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COLOMBIA

Colombian Emerald Reserves Inferred from Leached Beryllium of their Host Black Shale

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Abstract — We present a new approach for the evaluation of the potential of emerald reserves in gem projects using beryllium (Be) mobility. The distribution of Be in the different phases of hydrothermally altered (albitized) black shale hosting Colombian emerald deposits indicates that Be mobility is associated with the breakdown of Fe-Mn oxyhydroxide phases. The amount of Be that can be mobilized (~0.7 x 10^{-6} g/g; i.e., 0.7 ppm) may represent up to 18 wt% of the total Be contained in the black shale. A mass balance calculation applied to a well-constrained alteration zone of the Chivor mines, and based on the quantification of Be that can be mobilized under hydrothermal conditions, gives an estimate of the total gem reserves of these mines. **© 2000 Canadian Institute of Mining, Metallurgy and Petroleum. All rights reserved.**

Resumé — Une nouvelle approche, basée sur la mobilité du béryllium (Be) pour l'évaluation des réserves d'émeraude dans des projets d'exploitation de gemmes est présentée. La distribution du Be dans les différentes phases minérales de schistes noirs hydrothermalisés (albitisés) qui contiennent les gisements d'émeraude colombiens, indique que le Be mobilisable se trouve dans les oxyhydroxydes de fer et de manganèse. La quantité de Be potentiellement mobilisable (~0,7 x 10^{-6} g/g: soit 0,7 ppm) constitue jusqu'à 18% poids du Be global contenu dans le schiste noir. Un bilan de masse appliqué à la zone d'altération hydrothermale des mines de Chivor et basé sur la quantification du Be mobilisable, donne une première estimation des réserves à émeraude de ces mines.

Introduction

The legend of the royal couple, Fura and Tena, is part of the Muiscas Indians' mythology and explains the formation of Colombian emeralds. Fura wept at the death of her prince Tena, her tears permeated the Earth and became emeralds. Thus, the gemstones were derived from the sky as fruits of the gods. Ideas have moved on since, and the enigma of the formation of Colombian emeralds has been extensively discussed in the recent scientific literature (e.g., Renders and Anderson, 1987; Ottaway, 1991; Giuliani et al., 1992; Cheilletz et al., 1994; Ottaway et al., 1994; Giuliani et al., 1995; Cheilletz and Giuliani, 1996). These data suggest a model involving the interaction of basinal fluids with evaporites and organic-bearing black shales of Lower Cretaceous age. Colombian emeralds represent a unique example of beryllium mineralization developed in a black shale environment and genetically disconnected from magmatism. Beryllium (Be) and chromium (Cr) which combine to form emerald, are derived from the host black shale (Kozlowski et al., 1988; Ottaway, 1991; Giuliani et al., 1993; Ottaway et al., 1994). However, the phases which host the Be in the black shale remain unknown. In this study, we carried out a comprehensive evaluation of the distribution of Be between the different phases of the black shale in the El Toro and Chivor mines (eastern Colombian emerald zone). The data were obtained using sequential leaching techniques and atomic absorption spectrophotometry (AAS). They permit quantification of the amount of Be leached from the black shale, and allow us to estimate the mass of emerald crystals precipitated during hydrothermal activity.

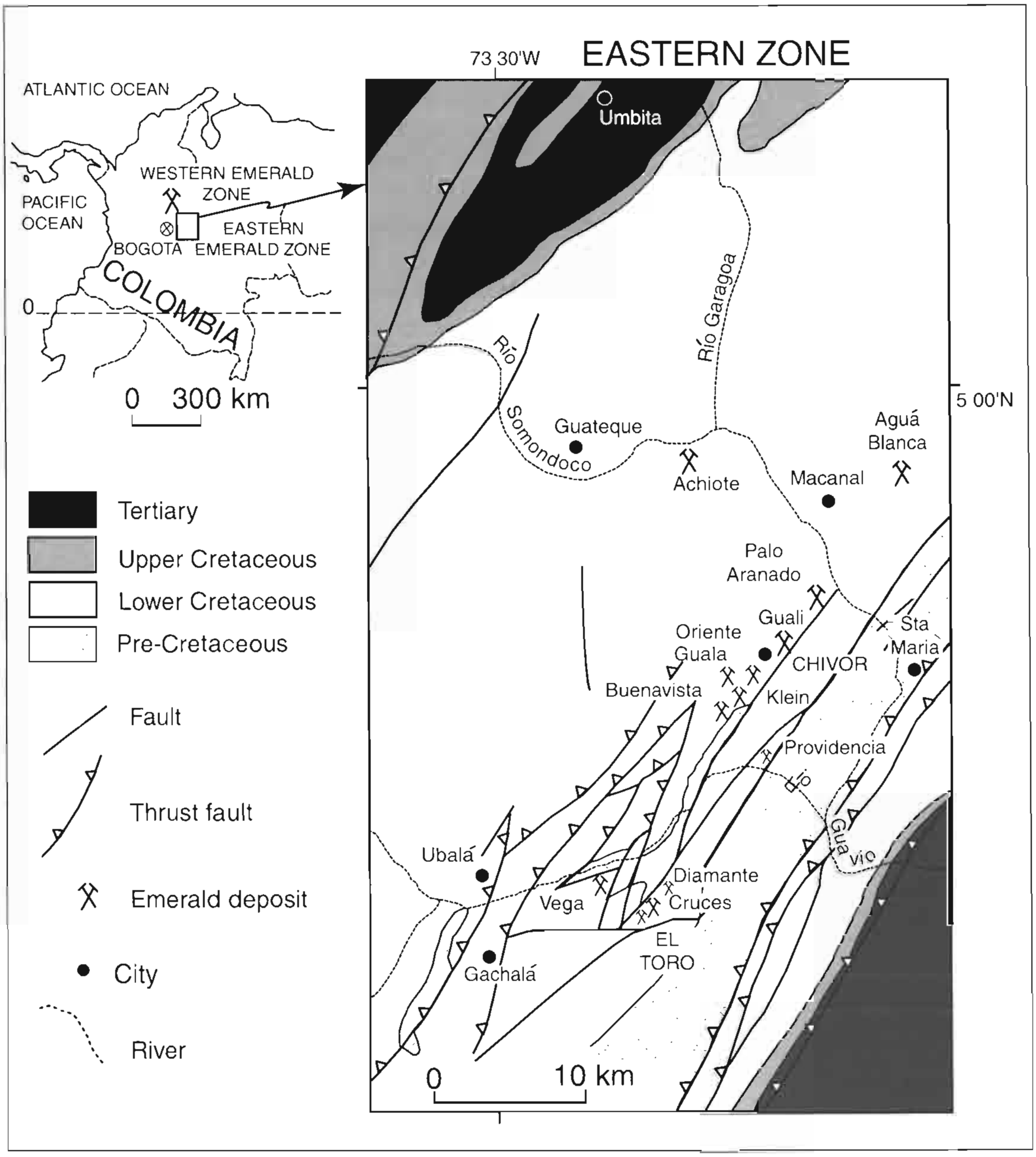

Fig. 1. Location and simplified geological map of the Colombian emerald deposits within the eastern emerald zone of the Eastern Cordillera (mining districts of Chivor and Gachala). The location of the eastern and western emerald zones are shown in the inset.

Geological Setting

Colombia has become the world's most important emerald producer with an estimated 1996 production of 15 million carats or 60% of total world production (Mineralco, 1996). This is followed by Zambia (15%), Brazil (10%), Russia (5%), Madagascar (3%), Zimbabwe (2%), Pakistan and Afghanistan (5%).

The Colombian emerald deposits are located at both the eastern and western borders of the Eastern Cordillera (Fig. 1). Two mineralized zones are hosted within Early Cretaceous black shale series: 1) the eastern zone comprises the mining districts of Gachalá (El Toro mine), Chivor (Porvenir, Klein, and Oriente mines), and Macanal; 2) the western zone includes mines in the Coscuez, Muzo, and La Palma-Yacopi districts.

The eastern emerald zone deposits are contained within the Lower Cretaceous series. The deposits are hosted by the Berriasian horizon of the Guavio series, which is overlain by siliceous black shales of the Valanginian Macanal series (Ulloa and Rodriguez, 1976). In the Chivor mining district, the Guavio series lies unconformably on a Paleozoic basement (Fig. 1). It is composed from bottom to top of: siltstones and siliceous black shales, carbonaceous black shale sequences that contain intercalations of limestone lenses and anhydrite beds evidenced by phantom nodules, chicken wire and tepee structures (Branquet et al., 1999), and shelly limestone grading to black shales intercalated with olistostromes.

The western emerald deposits are hosted within black shales and intercalated dolomitic limestones of Valanginian-Hauterivian ages (Rosablanca and Paja formations). The lithologies, from top to bottom, are: dolomitic limestones (Rosablanca formation), carbonate-bearing black shales (Hauterivian), which are the main emerald-hosting horizons, siliceous black shales (Hauterivian), and mudstones (Paja formation).

Two distinct formation ages have been obtained for the emerald deposits. An age of 65 Ma has been obtained for the Chivor mines (Cheilletz et al., 1997), and 38 Ma to 32 Ma for the Coscuez-Muzo mines (Cheilletz et al., 1994). In the western zone, the deposits are the consequence of a compressive tectonic phase characterized by thrusts and faults which contain the mineralization (Laumonier et al., 1996). In the eastern zone, emerald mineralization occurred during a halokinetic phase developed in an extensional regime (Branquet et al., 1999). In the two zones, emerald-bearing veins are spatially associated with stratiform breccias and albitites, the latter resulting from Na-metasomatism of the black shales. The veins consists of parallel, *en-echelon* or conjugate arrays grouped into two successive stages of extensional vein systems (Cheilletz and Giuliani, 1996). Stage 1 is characterized by décollement planes that focused the circulation of hydrothermal fluids which produced albite and calcite-dolomite alteration of the black shales. Stage 2 is marked by the formation of extensional vein sets and hydraulic breccias filled by muscovite, albite, calcite, dolomite and pyrite, and, lastly, by the precipitation of fluorite, apatite, parisite, dolomite, emerald and quartz in drusy cavities.

Methods

Sequential leaching procedures designed to determine the beryllium distribution among the phases constituting sediments were applied to twelve selected samples. This experimental method was previously developed, tested, and validated to estimate the potential of the authigenic $^{10}Be/^{9}Be$ as a marine sediment dating tool (Bourlès et al., 1989) and to quantify the mobility of beryllium in chemical conditions prevailing in extreme environments such as submarine hydrothermal systems (Bourlès et al., 1992). Its goal is to isolate the Be associated with the following phases (the quantities are those necessary for ~1 g of dry material, our typical sample size):

- Phase a, exchangeable (adsorbed): agitation for one hour at room temperature in 8 ml of 1 M $MgCl_2$ at pH 7.
- Phase b, calcium carbonate: agitation for five hours at room temperature in 8 ml of 1 M NaOAc adjusted to pH 5 with HOAc.
- Phase c, iron and manganese oxyhydroxydes: agitation for six hours at (96 ± 5°C) in 20 ml of 0.04 M $NH_2OH.HCl$ in 25% (V/V) HOAc.
- Phase d, organic matter: agitation for two hours at (80 ± 5°C) in 3 ml of 0.02 M HNO_3 and 5 ml of 30% H_2O_2 (pH 2 with HNO_3). Add another 3 ml H_2O_2 (pH 2 with HNO_3) and further agitation at (80 ± 5°C) for another three hours. After cooling, addition of 5 ml of 3.2 M NH_2Oac in 20% (V/V) HNO_3 to prevent readsorption of extracted Be onto the oxidized material.
- Phase e, residual silicates: complete dissolution in HF and H_2SO_4.

This experimental method was adopted mainly because:

1. the calcium carbonate fraction is extracted before dissolution of the other acid reducible phases. If not, the carbonate content can buffer the extractive solutions;

2. all the dissolution steps are executed in an acidic medium. At pH>5, beryllium tends to precipitate or to be strongly re-adsorbed on the walls of the container or on the remaining particles (Bourlès, 1992).

For each extraction, separation was carried out by centrifuging at 5500 rpm for 15 minutes. The supernatant was removed with a pipet and the residue washed twice with 2 ml portions of the leaching solution, which were then added to the supernatant.

Beryllium measurements in each extraction were made by flameless graphite-furnace atomic absorption spectrophotometry (AAS) (Perkin-Elmer Zeeman/3030 spectrophotometer, model HGA-600 furnace, model AS-60 auto sampler) using the method of standard additions and a Zeeman-effect background correction. The detection limit of this method is 6×10^{-11} g/g in a 1 ml sample with a constant conservative uncertainty of 5%.

Petrology and Analytical Results

The Berriasian black shales, which make up the wallrock of the El Toro and Chivor mines (Fig. 1), are characterized by high organic matter contents, averaging 2 wt%. The black shale parent rocks are composed of detrital components (mainly quartz, micas, and feldspars) and authigenic phases (organic matter, Fe-Mn oxhyhydroxides and biogenic precipitates such as silica and pyrite). Their total Be concentration (mean = $3.7 \pm 0.4 \times 10^{-6}$ g/g; n = 10) measured using plasma emission spectrometry (PES) is generally similar to that of average shale (Vine and Tourtelot, 1970). In the study area, the formation of emerald is related to a high-temperature hydrothermal event (T = 300°C; P = 1 kb; Cheilletz et al., 1994; Ottaway et al., 1994) involving the interaction of mesothermal brines (salinity of 38 wt% equivalent NaCl) with the black shales 65 Ma ago (Cheilletz et al., 1997). The resulting fluid/rock elemental exchanges, which lead to an albitic metasomatic alteration of the black shale, can be expressed by Na/Al ratio (Fig. 2), and to emerald-pyrite-carbonate crystallization in veins and vugs. Total Be concentrations for leached black shales (daughter rocks) range from 0.6 to 3.1×10^{-6} g/g (mean = $1.4 \pm 0.8 \times 10^{-6}$ g/g; n = 22).

Total Be concentrations, calculated by summing the Be concentration measured by atomic AAS in each sample constitutive phases are similar to those measured by plasma emission spectrometry (PES) in the whole rock samples. In the parent rocks, 3.84 ± 0.26 x 10^{-6} g/g (n = 7) of Be calculated using the AAS measurements is comparable to 3.73 ± 0.38 x 10^{-6} g/g (n = 7) of Be measured by PES, as is, in the daugther rocks, 2.93 ± 0.73 x 10^{-6} g/g (n = 5) calculated using the AAS measurements to 2.70 ± 1.00 x 10^{-6} g/g (n = 5) measured by PES (Table 1).

Discussion

In the El Toro mine, the parent rocks are carbonate-rich black shales (LC). Major element analysis of parent rock samples, G1, G2, and G3, and daughter rock samples, G7, G9, and G13, shows that increasing Na/Al values relate to increasing degree of metasomatic alteration, which in turn, correlates with a general decrease of the total Be (Fig. 2). The relative distribution of Be between the analyzed phases, when compared to the degree of alteration (as shown in Fig. 3) clearly indicates that this decrease mainly results from the leaching of Be associated with the Fe-Mn oxyhydroxides (phase c). The amount of Be associated with the Fe-Mn oxyhydroxides is indeed lower by 10 to 18 wt% in the parent rocks (G1, G2, G3), to less than 0.8% in the most Be-depleted daughter rock samples studied: (G9; Table 1 and Fig. 3). This implies a potential release of ~0.7 x 10^{-6} g of Be per gram of parent rock during the alteration process.

In the Chivor mine, where the parent rock is a siliceous black shale (LS, sample 95-37), hydrothermal alteration leads to the formation of a continuous stratigraphic level of greyish albitite daughter rocks which host the emerald veins. This structure has a geometry similar to a parallelepiped 900 m long, 30 m thick, and about 100 m deep in the workings of the Oriente, Klein, and Porvenir deposits. In spite of the difference in the parent rock (Fig. 3), Fe-Mn oxyhydroxides also appear to be the main mobile Be-bearing phase in this mine. The Fe-Mn oxyhydroxides contribution to the absolute total Be concentration decreases from 18 wt% (Fig. 3) in the parent rock to less than 3 wt% (Fig. 3) in the albitite-rich sample, G42 (Na/Al parameter = 0.69), implying a potential release of ~0.6 x 10^{-6} g of Be per gram of parent rock during the hydrothermal alteration. Sample G43, which contains ~20% of secondary muscovite, has a total Be concentration comparable to the parent rocks (Table 1). In this particular case, at least ~65% of the Be originally associated with the Fe-Mn oxyhydroxides was,

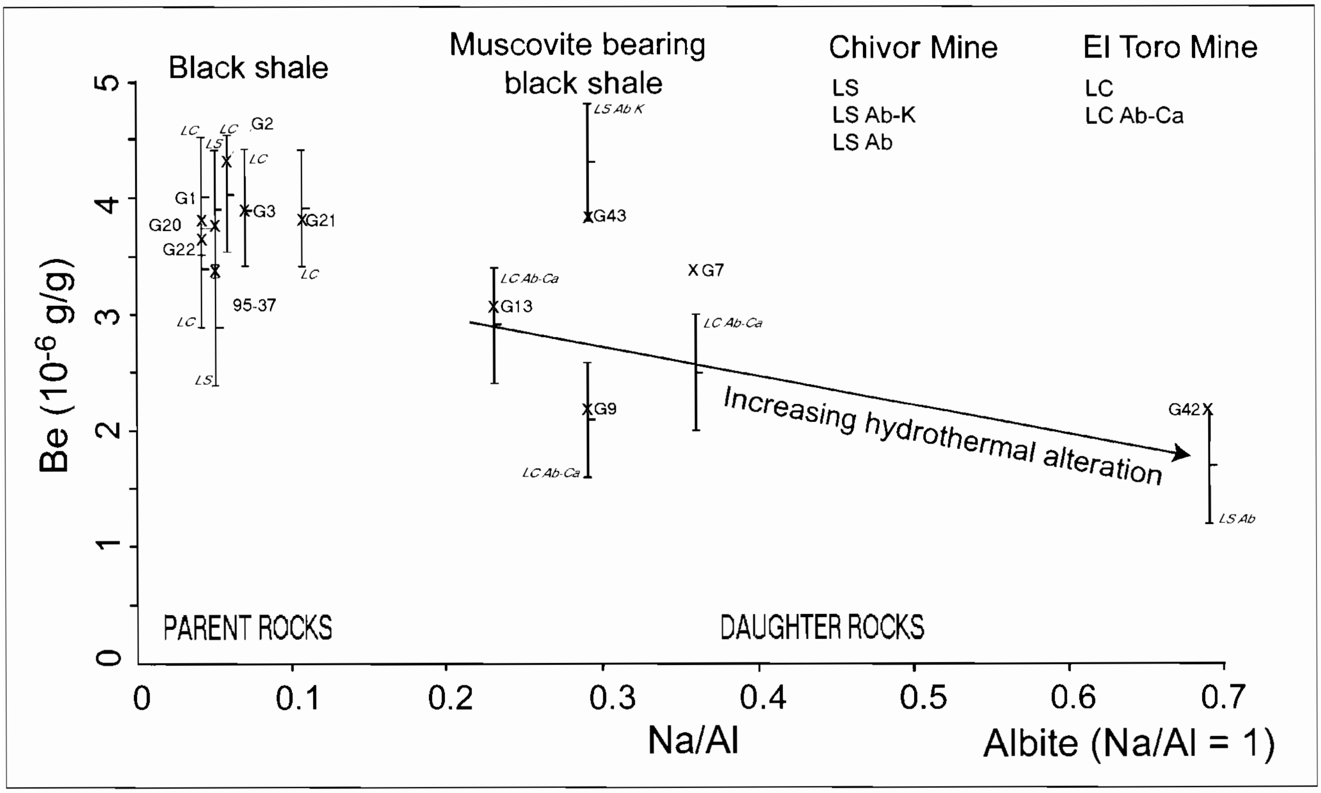

Fig. 2. ICP-MS (symbol: -) and Atomic Absorption Spectrophotometry (AAS) (symbol: x) Be concentrations as a function of the Na/Al alteration parameter for the parent and the hydrothermally altered daughter rocks from the Chivor and El Toro mines. LS: siliceous black shale; LS Ab-K: albitized siliceous black shale containing hydrothermal muscovite; LS Ab: albitized siliceous black shale; LC: calcareous black shale; LC Ab-Ca: albitized and carbonate altered calcareous black shale. The error bars correspond to the ICP-MS 0.5 x 10^{-6} g/g constant uncertainty. A constant conservative uncertainty of 5% is applied to each AAS measurement. The presented total AAS concentrations equal the sum of the different phases (see text). All samples except G7 are within the range defined by the ICP-MS uncertainty. The data show that an increase in hydrothermal alteration results in a significant decrease in Be concentration. Despite its elevated Na/Al value, sample G43 shows an abnormally high Be concentration, most likely due to the presence of secondary muscovite.

Table 1. Be concentrations measured by Atomic Absorption Spectrophotometry (AAS) in different phases (Phase a: adsorbed; Phase b: calcium carbonate; Phase c: iron and manganese oxyhydroxides; Phase d: organic matter; Phase e: residual silicates) extracted by sequential leaching techniques (see text) in parent rocks (PR) and daughter rocks (DR) from El Toro and Chivor mines. The Na/Al value increases with the degree of alteration. A constant conservative uncertainty of 5% is applied to each AAS measurement. The total AAS concentrations equal the sum of the different phases. They can be compared to the bulk rock ICP-MS concentrations having an associated uncertainty of 0.5 x 10^{-6} g/g (0.5 ppm).

Mineral Sample	Na/Al	Phase a (10^{-8} g/g)	Phase b (10^{-8} g/g)	Phase c (10^{-8} g/g)	Phase d (10^{-8} g/g)	Phase e (10^{-6} g/g)	Total AAS (10^{-6} g/g)	ICP-MS (10^{-6} g/g)
El Toro								
G1 PR*	0.05	33.56	12.59	39.63	6.24	2.81	3.73	4.00
G2 PR*	0.07	0.37	9.61	77.11	8.67	3.36	4.32	4.00
G3 PR*	0.08	1.09	13.94	59.91	5.86	3.08	3.88	3.90
G21 PR*	0.11	0.23	3.54	51.37	4.18	3.18	3.77	3.90
G22 PR*	0.05	0.12	6.66	11.56	1.54	3.68	3.88	3.40
G7 DR†	0.36	12.36	17.45	40.84	5.27	2.61	3.37	2.50
G9 DR†	0.29	0.30	9.39	1.72	3.92	2.02	2.18	2.09
G13 DR†	0.23	0.22	19.04	41.69	8.17	2.37	3.06	2.90
Chivor								
95-37 PR§	0.13	0.13	5.36	63.30	7.16	2.69	3.45	3.00
G20 PR§	0.06	11.24	13.05	44.39	7.06	3.06	3.82	3.90
G42 DR#	0.69	0.13	4.42	5.77	1.64	2.07	2.19	1.70
G43 DR$	0.29	25.61	4.97	15.40	5.96	3.31	3.83	4.30

* LC: calcareous black shale.
† LC Ab-Ca: albitized and carbonate altered calcareous black shale.
§ LS: siliceous black shale.
# LS Ab: albitized siliceous black shale.
$ LS Ab-K: secondary muscovite bearing albitized siliceous black shale.

however, preferentially leached by the hydrothermal fluids, as observed for the other daughter rocks (Table 1). Examination of the distribution of Be among the sample, G43, constitutive phases strongly suggests that the Be leached from the Fe-Mn oxyhydroxydes may have been simply transferred to the adsorbed phase of the OH-bearing muscovite (Table 1 and Fig. 3). This is in good agreement with the hydrolytic behavior of Be in aqueous solution (Chih Wong and Woolings, 1994).

The data set presented here demonstrates that within black shale the leachable Be necessary to form emerald is mainly associated with Fe-Mn oxyhydroxide phases. The amount of Be that could potentially be mobilized from the parent rock may represent up to 18% of the total Be contained in these rocks (Table 1), i.e., up to 0.7 x 10^{-6} g of Be per gram of black shale.

Inert Be appears to be associated with organic matter (phase d) and incorporated within the operationally defined residual phase (phase e). This last phase results from the total dissolution of all material remaining after the desorption step and dissolution of the carbonates, Fe-Mn oxyhydroxydes, and organic matter. Therefore, it probably corresponds to the Be within the matrices of resistant minerals such as phengites (white micas) and common heavy minerals. Table 1 shows that the Be concentrations measured within the daughter rocks residual phase are similar to those measured within the related parent rocks residual phase, whatever the type of parent rock considered (carbonated or siliceous black shale). As previously reported for hydrothermally altered marine sediments (Bourlès et al., 1992), this indicates redistribution of inert Be between sedimentary phases recrystallized during hydrothermally induced metasomatism. This uptake into resistant secondary minerals of the parent rock inert Be challenges the estimation of Cheilletz et al. (1999).

The results of this work can be used to estimate the amount of emerald crystals formed from the Be extracted during hydrothermal activity. For example, due to its well-constrained alteration zone (see above), the Chivor mine provides an ideal opportunity to model emerald formation. In this mine, gem extraction during the time period, 1926-1995, is estimated at 3985 kg (Feininger, 1971; Mineralco, 1996). This is equivalent to 300 000 crystals of 1 cm diameter and 5 cm length. Considering that the amount of mobile Be extracted from each gram of black shale by the hydrothermal fluids is in the order of 0.7 x 10^{-6} g, mass estimation of the alteration zone in the Porvenir, Klein, and Oriente workings leads to ~470 kg of Be leached, and therefore to the potential precipitation of ~720 000 emeralds (Fig. 4).

Conclusions

The hydrothermally induced breakdown of Fe-Mn oxyhydroxide phases is responsible for the mobilization of Be necessary to crystallize emeralds. Up to 18% of the total parent rock Be concentration can potentially be mobilized. Our data allow us to estimate gem reserves in a mining district using a mass balance calculation based on the quantification of hydrothermally mobilized Be, rather than using ore grade data. Considering the generally erratic distribution of ore in gem deposits, this new approach could be an important tool in the evaluation of the economic validity of gem projects, as it permits the estimation of the potential reserves at the initiation stage of mining evaluation.

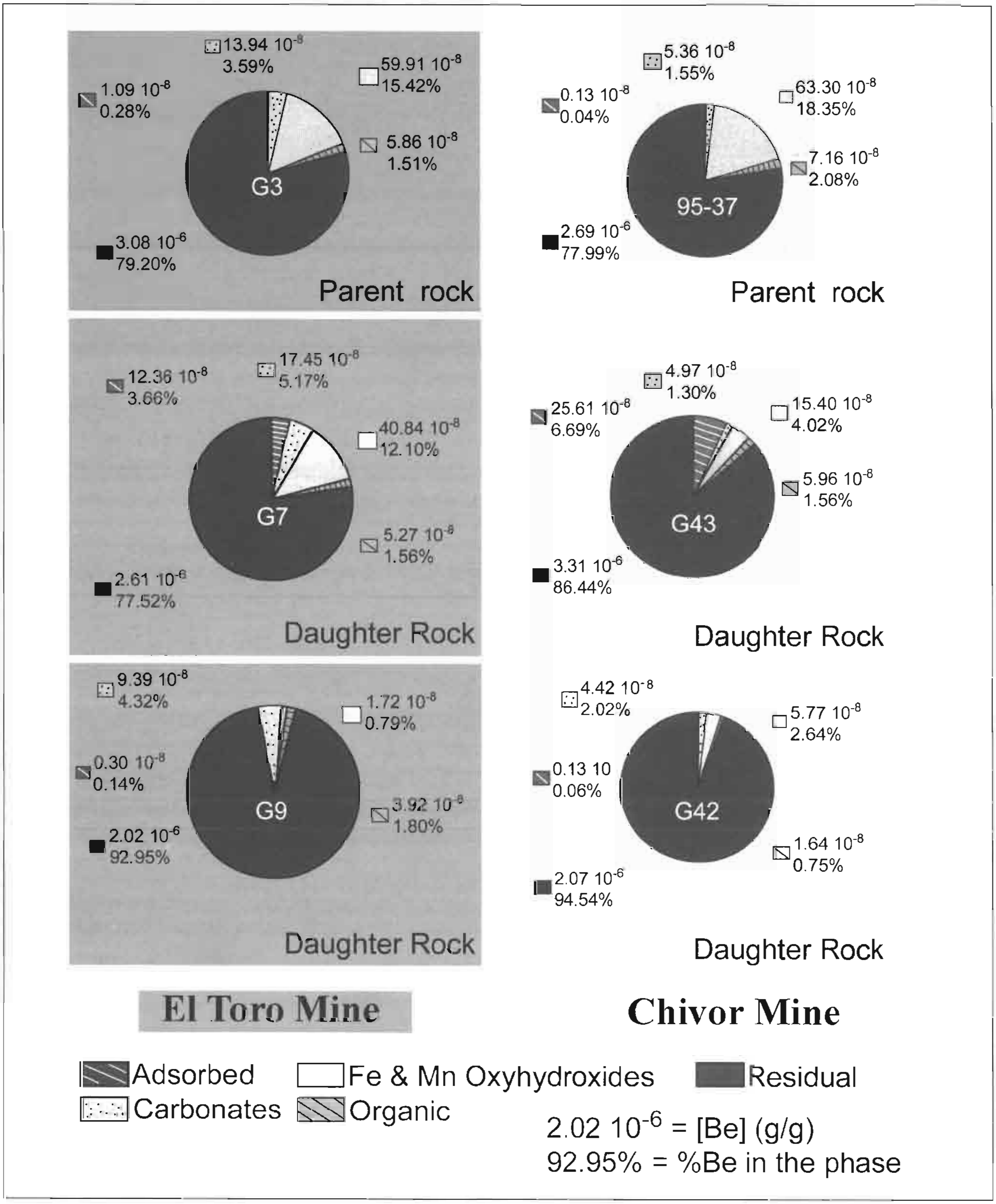

Fig. 3. Pie diagram representation of the concentration and relative distribution of beryllium between the different experimental phases extracted from parent and daughter rocks. For the El Toro mine, a sequence including a parent rock (G3) and two daughter rocks (G7 and G9) at different degrees of alteration (see Fig. 2) is presented. For the Chivor mine, the sequence includes a parent rock (95-37) and two daughter rocks: and G43 corresponds to a secondary muscovite-bearing daughter rock (see Fig. 2) and G42 to the sample with the highest degree of alteration (see Fig. 2). The data show that hydrothermal alteration is responsible for the leaching of Be associated with the Fe-Mn oxyhydroxides. In contrast, sample G43 suggests that the precipitation of secondary muscovite induces a transfer of Be to the adsorbed phase (see text).

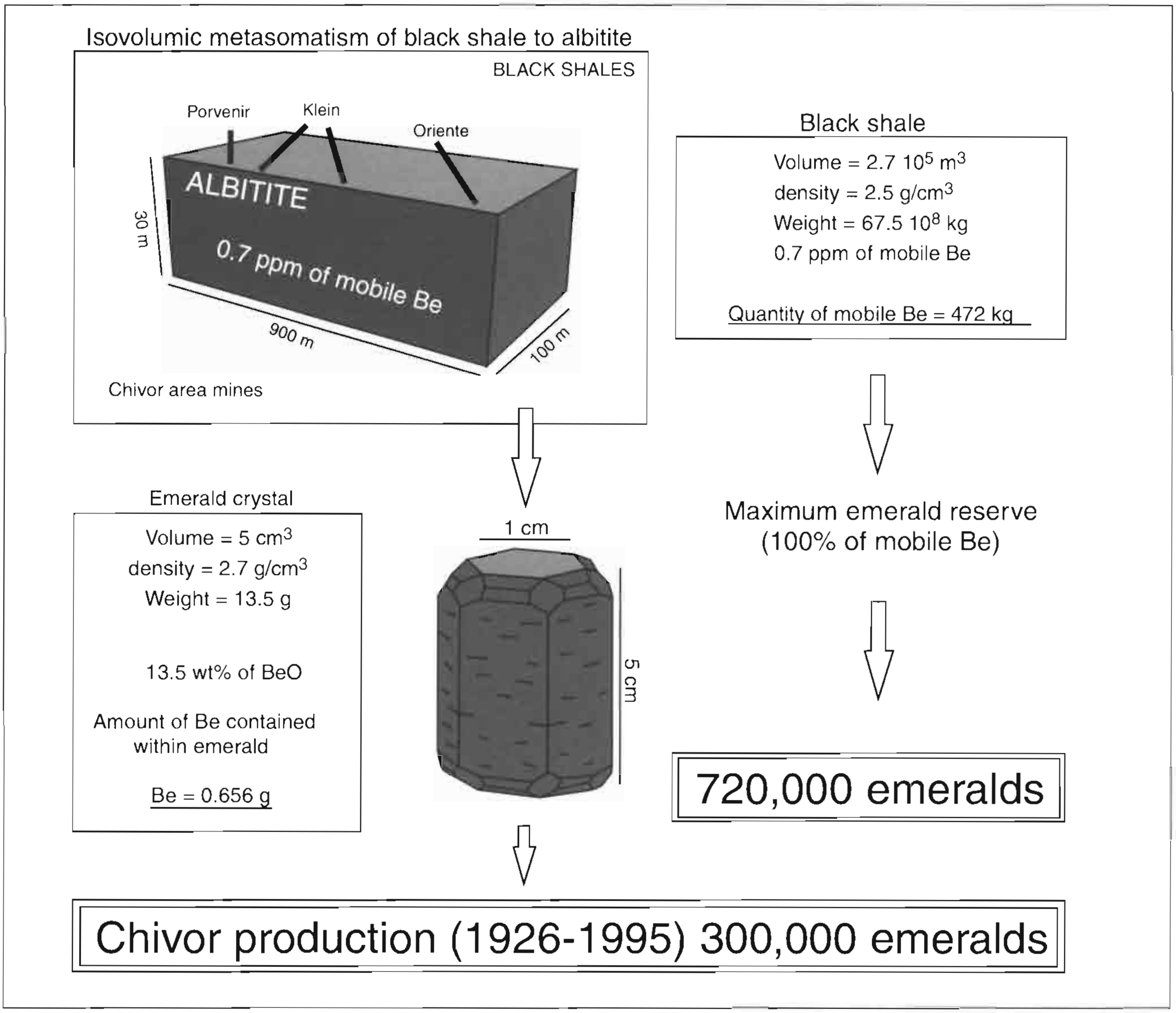

Fig. 4. Mass balance calculation of the potential amount of emerald crystals precipitated in the Chivor mine, based on an estimation of the amount of beryllium which may have been hydrothermally mobilized from the host black shale.

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