spiegel et al., 2000), which has been shown in a range of studies performed in orogens around the world (e.g., Bernet et al., 2009; Garver & Kamp, 2002; Stewart & Brandon, 2004). The number of age clusters or peaks that can be determined depends on the exhumation rates and relief in the drainage area, but the representation or significance of each peak depends on the number of grains dated per sample (Bernet, 2013; Naylor et al., 2015). The more grains that are analyzed, the more peaks can be fitted with peak–fitting routines if the age range is sufficiently large and the peaks are well separated (Brandon, 1996;

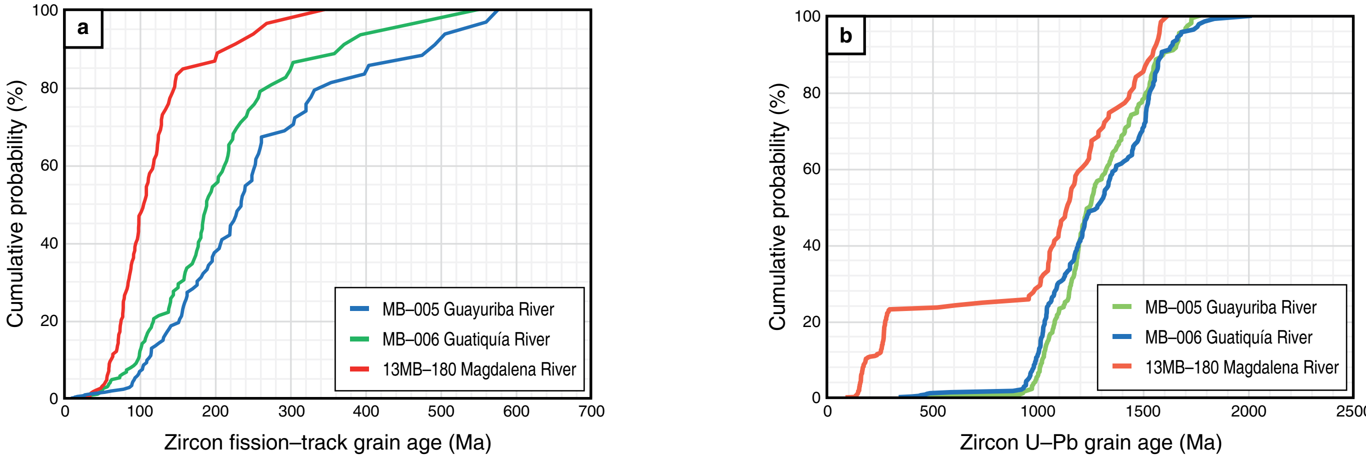

Figure 10. Cumulative probability density plots for Kolmogorov–Smirnov (KS–statistics) comparison of samples from three rivers: **(a)** Zircon fission–track grain age distributions and **(b)** U–Pb grain age distributions.

Galbraith & Green, 1990; Naylor et al., 2015). If bedrock zircon fission–track data are available, then modern river detrital zircon fission–track data can be compared to bedrock data to determine the sediment provenance (e.g., Bernet et al., 2001, 2004, 2009). This provides a baseline for provenance studies of ancient sandstones in associated sedimentary basins when studying long–term records of exhumation. In the absence of post–depositional thermal resetting, detrital zircon fission–track grain ages in ancient sedimentary rocks can only be the same or older than their actual sedimentation age.

In this study, we can compare the detrital zircon fission–track data of the Guayuriba and Guatiquía Rivers samples with the bedrock zircon fission–track data of Parra et al. (2009a, 2009b) from the Bogotá–Villavicencio section (Figure 2b). The documented bedrock zircon fission–track ages range from 5.9 ± 0.4 Ma (sample Z10, Quetame Group) to 165.9 ± 12.9 Ma (sample Z20, Brechas de Buenavista Formation) in the frontal fold–and–thrust belt of the Eastern Cordillera. This bedrock age range contrasts with the single grain age spectra of 8.8 to 530.1 Ma and 19.4 to 476.0 Ma obtained in the Guayuriba River and the Guatiquía River, respectively, in this study.

Parra et al. (2009a, 2009b) defined an exhumed zircon fission–track partial annealing zone along the Bogotá–Villavicencio profile. This means that the rocks containing partially annealed zircons remained at elevated temperatures of 180–220 °C for 50 my or more during their burial in the basin (Reiners & Brandon, 2006). Partially annealed zircons provide "apparent" cooling ages that neither precisely reflect orogenic cooling nor maintain a pre–depositional provenance signal but rather represent a partially reset age signal. However, fully annealed zircons reflect orogenic exhumation. The central ages of fully reset zircons range from 5.9 to 24 Ma from the Quetame Group to the middle of the Lower Cretaceous Macanal Formation, where the base of the exhumed ZFT partial annealing zone is located. The central ages of the partially reset zircons from within the partial annealing zone range from 61 to 166 Ma. The exhumation of the ZFT partial annealing zone started around 24 Ma; it was contemporaneous with major exhumation in the Santander Massif and the Antioquia Batholith in the Central Cordillera, which was linked to the collision of the Panamá–Chocó Block with the northwestern South American Plate (Amaya et al., 2017; Restrepo–Moreno et al., 2009). The exhumation of the ZFT partial annealing zone continued until the Pliocene – Pleistocene, and it was deformed and segmented during the formation and surface uplift of the Eastern Cordillera. This landscape is currently being eroded by surface processes, such as river incision, and its cooling and exhumation history is reflected in the detrital grain ages of the Guayuriba and Guatiquía River samples. The youngest detrital zircon fission–track age peaks of both rivers fall between 15 and 22 Ma. The zircons corresponding to these age peaks, which represent approximately 4–11 % of the analyzed grains, were derived from the fully reset zone (the Quetame Group to the middle Macanal Formation). The partially reset zircons with age peaks of approximately 50 to 110 Ma, which represent approximately 33–38 % of the analyzed grains, were derived from within the exhumed partial annealing zone (the upper Macanal to Chipaque Formations). Finally, 55–60 % of the Guayuriba and Guatiquía Rivers sample detrital zircons fall within the approximately 190–230 Ma age peaks, which were derived from Upper Cretaceous to Paleocene sedimentary rocks that were not affected by post–depositional partial annealing. Therefore, the two well–mixed modern river samples provide a complete representation of the bedrock cooling history in the drainage basins, including ages not observed in the 22 bedrock samples analyzed by Parra et al. (2009a, 2009b). Nonetheless, the Parra et al. (2009a, 2009b) data are critical for determining where the partial annealing zone is exposed in the drainage area, therefore making both datasets complementary. This demonstrates the usefulness of combining bedrock and modern river studies to understand orogenic evolution processes.

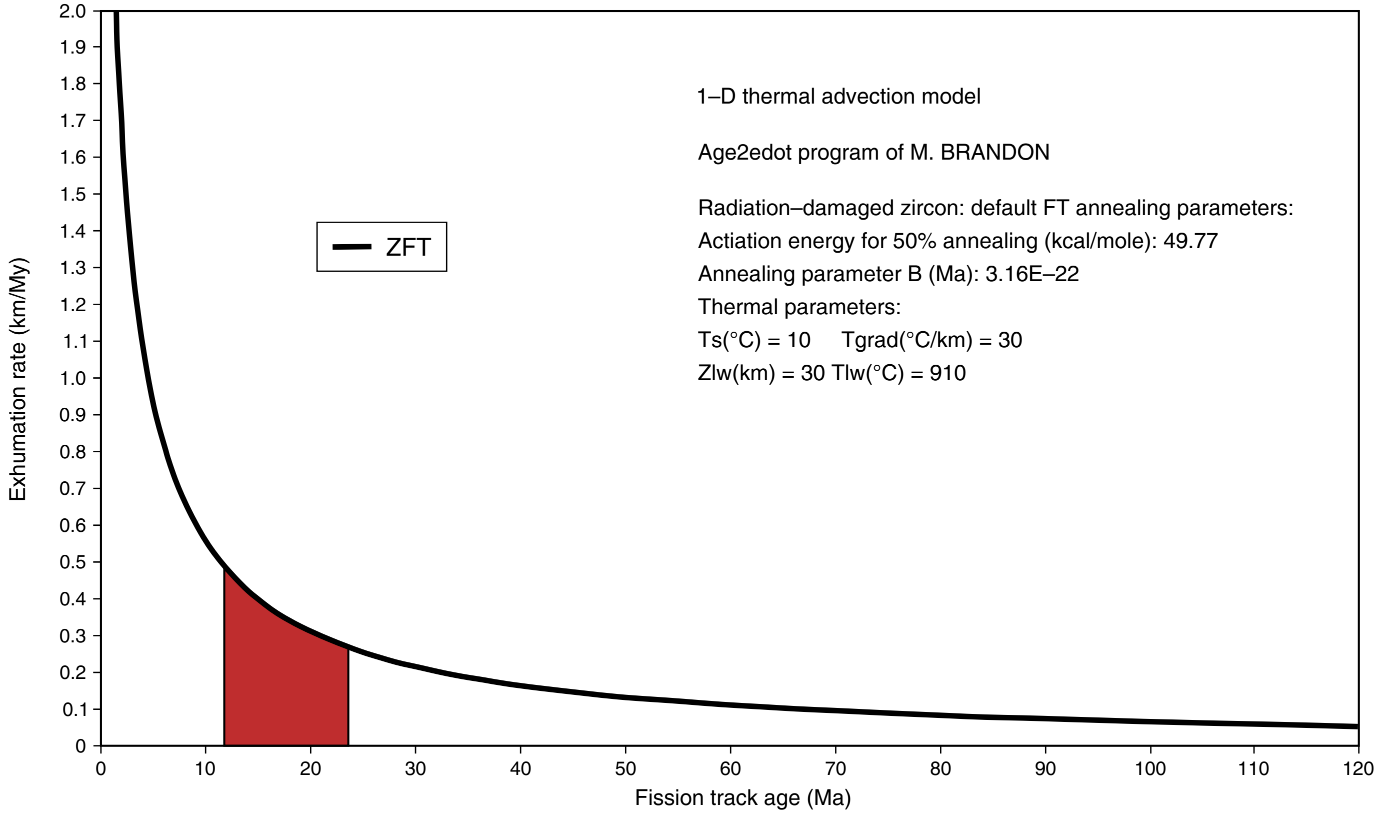

Figure 11. Model showing the estimated exhumation rates in relation to the zircon fission–track cooling ages of the younger age peaks of the Guayuriba and Guatiquía Rivers samples using the age2edot program of M. BRANDON (see Ehlers et al., 2005).

The detrital ZFT peak ages can be used to estimate the long–term average exhumation rates within the drainage basins, assuming a monotonous cooling history, using the age2edot software of Brandon (see Ehlers et al., 2005). Zircons with apparent Miocene cooling ages from the fastest exhuming areas in the Eastern Cordillera indicate exhumation rates on the order of approximately 0.3–0.4 km/My (Figure 11). These exhumation rates are moderately fast compared to those in other orogenic systems (see Montgomery & Brandon, 2002).

The Magdalena River sample, which yields a ZFT age spectrum of 28–328 Ma and age peaks at approximately 46, 92, and 230 Ma, does not show the same exhumation signal as the Guayuriba and Guatiquía Rivers samples (Figure 9). The youngest age peak of the Magdalena River sample is approximately 20–30 my older than the youngest age peak of the Guayuriba and Guatiquía Rivers. This means that the exhumation occurring in the Upper Magdalena River Basin is slower than that on the eastern flank of the Eastern Cordillera, thus reflecting the asymmetric evolution of the Eastern Cordillera (Mora et al., 2008) and highlighting that volcanic zircons with young cooling ages are too low in abundance and therefore could not be detected. Similar to the zircon U–Pb data, the ZFT ages of the Magdalena River indicate that many of these zircons were recycled from older sedimentary units.

6. Conclusions

The U–Pb and ZFT data of the bedrock and modern river sediments of the Eastern Cordillera between Bogotá and Villavicencio, as well as the Magdalena River at Girardot, highlight the applications and complexities associated with interpreting detrital zircon age data. The main challenge of this technique is to use the observed grain age distributions to assign statistical significance to the observed age groups or peaks, and then to use these to identify the sediment provenance, detect sediment recycling and determine the cooling and exhumation histories of sediment source areas.

The new U–Pb and ZFT data presented here allow us to make first–order observations about the provenance signals in modern rivers on the east and west flanks of the Eastern Cordillera, close to the Villavicencio and Girardot areas. On the east flank of the Eastern Cordillera, our data clearly show that the zircon U–Pb age spectra of the Paleozoic through Mesozoic sedimentary section being eroded are related to sources in the Amazon Craton, the magmatic Paleozoic basement of the same Eastern Cordillera, and the exhumation of proximal Precambrian basement blocks. On the other hand, the Magdalena River sample indicates the presence of these same Eastern Cordillera sources plus the addition of younger Permian – Triassic and Jurassic zircons derived from the reworking of Upper Magdalena Valley sedimentary units and/or the crystalline basement of the Central Cordillera.

The ZFT data presented here complement the existing record of recent exhumation for the Eastern Cordillera determined based on the dating of bedrock samples, thus indicating that moderate exhumation rates occurred over the last 20 my and verifying the asymmetric nature of the surface uplift occurring across the length of the Eastern Cordillera fold–and–thrust belt.

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Explanation of Acronyms, Abbreviations, and Symbols:

CA–ID–TIMS	Chemical abrasion thermal ionization mass spectrometry isotopic dilution
CL	Cathodoluminescence
ID–TIMS	Thermal ionization mass spectrometry isotopic dilution
LA–ICP–MS	Laser ablation inductively coupled plasma mass spectrometry
LA–SC–ICP–MS	Laser ablation single–cell inductively coupled plasma mass spectrometry
SEM	Scanning electron microscope
SGC	Servicio Geológico Colombiano
ZFT	Zircon fission–track

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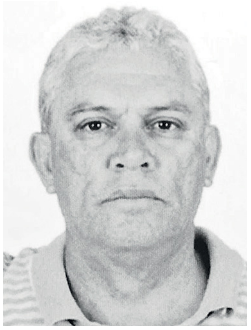

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