Triassic Amber of the Southern Alps (Italy)

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The Heiligkreuz-Santa Croce Formation (also known as Dürrenstein Formation, Upper Triassic) in the Dolomites contains one of the most ancient and substantial Triassic amber deposits in the world. The amber is found in sandstones and paleosols. It has an affinity to the conifer family Cheirolepidiaceae, and amber samples from the Julian and Carnic Alps (Southern Alps) also show an affinity to this family.

Physico-chemical investigations of the amber from the Dolomites by solid-state Fourier-transform infrared analysis (FTIR), nuclear magnetic resonance (NMR), pyrolysis-gas-chromatography/mass-spectrometry (pyr-GC/MS), thermogravimetry (TG), differential thermogravimetry (DTG), and automatized elemental analysis yielded a complete characterization of the amber, and allowed comparison with other ambers and younger resins (copals). FTIR revealed absorption bands typical of all fossil resins, and the spectrum region from 8–10 μm provided a fingerprint of the Triassic amber that differs from other known resins. The NMR spectrum also shows a typical pattern for fossil resins, but peculiar peak abundances permitted further characterization of the Triassic amber, both in the saturated (10–70 ppm) and unsaturated carbon region (100–160 ppm). The amber also lacks exomethylene resonances found in younger resins at 110 and 150 ppm. Pyrolysis-gas-chromatography/mass-spectrometry (pyr-GC/MS) experiments showed the amber was of class II, with some components of Class I. Thermogravimetric (TG) and differential thermogravimetric (DTG) analyses of combustion behavior of Triassic amber indicated a main exothermal event near 437°C, higher than that of other known resins. The elemental composition of Triassic amber is consistent with well-known constituents of natural resins, although the sulfur content was higher, likely due to high sulfur content in the embedding sediment. Triassic amber from the Dolomites appears to be a new kind of fossil resin with unique stratigraphical and physico-chemical characteristics.

INTRODUCTION

Triassic amber was first discovered in the early 19th century in the Dolomites (Southern Alps, Northern Italy; Fig. 1), and was mentioned in the famous posthumous work of Koken (1913) on the mollusc and vertebrate fauna of Heiligkreuz (Badia Valley, Dolomites, 46°37′N, 11°56′E). After these studies, this fossil resin was cited by Zardini (1973) and Wendt and Fürsich (1980), but no thorough study was performed before 1998 (Gianolla et al., 1998b). In 1996 and 1997, many samples of Triassic amber embedded in a sandstone matrix were collected in the Dolomites by researchers of the Museo delle Regole (Cortina d'Ampezzo, Italy) and by the authors. A reconnaissance field trip in 1999 led to discovery of fossil resin in a paleosol of the Heiligkreuz-Santa Croce Formation (also known as the Dürrenstein Formation) of a new and different shape, consisting of small, but perfectly preserved, drops. According to ammonoid and palynomorph data, all the above-mentioned Triassic fossil resins are from upper Julian–lowermost Tuvalian beds (Gianolla et al., 1998b; Roghi, 2004).

In Europe, scanty amounts of Upper Triassic amber have been reported from fluvial deposits of the Schilfsandstein Formation in Switzerland (Soom, 1984; Kelber, 1990), Raibler Schichten in Austria (Pichler, 1868), Lunzerschichten in Germany (Sigmund, 1937, quoted by Vávra, 1984), and Sándorhegy Formation of the Hungarian Transdanubian Range (Budai et al., 1999). In Arizona, Upper Triassic amber was found together with a rich palynological microflora in the Petrified Forest Member of the Chinle Formation (Litwin and Ash, 1991). It consisted of a discrete quantity of small and fractured amber; amber fragments also were recovered in various localities in the southwestern U.S.A. (Sidney Ash, pers. comm., 2002).

The abundance of excellently preserved little drops of fossil resin from the Heiligkreuz-Santa Croce Formation permitted characterization of the amber by means of various chemical and physical methods, such as infrared spectroscopy, nuclear magnetic resonance, pyrolysis- gas-chromatography/mass-spectrometry, and thermogravimetric analysis. Some preliminary data were published (Gianolla et al., 1998b; Ragazzi et al., 2003), and the study of perfectly preserved fossil microorganisms included in the amber is in progress, as well as of any affinity between the resin and fossil remains of the macroflora found in the sediment. Triassic amber, consisting of sparse material, also was found in the Julian Alps, Italy (Roghi et al., 2002) from a terrigenous-carbonate lithozone, attributed to the Rio del Lago Formation, which is older than the Dolomites amber described above.

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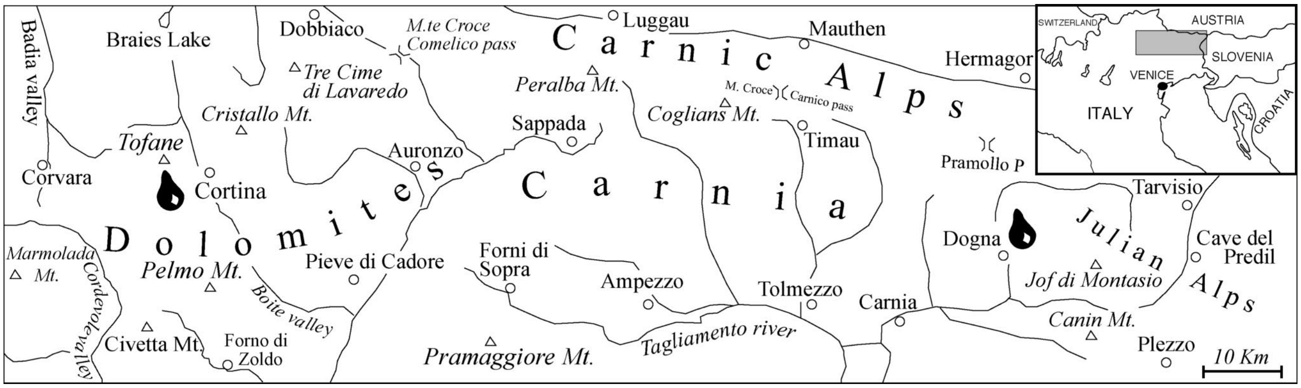

FIGURE 1—Original sites of discovery of amber (indicated by drops) in the Dolomites and Julian Alps.

The amber described in this paper represents one of the largest Triassic resin deposits known to date. The studied material is housed at the Department of Geology, Paleontology and Geophysics of the University of Padova, Italy (DGPGP in the text) and at the Museo delle Regole, Cortina d'Ampezzo, Italy (MRCA in the text).

GEOLOGICAL SETTING AND BIOSTRATIGRAPHY

Triassic fossil resins in the Southern Alps were found within at least two distinct stratigraphic units—one in the eastern Dolomites (Heiligkreuz-Santa Croce Formation) and the other in the Julian Alps (Rio del Lago Formation; Fig. 2). These units are situated in the eastern part of the Southern Alps, which were located in the westernmost part of the Tethyan margin of the Eurasian plate during Mesozoic time (Fig. 3).

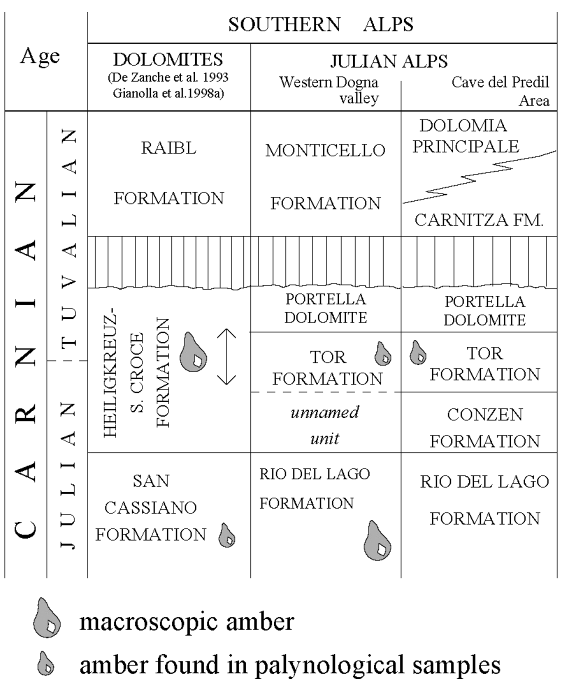

FIGURE 2—Correlations between Upper Triassic Formations from the Dolomites to the Julian Alps. The dimension of amber found in palynological samples is ≤200 μm. For aspects and dimensions of macroscopic amber specimens, refer to scale in Figure 5.

The Heiligkreuz-Santa Croce Formation recorded the flattening of complex topography of the lower Carnian and a period of anomalously abundant coarse siliciclastic supply (De Zanche et al., 1993; Gianolla et al., 1998a; Keim et al., 2001; Bosellini et al., 2003; Preto and Hinnov, 2003). It consists of mixed siliciclastic-carbonate successions, recording large shallow-water carbonate areas and zones subject to strong terrigenous input. The Heiligkreuz-Santa Croce Formation lies both on the shallow-water carbonate platforms of the Cassian Dolomite and on the basinal shales and limestones of the San Cassiano Formation. It is, in turn, unconformably overlain by the sabkha and paralic facies of the Raibl Formation. Its age is constrained to a narrow stratigraphic interval, close to the Julian–Tuvalian boundary (Carnian) by ammonoids and palynomorphs (Gianolla et al., 1998b; De Zanche et al., 2000; Roghi, 2004).

In the well-exposed outcrop below the Tofane Group (~300-m-thick section near Rifugio Dibona, 46°31′N, 12°04′E; Fig. 4), dolomitic limestone, dolomitic sandstone, and hybrid sandstone (sandstones with a high calcic content), with an increase upward of polygenic conglomerates and cross-bedded sandstone intercalated with paleosols and clay intervals, are present. The paleosols and clay beds that contain amber are very rich in plants, bivalves, and vertebrate remains, such as teeth and bones (Koken, 1913; Dalla Vecchia and Avanzini, 2002).

This part of the sequence ends with a carbonate mud-

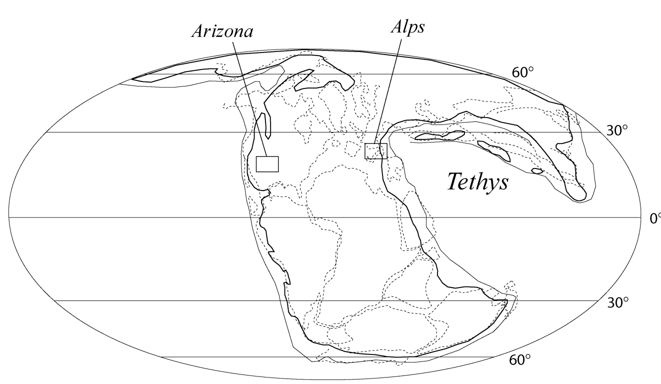

FIGURE 3—Map of Upper Carnian paleogeography showing the location of amber from east to west in the Alps (Italy, Germany, Austria, Switzerland) and in Arizona. Position of continents is taken from Owen (1983) and Scotese (2000).

FIGURE 4—Panoramic view of the Tofane mountains; A—San Cassiano Formation; B—Heiligkreuz-Santa Croce Formation; C—Raibl Formation; D—Dolomia Principale Formation. The amber site is within the Heiligkreuz-Santa Croce Formation (B).

stone and oolitic grainstone (a detailed lithofacies database is discussed in Preto and Hinnov, 2003). The last part of Heiligkreuz-Santa Croce Formation is composed of a more carbonate-rich lithozone, made of calcareous sandstones and massive dolostones. It forms an easily recognizable carbonate unit that can be traced for several kilometers, located below the Raibl Formation and Dolomia Principale (upper Tuvalian and Norian units; Fig. 4). The fossil resin was found in the lower part of the Heiligkreuz-Santa Croce Formation, which is more siliciclastic, well below the uppermost dolomite interval.

The palynofloras found in the strata with amber are characterized by typical Upper Triassic elements, including trilete levigate and ornamented spores, monosaccate and bisaccate pollen, and *Circumpolles*. Quantitative analysis of these associations indicates predominance of conifers (33–46%); pteridosperms (29%) and lycopsids (1%) are well represented (Table 1).

Stratigraphic research carried out in the Upper Triassic sequences of other Southern Alps localities allowed characterization of the paleobotanical features of the fossil resin. A few, small isolated granules of amber also were found in the Julian Alps (Dogna, 46°27′N, 13°19′E) in a lithozone attributed to the Rio del Lago Formation (Fig. 2; Preto et al., 2005), which consists mainly of intercalated marls and shales, suggesting a middle marine-platform environment. The Rio del Lago Formation is very rich in fossils such as ammonoids, pelecypods, crinoids, gastropods, cor-

TABLE 1—Palynological association, botanical affinity, and quantitative data from the amber-bearing beds of the Heiligkreuz-Santa Croce Formation (Dolomites). A general description of the palynological associations is given in Praehauser-Enzenberg (1970), Van der Eem (1983), Blendinger (1988), and Roghi (2004). Data are from samples SCS 16 and HEL 1, stored at DGPGP.

Species	Botanical affinity	%
Levigate and ornamented spores, genus *Calamospora*, *Todisporites*, *Concavisporites*, *Retusotriletes* and *Uvaesporites*	Lycopsids, Filicopsida and Sphenopsida	13
Spiritisporites spirabilis Scheuring, 1970	?Filicopsida	<1
Vallasporites ignacii Leschik *in* Kräusel and Leschik, 1956	Conifers	8
Enzonalasporites vigens Leschik *in* Kräusel and Leschik, 1956	Conifers	10
Patinasporites cf. *densus* (Leschik, 1956) Scheuring, 1970	Conifers	2
Patinasporites densus (Leschik, 1956) Scheuring, 1970	Conifers	1
Pseudoenzonalasporites summus Scheuring, 1970	Conifers	1
Samaropollenites speciosus Goubin, 1965	Conifers or Pteridosperms	1
Ovalipollis pseudoalatus (Thiergart, 1949) Schuurman, 1976	?Cycadales, ?Pteridosperms, ?Conifers	8
Lunatisporites acutus Leschik *in* Kräusel and Leschik, 1956	Pteridosperms (Peltaspermales), ?Conifers, ?Podocarpaceae	4
Infernopollenites parvus Scheuring, 1970	Pteridosperms (Peltaspermales), ?Conifers	<1
Triadispora spp.	Conifers (Voltziales)	8
Lueckisporites sp.	Conifers (Majonicaceae)	3
alete bisaccate	Pteridosperms (Peltaspermales), Conifers	29
Duplicisporites continuus Praehauser-Enzenberg, 1970	Conifers (Cheirolepidiaceae)	1
Paracirculina maljawkinae Klaus, 1960	Conifers (Cheirolepidiaceae)	3
Duplicisporites verrucosus (Leschik, 1956) Scheuring, 1970	Conifers (Cheirolepidiaceae)	1
Duplicisporites granulatus (Leschik, 1956) Scheuring, 1970	Conifers (Cheirolepidiaceae)	6
Camerosporites secatus Leschik *in* Kräusel and Leschik, 1956	Conifers (Cheirolepidiaceae), ?Pteridosperms	<1

TABLE 2—Palynological association, botanical affinity, and quantitative data from the amber-bearing beds of the Rio del Lago Formation in the Dogna locality. A general description of the palynological associations is given by Roghi (2004). Data are from samples CHUT 1, stored at DGPGP.

Species	Botanical affinity	%
Deltoidospora mesozoicus (Thiergart, 1949) Schuurman, 1977	Ferns (Marattiales)	5
Concavisporites sp.	Ferns (Filicales)	3
Retusotriletes mesozoicus Klaus, 1960	Lycopsids (Drepanophycales)	<1
Uvaesporites spp.	Lycopsids (Selaginellales)	3
Uvaesporites glomeratus Döring, 1965	Lycopsids (Selaginellales)	<1
Lycopodiacidites keupperi, Klaus, 1960	Lycopsids (Selaginellales), Ferns (Ophioglossales)	<1
Simplicisporites sp.	Undetermined affinity	<1
Kyrtomisporis ervii Van der Eem, 1983	Undetermined affinity	<1
Concentricisporites bianulatus (Neves 1961) Antonescu, 1970	Undetermined affinity	<1
alete bisaccate	Pteridosperms (Peltaspermales)	20
Parcisporites sp.	Conifers	20
Microcachrydites parvus Passoni, 1999	Conifers	3
Triadispora sp.	Conifers (Voltziales)	<1
Lueckisporites sp.	Conifers (Majonicaceae)	9
Ovalipollis pseudoalatus (Thiergart, 1949) Schuurman, 1976	?Cycadales, ?Pteridosperms, ?Conifers	<1
Praecirculina granifer Leschik, 1956	Conifers (Cheirolepidiaceae)	6
Camerosporites secatus Leschik, 1956	Conifers (Cheirolepidiaceae), ?Pteridosperms	<1
Duplicisporites verrucosus (Leschik, 1956) Scheuring, 1978	Conifers (Cheirolepidiaceae)	11
Duplicisporites sp.	Conifers (Cheirolepidiaceae)	9
algal spores		5

als, brachiopods, and plants, including conifer, pteridosperm, and horsetail remains. Palynological analysis from the same plant beds (Table 2) indicates a predominance of conifers (58%) and presence of pteridosperms (20%), ferns (8%), and lycopsids (<6%).

On the basis of ammonoid and palynological data, the Heiligkreuz-Santa Croce Formation belongs to the Austriacum and Dilleri ammonoid zone (upper Julian–lower Tuvalian; Gianolla et al., 1998b; Roghi, 2004). Based on the palynological association (Table 1), the Rio del Lago Formation in the Julian Alps belongs to Aonoides Zone (Julian; Roghi, 2004; Preto et al., 2005). The presence of *Kyrtomisporis ervi* and *Concentricisporites* cf. *bianulatus*, typical lower Julian forms (Van der Eem, 1983; Broglio Loriga et al., 1999), confirms an older age for the amber found in the Rio del Lago Formation compared to that found in the Heiligkreuz-Santa Croce Formation (Fig. 2).

TAPHONOMY OF TRIASSIC AMBER

Dolomites Amber

Two main sources of amber in the Dolomites can be distinguished. The first type was found mainly as fractured samples embedded in sandstone and hybrid-sandstone rocks, indicating that after production, the resin was transported. The second type consists of abundant, isolated, and well-preserved autochthonous drops, concentrated in paleosol deposits (Fig. 5I). The total amount of resin found (~500 g) consists of an estimated number of at least 50,000 drops; the resin collection from the paleosol was about 2–5% (in volume) of the sediment. The amber specimens found in sandstone range from circular to ovoid shapes and measure 1–2 mm in diameter and 2–10 mm in length, although a sample 30 mm in diameter was found. The amber found in the paleosol from the middle-upper part of the Heiligkreuz-Santa Croce Formation occurs as isolated specimens with a size range of 2–6 mm (most often 3 mm in diameter) and an evident droplet shape (Fig. 5A–H), often with a little stem (Fig. 5B, C, E, G). Also, bilobate drops with a second streaming down the first one were found (Fig. 5H).

The surface of the amber drops often shows desiccation fractures, indicating exposure to the air or sun (Fig. 5A, D, G; Fig. 6). Abundant plant fragments, cuticles, and amber in the paleosols and in the nearby layers suggest a rich vegetation in a coastal environment. Plant remains include horsetails (Fig. 7F), conifer twigs (Fig. 7D), and wood fragments (Fig. 7G). Preliminary analyses on cuticle and macrofossil fragments of some conifer shoots found in the amber-bearing paleosols indicate that the resin originated from representatives of the family Cheirolepidiaceae (J. van Konijnenburg-van Cittert, pers. comm., 2002; Roghi et al., 2002).

Julian Alps Amber

In the Rio del Lago Formation (Julian Alps), well-preserved conifer and pteridosperm remains were found (Fig. 7A), and fossil resin is directly associated with the conifers (Fig. 7B). Observation of plant macroremains under UV light (365 nm) showed fossil resin-filled spaces between leaves (Fig. 7C). Preliminary cuticle analyses of these samples indicate the same Cheirolepidiaceous affinity as the cuticles found in the Heiligkreuz-Santa Croce Formation.

PHYSICO-CHEMICAL CHARACTERISTICS OF TRIASSIC AMBER

Triassic amber from the Dolomites displays colors ranging from yellow to reddish-brown, with resinous brilliance and conchoidal fracture. The hardness is 2–3 (Mohs scale), the specific gravity is about 1.08, and it burns if exposed to flame, producing a resinous odor. The amber apparently is not soluble after the application of a drop of ethanol, dieth-

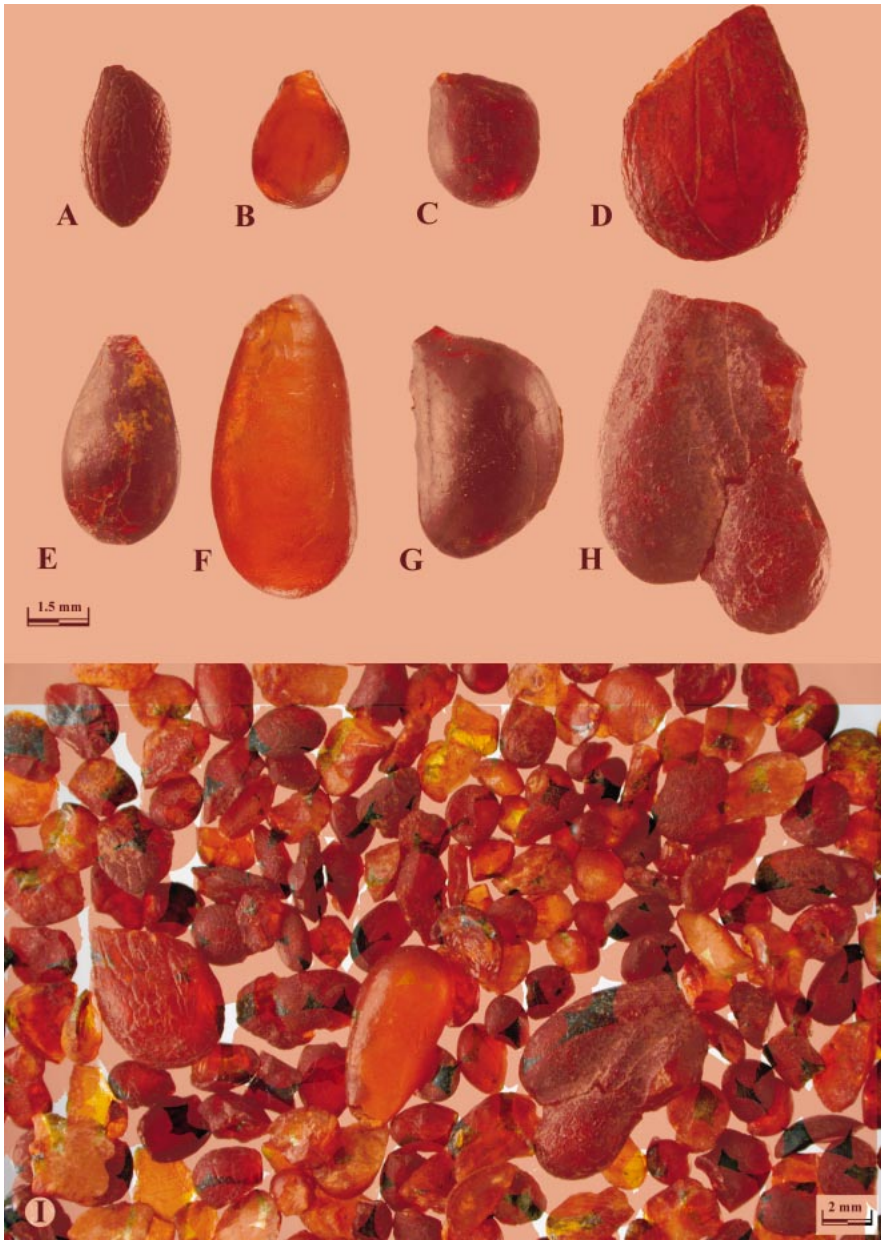

FIGURE 5—(A–I) Typical shapes of the Triassic amber found in paleosol from the Heiligkreuz-Santa Croce Formation in the Rifugio Dibona section. Samples are stored at DGPGP, no. NRE 103.

yl ether, or acetone to the surface for 30 seconds (this being a method of distinguishing between amber and other less-mature resins; see Currie, 1997). However, if the amber is left in the solvent for a longer time (up to 24 hours), it partially dissolves. An efficacious solvent was a mixture of diethyl ether/dichloromethane (1:1). When the solvent was removed, the remaining undissolved material was very brittle and crumbled easily. Another solvent able to dissolve the fossil resin is a mixture of acetone/oil of turpentine (4:1, according to the method of De Franceschi et al., 2000), and after 2–3 days, the fossil resin became soft and gummy.

The abundant availability of Triassic amber in the paleosol of the Heiligkreuz-Santa Croce Formation from the Dibona section permitted a more complete physico-chemical characterization, as reported below.

Infrared Spectroscopy

Solid-state Fourier-transform infrared analysis (FTIR) was performed on powdered samples after inclusion in potassium bromide pellets. A Perkin Elmer 1600 Series FTIR Spectrophotometer was used in the range 2.5–15.5 μm (4000–645 cm^{-1}). The FTIR spectrum of Triassic amber (Fig. 8) is typical of fossil resins where hundreds, if not thousands, of different compounds mixed together give rise to a mediated signal. It is possible to recognize the strong absorption band near 3.4 μm due to the presence of

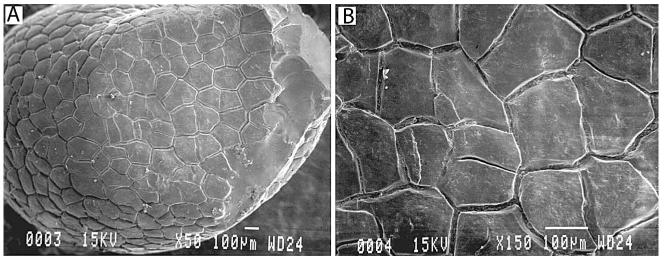

FIGURE 6—Scanning Electron Microscopy (SEM) images of the surface of a specimen of Triassic amber from Heiligkreuz-Santa Croce Formation in the Rifugio Dibona section. (A) Amber surface. (B) Close-up of surface shown in (A).

CH aliphatic groups, common in fossil resins, due to the stretching of aliphatic carbon-hydrogen bonds (Langenheim and Beck, 1968; Broughton, 1974; Vávra and Vycudilik, 1976). Bending motion of the same bonds, representing CH_2 and CH_3 functional groups, also produces absorption at 6.8 μm and 7.25 μm (Langenheim and Beck, 1968; Broughton, 1974; Vávra and Vycudilik, 1976). The band near 5.8 μm, called the carbonyl band (Langenheim and Beck, 1968), is caused by stretching motions of carbon-oxygen double bonds mainly due to carboxylic acid derivatives or carbonyl compounds. The presence of these carbon-based compounds is confirmed by the peak at 175–180 ppm in the ^{13}C-NMR spectrum. The presence of a shoulder at higher energy and a clean peak at lower energy may be diagnostic for this amber.

In the so-called fingerprint region between 8 and 10 μm, the absorbance due to carbon-oxygen single bonds (Langenheim and Beck, 1968; Vávra and Vycudilik, 1976) is of particular diagnostic value. The Triassic amber specimen shows unique patterns if compared to the spectra of other resins; the most typical band is at 8 μm and another at 8.5 μm. The Baltic shoulder in this region of the spectrum (Beck et al., 1964; Beck, 1986) is not evident, indicating a different paleobotanical origin and/or maturation in comparison with Baltic amber. Absorption above 10 μm is more difficult to assign, and must be used as fingerprints (Langenheim and Beck, 1968). The Triassic amber spectrum shows a weak band at 10.2 μm and another, weaker, but broader band, near 12.5 μm. The band described near 11–11.3 μm, due to out-of-plane bending of the two hydrogen atoms of a terminal methylene group (Langenheim and Beck, 1965, 1968; Broughton, 1974), is not present here, suggesting that the resin never had these functional groups, or, alternatively, that they were altered by prolonged exposure or maturation.

Carbon-13 Nuclear Magnetic Resonance

Carbon-13 nuclear magnetic resonance (^{13}C-NMR) spectroscopy is accepted as a useful technique to distinguish among fossil resins (Lambert and Frye, 1982; Grimalt et al., 1987; Lambert et al., 1988, 1996; Martinez-Richa et al., 2000). In order to characterize Triassic amber from the Dolomites by means of this technique, a 0.3-g powdered sample, obtained from a pool of 5 g of fossil resin consisting of several droplets collected at the Dibona section paleosol level, was used. A Bruker AMX300 spectrometer operating at 75.48 MHz on the ^{13}C resonance frequency was used for the CPMAS ^{13}C-NMR experiments. Cross-polarization with magic-angle spinning (CPMAS) was applied at 5 kHz. A contact time of 1 millisecond was used, while a pulse delay of 1 second was chosen. A rotor filled with sample was allowed to spin before each experiment in order to stabilize the sample packing and improve the field homogenization. Chemical shifts are given in parts per million (ppm).

The ^{13}C-NMR spectrum of Triassic amber (Fig. 9) shows the typical pattern of fossil resins (e.g., Lambert and Frye, 1982; Lambert et al., 1985, 1988, 1996), but with peculiarity of peak abundance, as far as it is permitted by the carbon integral. The saturated carbon region from 10–70 ppm is broad, with two main peaks at 14.26 and 30.61 ppm; an additional smaller peak is observed at 36.83 ppm. Methylene groups with adjacent branching that resonate near 30 ppm (Lambert et al., 1988) also were found in Baltic amber (Lambert and Frye, 1982; Lambert et al., 1985, 1988).

The unsaturated carbon region from 100–160 ppm (due to alkenic, cycloalkenic, and aromatic carbons; Lambert and Frye, 1982; Lambert et al., 1985) shows a broad peak at 125 ppm, ranging from 100 to 140 ppm (Fig. 9). This result is similar to that found in several fossil resins from the Cretaceous (Grimalt et al., 1987; Lambert et al., 1990, 1996). However, the saturated resonances of this Triassic resin contain two major peaks at about 14 and 30 ppm (the largest) that are different from those of Cretaceous amber specimens (examined by Lambert et al., 1996), which exhibit two major peaks at 25 and 40 ppm (the largest). Lambert et al. (1996) also showed the spectrum of an amber from Schliersee, previously attributed to the Triassic (Poinar, 1992; Poinar et al., 1993; Poinar and Poinar, 1994), but now attributed to Cretaceous (Schmidt et al., 2001). In this fossil resin, the saturated region is broader, and shows two peaks at 25 and 40 ppm (the largest).

The carbonyl region (170–190 ppm; Lambert and Frye, 1982; Lambert et al., 1985) exhibits a small peak at 179 ppm. The spectrum lacks exomethylene resonances ($C{=}CH_2$), which commonly are found at about 110 and 150 ppm in younger resins (Lambert et al., 1996), and are considered very sensitive to diagenesis and degradation (Lambert and Frye, 1982; Lambert et al., 1985, 1990, 1996). This datum is in agreement with the FTIR spectrum discussed above.

Differences in NMR spectra can result from several causes, including paleobotanical origin, age, maturation, and conditions of exposure (Lambert et al., 1985). The NMR spectrum of Triassic amber from the Dolomites is compatible with an old age, and a history of high-pressure exposure in the embedding sediment.

Pyrolysis-Gas-Chromatography/Mass-Spectrometry

In order to establish the class of the resin under investigation according to the classification proposed by Anderson et al. (1992), some pyrolysis-gas-chromatography/mass-spectrometry (pyr-GC/MS) experiments were performed.

The pyrolysis chamber (250°C) was linked directly to the injector of the GC/MS system. Pyrolysis-GC/MS experiments were performed using a FISON GC 8000/ MD 800 instrument operating in electron-impact conditions (70 eV, 200 A) and in full-scan mode (m/z 50–600). The GC col-

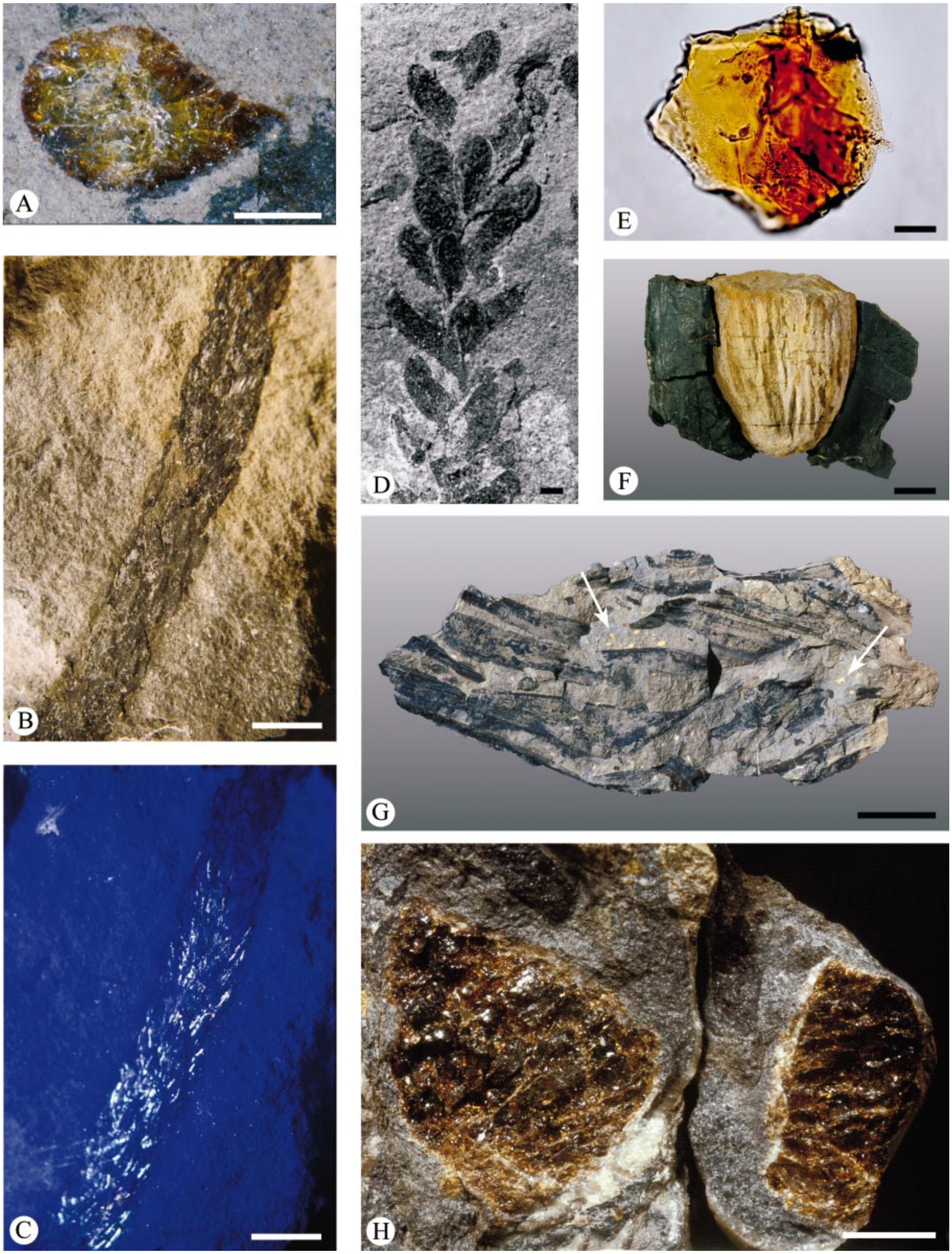

FIGURE 7—Amber and plant remains from the Upper Triassic deposits in the Southern Alps (Italy). Samples A–G are stored at the DGPGP, sample H at MRCA. (A) Amber drop from Julian Alps, CHIUT 16; scale bar = 1 mm. (B) Conifer fragment from Julian Alps, CHIUT 11; scale bar = 5 mm. (C) Conifer fragment shown in B under UV light (365 nm); white material is the fluorescent fossil resin filled between leaves; scale bar = 5 mm. (D) Conifer fragment from Lastoni di Formin, Heiligkreuz-Santa Croce Formation, LF 1; scale bar = 1 mm. (E) Amber fragment from palynological material, Dogna Valley, Tor Formation, TUB8; scale bar = 0.3 mm. (F) Plant remains belonging to an Equisetalean, SMA 12; scale bar = 10 mm. (G) Sandstone with plant fragment and little yellow amber drops (arrows) from Heiligkreuz-Santa Croce Formation, RUM 151; scale bar = 5 cm. (H) Amber from Heiligkreuz-Santa Croce Formation, MRCA 19371; scale bar = 0.8 cm.

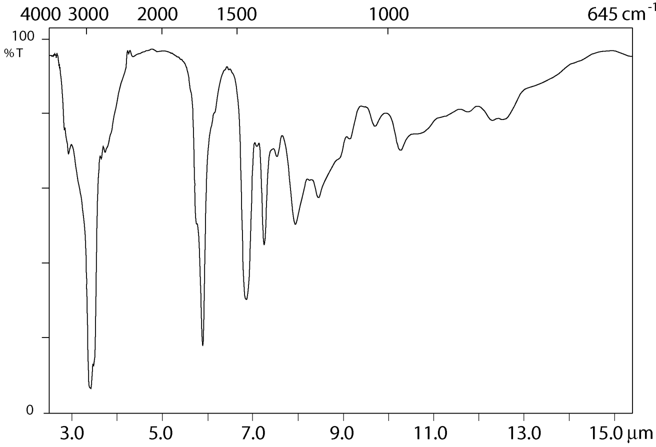

FIGURE 8—Solid-state FTIR spectrum of Triassic amber from the Dolomites.

umn was a DB-5ms (0.25 mm i.d., film thickness 0.25 µm). The temperature program for the GC oven was: isothermal at 40°C for 2 minutes; temperature programmed at 4°C/minute to 300°C; isothermal at 300°C for 2 minutes. About 0.5 mg of finely powdered sample was deposited on a 1.5-mm i.d. quartz tube, and inserted into the resistor of CDS (Chemical Data System) 100 pyroprobe. The pyrolysis conditions were: pyrolysis chamber at 250°C; pyrolysis final temperature at 480°C kept constant for 5 seconds; temperature ramp 20°C/millisecond.

A typical ion chromatogram obtained by this approach is shown in Figure 10. For the most abundant peaks, a comparison of the related mass spectra with library data was performed, and most of them were structurally identified. Classification of resinites by Anderson et al. (1992) gave some molecular fingerprinting for resin of different origins in terms of either space (geographical region) or time (geological age). Four main classes were defined by Anderson et al. (1992), each of which was characterized by the presence of well-defined, specific pyrolysis products.

Class III resins were excluded immediately due to the absence of peaks in the chromatogram representing styrene monomer, dimer, and trimer. Interestingly, most of the chromatographic peaks can be assigned to aromatic compounds, in particular substituted benzenes, naphtalenes, and phenantrenes (see peaks indicated in Fig. 10). These results strongly support assignment of the resin to class II. In addition, some data indicate assignment of the resin to class I, in particular, the peak at 18.78 min (see * in Fig. 10), which was assigned to L-camphor, a component generally present in class I resins.

Thermogravimetric (TG) and Differential Thermogravimetric (DTG) Analyses

By using a prototypal instrument (I.G.G.-C.N.R., Padua, Italy), denominated "Le Chatelier," consisting of a type S (Pt-10% Rh/Pt) thermocouple placed into an electric furnace and interfaced to a Mettler Toledo AB 104 balance, Triassic amber shows a TG combustion profile that begins after 200°C, with total combustion occurring before 600°C. Under differential thermogravimetric (DTG) analysis of combustion behavior, Triassic amber displays a main exo-

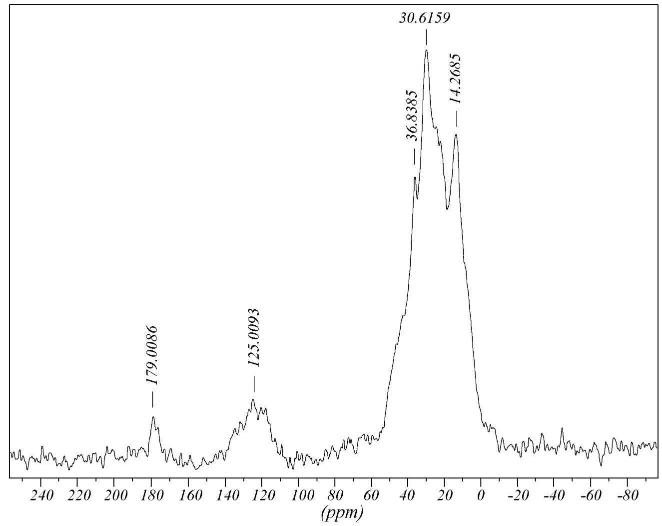

FIGURE 9—Solid-state carbon-13 nuclear magnetic resonance (^{13}C-NMR) of Triassic amber from the Dolomites.

thermal event as a consequence of a maximal rate of weight loss near 437°C (Fig. 11).

This datum is in accordance with that used to demonstrate that the age of the fossil resins can be correlated to the thermal behavior provided by TG and DTG analyses

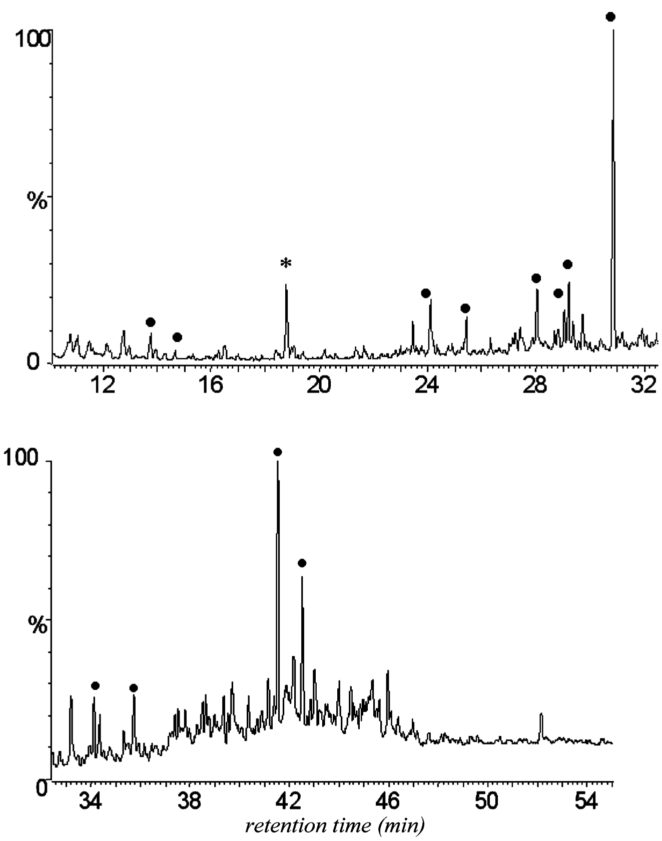

FIGURE 10—Typical ion chromatogram of Triassic amber, obtained by pyrolysis-gas-chromatography/mass-spectrometry. Dots indicate aromatic compounds, in particular, substituted benzenes, naphtalenes, and phenantrenes. The peak at 18.78 min (*) was assigned to L-camphor.

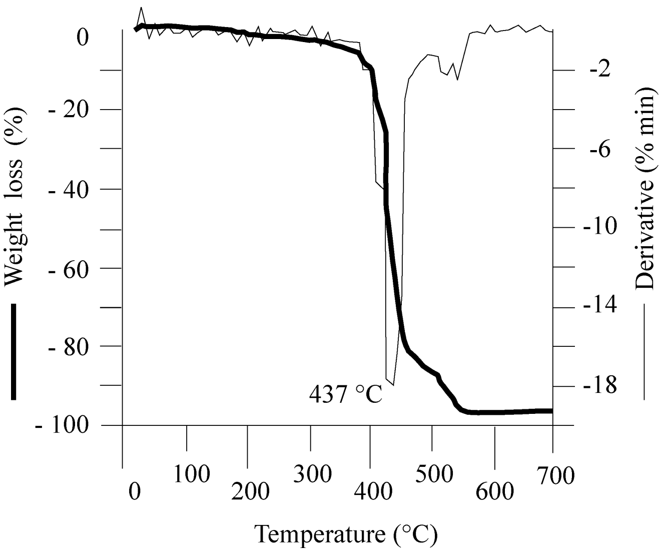

FIGURE 11—Thermogravimetric (thick line) and differential thermogravimetric (thin line) profile of Triassic amber from the Dolomites.

(Ragazzi et al., 2003). In fact, comparing the thermal behavior of Triassic amber to that of other resins, a linear correlation was found (Ragazzi et al., 2003), suggesting an increase in the main exothermal event according to the age of the resin. Table 3 summarizes the thermal analysis data (MTEP: main thermal event point) obtained with fossil resins from different locations and ages (from Ragazzi et al., 2003).

Elemental Analysis

A CE-Instruments EA 1110 Automatic Elemental Analyzer, equipped with an AS 200 autosampler and a Mettler Toledo AT21 Comparator, was used. The instrument is a simultaneous carbon-hydrogen-nitrogen and sulfur analyzer, based on dynamic-flash combustion and GC separation (He carrier gas), followed by thermal-conductivity detectors (TCD). The results obtained for two samples of Triassic amber (red or yellow color) are reported in Table 4.

DISCUSSION

The present work aimed to study the stratigraphy and physical chemistry of Triassic amber from the Dolomites. Based on palynological associations, the flora consists of lycopsids, filicopsids, sphenopsids, pteridosperms, and conifers. Spores are well represented, and quantitative analysis of the microflora indicated a predominance of conifers; among them, Voltziales, Majonicaceae, and Cheirolepidiaceae were found. Although the relative abundance of taxa within the sporomorphs is not necessarily a good indicator of botanical origin of the resin, it strongly suggests that the source of the amber belonged to one of these groups.

In the recent years, knowledge about Triassic macrofloras in the Southern Alps has increased remarkably. Very diverse floras have been collected in the Anisian (Broglio Loriga et al., 2002), Ladinian (Wachtler and van Konijnenburg-van Cittert, 2000), and Carnian (Passoni and van Konijnenburg-van Cittert, 2003). Despite the large number of fossil-plant specimens, no resin was found. However, the ages of the latter macrofloras and of the Dolomites amber are different. Similarly, based on the palynological assemblage (Passoni and van Konijnenburg-van Cittert, 2003), the Carnian age for plants from Monte Pora appears to be older than the age of the amber from the Heiligkreuz-Santa Croce Formation.

The formation of the Dolomites amber cannot be a single, fortuitous event. To explain the considerable amount of amber, three hypotheses can be suggested: (1) a massive production of resin was the consequence of a particular event that stressed the plants; (2) a new conifer taxon evolved that produced the resin; and (3) a relative sea-level rise covered the land where the resin-producing plants lived. The three hypotheses also may coexist.

To find fossil resin close to the Julian–Tuvalian boundary in both Europe and North America might indicate a large-scale event (Fig. 3). Quantitative palynological study around this boundary in the Julian Alps (Cave del Predil area) suggests climate change during this time. A decrease in typical xerophytic circumpollen forms, and the concurrent increase in azonotrilete forms (laevigate and apiculate spores), typical of hygrophytic assemblages, might indicate wetter paleoenvironmental conditions for

TABLE 3—Thermal-analysis data obtained with fossil resins from different locations and ages. MTEP = main thermal event point at DTG analysis (from Ragazzi et al., 2003).

Sample	Age	MTEP (°C)
Picea abies resin	Present-day	358
Madagascar copal	Holocene/Recent	384
Colombia copal	Pliocene/Recent	400
Blue Dominican amber	Oligocene–Miocene	400
Dominican amber	Oligocene–Miocene	400
Mexican amber	Oligocene–Miocene	441
Simetite	?Oligocene–?Miocene	405
Lessini amber	Lower/Middle Eocene	391
Baltic amber	Eocene	402
Cedar Lake amber	Upper Cretaceous	409
New Jersey amber	Upper Cretaceous	421
Red Triassic amber from the Dolomites	Upper Triassic	437
Yellow Triassic amber from the Dolomites	Upper Triassic	443

TABLE 4—Elemental analysis of two samples (typical red and yellow colors) of Triassic amber (from Ragazzi et al., 2003).

Sample	N (%)	C (%)	H (%)	S (%)	O and other elements in trace (%)
Red Triassic amber from Dolomites	0.050	81.27	10.35	1.74	6.59
Yellow Triassic amber from Dolomites	0.038	80.87	10.43	1.47	7.19

this part of the sequence (Roghi, 2001, 2004). Indication of a humid event in the Late Triassic has been suggested for other parts of Europe (Simms and Ruffel, 1989; Simms et al., 1995; Preto and Gianolla, 2001), and therefore, typical xerophytic conifers probably were stressed by this climatic perturbation.

Because the Upper Triassic is an important time in conifer evolution (Miller, 1977, 1982), the hypothesis that Triassic amber may originate from some new conifer group is possible. Additional support comes from the Dogna fossil flora and amber (Carnic Alps), where some plant macroremains are associated with the resin, suggesting that amber-producing species are related mainly to the family Cheirolepidiaceae, a group of typical Jurassic and Cretaceous conifers. The reported Carnian finding herein of macroremains attributed to the family Cheirolepidiaceae represents one of the oldest records of this taxon, and adds more evidence to the evolutionary history of these conifers during the Upper Triassic. Moreover, the younger Heiligkreuz-Santa Croce Formation amber deposit yielded associated cuticle specimens, the analysis of which showed evidence of the same cheirolepidiaceous affinity (Roghi et al., 2002).

A further cause of amber deposition is suggested by sequence stratigraphic analysis. Deposition of the main fossil-resin levels of the Heiligkreuz-Santa Croce Formation corresponds to transgressions (parasequences within the TST of CAR 3 depositional sequence in De Zanche et al., 1993). Sea-level rises could have covered the land near the coasts where the plants probably lived, therefore creating stressful conditions for the trees with optimal conditions for amber deposition and preservation. This hypothesis also is supported by the plants with cheirolepidiaceous affinity that were found in the coastal marshes or peat-bog environments represented in the Heiligkreuz-Santa Croce Formation, and are considered to be a typical coastal flora (Francis, 1983).

In the Southern Alps, at least two Carnian-age levels with fossil resin have been found. A recent discovery of lower Julian amber in palynological samples from the Prati di Stuores section (Badia Valley Dolomites, Broglio Loriga et al., 1999; Fig. 2) also suggests a correlation with the Carnic Alps amber deposits.

The physico-chemical characterization of Triassic amber was obtained by means of infrared spectrophotometry (FTIR), nuclear magnetic resonance (NMR), pyrolysis-gas-chromatography/mass-spectrometry (pyr-GC/MS), thermogravimetry (TG), differential thermogravimetry (DTG), and automatized elemental analysis. The elemental composition of Triassic amber is consistent with the well-known constituents of natural resins (C, H, O, N), although the sulfur content was higher—probably due to high sulfur content in the embedding sediment (Ragazzi et al., 2003).

Solid-state Fourier-transform infrared analysis (FTIR) permitted recognition of absorption bands typical of all fossil resins (see, e.g., a comparison in Beck et al., 1964; Langenheim and Beck, 1965, 1968; Broughton, 1974; Beck, 1986; Anderson and Crelling, 1995; Beck, 1999). The spectrum region from 8–10 μm provided a fingerprint of the amber that appeared to differ from that of other known resins. The particular pattern of the spectrum (e.g., absence of a band near 11–11.3 μm) suggests that Triassic amber never had these functional groups, or, alternatively, that they were altered by prolonged exposure during amberization (the process of maturation of fossil resins).

^{13}C-NMR spectroscopy suggests a complex history of maturation as well. The NMR spectrum shows a typical pattern for fossil resins, but peculiar peak abundances here permitted further characterization of the Triassic amber, both in the saturated (10–70 ppm) and unsaturated carbon region (100–160 ppm). In agreement with the FTIR spectra, the carbonyl region (170–190 ppm) presents a small peak at 179 ppm. The resin lacks exomethylene resonances at 110 and 150 ppm, which, in contrast, can be found in younger resins (Lambert et al., 1996).

Pyrolysis-gas-chromatography/mass-spectrometry (pyr-GC/MS) indicated the presence of several peaks in the ion chromatogram. The specific assignment to one of the classes of resinites proposed by Anderson et al. (1992) is not straightforward due to the presence of compounds that are found in more than one class. The absence of styrene monomer, dimer, and trimer suggests that class III should be excluded. In contrast, the presence of aromatic compounds supports the resin belonging to class II, and a few data suggest affinity for resins of class I. Therefore, due to its old geological age, the Triassic fossil resin includes a mixture of compounds, which, with further maturation, became differentiated from younger resins.

Thermogravimetric (TG) and differential thermogravimetric (DTG) analyses of combustion behavior of Triassic amber indicated a main exothermal event, as a consequence of maximal rate of weight loss, located near 437°C (Fig. 10). This temperature was higher than that of other known resins (Ragazzi et al., 2003). In addition, this analysis characterized this old fossil resin.

Triassic amber from the Dolomites appears to be a new kind of fossil resin with unique stratigraphical and physico-chemical characteristics; the data obtained suggest that it may be a source of further interesting information on Triassic climate and paleoenvironment.

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