Multiproxy provenance analysis of Lower to Upper Cretaceous synorogenic deposits in the Southern Andes (34-35°S): evidence of coeval volcanism during the onset of the Andean orogeny

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## **Credit Author Statement**

**Ricardo Gómez:** designed the research, provided lithological column information, collected the rock samples and wrote the paper with input from all authors.

Antonella Galetto: contributed to the discussion of fission track analysis.

Guadalupe Arzadún: performed fission track analysis.

Maisa Tunik: collected the rock samples and provided lithological column information.

Silvio Casadio: collected the rock samples and provided lithological column information.

Martin Parada: Prepared samples and photographs from SEM.

Lucas Lothari: collected the sediment samples and provided lithological column information

All authors discussed the results and commented on the final manuscript.

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20	Abstract
21	The combination of detrital low-temperature thermochronology with previous U-Pb
22	geochronology, petrological and sedimentological analyses, has proven to be a valuable
23	approach to constrain the provenance of non-marine Lower to Upper Cretaceous
24	synorogenic deposits in the northern Neuquén Basin. This work focuses on the study of
25	the Diamante Formation, a fluvial succession that represents the first synorogenic
26	products of the Andean foreland basin at 34-35°S. The results indicate that the deposition
27	of the Diamante Formation occurred simultaneously with the existence of an active
28	western volcanic arc during the onset of the foreland basin. The facies associations

evidence the transition between the backarc and the foreland basin stages as well as the 29 30 inception of fluvial sedimentation in the foredeep. Petrographic analyses, together with changes in the paleocurrents and the record of limestone clasts suggest a regional detrital 31 source shift. Apatite fission-track analyses (AFT) of a sample collected from the lower 32 part of the Diamante Formation indicate an Albian central cooling age. This sample also 33 evidences a remarkable presence of angular apatite and zircon crystals with subordinated 34 35 rounded and subangular grains. Zircon fission-track analyses (ZFT) of a sample from the upper part of the Diamante Formation yield two discrete populations of cooling ages, both 36 reflecting source-cooling during the Late Jurassic (~161 Ma) and the Permian (~265 Ma). 37 38 Finally, a comparison between the AFT and the U-Pb maximum depositional zircon-age reveals a short lag time (ca. 3 ma), likely related to the rapid magmatic cooling of a coeval 39 volcanic source at ~110 Ma (Albian). 40

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42 Keywords: Foreland Basin; Fluvial succession; Diamante Formation; latest Early
43 Cretaceous; Fission Track; U-Pb geochronology

44

## 45 **1. Introduction**

46 A combination of multiple techniques has been a useful tool to perform provenance studies in the last decade. The analysis of detrital crystals with particular 47 chemical and isotopic signatures reveals associations with source areas and tectonic 48 events in many geological scenarios (Carrapa et al., 2009; Carrapa, 2010; Cawood et al., 49 2012; Chew and Donelick, 2012; Peyton and Carrapa, 2013; Gehrels, 2014; Owusu 50 Agyemang et al., 2019). The changes of the allocyclic and autocyclic processes that 51 influence the sedimentation over time can generate sediments of variable compositions 52 derived from a similar source or, conversely, very similar sedimentary rock derived from 53

different sources. The recognition of different compositional patterns in the detrital 54 55 components of sedimentary sequences allows for the identification of diverse source signals (Cecil, 2003; Tyrrell et al., 2012; Franklin et al., 2019). Additionally, combined 56 geochronometry and low-temperature thermochronometry enhance the recognition of 57 syntectonic provenance information, by providing crystallization and thermotectonic 58 histories of the same source rocks feeding the basin (Malusá and Fitzgerald, 2019 and 59 60 references therein). Multiproxy analysis, including multidating methods, is a powerful tool for the reconstruction of the tectonic history of foreland basin systems (Bernet and 61 Spiegel, 2004; Umazano et al., 2009; Ghiglione et al., 2015; Suriano et al., 2017; 62 63 Thomson et al., 2017; Buelow et al., 2018; Bernet, 2019 and references therein).

64 The Neuquén Basin (Fig. 1) is a large depocenter developed during Late Triassic to Paleogene times in the southwestern margin of Gondwana (30-40°S) created by 65 66 continental-scale rifting processes in response to the break-up of the Pangea supercontinent (Mpodozis and Ramos, 2008; Charrier et al., 2015; D'Elia et al., 2020; 67 among others), with the potential influence of upper-plate movement and basement 68 fabrics interaction (Fennell et al., 2020 and references therein). It records a thick 69 70 Mesozoic sedimentary sequence of more than 7.000 m including marine and non-marine 71 sedimentary rocks. Between the Early to Late Cretaceous, the Neuquén Basin changed 72 from a backarc extensional basin to a retroarc foreland basin, in response to a westward acceleration of the South American plate during the Cretaceous (Mpodozis and Ramos, 73 74 1990; Howell et al., 2005; Ramos and Kay, 2006; Tunik et al., 2010). Congruent with this tectonic setting, several large-scale drainage systems have been interpreted for the Upper 75 76 Cretaceous non-marine deposits of the Neuquén Basin, based on stratigraphy and sedimentology (e.g., Di Giulio et al., 2012, 2017; Gómez et al., 2019, 2020). Provenance 77 analyses in the northern part of the basin (34-35°S) suggest that the foreland basin began 78

to form at approximately 100-107 Ma with the deposition of the Diamante Formation, 79 80 which is temporally equivalent to the youngest formation of the Neuquén Group described south of 35°S (Fig. 1; Gómez et al., 2019, 2020). A proposed source rock-model 81 includes a westward sediment dispersion derived from the Sierra Pintada System and the 82 83 San Rafael Block before the uplift of the Andes, which was then shifted eastwards with the onset of the Andean orogeny, associated to a new west-derived source (Tunik et al., 84 2010; Di Giulio et al., 2012, 2017; Balgord and Carrapa, 2016; Balgord, 2017; Fennell et 85 al., 2017a, Borghi et al., 2019; Gómez et al., 2019, 2020). However, the lack of detailed 86 sedimentological and provenance studies north of 35°S reflects the need for further 87 88 studies to better understand the tectonic evolution during latest Early to Late Cretaceous 89 times.

In the last few years, several discrepancies arose regarding the presence of a 90 91 coeval volcanic arc during the Upper Cretaceous along the western margin of the Neuquén Basin (e.g., Muñoz et al., 2018; Gómez et al., 2019, 2020). Some authors 92 93 speculated on a decreasing volcanic activity because of the absence of detrital zircons <100 Ma in the Neuquén Group. Moreover, an eastward arc migration is proposed in 94 95 response to a shallowing of the subducted oceanic slab at ~35°S (Fennell et al., 2017a; 96 Muñoz et al., 2018). This flat-slab stage produced an increase in the contractional 97 deformation and the generation of a first order unconformity known as the Patagonidic (Ramos, 1988; Leanza, 2009; Tunik et al., 2010; Fennell et al., 2017b; Asurmendi et al., 98 99 2017; among others). For this paleogeographic scenario, Muñoz et al. (2018) documented 100 differences in provenance patterns between the western and eastern Lower Cretaceous 101 synorogenic deposits of the Neuquén Basin (~35°S). Based on this finding, Muñoz et al. (2018) suggested the presence of a topographic barrier separating the eastern and western 102 domains, associated with the growth of the Andean fold-and-thrust belt during the Late 103

104 Cretaceous. More recently, Tapia et al. (2020) correlated this compressive stage with 105 maximum exhumation rates estimated for the western Paleozoic basement of the Coastal Cordillera, accompanied by the development of a topographic barrier that inhibited the 106 107 sediment supply derived from the contemporaneous volcanic arc to the eastern foreland basin. However, Gómez et al. (2019) found evidence of the influence of a volcanic arc 108 during the Lower to Upper Cretaceous (Albian-Campanian) non-marine sedimentation in 109 110 the foreland basin, and pyroclastic components associated with fluvial deposits were recognized within the Neuquén Group deposits (Corbella et al., 2004; Garrido, 2010; 111 Sánchez et al., 2008, 2013; Asurmendi et al., 2017). Furthermore, Gómez et al. (2020) 112 113 observed reworked tuff levels with ca. 0.60 m of thickness at 34-35°S in the Diamante Formation, which is interpreted as a direct evidence of volcanic activity during the 114 foreland basin deposition stage. Moreover, the U-Pb detrital zircon ages as well as a 115 116 petrographic analyses of the Neuquén Group and the Diamante Formation, reveal indirect evidence of the presence of an active volcanic arc after the Aptian (Tunik et al., 2010; 117 Borghi et al., 2019; Gómez et al., 2019; among others). 118

The goal of this paper is to constrain the sediment provenance of the Albian to Campanian synorogenic succession in the northern Neuquén Basin and to reconstruct its evolution. With this aim, a multiproxy approach was applied with the integration of new sedimentological, petrographic and low-temperature thermochronological data with previous geochronological results obtained by Gómez et al. (2019).

124 **2.** Tectonic setting

Three different kinematic regimes have been documented in the Andes: 1) a backarc extension as a result of a slab rollback rate exceeding the 'absolute' velocity (normal component) of the overriding plate; 2) dominant strike-slip kinematics with a local transtension to transpression during periods of oblique convergence; and 3) a

contractional deformation caused by the 'absolute' velocity (normal component) of the
overriding plate exceeding the rate of the slab rollback (e.g., Schellart, 2008; Ramos,
2010; Balgord, 2016).

The well-preserved sedimentary strata of the Neuquén Basin (30°-40°S) records 132 a complex tectonic history with spatial and temporal variations. According to Howell et 133 al. (2005), the tectonic evolution and its sedimentary infill can be grouped into three main 134 135 stages: (1) the tectonic extension phase during the Late Triassic–Early Jurassic period, manifested by isolated rift depocenters, each one showing a particular set of structural 136 and stratigraphic features (e.g., D'Elia et al., 2020; Bechis et al., 2020; and references 137 138 therein); (2) the Early Jurassic-middle Cretaceous marine and non-marine post rift basin 139 caused by a thermal subsidence stage (Uliana and Legarreta, 1993; Legarreta and Uliana, 140 1996; Schwarz et al., 2016); (3) the latest Early Cretaceous to Cenozoic retro-arc foreland 141 stage (Cobbold and Rossello, 2003; Ramos and Folguera, 2005; Mpodozis and Ramos, 2008; Tunik et al., 2010; Naipauer and Ramos, 2016; Horton, 2018). The basin was 142 143 bounded by three relatively tectonically stable areas: North Patagonian Massif to the 144 southeast and the San Rafael-Las Matras block to the north-east. To the west, the 145 boundary was conditioned by the evolution of the Andean arc.

146 Important geodynamic changes occurred during the latest Mesozoic with the 147 westward accelerated movement of the South American plate after its separation from the African plate, and the continuation of subduction processes along its western margin, with 148 149 the convergence between the Nazca-Farallón and the South American plates. This setting gave rise to compressive tectonics along the western margin of the South American plate, 150 151 thickening of the crust, the inversion and uplift of the Andean basins, and the continuation of the magmatic activity (Mpodozis and Ramos, 1990; Ramos and Kay, 2006; Somoza 152 and Zaffarana, 2008; Ramos, 2009). This interpretation has been modified by several 153

authors in the last few years suggesting that the onset of the contraction was triggered by
changes in a lower mantle convection (Faccenna et al., 2017; Schellart, 2017). Chen et al.
(2019) suggested that the subduction of the Farallón plate began at the northern Andes
(5°S) during the Late Cretaceous period (~ 80 Ma) and propagated southwards, reaching
40°S by the early Cenozoic period (~55 Ma), based on tomographic data. More recently,
Gianni et al. (2020) studied post-Gondwana synorogenic deposits and proposed that the
Andean uplift was a diachronic process that propagated northward.

The study area is located in the Cordillera Principal in the central-western sector of the Mendoza Province (~34°30'S, 69°40'W) (Fig. 2a-b). The main structural features of this area are associated with the Malargüe fold-and-thrust belt (MFTB), which is characterized by large basement structures coupled with thin-skinned thrust systems developed both on top of these basement structures and along the eastern thrust front (Kozlowski et al., 1993; Manceda and Figueroa, 1995; Giambiagi et al., 2008, 2009; Turienzo, 2010; Fuentes et al., 2016).

## 168 **3. Stratigraphic synthesis**

The non-marine Upper Cretaceous deposits recorded south of 35°S of the 169 170 Neuquén Basin are assigned to the Neuquén Group, where the sequence reaches a 171 maximum thickness of 1,600 m (Legarreta and Gulisano, 1989; Garrido, 2010; Orts et al., 2012). There, the synorogenic deposits of the Neuquén Group are subdivided into the Río 172 Limay, Río Neuquén and Río Colorado subgroups (Cazau and Uliana, 1973; Ramos, 173 1981). Cazau and Uliana (1973) pointed out that each subgroup is characterized by 174 upward fining and thinning sequences. However, there are still many problems regarding 175 176 the division and recognition of the formations. The Río Limay Subgroup represents the initial stage of the foreland basin deposition of the Neuquén Group, and it is composed 177 by the Candeleros, Huincul and Cerro Lisandro formations. The Río Neuquén Subgroup 178

includes the Portezuelo and Plottier formations, while the Río Colorado Subgroup is 179 180 composed of the Bajo de la Carpa and the Anacleto formations. Overall, the Neuquén 181 Group records important lateral and vertical facies changes integrating fluvial, aeolian, 182 lacustrine, deltaic, and estuaric deposits that are controlled by allocyclic and autocyclic processes. This configuration defines diverse local depocenters that integrate the entire 183 foreland basin (Cazau and Uliana, 1973; Leanza and Hugo, 2001; Leanza et al., 2004; 184 185 Garrido, 2010; Asurmendi et al., 2017; among others). Regionally, this cyclic pattern is interrupted by the overlying Maastrichtian to Palaeocene marine facies of the lower 186 Malargüe Group, which represents the first Atlantic-related marine succession on top of 187 188 the Neuquén Group deposits (Uliana and Dellapé, 1981; Barrio, 1990; Aguirre-Urreta et 189 al., 2008). The Neuquén Group overlies unconformably the Bajada del Agrio Group 190 (Barremian-Albian), which was originally defined by Méndez et al. (1995) as an 191 individual formation, and then formalized by Leanza (2003) including the Huitrín and Rayoso formations. The Huitrín Formation consists of alternating evaporitic and 192 193 marginal-marine deposits with an extensive geographical occurrence in the basin that predated the final disconnection of the paleo-Pacific Ocean with the Neuquén Basin. After 194 195 that, the sedimentation continued with the Rayoso Formation, which was accumulated in 196 a shallow perennial lake of variable salinity affected by long-lived hyperpycnal flows (Leanza, 2003; Zavala et al., 2006; Lazo et al., 2017). The unconformity that separates 197 the Bajada del Agrio Group and the Neuquén Group corresponds to a basin-scale angular 198 199 unconformity named the Patagonidic unconformity (cf. Fennell et al., 2017b). This 200 unconformity has been observed both in outcrops and in seismic data (Vergani et al., 201 1995; Mosquera, 2008). Nevertheless, in the study area, this angular unconformity was 202 not recognized. For this reason, and considering the facies analysis of this work, it is 203 assumed that the contact between the Rayoso and the Diamante formations is transitional

(see the discussion section for a detailed explanation). Balgord and Carrapa (2016)
proposed an erosion/non-deposition gap of 25 ma south of the study area, which includes
the entire Rayoso Formation, with the Huitrín Formation directly in contact with the nonmarine Late Cretaceous deposits. Similar features between both units were observed by
Balgord (2016) in the Aconcagua area (32-33°S).

North of 35°S, the reddish non-marine deposits overlying the Huitrín Formation 209 210 were included in the Diamante Formation initially defined by Groeber (1946). The age of 211 detrital zircons constitutes an important tool to demonstrate a correlation between the non-marine deposits cropping out in the Mendoza Province and the Neuquén Group, but 212 213 the lack of data at 34-35°S does not allow us to confirm this relationship until now. The Diamante Formation is ~300-1000 m thick in the Mendoza Province (Figs. 3-4), and 214 records fluvial (i.e., meandering and braided systems), alluvial fan and lacustrine 215 216 paleoenvironments (Cristallini and Ramos, 1996; Broens and Pereira, 2005; Balgord, 2016; Balgord and Carrapa, 2016; Mackaman-Lofland et al., 2019; Gómez et al., 2020; 217 Lothari et al., 2020). 218

The absence of radiometric ages for the scarce volcanic levels interbedded in the 219 220 Neuquén Group or the Diamante Formation makes it difficult to obtain an absolute age 221 for this unit. For that reason, the age of these units are still under debate. Based on paleomagnetic studies, Dingus et al. (2000) estimated an early Campanian age for the 222 Anacleto Formation (~83.5–79.5 Ma), while Corbella et al. (2004) obtained a zircon 223 224 fission track age of  $88 \pm 3.9$  Ma (Coniacian) for the base of the Huincul Formation of the Neuquén Group. Furthermore, several U-Pb dating on detrital zircons performed in 225 226 different localities of Neuquén Group and Diamante Formation, especially south of 35°S, indicate a maximum depositional age for this units between 107 and 97 Ma (Tunik et al., 227

228 2010; Di Giulio et al., 2012, 2017; Balgord and Carrapa, 2016; Fennell et al., 2017a;

Borghi et al., 2019; Gómez et al., 2019; Mackaman-Lofland et al., 2019).

In the Western Principal Cordillera of Chile, a well exposed continental Upper Cretaceous deposit named the BRCU series (Brownish Red Clastic Unit; Charrier et al., 1996) assigned to the latest Cenomanian-early Campanian by Muñoz et al. (2018), reveals sediment provenance from a coeval Cretaceous volcanic arc. According to these authors, the BRCU deposits have a chronological correlation with the Neuquén Group and the Diamante Formation in Argentina, but with differences in the provenance patterns of the U-Pb ages of their detrital zircons.

237

## 4. Multiproxy methodology

This paper contains results obtained through a combination of techniques to 238 239 determine the provenance of the Albian-Campanian non-marine deposits in the northern 240 part of the Neuquén Basin. Although several results from the application of this approach were published over the last decade, few papers include a detailed sedimentological 241 242 analysis in addition to the provenance studies (e.g., Surpless and Augsburger, 2009; Di Giulio et al., 2017; Suriano et al., 2017; among others), and none of them were from this 243 244 area. These analyses are the key not only to characterize the source regions, but also to 245 understand the processes and sedimentation patterns in the foreland basins.

## 246 **4.1. Stratigraphic sections, facies analysis and petrography**

To carry out a detailed sedimentological analysis, two areas were logged: (1) the Arroyo Oscuro area (34°36'15.45"S; 69°44'20.00"W) and, (2) the Arroyo Las Playas area (34°33'39.42"S; 69°44'44.91"W). Both areas were integrated with studies developed in the nearby areas of Vega Grande and Vega de Los Patos (Gómez et al., 2020; Lothari et al., 2020). All these areas are located between the Atuel and the Diamante rivers (Fig. 2bc). Thicknesses were determined using Jacob's staff. This procedure, in addition to the

rock descriptions with an emphasis on lithology (including texture and composition) and
on the sedimentary structures, allowed the characterization of the different sedimentary
facies. Field studies enabled the recognition of the geometry of the bedsets. The Arroyo
Oscuro area was the only area suitable for the analysis of the fluvial architecture, which
was then correlated with the other sections. The definition of facies and their associations
were used to discriminate the paleoenvironment of the Diamante Formation in the study
area.

Paleocurrent measurements were performed with a Brunton® compass, and were 260 corrected for magnetic declination in those places where exposure and outcrop-strata 261 262 orientations allowed accurate measurements. These measurements provided valuable information on paleocurrents in order to shed light on the paleotopography of the area 263 (e.g., Potter and Pettijohn, 1977; Buelow et al., 2018; Paredes et al., 2018). The 264 265 imbrication of pebbles and cross-stratification were used to obtain the paleocurrent directions. Nevertheless, three-dimensional exposures were necessary to obtain true dip 266 267 directions and to avoid erroneous current directions.

Measured stratigraphic areas provided the framework for a systematic collection 268 269 of sandstones samples. These samples were analysed under a magnifying glass for a 270 complete macroscopic description. Twenty-one standard 30 µm thin sections were impregnated with blue epoxy resin in order to highlight the porosity, stained with alizarin 271 red to distinguish the dolomite and the calcite, and stained with potassium ferricyanide to 272 273 distinguish the ferroan and the non-ferroan calcite following the method of Dickson (1965). After the petrographic analysis, 7 samples from the Arroyo Oscuro area and 5 274 275 samples from Arroyo Las Playas area were selected for the study of detrital modes and provenance analyses. The sandstones were classified following Folk et al. (1970) criteria, 276 and the Gazzi-Dickinson method was used for the provenance analyses based on a 400 277

clast count for each thin section (Ingersoll et al., 1984). All the surveyed data was
included in the discrimination provenance diagrams of Dickinson et al. (1983), along with
the samples previously obtained in the Vega Grande and the Vega de Los Patos locations
(Gómez et al., 2019).

**4.2. Detrital zircon U-Pb geochronology** 

In this study, we used the U-Pb detrital zircon ages previously published by 283 284 Gómez et al. (2019) from the lower part of Vega de Los Patos and the top of the Vega Grande area, with the aim of improving the multiproxy provenance analysis. These 285 samples consisted of a medium-grained sandstone and a reworked tuff, respectively 286 287 (VLP001 and VG24 samples, location in Figure 2c). U-Pb detrital zircon dating was conducted at the University of Arizona with a LA-ICP-MS equipment, following the 288 289 procedures outlined in Gehrels et al. (2006, 2008). For more details about laboratory and 290 analytical procedures see Gómez et al. (2019) and Appendix 4.

# 4.3. Low-temperature thermochronology: Apatite and Zircon Fission Track (AFTZFT)

The fission track method is based on the accumulation of narrow damage trails 293 called "fission tracks" in uranium-rich mineral grains (e.g., apatite, zircon) and natural 294 glasses, which form as a result of the spontaneous nuclear fission decay of <sup>238</sup>U in nature 295 (Price and Walker, 1963; Fleischer et al., 1975). Fission tracks start to be retained in the 296 crystal once the rock has cooled below the closure temperature (Tc) of  $240 \pm 20^{\circ}$ C for 297 ZFT and  $100 \pm 10^{\circ}$ C for AFT (Laslett et al., 1987; Brandon et al., 1998; Ketcham et al., 298 299 1999), and start to reduce their length within the thermal range of 200-300°C for ZFT (ZFT partial annealing zone, PAZ; Tagami, 2005) and 60-120°C for AFT (AFT partial 300 annealing zone, PAZ; Gleadow and Fitzgerald, 1987). When crystals are subjected to 301 302 temperatures within the PAZ for a long time, the tracks are erased or annealed by thermal

recovery, causing a reset of the isotopic system (Fleischer et al., 1975). Conversely, if the
crystals are maintained under the PAZ, then the fission tracks are preserved.

305 The fission-track cooling ages were performed at La.Te. Andes S.A. laboratory 306 (Salta, Argentina), and calculated for two samples collected from the bottom (AFT; VLP001) and the top (ZFT; 2119) of the Vegas de Los Patos stratigraphic section (Fig. 307 2c). The ages were obtained following the External Detector Method (EDM; Hurford and 308 309 Green, 1983) and using the TrackKey software (Dunkl, 2002) (See Appendix 3 for more details about the sample preparation and the analytical procedures). The Chi squared test 310  $-P(x^2)$ - was applied in each case in order to evaluate overdispersed data in relation to the 311 312 expectation of the statistical count for the radioactive decay process (Galbraith, 1981). For samples with  $P(x^2) < 5\%$ , the grain age distribution was decomposed by using the 313 binomial peak method (Galbraith and Green, 1990) through the Binomfit software 314 315 (Brandon, 2002) to identify discrete populations. A "lag time" was estimated by comparing the obtained FT cooling ages with the U-Pb detrital age of the same sample 316 317 from the Diamante Formation (Gómez et al., 2019). In sedimentary rocks, this parameter 318 represents the time a sample takes to cool below the PAZ temperatures in the source area, get transported and finally deposited (Garver et al., 1999). A rapid source cooling is 319 320 evidenced by short lag times, and can be associated with a rapid source-exhumation (Reiners and Brandon, 2006; Rahl et al., 2007), or with a magmatic-source when the lag 321 time is considerably short (Malusà et al., 2011; Malusà and Fitzgerald, 2019 and 322 323 references therein).

# 4.4. Electron Microscope Scanning: morphological analysis of apatite and zircon grains

326 Apatite fission-track and U-Pb detrital zircon dating of sample VLP001 was 327 complemented by a detailed morphological description of the apatite and zircon crystals

from the same sample, through the use of an Electron Microscope Scanning (EMS). The 328 329 main purpose of this approach was to distinguish populations of detrital grains with 330 potential equivalent provenances. With this objective, 530 detrital apatite grains and 508 detrital zircon grain were randomly picked under a binocular loupe, and dispersed on a 331 332 double-sided carbon tape placed on a carbon-coated aluminum stub. Each crystal was photographed with a ZEISS EVO MA15 scanning electron microscope (SEM). The 333 334 morphological analysis was performed at the Instituto de Investigación en Paleobiología y Geología, Universidad Nacional de Río Negro (General Roca, Argentina). The SEM 335 was executed in backscatter detector mode under a high vacuum with 20 kV and a 336 337 working distance of ~8.5 to 5 mm, where various magnifications (~700x to 1500x) were 338 used. The SEM was also equipped with an OXFORD X-Max 20 X-ray detector, which allowed the determination of the chemical composition of the analyses grains and verified 339 340 that they were apatite crystals. The morphology of each crystal was described as rounded, subangular and angular according to the standard classification of roundness (Cox, 1927). 341 342 Crystals with fractures that precluded the recognition of their morphology were not considered for the analysis. 343

344 **5. Results** 

## 345 5.1. Sedimentological analysis

To perform the paleoenvironment interpretation from the Arroyo Oscuro and Arroyo Las Playas localities, two stratigraphic sections were measured (Figs. 3-4). These localities had not been studied in detail before and included both the Bajada del Agrio Group and the Diamante Formation deposits. Considering that there is no evidence of the Patagonidic regional unconformity along the studied sections, a transitional boundary between both units was assumed (see discussion section). Towards the top of the Arroyo

Las Playas section, the Diamante Formation shows a transitional passage to marginal marine deposits of the Saldeño Formation (Tunik, 2003, 2004).

354 **5.1.1. Arroyo Oscuro** 

This section is 316 m thick (Fig. 3), where 221 m belong to the Diamante Formation while 70 m and 25 m belong to the Huitrín and Rayoso formations, respectively. Three main textural groups of sedimentary facies were identified: (1) conglomeratic and (2) sandy clastic facies, (3) calcareous and evaporitic facies. A total of nine sedimentary facies have been characterized (Table A1.1, Appendix 1). Furthermore, we classified these deposits into three main facies associations; A: restricted brackish lake, B: sheet-flood deposit, C: channelized fluvial deposits.

362 Conglomeratic facies (Table A1.1., Gmm, Gcm, Gct) are the least abundant in the 363 area and show fining-upward co-sets with variable thicknesses (up to 3 m) and lateral 364 extensions (from 1 to 27 m). The geometry of the sedimentary bodies is variable, with a predominance of lenticular and chaotic forms. Cut and fill structures have been observed 365 366 in these facies. The presence of isolated angular clasts of variable sizes (~6 cm) is common. This feature was frequently observed in the non-channelized fluvial deposits of 367 368 both sections. The most common arrangement begins with coarse to fine conglomeratic 369 (Gmm, Gcm, Gct), followed by coarse-sandstone facies and finishes with medium to fine sandstone facies (St, Sh, Sl) with bioturbation (Sm). A massive character predominates 370 in the sedimentary deposits, and the presence of 3D channelized sedimentary bodies and 371 372 imbricated clast is restricted; however, they allowed the measurements of paleocurrent 373 data (Fig. 3). Another important feature recognized is the difference in the composition of the conglomerates along the entire section. While the lower part of the section shows 374 a predominance of acid volcanic clasts (especially rhyolites), the middle and the upper 375 part are composed almost exclusively by clasts of limestone. 376

The sandy facies are predominant, with even more varied sedimentary structures 377 378 than the previously mentioned facies. Four facies have been recognized: St, Sh, Sm, and SI (Table A1.1, Appendix 1). Together, they represent a continuous succession of 379 channels and bars that are linked laterally and vertically, exhibiting both ribbon and sheet 380 geometries as well as individual bodies. Bioturbation is very common and frequent, 381 obliterating the primary structure. In the same way, an increase in bioturbation towards 382 383 the middle and upper part of the section has been observed. Regarding the ichnological content, Scoyenia isp., Skolithos isp., and Arenicolites isp. were recognized. Furthermore, 384 undifferentiated vertical and horizontal tubes have been identified. Additionally, the 385 386 presence of mottled massive sandstones with carbonate nodules is very common.

**5.1.2.** Arroyo Las Playas 387

A detailed examination of the succession in the Arroyo Las Playas area with a 388 389 total thickness of 614 m, allowed the identification of 532 m belonging to the Diamante Formation, while 70 m and 12 m belong to the Huitrín and Rayoso formations, 390 391 respectively. The essential characteristics of these facies are summarized in Table A1.1 (Appendix 1). Thirteen sedimentary facies were distinguished and divided into four 392 393 textural groups; (1) conglomeratic, (2) sandy and (3) mudstones clastic facies, and (4) 394 calcareous and evaporitic facies. As the result of the facies analysis, five facies associations were defined and interpreted; A: restricted brackish lake, B: ephemeral lake, 395 C: sheet-flood, D: channel and bars and E: floodplain. 396

397 In this area, as well as in the other sections, the conglomeratic facies were 398 comprised into sedimentary bodies with an erosive base and fining-upward trends. Internally, these bodies correspond to a clast-supported conglomerate with parallel 399 stratification (Ghc). Clasts are angular to subangular, composed by volcanic lithics and 400 reach up to 3-4 cm on the A-Axis. A massive matrix-supported conglomerate (Gmm) 401

with subangular to subrounded clasts of 6 cm long (along the A-axis), and a massive clast-402 403 supported conglomerate (Gcm) with volcanic and limestone clasts of up to 7 cm long 404 (towards the top of the section) have been also identified (Fig. 5a). Finally, clast-405 supported conglomerate facies with tangential and festoon cross-bedding (Gct) were identified, consisting mainly of angular to subrounded volcanic clasts, mainly of rhyolitic 406 composition, although clasts of dacite, trachyte, and andesite were also observed. Those 407 408 volcanic clasts present themselves in various ranges of sizes (1-7 cm). Conglomerates composed almost exclusively of limestone clasts can also be observed at the top of the 409 410 section, as well as subangular red-siltstone clasts.

Regarding sandy facies, medium to very fine sandstone with parallel stratification (Sh) have been identified, which eventually showed internal structures of parallel and ripple cross laminations, as well as some pedogenetic features such as mottling. Sandstone with parallel lamination (Sl), medium to fine-grained pebbly sandstone (SGm) with isolated clasts, and medium to very fine massive sandstone (Sm), which correspond to the most abundant sandstone within the surveyed area, have also been recognized (Fig. 5b).

The fine-grained facies are represented by massive mudstones (Fm), normally semi-covered, mudstones with parallel lamination (Fl), and mudstones with parallel bedding (Fh). These facies normally show pedogenetic features such as mottled, slickensides, blocky and subangular peds, and undifferentiated bioturbations.

The calcareous and evaporitic facies are present in the basal part of the succession of the Arroyo Las Playas area as well as the Arroyo Oscuro area (Figs. 3-4) and consists of 70 m-thick gypsum/massive anhydrite beds (Em) and less common gypsum/laminate anhydrite, which are interbedded with stratified limestone (Lh).

## 426 **5.1.3. Paleocurrent analysis**

A total of 44 paleocurrent directions were measured in the Arroyo Oscuro area, evidencing a significant change between the bottom and the top of the section (Fig. 3), revealing an important shift in the paleocurrent direction. They are east-derived (on average towards 285° Az) at the bottom and west-derived (on average towards 093° Az) at the top. In contrast, in the Arroyo Las Playas area, the paleocurrent data is limited to just one (towards 15° Az) at the top, due to the absence of 3D measurable sedimentary structures and the scarce levels with imbricated clasts.

434

## 4 **5.2. Petrographic description and modal analysis**

For the purpose of this study, we include new petrography data from the Albian 435 436 to Campanian deposits of the Arroyo Oscuro and the Arroyo Las Playas localities. They are compared with the data obtained from nearby study areas (Gómez et al., 2020; Lothari 437 et al., 2020). Petrographic observations facilitated a general description of the principal 438 439 components from the clastic fraction. Different types of quartz (Q), feldspars (F), and lithic fragments (L) were discriminated and quantified. Likewise, the features of the 440 441 cements and the presence of heavy minerals in the thin sections were observed. The sandstones were classified following the proposal of Folk et al. (1970) and were mainly 442 443 lithic feldarenite and feldespathic litharenite. The average value of the percentage in 444 weight for these samples is  $Q_{50}F_{24}L_{25}$  (Appendix 1-Fig. 7a). Quartz was the predominant component in all the samples (50%) and appears in the form of monocrystalline (33%) 445 and polycrystalline in subordinate proportions (7.2%). The most abundant type of quartz 446 447 is monocrystalline with straight extinction (26.4%), it generally appears well rounded, 448 and in some cases with inclusions. Quartz, as a fragment of a volcanic rock (0.9%) was 449 also recognized, as well as quartz with an undulatory extinction (5.7%). In particular, almost all samples contain embayment quartz (Fig. 6c). The variation in the quartz content 450 within the two areas shows a decrease in the middle part of both sections. Feldspars 451

constitute approximately 21-33% of the samples. Alkaline feldspar is the most common 452 453 type and was identified by Carlsbad or tartan twinning, when present. Plagioclase feldspar 454 was identified primarily by albite twinning. Both types of feldspars normally show 455 sericitic and argillic alteration, but it is more frequent in alkaline feldspar. Alkaline feldspar and plagioclase were recognized as volcanic rock crystals in subordinate 456 457 amounts. Furthermore, there was no correlation between either profile, or from the base 458 to the top. In the Arroyo Oscuro area, the plagioclase/total feldspar ratio does not vary and its value remains at 0.35, while in the Arroyo Las Playas area, there is a high value 459 at the bottom section, with a ratio of 0.65 and the ratio remains at 0.45 on average. 460 461 Recognition of different categories of lithic fragments is very important in provenance studies. Lithic fragments reach on average 25% of the clastic fraction and are almost 462 exclusively volcanic, and more specifically the paleovolcanic type following the criteria 463 464 used by Critelli and Ingersoll (1995). Paleovolcanic lithic fragments with granular (3%) and seriate (5%) textures are predominant, although volcanic fragments with lathwork 465 466 (0.6%), microlitic (0.2%) and pyroclastic (1%) with eutaxitic textures were also observed (Fig. 6a). It is very important to note the presence of calcareous lithic fragments in the 467 468 mid and top part of the Arroyo Oscuro section, although they were in low proportions (on 469 average 1%) (Fig. 6d). In contrast, altered lithic fragments were observed in relatively high proportions (on average 6%). Finally, sedimentary lithic grains, metamorphic rock 470 fragments and plutonic lithic clasts were observed, as well, in very low proportions (less 471 472 than 2%). Regarding the types of cement, ferruginous is the most common (5.9%), 473 followed by calcareous (4.2%) argillaceous (2%), and zeolitic (1.7%) types. The cement 474 appears as pore filling, pore lining and, more scarcely, as poikilotopic. The zeolitic cement corresponds to analcime type, which occurs as pore filling and in subhedral 475 crystals related to calcite cementation. The presence of this type of cement is important 476

for the provenance interpretation (see discussion below). Minor components correspond
to micas and opaques, along with heavy minerals such as zircons and apatite among others
(Fig. 6e-f).

Regarding sandstone point counting, a recalculated modal composition was carried out (Appendix 1) and plotted on a tectonic discrimination diagram of Dickinson et al. (1983). The Qt-F-Li graph indicates that the analysed samples from both sections correspond to recycled orogen. Furthermore, in the Qm-F-Lt diagram, the distributions of the samples are clustered in mixed and dissected arc fields. Figure 7b shows both graphs, as well as previous data obtained from other sections in the study area (Gómez et al., 2019).

## 487 **5.3. Apatite and zircon fission track**

The VLP001 sample from the lower part of the Diamante Formation reported an Early Cretaceous (Albian) AFT central cooling age of  $111.9 \pm 13.6$  Ma (Table 1), linked to a unique statistical population of grain-ages with a low degree of dispersion and a  $P(x^2)>5\%$  value (Fig. 8-Ia). The probability density distribution of the grain-ages indicates a major peak between 100 and 120 Ma, followed by a subordinated peak at ~200 Ma (Fig. 8-Ic); both correlative with those evidenced in the cumulative grain-age distribution plot (Fig. 8-Ib).

Thirty-seven zircon crystals from the Diamante Formation (sample 2119) dated by ZFT, did not pass the chi square test (P( $x^2$ )<<5%, Fig. 8-IIa), resulting in two discrete populations of grain-ages of  $P_1 = 161 \pm 16.4$  Ma (71%) and  $P_2 = 265.2 \pm 46.5$  Ma (29%) (Table 1, Fig. 8-IIa). Both populations are visible in the probability density distribution and the cumulative grain-age distribution plots (Figs. 8-IIb-c). See Appendix 3 for more details.

## 501 5.4. Morphological analysis of apatite and zircon crystals

From the total number of analysed apatite crystals (n=530) of the VLP001 sample 502 503 (Fig. 9), the morphological analysis of 383 apatites, showed a predominance of rounded crystals (Fig. 9c), corresponding to 56% of the total. Angular (Fig. 9a) and subangular 504 505 (Fig. 9b) crystals accounted for proportions of 21% and 23%, respectively, while 147 apatite grains with fractures were not considered for this analysis (See Appendix 5 for 506 507 details).

508 In the case of the zircon crystals from the VLP001 sample, from the total number of analysed grains (n=508) (Fig. 10), the morphological analysis of 361 zircons sample 509 showed a predominance of subangular crystals (Fig. 10b), corresponding to 40% of the 510 511 total. Angular (Fig. 10a) and rounded (Fig. 10c) crystals accounted for proportions of 33% and 27%, respectively, while 147 zircon grains with fractures were not considered 512 513 for this analysis (See Appendix 6 for further details).

514 6. Discussion

#### 6.1. Paleoenvironmental interpretation 515

516 The facies associations previously defined for the Arroyo Oscuro and the Arroyo Las Playas areas indicate particular depositional environments. 517

518 In the case of the Huitrín and the Rayoso formations (Bajada del Agrio Group), 519 the facies associations represent a restricted marginal marine system that evolved to an ephemeral lacustrine environment. The Huitrín Formation is linked to an inland 520 hypersaline shallow sea with high temperatures resulting in high evaporation rates, as 521 522 well as periodic siliciclastic depositional stages. The presence of fine-grained siliciclastic 523 facies at the top of the succession could suggest meteoric sediments coming from emerged areas or/and changes at the base level (Roulston and Waugh, 1983). The connection with 524 the proto-Pacific Ocean located to the west was limited by a magmatic arc, which was a 525 topographic barrier that partially restricted the influx of seawater. This sedimentary 526

sequence depicts the transition between the backarc and the foreland stages during the
Early to the Late Cretaceous periods (Veiga and Vergani, 2011; Gabriele, 2016; Lothari
et al., 2020; among others).

530 The Arroyo Oscuro and the Arroyo Las Playas areas, as well as previously studied sections of Vega Grande and Vega de los Patos (Gómez et al., 2020; Lothari et al., 2020), 531 532 reveal that the Diamante Formation comprises a complex fluvial system that reflects 533 changes in depositional controls (e.g., tectonic and climate), and in the accommodation and sediment supply conditions represented by shifts in the floodplain deposit's thickness 534 and the stacking of channels (Table A1.1, Appendix 1). Furthermore, the changes of the 535 536 paleo-slope resulted in a variation in the facies associations and the geometry of the sedimentary bodies (Schumm, 1981; Ramon and Croos, 2002; Bridge, 2003; Miall, 537 2014). Based on these characteristics, we propose that the Diamante Formation was 538 539 deposited by a braided fluvial system that evolved over time to a meandering fluvial system. Furthermore, we argue that the Diamante Formation would have been part of the 540 541 medial zone of a fluvial fan or Distributive Fluvial System (DFS) (Nichols and Fisher, 2007; North and Warwick, 2007; Cain and Mountney, 2009; Hartley et al., 2010; 542 543 Weissmann et al., 2010; Miall, 2014).

544 The coarse-grained facies (Gmm, Gcm, Gct, Sgm, St, Sh) of the Arroyo Oscuro and the Arroyo Las Playas areas show distinctive processes associated with braided and 545 meandering fluvial systems, with the stacking of channels and bars limited by erosive 546 547 surfaces, and the presence of cut and fill structures that represent the overlapping of 548 various events, leading to multi-story channel. The presence of juxtaposed younger and older individual channels lumped together into a composite body is very common, and is 549 associated with successive relocations of the river (avulsion). The ratios between the 550 width and the thickness of fluvial-channel bodies is an average of 0.5 to 9, which is 551

evidence of narrow to broad ribbons based on the classification of Gibling (2006) (Fig. 552 553 5b). Considering the geomorphic setting, the geometry, and the internal characteristics of these fluvial-channel bodies, they can be collectively defined as parts of distributive 554 555 systems (Gibling, 2006). A multiepisodic sandy-gravel channelized complex is recognized within the facies association C in the middle part of the Arroyo Oscuro area 556 (Fig. 5c-d), based on the methodology suggested by Miall (1985, 1996) and Cain and 557 558 Mountney (2009 and references therein). This architectural element describes a simple lateral and vertical arrangement of sedimentary facies of clast-supported conglomerates 559 with tangential and festoon cross-beddings (Gct), medium to fine sandstones with planar 560 561 parallel stratifications (Sh), and fine to very fine massive sandstones with bioturbations (Sm). This setting conforms to packages of amalgamated macro-channels of a 2 to 6 m 562 thickness, interbedded with non-channelized sandy facies (Fig. 5e-f). 563

564 There is a predominance of massive structures, both in non-channelized conglomerates and sandy facies (Gmm, Gcm, Sgm, Sm), probably associated with 565 566 flooding events responsible for debris flow deposits. These deposits describe irregular and sharp – often non-erosional – bases, and form lobes, ribbons or sheets (e.g., Miall, 567 1985, 1996; Bridge, 2003). The presence of isolated clasts within these deposits is 568 569 interpreted as traction-carpets deposited by a high-density gravel turbidity current (Lowe, 1982; Mutti, 1992). Furthermore, it cannot be ruled out that the massive character of these 570 facies could be related to the destruction of primary depositional structures by 571 bioturbation. 572

573 Fine-grained facies (Fm, Fl, Fh), are part of floodplain facies associations and 574 show vertical accretion deposits with a maximum thickness of ~80 m. The main processes 575 associated with these sedimentary facies are the decanting of the fine material produced 576 during the final stage of the decelerated flows. The high level of bioturbation in these

facies and the presence of mottled, slickensides, blocky and subangular peds, are evidence 577 578 of pedogenetic processes. The high percentage of floodplain facies associations in the 579 study area, as well as in nearby localities (Gómez et al., 2020; Lothari et al., 2020), could 580 be related to a combination of tectonic and climate factors during sedimentation processes. In the studied sections, these deposits are frequently truncated by isolated 581 channel-bodies with  $\sim 2$  m of thickness. The presence of these isolated deposits would be 582 583 explained by a typical mechanism of deposition called "incisional avulsion" (Slingerland 584 and Smith, 2004).

## 585 6.2. Provenance analysis: Clasts composition and paleocurrent data

586 Acid and intermediate volcanic (rhyolites-trachytes) clasts are the most common rock constituents in the studied succession. These fragments could be associated with the 587 588 Choiyoi Group (Permian-Triassic), which integrates the structural basement of the Neuquén Basin, and are very common in other sections of the Neuquén Group from 589 590 different sectors of the basin (Garrido et al., 2010; Balgord and Carrapa et al., 2016; Borghi et al., 2019; among others). These clasts could derive from the erosion of the 591 592 eastern San Rafael Block, if we consider this area as a positive forebulge as Balgord and 593 Carrapa (2016) proposed. A minor number of andesitic-dacitic volcanic clasts was also 594 recognized in the Arroyo Oscuro and the Arroyo Las Playas areas probably associated with the Choiyoi Group. More recently, Martos et al. (2020) described the presence of 595 596 metamorphic clasts and measured north-derived paleocurrents in Vega de Los Patos area, which would indicate that the Frontal Cordillera could have been a potential source. 597

Red-siltstones are present as subangular clasts in conglomerate facies at the top of Arroyo Las Playas section and can be associated with the erosion and redeposition of the underlying Rayoso or Tordillo formations. Of particular relevance for the provenance analysis is the occurrence of limestone lithic clasts starting from the middle part of both

602 sections and persisting to the top of the sequence. This macroscopic observation was also 603 recognized in neighbouring study areas (Gómez et al., 2019; Lothari et al., 2020). 604 Moreover, limestone lithic clasts within the Diamante Formation are evident to the west 605 (Broens and Pereira, 2005). The regional occurrence of these calcareous clasts (Tunik, 2001; Balgord and Carrapa, 2016; Fennell et al., 2017a; Gómez et al., 2019; Borghi et al., 606 607 2019; Lothari et al., 2020), would suggest the presence of a common regional source 608 associated with the exhumation of the Andean orogen west of the study area, and the erosion of the Mendoza Group (Upper Jurassic-Lower Cretaceous) or the Lotena Group 609 (Middle-Upper Jurassic), representing a regional depositional event. 610

611 Paleocurrent measurements from the Arroyo Oscuro area (n=44; Fig. 3) evidence 612 an important change of direction in the middle part of the section. The orientation of 613 imbricated clasts and 3D measurable sedimentary structures describes a W-directed 614 paleoflow for the first part of the section (average =  $285^{\circ}$ Az; n=35), followed by an Edirected paleoflow in the upper part (average =  $093^{\circ}$ Az; n=9), possibly derived from the 615 616 San Rafael Block located to the east, and the Andean orogen situated to the west, respectively (see the U-Pb detrital zircons peaks to compare, Fig. 8-III). Even though the 617 618 role and exhumation of the San Rafael Block during Mesozoic times remains unclear, the 619 presence of a positive topography eastwards becomes evident. This interpretation is 620 consistent with the hypothesis of a forebulge or peripheral bulge area associated with the Andean fold-and-thrust belt foreland (Tunik et al., 2010; Di Giulio et al., 2012; Borghi et 621 622 al., 2019). In fact, if we consider the proximity (~50 km) between the non-marine deposits 623 of the Diamante Formation in the study area and the San Rafael Block, we can infer that 624 the variations in the provenance patterns are part of a coalescent fluvial system with a predominance of W-directed paleocurrents during the initial deposition of the Diamante 625

Formation deposits. Then, a shift in the paleocurrent direction occurred, provoked by theonset of the Andean orogeny (Fig. 11).

## 628 6.3. Onset of foreland basin deposition: diachronism or synchronism?

The analysis of the U-Pb maximum depositional ages documented for non-marine 629 Lower to Upper Cretaceous synorogenic deposits throughout the Neuquén Basin in the 630 631 last decade, integrated with those obtained from the base of the Diamante and the Candeleros formations, should permit us to better constrain the beginning of the foreland 632 basin infill (Fig. 12). Nevertheless, differences exist when comparing all maximum 633 depositional ages proposed by each author from different sectors of the basin. The 634 Candeleros Formation yields a maximum depositional age of  $100.5 \pm 2.1$  (n=1) and 104.3635 636  $\pm$  2.5 Ma (n=1) (Tunik et al., 2010); and Di Giulio et al. (2012) obtained similar ages of  $102 \pm 2$  Ma (n=5) and  $100 \pm 8$  Ma (n=1), both for the Agric fold-and-thrust belt (37°-637 38°S). In the southern Mendoza province (35°-36°S), Fennell et al. (2017a) obtained a 638 639  $100.2 \pm 2.1$  Ma age (n=1) and Balgord and Carrapa (2016) a 97  $\pm 2$  Ma age (n=4) for the base of the Río Limay-Subgroup and the Diamante Formation, respectively. Recently, at 640 the same latitudes, Borghi et al. (2019) defined two maximum depositional ages for the 641 Río Limay-Subgroup, one of  $101.6 \pm 2.6$  Ma (n=2) near the contact with the Rayoso 642 643 Formation, and another of 91.4  $\pm$  2.3 Ma (**n=4**) 70 m above the first age obtained. Our previous studies in a nearby locality yielded two maximum depositional ages of  $107.2 \pm$ 644 645 1.4 Ma (n=4) for a litharenite deposit from the lower part of the Vega de Los Patos area (Fig. 8-III), and 91.1  $\pm$  2.2 Ma (**n=3**) for a reworked-tuff from the top of Vega Grande 646 647 section (Gómez et al., 2019).

648 The lack of a tight correlation between the maximum depositional ages for the 649 first Andean synorogenic deposits, likely relates to the method applied for the estimation 650 of the ages in each case, rather than a diachronic deposition. The methods applied for that

purpose range from the most robust (e.g., two or more young grain-ages that overlap) to 651 652 the least robust (e.g., the -unique- youngest grain-age) (Dickinson and Gehrels, 2009), sometimes involving a certain grade of subjectivity. This becomes evident when a 653 654 detailed comparison between the distributions of the youngest grain-ages of each sample is performed. If all youngest U-Pb detrital zircons from previous studies (Tunik et al., 655 656 2010; Di Giulio et al., 2012; Balgord and Carrapa, 2016; Fennell et al., 2017a, Borghi et 657 al., 2019; Gómez et al., 2019) are analysed in detail, a common group of overlapped grainages which cluster around ~109-107 Ma (Fig. 12) can be recognized for each sample. 658 Considering that the number of zircons involved in this interval of ages is statistically 659 660 more robust than selecting a unique age in each case (Tucker et al., 2013; Coutts et al., 2019; Vermeesch, 2021), we argue for an Albian age (Lower Cretaceous) for the 661 Candeleros Formation (or for the base of the Diamante Formation). 662

663 **6.4.** 

## 6.4. Lower Cretaceous to Upper Cretaceous volcanic activity

The application of a multiproxy approach for the provenance analysis of the 664 665 Arroyo Oscuro and the Arroyo Las Playas areas, and its integration with pre-existing data (Gómez et al., 2019; Lothari et al., 2020), allowed us to identify a potential signal of 666 volcanic activity at 34-35° for the Albian. The petrographic analysis developed in the 667 668 Arroyo Oscuro and the Arroyo Las Playas areas evidence an important presence of volcanic lithic fragments, where felsitic and granular textures are common (Figs. 6-7). 669 The same analysis from the Vega Grande area also showed subordinated vitric textures 670 671 (Gómez et al., 2019). These characteristics could be indicative of a potential volcanic 672 source related to an incipient Late Jurassic-Early Cretaceous magmatic arc located to the west, as already proposed by Vergara et al. (1995). According to Affolter and Ingersoll 673 (2019), the granular textures are usually related to high-SiO<sub>2</sub> sources. These authors also 674 explain the importance of considering the preservation of textural and compositional 675

676 types. Vitric textures are the most reactive because the glass is fragile, especially in the 677 form of glass shards and bubble walls. Conversely, lithic fragments with high-SiO<sub>2</sub> 678 content are generally more stable during weathering. This could explain the lack of vitric 679 textures in the petrographic thin sections of the Arroyo Oscuro and the Arroyo Las Playas areas. Furthermore, pyroclastic lithic fragments with eutaxitic textures, embayment on 680 the quartz, and analcime as cement, have also been observed. The analcime is very 681 682 common in volcanic environments since it could be formed by dissolution and precipitation from the volcanic glass. Sandstone samples show medium to low 683 plagioclase/total K-feldspar ratios, ranging from 0.65 to 0.45 (from the base to the top of 684 685 the Arroyo Las Playas area). This trend could indicate a felsic plutonic provenance (e.g., Critelli and Ingersoll, 1995; Critelli and Nilsen, 2000). 686

AFT analysis of VLP001 reveals a unique central cooling age of  $111.9 \pm 13.6$  Ma 687 688 (Albian) ( $P(x^2) > 5$ , Fig. 8a). This age could reflect diverse hypothetical scenarios: (1) Albian cooling/exhumation in situ, with an erased inherited signal by a total reset after 689 690 deposition; (2) cooling/exhumation of the source during Albian times; or (3) magmatic cooling of a volcanic source during Albian times. Considering that the maximum 691 692 thickness of the sediments registered since Albian times in the study area are in the range 693 of ~1,165-1,373 m (Turienzo et al., 2012), a total reset of the AFT isotopic system of the VLP001 sample after deposition is not feasible. The estimation of a lag time between both 694 the AFT and the U-Pb maximum depositional age of the VLP001 sample (111.9  $\pm$  13.6 695 696 Ma and  $107.2 \pm 1.4$  Ma, respectively) results in ca. 3 ma, with both ages overlapped, 697 considering their range of analytical uncertainty. This time lapse is considerably tight to account for the exhumation of the source, erosion, transport and deposition, being better 698 explained by a rapid magmatic cooling of the source (e.g., Malusà et al., 2011; Malusà 699 700 and Fitzgerald, 2019 and references therein).

It is important to highlight that most of the apatite crystals of the VLP001 sample that were analysed by AFT, have euhedral to subhedral morphologies. Rounded (anhedral) apatite-crystals were not included for measuring purposes because of the problems of finding crystals with an appropriate orientation (Dpar parallel to the ccrystallographic axis). Therefore, we infer that the central cooling age obtained represents the same population of apatites derived from a volcanic source, as suggested by the estimated short lag time.

The morphological analysis of 530 apatite and 508 zircon crystals from the 708 VLP001 sample through SEM, reveals the presence of three populations of grains: 709 710 rounded (56%), subangular (23%), and angular (21%) (Fig. 9) for the apatites analysis, and subangular (40%), angular (33%) and rounded (27%) (Fig. 10) for the zircons 711 analysis. This approach was applied to observe morphological features of these grains 712 713 and used as a fingerprint of the provenance source in the foreland basin deposits (e.g., Fedo et al., 2003; Finzel, 2017). The high proportion of angular and subangular apatites 714 715 and zircons is in good agreement with a volcanic provenance whereas the prevalence of 716 rounded grains in the sample could evidence the recycling of the Mesozoic sedimentary 717 units.

718 Based on the petrological and sedimentological analysis, and its integration with AFT and U-Pb ages of the VLP001 sample, we argue for the presence of a volcanic arc 719 720 located west of the study area, coeval with a non-marine Diamante Formation deposition 721 during the latest Early Cretaceous times (119-102 Ma). The volcanic activity towards the 722 Late Cretaceous becomes evident given the recognition of the reworked-tuff interbedded in the Vega Grande area where one of these layers yielded a maximum depositional age 723 of 91.1  $\pm$  2.2 Ma (Gómez et al., 2019). The youngest detrital zircons of this sample (~92-724 80 Ma), is also correlative with the U-Pb detrital zircon-ages provided for the Mesozoic 725

units from the western Andean slope, which are contemporaneous with the deposition ofthe Diamante Formation (Muñoz et al., 2018).

728 Our proposal matches the existence of a continuous activity of a magmatic arc 729 suggested for the Late Jurassic-Late Cretaceous period and the Lower Cretaceous contractional episode, both suggested for the western slope of the Andes at these latitudes 730 (Charrier et al., 2007; Oliveros et al., 2018; Tapia et al., 2020) (Fig. 11). Additionally, 731 732 Balgord (2017) observed continuous volcanism from 190 to 140 Ma, followed by major age populations at 129 Ma, 110 Ma, 67 Ma, 52 Ma, 16 Ma, and 7 Ma, based on a 733 combination of a large bulk of detrital zircon data from the Neuquén basin between 34° 734 735 and 40°S. This author proposed a lull in volcanic activity during the retroarc foreland basin deposition, with a hiatus of 40 ma between 110 Ma and the first high-flux event 736 after the initial shortening at 70 Ma. 737

Mesozoic units from the Chilean slope of the Andes (33-36°S) evidence a 738 739 synorogenic nature; as well as a volcanic provenance represented by volcanoclastic and volcanic deposits of the BRCU, Las Chilcas, and the Colimapu formations (Boyce et al., 740 2014; Muñoz et al., 2018; Tapia et al., 2020). The U-Pb zircon-ages indicate an Albian 741 depositional age for the Las Chilcas Formation in the forearc (Godoy et al., 2009; Boyce 742 743 et al., 2020; Contreras and Schilling, in press), coincident with the maximum depositional age of  $107.2 \pm 1.4$  Ma obtained from the Vega de Los Patos area located in the retroarc 744 745 foreland basin (Gómez et al., 2019). However, Tapia et al. (2020) proposed that the compressive deformation along the Coastal Cordillera at ~35° S would have begun at 113 746 747 Ma with the deposition of the Las Chilcas Formation along the eastern slope of the late 748 Early Cretaceous orogen, in agreement with an accelerated exhumation period registered between 113 and 80 Ma along the Paleozoic metamorphic basement of the same region 749 750 (Willner et al., 2005). Moreover, the maximum depositional age of  $107.2 \pm 1.4$  Ma

matches the onset of the Andean exhumation and denudation proposed by Galetto et al. (2021) for the Albian (~110 Ma), based on inverse thermo-numerical modeling of multiple thermochronometers from the northern Chos Malal fold-and-thrust belt (36-37°S). At an intercontinental scale, this proposal coincides with a remarkable increase in plate spreading rates, interpreted as the responsible for the consequent Andean compressional stage (Somoza and Zaffarana, 2008; Matthews et al., 2012; Müller et al., 2016).

6.5. Significance of Permian and Late Jurassic zircon-ages

### 758

A ZFT analysis of the top of the Diamante Formation in the Vega de Los Patos 759 area (2119 sample), yielded two discrete populations of grain-ages of  $P_1 = 161.1 \pm 16.4$ 760 761 Ma and  $P_2 = 265.2 \pm 46.5$  Ma (Table 1, Fig. 8). These results reveal a non-reseted ZFT isotopic system, where both ages reflect source-cooling. This is in agreement with the 762 maximum thickness of the sedimentary sequence registered since Albian times in the 763 study area (~1165-1373 m, Turienzo et al., 2012), not enough to reset/partial reset the 764 ZFT isotopic system of the 2119 sample. Both populations are consistent and overlapped 765 (considering the range of analytical uncertainty) with the second and third peaks visible 766 on the frequency histogram and the relative probability plot of the U-Pb detrital ages from 767 the VLP001 sample (148 and 267 Ma, respectively; Fig. 8). In this sense, both the Upper 768 Jurassic U-Pb and the ZFT zircons-ages (U-Pb =  $\sim$ 148 Ma, ZFT  $P_1$  = 161.1 ± 16.35 Ma) 769 770 could derive from the Jurassic volcanic arc situated in the Coastal Cordillera by that time (Oliveros et al., 2006; Tapia et al., 2020), as well as from the erosion and recycling of the 771 772 Mesozoic units (Rossel et al., 2014; Naipauer and Ramos, 2016; Naipauer et al., 2018). The Permian U-Pb and the ZFT zircon-ages (U-Pb = ~267 Ma, ZFT  $P_2 = 265.2 \pm 46.5$ 773 Ma) could derive from the reworked pre-Mesozoic units of the Choiyoi Group (Sato et 774 775 al., 2015) (Fig. 11). Regarding the absence of the cretaceous cooling age-population in this sample, it could indicate a decrease in the volcanic input to the top of the Vega deLos Patos section.

778 **7. Conclusions** 

A multiproxy provenance analysis developed on the Arroyo Oscuro and the Arroyo Las Playas areas, and its combination with previous studies, evidence the presence of coeval volcanic activity during the onset of the Andean foreland basin at 34-35°S. The evidence that support our proposal are summarized below, integrated with paleoenvironment and regional tectonic scenarios:

- The stratigraphic sequence of the study area records the transition between the 784 backarc and the foreland basin stages in the Neuquén Basin. Both stages are represented 785 786 by a transitional boundary between the Bajada del Agrio Group and the Diamante Formation. The Rayoso and the Huitrín formations (Bajada del Agrio Group) correspond 787 to a restricted marginal marine system, evolving over time towards an ephemeral lake 788 789 environment. The analysis of the non-marine deposits of the Diamante Formation allow the identification of a braided fluvial system that evolves towards a meandering fluvial 790 791 system, as part of the medial zone of a fluvial fan or Distributive Fluvial System (DFS). 792 Our results evidence that the Diamante Formation was deposited in a foredeep depozone 793 with sediments derived both from the Andean fold-and-thrust belt, and the magmatic arc located to the west, as well as from the forebulge located to the east. 794

- There is a predominance of volcanic clasts with acid to intermediate compositions through the Arroyo Oscuro and the Arroyo Las Playas sections, with a particular occurrence of calcareous clasts in their middle parts. This change was accompanied by a shift in the paleocurrent direction (more evident in the Arroyo Oscuro area) that reveal a mixed provenance from the western and the eastern areas (Andean Range and basement forebulge, respectively) for the first foreland deposits, as well as a

notable increase in the western input for the second half of the sections. Additionally,
petrographic analyses show a predominance of feldspathic litharenite according to the
Folk et al. (1970) classification, while the source areas are mainly recycled orogen
(diagram QFL) and mixed and dissected arc (diagram QmFLt), according to Dickinson
et al. (1983).

806 - The analysis of the U-Pb maximum depositional ages documented for nonmarine Upper Cretaceous deposits throughout the Neuquén Basin, integrated with those 807 808 obtained from the bottom of the Diamante and the Candeleros formations, evidence a 809 persistent group of overlapped grain-ages that cluster around ~109-107 Ma. Based on this correlation, and on the pursuit of reviewing the methods applied for the statistical analysis 810 811 of the U-Pb detrital zircon-ages, we propose an Albian age for the base of the Diamante Formation and the Candeleros Formation, and we argue for a re-evaluation of the 812 diachronism between the first foreland deposits of the Neuquén Basin. 813

- Apatite fission track analyses from the bottom of the Diamante Formation 814 815 (VLP001) yield an Albian central cooling age, derived from the measurement of euhedral to subhedral apatite crystals. The comparison between the AFT central age and the U-Pb 816 817 maximum depositional age of the same sample allows the estimation of a short lag time of ca. 3 ma, interpreted as evidence of a volcanic arc provenance during Albian times. In 818 819 this scenario, the AFT central age would represent the rapid cooling of a magmatic source. 820 Additionally, morphological analysis of apatite and zircon crystals from VLP001 through 821 SEM images, reveal a notable presence of angular (euhedral) crystals, likely derived from 822 a magmatic source, with subordinated rounded crystals that reflect a sedimentary input as 823 well.

Zircon fission track analyses of sample 2119 collected from the top of the
Diamante Formation yield two discrete populations of grain-ages of ca. 161.1 Ma and ca.
265.2 Ma. Both are interpreted as inherited signals, in reflection of source-cooling. The
first population of apatites could derive from the erosion of the Upper Jurassic magmatic
arc, therefore revealing its magmatic cooling. The second population could derive from
the erosion of the Choiyoi Group units, reflecting their magmatic cooling during Permian
times.

- The results presented in this work suggest a coeval volcanic activity during the Diamante Formation deposition, and are consistent with the volcanic and volcaniclastic processes documented in the Chilean Mesozoic sediments west of the study area. The new data provided in this work highlight that deeper studies are needed to better constraint the onset of the foreland basin at these latitudes, and to better define the role of the volcanic arc and its influence on the foreland deposits in the Southern Andes during the latest Early-Late Cretaceous period.

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1395 Figure captions

Figure 1. Regional DEM showing the location of the study area in the context of South
America and the Neuquén Basin extension in the Jurassic-Cretaceous period. The main
morphostructures are shown together with the maximum eastward progression of the Late
Cretaceous orogenic front, and the extension of the Neuquén Group outcrops at present.

Figure 2. a) Local DEM of the study area with detail of the main morphostructures. b)
Local geological map showing the main units, structural features and the location of
stratigraphic sections (after Lothari et al., 2020). 1: Vega Grande; 2: Vega de Los Patos;

1403 3: Arroyo Oscuro; 4: Arroyo Las Playas. Sections 1 and 2 correspond to the synthetic and

schematic sections created by Gómez et al. (2019). Sections 3 and 4 belong to this work.

- 1405 c) Schematic sections of Vega Grande and Vega de Los Patos with the location of the
- samples collected for the provenance analysis (see Fig. 3 for the references) (after Gómez
- 1407 et al., 2019).

1408 **Figure 3.** Measured stratigraphic section from the Arroyo Oscuro area.

1409 **Figure 4.** Measured stratigraphic section from the Arroyo Las Playas area.

Figure 5. Photographs of the analysed outcrops. a) Conglomerate from Arroyo Las Playas 1410 1411 section, composed almost exclusively of limestone rock fragments. b) Stacking of narrow 1412 to broad ribbon fluvial-channel deposits from the Arroyo Las Playas area. c) Outcrop view with: d) schematic panel depicting multiepisodic sandy-gravel channelized complex 1413 from the Arroyo Oscuro area. This architectural element shows paleoflow data and the 1414 1415 typical sedimentary facies, as well as the hierarchy of the architectural units according to Miall (1996). e) Lateral view of the same outcrop showing the thickness of the fluvial 1416 macroform. f) Detail of the sedimentary facies. 1417

Figure 6. a) AO01-17A sample. Photomicrographs of the framework's composition: 1418 monocrystalline quartz (Qm), plagioclase (Fpl), altered and undetermined lithic grain 1419 1420 (La) and pyroclastic paleovolcanic lithic fragment (Lp) with eutaxitic textures. Calcite as 1421 a main type of cement (Cca). Photomicrographs on NP and NX. Scale: 100 µm. b) 1422 ALP06-17 sample. Photomicrographs of framework composition: monocrystalline quartz (Om), altered and undetermined lithic grain (La), serial (Lps) paleovolcanic lithic 1423 1424 fragment. Analcime as a type of zeolitic cement (Cc). Photomicrographs on NP and NX. Scale: 60 µm. c) AO08-17 sample. Photomicrographs of framework composition: high 1425 percentage of monocrystalline quartz (Qm) showing embayment, plagioclase (Fpl), 1426

calcite cement (Cca) and different types of paleovolcanic lithic fragment. 1427 1428 Photomicrographs on NX. Scale: 100 µm. d) ALP06-17 sample. Photomicrographs of framework composition: limestone lithic fragments (Lc), monocrystalline quartz (Qm), 1429 1430 granular (Lpg) paleovolcanic lithic fragment, alkaline feldspar (Fk), altered and undetermined lithic grain (La), iron cement (Cf) and porosity (P). Photomicrographs on 1431 NP. Scale: 100 µm. d-e) ALP10-17 sample. Photomicrographs of framework 1432 1433 composition: monocrystalline quartz (Qm), altered and undetermined lithic grain (La), serial (Lps) paleovolcanic lithic fragments and opaques (Op). These photomicrographs 1434 also show one rounded zircon grained  $(34.563 \mu)$  and two apatite/zircon angular grained 1435 1436  $(50.755 \text{ and } 29.470 \,\mu)$ . Photomicrographs on NP. Scale: 20  $\mu$ m.

Figure 7. a) Sandstone classification QFL plot according to Folk et al. (1970) of the
analysed samples. b) QFL and QmFLt plots of sandstones from Diamante Formation to
discriminate provenance areas. On the left, QFL diagram from Dickinson et al. (1983).
On the right, QmFLt from Dickinson et al. (1983). Q: total quartz, F: total feldspar, L:
total lithic fragments, Qm: monocrystalline quartz, Lt: total lithic fragments plus
polycrystalline quartz.

1443 Figure 8. Graphical representation of AFT(I), ZFT(II) and U-Pb(III) data from samples VLP001 and 2119-Diamante. Fission track data is displayed by three different graphical 1444 1445 devices: a- radial plot of single grain-ages; b-cumulative grain-age distribution; c- Kernel 1446 and probability density distributions. Plots a- and c- were made using a Density Plotter 1447 (Vermeesch, 2012). The U-Pb ages from analysed zircons are displayed with a frequency 1448 histogram and relative probability plot, focused on the youngest ages considering the 1449 correlation purpose (see Appendix 4 for complete plot and Gómez et al., 2019 for further 1450 details). In the I and III plots, the maximum depositional age of sample VLP001 is depicted with a dashed black line, and the AFT central age of the same sample is depicted 1451

with a red shadow, including the range of analytical uncertainty. In the II plots, ZFT ages of discrete populations ( $P_1$ ,  $P_2$ ) from sample 2119-Diamante are indicated with continuous red and yellow lines, and the range of the analytical uncertainty is highlighted with the same colored shadow in each case.

Figure 9. Scanning Electron Microscope images (SEM) of apatite grains from the
VLP001 sample showing the morphological classification proposed. a) Angular. b)
Subangular. c) Rounded. The bar in each image represents 20 µm.

Figure 10. Scanning Electron Microscope images (SEM) of zircon grains from the
VLP001 sample showing the morphological classification proposed. a) Angular. b)
Subangular. c) Rounded. The bar in each image represents 20 µm.

Figure 11. Schematic block diagrams (unscaled) illustrating a paleogeographic 1462 reconstruction of the Andean Orogen (34-35°S). a) Early Cretaceous (Aptian) backarc 1463 1464 stage showing the final disconnection of paleo-Pacific Ocean with the Neuquén Basin during the deposition of the Huitrín Formation. b) the non-marine deposition of Rayoso 1465 Formation coeval with Colimapu Formation. c) the onset of foreland basin (Albian) with 1466 the Diamante Formation deposition (lower interval-107 Ma MDA) coeval with Las 1467 Chilcas Formation. d) the Late Cretaceous (Turonian?) reconstruction with the Diamante 1468 1469 Formation and BRCU deposition, with the presence of topographic barrier dividing the foreland basin (based on Mescua et al., 2013; Balgord and Carrapa, 2016; Muñoz et al., 1470 1471 2018; Gómez et al., 2019; Tapia et al., 2020; Boyce et al., 2020).

Figure 12. a) Regional DEM with the location of the samples from the Diamante
Formation (1 and 2; Balgord and Carrapa, 2016; Gómez et al., 2019) and the Neuquén
Group (3 to 6; Tunik et al., 2010; Di Giulio et al., 2012; Fennell et al., 2017; Borghi et
al., 2019) in the context of the Neuquén Basin. CF: Cordillera Frontal; CP: Cordillera

Principal; PCN: Precordillera Neuquina; CNP: Cordillera Nordpatagónica. b) 1476 Comparative probability density plots (PDPs) of detrital zircon U-Pb ages from the 1477 samples indicated in a. Zircons younger than 500 Ma were considered. Colored bars 1478 represent provenance signatures and the green vertical line marks the relative 1479 predominance of ca.107 Ma detrital zircons. 1480

Tables 1481

Table 1. Description of the samples analysed and the apatite and zircon fission track 1482

analytical data. 1483

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# Table 1

## Table 1

Description of the samples analysed and apatite and zircon fission track analytical data

Sample	Latitude	Longitude (W)	Stratigraphic	Stratigraphic	No. of	Spontaneous track density $\rho_s \ge 10^5$ tracks/cm <sup>2</sup> (Ns)	Induced track density $\rho_i \ge 10^6$ tracks/cm <sup>2</sup> (Ni)	Dosimeter track density $\rho_d \times 10^6$ $cm^2$ (Nd)	Age (Ma)	$\mathbf{P}(\mathbf{r}^2)$	$P_1$	$P_2$
Sample	(6)	$(\mathbf{w})$	um	Age	grams		(14)	(140)	± 10	I ( <i>x</i> )	(70)	(70)
VLP001	34°39'31.04"	69°41'33.68"	Diamante Fm.	Albian	42 (Ap)	1.601 (242)	1.91 (289)	7.47 (5000)	$111.9 \pm 13.6$	31.13	x	x
2119	34°39'29.19"	69°42'3.21"	Diamante Fm.	Turonian?	37 (Zr)	96.01 (4778)	11.07 (551)	3.33 (5000)	$184.0\pm13.1$	0.75	$161.1 \pm 16.4$ (71%)	$265.2 \pm 46.5 \\ (29\%)$

*Note.* Abbreviations are Ns, total number of spontaneous tracks; Ni and Nd, total numbers of induced and dosimeter tracks;  $P(x^2)$ ,  $x^2$  probability. No confined track lengths were measured.  $\zeta$  zeta value: 131.3 ± 5.1 (ZFT\_2119); 352.4 ± 22.9 (AFT\_VLP001). Counted by Dr. Arzadún G. in La.Te. Andes S.A. Etching conditions: 5.5 N (HNO<sub>3</sub>) for 20 s at 20°C (AFT\_VLP001); NaOH-KOH eutectic solution of 8 g of sodium hydroxide (NaOH) and 11.5 g of potassium hydroxide (KOH), melted at 210°C (ZFT\_2119). Dosimeter glasses: IRMM540 (AFT\_VLP001) and IRMM541 (ZFT\_2119). All samples were irradiated in the RA-3 Reactor in Centro Atómico Ezeiza (Buenos Aires, Argentina). See Appendix 3 for further information.






















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## HIGHLIGHTS

• A multiproxy provenance analysis was applied to the Diamante Formation in the northern Neuquén Basin.

• Apatite Fission Track analysis of the Diamante Formation reveals an Albian rapid cooling for its provenance-source.

• A lag time of 3 ma. for the Diamante Formation suggests a volcanic arc provenance during Albian times.

• Diamante Formation records the transition between the backarc and the foreland basin.

• Diamante Formation at 34-35°S was deposited in a foredeep depozone through a Distributive Fluvial System.

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## **Declaration of interests**

 $\boxtimes$  The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

