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FIRST CHARACTERIZATION OF OBSIDIAN FROM COLOMBIAN AND ECUADORIAN SOURCES USING ICP-AES AND ICP-MS

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41 obsidian samples from geological sources of SW Colombia and Ecuador were analysed by ICP-AES and ICP-MS to contribute to the build-up of a database for obsidian provenience studies. It is shown that obsidians from the Colombian Paletara caldera (Río Hondo and Río Granates compositional groups), the Ecuadorian sierra de Guamaní (Mullumica/Callejones and Quiscatola/Yanaurcu groups) and North Cotopaxi volcano (Quebrada Colorada group) can be easily distinguished from each other. It is also shown that obsidians with a given chemical composition may be found over a large distance, up to >100 km from volcanic structures (e. g. from ignimbrites to calites in NW of Paletara caldera).

KEYWORDS: OBSIDIAN SOURCES, COLOMBIA, ECUADOR, ICP-MS, ICP-AES.

Introduction

Obsidian was widely used by the prehispanic cultures of Colombia and Ecuador (Septentrional Andean Area, SAA), especially from the Formative period. Chemical characterization of a few hundred artefacts from this area proved that at least 7 obsidian sources were involved (Asaro et al. 1994; Burger et al. 1994; Bellot-Gurlet et al. 1998; Gnecco et al. 1993). Fission track dating showed that more sources than suggested by chemical analysis were used (Dorighel et al. 1997). However, a comparatively modest number of obsidian sources are known in the SAA and frequently only a small number per source have been analysed or only partial chemical analysis is available. Only three of these sources have been proved to be possible origins for certain artefacts of the SAA. These are, in Ecuador, the Mullumica flow and the blocky obsidian outcrops associated with a nearby deeply eroded volcano in the sierra de Guamaní of the *Cordillera Real* (Asaro et al. 1994; Burger et al. 1994) and in Colombia the Río Hondo source, South-East of Popayán (Gnecco et al. 1993).

In order to contribute to a better characterization of the SAA obsidian sources, we analysed by ICP-AES and ICP-MS forty-one samples coming from the presently known obsidian occurrences where this material is estimated to be of good knapping quality. The obsidians analysed were either directly collected by one of us (O. D.) during two field trips (1995, 1997) or sent us for analysis.

Sampling

Each of the sampled source zones (Fig. 1) presents several obsidian outcrops and have been already more or less extensively studied. Some of our obsidians are aliquots of samples previously used in other laboratories for fission track dating (Bigazzi et al. 1992) or INAA (Bigazzi et al. 1992; Gnecco et al. 1993; Glascock, pers. com.). In the following we describe briefly these obsidian sources and the precise origin of our samples. When a precise enough information on sample locality is known, it is marked on a map.

Colombia

In southwestern Colombia, obsidian is found in two types of deposits, either associated with the large ignimbritic nappes around the Paletara caldera, or at some distance to the North, as single nodules dispersed on the ground. We analysed samples collected in these two types of occurrence.

The Paletara caldera ignimbritic field. The ignimbrites of the Cauca and Huila Departments of SW Colombia expand both over the oriental and occidental flanks of the Central Cordillera (Kroonenberg et al. 1981; Murcia & Pichler 1986; Torres et al. 1992; Orrego et al. 1993) but it is only recently that their origin was related to a vast, about 40 km diameter, caldera (Torres et al. 1995). Many of the permanent and non-permanent mountain streams radiating from inside or outside this structure are cutting through the ignimbritic nappes, exposing fresh to reworked volcanic materials bearing obsidian. This is the case, for instance, for the Río Hondo and Río Negro to the NW of the caldera and the Río Granates, to the SE. The eleven samples we obtained come from these rios. In the Río Negro, four samples were taken in an c. 2 m² test pit in the river bank as cm sized obsidian nodules within ignimbritic blocks embedded in a volcano-sedimentary matrix. The Río Hondo "source" is located upstream, following C. Gnecco (pers. com.), and would consist in large nodules within an apparently altered volcanic matrix. Four of our samples come from this location (O. D. and C. G., 1995). South-east of the caldera three samples were taken by one of us (O. D.) along the Río Granates over a distance of about 5 km. These samples are obsidian nodules collected either in the river bed (two samples) or in an eroded river bank.

Calites. These obsidians, also previously known as "americanites", were once considered as possible tektites, because of their occurrence as dispersed glassy nodules lying on the ground surface over a large area in Southern Colombia between Cali and Popayán (Stutzer & Doring 1928; Martin 1934; Martin & De Sitter-Koomans 1955). Among the four "calites" analysed in this work, two were sampled by us in the northernmost part of the Department of Cauca near La Balsa

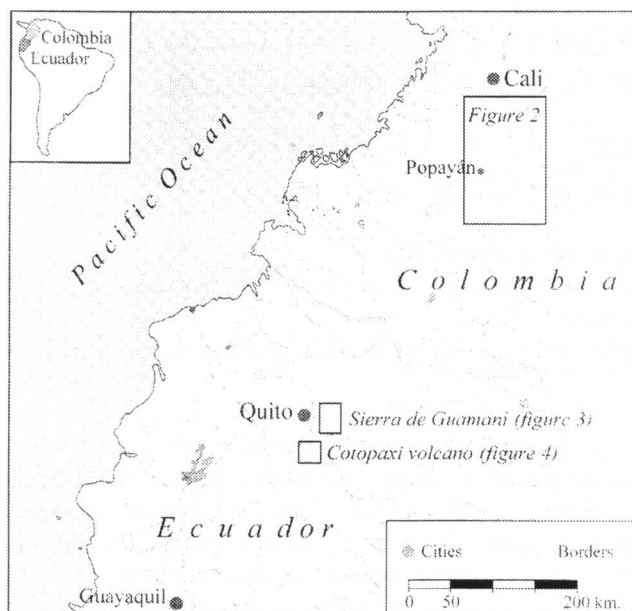


Fig. 1. Location of the sampled areas in Colombia and Ecuador. Rectangles : location of maps in Figs. 2 to 4.

and the other two, collected by Victoria Mejia, come from the Hacienda Corcovado about 10 km SE from our sampling point (Fig. 1).

Ecuador

Sierra de Guamaní. The sierra de Guamaní, also known as Chacana caldera (Hall & Mothes 1997) is located East of Quito in the Cordillera Real. It is there, in an approximately 200 km² area, that the major known obsidian occurrences of Ecuador are concentrated. We analysed samples from the three main sources in the area. Two of these (Mullumica and Callejones) are upper Pleistocene lava flows whereas the third one, a few km southwest, is composed of various outcrops related to an older, Plio-Pleistocene, activity.

The Mullumica source was discovered in September 1977 by Ernesto Salazar (Salazar 1980). It is a large rhyolitic lava flow, made of two overlapping lobes. The lower lava flow extends over a length of some 6 km and presents an average thickness of 150 m, whereas the upper flow is less important, being no more than 1.5 km long. Obsidian is found in many occurrences along the borders of the flow, where it appears as elongated bands (Salazar 1985; Bigazzi et al. 1992). Eleven samples taken in various locations along the flow were analysed. Seven are new samples, the others were previously analysed by INAA, three by Bigazzi et al. (1992) one by Glascock (pers. com.). The sampling locations of our samples and of those analysed by Bigazzi et al. (1992) are reported in the map of Fig. 2. The Glascock sample comes from an imprecise point of the lower Mullumica flow.

The Callejones source is a c. 3 km long lava flow located about 2 km east from the northern end of the Mullumica flow. The Mullumica and Callejones flows are located on each side of the local Andean watershed line, the latter facing the Amazonas basin. In 1997, Olivier Dorighel and Michel Monzier sampled several obsidians near to the village of Oyacachi at the base of a >100 m thick flow which is interpreted as one lobe of the Callejones flow previously mapped by Coltelli (Bigazzi et al. 1992), as illustrated in

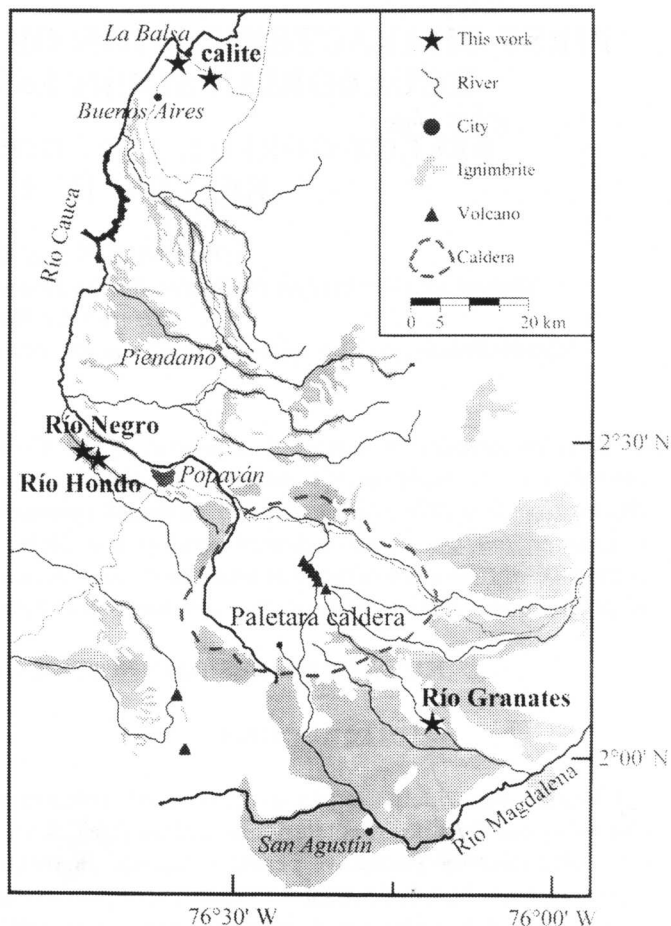


Fig. 2. Simplified map of the ignimbritic field associated to the Paletara caldera, SW Colombia (modified from Torres et al. 1995) and location of analysed samples.

Fig. 2. Among our six samples, two were freshly sampled, others were studied by INAA, two samples by Bigazzi et al. (1992) and two by Glascock (pers. com.).

The third obsidian source of the sierra de Guamaní is composed of several outcrops, apparently related to the same volcanic complex. Among these, the Cerro Yanaurco and Loma Quiscatola are two summits located in the southwestern part of the Chacana caldera. Following Bigazzi et al. (1992), this volcanism, older than that of Mullumica and Callejones, belongs probably to the top of the Plio-Pleistocene Basal Volcanic Complex (BVC) of Barberi et al. (1988). The first description of these deposits is given by Salazar (1980) who discovered them. In both cases they correspond to concentrations of obsidian blocks in the upper part of mounts interpreted by Bigazzi et al. (1992) as probable “erosional remnants of obsidian flows”. Six samples from these BVC materials were analysed, of which four had been previously analysed by NAA, three by Bigazzi et al. (1992) and one by Glascock (pers. com.). The Glascock sample is referred to the Quiscatola source but without field positioning information. The two other samples were recently (1997) collected by Pete Hall and Patricia Mothes in the Quebrada Encanada (Fig. 2).

Cotopaxi volcano. Located about 55 km South of Quito on the Central Cordillera, the Cotopaxi is the highest active volcano of the world. It produced obsidians both during an early – Palaeo-Cotopaxi – phase of activity, and more recently, after an andesitic effusive phase which resulted in the building of a cone (Bigazzi et al. 1997). The only data on

these obsidians are the fission track ages obtained by Bigazzi et al. (1997) for five nodules from pyroclastic ash-flow. Two samples from the Palaeo-Cotopaxi have plateau-ages around 0.55 Ma and three others, from the Cotopaxi, present apparent ages in the range 0.008–0.31 Ma.

The four samples we collected over a distance of some 600 m come from the Quebrada Colorado, on the Northern flank of the Cotopaxi cone (Fig. 3). The obsidians were in the form of small (<5 cm) nodules embedded in a pyroclastic matrix.

ICP-AES and ICP-MS analytical procedures

Obsidian samples were crushed and clear interior fragments (5–30 mg each) were selected. For each sample, about 150 mg of these chips were dissolved in a screw-top Teflon bomb using HF and HClO₄ at 120°C. Obsidians were without acid resistant phases and complete dissolution was always achieved. After evaporation to dryness of the HF-HClO₄ mixture, the samples were taken up in 40 mg of 6M HCl. Aliquots were then taken from this “mother solution” for the determination of elemental abundances (Barrat & Nesbitt 1996; Barrat et al. 1997).

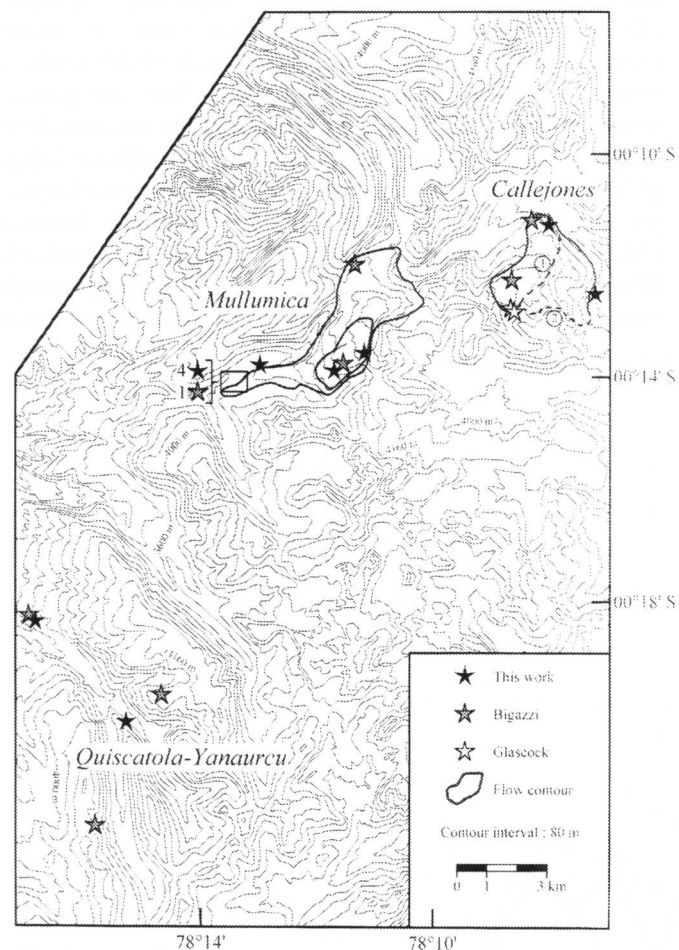


Fig. 3. Simplified topographic map of the sierra de Guamaní and location of analysed samples.

From the 1/100,000 topographic maps of the Instituto Geografico Militar, Quito, Ecuador (Sangolqui CTN-III-B-3993, Serie J 621 and Pintag CTN-III-D-3992, Serie J 621).

Dotted lines: 1, Southern limit of the Callejones flow as proposed in Bigazzi et al. (1992); 2, proposed modified contour; this work.

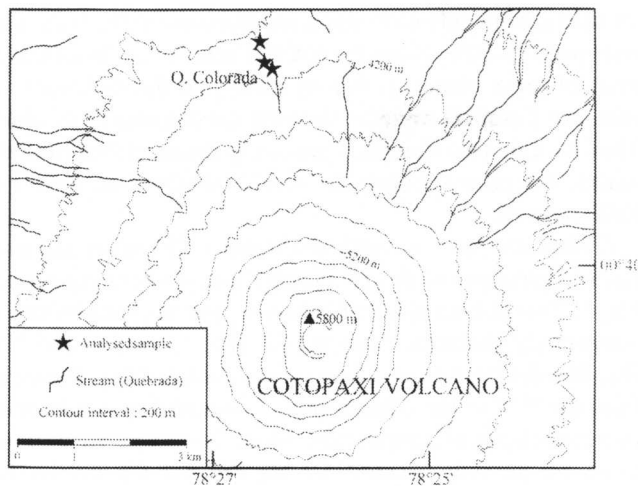


Fig. 4. Simplified topographic map of the Cotopaxi volcano and location of analysed samples from the 1/50,000 topographic maps of the Instituto Geografico Militar, Quito, Ecuador (Sincholagua CTN-III-D3, Serie J 721 and Cotopaxi CTN-III-F1, Serie J 721).

During ICP-MS measurements the signal drift was monitored by means of an In and Re spike added to the solution and a linear drift correction was applied. The ICP-AES signal drift was monitored by running a calibration standard solution every four samples and a linear drift correction applied accordingly. Blank solutions were prepared along with sample solutions and analysed together with them. Appropriate corrections were then applied for element concentration determinations.

The content of Al₂O₃, Fe₂O₃, MnO, MgO, CaO, Na₂O, K₂O, TiO₂ and P₂O₅ were determined by ICP-AES using a Jobin Yvon JY24 spectrometer. According to the measured elements the mother solution was diluted by a factor of about 200 to 20,000 for analysis. Silica, evaporated after sample dissolution, was not measured but its content can be estimated by difference. Elemental concentrations were calculated by reference with standard solutions and controlled by measurements of international standards (BIR-1, WS-E, RGM-1, UBN and BHV-01).

The contents of Co, Rb, Sr, Y, Zr, Nb, Ba, Tm, Hf, Ta, Th and U were determined by ICP-MS using a Fisons Plasma Quad turbo 2+ spectrometer. The solutions were diluted by a factor of about 2000–3000 for measurements. Elemental concentrations were calculated by reference to five geological international standards, BIR-1, WS-E, RGM-1, UBN and BHV-01 taking into account the sample dilution factor used for each sample.

The rare earth elements (REE) : La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb and Lu were determined with the same ICP-MS but after a Tm spiking and ion exchange separation of REE on an aliquot of the mother solution (Barrat et al. 1996). This preparation allows the determination of REE without various mass interferences from lighter elements. The REE abundances in the sample are calculated using the amount of Tm added (Barrat et al. 1996).

Results

The major element contents show that while the Cotopaxi samples are at the low end of the range of silica content of

obsidians, with only 67% silica, all others are at the high silica end, with 75–80%. From the A/CNK scheme of classification most of these obsidians belong clearly to the peraluminous clan, the Cotopaxi samples showing metaluminous affinities. Thus the Cotopaxi obsidians are easily distinguished from the others. We present below the results obtained on trace and REE element.

Calites and Paletara caldera obsidians. The major and trace element patterns for the 15 samples analysed are reported in Fig. 5. Two distinct kinds of behavior appear, characteristic of respectively obsidians from the SE (Río Granates) and NW (Río Hondo, Río Negro, Calites) of the caldera. The 12 samples from the NW of the caldera show a remarkably similar trace elements chemical composition. Slight deviations from sample to sample are only observed for Ba, Sm, Eu, Gd and Tb. For the last four elements, the samples split into two discrete groups having both sources and Calites representative of Río Hondo and Río Negro. The differences in element contents between these two groups are however subtle: at most, in the Eu case, it amounts to 60 ppb.

The three Río Granates samples SE of the caldera, collected at distance of a few km from each other, exhibit also a very homogeneous composition with only very slight variations from Sm to Gd. These obsidians are easily distinguishable from the obsidians of the NW of the caldera by their difference in element contents, especially their Ba abundances, but also Cs, Th, U and Sr to Er, in the spidergram of Fig. 5.

Sierra de Guamaní obsidians. The spidergram of the Mullumica and Callejones obsidians shows a larger compositional dispersion than observed for the Paletara caldera (Fig. 6). The similarity of the trace and REE element behaviors observed for samples from these two flows suggests that it might not be easy to find some distinctive com-

positional criteria between their obsidians. The six samples from the BVC in the vicinity of Loma Quiscatola and Cerro Yanaurcu (Fig. 3) show also some variations in their element spidergrams. However, as it appears in Fig. 7, this group of samples differs in some respects from that of the Mullumica-Callejones group in their spidergrams. In particular, elements like La, Ce and from Pr to Y are systematically less abundant than in the Mullumica and Callejones flows.

Cotopaxi volcano. The three obsidians from the Quebrada Colorada of the Cotopaxi volcano show a remarkably consistent element pattern (Fig. 8). When compared to the other obsidians analysed here, they have lower contents in Cs, Rb, Th, U, Nb, Ta and heavy REE, which allows a clear-cut distinction.

Discussion

Before this work was carried out, the data base on most of the sources analysed, relying on XRF or NAA, was therefore heterogeneous. With these more complete ICP analyses, one can re-examine the potentialities and limits of the chemical characterization of obsidian sources in an obsidian artefact provenience study perspective in the South Colombia-Ecuador area. One first point is that considering only major element contents, only one source can be easily distinguished: that of the Quebrada Colorada of the Northern Cotopaxi volcano flank. Discrimination between sources must therefore essentially remain on REE and trace elements patterns. The spidergrams of Figs. 5 to 8 have the advantage of presenting the general behavior of these elements but they do not directly offer ways of distinguishing between chemical compositions. Binary element-element or elements ratio versus elements ratios diagrams are more frequently used in provenience studies contexts. We give in Fig. 9 one

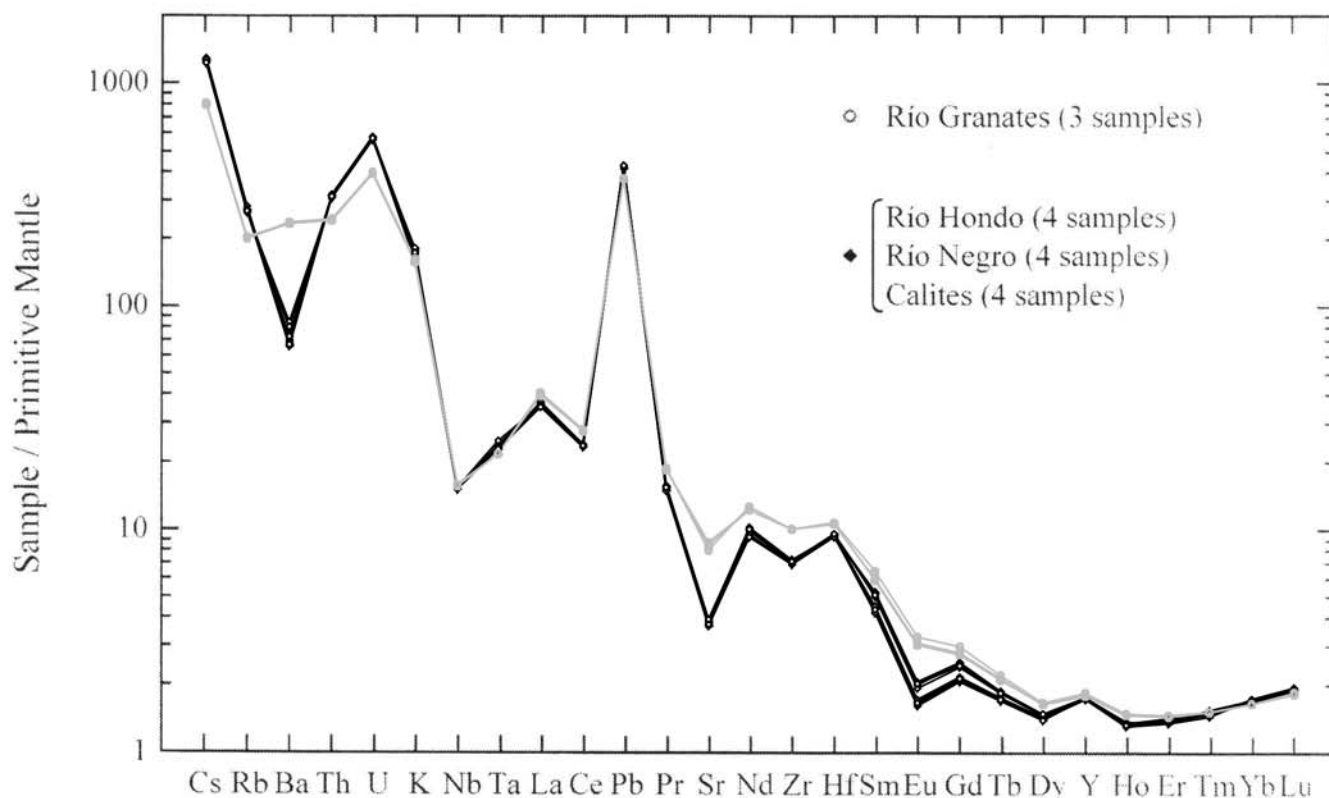


Fig. 5. REE and trace element abundance patterns normalized to the primitive mantle (Sun & McDonough, 1989) for south Colombian samples.

binary diagram in which our samples appear to be distributed into five discrete groups. Other kinds of binary diagrams would show the same sample groupings. We discuss them separately below.

Mullumica-Callejones compositional field. In this field, the 17 samples of the Mullumica and Callejones flows are present. In any binary diagram, the Callejones flow samples are essentially located at one end of the linear array. However, the Easternmost sample of the flow (Fig. 4) is well inside the Mullumica compositional field (see Fig. 6).

The linear behavior of the 11 Mullumica flow samples is strongly suggestive of a two-component magma mixing. This had also been inferred by Asaro et al. (1994) on the basis of the linear array shown in element-element binary diagrams built from five Mullumica flow obsidians and obsidian artefacts coming from various Ecuadorian archaeological sites.

As the Mullumica and Callejones obsidians have overlapping compositional fields, we consider that they cannot be distinguished from each other on a geochemical basis. This is why in the diagram of Fig. 9, we propose a unique field for the Mullumica-Callejones points. As these two flows have also indistinguishable fission track ages of 0.16–0.19 Ma (Bigazzi et al. 1992), we propose to name these two flows the Mullumica-Callejones source.

BVC samples of the Loma Quiscatola-Cerro Yanaurcu-Rodeo Corrales area. The six samples from this eroded structure of the BVC are arranged following a boomerang-like field in Fig. 9. As these samples come from local occurrences of obsidians over about an area of 20 km² they probably originated from various obsidian-producing events having related but different chemical compositions and ages. The fission track ages of obsidian samples from Loma Quiscatola, Cerro Yanaurcu and Rodeo Corrales, between

suggest these events might have occurred some 1.4–1.7 Ma ago (Bigazzi et al. 1992).

NW of Paletara caldera. Geochemical data are available for obsidians from three sectors outside the Paletara caldera. To the NW, samples from the Río Hondo source exhibit a rather constant composition, as can be shown on the 12 samples of Fig. 9 or the 17 samples analysed by Gnecco et al. (1993) by INAA. As same composition is found downstream for the Río Negro obsidians and especially northward up to the Cali-Popayán area with the calites (Fig. 9), the implication for provenience studies is that obsidian artefacts with a Río Hondo-type composition may come from a vast region from the NW border of the caldera to at least Cali, at some 110 km North.

SE of Paletara caldera. The chemical composition of obsidians from the Río Granates, well individualized in Fig. 9 is very close to that of the so-called Sotara source analysed by Gnecco et al. (1993). However the samples analysed by Gnecco et al. are not geological samples from the Sotara volcano (SW of the Paletara caldera), as could be understood from their report. Rather, they are artefacts (Gnecco, pers. com. 1997) from the archaeological site of Morelia (Lanos-Vargas 1988) located about 500 m from one of our Río Granates sampling points. The good agreement between the Gnecco et al. (1993) analyses of artefacts and our Río Granates samples suggests a local origin for the archaeological materials.

Fission track ages of samples from the Río Hondo and Río Granates are in the range 3.4–4.2 Ma (Dorighel et al. 1997 and Grenoble unpub. data), in agreement with two K-Ar ages of 3.7±0.2 Ma (Murcia & Pichler 1986) and 4.5 Ma (James et al. 1986) for ignimbrites of respectively the Huila and Cauca (Piendamón) regions. These obsidians appear as the oldest obsidian sources of Colombia and Ecuador, with the

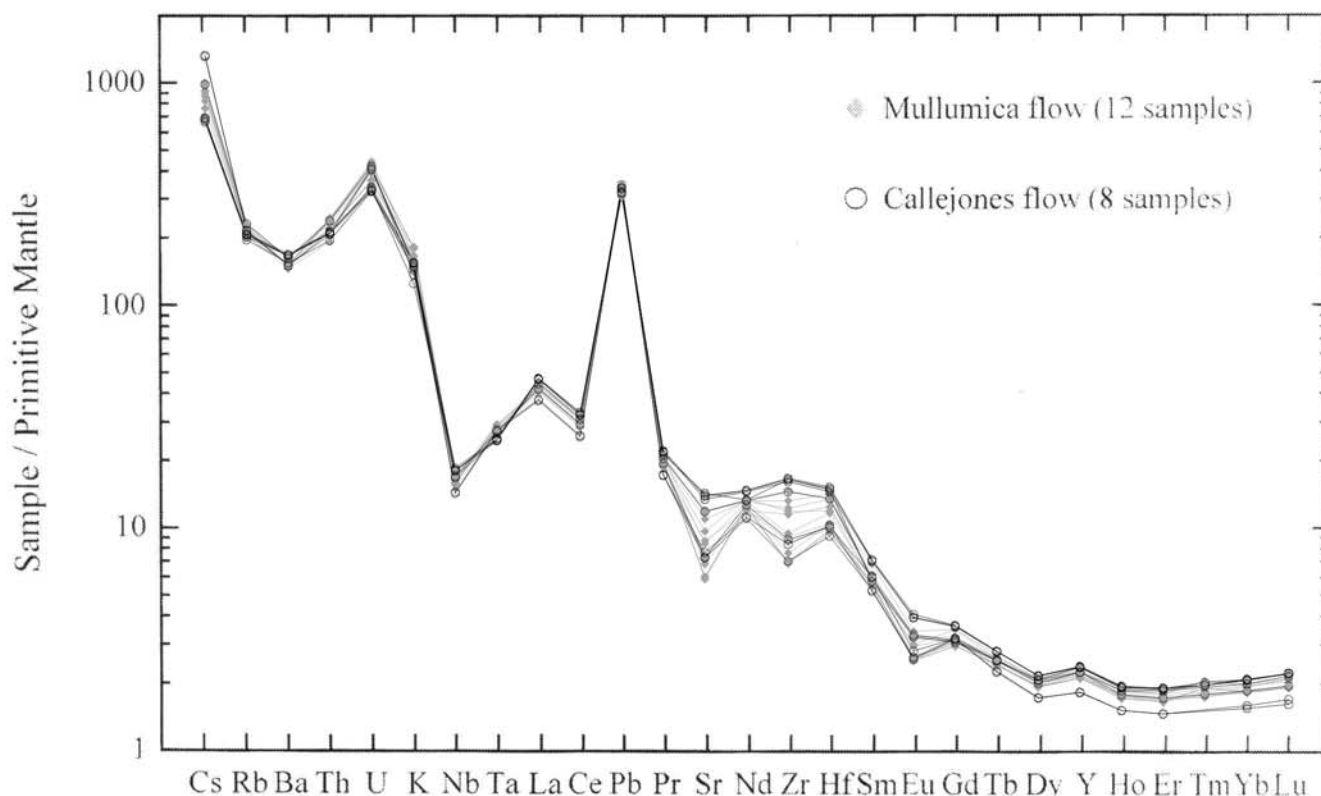


Fig. 6. Normalized REE and trace element abundance patterns for the Mullumica and Callejones flows, sierra de Guamaní samples.

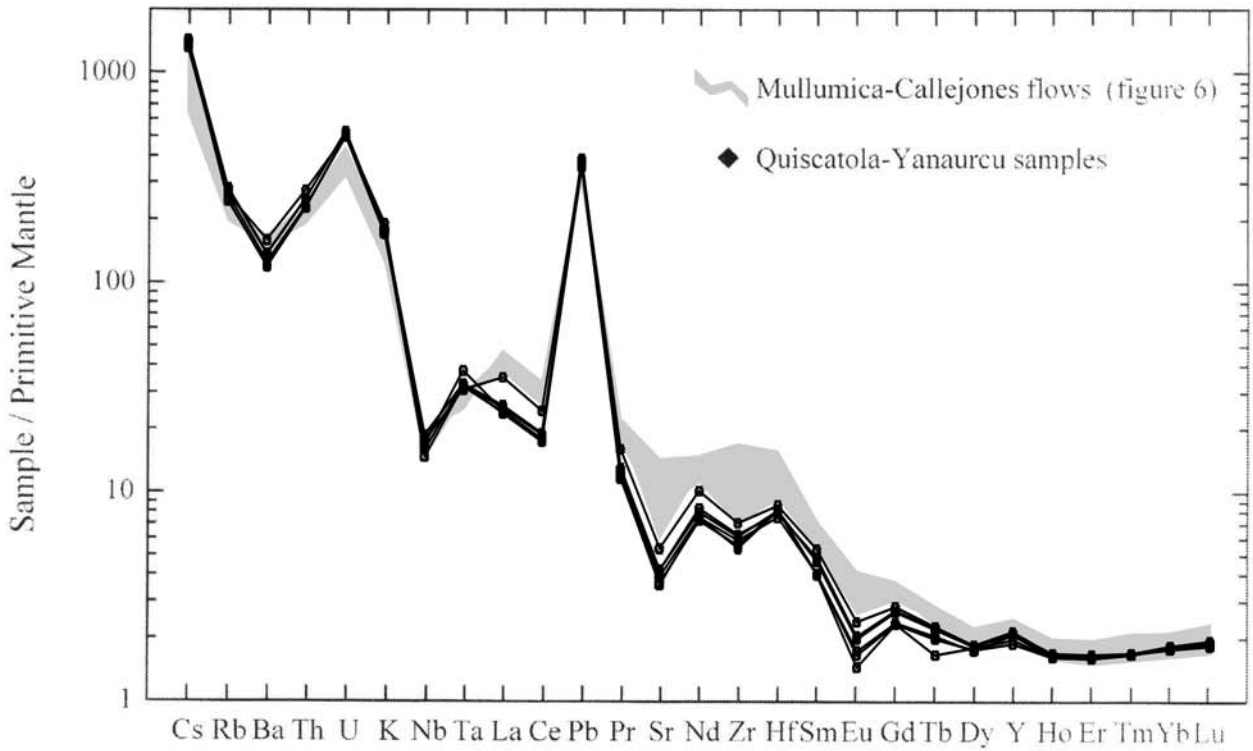


Fig. 7. Normalized REE and trace element abundance patterns for obsidians of the Basal Volcanic Complex, Loma Quiscatola-CerroYanaurcu area, sierra de Guamaní. Stippled: overall Mullumica and Callejones flows element patterns (from Fig. 6).

exception of an earlier fission track age of 6.5–10.2 Ma reported for one Colombian “americanite” by Storzer and Poupeau (1973).

Quebrada Colorada samples of the Cotopaxi volcano. The obsidians from the Quebrada Colorada appear presently as the easiest source to characterize geochemically as this is possible without ambiguity both from their major element

composition and their REE and trace element behavior. The age of our obsidians has not yet been determined.

Summary and conclusion

We re-investigated the chemical composition of obsidians known for their good knapping qualities in Southern

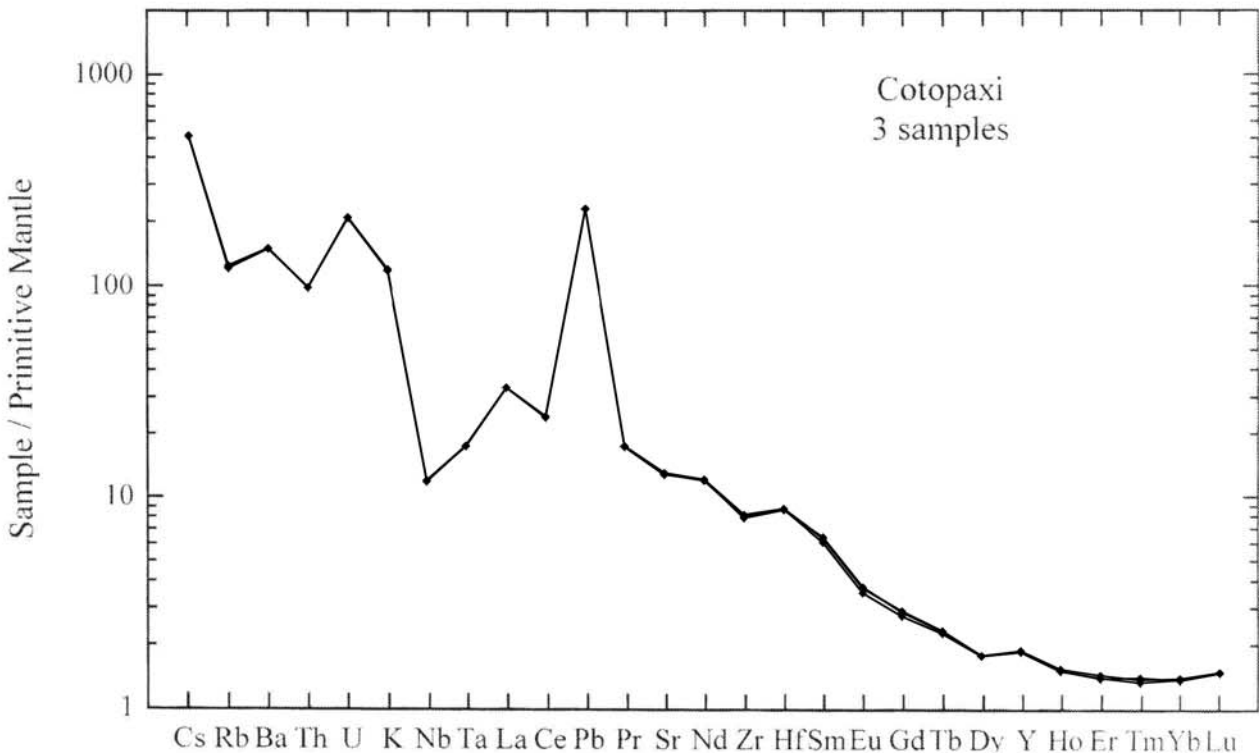


Fig. 8. Normalized REE and trace element abundance patterns for obsidians of the Quebrada Colorada, north flank of the Cotopaxi volcano.

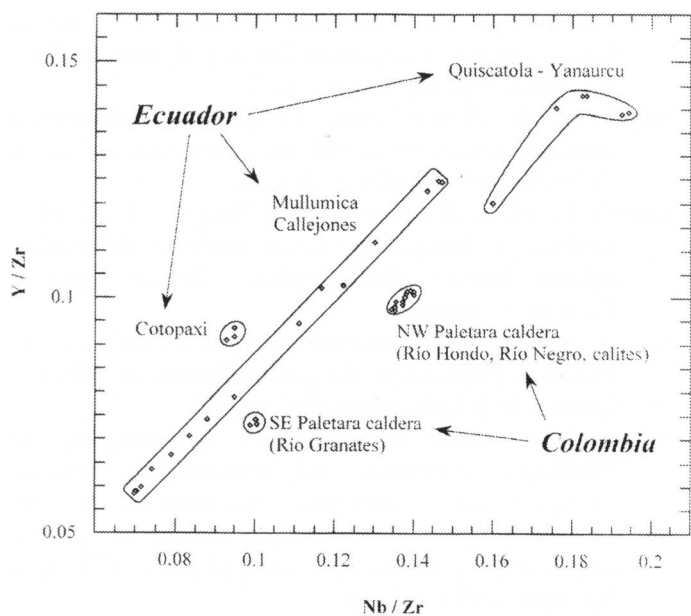


Fig. 9. Binary diagram comparing the Y/Zr versus Nb/Zr elementary ratios for the 41 obsidians studied.

Colombia and Ecuador, based on the measurement of 36 element contents by ICP-AES and ICP-MS. In Colombia, obsidians were collected in six locations on the SE and NW sides of the Paletara caldera but belong to only two compositional groups, the already known "Río Hondo" group (Gnecco et al. 1993) and the new "Río Granates" group. Obsidians of the Río Hondo group are found over a wide territory to the NW of the caldera. The spatial extension of the obsidian occurrences with a Río Granates composition remains, as for those of the Río Hondo type, to be determined.

In the sierra de Guamaní, we substantiate the binary mixing nature of the Mullumica magma, as suggested previously by Asaro et al. (1994) on the basis of the common behavior of elemental composition of obsidian artefacts and five samples from the flow itself. We show that the Callejones and Mullumica flows cannot be distinguished from their chemical composition, as suspected by Bigazzi et al. (1992) from a dendrogram of the chemical composition of the sierra de Guamaní obsidians. Thus obsidians with a "Mullumica"-type of composition must be referred to as coming from one of these two flows. We have also shown that the obsidian block-bearing remains of an eroded volcano from the Basal Volcanic Complex, in the Loma Quiscatola-Cerro Yanaurcu-Rodeo Corrales area of the sierra de Guamaní, albeit somewhat heterogeneous compositionally, can be easily discriminated from the "Mullumica" compositional type.

Finally, preliminary results are presented for the Cotopaxi volcano. Our samples, of ages not yet determined, are chemically very different from all other obsidians of the area. Other samples from this volcano were recently dated by fission tracks around 0.55 Ma (plateau ages) or 0.008–0.18 Ma (apparent ages), (Bigazzi et al. 1997).

Thus a regional discrimination is now possible on the basis of geochemical analysis or age determination, if looking for a local quarry origin for an artefact still appears frequently an premature goal. Further works must be pursued in several directions, such as to continue the documentation and analysis of samples for sources presently characterized by

only a few samples (e.g. Río Granates, Quebrada Colorada), to have a better coverage of some areas, as around the Paletara caldera and to prospect for yet unknown sources used in the past, as implied from fission track data (Doriguel et al. 1997) and chemical characterization (Bellot-Gurlet et al. 1997) of obsidian artefacts from the Septentrional Andean Area. Although artefacts are most often made of transparent obsidians, the investigation of sources where obsidians is of "low" knapping quality should not be neglected.

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