1	Neoproterozoic Re-Os systematics of organic-rich rocks in
2	the São Francisco Basin, Brazil and implications for
3	hydrocarbon exploration
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16	
17	Abstract
18	The São Francisco Basin contains a remarkable archive of Neoproterozoic strata and
19	its hydrocarbon-bearing strata are receiving increasing attention as global oil and gas
20	exploration targets progressively deeper and older rocks. New Re-Os geochronology
21	for the Paracatu Slate Formation of the Canastra Group, Brazil yields a depositional
22	age of 1002 ± 45 Ma. This age represents the first successful application of the Re–Os
23	system to rocks of this group and indicates excellent agreement with a previously

24 published U-Pb detrital zircon age (Rodrigues et al., 2010). Together with TOC 25 values of ca. 2 wt.% (despite greenschist metamorphism), it might be argued that the 26 São Francisco Basin has had the potential for hydrocarbon generation since the 27 Tonian (1000 – 850 Ma). In addition, we also report an imprecise Re–Os age (1304 \pm 28 210 Ma) for the Serra do Garrote Formation, a further potential source rock of the 29 Vazante Group. We suggest, based on petrological evidence, that the Re-Os 30 systematics were disturbed by post-depositional fluid flow that was most likely 31 associated with Vazante ore deposit mineralization. An attempt to determine a Re-Os 32 date for the Sete Lagoas Formation, a putative post-Sturtian cap carbonate, is 33 precluded owing to low Re abundances (<100 ppt). Major environmental changes in 34 the aftermath of the Jequitaí glaciation, particularly the development of 35 palaeotopography such as subglacial tunnel valleys, may account for the apparent 36 random distribution of TOC enrichment in these Cryogenian/Ediacaran post-glacial 37 deposits. This scenario might thus have major implications for the hydrocarbon 38 prospectivity of this post-glacial succession.

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40 *Keywords:* Re–Os, Neoproterozoic, Canastra, Vazante, Bambuí, source rock

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51 The São Francisco Basin and its surrounding belts contain an extensive stratigraphic 52 archive of Proterozoic time, extending at least from the Statherian (1750 Ma) to Late 53 Ediacaran (560 Ma) (Alkmim and Martins-Neto, 2012; Paula-Santos, 2013; Warren et 54 al., 2014). Over a wide area, diamictites and associated cap carbonates have been 55 correlated and linked to major Earth events, as the Sturtian (Babinski and 56 Kaufman, 2003; Azmy et al., 2006; Babinski et al., 2007; Vieira et al., 2007) and 57 Marinoan (Caxito et al., 2012) glacial epochs. In addition, the São Francisco Basin 58 has multiple hydrocarbon gas shows, which are probably sourced from Meso-59 Neoproterozoic organic-rich rocks (Craig et al., 2013 and references therein). 60 However, the lack of accurate geochronological data throughout the stratigraphy 61 hinders attempts to develop a chronological framework for the São Francisco Basin 62 and its surrounding belts. An understanding of the hydrocarbon potential of these 63 rocks and the relationship of organic-rich horizons to global geological events requires a precise geochronological framework. The absence of volcanic ash layers 64 65 for U-Pb and Ar-Ar geochronology has hindered attempts to gain absolute 66 geochronological age data (Misi et al., 2011). In addition, age control in these strata 67 is further hampered by the general lack of biostratigraphic constraints, intense 68 deformation and metamorphism. Placing accurate geochronological constraints on 69 organic-rich horizons is integral to understanding of the nature of the depositional 70 environment and fossil hydrocarbon system in this vast basin.

The rhenium–osmium (Re–Os) geochronometer is an increasingly recognized
tool for determining depositional ages of organic-rich rocks (Ravizza and Turekian,
1989; Cohen et al., 1999; Selby and Creaser, 2005a; Georgiev et al., 2011; van Acken

74 et al., 2013) and hydrocarbon deposits (Selby et al., 2005; Selby and Creaser, 2005b). 75 The method has yielded absolute dates for Neoproterozoic strata with precision 76 approaching 0.5 % uncertainty (2σ) in units up to greenschist facies (Kendall et al., 77 2004; Rooney et al., 2014). In the São Francisco Basin of Brazil (Fig. 1A) the Re-Os 78 radioisotope system has been previously utilized with limited success to provide 79 depositional ages for the Vazante Group (Geboy, 2006; Azmy et al., 2008; Geboy et 80 al., 2013). Here we evaluate and discuss Re-Os geochronology of the Canastra, 81 Vazante and Bambuí groups with the aim of constraining the depositional age of the 82 organic-rich strata of the Paracatu, Serra do Garrote and Sete Lagoas formations. 83 More widely, this study contributes to the radiometric calibration of the Proterozoic 84 rock record in Brazil, and hence to a better understanding of the geological evolution 85 of the Brasília Belt and São Francisco Basin.

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87 2. Geological setting and existing chronostratigraphy

88 The São Francisco Craton (Fig. 1A) is one of the oldest portions of the Precambrian 89 nucleus of South America (Almeida et al., 2000). Together with the other cratons of 90 South America, it represents the internal portions of plates involved in the assembly 91 of West Gondwana toward the end of the Proterozoic Era (Alkmim and Martins-Neto, 92 2012). The Brasiliano/Pan-African Orogenic Belts, meanwhile, fringe those ancient 93 plates and also include marginal metasediments accreted during collision (Almeida et 94 al., 2000; Alkmim et al., 2001; Alkmim and Martins-Neto, 2012). The Brasilia Belt, 95 which flanks the São Francisco Basin to the west, exhibits a fundamentally complex 96 tectonic character and variable metamorphic grade. Therefore, it is essential to briefly 97 outline the structural character, the stratigraphy, and present geochronology of both 98 the Brasília Belt and the São Francisco Basin.

101

102 2.1 The Brasilia Belt and São Francisco Basin

The Brasilia Belt, located on the western margin of the São Francisco Craton (**Fig. 104 1B**), is the product of a collision between the Amazon, São Francisco-Congo and Paranapanema paleocontinents (ca. 850-550 Ma; Pimentel et al., 1999) during the amalgamation of Gondwana (Li et al., 2008; Pimentel et al., 2011; Rodrigues et al., 2012). This belt is composed of thrust sheets verging eastward towards the São Francisco platform (**Fig. 1B**). Metamorphic grade increases progressively westward, reaching granulite facies conditions in the central part of the belt (Dardenne, 2000).

110 The southern Brasília Belt involves sedimentary rocks grouped into several 111 lithostratigraphic units (Fig. 1B): the Araxá, Paranoá, Canastra, and Ibiá groups (Pimentel et al., 2011). Intense deformation, the lack of intercalated volcanic 112 113 horizons, and the absence of biostratigraphic controls results in multiple possible 114 interpretations for this supracrustal succession (Dardenne, 2000; Valeriano et al., 115 2008; and references therein). Provenance studies suggest that the Paranoá and 116 Canastra groups are passive margin deposits of the São Francisco paleocontinent, while the Araxá, and Ibiá groups are synorogenic (fore- or back-arc) basin fill 117 118 (Pimentel et al., 2001; Rodrigues et al., 2010; Pimentel et al., 2011).

The São Francisco Basin occupies the ca. 800 km-long NS-trending lobe of the São Francisco Craton (**Fig. 1A**). Bounded to the west and to the east by emergent thrusts of the adjacent Brasília and Araçuaí Orogenic Belts respectively, the basin is filled by Palaeo-Mesoproterozoic units (Espinhaço Supergroup and Paranoá Group), and Meso-Neoproterozoic strata of the Vazante Group, Jequitaí Formation, and
Bambuí Group (Alkmim and Martins-Neto, 2012).

- 125
- 126 *2.2 Canastra Group*

127 The Canastra Group subdivides into three formations (Serra do Landim, Paracatu and 128 Serra dos Pilões). These rocks, which are mainly present in the southern portion of the 129 eastern Brasilia Orogen (Fig. 1B), comprise phyllite and quartzite with common 130 carbonate beds (Pereira et al., 1994; Dias, 2011). These have experienced lower 131 greenschist (2-3 kbar and 350-380 °C) facies metamorphism (Freitas-Silva, 1996; 132 Dardenne, 2000). Thrust contacts characterize the boundaries between the Canastra 133 and lower grade metamorphic strata of the Vazante, Paranoá and Bambuí groups 134 (Pereira et al., 1994). It has been suggested that the Canastra Group is a lateral 135 equivalent of the Paranoá Group (Dardenne, 2000; Pimentel et al., 2011).

136 The lithostratigraphy of the Canastra Group is difficult to unravel owing to numerous thrust faults (Rodrigues et al., 2010) (Fig. 2), especially for the basal Serra 137 138 do Landim Formation (chlorite-rich calc-phyllite and calcschist) and the upper units 139 (Paracatu and the Chapada dos Pilões formations). The Paracatu Formation, which 140 can reach up to ca. 2500 m in thickness (Dias, 2011) comprises slope turbidites and 141 basinal, carbonaceous phyllites rich in diagenetic pyrite, whereas the Chapada dos 142 Pilões Formation consists of shallow marine wave and storm-influenced clastics 143 (Pereira et al., 1994). The coarsening upward succession in the upper Canastra Group 144 thus records a regressive continental platform megasequence (Pereira et al., 1994).

For the Canastra Group, detrital zircon ages reveal Paleoproterozoic (ca. 1.8 and 2.1 Ga) and Mesoproterozoic (1.1-1.2 Ga) peaks, particularly for the Paracatu Formation (Rodrigues et al., 2010). The absence of Neoproterozoic zircon grains related to the active margin of the Brasília Belt, in addition to homogeneous
Paleoproterozoic Sm–Nd model ages (ca. 2.2 Ga) (Pimentel et al., 2001) suggests the
São Francisco-Congo Craton as the main source region. This led to the interpretation
of the Canastra Group as a passive margin succession (Pimentel et al., 2001, 2011).
The youngest detrital zircons in the Paracatu Formation are ca. 1040 Ma (Valeriano et
al., 2004; Rodrigues et al., 2010; Dias, 2011) (Fig. 2); the origin of the
Mesoproterozoic (ca. 1.2 Ga) population remains uncertain.

The diagenetic age of the ore-hosting carbonaceous phyllites of the Morro do
Ouro Member of the Paracatu Formation has been estimated at 1000-1300 Ma based
on Rb-Sr and K-Ar chlorite, and Pb-Pb galena ages (Freitas-Silva, 1996).
Metamorphism and gold enrichment of this unit is related to the Brasiliano Event at
ca. 680 Ma (Freitas-Silva, 1996).

160

161 "Insert Figure 2 here"

162

163 2.3 Vazante Group

164 The Vazante Group positioned in the western margin of the São Francisco Basin (Fig. 165 1B), comprises thick (ca. 5000 m) siliciclastic-dolomite (Fig. 3) of marine origin 166 (Azmy et al., 2008; Misi et al., 2011). The Vazante Group is divided into seven 167 formations (Santo Antonio do Bonito, Rocinha, Lagamar, Serra do Garrote, Serra do 168 Poco Verde, Morro do Calcário and Lapa; Dardenne, 2000), which experienced 169 greenschist facies metamorphism. Neoproterozoic Brasiliano thrusts and nappes 170 obscure many sedimentary contacts (Dardenne, 2000), particularly with the Canastra 171 Group to the west and the Bambuí Group to the east (Rodrigues et al., 2012). Intense 172 deformation in the outcrop area raises major uncertainties about the internal 173 stratigraphy and lateral correlation of the units.

174 In this paper, we analyzed the Serra do Garrote Formation (Fig. 3). This 175 formation, which can reach up to 1000 m in thickness (Misi et al., 2011), is 176 dominantly carbonaceous and pyrite-bearing slate, intercalated with fine quartzite 177 beds, representing an open marine succession deposited below storm wave base 178 (Madalosso, 1980). The Serra do Poco Verde Formation lies conformably over the 179 Serra do Garrote Formation and is dominantly dolomitic. The presence of glendonite 180 pseudomorphs after ikaite and dropstones in slates was interpreted as suggestive of 181 paraglacial depositional conditions (Olcott et al., 2005). The Morro do Calcário 182 Formation, a carbonate-stromatolitic succession, often diamictitic (Dardenne, 2000), 183 conformably overlies the former unit. The Morro do Calcário Formation is truncated 184 by an unconformity at the base of the overlying Lapa Formation (Misi et al., 2005), 185 which contains diamictites, organic-rich shale, and cap carbonates with a characteristic negative $\delta^{13}C_{carbonate}$ excursion (ca. -8 to 0 ‰) interpreted to record the 186 187 resumption of primary productivity in the aftermath of glaciation (Azmy et al., 2006; 188 2008). A reassessment of the core used by Azmy et al. (2006, 2008) concluded that 189 the unit is part of the Morro do Calcário Formation rather than the Lapa Formation as 190 previously thought (Geboy et al., 2013).

Based on C and Sr isotope profiles, the Morro do Calcário Formation is correlated with the Sturtian glacial epoch (Azmy et al., 2006; ca. 717 Ma; Macdonald et al., 2010). Globally, the chronometry of the Sturtian glaciation is considered to encompass a ca. 60 Myr window, based on U-Pb zircon and Re-Os geochronology of syn- and post-glacial deposits associated with the Rapitan glacial succession in NW Canada (Macdonald et al., 2010; Rooney et al., 2014). However, Re-Os analyses yield Mesoproterozoic depositional ages for organic-rich shales of the Serra do Garrote 198 $(1353 \pm 69 \text{ Ma})$ and Serra do Poço Verde $(1126 \pm 47 \text{ Ma})$ formations, respectively 199 (Fig. 3). The same technique together with U-Pb measurements on detrital zircons of 200 the Morro do Calcário Formation, suggest deposition at ca. 1000-1100 Ma (Azmy et 201 al., 2008; Fig. 3). As a result, the Vazante Group is considered late Mesoproterozoic, 202 rather than Sturtian (Azmy et al., 2008). Additional, SHRIMP U-Pb detrital zircon 203 analyses (Rodrigues et al., 2012) of five formations of the Vazante Group has 204 identified the youngest population ages (ca. 930 Ma) at the base of the group, and 205 older populations (ranging ca. 1200-1000 Ma) toward the top (Fig. 3). A reverse fault 206 identified between the Rocinha and Lagamar formations solve the paradoxical 207 stratigraphic age inversion (Geboy et al., 2013). The same authors also present Re-Os 208 ages for the Serra do Garrote and Morro do Calcário formations of 1354 ± 88 Ma and 209 1112 ± 50 Ma, respectively (Fig. 3).

210 Despite the complex history of the Vazante Group, the detrital zircon age pattern of the Serra do Garrote Formation (ca. 1.29 Ga, Rodrigues et al., 2012) is 211 212 coherent with Re-Os isochron ages obtained for the same formation (ca. 1.35 Ga, 213 Geboy, 2006; Geboy et al., 2013). However, high Mean Square of Weighted Deviates 214 (MSWD) values of 26 and 49 is associated with these Re-Os isochrons rendering 215 them imprecise and less than conclusive with regards to the true age of the Vazante 216 Group (Geboy, 2006; Geboy et al., 2013). Therefore, further provision of radiometric 217 ages is clearly necessary, and motivates our attempts to date the Serra do Garrote 218 Formation.

219

220 "Insert Figure 3 here"

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222 2.4 Neoproterozoic glacials and the Jequitaí Formation

223 Evidence for glaciation in the Jequitaí Formation in the São Francisco Basin and its 224 correlatives, the Bebedouro Formation (northern São Francisco Craton) and 225 Macaúbas Group (Araçuaí Belt), is compelling (e.g. Cukrov et al., 2005; Uhlein et al., 226 2007; Chaves et al., 2010). The preceding authors reported a striated pavement cut 227 into the Espinhaço Supergroup in the northeastern portion of the São Francisco Basin, 228 together with abundant diamictites with exotic lonestones, some of which are well 229 stratified and exhibit unequivocal impact structures implying ice-rafted debris. 230 Furthermore, on the western margin of the São Francisco Basin, Martins-Ferreira et 231 al. (2013; Fig. 1) describe a ca. 4 km-wide valley carved in sandstone of the Paranoá 232 Group and filled by a ca. 40 m package of sandstone, diamictite and tillite of the 233 Jequitaí Formation. These glaciogenic rocks are, in turn, covered by a cap carbonate 234 that marks the base of the Bambuí Group in the São Francisco Basin (Fig. 4). With 235 the exception of the striated pavement, each of these facies are recognised in 236 proprietary cores across the subsurface of the basin. Zircons U-Pb systematics 237 extracted from the Jequitaí Formation and the correlative Macaúbas Group yield 238 maximum deposition ages of 880 Ma and 864 Ma, respectively (Pedrosa-Soares et al., 239 2000; Rodrigues et al., 2008).

240

241 2.5 Bambuí Group

These epicontinental deposits, of alternating siliciclastics and carbonates are the most widely distributed unit in the São Francisco Basin (**Fig. 1**), draping the Jequitaí diamictites and sandstones. They form a shallowing upwards sequence (Dardenne, 2000; Santos et al., 2000), divisible into three coarsening upward megacycles (Dardenne, 2000; Martins-Neto, 2009). The first megacycle is represented by the Sete Lagoas Formation, the second includes the Serra de Santa Helena and Lagoa do Jacaré formations and the last cycle comprises the Serra da Saudade and Três Marias formations (Martins and Lemos, 2007) (**Fig. 4**). The absence of volcanic ash horizons throughout the Bambuí Group, in addition to hampering geochronology, has promoted debate regarding the tectonic setting for this group (e.g. Alkmim and Martins-Neto, 2001; Zalán and Silva, 2007) and its relationship with the Jequitaí diamictites (Babinski et al., 2007, 2012; Misi et al., 2011; Caxito et al., 2012).

254 The Sete Lagoas Formation, for which we present data in this paper, comprises a ca. 200 m succession (Vieira et al., 2007) of siliciclastic-calcareous 255 256 sediments, grading upwards into microcrystalline limestones and dolostones. Its upper 257 section contains the most extensive shallow water carbonates of the basin with 258 laminated and columnar Gymnosolenide stromatolites (Dardenne, 1978) and evidence 259 for subaerial exposure (Martins and Lemos 2007). Its basal contact is characterized by 260 an unconformity: the formation rests on granite-gneiss basement, on the glaciogenic 261 Jequitaí Formation, or on conglomerates of the Carrancas Formation, exposed along 262 the southern border of the basin (Dardenne, 2000, Alkmin and Martins-Neto, 2001, 263 Vieira et al., 2007).

Several isotopic studies have demonstrated a negative excursion of $\delta^{13}C_{carbonate}$ 264 265 (ca. -4 to 0 ‰) for the base of the Sete Lagoas Formation (e.g. Alvarenga et al., 2007; Kuchenbecker, 2011). The δ^{13} C signature, together with its stratigraphic position, 266 267 sitting on top of the Jequitaí diamictite deposits, has led to interpretations of a typical 268 postglacial cap carbonate sequence related either to Sturtian (Babinski and Kaufman, 2003; Babinski et al., 2007; Vieira et al., 2007) or Marinoan (Caxito et al., 2012) 269 270 deglaciation. Despite a large suite of isotopic and chemostratigraphic data available 271 the depositional age has been unknown until recently. A Pb-Pb age of 740 ± 22 Ma 272 (Fig. 4) from basal carbonates of the Sete Lagoas Formation (Babinski et al., 2007) was the first estimate for its depositional age. Subsequently, U-Pb detrital zircons in siliciclastics intervals provided maximum depositional ages of 610 Ma (Rodrigues, 2008) and 557 Ma (Paula-Santos, 2013) (Fig. 4). An Ediacaran fossil assemblage containing *Cloudina sp.* has recently been discovered in the central-eastern part of the basin (from the middle part of the Sete Lagoas Formation) (Fig. 4): this suggests a narrow time window (ca. 550-542 Ma) on account of the known global biozone that this assemblage represents (Warren et al., 2014 and references therein).

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From the above, it is clear that the depositional age of the Sete Lagoas Formation spans ca. 200 Myr. Thus, the different ages may imply that this formation contains a substantial hiatus within it. On the other hand, identical ⁸⁷Sr/⁸⁶Sr values (0.7074-0.7076) obtained both below and above the supposed unconformity separating the Cryogenian from the Ediacaran carbonates, argue against this hypothesis (Caxito et al., 2012).

In view of the uncertainties described earlier, and the necessity of understanding the context of deposition of the Bambuí Group, further provision of radiometric ages are required. In addition, most of the geochronological determinations derive from outcrops along the eastern margin of the basin (Babinski et al., 2007; Rodrigues, 2008; Paula-Santos, 2013). Thus, access to core material from the southwestern margin in the present study will extend knowledge to other parts of the carbonate platform.

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297

3. Methods

299 *3.1 Sampling*

300 Samples of the 3 formations in this study were collected from proprietary drill cores 301 (Fig. 1). Drill cores intersecting the Paracatu and Serra do Garrote formations were 302 provided by Votorantim Mine and a mining company from the Arcos region supplied 303 the Sete Lagoas Formation samples. In the MASW03 (Paracatu Formation) core (Fig. 304 **5A**) the sampled interval spans 47.10 to 55.70 m (MD) and include dark grey to black 305 slates, with sporadic quartz as thin veins together with pyrite. VZCF001 (Serra do 306 Garrote Formation) core samples (Fig. 5B) extend from 280.10 to 292.65 m and 307 include black slates, with considerable carbonaceous material (staining). Pyrite is 308 present, both as lamina-parallel mineralization, and as crosscutting veins and 309 framboid nodules. Finally, LMR1009 (Sete Lagoas Formation) core samples (Fig. 310 5C) were obtained from four intervals; 1, from 36-47 m (microbial dolomite and 311 mudstones); 2, from 111-118 m (laminated limestones with carbonaceous seams); 3, 312 from 144-157 m (clay-rich limestones); 4, from 158-165 m (argillites). Following 313 Kendall et al. (2009a), ca. 100g samples were collected at 1 m intervals in each core. 314 Sub-sampling at further 0.4 m intervals was undertaken to detect further changes in 315 Re and Os abundance and isotope composition.

We note that petrographic inspection of the Serra do Garrote Formation in core VZCF001 revealed faulting, brecciation of the host rock and pervasive quartz veining. Care was taken to avoid these zones of hydrothermal alteration and mineralization.

320

321 *3.2 Total organic carbon (TOC)*

322 TOC values for the all samples were determined at the School of Civil Engineering323 and Geoscience of Newcastle University, UK. An accurately weighed 0.1 g of

324 powdered rock was digested in hot (60-70 °C) hydrochloric acid (4 mol/L) to remove 325 the inorganic (carbonate) carbon. The decarbonated and washed samples (in deionised 326 water) were then dried overnight in an oven at 65 °C. The organic carbon in the 327 decarbonated samples was determined using a Leco CS230 Carbon-Sulphur analyser 328 (previously calibrated on standard samples; standard deviation 3 %).

329

330 *3.3 Re-Os geochronology*

331 For Re-Os analysis, the core samples were polished using a diamond coated polishing 332 pad to eliminate any metal contamination (e.g. cutting and drilling marks). Each 333 sample was dried at 60 °C for 24 h and then crushed to a powder (c. 30 µm) in a 334 zirconium dish using an automated shatterbox. Re and Os isotope analyses were 335 carried out at Durham University's TOTAL laboratory for source rock geochronology 336 and geochemistry at the Northern Centre for Isotopic and Elemental Tracing (NCIET) 337 using methods outlined in Selby and Creaser (2003) and Selby (2007). Between 0.2 338 and 0.4 g of each sample was digested and equilibrated in a borosilicate carius tube in 8 ml of Cr^{VI}–H₂SO₄ together with a mixed tracer (spike) solution of ¹⁹⁰Os and ¹⁸⁵Re at 339 220 °C for 48 h. The Cr^{VI}–H₂SO₄ solution was used to liberate hydrogenous Re and 340 341 Os, restricting the incorporation of non-hydrogenous Re and Os (Kendall et al., 2004). 342 Solvent extraction (CHCl₃) for Re and Os purification, micro-distillation and anion 343 chromatography methods were employed as outlined by Cumming et al. (2013). The 344 purified Re and Os fractions were loaded onto Ni and Pt filaments, respectively 345 (Selby, 2007), with the isotopic measurements determined by Negative Thermal 346 Ionization Mass Spectrometry using a ThermoScientific mass spectrometer via static 347 Faraday collection for Re and ion-counting using a secondary electron multiplier in 348 peak-hopping mode for Os. Total procedural blanks during this study were $14.6 \pm$

349 0.16 pg and 0.05 \pm 0.01 pg (1 σ S.D., n = 3) for Re and Os, respectively, with an average 187 Os/ 188 Os value of 0.61 ± 0.03 (n = 3). Uncertainties for 187 Re/ 188 Os and 350 ¹⁸⁷Os/¹⁸⁸Os were determined by error propagation of uncertainties in Re and Os mass 351 spectrometer measurements, blank abundances and isotopic compositions, spike 352 353 calibrations and reproducibility of standard Re and Os isotopic values. The Re-Os isotopic data including the 2σ calculated uncertainties for ${}^{187}\text{Re}/{}^{188}\text{Os}$ and ${}^{187}\text{Os}/{}^{188}\text{Os}$ 354 355 and the associated error correlation function (rho) were regressed to yield a Re-Os date using Isoplot V. 4.0 and the λ^{187} Re constant of 1.666 \times 10⁻¹¹ a⁻¹ (Smoliar et al., 356 1996; Ludwig, 2003). The age uncertainty including the uncertainty of 0.35 % in the 357 ¹⁸⁷Re decay constant only affects the third decimal place (Smoliar et al., 1996; Selby, 358 359 2007).

To evaluate mass spectrometry reproducibility, two in-house Re and Os 360 361 (Durham Romil Osmium Standard=DROsS) solution standards were analyzed. The Re solution standard yields an average ${}^{185}\text{Re}/{}^{187}\text{Re}$ ratio of 0.598071 ± 0.001510 (1 362 363 S.D., n = 67), which is in agreement with the value reported for the AB-1 standard (Rooney et al., 2010). The measured difference in ¹⁸⁵Re/¹⁸⁷Re values for the Re 364 standard solution and the accepted 185 Re/ 187 Re value (0.5974; Gramlich et al., 1973) is 365 used to correct the measured sample Re isotope composition. The Os isotope 366 reference solution (DROsS) gave an 187 Os/ 188 Os ratio of 0.160892 ± 0.000559 (1 S.D., 367 n = 67), which is in agreement with previous studies (Rooney et al., 2010). 368

369

370 **4. Results**

371 *4.1 TOC*

The TOC results for all samples are presented in **Table 1** and **Fig. 5A**, **B** and **C**. The Sete Lagoas Formation has the lowest TOC of the 3 analyzed cores (<0.01 to 0.49)

374 wt%), while the Serra do Garrote and Paracatu formations possess the highest TOC 375 values (0.75 to 2.12 wt % and 0.07 to 2.15 wt %, respectively). According to these 376 samples, the basin possesses fair quality as a potential hydrocarbon source rock, both 377 in carbonates and shales (c.f. Craig et al., 2013). Re-Os geochronology has been applied successfully to rocks with < 0.5 % TOC (Kendall et al., 2004), thus this cut 378 379 off value was used to select the samples for Re-Os analysis. Only the samples from 380 the Paracatu and Serra do Garrote formations provided ≥ 1 wt% TOC (Fig. 5A and B), 381 in low-grade metamorphic rocks. Therefore, maturation analyses (Rock Eval) were 382 not performed.

383

384 "Insert Table 1 here"

385 "Insert Figure 5 here"

386

387 4.2 Paracatu Slate Formation: Re-Os data

388 The Paracatu Slate Formation samples have Re (0.3 - 4.1 ppb) and Os (53 - 297 ppt)389 abundances (Table 2) that are close to or less than that of average continental crustal 390 values of 1 ppb and 50 ppt, respectively (Esser and Turekian, 1993; Peucker-Ehrenbrink and Jahn, 2001; Hattori et al., 2003). The ¹⁸⁷Re/¹⁸⁸Os ratios display a 391 limited range from 24.2 to 79.6 and present-day ¹⁸⁷Os/¹⁸⁸Os ratios range from 0.667 to 392 393 1.593 (Table 2). Regression of the Re–Os isotope data yield a Re–Os age of $1002 \pm$ 45 Ma (2σ , n = 4, Model 1, MSWD = 1.2, initial ¹⁸⁷Os/¹⁸⁸Os = 0.25 ± 0.04; Fig. 6A). 394 This initial 187 Os/ 188 Os (hereafter Os_i) value is remarkably unradiogenic and will be 395 396 discussed further in the following section.

397

398 "Insert Table 2 here"

401 *4.3 Serra do Garrote Formation: Re-Os data*

402 The Serra do Garrote slates are enriched in Re (4 - 28 ppb) and Os (137 - 585 ppt; Table 2) and present a large spread in ¹⁸⁷Re/¹⁸⁸Os ratios (205.1 - 601.2) and 403 ¹⁸⁷Os/¹⁸⁸Os ratios (3.628 - 12.207) (Fig. 6B). Replicate analysis of one Serra do 404 405 Garrote sample (VZCF-6r) show good reproducibility in Re and Os abundances and ¹⁸⁷Re/¹⁸⁸Os and ¹⁸⁷Os/¹⁸⁸Os ratios. Contrary to the Paracatu Slate Formation, 406 407 regression of the isotopic composition data for the Serra do Garrote Formation yield 408 an imprecise, Model 3 age of 1304 ± 210 Ma (MSWD = 96), with a negative initial 409 Os isotope value of -1.0 ± 1.4 .

410

411 4.4 Sete Lagoas Formation: Re-Os data

The samples of the Sete Lagoas Formation have very low Re abundances <100 ppt, which are lower than estimated average (present-day) upper continental crust. These samples are not amenable to Re-Os geochronology as the low Re abundances would result in very large (>100 %) uncertainties in isotopic measurements using current analytical techniques.

417

418 **5. Discussion**

419 5.1 Paracatu Slate Formation

New Re–Os geochronology for the Paracatu Slate Formation yields a depositional age
of 1002 ± 45 Ma, which agrees, within uncertainty, with U–Pb geochronology data
(detrital zircons ca. 1040 Ma; Valeriano et al., 2004; Rodrigues et al., 2010; Dias,
2011). This relatively precise age represents the first successful application of the Re–

424 Os geochronometer in samples of the Canastra Group. The new Re–Os 425 geochronology data adds credence to previous studies that suggest that there is no 426 significant disturbance in the Re-Os systematics of carbonaceous organic-rich rocks 427 that have experienced anhydrous greenschist facies metamorphism (Kendall et al., 428 2004; Rooney et al., 2011).

429 The Os_i value for seawater at the time of deposition of the Paracatu Formation 430 (0.25 ± 0.04) is much less radiogenic than that of modern-day seawater (ca. 1.06; 431 Levasseur et al., 1998) indicating that the dominant input of Os to seawater was non-432 radiogenic. This Os_i value is consistent with a marine Os budget dominated by 433 extraterrestrial and ultramafic-mafic magmatic/hydrothermal inputs with minor 434 contribution of radiogenic Os from continental weathering sources. The calculated Os 435 isotope composition of continental crust at 1 Ga ranges from 0.5-1.0 thus indicating 436 that this source played a minor role in the oceanic budget during this time as has been previously postulated (Kendall et al., 2009a; Rooney et al., 2010; van Acken et al., 437 438 2013). The Paracatu Slate Formation Os_i value provides an additional data point to the 439 record for Precambrian seawater, evidencing that the change in global patterns of 440 oxidative weathering and Os influx was of little importance, at least until the earliest 441 Tonian (van Acken et al., 2013) although this remains contentious (Sperling et al., 442 2014).

Based on our Re-Os data, the Canastra Group was deposited at or around the Meso-Neoproterozoic boundary. This places the Canastra Group as a possible correlative of the Paranoá Group, considering the diagenetic xenotime U-Pb age of 1042 ± 22 Ma of the latter (Matteini et al., 2012). Moreover, the Re-Os age endorses tectonostratigraphic models of a passive margin sequence, deposited along the SW margin of the São Francisco-Congo paleocontinent (Pimentel et al., 2001, 2011;

Rodrigues et al., 2010). As the detrital zircons and depositional Re-Os isochron of the Canastra Group are similar, this requires the rapid exhumation of the Mesoproterozoic source region (main peak at ca. 1.2 Ga (Stenian); Rodrigues et al., 2010). On the other hand, the youngest zircon population of the Paranoá Group interpreted as a maximum depositional age (1540 Ma (Calymmian); Matteini et al., 2012) necessarily imposes source isolation later in the evolution of the passive margin, if both units are indeed chrono-correlatives.

456 Considering the amount of organic matter preserved even after maturation (ca. 2 wt %,), it is likely that the Paracatu Slate Formation of the Canastra Group 457 458 constituted an extensive hydrocarbon source rock. Despite no remaining potential for 459 further hydrocarbon generation, it is not implausible that between deposition (ca. 460 1000 Ma) and prior to the last tectono-metamorphic event recognised in the Brasília 461 Belt (ca. 600 Ma; Pimentel et al., 1999), the rock expelled hydrocarbons. However, 462 the presently available data is insufficient to determine the precise timing of 463 hydrocarbon generation/migration, as the intense deformation during the 464 Neoproterozoic Brasiliano Event and the posthumous erosion has obliterated true 465 stratigraphic thicknesses.

466

467 5.2 Serra do Garrote Formation

The Serra do Garrote Formation, which similarly to the Paracatu Formation has experienced regional Brasiliano metamorphism (Dardenne, 2000), shows a large scatter about the Re–Os regression line (Model 3, 1304 ± 210 , MSWD = 96) together with a negative initial Os isotope composition (-1.0 ± 1.4) suggestive of disturbances to the Re–Os systematics. This imprecise age may result from either depositional and/or post-depositional processes. The presence of detrital Os with variable

¹⁸⁷Os/¹⁸⁸Os values, may result in imprecise and geologically meaningless ages 474 (Kendall et al., 2004, 2009a), but is considered an unlikely cause because the Cr^{VI}-475 476 H₂SO₄ digestion technique preferentially liberates hydrogenous Os. Another feasible 477 cause of geological uncertainty for the Re-Os systematics may be variations in initial 478 Os isotope compositions during deposition (Selby and Creaser, 2003). In order to 479 avoid these heterogeneities, short stratigraphic sampling intervals (ca. 0.6 m) were 480 employed. The Os isotope composition of the Serra do Garrote Formation, however, 481 show variations that exceed those expected from temporal evolution in seawater 482 (unless sedimentation rates were anomalously low). Thus, these variations do not 483 fully account for the complex Re-Os systematics in the Serra do Garrote Formation.

484 Weathering and metamorphism are unlikely explanations for the scattered Re-485 Os isotope systematics because drill core provided access to material showing no 486 apparent evidence of surficial alteration. Additionally, metamorphic conditions of the 487 Serra do Garrote Formation related to the Brasiliano Orogeny did not exceed 488 greenschist facies (Dardenne, 2000; Misi et al., 2005, 2007). Petrological evidence 489 (coarse pyrite aggregates, quartz veinlets and pervasive faulting and fracturing) 490 suggests the Serra do Garrote Formation has been affected by hydrothermal fluid 491 flow. Although we avoided sampling material with abundant quartz veins, the scatter 492 in the Re–Os regressions for the Serra do Garrote Formation and the Os_i signature of 493 the samples is indicative of a hydrothermal alteration origin, implying that there might 494 have been some mobilization of Re and Os by fluid flow. Similar Re-Os behaviour 495 has been observed by Rooney et al. (2011) for the Leny Limestone and by Kendall et 496 al. (2009b) for the Wollogorang Formation. Although the Vazante Ore hypogene 497 deposit is located in the overlying Serra do Poço Verde Formation (Monteiro et al., 498 2006), we do not discount the possibility that the same mineralizing and oxidizing 499 fluids may have affected the Serra do Garrote Formation due to the proximity of well 500 VZCF001 to brecciated metadolomites and epigenetic willemitic ore bodies along the 501 Vazante Shear Zone (Fig. 1B). Therefore, it is possible that the high-temperature (> 502 250 °C), oxidizing and moderate saline (ca. 15 wt. % NaCl equiv.) brines that leached 503 base metals from the basement and ascended to finally interact with the host 504 dolostones of the Serra do Poco Verde (Monteiro et al., 2003; Misi et al., 2005) have 505 hydrated the Serra do Garrote slates, resulting in disturbance of the Re-Os 506 geochronometer. Similar alteration of Re-Os systematics by mineralizing fluid 507 circulation might also explain why previous attempts to date the Serra do Garrote and 508 Morro do Calcario formations with the Re-Os geochronometer (Geboy, 2006; Azmy 509 2008; Geboy et al., 2013) were unsuccessful.

510 It is likely that the extensive hydrothermal activity recorded in the Vazante 511 Group, and associated with the abundant Zn deposits (Monteiro et al., 2006) had 512 intrinsic relation with hydrocarbon generation, possibly sourced by the Serra do 513 Garrote Formation. Pyrobitumen has been observed within hydrothermal veins in the 514 carbonates of the overlying Morro do Calcário Formation (Rubo and Monteiro, 2010; 515 Tonietto, 2011) and hydrocarbon inclusions were described in sulfides of the Vazante 516 ore deposit (L. Monteiro pers. comm.). Future dating of these hydrocarbon products with the ¹⁸⁷Re-¹⁸⁷Os radioisotope system (e.g., Selby and Creaser, 2005b) could help 517 518 constraining the timing of emplacement, the source of migrated hydrocarbons and the 519 temporal relation of the mineralization and hydrocarbon accumulation.

520

521 5.3 Sete Lagoas Formation

522 The low Re and Os abundance in the carbonate of the Sete Lagoas could be 523 intrinsically associated with the low TOC observed for the unit, and/or be directly related to the depositional environment and the organic matter type (Colodner et al., 1993; Crusius and Thomson, 2000; Selby and Creaser, 2003; Cumming et al., 2012; Harris et al., 2013). Further, the observed low organic content can also be related to thermal maturation (Peters and Cassa, 1994) which may cause a loss of 30-50 % of the assumed original amount of TOC (Buchardt et al., 1986). Despite the intention of accounting for the effects of maturation using biomarker studies, the results proved inconclusive likely due to low volumes of organic matter analysed.

531 The recognition of palaeovalleys in outcrop (Martins-Ferreira et al., 2013), 532 and information of their infill with the Jequitaí Diamictites and post-glacial cap 533 Bambuí carbonate may have important implications for the distribution of organic-534 rich facies. If indeed the Jequitaí glaciation left a sculpted palaeotopography, then 535 factors linked to restricted/open circulation within/out palaeovalleys may explain oxic 536 versus anoxic conditions for organic preservation and associated Re and Os complexation. The lack of diamictites underlying carbonates of the Sete Lagoas 537 538 Formation in well LMR1009 (opposed to other cores of the region; F. Pimenta pers. 539 comm.; Kuchenbecker 2011; Kuchenbecker et al., 2011, 2013) could indicate a distal 540 position from paleodepressions, resulting in the low TOC values observed in this 541 particular location. This interpretation can only be tentative, however, because 542 seismic sections are not yet available in this region. Nevertheless, considering that 543 similar distributions are recognised in other Neoproterozoic post-glacial successions 544 (Bechstädt et al., 2009), and that organic-rich post-glacial facies in North Africa have 545 charged more than 50 major oil and gas fields (Lüning et al., 2000), the Sete Lagoas 546 Formation may yet have good source rock potential. Further studies are clearly 547 required to unravel the complex depositional history of this unit.

549 **6.** Conclusions

New Re–Os geochronology for the Paracatu Slate Formation yields a depositional age of 1002 ± 45 Ma and is in agreement, within uncertainty, of U–Pb detrital geochronology. This relatively precise age provides a more precise chronostratigraphic framework for understanding the tectonic evolution of the Canastra Group and the onset of sedimentation within the São Francisco Basin.

555 Disturbance of Re-Os systematics in the Serra do Garrote Formation is 556 evident by an imprecise and inaccurate age along with a negative value for the Os_i 557 value. These factors together with petrological evidence strongly suggest that the Re-558 Os system was disturbed in response to hydrothermal fluid flow, possibly associated 559 with the Vazante ore deposit mineralization events. The circulation of oxygenated 560 fluids through the Vazante Group is suggested to be the cause for disturbance to the 561 Re-Os geochronometer. Consequently, care is necessary when applying the Re-Os 562 deposition-age geochronometer to sedimentary rocks that have experienced tectonic 563 deformation and hydrothermal fluid flow.

The lack of Re enrichment in the base of the Sete Lagoas Formation could be explained by the distribution of the organically-rich facies which, similarly to the deglacial shales in North Africa (Lüning et al., 2000), was inherited from glacial topography.

568

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570

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952 Figure Captions

953

Fig. 1. Location and geology of the study area. (A) São Francisco Craton, São
Francisco Basin and surrounding belts (BFB=Brasilia Fold Belt); (B) Simplified
geological map of the Brasília Belt (after Dardenne, 2000).

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Fig. 2. Lithostratigraphic column of the Canastra Group (modified from Dardenne,
2000). Youngest concordant age interpreted as maximum depositional age:
⁽¹⁾Rodrigues et al. (2010); ⁽²⁾Dias, 2011; ⁽³⁾ Valeriano (2004). Average thickness from
Pereira et al. (1994).

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Fig. 3. Lithostratigraphic column of the Vazante Group (modified from Dardenne,
2000). Youngest concordant age interpreted as maximum depositional age:
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al. (2011).

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Fig. 4. Lithostratigraphic column of the Bambuí Group (modified from Dardenne,
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Fig. 5. Stratigraphic levels used for TOC and Re–Os measurements (A) Paracatu
Formation, drillhole MASW03; (B) Serra do Garrote Formation, drillcore VZCF001;

977 (C) Sete Lagoas Formation, drillcore LMR1009.

979	Fig. 6. Re–Os isochron diagram (A) Paracatu Formation organic-rich slates, drillhole
980	MASW03; (B) Serra do Garrote Formation organic-rich slates, drillhole VZCF001.
981	
982	
983	Tables
984	
985	Table 1: TOC content for the Canastra, Vazante and Bambuí groups.
986	
987	Table 2: Re-Os isotope data for the Paracatu and Serra do Garrote formations. *Rho is
988	the associated error correlation at 2σ (Ludwig, 1980). $^{\$}Os_{i}$ is the initial $^{187}Os/^{188}Os$
989	isotope ratio calculated at 1002 Ma for the Paracatu Formation and 1300 Ma for the
990	Serra do Garrote Formation. VZCF-6r is a repeat analysis and was not included in the
991	regression
992	

Sample	Re	±	Os	±	¹⁸⁷ Re/ ¹⁸⁸ Os	±	¹⁸⁷ Os/ ¹⁸⁸ Os	±	Rho [*]	Osi§
-	(ppb)									
Paracatú										
MASW03-36	0.30	0.001	64.8	1.2	24.2	0.3	0.667	0.038	0.705	0.260
MASW03-38	0.36	0.001	52.7	1.0	36.1	0.7	0.847	0.030	0.705	0.239
MASW03-40	4.11	0.013	296.5	2.1	79.6	0.8	1.593	0.040	0.656	0.253
MASW03-42	1.31	0.004	175.1	1.0	39.8	0.6	0.934	0.030	0.654	0.264
Serra do Garrote										
VZCF001-6	18.7	0.06	507.2	5.2	317.9	2.8	6.167	0.070	0.656	-0.793
VZCF001-6r	18.9	0.06	515.7	9.3	314.3	6.4	6.136	0.174	0.698	-0.746
VZCF001-11	9.2	0.03	260.2	2.5	269.9	2.4	4.654	0.053	0.657	-1.255
VZCF001-13	28.3	0.09	584.6	7.0	601.2	5.2	12.207	0.139	0.656	-0.956
VZCF001-3	4.0	0.01	136.8	2.2	205.1	4.2	3.628	0.103	0.699	-0.862

Formation	Sample	Depth	TOC
	I I I	[m]	[wt%]
	VZCF001-1	280.23	0.85
	VZCF001-2 VZCF001-2	281.33	1.53
	VZCF001-3	281.33	0.07
	VZCF001-4	281.78	0.87
	VZCF001-5	282.23	2.10
C I .	VZCF001-7	285.78	0.72
Serra do	VZCF001-8	286.63	0.20
Garrote	VZCF001-9	287.78	0.93
	VZCF001-10	288.43	1.48
	VZCF001-11	288.98	0.62
	VZCF001-12	289.15	1.98
	VZCF001-13	289.60	1.50
	VZCF001-14	289.98	1.38
	VZCF001-15	292.55	1.89
	MASW03-33	47.6	2.12
	MASW03-34	48.0	1./6
	MASW03-33	40.3 18 6	1./3
	MASW03-30 MASW03-37	40.0 10 0	1.30
	MASW03-38	49.9 50.4	1.40
	MASW03-39	50.7	1.03
Paracatú	MASW03-40	51.0	0.92
1 ur uvuvu	MASW03-41	52.3	1.23
	MASW03-42	52.6	1.19
	MASW03-43	52.9	1.16
	MASW03-44	53.2	0.99
	MASW03-45	53.5	1.59
	MASW03-46	55.6	0.75
	MASW03-47	55.9	1.13
	LIMR1009-U4S15	35.85	0.08
	LIMR1009-U4S14	36.85	0.17
	LIMR1009-04813	37.85	0.03
	LIMR1009-04512	30.85	0.03
	LIMR1009-04511	40.85	0.02
	LIMR1009-U4S9	41.85	0.07
	LIMR1009-U4S8	42.85	0.02
	LIMR1009-U4S7	43.85	0.10
	LIMR1009-U4S6	44.85	0.03
	LIMR1009-U4S5	45.85	0.04
	LIMR1009-U4S4	46.25	0.02
	LIMR1009-U4S3	46.65	0.01
	LIMR1009-U4S2	47.05	0.02
	LIMR1009-U4S1	47.57	0.01
Sata Lagoos	LIMR1009-U3S8	112.34	0.01
Sele Lagoas	LIMR1009-U3S7	113.34	0.01
	LIMR1009-U3S6	114.34	0.02
	LIMR1009-U3S5	115.34	0.08
	LIMR1009-U3S4	116.34	0.01
	LIMR1009-U3S3	116.74	0.04
	LIMR1009-U3S2	117.14	0.03
	LIMR1009-U3S1	117.54	0.02
	LIMR1009-U2S15	145.75	0.00
	LIMR1009-U2S14	146.75	0.00
	LIMR1009-U2S13	147.75	0.00
	LIMR1009-U2S12	148.75	0.02
	LIMR1009-U2S11	149.75	0.00
	LIMR1009-U2S10	150.75	0.02
	LIMR1009-U2S9	151.75	0.08
	LIMR1009-U2S8	152.75	0.00

LIMR1009-U2S7	153.75	0.01
LIMR1009-U2S6	154.75	0.01
LIMR1009-U2S5	155.75	0.00
LIMR1009-U2S4	156.15	0.01
LIMR1009-U2S3	156.55	0.06
LIMR1009-U2S2	156.95	0.02
LIMR1009-U2S1	157.35	0.13
LIMR1009-U1S1	158.15	0.32
LIMR1009-U1S2	158.55	0.22
LIMR1009-U1S3	158.95	0.24
LIMR1009-U1S4	159.35	0.24
LIMR1009-U1S5	159.75	0.22
LIMR1009-U1S6	160.75	0.06
LIMR1009-U1S7	161.75	0.36
LIMR1009-U1S8	162.75	0.14
LIMR1009-U1S9	163.75	0.49
LIMR1009-U1S10	164.75	0.33





Group	Formation		Lithology	Youngest concordant age (Ma)	Depositional age (Ma)
	Lapa		Carbonaceous phyllite, carbonatic metasiltstone quartzites, conglomerate and slate	1084±14 ⁽¹⁾	
	Morro do Calcário		Dolomitic biostromes and bioherms, breccia, dolorudite, oolitic dolarenite and oncolits	1137±8 ⁽¹⁾	993±46 ⁽³⁾ 1100±77 ⁽³⁾ 1112±50 ⁽⁴⁾
	Serra do Poço Verde		Limestones with stromatolitic mats and mud crack		
			Slate with intercalations of dolomite		1126±47 ⁽²⁾
		1 2 0	Dolomite with stromatolitic mats and bird's eyes		
			Dolomite with layers of breccias and doloarenite		
VAZANTE (~5000 m thick)	Serra do Garrote		Carbonaceous pyrite-bearing slate with rare fine quartzite intercalations	1296±13 ⁽¹⁾	1353±69 ⁽²⁾ 1354±88 ⁽⁴⁾
	Lagamar		Stromatolitic bioherma interdigitated with carbonate-bearing metasiltstone and slate. Intraformational dolomitic breccia. Conglomerate, quartzite, metasiltstone and slate		
	Rocinha	/	Phophoarenite rich in intraclasts and pellet		
			Slate, with pyrite and phosphorite Rhythmic package of slate and metasiltstone	935±14 ⁽¹⁾	
	Santo Antonio do Bonito/Retiro	A - 7A	Quartzite, intercalated with slate. Diamictite	997±29 ⁽¹⁾	



